

# THE ANALYST

Volume X

1885

W. Heffer & Sons Ltd.  
Cambridge, England  
Johnson Reprint Corporation  
New York 3, New York

6231

*Reprinted photographically in Great Britain by  
Lowe & Brydone (Printers) Ltd. for W. Heffer & Sons Ltd., Cambridge*

*Distributor for North Central and South America  
Hawaii and the Philippines :  
Johnson Reprint Corporation  
New York 3, New York*

The original numbers of *The Analyst* from which this reprint was produced were kindly loaned by T. McLachlan, Esq., D.C.M., A.C.G.F.C., F.R.I.C.

# The Analyst,

INCLUDING THE PROCEEDINGS OF

THE "SOCIETY OF PUBLIC ANALYSTS."

A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT OF THE  
ANALYSIS OF FOOD AND DRUGS, AND OF GENERAL  
ANALYTICAL AND MICROSCOPICAL RESEARCH.

---

EDITED BY

JOHN MUTER, M.A., Ph.D., F.I.C., F.C.S., &c.,

*Late President of the Society of Public Analysts; Author of several Text-Books on  
Pharmaceutical Chemistry, Analytical Chemistry and Materia Medica; Public  
Analyst for Lambeth, Wandsworth, Southwark, Bermondsey, Rotherhithe, Newington,  
Tenterden, &c.; Analyst to the Metropolitan Asylums Board.*

VOL. X.

1885.

---

LONDON:

PUBLISHED FOR THE PROPRIETOR, BY MESSRS. BAILLIERE, TINDALL & COX,  
KING WILLIAM STREET, STRAND, W.C.

# INDEX.

	PAGE	PAGE
<b>A</b>		
Acid, Phosphoric, Reverted, Estimation of, MOHR .. .. .	16	
Acidity in Sour Milk and Koumiss, Estimation of, H. W. WILEY .. .. .	204	
Action of Tannin on Fehling's Solution, SONNENSCHNEIN .. .. .	205	
Active Principle of Goa Powder, PETIT .. .. .	207	
ADAMS, M.A., on a New Method for the Analysis of Milk .. .. .	46	
"    "    the Treatment of Sour Milk intended for Analysis .. .. .	99	
Address, Inaugural, by Dr. HILL, President .. .. .	42	
Admixtures of Malt-Extract with certain Fixed Oils, E. GODWIN CLAYTON .. .. .	140	
Adulterated Pepper, Note on a Sample of, Dr. A. BOSTOCK HILL .. .. .	122	
"    Sample of Tea, Note on a Sample of, Dr. A. BOSTOCK HILL .. .. .	71	
Adulteration Acts in Dublin, Operation of the, SIR C. CAMERON .. .. .	175	
"    of Argol, ZIEREK .. .. .	189	
"    of Carbon Bisulphide, M. VAN ENGELN .. .. .	189	
"    of Menthol by Thymol, Detection of, BERNBECK .. .. .	37	
"    of Olive Oil, HIEPE .. .. .	205	
"    of Saffron, Prof. J. M. MAISCH .. .. .	200	
Æsculine, New Colour Test for, RABY .. .. .	208	
Albumen, New Method for Separating Globuline from, WICHAIRLOW .. .. .	211	
"    New Reaction for, D. OXENFELD .. .. .	167	
"    Serum, Behaviour of, in the presence of Acids and Neutral Salts, E. JOHANNSEN .. .. .	184	
Albuminous Compounds in Milk, J. SEBELIEN .. .. .	184	
Alkaloids in old Flour, BALLAND .. .. .	228	
"    and Glucosides, Reaction of, SERENA .. .. .	149	
"    Separation of, from Human Remains, H. BECKURTS .. .. .	191	
Alkanet in Wine, Detection of, H. HEERZ .. .. .	146	
ALLEN, A. H. Note on the Optical Estimation of Milk-Sugar .. .. .	72	
"    on Reichert's Method of Examining Butter Fat .. .. .	103	
Aloes in Mixtures, Quantitative Determination of, H. HAGER .. .. .	95	
"    New Test for, CRIPPS & DYMOND .. .. .	60	
Aloin, J. DIETRICH, .. .. .	186	
Alumina and Iron in the presence of Phosphates, Determination of, M. KRETZSCHMAR .. .. .	151	
Alumina and Iron in the presence of Phosphoric Acid, Determination of, R. LUCION .. .. .	192	
Alumina and Iron, Separation of, ILINSKY and KNORKE .. .. .	232	
American Society of Public Analysts:—		
Report of April Meeting .. .. .	105	
"    May .. .. .	123	
"    June .. .. .	143	
"    October .. .. .	223	
Ammonium Citrate in the Analysis of Precipitated Phosphates, H. H. B. SHEPHERD .. .. .	161	
Analysts, Proceedings of the Society of Public, 1, 21, 41, 61, 81, 99, 117, 135, 155, 175, 195, 215 .. .. .	161	
Antipyrin, Detection and Properties of, O. SCHWEISSINGER .. .. .	188	
Apparatus for Fat Extraction, F. WALLIS STODDART .. .. .	108	
"    for Milk Analysis, W. JOHNSTONE .. .. .	81	
"    for filtering small Quantities of Liquid, WILEY .. .. .	204	
"    for the Determination of Starch, R. REMPEL .. .. .	94	
"    New Extraction, T. WEYL .. .. .	153	
Appointments .. .. .	40	
Arata, P.N., on the Detection of Colouring Matters in Wine .. .. .	186	
Argol, Adulteration of, ZIEREK .. .. .	189	
Artificial Colours in Butter, &c., Method of Separating and Determining, E. W. MARTIN .. .. .	163	
"    Honey, H. HAGER, .. .. .	147	
<b>B</b>		
Baking Powder, Substitutes for Tartaric Acid in, J. NAPIER .. .. .	124	
BALLAND on Alkaloids in Old Flour .. .. .	228	
BECKER, F. on the Determination of Tannin .. .. .	112	
BECKURTS, H. on the Separation of Alkaloids from Human Remains .. .. .	191	
Beef Tea, Note on, O. HEHNER .. .. .	221	
BEHAL, A. on the Separating of Copper and Cadmium .. .. .	129	
Behaviour of Serum Albumen in the presence of Acids and Neutral Salts, E. JOHANNSEN .. .. .	184	
BENEDICT on the Determination of Glycerine in Dilute Aqueous Solutions and in Fats .. .. .	205	
BERGLUND, E., on the Separation of Sn, Sb, and As .. .. .	17	

	PAGE		PAGE
BERNECK on the Detection of the Adulteration of Menthol by Thymol ..	37	CAUSSE on the Separation of the Halogens Chalybeate Water and Saline Deposit from Southbourne-on-Sea, C. T. KINGZETT ..	26
"    on Secale Cornutum ..	169	Cheese Analysis, DR. MUTER ..	2
BRUSEMANN, on the Melting points of Fats	204	Chemical Candidate for Parliament, A. ..	213
BIARD on the Determination of Cane-sugar, Grape-sugar, and Dextrin in the same Solution ..	125	"    Examination of Pepper, LENG ..	10
Bile in Urine, Test for, C. DEUBNER ..	190	Chloral Hydrate, Reaction for, M. HIRSCHFELD ..	78
Blow-Pipe Reagent, Silver Iodide as a, P. CASAMAJOR ..	114	Chlorine, Diphenylamine as a Reagent for, H. HAGER ..	154
BLUM on the Determination of Silicon in Iron ..	213	"    New Method for the Volumetric Determination of, E. BOHLIG ..	171
BODENBENDER, H., on the Direct Determination of Invert Sugar ..	112	Chlorides, Distinguishing between Bromides, Iodides and, H. HAGER ..	97
BODMER, R., on the Determination of Mixtures of Milk-sugar and Cane-sugar ..	62	"    in the presence of Bromides and Iodides, Detection of, L. de KONINCK ..	173
BOHLIG, E., on a New Method for the Volumetric Determination of Chlorine ..	171	Cinchona Alkaloids, Quantitative Determination of, SHIMOYAMA ..	207
Books, &c., Received 20, 40, 60, 80, 154, 174, 194, 214 and 234		Cinchonidine in Quinine Sulphate, Determination of, DE VRIJ ..	148
BOSSHARD, E., on KJELDAHL'S Method for the Determination of Nitrogen ..	127	Citric Acid, Detection of, G. MANN ..	126
Bread, Musty, A. PERCY SMITH ..	181	CLASSEN, A., on Quantitative Analysis by Electrolysis ..	154, 192
BROADBENT, on the Estimation of Water in Honey ..	204	CLAYTON, E. GODWIN, on Admixtures of Malt-Extract with certain Fixed Oils ..	140
Bromides, Distinguishing between Chlorides, Iodides and, H. HAGER ..	97	CLERE, on the Determination of Carbon in Iron ..	212
Bromides and Iodides, Detection of Chlorides in the presence of, L. de KONINCK ..	173	Cobalt and Nickel, Separation of, ILINSKI AND KNORRE ..	189
BROMMA, on Tests for the Purity of Iodoform	37	Cocain, A. PERCY SMITH ..	55
BRUNNEMANN, on the Determination of Phosphoric Acid in Phosphatic Slags ..	212	Cocoa-Nut Oil, Relation of, to the Various Methods of Butter Analysis, R. W. MOORE ..	224
Buckwheat and Rice, Distinction between, Butter Analysis, The Relation of Cocoa-Nut Oil to the Various Methods of, R. W. MOORE ..	60	Cod Liver Oil, Iodized ..	20
Butter-Fat, REICHERT'S Method of Examining, A. H. ALLEN ..	103	Codine, New Colour Test for, RABY ..	208
Butter and Oleomargarine, Examination of, M. LIEBSCHUTZ ..	111	Colours, Artificial, in Butter, &c., Method of Separating and Determining, E. W. MARTIN ..	163
"    &c., Method of Separating and Determining Artificial Colours in, E. W. MARTIN ..	163	Colouring Matters in Granulated Sugar, HEWITT ..	203
		"    in Wines, Detection of, P. N. ARATA ..	186
		Composition and Adulteration of Honey, LENZ AND SIEBEN ..	34
		Composition of Mare's Milk and Koumiss, Dr. VIETH ..	218
		Concentrated Meat Preparations, Analysis of, Dr. A. STUTZER ..	57, 73
		Continuous Etherification, L. M. NORTON AND C. O. PRESCOTT ..	13
		Contributors, Notices to ..	20, 40, 154, 234
		Copper and Cadmium, Separation of, A. BEHAL ..	129
		"    Separation of Cadmium from, P. GUCCI ..	79
		Correspondence 40, 80, 116, 134, 194, 214, 233	
		Correspondents, Answers to ..	40, 214, 234
		CRIPPS on a New Test for Aloes ..	60
		Crude Tartar and Wine Lees, Determination of Potassium Bitartrate in, F. KLEIN ..	170
		Cupreol, O. HESSE ..	187
		CURTMANN on Pyrogallic Acid as a Reagent for Detecting Minute Quantities of Nitric Acid ..	212
		"    on the Detection of Salicylic Acid	
		Cyanogen in a Mixture of Gases, Estimation of, G. JAQUEMIN ..	190

## C

Cadmium and Copper, Separation of, A. BEHAL ..	129
Calculation of Milk Results, HEISCH ..	22
Caoutchouc, Vulcanised, Analysis of, B. UNGER ..	131
CAMERON, Sir C., on the Operation of the Adulteration Acts in Dublin ..	175
Cane-Sugar, Grape-Sugar and Dextrin in the same Solution, Determination of, BIARD AND PELLET ..	125
Caramel, Detection of, AMTHOR ..	36
Carbon Bisulphide, Adulterant of, M. VAN ENGELEN ..	189
"    in Iron, Determination of, CLERE ..	212
Casein in Cow's Milk, Determination of, FRENZEL ..	95

	PAGE
<b>D</b>	
DARNEY, C. W. JUN., on the Estimation of Nitrogen in Fertilizers ..	15
DEMARCAZ, E. on the Separation of Titanium from Niobium and Zirconium ..	114
DEMSEI on the Detection of Rosin Oil in Mineral Oils ..	231
DEUBNER, C., on Tests for Bile in Urine ..	190
DE VRIJ, on Cinchonidine in Quinine Sulphate ..	148
DIETRICH, J., on Aloin ..	186
Dimethylamidoazobenzol as an Indicator, FISCHER ..	152
Diphenylamine as a Reagent for Chlorine, H. HÄGER ..	154
DOUMER on the Spectroscopic Behaviour of Fatty Oils ..	148
DRINKWATER, DR. on a New Variety of Pyrites for Sulphuric Acid Manufacture ..	109
DUBENARD on the Volumetric Determination of Potassium ..	210
Dublin, Operation of the Adulteration Acts in the City of, SIR C. CAMERON ..	175
DUPRE, DR., on the Estimation of Dissolved Oxygen in Water ..	156
"    Observations on the Permanganate Test in Water Analysis ..	118

**E**

EARL, Patrick W., Obituary Notice ..	174
Editor, Letters to the, 40, 80, 116, 134, 194, 214, 233 ..	
Electrolysis, Quantitative Analysis by, A. CLASSEN and R. LUDWIG ..	154, 192
EMMERLING on the Volumetric Determination of Phosphoric Acid in Superphosphates ..	
"    on the Examination of Food Stuffs ..	229
ENGEL, R., on New Indicators for Use in Determination of the Caustic Alkalies in the presence of Carbonates ..	128
ENGELEN, M. VAN, on an Adulterant of Carbon Bisulphide ..	189
Enriched Skim Milk Cheese, DR. VIETH ..	7
ESTCOURT, C., on the Valuation of Milk Solids instead of a Limit or Standard ..	54
Ether, Detection of Petroleum Ether in, D. VITALI ..	95
Etherification, Continuous, L. M. NORBON and C. O. PRESCOTT ..	13
Eucalyptol, C. JAHNS ..	126
Examination and Composition of Milk and Milk Products, Notes on, DR. VIETH ..	67
"    of Butter and Oleomargine, M. LIEBSCHUTZ ..	111
"    Food Stuffs, A. EMMERLING ..	229
"    Pepper, C. NEUSS ..	76
Extraction Apparatus, New, T. WEYL ..	153

	PAGE
<b>F</b>	
Fat Extraction, Apparatus for, F. WALLIS STODDART ..	108
"    in Milk, Simple Apparatus for the Estimation of, A. C. WILSON ..	55
Fats, Melting Points of, BEUSERMANN ..	204
"    and Oils, Mutual Detection of, HUBL ..	9
"    Determination of Glycerine in, BENEDIKT and ZSIGMONDY ..	205
Fatty Oils in Mineral Oils, Detection of, F. LUX ..	169
"    Spectroscopic Behaviour of, DOUMER ..	148
Fehling's Solution, Action of Tannin on, SONNENSCHNEIN ..	205
Filtering Small Quantities of Liquid, Apparatus for, WILEY ..	204
FISCHER on Dimethylamidoazobenzol as an Indicator ..	152
Flour, Old, Alkaloids in, BALLAND ..	228
Fluorescent Substances in Lard Oil, Note on the Detection of, PROF. TICHOBOENE ..	161
Food Exhibition ..	174
"    Notices ..	194
"    Stuffs, Examination of, A. EMMERLING ..	229
FRENZEL on the Determination of Caseine in Cows' Milk ..	95
FRESENIUS, R., on the Determination of Tin in Tin Scorise ..	193
"    on a New Reaction for Titanic Acid ..	172
Fusion of Silicates, Substitute for Sodium Carbonate in, CARL HOLTHOF ..	18

**G**

GAWALOSKI, A., on Petroleum Ether for the Extraction of Wet Substances ..	185
GLASER, C., on the Determination of Phosphoric Acid ..	127
Globaline, New Method for Separating Albumen from, WICHAIRLOW ..	211
Glucosides and Alkaloids, Reactions of, SERENA ..	149
Glycerine in Dilute Aqueous Solutions and in Fats, Determination of, BENEDIKT and ZSIGMONDY ..	205
Goa Powder, Active Principle of, PETT ..	207
GRAEBE, G., on the Detection of Nitrogen in Organic Compounds ..	190
Grain, Determination of Starch in, M. MARKEE ..	94
Grape-Sugar and Milk-Sugar, New Tests for, RUBNER ..	168
Granulated Sugar, Colouring Matter in, HEWITT ..	293
Graphite in Minerals, Determination of, J. B. MACKINTOSH ..	113
Gravimetric Determination of Manganese, H. VON JUPTNER ..	149

	PAGE
GROCCO, P., on Phenylhydrazine as a Test for Sugar in Urine .. ..	174
GUCCI, P., on the Separation of Copper from Cadmium .. ..	79
Gum Arabic, Examination of, for Artificially Prepared Gums, H. HAGER .. ..	230

## H

HAGER, H., on Artificial Honey .. ..	147
"   the Detection of Cu and Pb in Wine .. ..	77
"   Diphenylamine as a Reagent for Chlorine .. ..	154
"   the Examination of Gum Arabic for Artificially Prepared Gums .. ..	230
"   the Quantitative Determination of Aloes in Mixtures .. ..	95
Halogens, Separation of the, CAUSSE .. ..	230
HAMBURGER, H. J., on the Estimation of Urea .. ..	150
HAMPEL on the Volumetric Analysis of Manganese .. ..	191
HAUPE, W., on the Separation of Zinc .. ..	152
HEHNER, Note on Beef Tea .. ..	221
"   Honey .. ..	217
HEINER on the Quantitative Valuation of Resin in Soap .. ..	209
HEISCH on the Calculation of Milk Results .. ..	22
HEPPE, G., on the Detection of Turpentine in Oil of Lemon .. ..	187
HERFF, B. VON, on the Estimation of Nitrogen in Fertilizers .. ..	15
HERZ, H., on the Detection of Alkanet in Wine .. ..	146
HESSE, O., on Cupreol .. ..	187
HEWITT on Colouring Matter in Granulated Sugar .. ..	203
HIEZELMANN on the Determination of Salicylic Acid in Wine .. ..	77
HIEPE on an Adulteration of Olive Oil .. ..	205
HIRSCHFELD, C., on a Reaction for Chloral Hydrate .. ..	78
HILL, DR. ALFRED, Inaugural Address by, .. ..	42
"   Remarks on Sewage Farm Milk and Butter .. ..	135
HILL, DR. A. BOSTOCK, Note on an Adulterated Sample of Tea .. ..	71
"   Note on a Sample of Adulterated Pepper .. ..	122
HOLTHOF, CARL, on a Substitute for Sodium Carbonate in the Fusion of Silicates .. ..	18
Honey, Artificial, H. HAGER .. ..	147
"   Composition and Adulteration of, LENZ and SIEBEN .. ..	34
"   Estimation of Water in, WILBY and BROADBENT .. ..	204

	PAGE
Honey, Note on, O. HEHNER .. ..	217
HUBL on the Mutual Detection of Fats and Oils .. ..	9
Human Remains, Separation of Alkaloids from, H. BECKURTS .. ..	191

## I

ILLINSKI on the Separation of Nickel and Cobalt .. ..	189
"   "   "   Iron and Alumina .. ..	232
Inaugural Address, DR. HILL .. ..	42
Improved Apparatus for Milk Analysis, W. JOHNSTONE .. ..	81
Indicator, Dimethylamidoazobenzol as an, FISCHER .. ..	152
"   Lakmoid as an, M. C. TRAUB .. ..	79
Indicators, New, for use in Determination of Caustic Alkalies in the Presence of Carbonates, R. ENGEL and J. VILLE .. ..	123
Indigo, Analysis of, H. M. RAU .. ..	78
Invert Sugar, Direct Determination of, H. BODENBENDER .. ..	112
Iodide of Silver as a Blow-Pipe Reagent, P. CASAMAJOR .. ..	114
Iodides and Bromides, Detection of Chlorides in the presence of, L. DE KONINCK .. ..	173
"   Chlorides and Bromides, Distinguishing between, H. HAGER .. ..	97
Iodized Cod Liver Oil .. ..	20
Iodol, Test for, VULPIUS .. ..	229
Iron and Alumina in the Presence of Phosphates, Determination of, KRETZSCHMAR .. ..	151
"   and Alumina in the Presence of Phosphoric Acid, Determination of, LUCION .. ..	192
"   and Alumina, Separation of, ILLINSKY and KNORRE .. ..	232
"   Determination of Carbon in, CLEERE .. ..	212
"   "   Silicon in,—BLUM .. ..	213
"   and Iron Ores, Determination of Titanic Acid in, A. LEDEBUE .. ..	96
"   Ores, Titration of, W. HEMPEL .. ..	129

## J

JAHNS, C., on Eucalyptol .. ..	126
JAQUEMIN, G., on the Estimation of Cyanogen in a Mixture of Gases .. ..	190
JOHANNSEN, E., on the Behaviour of Serum Albumen in the Presence of Acids and Neutral Salts .. ..	184

	PAGE
JOHANSON on the Determination of Tannin	113
JOHNSTONE, W., on an Improved Apparatus for Milk Analysis	81
JUPTNER, H. VON, on the Gravimetric Determination of Manganese	149

**K**

KAYSER, R., on the Detection of Liquorice in Beer	125
KINGZETT, C. T., on a Chalybeate Water and Saline Deposit from Southbourne-on-Sea	26
KJELDHAHL'S Method for Determination of Nitrogen, E. BOSSHAED	127
,, Nitrogen Method, Note on, D. PFEIFFER and F. LEHMANN	172
KLEIN, F., on the Determination of Potassium Bitartrate in Crude Tartar and Wine Lees	170
KNOBE on the Separation of Nickel and Cobalt	189
,, on the Separation of Iron and Alumina	232
KOHNSTEIN, B., on the Determination of Free Sulphuric Acid in Vinegar	126
KONINCK, L. DE, on the Detection of Chlorides in the Presence of Bromides and Iodides	173
Koumiss, Estimation of Acidity in, H. W. WILEY	204
,, and Mare's Milk, Composition of, DR. VIETH	218
KREMEL, A., on the Detection of Monocarbonate in Sodium Bicarbonate	79
KRETZSCHMAR, M., on the Determination of Alumina and Iron in the Presence of Phosphates	151
KRUTWIG on the Separation of Cl and Br in the Dry Way	37

**L**

LACH, B., on the Valuation of Ozokerit	153
Lakmoid as an Indicator, M. C. TRAUB	79
Lard Oil, Note on the Detection of Fluorescent Substances in, Prof. TICHBORNE	161
Law Reports	98, 234
LEDEBUR, A. on the Determination of Titanic Acid in Iron and Iron Ores	96
LEHMANN, F. Note on Kjeldahl's Nitrogen Method	172
LENG on the Chemical Examination of Pepper	10

	PAGE
LENZ on the Composition and Adulteration of Honey	34
Letters to the Editor	40, 80, 116, 134, 194, 214, 233
LIEBSCHUTZ, M. on the Estimation of Zinc in Tutty Powder	113
,, on the Examination of Butter and Oleo-margarine	111
LIEBERMANN, on the Determination of Milk Fat	12
LIPP, F. on the Analysis of Tungsten Steel	128
Liquorice in Beer, Detection of, R. KAYSER	125
LUCRON, R. on the Determination of Iron and Silica in the Presence of Phosphoric Acid	192
LUDWIG on Quantitative Analysis by Electrolysis	154, 192
,, the Quantitative Determination of Uric Acid	130
LUX, F. on the Detection of Fatty Oils in Mineral Oils	169
LYONS, A. B. on the Estimation of Urea	150

**M**

MACKINTOSH, J. B. on the Determination of Graphite in Minerals	113
MAISCH, PROF. J. M. on the Adulteration of Saffron	200
Malt—Extract with Certain Fixed Oil, Admixture of, E. GODWIN CLAYTON	140
Manganese, Gravimetric Determination of, H. VON JUPTNER	149
,, Volumetric Analysis of, HAMPE	191
MANN, G. on the Detection of Citric Acid	126
Manures containing Nitrates, Determination of Nitrogen in, REITMAR	212
Mares' Milk and Koumiss, Composition of, DR. VIETH	218
MARKER on the Determination of Starch in Grain	94
MARTIN, E. W. on a Method of Separating and Determining Artificial Colours in Butter, Fats and Oils	163
Meat Preparations, Concentrated, Analysis of, DR. STUTZER	57, 73
Meetings of the Society, Reports of, 11, 41, 81, 99, 117, 155,	61, 215
MEINKE on a New Method for the Determination of Phosphoric Acid by Molybdenum	129
Melting Points of Fats, BAUSERMANN	204
Milk, Albuminous Compounds in, J. SEBELIEN	184
,, Analysis, an Improved Apparatus for, W. JOHNSTONE	81
,, and Butter, Sewage Farm, Remarks on, DR. HILL	135
,, Committee, Report of	216
,, Detection of Water in, UFFLEMAN	146



	PAGE
Milk, Determination of Casein in, FRENZEL and WEYL ..	167
„ Fats, Determination of, LIEBERMANN ..	12
„ New Method for the Analysis of, M. A. ADAMS ..	46
„ Results, Calculation of, HEISCH ..	22
„ Solids, Valuation of, instead of a Limit or Standard, C. ESTCOURT ..	54
„ Some Analyses of, C. W. STEPHENS ..	30
„ Sugar and Grape-Sugar, New Tests for, RUBNER ..	168
„ „ Note on the Optical Estimation of, A. H. ALLEN ..	72
Mineral Oils, Detection of Fatty Oils in, F. LUX ..	169
„ Detection of Rosin Oil in, DEMSKI and MORAWSKI ..	231
Minerals, Determination of Graphite in, J. B. MACKINTOSH ..	113
MOHR, C. on the Estimation of Reverted Phosphoric Acid ..	16
Molybdic Acid Residues, W. VENATOR ..	191
Molybdenum, Determination of, VON DER PFORDTEN ..	39
MOORE, R. W., on the Relation of Cocoa-Nut Oil to the Various Methods of Butter Analysis ..	224
Morphia in Opium, Determination of, V. PERGER ..	77
Musty Bread, A. PERCY SMITH ..	181
MUTER, DR., on Cheese Analysis ..	2
„ „ the Processes and Standards of Food Analyses in use at the Municipal Laboratory of the City of Paris ..	143, 163, 179, 195

## N

NAPIER, J., on Substitutes for Tartaric Acid in Baking Powder ..	124
NEUSS, C., on the Examination of Pepper	76
Nickel and Cobalt, Separation of, ILINSKI and KNORRE ..	189
Niobium and Zirconium, Separation of Titanium from, E. DEMARCAV ..	114
Nitrates in Water, New Test for, A. PERCY SMITH ..	199
Nitric Acid in Minute Quantities, Pyrogallic Acid as a Reagent for Detecting, CURTMAN ..	212
„ Quantitative Determination of, WILDT and SCHEIBE ..	190
„ Volumetric Estimation of, LONGI ..	38
Nitrogen in Fertilizers, Estimation of, C. DABNEY and B. VON HERFF ..	14
„ KJELDAHL'S Method for the Determination of, E. BOSSHARD ..	127
„ in Manures containing Nitrates, Determination of, O. RETTMAR ..	212

	PAGE
Nitrogen, Note on KJELDAHL'S Method, PFEIFFER and LEHMANN ..	172
„ in Organic Compounds, Detection of, C. GRAEBE ..	190
NORTON, L. M., on Continuous Etherification ..	13
Note on Beef Tea, O. HEHNER ..	221
„ Honey, O. HEHNER ..	217
Notices to Correspondents ..	60, 154, 174

## O

Obituary Notice, PATRICK W. EARL ..	174
Oil of Lemon, Detection of Turpentine in, G. HEPPE ..	187
Oleomargarine and Butter, Examination of, M. LIEBSCHUTZ ..	111
„ Rapid detection of, Dr. T. TAYLOR ..	13
„ „ Test for, PIALLAT ..	185
Olive Oil, Adulteration of, HIEPE ..	205
Operations in Dublin of the Adulteration Acts, Sir C. CAMERON ..	175
Opium, Determination of Morphia in, V. PERGER ..	77
Optical Estimation of Milk-Sugar, Note on, A. H. ALLEN ..	72
Ores, Iron, Titration of, W. HEMPEL ..	129
Organic Matter in Water, Determination of, M. PETIT ..	147
OXENFELD, D., on a New Reaction for Albumen ..	167
Oxygen in Water, Estimation of Dissolved, Dr. DUPRE ..	156
Ozokerit, Valuation of, B. LAOH ..	153

## P

Parliament, A Chemical Candidate for ..	213
PELLET on the Determination of Cane-Sugar, Grape-Sugar, and Dextrine in the same Solution ..	125
Pepper, Chemical Examination of, LENG ..	10
„ Examination of, C. NEUSS ..	76
„ Note on an adulterated Sample of, Dr. A. BOSTOCK HILL ..	122
Pepsin, Determination of Value of, O. SCHLICKUM ..	147
PERGER, VON, on the Determination of Morphia in Opium ..	77
Permanganate Test in Water Analysis, Some observations on the, Dr. DUPRE ..	118

	PAGE
<b>PETIT</b> on the Active Principle of Goa Powder ..	207
"    Determination of Organic Matter in Water ..	147
<b>PETROLEUM</b> Ether in Ether, Detection of, <b>D. VITALI</b> ..	95
"    for the Extraction of Wet Substances, <b>A. GAWALOSKI</b> ..	185
<b>PFEIFFER</b> on the Volumetric Determination of Urea ..	211
"    Note on <b>KJELDAHL'S</b> Nitrogen Method ..	172
<b>PFORSTEN, VON DER</b> , on the Determination of Molybdenum ..	39
"    on the Determination of Tungsten ..	38
<b>Phenylhydrazine</b> as a Test for Sugar in Urine, <b>P. GROCCO</b> ..	174
<b>Phosphoric Acid</b> , Determination of, <b>C. GLASER</b> ..	127
"    Iron and Alumina in the Presence of, <b>R. LUCION</b> ..	192
"    New Method for the Determination of, by Molybdenum, <b>MEINEKE</b> ..	129
"    Reverted, Estimation of, <b>MOHR</b> ..	16
"    Soluble, in Superphosphates, Volumetric Determination of, <b>A. EMMERLING</b> ..	230
"    in Phosphate Slag, <b>BRUNNEMANN</b> ..	212
<b>Phosphates</b> , Precipitated, On the Use of Ammonium Citrate in the Analysis of, <b>H. H. B. SHEPHERD</b> ..	161
<b>PIALLAT</b> on a Rapid Test for Oleomargarine ..	185
<b>Potassium Bitartrate</b> in Crude Tartar and Wine Lees, Determination of, <b>F. KLEIN</b> ..	170
"    Carbonate. Determination of Small Quantities of Sodium Carbonate in, <b>ROTTGER</b> and <b>PRECHT</b> ..	209
"    Volumetric Determination of, <b>DUBENARD</b> ..	210
<b>Potato Starch</b> , Water in, <b>SAARE</b> ..	146
<b>PRECHT</b> on the Determination of Small Quantities of Sodium Carbonate in Potassium Carbonate ..	209
<b>PRESCOTT, C. O.</b> , on Continuous Etherification ..	13
<b>Proceeding of the Society</b> 1, 21, 41, 61, 81, 99, 117, 135, 155, 175, 195, 215	
<b>Processes and Standards of Food Analysis</b> in Use at the Municipal Laboratory of the City of Paris, <b>DR. MÜTTER</b> ..	143, 163, 179, 195
<b>Pyrites</b> , New Variety, for the Manufacture of Sulphuric Acid, <b>DR. DRINKWATER</b> ..	109
<b>Pyrogallic Acid</b> as a Reagent for Detecting Minute Quantities of Nitric Acid, <b>CWETMAN</b> ..	212

## Q

	PAGE
<b>Quinine Sulphate</b> , Quantitative Determination of Cinchonodine in, <b>DE V&amp;U</b> ..	148

## R

<b>RABY</b> on a New Colour Test for Codeine and Æsculine ..	208
<b>RAU, H. M.</b> , on the Analysis of Indigo ..	78
<b>REICHELT'S</b> Methods for Examining Butter Fats, <b>A. H. ALLEN</b> ..	103
<b>REITMAR, O.</b> , on the Determination of Nitrogen in Manures containing Nitrates ..	212
<b>REMPEL, R.</b> , on a New Apparatus for the Determination of Starch ..	94
Report of Milk Committee ..	216
Reports of the Proceedings of the American Society of Public Analysts 105, 123, 143, ..	223
"    Proceedings of the Society 21, 41, 61, 81, 99, 117, 135, 155, 175, ..	195
<b>Resin in Soap</b> , Quantitative Valuation of, <b>HEINER</b> ..	209
<b>REVIEWS</b> ;—	
"Acetic Acid, &c. (Churchill's Technical Handbooks)." ..	98
"Allan's Commercial Organic Analysis" ..	133
"Aids to the Analysis of Food and Drugs" ..	115
"Fresenius's Quantitative Analysis" ..	97
" Inorganic Chemistry," by C. Frankland and F. R. Japp ..	19
"Life of Scheele," by Hays ..	40
"List of Tests (Reagents), in Alphabetical Order, according to the Names of the Originators," by Schneider and Wilder ..	40
"Simon's Manual of Chemistry" ..	132
<b>Rice and Buckwheat</b> , Distinction between, <b>ROSE</b> ..	60
<b>Rosin Oil</b> in Mineral Oils, Detection of, <b>DEMSEK AND MORAWSKI</b> ..	231
<b>ROTTGER</b> , on the Determination of Small Quantities of Sodium Carbonate in Potassium Carbonate ..	209
<b>Rye Flour</b> , Detection of Wheat in, <b>WITTMACK</b> ..	169

## S

<b>Saffron</b> , Adulterations of, <b>PROF. J. M. MAISOH</b> ..	200
<b>Saponin</b> , <b>M. C. SCHIAPARELLI</b> ..	148



	PAGE		PAGE
<b>U</b>			
UFFLEMAN on the Detection of Water in Milk .. .. .	146	Water Estimation of Dissolved Oxygen in, DR. DUPRE .. .. .	156
UNGER, B., on the Analysis of Vulcanized Caoutchouc .. .. .	131	„ in Honey, Estimation of, WILEY AND BROADBENT .. .. .	204
Urea, Determination of, PFEIFFER .. .. .	211	„ in Milk, Detection of, UFFLEMAN .. .. .	146
„ Estimation of, H. J. HAMBURGER .. .. .	150	„ New Test for Nitrates in, A. PRECY SMITH .. .. .	199
„ „ A. B. LYONS .. .. .	150	„ in Potato Starch, SAARE .. .. .	146
Uric Acid, Quantitative Determination of, E. LUDWIG .. .. .	130	WEYL, on the Determination of Caseine in Milk .. .. .	167
Urine, Phenylhydrazine as a Test for Sugar in, P. GROCCO .. .. .	174	„ on a New Extraction Apparatus .. .. .	153
„ Tests for Bile in, C. DEUBNER .. .. .	190	Wheat in Rye Flour, Detection of, WITTMACK .. .. .	169
—			
<b>V</b>			
VENATOR, W., on Molybdic Acid Residues .. .. .	191	WICHAIBLOW, on a New Method of Separating Globuline from Albumen .. .. .	211
VIETH, Dr. P., on the Composition of Mares' Milk and Koumiss .. .. .	218	WILDT on the Quantitative Determination of Nitric Acid .. .. .	190
„ on Enriched Skim Milk Cheese .. .. .	7	WILEY, H. W., on an Apparatus for Filtering Small Quantities of Liquid .. .. .	204
„ Notes on the Examination and Composition of Milk and Milk Products .. .. .	67	„ on the Estimation of Acidity in Sour Milk and Koumiss .. .. .	204
VILLE, J., on New Indicators for Use in Determination of Caustic Alkalies in the Presence of Carbonates .. .. .	128	„ on the Estimation of Water in Honey .. .. .	204
Vinegar, Estimation of Free Sulphuric Acid in, B. KOHNSTEIN .. .. .	184	WILSON, A. C., on a Simple Apparatus for the Estimation of Fat in Milk .. .. .	55
VITALI, D., on the Detection of Petroleum Ether in Ether .. .. .	95	Wine, Detection of Alkanet in, H. HERZ .. .. .	146
Vulcanized Caoutchouc, Analysis of, B. UNGER .. .. .	131	„ „ of Colouring Matters in, P. N. ABATA .. .. .	186
VULPIUS on a Test for Iodol .. .. .	229	„ „ of Cu and Pb in; H. HAGER .. .. .	77
—			
<b>W</b>			
Water Analysis, Some Observations on the Permanganate Test in, DE. DUPRE .. .. .	118	„ Determination of Salicylic Acid in, G. HEINZELMANN .. .. .	77
„ Chalybeate, from Southbourne-on-Sea, C. T. Kingzett .. .. .	26	„ Lees, Determination of Potassium Bitartrate in, F. KLEIN .. .. .	170
„ Determination of Organic Matter in, M. PETIT .. .. .	147	WITTMACK on the Detection of Wheat in Rye-Flour .. .. .	169
—			
<b>Z</b>			
		Zirconium and Niobium, Separation of Titanium from, E. DEMARÇAY .. .. .	114
		ZIEREK on the Adulteration of Argol .. .. .	189
		Zinc, Separation of, W. HAUPE .. .. .	152
		„ in Tutty Powder, Estimation of, M. LIEBSCHUTZ .. .. .	113
		ZSIGMONDY on the Determination of Glycerine in Dilute Aqueous Solutions and in Fats .. .. .	205

# THE ANALYST.

JANUARY, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
(a) REPORT OF MEETING .. .. .	1	(a) ESTIMATION OF COMBINED CARBON IN IRON AND STEEL, ZABOUDSKY ..	14
(b) CHEESE ANALYSIS, By DR. MUTER ..	2	(b) ESTIMATION OF NITROGEN IN FERTILIZERS, DABNEY AND VON HERFF ..	15
(c) ENRICHED SKIM MILK CHEESE, By DR. VIETH .. .. .	7	(c) ESTIMATION OF REVERTED PHOSPHORIC ACID, C. MOHR .. .. .	16
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—		(d) SEPARATION OF Sn, Sb, AND As, BERGLUND .. .. .	17
(a) THE MUTUAL DETECTION OF FATS AND OILS, HUBL .. .. .	9	(e) SUBSTITUTE FOR SODIUM CARBONATE IN FUSION OF SILICATES, HOLTHOF ..	18
(b) CHEMICAL EXAMINATION OF PEPPER, LENG ..	10	REVIEW:—	
(c) DETERMINATION OF MILK FAT, LIEBERMANN .. .. .	12	INORGANIC CHEMISTRY, By FRANKLAND AND JAPP .. .. .	19
(d) RAPID DETECTION OF OLEOMARGARINE, TAYLOR .. .. .	13	MONTHLY RECORD OF ADVANCES IN THE PREPARATION OF FOOD AND DRUGS:—	
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—		IODIZED COD LIVER OIL .. .. .	20
CONTINUOUS ETHERIFICATION, NORTON AND PRESCOTT .. .. .	13	BOOKS RECEIVED—NOTICE TO CONTRIBUTORS..	20

### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of this Society was held at Burlington House, on Wednesday, the 17th December last, Mr. C. Heisch, vice-president, in the chair.

The minutes of the previous meeting were read and confirmed.

Messrs. Kingzett and Johnstone were appointed auditors.

The Scrutineers, having opened the ballot papers, reported that the following gentlemen had been duly elected:—

As Members—H. F. Cheshire, F.C.S.; Sandford Moore, M.B.; D. A. Sutherland; R. C. Woodcock, F.C.S.

As Associate—J. K. Colwell, Assistant to Mr. Wynter Blyth.

The following gentlemen were proposed, and will be balloted for at the next meeting:—

As Members—W. Newton, Analyst, London; C. A. Smith, Analyst, Sydney, New South Wales.

As Associate—Mr. Bredon, Assistant to Dr. Bernays.

The following papers were then read and discussed:—

“On the Calculation of Milk Results,” by C. Heisch, F.I.C., F.C.S.

“Some Analysis of Pure Milk,” by C. W. Stephens.

“On a Chalybeate Water and Saline Deposits from Southbourne-on-Sea,” by C. T. Kingzett, F.C.S., F.I.C.

The paper announced to be read by Mr. Hehner, on “Honey and Wax from Sugar-Fed Bees,” was postponed, owing to the author’s absence, caused by recent domestic affliction.

At the conclusion of the ordinary meeting, an extraordinary one was held, for the purpose of considering an alteration in the rules of the Society, whereby all past Presidents of the Society now, and for the future, would be eligible for re-election from year to year as vice-presidents and *ex officio* members of the Council, in addition to three

other vice-presidents, who have not yet passed the chair, to be elected annually without any reduction of the number of ordinary members of Council.

Dr. Dupré, in proposing the alteration, said it was desired to avoid the chance of all the London members who had passed the chair, being removed from the Council, and this would happen if some such alteration were not made, unless they were continually re-elected, and so prevent new blood being introduced on the Council; but it was necessary to have a large number of London members on the Council, or there would never be a quorum. It was not likely that the past presidents would swamp the ordinary members—in the Chemical Society they had such a rule—and there were now only 17 vice-presidents after about 44 years.

The alteration was unanimously agreed to.

## NOTES FROM THE PRACTICE AT THE SOUTH LONDON CENTRAL PUBLIC LABORATORY.

BY JOHN MUTER, Ph.D., F.I.C.

### CHEESE ANALYSIS.

(*Read before the Society at the Meeting on 19th November, 1884.*)

A GOOD deal has been written from time to time on this subject, and both processes and typical analyses have been given in the recent works of Dr. Bell and Mr. Wynter Blyth. There is, therefore, perhaps, nothing very novel in what follows, but as the analysis is of growing importance, owing to the increasing importation of oleomargarine cheese into this country from America (which specially finds its way into the shops in the spring months), I have thought it well to publish our method of working, together with a series of the results of the examinations of such of the various cheeses of commerce as have lately come under our notice: the actual analyses quoted being chiefly the work of Mr. José M. Várgas.

The first point is so to deal with the sample, that the small quantity (rarely exceeding  $\frac{1}{4}$  lb.), usually brought by the Inspector, shall suffice for everything. The only adulteration ever now found in cheese being the introduction of oleomargarine, the best thing is at once to separate the fat and to make a preliminary test by Koettstorfer's process. The simple method by gravity is not available with cheese, unless we can work upon a much larger quantity, and besides, the solvents used to extract the fat are apt, unless great care be exercised, to render it a little too light. While extracting the fat, it is well to obtain not less than 6 grammes, so that there will be enough for all future proceedings, should this test turn out unfavourably. The poorest cheese in fat in the market is English cream cheese, and this never contains less than 15 per cent. We, therefore, work upon 45 grammes of cheese, to be sure that we obtain enough fat, thus consuming a little over  $1\frac{1}{2}$  ounces of our sample. There are three steps taken in this part of the analysis, as follows:—

1. *Determination of the total acidity in the presence of alcohol.*—This is necessary, as a guide, in future parts of the work, and is done by rubbing up 5 grammes of the cheese (broken up small if a hard one) in a small mortar, with 10 c.c. of alcohol, and a few drops of alcoholic solution of phenol-phthalein, and titrating with decinormal soda, until

a faint pink colour is obtained, which does not disappear on farther rubbing. The number of c.c. of soda used is noted.

2. *Extraction of the fat.*—45 grammes of the cheese are melted in a basin on the water bath, and 8 grammes of pure sand having been stirred in with a thick glass rod (or rather a glass pestle, somewhat longer and thinner than usual), sufficient decinormal soda is added from a burette, to exactly neutralize all acid; this amount being calculated from experiment No. 1, above described. Another 8 grammes of sand are then stirred in, and the whole dried on the water bath, with frequent stirring. When dry, the contents of the basin are broken up, transferred to a Soxhlet extractor, and acted upon therein by petroleum spirit, which has been redistilled under 200° F. When the extraction is complete, the petroleum is distilled off, and the fat dried by heating to 212° F. with a current of air passing through the flask containing it, until not the slightest vestige of odour of petroleum remains. In the flask will now be left a minimum of 6 grammes of fat.

3. *Ascertaining the amount of alkali consumed in saponifying the fat.*—A small light wide-mouthed flask of a little over 100 c.c. capacity, is thoroughly dried and tared on the balance, and about 2 grammes of the melted fat are poured in, so that all collects exactly in the bottom of the flask, and no drops fall upon the neck or sides. The whole is then weighed, and the tare having been deducted, the balance is the weight of fat taken for analysis. Another similar flask is also got ready for a check blank experiment, without any fat. Into each of these flasks 25 c.c. of a normal solution (56 grms. per litre) of potassium hydrate in alcohol of .835 sp. gr., are very accurately measured by dropping it straight into the bottom, and the mouths of the flasks having been lightly closed by placing a watch glass over each, the contents are brought to the boil, and kept very gently boiling for fifteen minutes. 10 c.c. of alcohol, and a few drops of alcoholic solution of phenol-phthalein are then added to each flask, and the contents are titrated with seminormal hydrochloric acid (18.25 grms. real HCl per litre), until the colour just changes from pink to yellow. By mutual calculation of the check blank experiment, and the analysis, the amount of real KHO consumed in saponifying the fat is obtained, and expressed in millegrammes for each gramme of fat taken. Genuine cheese fat should not consume less than 220 millegrammes of KHO for each gramme started with. That this is so, will be evident from the following table, showing the mean of many analyses of the cheeses named.

Cheddar	..	..	..	..	..	..	227.5
Double Glo'ster	..	..	..	..	..	..	229.3
Stilton	..	..	..	..	..	..	231.7
English Cream	..	..	..	..	..	..	220.0
Dutch	..	..	..	..	..	..	228.7
Gruyère	..	..	..	..	..	..	228.0
Rochefort	..	..	..	..	..	..	229.3
Camembert	..	..	..	..	..	..	229.0
Bondon	..	..	..	..	..	..	228.0
American	..	..	..	..	..	..	220.2

If, however, the KHO consumed exceeds that limit the cheese is probably adulterated, and it is then necessary to confirm by taking the soluble and insoluble fatty acids, using for that purpose the rest of the extracted fat. As a check the

adulteration may be calculated by Koettstorfer's formula, taking the lowest possible estimate of genuine cheese fat thus ( $n =$  millegrammes KHO consumed)—

$$\frac{(220-n) \times 100}{(220-195.5)} = x$$

but we never report on this test alone without confirming by taking the fatty acids. We prefer to use seminormal acid, as the readings are more delicate than with the normal. The addition of the extra 10 c.c. of alcohol after saponification is also an improvement, as there is then never any separation of fatty acid during the titration. The alcoholic potash should be measured from a special burette fitted with a glass stopcock, and provided with a float made to work in a solution of a like specific gravity. The potash solution in alcohol may be made by starting with rectified spirit of .835 gravity and dissolving such an amount of commercial potash (usually about 60 grammes) as will bring it (when cold and after decanting from the sediment) to a specific gravity of .987. It will then be ready for testing against the seminormal acid and diluting to the accurate point. The best way of making the acid is to take 60 c.c. of the ordinary pure hydrochloric acid of the B.P., and make it up to one litre with water; 100 c.c. of this acid is then to be exactly neutralized by pure sodium hydrate, and titrated with decinormal solution of argentic nitrate with potassium chromate as an indicator, and the amount of dilution required having been calculated from this result, the finished acid is once more tested to ensure accuracy.

If the cheese should give unfavourable indications with Koettstorfer's test, then we proceed to:—

4. *The estimation of the soluble and insoluble fatty acids.*—On this point our views have undergone some modification since I first wrote about it in 1877. We now start with about four to five grammes of the fat weighed (by difference from the flask containing it) into a flat bottomed flask of very thick glass. To this we add 25 c.c. of the normal alcoholic potash, close it with a good india-rubber cork, and saponify under pressure for at least an hour at a temperature of 165° F. The flask is then allowed to get perfectly cold, so that the whole soap in it solidifies. It is then washed into another ordinary flask with about 200 c.c. of water, and the alcohol having been got rid of, the acids are thrown up with a slight excess of seminormal sulphuric acid. The acids are collected upon a well wet filter, washed with boiling water until free from acidity, the filter always being kept full. When this point is reached the bottom of the filter is closed by a clamp, and the fat cake allowed to form. When cold, the water is run off from beneath, and the whole allowed to become air dried. The cake is removed to a tared platinum dish, and the filter and funnel, having been rinsed with absolute alcohol, and the rinsings added to the dish, the whole is evaporated, dried at 212° F., and weighed. The soluble acids are obtained in an aliquot part of the filtrate with decinormal soda solution, and, allowance having been made for the excess of seminormal sulphuric acid already added, the result is multiplied by .0088, and expressed as butyric acid. I now agree with Messrs. Angell and Hehner that *with care and experience* the loss by filter washing of fatty acids may be reduced to a minimum, especially if any distinct globules of fat forming on the filtrate when cold, be carefully picked off the surface and added to the main cake. I



also agree with Dr. Dupré in the statement, that, given this method of procedure, the total fatty acids of cheese should add up to about 93·3 per cent., otherwise the analysis is not reliable and should be gone over again.

The following are the average amounts of fatty acids we have found in the under-noted cheeses, which are slightly lower in insoluble acids than those recorded by Dr. Bell in some cases, but the results accord well with the average Koettstorfer's indication :—

Name.	Insoluble acids.	Soluble acids	Total.
Cheddar ..	87·66 ..	5·60 ..	93·26
Double Glo'ster..	87·00 ..	6·28 ..	93·28
Stilton .. ..	86·20 ..	7·02 ..	93·22
English Cream ..	90·01 ..	3·26 ..	93·27
Dutch .. ..	87·20 ..	6·09 ..	93·29
Gruyère .. ..	87·32 ..	5·98 ..	93·30
Rochefort ..	87·00 ..	6·27 ..	93·27
Camembert ..	87·15 ..	6·09 ..	93·26
Bondon .. ..	87·34 ..	5·95 ..	93·29
American } Cheddar }	89·98 ..	3·30 ..	93·28

In deciding upon the adulteration of cheese, the *limit* should therefore be, in my opinion, 90 per cent. of insoluble fatty acids, but if it passes that limit markedly then the *calculation* should be made upon 88·5 per cent. The American oleomargarine cheese shows from about 90·5 to 92 per cent.

The remainder of the ordinary analysis of cheese for the purposes of a quantitative report is then, if necessary, performed as follows :—

5. *Total Solids*.—5 grammes of the cheese (broken up if hard) are placed in a fairly commodious tared platinum dish, and 5 c.c. of absolute alcohol having been added, the whole is allowed to soak for half an hour, and then placed to dry in the water oven till the weight is constant. This experiment is to be performed in duplicate, reserving one portion (hereafter called A) for the fat &c., and the other (hereafter called B) for the ash determination.

6. *Fat*.—The residue A is treated in the dish with 50 c.c. of petroleum spirit and placed on the top of the water bath till it just boils. The whole is stirred to detach the residue, and the spirit having been decanted into the tared flask of a "Soxhlet" or other fat extractor, the now loose residue is transferred to the body of the apparatus, and the extraction conducted in the usual manner. The spirit is then distilled off from the flask and the residue dried at 212°F., and weighed. It is here to be noted that for the purposes of cheese analysis, the petroleum spirit used must have been previously redistilled under 200°F.

7. *Acidity as Lactic Acid*.—The residue from the fat determination is transferred from the extractor to a basin and treated with water. After steeping for some time the acidity is taken with decinormal soda, using litmus paper as an indicator (carefully avoiding any excess of the soda) and calculated to lactic acid.

8. *Lactose*.—The contents of the basin are filtered through a plaited filter and washed, and the lactose is determined by Fehling's solution gravimetrically. As previously pointed

TABLE SHOWING THE AVERAGE COMPOSITION OF CHEESE.

Name of Cheese	Insoluble Acids	Soluble Acids	KHO consumed by one gramme	Water	Fat	Lactic Acid	Lactose	Insoluble Ash	Soluble Ash	Salt
Cheddar .. .. .	87.66	5.60	227.5	33.40	26.60	1.53	—	2.30	2.00	1.52
Double Glo'ster .. .	87.00	6.28	229.3	37.20	22.80	1.80	—	2.56	2.00	1.64
Stilton .. .. .	86.20	7.02	231.7	28.60	30.70	1.08	—	1.80	2.22	.75
English Cream .. .	90.01	3.26	220.0	63.64	15.14	.90	.90	.72	.20	.012
Dutch .. .. .	87.20	6.09	228.7	42.72	16.80	1.35	—	2.26	9.10	4.02
Gruyère .. .. .	87.32	5.98	228.0	33.20	27.26	1.35	—	3.12	1.58	1.05
Rochefort .. .. .	87.00	6.27	229.3	21.56	35.96	.72	—	1.70	8.54	3.42
Camembert .. .. .	87.15	6.09	229.0	48.78	21.35	.36	trace	.16	8.64	3.46
Bondon .. .. .	87.34	5.95	228.0	55.20	20.80	.90	.74	.52	6.46	3.16
American Cheddar .. .	89.98	3.30	220.2	29.70	30.70	.90	trace	2.16	1.54	1.20

out by me when discussing the detection of cane sugar in milk (see ANALYST, vol. 5 page 37) the direct weighing of the precipitated cuprous oxide dried at 212°F. is sufficiently accurate, and better than the ordinary volumetric method in this special case. The determination of lactose is only necessary in the soft or cream cheeses.

9. *Casein*.—Is found by difference.

10. *Soluble and Insoluble Ash*.—The residue B is calcined at a very gentle heat until perfectly charred. It is then treated with water, filtered, and the filtrate evaporated to dryness, gently heated and weighed. The insoluble portion is dried on the filter, strongly ignited till white, weighed, and the weight of the filter ash deducted.

11. *Chlorine in the Ash*.—The soluble ash is dissolved in water, and the solution having been stirred up with a pinch of calcium sulphate to remove soluble phosphates, is filtered and titrated with decinormal solution of argentic nitrate, using potassium chromate as an indicator.

### ON SKIM MILK AND ENRICHED SKIM MILK CHEESE.

BY DR. P. VIETH, F.C.S.

*Read before the Society at the Meeting on 19th November, 1884.*

FOR dairies, in which not cheese-making but the manufacture of butter is the main object, the difficulty arises to dispose of the skim milk. When the milk is "set" in order to raise the cream, and kept standing for 36 or 48 hours, or even longer, as it used to be done exclusively, the skim milk obtained is, if not already curdled, certainly more or less sour, and therefore diminished in its value to some extent, whatever the way of using it up might be. But now-a-days one is enabled by employing machines in which the centrifugal force acts upon the milk to separate the latter into cream and skim milk immediately after milking, and thereby gain a skim milk which is equal to fresh milk in every respect, except its poorness of fat. Instead of, say, 3·5 or 4 per cent. of fat, as present in fresh milk, the separated skim milk contains not more than 0·5 per cent., and in many instances less.

Investigations made on the subject have shown that the fat in milk is easier digestible and more readily assimilated than any other fat, and this cannot be astonishing considering the minutely divided state of tiny globules of microscopic size, in which the fat is present in milk, not to speak of the difference in chemical composition. Skim milk therefore is not to be considered a proper food for infants, but may be taken with the greatest advantage as a wholesome drink and addition to food by human beings—children and adults—with greater digestive powers, enabling them to make up for the deficiency in fat in some other way. It is, especially in large towns with their dense population, to the poorer part of which fresh milk is a too costly and almost unknown luxury, that skim milk ought to be made a regular article of diet. Instead of buying a quart of a watery mixture containing fermented starch sugar, alcohol, salt, and perhaps worse things besides, and called beer, two-pence would be spent much better for a quart of wholesome, nourishing and delicious sweet skim milk.

There are, however, objections to trading with skim milk, and a great deal of prejudice must be overcome, before it would sell in an honest way to any appreciable amount. But even if that could be brought about, only part of the skim milk produced could be disposed of in this way. There are some other ways for using up skim milk, viz., rearing and fattening calves and feeding pigs, and making skim milk cheese; the former will pay only under certain conditions, and skim milk cheese is not in favour with the population of this country.

It was first in America suggested and carried into practice, to sell the fat of milk in the best paying form of butter, and substitute it by a cheaper fat in order to make a rich cheese of poor skim milk. Two years ago I had the opportunity of analysing two specimens of enriched skim milk cheese manufactured in America, and published the analysis in *THE ANALYST* (Vol. VII., 1882, page 137). The cheeses had been made, the one with the addition of oleomargarine, the other with the addition of lard. These substances cannot be introduced into the curd directly, but have first to be brought into a state similar to that in which fat is present in milk, *i.e.*, the state of emulsion. An American with the name of Cooley, the inventor of other dairy machinery, has brought out a centrifugal apparatus for emulsifying fat, and judging by the products I had the opportunity of examining, this apparatus does its work very well. The machine, however, is expensive, rather complicated, and requires an unproportionately great driving power, and will for these reasons most likely not find a very extensive application.

At the Dairy Show held last year, at the Agricultural Hall, Islington, a very simple looking apparatus for the same purpose was shown by Messrs. Lawrence and Co., St. Mary Axe, and again this year exhibited in an improved form at the same place, and at the Health Exhibition, South Kensington. The apparatus is called, "Lactoleoextract," and meant for making emulsions of any kind, though in the first place of those to be used in the manufacture of enriched skim milk cheese. It consists chiefly of a steam injector to which two pipes, fitted with taps, and connected with receiving vessels, are joined. The apparatus is worked as follows:—One of the receiving vessels is charged with the fat, if necessary in a melted state, the other with the liquid, with which the fat is to be emulsified. The steam injector is set to work, and the taps of the feeding pipes are opened, care being taken to run the fat in last. The steam jet breaks up the fat into minute globules, which get mixed with the liquid in a most intimate manner, and a continuous stream of a very perfect emulsion is delivered from the discharge pipe and collected; it may, however, if thought necessary, run through the apparatus over and over again. The apparatus, which is worked with a steam pressure of from 5 to 10 pounds, is very effective. A small one, looking like a toy, produces 15 gallons of emulsion, of any required percentage of fat, per hour, and a larger apparatus 80 gallons in the same space of time. I saw an emulsion made of 1 quart of skim milk and  $\frac{1}{2}$ -pint of melted oleomargarine; it was done in almost no time. A sample of this emulsion, which I took with me, showed, when examined microscopically, the fat subdivided in a most thorough manner. When left at rest, the emulsion threw up a thick layer, which broke almost like tallow, but

diffused by gently shaking the bottle, after having been kept in warm water for a little while. The emulsion was of the following composition:—

## ANALYSIS OF EMULSION OF OLEOMARGARINE IN SKIM MILK.

Solids	..	..	..	..	28·01 per cent.
Fat	..	..	..	..	21·16 ,,
Solids not fat	..	..	..	..	6·85 ,,

The low amount of solids not fat cannot surprise, as, of course, steam is condensed, and the water gets mixed with the emulsion.

As mentioned already, the emulsion can be made of any required degree of richness in very wide limits.

For the purpose of making enriched skim milk cheese, the emulsion is to be mixed with the skim milk, and the mixture then treated in the usual manner. A specimen of such a cheese, similar in shape, size, and appearance to the well-known Cheddar, was shown at this year's Dairy Show, and a sample of it very kindly given to me. An analysis of it gave the following results:—

## ANALYSIS OF ENRICHED SKIM MILK CHEESE CONTAINING OLEOMARGARINE.

Water	..	.	..	..	34·47 per cent.
Fat (Ether extract)	..	..	..	..	18·05 ,,
Casein, etc.	..	..	..	..	42·96 ,,
Ash	..	..	..	..	4·52 ,,
Cl. in Ash	..	..	..	..	0·61 ,,
Equal to NaCl	..	..	..	..	1·01 ,,

In a larger quantity of fat, extracted with every precaution to obtain it as pure as possible, the insoluble fatty acids were determined and found to amount to 90·78 per cent.

I do not pretend to be a judge on English cheeses, which, I cannot help to say, I am not very fond of. But with this reserve, I must declare the cheese in question of very good quality, regarding to taste, as well as to texture. I need hardly add that the determination of the insoluble fatty acids present in the fat of such a cheese gives a ready means for detecting what it was made of.

## CONCLUSION OF THE PROCEEDINGS OF THE SOCIETY.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

In *Dingler's Polytechnisches Journal*.—Hübl gives the following method for the mutual discrimination of fats and oils. It is based upon the fact that nearly all fats are composed of the glycerides of members of three groups of fatty acids. These are the acetic, acrylic and tetrolic acid series. The relative proportion of these acids in any given fat or oil is constant within certain limits, and this proportion varies only in different kinds of oils. The members of the three groups of acids, however, exhibit a very different behaviour towards chlorine, bromine and iodine, while under ordinary circumstances the acids of the first series are indifferent, those of the second and third series readily unite with definite quantities of the halogens and substitution products of the first series are formed.

If, therefore, it is possible to make a fat unite with a halogen under circumstances which preclude the possibility of substitution, and the amount which enters into the compound be accurately determined, the number thus obtained would be a constant, and would be dependent upon the amount of unsaturated acids present in the fat. An alcoholic solution of iodine and mercuric chloride was found to give better satisfaction than iodine alone. The standard solution was prepared by dissolving 25 grams of iodine and 30 grams of mercuric chloride, each in a half litre of 95 per cent. alcohol and uniting the solutions. After standing from 6-12 hours the strength of the solution was determined by means of a standard sodium hyposulphite solution.

An oil is examined by weighing out from .2 to .8 gram and dissolving in ten cubic centimetres of chloroform; an excess of standard iodine solution is added, and after standing from 1½ to 2 hours the excess of iodine solution determined by means of the sodium hyposulphite solution. By a simple calculation the number of grams of iodine taken up by 100 grams of fat is determined, and this number is a constant for the fat examined. Hübl has determined this constant for a large number of oils and fats. The following are some of his results:

Linseed oil . . . . .	158
Walnut oil . . . . .	143
Poppyseed oil . . . . .	136
Cottonseed oil . . . . .	106
Rapeseed oil . . . . .	100
Olive oil . . . . .	82.8
Lard . . . . .	59
Oleomargarine . . . . .	55.3
Palm oil . . . . .	51.5
Tallow . . . . .	40
Butter . . . . .	31
Cocoanut oil . . . . .	8.9
Japanese wax . . . . .	4.2

The method affords a ready means of determining the nature of a fat. In case of a mixture of two known fats or oils the proportion of each present can be readily calculated. Thus let  $x$  be the per cent. of one fat and  $y$  that of the other, then  $x+y=100$ ; and if  $m$  is the iodine constant for the fat  $x$ , and  $n$  that of the fat  $y$ , and if the number found for the mixture is  $J$ ; then the quantity of the fat  $x$  present in mixture is readily calculated from the equation

$$x = \frac{100(J-n)}{m-n}$$

For example, a commercial olive oil gave on examination 97 as the iodine constant, it was therefore adulterated with a considerable quantity of some other oil. The melting point of the free fatty acids was found to be 30°. This indicated the presence of cottonseed oil, which was confirmed by other tests. Then by substituting in the above formula, the quantity of cottonseed oil present was found to be 60 per cent.

THE CHEMICAL EXAMINATION OF PEPPER. *By W. Leng. Zeitschrift für Anal. Chem.* 23-501.—It is well-known that the methods now in use for examining pepper, such as the determination of the aqueous, alcoholic and ethereal extracts, are of little real use in detect-

ing and estimating adulteration, it being possible by judicious admixture to produce an adulterated pepper which shall give any desired extract. The commonest adulteration is with pepper husks and palm kernel powder (palmkernmehl); and E. Geissler showed a short time back that it was impossible to detect this adulteration by extraction. The determination of the alcoholic and ethereal extracts, as now practised, is therefore worthless; and the same may be said of the extraction with petroleum-ether, proposed by A. W. Blyth. The author then proceeds to show that the different extraction apparatus in use give remarkably different results. Thus, Tollen's apparatus gave a petroleum-ether extract of 4.55 per cent in 11 hours; while with Soxhlet's only 1.98 per cent. was obtained in 12 hours. Induced by this condition of things the author has entered upon a series of investigations to work out a method for the purely chemical examination of pepper; and the present paper contains an account of his first results. The method depends upon the fact that pepper is characterised, in contra-distinction to its possible adulterants by the comparatively large amount of starch it contains, and is based upon the inversion of this starch and the determination of the sugar produced. It must not, however, be forgotten that other substances besides starch are inverted by hydrochloric acid and that sugar is not the only substance that reduces copper solution. To obtain good results, therefore, the following directions must be exactly followed:— 3 to 4 grams of the powder to be examined are digested for 3 to 4 hours with  $\frac{1}{4}$  litre distilled water, (the flask being repeatedly shaken), filtered, washed with water, and the damp powder washed back into the flask, which is then filled up so as to contain 200 c.c. To this 20 c.c. of a 25 per cent. solution of hydrochloric acid are added, and the flask is closed with a cork, through which passes a glass tube about a metre long to act as condenser. The flask and contents are then heated for exactly three hours on the water bath, with occasional shaking. The liquid having been allowed to cool, is filtered into a 500 c.c. flask, washed with cold water, the filtrate neutralised with sodium hydrate and diluted up to the mark. The "reduction-value" of this solution is determined with 10 c.c. Fehling's solution diluted with 40 c.c. water. In calculating the reducing power, 10 c.c. Fehling's solution were taken as equal to 0.05 gram sugar. If the hot solution does not clear, a few drops of zinc chloride solution are added (as proposed by F. Mayer.) The disappearance of the red colour with potassium ferrocyanide in a drop of the filtrate acidulated with acetic acid may be used as an indicator.

The author gives the following as his mean results:—

*Black Pepper*, 52 per cent. sugar in the substance, free from ash.

*White Pepper*, 60 per cent. (nearly) sugar in the substance, free from ash.

*Palm Nut Powder*, (Palmkernmehl) 22.6 per cent. sugar in the substance, free from ash.

*Pepper Husks*, 16.3 per cent. sugar in the substance free from ash.

No pure pepper should give less than 50 per cent. reducing sugar, calculated upon the ash-free substance. Taking 23 as the mean percentage for palm nut powder and 52 for pure pepper, the amount of the former contained in a given pepper may be calculated by subtracting the percentage of sugar found from 52 and dividing the difference by 0.29

$$= \left( \frac{52 - 23}{100} \right)$$

F. H. H.

ON THE DETERMINATION OF MILK FAT. BY PROF. DR. LIEBERMANN.—*Zeitschr. für Anal. Chem.* 23, 476.

This is a short paper defending the author's volumetric method for the determination of milk-fat, *Zeitsch. für Anal. Chem.*, 22, 383, which had been called in question by C. H. Wolf, *Pharm. Centralhalle*, 1883, p. 435, or *Zeitschr. für Anal. Chem.*, 23, 87. The latter maintained that the method gives 0.2 per cent. more fat than that obtained gravimetrically (by evaporating to dryness on the sand-bath, and extracting with ether); he tried to explain the difference by assuming that milk and ether, on being mixed, undergo, like water and ether, a change in volume. To obviate this source of error, Wolf recommended using for the extraction 55 c.c. or 44 c.c. instead of 50 c.c. ether.

The author, however, shows that a mixture of ether and milk undergoes no such alteration in volume as is undergone by water and ether. To test the method he made several determinations of milk-fat both gravimetrically, and according to his method with and without Wolf's modification. The following is an example:—

*Milk-Fat.*—

Gravimetrically	.. .. .	4.229 per cent.
The Author's method	} Residue weighed .. .. .	4.187 per cent.
(with 50 c. c. ether)		„ determined volumetrically .. .. .
Wolf's modification	} Residue weighed .. .. .	3.872
(with 54 c. c. ether)		„ determined volumetrically .. .. .

As was to be expected, the results obtained with Wolf's method are too low. The author can only explain Wolf's not obtaining good results with his method by assuming that he did not sufficiently dry the fat. For safe results the fat should be dried at 110° C. for at least half an hour.

As the author has slightly modified the process, he gives the following short description. 50 c.c. milk are well shaken up in a cylindrical vessel with 50 c.c. Potash (Sp. G. 1.27), and allowed to stand 5 minutes; on the lapse of this period 50 c.c. aqueous ether are added, and the mixture shaken moderately for 10 seconds. The operator must then give the vessel one or two vertical taps every half-minute for 20 minutes, during which time the ether separates out clear. Of this clear ether solution 20 c.c. are drawn off with a pipette and run into a flask. The solution is evaporated, and the residue dried for half an hour at 110° or, what is still better, heated over a small gas-flame until a smell of decomposed butter is given off. The residue, after being allowed to cool in the dessicator, can either be weighed directly or determined volumetrically, according to the method described by the author in a previous communication, 22, 383, *Zeitsch. für Anal. Chem.*

Soxhlet's areometric method is recommended for cases when the author's cannot be conveniently used—for example, when the operation takes place in a small and badly ventilated room, where large quantities of ether cannot be evaporated. A drawback to its popular use is that the milk must often be submitted to two examinations, first, for normal milk, and, when this does not succeed, with the areometric method for milks poor in fat.

F. H. H.



**RAPID DETECTION OF OLEOMARGARINE.**—Dr. Thomas Taylor reports to the Department of Agriculture that he has made a series of experiments with oleomargarine of different fats, using a variety of acids to ascertain what permanent change of colour would take place by oxidation, etc. Of the various acids employed, sulphuric acid gave the most satisfactory results. The test is a very simple one. If a few drops of sulphuric acid be combined with a small quantity of pure butter, the butter will assume first an opaque whitish-yellow colour, and, after the lapse of about ten minutes, it will change to a brick red. Oleomargarine made of beef fat, when treated in the same manner, changes at first to clear amber, and after the lapse of about twenty minutes, to a deep crimson.

That the changes in colour do not arise from the action of the sulphuric acid on the artificial colouring matter (annatto) is certain, as I find that when annatto is combined with sulphuric acid a dark bluish-green colour is produced, entirely unlike any of the changes mentioned.

Owing to the active corrosive properties of the sulphuric acid, in making these tests a glass rod should be used in combining these substances.—*Scientific American*.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

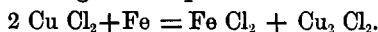
In the *American Chemical Journal*, edited by Dr. Remsen, there is a very interesting paper on "Continuous Etherification," by L. M. Norton and C. O. Prescott, which contains the following remarks, having a distinct bearing upon the analytical method commonly employed for the detection and estimation of ethyl alcohol in methyl alcohol, as usually performed for excise purposes:—

"The authority for the statement that mixed ethers can be obtained by the process of continuous etherification rests upon two experiments of Williamson. It seemed desirable to test the applicability of this process to the formation of mixed ethers anew, especially as Guthrie was unable to repeat one of Williamson's experiments. We chose for an experiment a mixture of methyl and ethyl alcohols, containing 1 mol. of  $\text{CH}_3\text{OH}$  to 1 mol. of  $\text{C}_2\text{H}_5\text{OH}$ . This mixture was treated in the usual manner at  $140^\circ$ , and the utmost care was observed in condensing the products. A colourless distillate, free from sulphurous anhydride, was obtained, and at the same time a gas, which we were unable to condense, escaped from the apparatus. This gas possessed a strong ethereal odour and was doubtless methyl ether, which boils at  $-23^\circ$ . The distillate resolved itself upon fractionation into three portions—one boiling from  $10^\circ$ – $15^\circ$ , the second from  $30^\circ$ – $40^\circ$ , and a third portion consisting of undecomposed alcohols. We obtained from the portion boiling lowest a considerable quantity of a liquid boiling between  $10^\circ$  and  $13^\circ$ , possessing a strong ethereal odour, and corresponding in every respect with the methyl-ethyl ether prepared by Williamson from sodium alcoholate and methyl iodide, and also by Würtz, by the action of a mixture of methyl iodide and ethyl iodide upon silver oxide. The portion of the distillate boiling between  $30^\circ$  and  $40^\circ$  consisted mainly of ethyl ether. The yield of the mixed ether was very good. The three possible ethers appear to be formed simultaneously, but the mixed ether is formed in much the largest proportion."

MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL  
CHEMISTRY.

In the *Bulletin de la Société chimique de Paris* are found some researches by Zaboudsky into the exact nature of the so called residue of carbon left when iron and steel are treated by Boussingault's method with cupric sulphate. He finds in confirmation of the views of Schützenberger and Bourgeois that this is not pure carbon, but a hydrated carbon having the composition  $C_{12}H_6O_3$ . This body is insoluble in water, alcohol, ether hydrochloric and sulphuric acids, and it is impossible to convert it into pure carbon by heat alone. With nitric acid it dissolves entirely, forming a reddish brown compound having the formula  $C_{24}H_{16}(NO_2)O_{12}$ . That it is a definite "hydrate of combined carbon" is further proved by its behaviour with the halogens and not even decomposition by electrolysis can cause it to yield pure carbon. Upon these researches the author bases the following process for:—

*The determination of combined carbon in iron and steel.*—For the purpose of decomposing the iron or steel, a dry mixture of chloride of copper and chloride of sodium is used. This mixture is obtained by evaporating to dryness a saturated solution of sulphate of copper and chloride of sodium. The metal should be finely pulverised and carefully mixed with this mixture in a mortar. Enough water should then be added to make a paste, and the mass triturated with a pestle, care being taken to keep the mortar cold. The decomposition is effected according to the equation:



Theoretically 4.8 grams of chloride of copper, or 14 grams of the mixture as prepared above, are required to dissolve 1 gram of iron. Practically, however, 20 grams of the mixture are found to be more convenient.

After half an hour's trituration the pasty mass is raised from the mortar and placed in a beaker, and the mortar washed with ferric chloride (1 pt.  $Fe_2Cl_6$  to 4 pts. water). The glass containing the pasty mass, together with the wash water, is then heated gently, and, after warming, a little hydrochloric acid is added. This operation lasts about forty-five minutes, when the residue can be collected on a filter. This is then dried and burned in the ordinary manner. The loss in weight corresponds to the weight of the hydrated carbon and is *not* the weight of pure carbon, as was formerly supposed.

In order to obtain the true weight of the combined carbon, factors must be used. These factors represent the amount of carbon in the hydrate of carbon that has been burned.

In order to determine the mean value of these factors for the various grades of iron Zaboudsky examined a large number of specimens, and gives the following numbers as representing the amount of carbon in the combustible portion of the residue obtained by treating iron and steel in the manner given above:

Pure specular cast-iron	..	..	..	..	..	.720
Ferro-manganese	..	..	..	..	..	.700
Specular manganiferous cast-iron		..	..	..	..	.685
White cast-iron	}					
Pure grey cast iron		..	..	..	..	..

Steel (cast for guns)	..	..	..	..	..	..	.660
Hard steel	..	..	..	..	..	..	.675
Iron	..	..	..	..	..	..	.690

In the *American Chemical Journal* is found a long paper on the determination of nitrogen in fertilizers, in which is given the results of an exhaustive series of experiments made at the agricultural experimental station, Raleigh, N.C., performed by Messrs. C. W. Dabney, Jr., and B. Von Herff, with the view of determining the actual relation of a so-called commercial combustion (like the improved soda-lime process described by Ruffle in 1881 at the Chemical Society) to that of the *absolute* method of Dumas or one of its modifications. The Ruffle process was carried out exactly as directed by the authors, except that glass tubes were used instead of the iron ones originally described. This method has the advantage of being simpler, cheaper and somewhat quicker than the copper oxide method. There are fewer risks to run, fewer combustion tubes break, and for several reasons a much larger per cent. of complete analyses are obtained by it.

The tube used was exactly like the ordinary soda-lime combustion tube. Charcoal, free from nitrogen, was obtained, and the mixture with sulphur was weighed out in 2 gram portions. The soda-lime and fused hyposulphite were mixed by eye. In making the combustion a noteworthy precaution is to heat the tube very slowly at first, especially in case of compounds containing high per cents. of nitrogen. In some cases the determination may require three hours. If the combustion is too much hastened the results will be too low. Ordinarily, an hour and a half is necessary for making two such determinations together, the weighing and filling the tubes included. Occasionally, the final reaction in titrating is rather indistinct when this combustion had been made. The copper oxide method followed was that described by Prof. Johnson, in which the air is removed by a Sprengel pump. It was not found necessary to pass oxygen, however, in most cases, and they, therefore, omitted the chlorate of potash from the end of the tube. In mixtures containing a good deal of horn, where resistant cyanogen compounds are formed, this oxygen may be of some aid in completing the combustion. In analysing compounds rich in nitrogen, and especially those containing much nitrate, the addition of charcoal powder to the substance causes the nitrogen to come off more regularly, and gives generally better results. For getting the air, before combustion, and the nitrogen afterwards, out of the tube, the Authors used carbon dioxide without a pump and obtained excellent results, though more time is consumed in this way than with a good pump. Magnesite or carbonate of manganese, put in the back end of the tube, are the best sources of carbon dioxide for this purpose. They give off their carbon dioxide slowly and uniformly. In the Authors' experience this method of driving the nitrogen out is far preferable to working with a poor, or slow pump, and they recommend it to those who do not want to invest in a Sprengel pump. If one can get a fast-working, tight pump, a combination of both plans of getting the nitrogen out is the best and quickest method.

The authors experimented upon four typically distinct nitrogenous bodies with the following results:—

แผนกห้องสมุด กรมวิทยาศาสตร์  
กระทรวงอุตสาหกรรม

Material.	Ruffe.	CuO.	Theory.
Paranitrobenzanilid .. .. .	11.495	11.670	11.570
Nitrate of potash .. .. .	13.735	13.70	13.861
Nitrosalicylic acid. . . . .	7.450	7.655	7.690
Dinitrophenyltoluidid .. .. .	12.620	12.910	12.844

They then extended their experiments to ten distinctive fertilizers, with the sub-joined effects :--

Material.	Ruffe.	CuO.	Difference.
Ammoniated cotton fertilizer .. ..	2.085	2.065	+ 0.020
Fertilizer, with tobacco refuse .. ..	2.125	2.195	- 0.07
Ammoniated superphosphate with 1 p. c. N from nitrate .. .. .	2.110	2.075	+ 0.035
Ammoniated superphosphate with animal and vegetable matter and nitrate .. ..	2.080	2.095	- 0.015
Shoemaker's bone meal .. .. .	6.715	6.775	- 0.060
N. C. fish-scrap .. .. .	7.515	7.675	- 0.160
Cotton-seed meal, extracted .. .. .	7.865	8.075	- 0.210
Chincha Island Peruvian .. .. .	7.255	7.270	- 0.015
Shoemaker's "Ammonite B." .. .. .	12.685	12.725	- 0.035
Dried blood .. .. .	14.585	14.770	- 0.185

In conclusion, they point out that these experiments, confirmed by many others in their possession, clearly show that the Ruffe and Dumas methods give equally good results. In general it may be said that the Ruffe process is as well suited to come sufficiently near the truth as the cupric oxide, and that in both we have two well defined "absolute" methods of estimating nitrogen in fertilizers generally.

#### ON THE DETERMINATION OF REVERTED PHOSPHORIC ACID AND THE PHOSPHORIC ACID IN DI-CALCIUM PHOSPHATE.—BY CARL MOHR. *Zeitschr. f. Anal. Chem.* 23-487.

The analysis of commercial phosphates (superphosphates, &c.) has led to considerable differences in the manure trade; and it is therefore of the utmost importance to have a method giving better results than the existing ones.

The method practised by French and Belgian (also by many English) chemists consists in determining the phosphoric acid, soluble in ammonium citrate, by direct precipitation; while, in Germany, the aqueous solution is still preferably employed.

Regarding the ammonium citrate method, the author remarks that it is often likely to fail because of the difficulty in hitting off the right proportion of ammonium citrate to phosphoric acid; further, the filtrate from the precipitate with magnesia mixture, always gives a slight precipitate when treated with fuming nitric acid in excess and ammonium molybdate, and heated for an hour at 85° C. It is on this latter reaction that the author has founded a method, which gives results agreeing satisfactorily with those obtained by the difference method. About five grammes of the substance are treated with hot water in a mortar, and filtered into a 200 or 250 c.c. flask. After three successive treatments with hot water, the insoluble powder in the mortar and on the filter is brought into a flask and digested for an hour at 60°-70°, with 25 c.c. of an alkaline solution of ammonium citrate. The solution thus obtained is filtered and added to the first filtrate, the flask containing both being then filled up to the mark. If the liquid is not acid, it is acidulated with a few drops of nitric acid. To 10-20 c.c. of this solution an equal volume of fuming nitric acid is added, and then molybdate solution in

excess. To ensure complete precipitation the mixture must be digested for an hour at 85° C. on the sand-bath, and allowed to cool before filtering, as the hot acid solution rapidly attacks the filter paper. The yellow residue on the filter is dissolved in ammonia, filtered and precipitated with magnesia mixture. The precipitate can be either weighed directly or titrated with a standard uranium solution. An important advantage of this process that the direct precipitation method does not possess, is that it can be used for the examination of phosphates, manures, &c., containing magnesium compounds.

The following example is taken from the author's results.

The substance examined was a precipitated phosphate from Anvelais in Belgium, which had been previously analysed by two chemists, M. and G., using the direct precipitation method.

<i>Difference method :</i>				
Total quantity	..	..		31.50 per cent.
Insoluble in ammonium citrate				5.35 ,,
Soluble in ammonium citrate	..			26.15 ,,
<i>The Author's method :</i>				
(1) ..	..	..	..	26.15 ,,
(2) ..	..	..	..	25.50 ,,
Mean	..	..	..	25.82 ,,
<i>Direct precipitation method :</i>				
M. obtained	..	..	..	23.55 ,,
G. ,,	..	..	..	23.552 ,,

The author's results thus agreeing much better with the calculated ones than those obtained by the direct precipitation method.

#### A NEW METHOD FOR THE QUALITATIVE SEPARATION OF TIN, ANTIMONY, AND ARSENIC.— BY EMIL BERGLUND.\* Ber. d. Deutschen Chem. Gesell, 17-95.

The method is based upon the fact that the sulphides of tin, antimony, and arsenic, when boiled with oxide of copper, lose their sulphur, and are converted into the higher oxides. Thus, if tin sulphide be dissolved in sodium hydrate, and boiled with copper oxide, stannic acid is obtained. In the same way antimony and arsenic sulphides are converted respectively into antimonic and arsenic acids. The copper oxide required for this reaction may be prepared by precipitation with sodium carbonate, and drying at 100°–150° C. The process is the following:—

The sulphides, obtained by dissolving in ammonium sulphide, and reprecipitating with hydrochloric acid, are well washed, and brought into a porcelain dish, with a moderate quantity of water. The liquid is then boiled, and, while stirring, sodium sulphide carefully added (a large excess being avoided) till a completely clear solution is obtained, or, at the most, only black-brown copper sulphide remains, which sometimes gets dissolved by the ammonium sulphide. Neglecting this residue, copper oxide is now added, the solution being meantime kept boiling and well stirred. If sufficient oxide of copper be added, the copper sulphide sinks as a heavy powder to the bottom, leaving the supernatant liquid clear. The solution, having been thus "desulphurized," is filtered warm. In the filtrate are tin, antimony, and arsenic, as sodium salts of the

\* From Zeitsch. für Anal. Chem. 23, 573.

respective acids. If much antimony be present it now shows itself by the formation of a white granular precipitate. On cooling, the liquid is mixed with one-third to a quarter its volume of alcohol; the sodium antimonate then separates out as an extremely fine white precipitate, which, after being allowed to settle, is filtered. The filtrate must be passed several times through the filter, as there is some difficulty in getting it clear. The clear filtrate is boiled till free from alcohol, and then mixed with excess of a concentrated solution of ammonium chloride. If a large quantity of tin be present, a milk-white precipitate is now formed. If no precipitate be produced, there can only be, at the most, small traces of tin in the solution. If tin and arsenic be both present, the precipitate contains  $2\text{SnO}_2$ ,  $\text{As}_2\text{O}_5$ . However, whether ammonium chloride causes a precipitate or not, a few drops of ammonium hydrate are added, and sulphuretted hydrogen passed in. If a precipitate be produced, the current of  $\text{H}_2\text{S}$  is continued until it redissolves, leaving, possibly, a few flakes of insoluble silica or alumina (from the porcelain dish). To the clear solution is now added one-third its volume of ammonium hydrate and magnesia mixture to precipitate the arsenic as ammonium-magnesium arseniate.

If tin has not been detected in the course of the operation, the liquid, after having been allowed to stand an hour, is filtered, and the filtrate acidulated with hydrochloric acid. The direct production of a yellow precipitate shows tin, otherwise a slight white precipitate of sulphur is formed.

---

A SUBSTITUTE FOR SODIUM CARBONATE IN THE FUSION OF SILICATES. By *Carl Holthof*.—*Zeitsch. für Anal. Chem.* 23—499.

THE Author has for many years used sodium bicarbonate instead of the monocarbonate, or a mixture of the carbonates of sodium and potassium as a flux. It possesses several advantages; it can easily be obtained pure, allows of being finely powdered, is not hygroscopic, and when slowly heated gives off its water of crystallisation, and the second  $\text{CO}_2$  molecule without decrepitation. The fusion takes place easier than with the monocarbonate, in most cases below the melting point of the mass, the evolution of the carbonic acid causing the acids to exchange bases sooner than they otherwise would.

The Author proceeds thus:—Pure, dry, and finely powdered sodium bicarbonate, from 12 to 15 times the weight of the silicate under examination, is used. A quarter of the weight is placed directly in the crucible, a second quarter intimately mixed with the powdered substance in a warm dish, and this, after mixing with another quarter on glazed paper, is filled into the crucible, the remaining quarter being used to wash the dish and paper. The crucible, which may be quite half full, is first heated carefully over a moderate flame for a quarter of an hour; the heat is then slowly increased during the next quarter of an hour till the lower half of the crucible is just red. Finally, the heat is raised until the mass melts and becomes clear; the fusion is then complete.

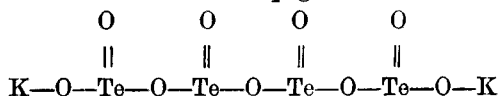
F. H. H.

---

## REVIEW.

INORGANIC CHEMISTRY. *By E. Frankland, Ph.D., F.R.S., &c., Professor of Chemistry in the Normal School of Science, and Francis R. Japp, M.A., Ph.D., &c., assistant Professor of Chemistry.* London : J. & A. Churchill.

In the preface the Authors say :—" The lecture notes for chemical students, already published by one of us, and now in their third edition, were always intended to be the precursors of text books on Mineral and Organic Chemistry. The present volume fulfils this intention so far as Inorganic Chemistry is concerned. It is constructed on those principles of classification, nomenclature, and notation, which, after an experience of nearly twenty years, have been found to lead most readily to the acquisition of a sound and accurate knowledge of elementary chemistry." Now had the authors introduced the saving phrase "in our opinion" after "have been found" then we would not have had anything to say. We have no wish to fight over again the battle for and against graphic structural formulæ, but we feel bound to protest mildly on behalf of other teachers who have also had a similar experience, to the implied conclusion that any system having for its basis the excessive use of such formulæ is admitted by all to lead most readily to the acquisition of a sound and accurate knowledge of elementary chemistry. Every author of a book is entitled to his opinion, and to have that opinion respected so long as he clearly puts it forward simply as his own, but the moment he claims universal acceptance for it, he excites criticism and contradiction. Such criticism and contradiction will not however come from us on the present occasion, because we do not wish to allow our disagreement with some of the notation to warp our ideas of the book as a whole. Before leaving the subject we would simply take by chance the formula of dipotassic tetratellurite which heads page 313, viz. :—



as a specimen of the kind of formula likely to lead "*most readily*" to the acquisition of elementary chemistry and against that to place the words of warning recently spoken by Dr. Remsen, in his able work on theoretical chemistry, to the effect that we are now in a period of formula worship, and the great danger is, lest in the estimation of students more value be attached to the construction of a formula, than to a really practical knowledge of the nature and properties of the substance represented.

Having thus made our protest, we now proceed to examine the general merits of the work, and happily here we are quite in accord with the authors, because after a most careful perusal of the book, we have formed a very high estimate of its general excellence. It opens with a series of 20 chapters (occupying 130 pages), entirely devoted to the theories of chemistry ; and in this portion, the most modern ideas are introduced, and the relations of the various recent researches in chemical physics, to the elucidation of modern chemical theories, are carefully and individually explained. Then follow the non-metals, and the metals in the order usual in text books :—the descriptions of the more important compounds being printed in ordinary type, and the rarer ones in small type. This might be objected to by some purists as savouring of cramming, but

it is, after all really, in our opinion, a legitimate assistance to the student, and, had it not been done, the work would have been of a size altogether too unwieldy for comfortable reading. Besides the excessive use of structural formulæ, there is another part of the book that might have also been modified, and that is the lengthy remarks upon potable water. Here again we meet with another of the points upon which all the chemical world does not absolutely agree with the senior author, and we have no intention of treading upon his toes when we say, that, out of a work of about 640 pages of descriptive chemistry, 26 pages are too much to devote to the single subject of potable water. This is the more marked, because no single instruction as to the practical analysis of water is attempted, and indeed we are told (page 546), that the proper chemical examination of a sample of water "requires weeks, sometimes even months, for its completion." Thus the whole of these 26 pages are simply devoted to a sort of exposition of Dr. Frankland's views upon water, and the deductions to be drawn by those using his organic carbon and nitrogen process. We submit that this section would have much more appropriately formed the nucleus of a special work, than being as it is, placed in a general manual in conjunction with the metal calcium. We are not to be taken as dissenting altogether from the matter of this section, which is in many respects very sound and ably written, but we look upon it as the Professor did on dust, viz., as an excess of matter in the wrong place.

Taking the whole work, however, it is, as we have before said, a most excellent one, and likely to be of great service to students of general inorganic chemistry.

#### MONTHLY RECORD OF ADVANCES IN THE PREPARATION OF FOOD OR DRUGS.

*Iodized Cod-Liver Oil.*—While abstracting Hubl's paper on the absorption of iodine by oils, we received from Messrs. Walker and Moore a sample of cod-liver oil treated in a similar manner for medicinal purposes. It is considered by some authorities that the small, but variable, amount of iodine found naturally in this oil is an important factor in its therapeutical action, and the idea of the originators of the article under review, is to present an oil always containing a definite amount of that element. On analysis, the sample submitted proved to be iodized to the extent of 25 per cent. The oil also answered the tests for genuine cod-liver oil, and we have no doubt that it will find favour with those Physicians who desire to be always able to prescribe an article of definite composition in preference to running the ordinary commercial chances of both strength and purity.

#### BOOKS, &c., RECEIVED.

Report (Seventh Annual) of the Board of Health of the State of New Jersey; Sanitary Control of the Food Supply, by W. K. Newton, M.D.; Some Facts about the New Jersey Milk Adulteration Act, by W. K. Newton, M.D.; American Chemical Review; American Grocer; British Medical Journal; The Chemist and Druggist; The Country Brewers' Gazette; The Cowkeeper and Dairyman's Journal; The Grocer; The Grocers' Gazette; Independent Journal; Invention and Inventors' Mart; The Lancet; Le Mouvement Hygienique; The Medical Record; The Medical Press; The Miller; The Monthly Magazine of Pharmacy and Chemistry; The Pharmaceutical Journal; The Polyclinic; San Francisco News Letter; Science; Scientific American; The Students' Journal.

#### NOTICE TO CONTRIBUTORS.

All Letters and M.S.S. intended for the literary part of this Journal should be addressed to the Editorial Department, 325, Kennington Road, London, S.E.



# THE ANALYST.

FEBRUARY, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
(a) REPORT OF ANNUAL MEETING .. .. .	21	(a) QUANTITATIVE SEPARATION OF Cl AND Br IN THE DRY WAY, KRUTWIG .. .. .	37
(b) THE CALCULATION OF MILK RESULTS, By MR. HEISCH .. .. .	22	(b) VOLUMETRIC ESTIMATION OF NITRIC ACID, LONGI .. .. .	38
(c) DISCUSSION ON MR. HEISCH'S PAPER .. .. .	25	(c) DETERMINATION OF TUNGSTEN, VON DER PFORDTEN .. .. .	38
(d) A CHALYBEATE WATER AND SALINE DEPOSIT FROM SOUTHBORNE-ON-SEA, By MR. C. T. KINGZETT .. .. .	26	(d) DETERMINATION OF MOLYBDENUM, VON DER PFORDTEN .. .. .	39
(e) SOME ANALYSIS OF MILK AND THE DETERMINATION OF MILK SUGAR, By MR. C. W. STEPHENS .. .. .	30	REVIEWS:—	
(f) DISCUSSION ON MR. STEPHENS'S PAPER .. .. .	32	(a) LIST OF TESTS (REAGENTS) IN ALPHABETICAL ORDER, ACCORDING TO THE NAMES OF THEIR ORIGINATORS, SCHNEIDER AND WILDER .. .. .	40
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—		(b) LIFE OF SCHEELE, HAYS .. .. .	40
(a) THE COMPOSITION AND ADULTERATION OF HONEY, LENZ AND SIEBEN .. .. .	34	CORRESPONDENCE .. .. .	40
(b) THE DETECTION OF CARAMEL, AMTHOR .. .. .	36	ANSWERS TO CORRESPONDENTS .. .. .	40
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—		APPOINTMENT .. .. .	40
(a) TESTS FOR PURITY OF IODOFORM, BROUMA .. .. .	37	BOOKS, &c., RECEIVED .. .. .	40
(b) DETECTION OF ADULTERATION OF MENTHOL BY THYMOL, BERNECK .. .. .	37		

### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE ANNUAL meeting of the Society was held on Wednesday, 28th January, at Burlington House, and was numerously attended. Dr. Hill, Vice-President, in the chair.

The minutes of the previous meeting were read and confirmed.

The Auditors' report that they had examined the accounts and found them to be correct was read, and a copy of the balance sheet will be posted to each member.

The election of officers for the current year was then proceeded with, and on opening the ballot papers the scrutineers Mr. Baynes and Mr. S. Harvey, reported the election to be as follows:—

#### President.

A. HILL, M.D., F.I.C.

#### Vice-Presidents.

(Who have filled the office of President.)

A. DUPRE, Ph.D., F.R.S., F.I.C.

C. HEISCH, F.C.S., F.I.C.

J. MUTER, Ph.D., M.A., F.C.S., F.I.C.

#### Vice-Presidents.

(Who have not filled the office of President.)

M. A. ADAMS, F.R.C.S., F.C.S.

A. WYNTER BLYTH, M.R.C.S., F.C.S., F.I.C.

C. T. KINGZETT, F.C.S., F.I.C.

#### Treasurer.

C. W. HEATON, F.C.S., F.I.C.

#### Hon. Secretaries.

BERNARD DYER, F.C.S., F.I.C.

OTTO HEHNER, F.C.S., F.I.C.

#### Other Members of Council.

A. ANGEL, Ph.D., F.I.C.

A. J. BERNAYS, Ph.D., F.C.S., F.I.C.

W. JOHNSTONE, F.C.S., F.I.C.

J. F. HODGES, M.D., F.C.S., F.I.C.

P. VIETH, Ph.D., F.C.S.

C. R. A. WRIGHT, D.Sc., F.R.S., F.C.S., F.I.C.

The names of those Members of Council whose term of office has not yet expired, and who, consequently, do not retire this year, are J. Baynes, jun., F.C.S., F.I.C.; C. Estcourt, F.C.S., F.I.C.; R. H. Harland, F.C.S., F.I.C.; A. Smetham, F.C.S., F.I.C.; T. Stevenson, M.D., F.R.C.P., F.C.S., F.I.C.; and J. W. Tripe, M.D.

The following gentlemen were also balloted for and elected. As members:—Charles A. Smith, Analytical Chemist, Sydney, New South Wales; William Newton, Analyst, London; J. P. Laws, F.C.S., Analytical Chemist, Oxford. As associates (for re-election in accordance with the Rules):—W. Bouchier, F. W. Gear, F. Heron, J. Nimmo, H. H. Slater, L. Stansell, C. G. Stewart, J. W. Taylor.

The new President then returned thanks for his election and proceeded to deliver the annual address.

The usual votes of thanks to the Chemical Society for the use of their rooms, and to the Honorary Secretaries and Treasurer, were proposed and cordially carried.

Mr. M. A. Adams, F.R.C.S., then read a paper entitled, "A New Method for the Analysis of Milk," the discussion on which was adjourned until the March meeting, the Milk Committee in the meantime to examine the process.

The dates of ordinary meetings for the year 1885 were then fixed as follows:—

Wednesday, February 11th.	Wednesday, June 10th,
„ March 11th,	„ November 11th,
„ April 8th,	„ December 9th,
„ May 13th	„ Jan. 13th, 1886 (Annual

Meeting).

The Presidential Address, together with a full report of all the speeches, and of Mr. Adam's paper, will appear in our next issue, the time being too short to admit of justice being done to them in the present number without unduly disturbing the publication arrangements of THE ANALYST.

The members afterwards dined together at the Criterion, where a pleasant evening was passed.

## ON THE CALCULATION OF MILK RESULTS.

BY CHAS. HEISCH, F.I.C., F.C.S.

*Read before the Society at the Meeting on 17th December, 1884.*

IN the discussion of the Manchester Appeal Case, which took place at the end of last year, a general feeling seemed to prevail that, if a sample of milk contained a good proportion of fat, it, to a certain extent, made up for a deficiency in non-fatty solids; and Mr. Escourt, in a paper read on that occasion, proposed a mode of valuation, the great recommendation of which seemed to be that it would pass many more samples than we do at present. I then spoke strongly on the subject of the superior value of solids not fat, to fat in milk considered as a food; and the more I consider the question, the more strongly I feel, that fat should not be taken as in any sense an equivalent for non-fatty solids. I should, therefore, propose that the valuation of milk should include two separate statements—the percentage of fat in the milk, and the percentage of non-fatty solids in the milk deprived of its fat, or we might, for brevity sake, call it the skimmed

milk. This would sometimes lead to a milk being passed which is now (if not actually called adulterated) at least classed as poor; but on looking through all the analyses I have made of samples since the first Adulteration Act was passed, I find comparatively few samples in which it makes a material difference, the general experience being that low fat accompanies low non-fatty solids. The great thing that I observe in genuine samples being, that, though the fats differ considerably, the non-fatty solids calculated on the skim milk approach much more nearly to uniformity than when calculated on the whole milk.

Concluding, for the present, that the limits taken by the Society are correct, this would leave the fat, as now, at 2·3 per cent., and the non-fatty solids in the milk deprived of its fat at 9·2 instead of 9 per cent. on the whole milk. These limits will, of course, be liable to variation after the report of the Milk Committee has been discussed. In Mr. Escourt's paper he mentions a milk containing 6 per cent., non-fatty solids with 4·76 of fat, which he would pass by his valuation, in which he takes 3 as the lowest limit of fat, thereby showing that he considers 1·7 fat as good as 3 solids not fat, which I consider simply monstrous. It is well known that invalids are now frequently put on a skim milk diet, and to such, the milk in question would be distinctly injurious. I know of several such cases in which nothing but skim milk has been taken for 15 or 16 years, any attempt at change of diet being followed by a recurrence of diabetic symptoms, while perfect health is maintained as long as no food but the skim milk is taken. One gentleman, whose case I know most intimately, takes one gallon of skim milk every day. If this contained even 9 per cent. solids, he gets about 14½ ozs. of dry solids per diem; if the solids not fat were reduced to 8 per cent. he gets only 12·8 ozs.; while, if he got Mr. Escourt's milk, his nourishment would be reduced by one-third, or he would have to drink one-third gallon more to get the same amount of nourishment, which is a very serious consideration.

I recently had brought me a sample which shows the impropriety of valuing fat so highly as proposed by Mr. Escourt even more strongly; it contained T. S. 14·4, S. n. F. 6·01, F. 8·39. The percentage of S. n. F. in the skim milk would consequently be 6·5. This sample, I found from the inspector, was dipped from the top of a churn of milk standing in the shop, I have no doubt with the idea that we should be deceived by the amount of fat. According to those who value fat so highly, it was a very good rich milk. I returned it as containing 29 per cent. added water, and my friend, Mr. Hehner, who kindly repeated the analysis, came to the same conclusion. I reported it as unfit for children or invalids. Unfortunately the inspector made a slip in taking the sample, which created a legal objection, viz., that, the milk not having been paid for at the time, the purchase was not complete when the sample was divided. The magistrate took time to consider the answer of our solicitor, which was this—that a purchase was complete when the buyer had the article, inasmuch as the vendor could sue for the money. He pointed out also, that, if this were not the case, any article, however bad, might be sold wholesale, as such transactions were always on credit. After consulting his colleagues, the magistrate came to the conclusion that our solicitor was right, but offered to grant a

case, if defendant wished it. This he refused, and, being thus in a corner, did not deny the watering of the milk, and was fined accordingly. This case was pressed especially as the man supplied the Workhouse Infirmary, where nourishment, not fattening, is specially required.

I, perhaps, feel on this point more strongly, because since I was, as you know, laid up with typhoid fever, I have been, to a large extent, on a milk diet, and, as the last thing in the world which I want is fattening, a milk with high fat and low solids not fat is no use to me. The same is the case with children; the nitrogenous part of the milk and the mineral matters, phosphates especially, being the part of the milk they really want.

Fat.	S. N. F.		Total.	Ash.	
	Whole.	Skim.		Whole.	Skim.
3·57	8·88	9·208	12·45	·72	·74
4·0	9·00	9·37	13·0	·66	·687
4·37	9·15	9·56	13·52	·78	·81
Milk from an Alderney cow allowed to stand and a dip taken off top.					
6·57	10·2*	10·7*	16·77	·72	..
Sample dipped off top of a churn.					
8·39	6·01*	6·56*	14·4	·478	·51
Dipped off top of one pint ordinary milk.					
5·42	9·114	9·63	14·539	·70	·74
Same milk mixed.					
2·26	9·31	9·52	11·57	·675	·69

I have put on the board one or two examples of milks, in which, taken on the whole milk, the non-fatty solids appear low, while (owing to the amount of fat), taken on the skim milk, they are, to say the least, fair, that is, up to our present limit. I have tabulated a number of dairy samples, from various dairy farms in Kent, taken at the railway stations, from which you will see the much greater uniformity in the non-fatty solids, and ash, of skim, than of whole milk, these I need not put on the board at present; but I trust I have shown reason for the plan I propose, and that it is one equally fair to the vendor and purchaser.

Total Solids	Non-Fatty Solids	Non-Fatty Solids in Milk less Fat.	Fat.	Ash in		Sp. gr.
				Ash	Milk less Fat.	
12·29	9·29	9·57	3	·7	·72	1032
12·41	9·17	9·41	3·24	·68	·702	?
12·90	9·08	9·34	3·82	·68	·70	1032·5
12·57	9·28	9·59	3·29	..	..	1031·5
12·27	9·35	9·63	2·91	·68	·70	1031·
12·81	9·23	9·58	3·58	·72	·74	1032·5
14·17	9·39	9·86	4·78	·70	·73	1034
13·50	9·17	9·566	4·33	·68	·71	1132
13·45	9·39	9·78	4·06	·65	·677	1032·5
13·15	9·27	9·64	3·88	·72	·749	1034
12·79	9·36	9·69	3·43	·68	·704	1033

\* These two show the difference between good and bad milk treated the same way.

## DISCUSSION.

DR. VIETH said he thought all such calculations were of doubtful value, as long as one did not know what methods of fat extraction was employed. He felt quite sure that in the case where there were 8 per cent. of fat present in the milk, the extraction was much more complete than in that where only 2 per cent. were present. If the fat was extracted from the total solids directly, that, of course, would influence the solids, not fat, to a vast degree. He thought it was only possible to make such calculations, in a way to be of value, if it were quite certain that the fat was perfectly exhausted.

MR. ALLEN said that he thought favourably of the suggestion of Mr. Heisch. At the same time he would take that opportunity of stating that he was afraid that very often there was a misunderstanding on the part of the magistrates as to the extent of the adulteration. He himself very much objected to the method of stating the results frequently adopted. Supposing one gallon of water were added to two gallons of milk, many analysts would say that the sample contained  $33\frac{1}{3}$  per cent. of added water; but he himself would say it had been adulterated with 50 per cent. of water. He, however, preferred not to use percentages at all; he would certify for example, that a sample of milk was "adulterated with water, and that he estimated the proportion of water added at one gallon to every four gallons of milk, provided the milk was originally of fair average quality; or at somewhat less than that proportion if it happened originally to be of inferior quality." By adopting this method of expression the magistrate at once saw that the analyst had taken into account the natural variations in the quality of milk. He had not had an opportunity of mentioning this before, and it had struck him that it might be of interest.

MR. HEATON said that he reported substantially in the way mentioned by Mr. Allen, viz., that to every gallon of milk there had been added water not less than so and so.

MR. BAYNES thought that they should take 9 as the limit for solids, not fat—not 9·3. He considered it rather unfair to run magistrates down. He thought it was quite sufficient if they used percentages, and if a magistrate had any education at all he would have no difficulty in calculating them.

MR. ADAMS said that on one point he disagreed with the author, viz., as to the relative value of fat and solids not fat. Fat was a most important force producer, and, from that point of view, he thought it was of equal value with the solids not fat. With that exception he quite agreed with Mr. Heisch.

DR. DUPRE said he had repeatedly thought of adopting some similar plan, but had never done so. There was a great deal to be said in favour of Mr. Heisch's plan. The great advantage was, that it did away with any possible variation of the fat, and that was sometimes a great difficulty to the magistrates, and he was afraid possibly to some of their less experienced brethren. Men who had never had much experience themselves were so apt to pin their faith to a standard, and at once say a sample is adulterated if it is less than the standard. He, himself, had always taken into consideration that their standard of 9 should be coupled with 2·5 per cent. of fat, and if more fat were present he made an allowance for that. One might get a milk with 5 per cent. of fat, and only

8·8 per cent. solids not fat, because it had been "diluted with fat," as Dr. Muter had once well expressed it. In the Manchester milk case the opposition wanted to pin them down to that. They were asked, "What was their standard?" and endeavours were then made to fix every analyst to that, irrespective of fat. With regard to the value of fat he agreed somewhat with Mr. Adams. As a rule the fat was as useful as the solids not fat. What the public should have was a fair average milk both in fat and solids not fat. The great difficulty in taking a standard of total solids is that the analyst would so often get samples up to the limit of total solids and yet watered. He had himself had one with 20 per cent. of total solids, and only about  $4\frac{1}{2}$  per cent solids not fat. The milk had evidently been very largely watered, and he reported it as adulterated; the man was fined and never denied that he had watered it. With regard to calculation, magistrates were not always blessed with special mathematical or chemical knowledge, and it was sometimes difficult to persuade them of the difference between percentage present and percentage added. Now he always gave the details, solids not fat, fat, natural moisture and added water, and he never had any difficulty with magistrates. He used also to calculate on 9·3, but since the Manchester case had taken 9 and reported against every case that fell below 9.

Mr. HEISCH, in reply, said there would be a difference in the value of the fat and solids not fat. For instance, if children were given too much fat it would do them harm. As to stating results for magistrates he himself certified, "This consists of 80 per cent. of milk and 20 per cent. of added water."

---

#### ON A CHALYBEATE WATER AND SALINE DEPOSIT FROM SOUTHBOURNE-ON-SEA.

By C. T. KINGZETT, F.I.C., F.C.S.

*Read before the Society at the Meeting, on 17th December, 1884.*

SOUTHBOURNE-ON-SEA is situate towards the eastern extremity of Bournemouth Bay, and was founded some thirteen years ago by Dr. T. A. Compton, a retired physician, formerly of Bournemouth. It is a remarkably healthful and charming table-land, with full southern exposure.

During the excavations which were recently made for the foundations of a Sea-wall and Esplanade, which are constructed seven feet in and below a bed of solid greyish black clay, and about ten or eleven feet (on an average) below the upper surface of the sandy beach, there was discovered a spring of water, which was thought by the workmen to be derived from the sea by permeation through the clay at high tide. Dr. Compton was led, however, to form a different opinion, in which he was confirmed by tasting the water, which has an inky taste, not shared by any of the spring waters above the surface level of the beach; and having sunk a well about 20 feet north of the Sea-wall, samples of the water and a saline deposit found upon the clay were sent to me for analysis, the results of which Dr. Compton has very kindly allowed me to publish in this communication.

The saline deposit strikingly resembled alkali waste, in appearance, but it was more sand-like in character, being impregnated with green and yellow granules.

From the analytical results the composition of the deposit was calculated as follows:—

		<i>Analysis.</i>		
Chloride of Sodium	(NaCl)	..	..	1.54
Sulphate of Sodium	(Na <sub>2</sub> SO <sub>4</sub> )	..	..	5.69
Sulphate of Potassium	(K <sub>2</sub> SO <sub>4</sub> )	..	..	.33
Sulphate of Calcium	(CaSO <sub>4</sub> )	..	..	1.00
Ferrous Sulphate	(FeSO <sub>4</sub> )	..	..	5.46
Ferric Sulphate	(Fe <sub>2</sub> 3SO <sub>4</sub> )	..	..	4.82
Sulphate of Aluminium	(Al <sub>2</sub> 3SO <sub>4</sub> )	..	..	11.23
Water	(H <sub>2</sub> O)	..	..	13.73
Organic and undetermined matter		..	..	.64
				———— = 44.44 % soluble matters
Ferric Oxide	(Fe <sub>2</sub> O <sub>3</sub> )	..	..	4.72
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	..	..	1.24
Silica (sand)	(SiO <sub>2</sub> )	..	..	46.68
Organic matter		..	..	2.92 = 55.56 % insoluble matters
				—————
				100.00 100.00

Magnesium was present only in traces; nitrates were absent; other metals were absent.

The water was quite clear when first received, but after opening the bottles, upon standing, it threw down a heavy deposit. In reaction the water was acid.

<i>Analysis.</i>		<i>Grains per gallon.</i>	
The total solid residue dry at 100° C. amounted to	..	..	275.00
Loss on ignition of residue	.. .. .	..	44.96
Chloride of Sodium (NaCl)	.. .. .	..	19.16
Sulphate of Sodium (Na <sub>2</sub> SO <sub>4</sub> )	.. .. .	..	56.75
Sulphate of Calcium (CaSO <sub>4</sub> )	.. .. .	..	13.72
Ferrous Sulphate (FeSO <sub>4</sub> )	.. .. .	..	33.49
Ferric Sulphate (Fe <sub>2</sub> 3SO <sub>4</sub> )	.. .. .	..	65.80
Ferric Oxide (Fe <sub>2</sub> O <sub>3</sub> )	.. .. .	..	5.40
Sulphate of Aluminium (Al <sub>2</sub> 3SO <sub>4</sub> )	.. .. .	..	27.47
Silica (SiO <sub>2</sub> )	.. .. .	..	6.75
—————			
Traces of Nitrates, Nitrites, Potassium and Magnesium	..	..	273.50

The water contained no free ammonia, and the albumenoid ammonia amounted to 0.05 part per 100,000.

I have not made an examination of the clay underlying the sand through which this water wells, but there can be no doubt that this chalybeate water results from the action of fresh water upon a ferruginous and aluminous soil, containing also much silica, the large amount of which substance held in solution, is possibly due to the acids which are generated in the slow decay of substances of an animal and vegetable character, and which are well known to exercise a powerful solvent action upon silica.

It may be of interest to compare the composition of the Southbourne "Chalybeate" water with some other ferruginous waters, which have been analysed and are known to be of medicinal value.

There is a spring at Trefiew (on the left bank of the Conway, about  $2\frac{1}{2}$  miles from Llanrwst) which yields water, valued as a remedy in the treatment of certain diseases of the digestive organs and the skin, which, according to the analysis of T. Carnelly,\* contains 286 grains of ferrous sulphate, and 95 grains of sulphate of aluminium per gallon: it is free from ferric sulphate, but contains 10.99 grains  $\text{SiO}_2$  per gallon. In this instance there is no difficulty in accounting for the composition of the water, for "the mountains, at the base of which the wells are situated, consist chiefly of beds of limestone, ironstone, alum, slate, and iron pyrites, together with varying proportions of silicates, very much fractured and disintegrated, forming the northern extremity of the Bala or Caradoc beds. Up in the mountains and on these beds lie some small lakes, from which the springs are supposed to derive their principal supply of water, which, after percolating through the above beds and dissolving large quantities of their constituents, finds its exit near the base of the mountain, Alcae Coch, where it issues from the slate bed (Black Band), and between it and the ironstone."

The Orchard Alum Spring yields water containing, according to the analysis of Dr. Thresh,† among other constituents:—

$\text{Fe}_2 \text{SO}_4$	=	174.426	grains per gallon.
$\text{Fe}_2 \text{O}_3$	=	6.275	" "
$\text{Al}_2 \text{SO}_4$	=	72.908	" "
$\text{Fe SO}_4$	=	1.596	" "
$\text{Na}_2 \text{SO}_4$	=	.537	" "
$\text{Mg SO}_4$	=	21.055	" "
$\text{Ca SO}_4$	=	14.381	" "
$\text{Si O}_2$	=	5.776	" "

This spring originates in a disused coal mine, near the summit of Axe Edge, the highest point in the Peak district. The water is used as a vermifuge, and as an external application for various skin diseases. Dr. Thresh writes:—"We have not far to look for the source of the more abundant constituents. The spring arises, or rather issues, from the hillside, immediately above the upper surface of the millstone grit formation, which is here exposed and forms the bed of the rivulet into which the water flows. Overlying the sandstone is a layer of aluminous shale, of a blue grey colour, and doubtless, by the action of the air and water, this suffers such decomposition as to produce the soluble salts found in the spring. The exposed edges of this shale are encrusted with ferric oxide, and here and there, after rain, the water dripping off is seen to be distinctly coloured."

Dr. Thresh adds, that on the opposite side of the hill the water flowing from a similar mine contains 50 grains of crystallised ferrous sulphate to the gallon, with only a trace of ferric salt.

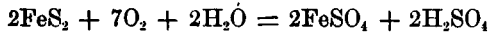
\* *Chem. News*, Vol. xxxi., p. 27.

† *Chem. News*, Vol. xlv., p. 226.

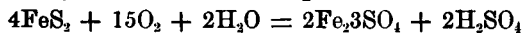


The Flitwick natural water contains, according to the analysis of Dr. Redwood,\* 144 grains of oxide and carbonate of iron, and as much as 59.2 grains of the sulphates of magnesium and sodium. In this case "the surrounding country is flat, and is constituted principally by an undrained valley, the surface of which consists of a crumbly black peat that readily dries, and that is remarkable in containing a large quantity of 'meadow' or 'bog iron ore,' while underneath the peat, there lies, in the immediate vicinity of the well, a stratum of marly clay, and under this again is a deep stratum of sand, known to geologists as the lower green sand, and which ordinarily underlies the chalk. This green sand, which belongs to the Oolitic or Jurassic period, is known to be rich in iron shale, and to be studded with ferruginous nodules, while it owes its colouration to the silicate of iron that enters into its composition."

As regards the Southbourne Chalybeate water, there can be no doubt of the history of its formation from a chemical point of view, and judging from the absence of magnesium, and the small amount of chlorides present, it may be safely concluded that it is derived by the action of fresh water (not sea water) upon ferruginous and aluminous deposits. The formation of ferrous sulphate from pyrites, by the action of water and air, is generally represented by the equation—



and the sulphuric acid thus set free, by its action upon clay and other bases, will produce the sulphates of aluminium and calcium. The sulphate of sodium may possibly be derived by the action of sulphuric acid upon common salt, and its large amount reminds one of the Cheltenham spring water, in which sulphate and chloride of sodium are the predominating constituents. The ferric sulphate is produced by the further oxidation of pyrites, as suggested by Dr. Thresh in the equation—



and by the action of ferric sulphate upon calcic carbonate, ferric oxide and basic ferric sulphate would result coincidentally with calcic sulphate.

The medicinal properties of the Southbourne mineral water must be of a pronounced and valuable character, superior to those of the Trefiew water, since it contains a considerable quantity of ferric sulphate, and at the same time smaller quantities of ferrous sulphate and sulphate of aluminium; superior to the Orchard Alum spring, because, while sufficiently ferruginous, it contains far less sulphate of aluminium, which is an undesirable constituent, and superior to the Flitwick water, inasmuch as it contains twice as much silica, a constituent which is highly prized in the treatment of some diseases. The large amount of sulphate of sodium which the water contains is calculated to obviate certain secondary effects, which the salts of iron and aluminium might otherwise produce in the system.

Dr. Hermann Weber considers that the Southbourne water, properly diluted, "would be likely to act beneficially in many cases of anæmia," particularly if the dilution be effected by admixture with Apollinaris, Vichy, or Vals water. There can be no doubt, also, that in the treatment of other diseases this newly discovered chalybeate

\* Quoted in a Paper by Dr. R. T. Cooper, in the *Monthly Homœopathic Review*, November 1, 1884.

water may be prescribed with great advantage. It is fortunate, moreover, that the locality of the spring is so healthful and beautiful in itself, and this combination of circumstances should secure to Southbourne a great deal of the patronage that has hitherto been extended—almost exclusively—to continental towns. Apart from the medicinal value of its chalybeate spring water, Southbourne offers many advantages to persons suffering from anæmia and lung complaints, for the table-land air is pure and bracing, and the place is surrounded by pine woods, which give to the atmosphere at once a fragrance and a chemical potency for effecting beneficial changes in the sick and jaded.

As regards the supply of the spring water it is difficult to speak with any certainty at present, but the amount is apparently very considerable. The one well which has been sunk is seven feet in diameter, and is thickly cemented at the sides, so that the supply comes only from the bottom. When it contains a depth of five feet of the water, and this is pumped out, it fills again in course of a few hours. None of the water apparently comes from above the clay bed, but it commences to flow about two feet below it, and so far as can be judged the greater quantity flows in five feet below it.

#### SOME ANALYSES OF MILK AND THE DETERMINATION OF MILK SUGAR.

BY C. W. STEPHENS, F.C.S.

*Read before the Society at the meeting on the 17th December, 1884.*

THESE experiments were undertaken in the hope of slightly aiding the Milk Committee of the S. P. A. ; also (at the suggestion of Mr. Kingzett) to ascertain if it is practicable to estimate milk sugar without first separating the caseine. If this can be done, not only will time be saved in the analysis of milk, but it is possible that the estimation of the lactose in milk may serve as an additional standard for determination of purity and adulteration. The "total solids not fat" have not been estimated.

The cream was estimated in a glass cylinder capable of holding 100 c.c., and graduated to one c.c., by allowing the milk to stand in the measure during 24 hours. I find the cream comes up best, when 50 c.c. of the milk are run into the cylinder, and then filled up with water to the 100 c.c. mark.

The specific gravity was taken by weight (with the exception of the first three, in which cases it was taken by the hydrometer) using a sp. gr. bottle of 25 c.c. capacity, and a "Becker's balance," indicating half a milligramme. The "total solids" were estimated by evaporation in a platinum dish, until the weight was constant. After several trial experiments, in which the caseine was first precipitated in the usual manner and removed before the estimation of the milk sugar, I found that, if from 6 to 8 grammes of the milk be carefully weighed in a tared beaker, and then made up to 100 c.c. with water, the sugar can be estimated at once without the separation of the caseine. The method I have adopted is as follows:—

Twenty-five (25) c.c. of "Fehling's solution" (34.65 grammes of sulphate of copper dissolved in 200 c.c. water and mixed with a solution containing 173 grammes of double tartrate of potassium and sodium in 480 c.c., caustic soda solution of sp. gr. 1.14,

and the mixture made up the one litre) is taken and placed in a beaker or porcelain dish; when boiling, the milk solution is run in from a burette (graduated to 0.2 c.c.) until decolourisation is nearly effected, the solution being tested, as usual, from time to time, with potassium ferrocyanide, and addition of the milk solution continued until the mixture ceases to give the red colouration. Most of the results have been controlled by the gravimetric process, which was conducted as follows:—A slight excess of the “Fehling’s solution” was boiled; a known quantity of the milk solution was then added; the precipitate was filtered off, well washed in order to remove the excess of copper solution, and then dissolved on the filter with hot dilute nitric acid, the copper being afterwards precipitated in the usual way, with caustic soda, roasted in a porcelain crucible and weighed as cupric oxide. I have found that the two processes give fairly accordant results. The ash was estimated by burning the “total solids” in a platinum dish; the solids and the ash being determined in duplicate. The lactose was checked as said, or by a duplicate volumetric estimation, 10 c.c. of the copper solution have been estimated as equal to 0.067 gramme milk sugar, dried at 100° c. I also tested the “Fehling’s solution” against pure lactose. I will now give an example of the method followed for determining the lactose:—6.743 grammes milk were diluted with water to 100 c.c.; 58 c.c. of the milk solution were found necessary to reduce 25 c.c.

copper solution  $\therefore \frac{25 \times 100}{58} = 43.1034$  copper solution required for the 6.743 grammes

milk,  $\therefore$  the value in lactose for 100 grammes milk =  $\frac{67 \times 43.1034}{6.743} = 4.28$  grammes

lactose.

Number of experiment	Cream per cent. by measure	Sp. gr. corrected to 60° F.	Total solids dried to constancy	Lactose gravimetrically		Lactose determined volumetrically	Ash per cent. by weight	Remarks
				Copper oxide, per cent., weighed	Lactose calculated therefrom			
1	11	By hydrometer 1.0347	12.75	7.675	4.64	..	.69	No. 1, evening's milk, authenticated.
2	8	1.0304	12.2055	7.527	4.569	..	.71	No. 1, morning's milk, authenticated.
3	13	Coagu.	14.1225	6.8041	4.129	..	.86	No. 2, evening's milk, authenticated.
4	8	1.0389	12.1375	6.90529	4.1904	..	.77	No. 2, done again because of coagn.
5	24	By weight 1.03414	14.8869	6.7522	4.098	..	.77	No. 3, near calving, do not give much milk, morning, authenticated.
6	23	weighed 1.03117	14.7159	7.8682	4.776	4.4423	.808	No. 3, evening's milk, authenticated.
7	12	1.02712	12.8688	6.447	3.9	4.1	.7	No. 4, evening's milk, lately losing flesh, but no particular sign of disease, authenticated.
8	12	1.03053	12.87	7.0347	4.27	4.53	.836	No. 5, morning's milk, authenticated.
9	11	1.02765	13.88?	Lost	..	4.1946	.7	No. 5, evening's milk, authenticated.
10	12	1.02538	14.6142	7.4927	4.581	4.548	.8	Ditto ditto.
11	10	not taken	12.081	7.2371	4.39303	4.2728	.698	From a herd, unauthenticated.
12	12	1.02944	12.629	omitted	..	4.5604	.725	No. 6, morning's milk, authenticated.
13	12	1.02838	12.199	omitted	..	4.16204	.74	From a herd, unauthenticated.
14	16	1.03228	12.99315	6.6922	4.0622	4.208	.708	No. 7 morning, authenticated.
15	12	1.02947	13.4225	omitted	..	4.77174	.715	From a herd, unauthenticated.
16	22	1.02556	14.112	omitted	..	4.08829	.6638	No. 7, evening's milk, authenticated.

The numbers 1, 2, 3, &c., given in the remarks in the table of analyses, refer to individual cows, numbered in that way by the milkman and myself to distinguish each cow. The cows so marked are fairly fed, all under seven years of age. The milk marked from a "herd" is the only mixed milk. "Authenticated," of course means it is known to be genuine, and "unauthenticated," means there is not that certain knowledge; although the samples appear to be genuine from the analyses, saving that some cream may possibly have been removed. As to the determination of lactose by the gravimetric process, I am not perfectly satisfied with the results, because I have had to do the analyses in broken periods of time; I am sure, however, that the results of the gravimetric and volumetric processes could be brought more into accordance. It may be useful to note that, if any "standard" be adopted for milk sugar, it would be useful to use the number 600 c.c. as representing the minimum of "Fehling's solution" that should be required per 100 grammes of milk, this would be better than using a per-centage of lactose, because the number 600 is comparatively large. What I mean is, it takes *about* 600 c.c. of the copper solution for the 100 grammes of milk; thus we get the factors  $\frac{6 \times 67}{100} = 4.02$  lactose.

In calculating from the results of the gravimetric process, I have taken the value of the cupric oxide in terms of lactose from 0.067 gramme per 10 c.c. of "Fehling's solution." Example:—cupric oxide found = 0.595 gramme for 8.458 grammes milk  $\therefore \frac{.595 \times 100}{8.458} = 7.0347$  grammes, cupric oxide for 100 grammes milk; but what is the value of this in terms of lactose?

From the knowledge that 79.5 grammes CuO are reduced by 6.7 grammes lactose, we learn by calculation that 7.034 grammes CuO (got by oxidation of the reduced  $\text{Cu}_2\text{O}$ ) is equivalent to 4.2 lactose.

#### DISCUSSION.

MR. KINGZETT said:—In the ordinary course of professional work, I have been in the habit of estimating the milk sugar volumetrically, and without first precipitating the caseine, as I found this to be unnecessary. The agreement between the volumetric and gravimetric determinations made by Mr. C. W. Stephens is not so entire as could be wished, owing partly, in all probability, to the fact that the work was conducted in broken periods of time and not continuously. At the same time the results approximate sufficiently to show that milk sugar may be accurately estimated by the direct titration of the diluted whole milk. That being so, I think chemists should pay as much attention to this factor, in the determination of the purity or adulteration of milk, as to other factors. At least, as regards watering of milk, the evidence to be obtained by determining the sugar is as important as that furnished by the ash, and certainly more valuable than that of fat and total solids. I can give a case in point which relates to a watered milk supplied to my own household.

The specific gravity of the milk (taken by weight) was 1.025.

<i>Standard taken for Calculation.</i>	<i>Found.</i>	<i>Inference.</i>
·73 Ash	·57	78 c.c. Milk in each 100 c.c.
12.40 Total solids	9.87	79 c.c. " " " "
4.30 Sugar	3.35	78 c.c. " " " "
Solids not fat ÷ .0965	7.75	80 c.c. " " " "

I am aware that some chemists determine milk sugar by precipitating the caseine and weighing the suboxide of copper obtained in the volumetric process, but I should be very strongly inclined to question the accuracy of this method, since the reduced oxide in the presence of alkali very readily absorbs oxygen and becomes converted into the higher oxide. So long ago as in 1872\* I showed that the precipitate obtained by reduction of cupric sulphate with sugar in the presence of potassic hydrate, after washing and drying, is not free from cupric oxide and that the whole of it may be effectually converted into cupric oxide by suspending it in water rendered slightly alkaline, and passing a current of air through the mixture for a few hours. I am satisfied, therefore, that, considering the rapidity of this change and the considerable extent to which the suboxide of copper is exposed to the action of the atmosphere during filtration and washing (while alkali is still present), perfectly accurate results could not be obtained. If it be desired to employ a gravimetric method, the suboxide must be converted wholly into the higher oxide and weighed as such. It would be probably an improvement upon the method followed by Mr. Stephens, to separate, wash, and dry the cuprous oxide, and convert it finally into cupric oxide by merely moistening it with strong nitric acid, followed by ignition in a platinum crucible.

DR. DUPRE said that the estimation might, of course, be of considerable value, but there were very strong reasons for supposing that the amount of milk sugar in milk was not even fairly constant. It was not an uncommon thing to have two samples of milk perfectly genuine, of which one might have twice as much milk sugar as the other. Two years ago he read a paper, giving some experiments, in which the amount of milk sugar varied to an enormous extent. It would require a very large number of experiments to get out any reliable data.

MR. ALLEN described a method for the determination of milk sugar communicated to him by Professor S. P. Sharples, of Boston, U.S.A. The process consisted in treating 65 grammes of the sample of milk with 5 c.c. of the solution of basic acetate of lead employed for clarifying sugar solutions. The mixture was shaken, the liquid diluted to 100 c.c., and at once filtered through a dry filter. If properly done, the treatment produced a perfectly clear filtrate, which gave no precipitate on boiling. The essential condition was to employ *no heat*, or effectual filtration became impossible. A 2-decimetre tube was then filled with the clear solution, and the rotation observed with a polarimeter. With an instrument employing white light, and constructed for use with 26.048 grammes of cane sugar, half the number of sugar-units of rotation gave the

\* *Chemical News*, January 5, 1872.

grammes of hydrated milk sugar in 100 c.c. of the milk.\* The whole operation occupied from 12 to 15 minutes. Mr. Allen suggested that, besides being useful as a means of estimating milk-sugar, the process might probably be advantageously employed for the extraction of the fat, as it would merely be necessary to dry the filter, and treat it in a Soxhlet's tube. A few experiments he had made in this direction had given very encouraging results.

CONCLUSION OF THE PROCEEDINGS OF THE SOCIETY.

MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

ON THE COMPOSITION AND ADULTERATION OF HONEY.—*Zeitschrift für Anal. Chem.*  
24, 135.

THIS is a paper giving the results of investigations which have lately been brought to completion by several German analysts. Among others, those of W. Lenz (*Chemiker Zeitung* 8.613), are of an interesting nature. According to him, the sugar in honey is essentially invert-sugar. The chief adulterants are water and starch-sugar (glucose). To detect these Lenz proposes the following process:—

30 grammes of the honey under examination are dissolved in exactly twice the quantity of water. The Sp. G. of this solution should not be less than 1.111 (at 17°C.), or the honey is adulterated with water. If any considerable quantity of flour is present, it remains undissolved, and can be examined with the microscope. A few starch-grains do not necessarily prove adulteration. For the optic examination, 50 c.c. of the honey-solution are mixed with 3 c.c. of a solution of basic plumbic acetate and 2 c.c. of a concentrated solution of sodium carbonate. The mixture is filtered, and the filtrate polarised in the 220 m.m. tube of Wild's instrument. Pure honey never gives a rotation of less than minus 6° 30'.

Ernst Sieben, in a paper on "Honey and its Adulteration" (*Zeitschrift des Vereins für die Rüben-zucker Industrie des deutschen Reiches*, p. 837), gives four different methods for proving the presence of grape-sugar (glucose) in honey, from which the following has been abstracted:—

(1.) 25 grammes of honey are dissolved in water with 12 grammes of German yeast (free from starch), making in all about 200 c.c. After being allowed to ferment for 48 hours at the temperature of the room, aluminium hydrate is added, and the solution made up to 250 c.c. 200 c.c. of the clear filtered solution are then evaporated to 50 c.c., and polarised in the 200 m.m. tube. Pure honey, after fermentation, leaves no substance exerting an influence upon polarised light; whereas glucose (starch-sugar) produces heavy dextro-rotatory substances, which, on heating with hydrochloric acid, give sugar.

\* Strictly speaking, the amount of milk which should be employed with such an instrument is 65.7 grammes, or 64 c.c. If the instrument be one intended for use with sodium light (the standard weight of sugar being 26.048 grammes as before), 70.27 grammes (or 68 c.c.) of milk should be employed, when each *circular degree* of rotation will correspond to 1.5 grammes of milk-sugar in 100 c.c. of the sample. If 16.35 grammes be the standard weight of sugar intended to be used with the instrument, the most convenient quantity of milk to employ is 61.62 grammes (= 60 c.c.) when one-third of the cane-sugar-units indicated will be the number of grammes of milk-sugar in 100 c.c. of the sample.—A. HILL.

With a Wild's instrument pure honey containing

	5 per cent. starch-sugar	rotates	+ 1.2°
10	„ „ „	„	+ 3.0°
20	„ „ „	„	+ 7.2°
40	„ „ „	„	+ 18.4°
50	„ „ „	„	+ 22.2°

(2.) 25 c.c. of the solution, as used for the polarisation, are heated with 25 c.c. water and 5 c.c. concentrated hydrochloric acid for an hour on the water bath, neutralised, and made up to 100 c.c. In 25 c.c. of this solution the sugar is determined as grape-sugar, according to Allihn's method (R. Fresenius' *Anleitung zur Quant. Analyse*; 6 Edition, vol. II., p. 595). This, multiplied by 40, gives the amount of grape-sugar to 100 grammes honey—

Honey containing 5 per cent. starch-sugar gives 1.472 grammes.

„	„	10	„	„	„	3.240	„
„	„	20	„	„	„	6.392	„
„	„	40	„	„	„	8.854	„

Both methods suffice to detect with certainty an adulteration with starch-sugar above 5 per cent.

(3.) 14 grammes of honey are dissolved in about 450 c.c. water, heated with 20 c.c. semi-normal hydrochloric acid (to invert the cane-sugar), neutralised, and made up to 500 c.c. 100 c.c. Fehling's solution are then titrated with this solution (23–26 c.c. being used), and, according to the result thus obtained, 100 c.c. Fehling's solution are boiled with a quantity of honey solution 0.5 c.c. less than was necessary for the reduction of the whole quantity of copper. The reduced liquid is filtered through an asbestos filter, washed with a few c.c. of hot water, the filtrate neutralised with concentrated hydrochloric acid,  $\frac{1}{10}$  the volume of concentrated hydrochloric added, the whole heated an hour on the water-bath, nearly neutralised with concentrated p. soda (leaving the solution just acid), and finally made up to 200 c.c. The solution on cooling is filtered from separated wine-stone, 150 c.c. of the filtrate treated with 120 c.c. Fehling's solution and 20 c.c. water, filtered and weighed. The grape-sugar is calculated from the weighed copper, according to Allihn. 2 milligrammes Cu is the maximum for pure honey.

Pure honey (with 75 per cent. invert sugar) containing

	5 per cent. starch-sugar	gives	20	millegrammes Cu.
10	„ „ „	„	40	„
20	„ „ „	„	90	„
30	„ „ „	„	140	„
40	„ „ „	„	190	„
50	„ „ „	„	250	„
60	„ „ „	„	330	„
70	„ „ „	„	410	„
80	„ „ „	„	500	„

This method is extremely delicate ; by its use the smallest quantity of starch-sugar can be detected.

Sieben has examined 60 samples of pure bee-honey, and gives the following results :—By titration, with Fehling's and Sach's solution, he obtained for grape-sugar the maximum 44·71 per cent. (to 33·92 per cent. Levulose), the minimum 22·23 per cent. (to 46·89 per cent. Levulose), mean 34·71. Levulose varies from 46·89 per cent. to 32·15 per cent. mean 39·24 per cent.

By titration with Fehling's solution for invert sugar, he obtained the numbers—79·57–67·92, mean 70·30. For cane-sugar 8·22–0 per cent., mean 1·08 per cent.

The total sugar (Dextrose, Levulose, and cane-sugar) varied from 81·74 to 68·91, mean 75·02 per cent. The dried residue was 83·72–75·05, mean 80·03 per cent. ; the non-saccharine substance 8·82–1·29, mean 5·02 per cent.

F. H. H.

---

THE DETECTION OF CAMEL. BY DR. CARL AMTHOR. ZEITSCHRIFT FÜR ANAL.  
CHEMIE. 24. 30.

THE author has founded a method for the detection of caramel in white wines and spirits upon the following reaction :—If para-aldehyde be added to an alcoholic liquid coloured with caramel, a brown precipitate is formed, and on adding sufficient para-aldehyde the liquid is decolourised.

The author proceeds as follows :—To 10 c.c. of the liquid to be examined are added 30–50 c.c. para-aldehyde (according to the intensity of the colour), alcohol being added in sufficient quantity to cause the liquids to mix. (For wine 15–20 c.c. alcohol are sufficient.) If caramel be present, a brownish-yellow to deep-brown precipitate is formed after 24 hours. The supernatant liquid is decanted, and the para-aldehyde washed out with absolute alcohol. The precipitate is then dissolved in hot water, filtered, and concentrated to 1 c.c. The amount of caramel can now be roughly estimated from the intensity of the colour. If only a small quantity of caramel be present in the wine, it must be concentrated over sulphuric acid, and then proceeded with as above. Evaporation by warming is to be avoided, as substances resembling caramel are very easily formed and lead to false results.

The caramel precipitate with para-aldehyde can be confirmed by adding a solution of phenylhydrazin hydrochloride (2 parts of phenylhydrazin hydrochloride, 3 parts of sodium acetate, and 20 parts water) to the filtered solution of the caramel precipitate in hot water. After 24 hours an amorphous brown precipitate separates out. If only a small quantity be present, the solution becomes just turbid, and the precipitate forms after 24 hours.

The solution of the white precipitate formed by adding para-aldehyde to a pure white wine, gives no precipitate with phenylhydrazin.

F. H. H.



## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

## THE PURITY OF IODOFORM.

IN addition to the usual or possible impurities contained in commercial iodoform, which are readily detected by a systematic analysis, or by the special tests given in some pharmacopœias, there appear to be often certain others present, the nature of which is not quite understood, and which have probably not been exactly identified. Dr. Brouma, in Leyden, was led to believe that the discordant results obtained by various experimenters with iodoform were, at least partly, due to the presence of certain impurities; and, after having made a large number of experiments with *pure* iodoform which had been found to stand the (additional) test given below, he found that it did not produce the toxic symptoms reported by others—or, at least, but rarely. The test is the following:—A certain quantity of iodoform is shaken for some time briskly with distilled water, and the mixture then filtered. The filtrate is mixed with a little alcoholic solution of silver nitrate, and the mixture set aside for twenty-four hours. If the iodoform was impure, a black precipitate, consisting of reduced silver, will be formed; while, if the preparation was pure, there will only be a faint whitish-gray turbidity. All samples of iodoform which produced toxic symptoms, when tested in the above manner, reduced the silver solution.—*American Druggist*.

## ADULTERATION OF MENTHOL BY THYMOL.

THE appearance and solubilities of thymol are quite similar to those of menthol, and the adulteration of the latter by the former is asserted. There is no doubt such is the case, and the writer has had opportunities for knowing that such a practice is in vogue. The test for solubility in alcohol of menthol will not answer; and we note that Herr Bernbeck, in the *Pharmaceutische Zeitung*, calls attention to a new test for the presence of thymol. If the suspected article is mixed with four times its weight of sulphuric acid, and thymol is present, there will be developed a deep rose-colour on the application of heat. The solution obtained by this may then be poured into ten times its bulk of water, and digested with excess of plumbic oxide, and filtered. The filtrate, when tested with solution of ferric chloride, will give a violet colour. If the suspected sample of menthol is dissolved in chloroform or alcohol, and heated with solution of potassium hydrate, a red colour will be developed if thymol is present.—*Pharmaceutical Record*.

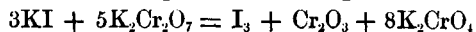
## MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

THE QUANTITATIVE SEPARATION OF IODINE AND CHLORINE IN THE DRY WAY.—*Zeitsch.*

f. Anal. Chem. 24, 96.—By JEAN KRUTWIG.—Ber. d. deutsch. Chem. Gesellsch. zu Berlin, 17,341.

THE author proposed the following method for the separation of iodine and chlorine, which is characterised by the facility and rapidity with which it may be carried out.

If a mixture of potassium iodide and acid chromate of potash be heated together, the iodine is driven off, and the following reaction takes place:—



The iodine and chlorine in a mixture of potassium iodide and sodium chloride can therefore be separated by heating the mixture with acid chromate of potash in a porcelain crucible. The iodine is calculated from the loss of weight, or from the weight of the chromium oxide formed. The latter is determined by warming the fused mass with water in the water-bath; the neutral and acid chromates then dissolve, leaving the insoluble dark green chromium oxide, which is then filtered, dried, and weighed. The chlorine is determined in the filtrate by precipitation with silver nitrate in presence of nitric acid.

F. H. H.

---

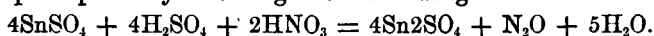
METHOD FOR THE VOLUMETRIC ESTIMATION OF NITRIC ACID.—By ANTONIO LONGI.—*Zeitschrift für Anal. Chemie.*, 24, 23.

THIS method consists in destroying, with a standard tin solution, the blue colour produced by adding diphenylamine to the strongly acid nitrate solution.

The standard tin solution is prepared in the following way:—40 grms. of Marignac's salt—*Ann. des Mines* [5] C. XII. p. 54—(a combination of stannous oxide with potash) was added to 800 c.c. of dilute sulphuric acid (1·1), and brought into solution by adding a small quantity of hydrochloric acid. This solution is titrated according to Fresenius' method—*Anleit. 3 Quantung Analyse*, 6 Ed. Vol. I. p. 291—and sufficient dilute sulphuric acid added to give the solution decinormal strength (1 c.c. = ·0118 grm. tin as oxide).

The method is as follows:—3·5 vols. of pure concentrated sulphuric acid are added to 1 vol. of the nitrate solution. On cooling, one drop of a solution of diphenylamine in sulphuric acid is added. The liquid soon turns deep blue. The decinormal tin solution is then run in until the blue colour disappears and does not return on standing; the liquid is then yellow.

This takes place probably according to the following reaction:—



The method is best suited to the determination of small quantities of nitric acid.

F. H. H.

---

THE DETERMINATION OF WOLFRAM (*Tungsten*). By OTTO FREIHERR VON DER PFORDTEN.  
—*Zeitsch für Anal. Chemie.* 24–92.

THE author has been testing the gravimetric methods for the determination of Wolfram, and has worked out a new volumetric method.

*Gravimetric Methods.*—The substance used for analysis was recrystallized normal sodium tungstate ( $\text{Na}_2\text{QWO}_3 + \text{H}_2\text{O}$ ). The salt was freed from water by heating, and then had the formula,  $\text{Na}_2\text{OWO}_3$ .

The precipitation of tungstic acid with mercurous nitrate, proposed by Berzelius, gives good results. The modifications proposed by C. Scheibler and Wolcott Gibbs, in which ammonia or mercurous oxide are added to aid precipitation, are not necessary. The precipitation should be conducted in the following manner:—To the concentrated solution excess of a concentrated solution of mercurous nitrate is added, the mixture well stirred until the precipitate separates well out, and filtered.

Scheele's method by evaporation with hydrochloric acid also gives good results; the conditions to be observed are the same as for the estimation of silica.

*The Volumetric Method.*—The method consists in reducing the tungstic acid with zinc in acid solution, and titrating with standard permanganate of potash. The author's experiments show that the reduction takes place best in hydrochloric acid solution, as sulphuric acid and dilute hydrochloric produce a blue-black turbid solution. Reduced with hydrochloric acid and zinc, the solution undergoes the following changes of colour:—Blue, black, blackish-green, and finally, after about ten minutes, deep brown-red.

The process is the following:—The solution of the salt (containing 19  $\text{Na}_2\text{O} \cdot \text{WO}_3$ ) is warmed with a small quantity of water on the water bath, and 70–80 c.c. of a 27 per cent. HCl solution, together with 14–15 grms. zinc (in large pieces), added. The reaction takes place rapidly, and does not require to be supported by warming. As soon as the solution has assumed the red colour, the flask is cooled, and the contents carefully washed into a porcelain dish, which already contains excess of permanganate solution, 70–100 c.c. dilute sulphuric acid, and 40 c.c. of a solution of manganese sulphate. The whole is then diluted to about a litre, excess of a solution of ferrous sulphate run in, and the mixture finally titrated with permanganate until the liquid assumes a pale pink colour.

F. H. H.

THE DETERMINATION OF MOLYBDENUM.—By OTTO FREIHERR VON DER PFORDTEN.—  
Ber. d. deutsch. Chem. Gesellsch. Zu Berl. 17,731.

THE Author communicates that both the trisulphide and disulphide of molybdenum can be converted into metallic molybdenum by reduction in a stream of pure and dry hydrogen. The reduction can be made in a platinum or porcelain tube; or a Rose's crucible may be used. This discovery is in opposition to Rose's views, (Gmelin-Krant Handbuch der unorganisch. Chemie. II. 2, 175,) who maintained that the disulphide remains unaltered in a current of hydrogen; this, however, is the case only when the hydrogen has been insufficiently dried. Besides the precipitated sulphides, native molybdenum sulphide can also be reduced in this way, the reduction being naturally somewhat slower. The author reduces the finely divided substance in a platinum dish, under a current of hydrogen, with the extreme heat of a good blowpipe.

Since molybdenum sulphide thus permits of further reduction, it is necessary to take some precautions in the estimation of molybdic acid, according to L. Paul Liechti and Bernhard Kempes method (Zeitsch. f. Anal. Chemie 23, 416,) which consists in reducing the trisulphide to disulphide in a current of hydrogen; and as the method requires considerable time and care, the author proposes the following modification:—The dried precipitate is separated as far as possible from the filter paper, and brought into the crucible, the filter paper folded and burnt. The whole substance is now reduced to metal in a current of hydrogen, a platinum crucible being used in preference to a porcelain one. This method is analagous to the author's method for the reduction of molybdic acid to molybdenum, to which I therefore refer for details. See the Analyst, IX., p. 203.

## REVIEWS.

**LIST OF TESTS (REAGENTS) ARRANGED IN ALPHABETICAL ORDER ACCORDING TO THE NAME OF THE ORIGINATORS.** Translated from the German of *H. Schneider*, by *H. M. Wilder*. New York: P. W. Bedford, 36, Beekman Street.

THIS unpretentious little pamphlet (reprinted from the *New York Pharmaceutical Record*) is really, in its way, a happy thought. Every one has, no doubt, felt (especially in reading abstracts from foreign journals) that now and then he comes to a mention of some test under the name simply of "so-and-so's reagent," and he has to pause for a moment and think, "Let me see, what is that?" With this little book on the table, no difficulty will be experienced, as the information is already to hand. We should fancy that it is just one of those little brochures which would have a considerable circulation as a most convenient ready reference.

**THE LIFE WORK OF CARL WILHELM SCHEELE.** A Paper read before the Alumni Association of the College of Pharmacy of the City of New York, by *B. Frank Hays*, Ph.G. New York: P. W. Bedford, 36, Beekman Street.

THIS is a well written and interesting pamphlet, also reprinted from the *New York Pharmaceutical Record*.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

TO THE EDITOR OF "THE ANALYST."

SIR,—The enclosed report of a prosecution under the Food and Drugs Act contains a point of considerable interest both to Analysts and Inspectors. The assistant to the Inspector, in making the purchases, asked for and obtained a bill in each case, and the production of these bills in court settled the matter at once. If this method were generally adopted in obtaining such samples as butter, coffee, &c., a good deal of hard swearing as to what was asked for would be prevented, and I think convictions in such cases more easily obtained. The idea is entirely due to Inspector H. Wharton. The report is from the *Cheshire Observer*.

Chester, January 19th, 1886.

I am, Yours truly,

W. F. LOWE.

[The report of the case enclosed by our correspondent is too long to admit of insertion in full, owing to the press of other matter; but it simply amounts to what is stated in Mr. Lowe's letter, viz.:—that through the Inspector's sharpness in asking for a bill in which the article was described "butter" all the usual excuses fell to the ground and conviction followed. ED. ANALYST.]

## TO CORRESPONDENTS.

MR. ABBOTT (Massachusetts), is thanked for his letter and enclosure, both of which will be referred to at an early date.

MR. P. W. BEDFORD will see that a part of his communication has been anticipated. Many thanks for enclosure in first letter which will be reciprocated by post.

MR. E. WALLER's communication received, and will be replied to by post.

MESSRS. A. C. WILSON and A. PERCY SMITH are thanked for their papers, which are under consideration.

MR. J. MACALLAN will be answered by post.

## APPOINTMENTS.

SALE OF FOOD AND DRUGS ACT. Mr. Bernard Dyer, F.I.C., F.C.S., has been appointed Public Analyst for Southend.

## BOOKS, &amp;c., RECEIVED.

The Life Work of Scheele, by B. Frank Hays, Ph.G.; List of Tests (Reagents), by H. M. Wilder; Report (Fifth Annual) of the Board of Health of Massachusetts; Report on Fresh and Condensed Milk, by C. C. Mansall, Ph.D.; American Chemical Review; American Druggist; American Grocer; British and Colonial Druggist; Brewers Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewers Guardian; Cowkeeper and Dairyman's Journal; Independent Journal; Invention; Journal of Chemical Industry; Journal of Microscopy and Natural Science; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Publishers' Circular; The Sanitarian; San Francisco News Letter; Science; Scientific American; Scientific Californian; Society of Arts Journal; Students' Journal.

## NOTICE TO CONTRIBUTORS.

All Letters and M.S.S. intended for the literary part of this Journal should be addressed to the Editorial Department, 325, Kennington Road, London, S.E.

# THE ANALYST.

MARCH, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—	
(a) REPORT OF MEETING . . . . .	41	DISTINCTION BETWEEN RICE AND BUCK-	
(b) INAUGURAL ADDRESS, By THE PRESIDENT	41	WHEAT (REST POSTPONED) . . . . .	60
(c) A NEW METHOD FOR THE ANALYSIS OF MILK, By M. A. ADAMS, F.R.C.S. . .	46	MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
(d) VALUATIONS OF MILK SOLIDS, INSTEAD OF A LIMIT OR STANDARD, By C. ESTCOURT, F.I.C. . . . .	54	NEW TEST FOR ALOES—CRIPPS AND DYMOND	60
ORIGINAL ARTICLES:—		MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
(a) ON A SIMPLE APPARATUS FOR THE ESTIMATION OF FAT IN MILK, By A. C. WILSON, F.C.S. . . . .	55	(ALL POSTPONED THIS MONTH) . . . . .	60
(b) ON COCAIN, By A. P. SMITH, F.I.C. . . . .	55	NOTICES TO CORRESPONDENTS . . . . .	60
(c) ON THE ANALYSIS OF CONCENTRATED MEAT PREPARATIONS, By DR. A. STUTZER . . . . .	57	BOOKS, &c., RECEIVED . . . . .	60

## SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of this Society was held at Burlington House, on Wednesday, the 11th February, the President, Dr. Hill, in the chair.

The minutes of the annual meeting were read and confirmed.

Dr. Alder Wright and Mr. Baynes were appointed scrutineers, and on opening the ballot papers they reported that the following gentlemen had been elected:—

As member:—E. Seaton, M.D., M.R.C.P., Public Analyst for Chelsea.

As Associates:—W. W. Dry, Assistant to Mr. Eastick and G. H. Gemmell, Assistant to Dr. Campbell Brown.

The following gentlemen were proposed for election at the next meeting:—

As Members:—J. Ruffle, M.R.A.C., Analytical Chemist, Plaistow, and R. Bodmer F.C.S., Analytical Chemist, London.

As Associate:—W. Hogben, Assistant to Dr. Drinkwater.

The following papers were read and discussed:—

“On the determination of mixtures of Milk Sugar and Cane Sugar,” by A. W. Stokes, F.C.S., and R. Bodmer, F.C.S.

“Note on an Adulterated Sample of Tea,” by Dr. Bostock Hill, F.I.C.

“On the Examination and Composition of Milk and Milk products,” by Dr. P. Vieth, F.C.S.

The next meeting of the Society will be held at Burlington House, on Wednesday, the 11th March, when the discussion on Mr. Adams' paper, read at the Annual Meeting, will be resumed, and other papers will be read.

## INAUGURAL ADDRESS, BY DR. HILL, PRESIDENT.

(Delivered at the Annual Meeting, 28th January, 1885.)

In assuming the chair, as your president for the coming year, my first duty and my first impulse are to express to you my best thanks for the honour you have done me in electing me to the office, an honour which I greatly appreciate on many grounds, and not least because this is the first occasion on which a provincial member of our Society has been so elected. There are, of course, many reasons why metropolitan members should have been chosen in the past, particularly in the early part of the Society's existence, when matters of a pressing nature required that prompt and frequent attention which could necessarily best be given by gentlemen living on the spot, and there cannot be a doubt that the various duties of the officers have been most ably and efficiently performed. To this fact I am glad of the present opportunity of bearing testimony.

Again, it might naturally be supposed that members living at a distance would be either unable, or unwilling, to undertake the duties of the honorable office of President if elected, and the Society might, therefore, having done so well hitherto, have preferred to proceed "per antiquas vias."

The Society has, however, now happily got safely through the feebleness and dangers incidental to infancy, and may truly be said to have attained a healthy and vigorous adolescence although dating only from ten years back, so that it is now doubtless felt that the time has come when all the privileges and honours which the Society has to bestow, without exception, may be, under certain conditions, extended to all its members. I trust this new departure may be, as I believe it will be, justified by the results. In any case I am desirous of acknowledging the compliment paid to myself, and through me to the whole body of provincial members, and to say, on my own behalf, that I shall feel bound to do my best to perform the duties which the acceptance of office imposes, and do all in my power to preserve the honour and interests of the Society and to promote its usefulness.

In addressing you from this chair, I am forcibly reminded that your late President would, had he lived, have been occupying it probably at the present moment, and I am sure I am expressing your sentiments as well as my own when I say how greatly I deplore his untimely decease. Mr. Wigner had been so closely identified with the Society from its commencement, and had been so uninterruptedly bound up with its fortunes and its labours, he had especially so much contributed in various capacities to its success, that everyone of us must be deeply sensible of the irreparable loss which we have sustained by his death. He was constitutionally endowed with great energy, industry, zeal and ability, and he employed his faculties with characteristic devotion to the advancement of our special department of chemical science, and the general interests of our Society. Whether engaged as an original promoter and founder of the Society, as Secretary from its commencement to his resignation of the office in 1883 in order to assume the Presidency, as editor, either sole or conjoint, from the publication of the *Proceedings* in 1875, and of *THE ANALYST* from its first publication in 1876 until the year of his death in 1884; as a practical worker on many of the principal articles of Food and the Water supplies of London and other towns, or in the extra-professional, but not less useful duty

of contributing suggestions and information for the improvement of defective Adulteration Acts, in all these capacities and some others, I say, Mr. Wigner was conspicuous for his earnestness, his ability and his usefulness. The regret felt for his too early removal in the prime of his manhood, and when his ripe experience was calculated to be increasingly beneficial, is tempered by the reflection that he has done so much good work and has left behind him a record of it, and the benefit of his example for those who remain, and will succeed him. "Requiescat in pace."

Few societies can lay a stronger claim to the *raison d'être* than ours, for it is concerned with a department, or an application of chemico-physical science which, until thirty years ago, had received comparatively little attention, not indeed until the labours of Dr. Hassall attracted public notice to the extensive and dangerous character of Food adulteration and impurity, resulting in the passing in the year 1860 of "An Act for preventing the Adulteration of Food or Drink."

The work done by Dr. Hassall was not only very valuable in educating the public mind, and so conducing to the passing of the first Adulteration Act, but it formed a basis of operations and a guide to those who were disposed to study the subject of Adulteration, particularly to the analysts first appointed under the Act, who were very few in number, and who would have had much difficulty in performing their new duties, in the absence of the Reports of the Analytical Sanitary Commission, and of the work which followed under the title of "Adulteration Detected." I have a lively recollection of the interest with which I studied the Reports in the *Lancet* as they appeared from week to week, illustrated by excellent engravings of the microscopical characters of the various foods, and their adulterants. I was so interested in the novel subject, that I gave several public lectures on it more than five and twenty years ago, and this early interest in it was, probably, one of the causes of my being elected to my present appointment of Analyst to the Borough of Birmingham in the February (1861) of the year following the passing of the Act; so that I am probably the oldest Public Analyst living, and I am not sure that I was not the first appointed under the original Act.

I trust you will pardon me referring to events and recollections so personally interesting to myself, but I shall ever look back with appreciation and a feeling of deep indebtedness to the labours of Dr. Hassall. They possessed a novelty, an originality and a value, and they evinced a courage, an ability and an exceptional knowledge of a new, difficult, and many-sided subject which cannot fail to elicit our respect and admiration.

Excellent and useful as were these early labours in their day, there was naturally much room for improvement of chemical processes. This is especially the case with regard to such articles as Milk, Flour and Bread, Butter, Coffee and Tea, which constitute the principal portion of our dietary, and which are, therefore, tempting objects of falsification to the unprincipled trader. It has been the privilege of members of this Society to simplify and perfect the processes of examination of these articles, and to give to them an uniformity and an exactness which enable them to compare, not unfavourably, with some results of ordinary mineral analysis.

Uniformity of procedure can only be secured by the concerted labours of a number of workers, and the detailed description, comparison, and discussion of the processes employed. Without such criticism and frequent interchange of views and experience, Public Analysts would, by adopting different methods, inevitably obtain discordant results; their work would appear untrustworthy; they would have less weight in Courts of Law; and, what is still worse, they would often find themselves pitted against each other in most undesirable opposition. But, since several analyses of the same sample of milk, even in different hands—thanks to the excellent and simple process of Wanklyn, by which he discovered that the amount of solids-not-fat forms a constant factor, not only in the case of whole Milk, but also in that of skimmed Milk, and even in that of Cream—are found to agree, even to the decimal places of figures; since Alum can not only be detected in Flour and Bread with ease, but be determined with certainty by the well-devised process of Dupré; since Butter and other fats can be made to yield their acids with great precision by the ingenious method originally proposed by Hehner and Angell, and supplemented, elaborated, and perfected by Dupré, Muter and others; since sufficiently close results for practical purposes can be obtained in the analysis of Coffee and its mixtures; and so with some other important articles of Food and Drink, we have the comfortable assurance that, in the hands of the Analysts of experience and position, concordant results may be obtained, and so unseemly differences and conflicts among them in matters of procedure and interpretation be avoided.

This desirable result was illustrated in my own practice, some months ago, when our late President was brought down to Birmingham, on very short notice, to contest my certificate and oral evidence in a milk case; on going into court, I was considerably surprised, and a little annoyed to see Mr. Wigner sitting at the elbow of the opposing Counsel, feeling as I did that the only object of his being there could but be intended opposition to my evidence. To my surprise, however, my friend was not called for the defence, and I have no doubt, that he advised Counsel, after hearing my evidence, that the defence could not be sustained. I attribute this satisfactory result to the perfection of the method of milk analysis, and to the fact that the chemical witnesses were not "incompetent" analysts.

These facts, then, with many others, into which time will not allow me to enter, constitute our *raison d'être*, and practically and amply justify our act of association ten years ago. Such advances in food analysis, such satisfactory practical results could not possibly have been secured if the Public Analysts of this country had not formed themselves into an united body, affording another illustration of the axiom that "union is strength."

England was not only the first country to move in the direction of repressing adulteration, but her chemists, who at the same time are or have been members of this Society, have had the distinction of being foremost in the invention and perfection of processes for the analysis of articles of food and drink, processes so satisfactory that they have not been superseded by anything done since in any other country, although the subject has been industriously pursued by many able chemists in all parts of the world.



But, though England was the leader in the anti-adulteration crusade, she has been so well followed by France, Germany and America, that there is a danger that our pupils "will better the instruction" of their teacher; and, owing to our defective legislation, and that morbid sympathy which, in this country, is tending constantly more and more to protect, or let go, the wrong-doer at the expense of his victim, will have a better system of adulteration-prevention than we have ourselves. It should continue to be an object of our Society to embrace every opportunity of improving our parliamentary Act; and, above all, we should strive to keep at its highest level the scientific character and work of our members, without regard to other considerations. While admitting, according to our written law, "all analysts in actual practice," we must be careful that our organisation protects practical men from the intrusion of mere theorists, and competent chemists from ambitious, but unqualified aspirants. In this way only is the ability and reputation of our Society to be upheld.

It is customary, at the annual meeting, to give a statement of our position, which, through the kindness of one of our honorary secretaries, I am able to do.

During the past year thirteen members and five associates have been elected, while two associates have become members.

Against this increase of 18, we have to place a decrease of 6 members; 4 by death, viz., Dr. Angus Smith (honorary), Mr. Robert Harvey, of Madras, Mr. La Rue, of Quebec, and our late President, Mr. Wigner; and two by resignation—Mr. McAlley, of Falkirk, and Mr. de Koningh, of London.

Our Society is thus larger by 12 members and associates than it was twelve months ago, our total roll being 173, made up of 10 honorary members, 142 ordinary members, and 21 associates.

From the nature of our work, and the conditions of membership, it is not likely that our number will ever be very large, but I don't know that that is any matter for regret so long as the quality of our members continues of the desired standard; it is quality, and not quantity, that is most to be aimed at, and above all things we must not, in our wish to extend our subscription list, lose sight of this fact, as no number of additional subscriptions would compensate for the disadvantages entailed by a departure from this policy; but our actual requirements being moderate, there would, I believe, be no difficulty on this point if all our members would be good enough to promptly pay their subscriptions.

During the year 18 papers were read at our meetings which were fairly well attended, and the papers were of considerable utility and interest, as was proved by the animated discussions to which they gave rise.

Some very interesting subjects were introduced and discussed in connection with food at the International Health Exhibition held last year, a full account of which appears in the last volume of THE ANALYST, and I must not close my remarks without referring to the novel exhibit of our Society, arranged, if not suggested, by our late President, Mr. Wigner. This interesting and valuable collection included substances used as adulterants, adulterated articles of food sold in England, examples of articles whose adulteration has been suppressed, adulterations found to prevail in foreign

countries, and such mixtures as are protected by labelling. It is probable that this display did not receive its due share of attention, seeing that it did not form a part of the exhibition until towards its close, but I understand that it was considered by competent judges to possess much value, and that it is to be preserved with a view to its further usefulness. A practical and gratifying proof that the exhibit was appreciated by those competent to judge, exists in the fact that it was awarded the diploma of honour.

The labours of the Committee appointed to make concurrent experiments on the same samples of milk with the various processes in use in its analysis, with a view to obtain the greatest possible uniformity of procedure and results, have not yet been brought to a close. Everyone must admit the desirability of such uniformity of procedure in complicated and difficult analyses, especially in the case of certain natural organic and animal products, the composition of which has a certain range of variation. Absolute uniformity of results is, of course, not to be expected between different operators, if from no other cause than the disturbing influence of the "personal equation," as it is called, but given a good process, the main condition necessary to obtain practically concordant results by it in different hands is scrupulous care.

In conclusion, I consider we have good cause to be satisfied with our progress during the past ten years, and I have only to express the hope that our success will be at least as great during the next decade.

---

#### ON A NEW METHOD FOR THE ANALYSIS OF MILK.

By MATTHEW A. ADAMS, F.R.C.S., F.C.S.

(*Read at the Meeting on 28th January, 1885.*)

TWELVE months ago, I was possessed of the belief that the analysis of milk, for the purposes of the Food and Drug's Act, was simple, easy, and accurate. I then supposed that any twelve practical men might easily arrive at concordant results on the same sample, and that there was no ground for discrepancy of opinion as to the interpretation of results, except such as arose from the diverse views held by different authorities, as to the natural limits of variation of genuine milk.

This comfortable belief was somewhat shaken by the Manchester trial, since which a Milk Committee was, as you know, appointed by this Society, for the purpose of enquiring into the whole subject of milk analysis. Having taken part in the labours of that Committee, I was soon convinced that although any one man, working in his own special way, with his own special apparatus, might, for himself, obtain concordant results, there is no such concordance to be expected between different workers employing different processes, or may be, slightly different forms of apparatus, or merely the personal equation even, is enough to create discordance. This being so, it follows that there can have been, not only no common ground for a judgment of the quality of a given sample of milk, but it is also proved that most, perhaps all, of the processes hitherto employed fail in point of absolute accuracy.

I say this conviction has been brought about by a study of the results of analyses undertaken on samples of the same milks, by the members of the Committee, each

member working in his own way. Of course I am not at liberty, or in a position to anticipate the conclusions hereafter to be reported by that Committee, yet in what I shall have to say, I cannot altogether avoid some allusion to the results of our joint work, for which I hope to be excused, holding myself responsible for any opinions herein expressed.

Well then, the general outcome of our investigation, upon which I wish to fix your attention is:—

1st. That fairly concordant results, as to the *Total Solids*, are the rule.

2nd. That the results as to the *Fat* differ very widely with the process employed, and the operator.

3rd. That this difference is less in samples rich in fat, but far greater in skim milk.

4th. As a matter of course, the disagreement in the solids-not-fat, vary in like proportion with the disagreement in the fat, only in the opposite direction.

In short, it has been shown that there is a great difficulty, especially in poor milks, in completely removing the whole of the fat from the solids-not-fat, the reason of which is pretty clear; it is, there is no doubt, because a rather considerable residue of fat becomes enveloped and imprisoned in the dried proteids in such a way that the solvent cannot get at it, and, in accordance as the milk is rich or poor in fat, on drying the solid residue becomes spongy, or horny, and in proportion presents more or less facility for the extraction. Those who employ Plaster of Paris, sea sand, or mechanically granulate, or otherwise treat the milk solids, so as to secure more perfect access for the solvent, practically acknowledge this difficulty; yet notwithstanding the various schemes in use, no two workers seem to attain to concordant results, even when working on the same scheme, so much depending upon minute details of working; indeed, as I shall endeavour to show, it appears that a certain residue of fat, is in almost every case left behind.

It was in these circumstances that it occurred to me to seek for an improvement which might be depended upon for absolutely exhausting the solids of the last trace of fat by some simple expedient that could be easily worked by any number of competent persons.

The method I am about to describe, accomplishes this thoroughly, and in a most simple way.

Reflecting upon the fact that rich milks are much more easily exhausted than poor milks—and that this must be due to the different physical conditions of the residues—once naturally sought to bring about the most extended surface and spongy texture possible for the deposition of the total solids.

With this end in view, I took 5 c.c. of milk and discharged it on a level surface of glass, to ascertain the area it would extend itself over, by mere gravity, this I found to be somewhere about 97 square centimetres. Repeating the experiment on a surface of blotting paper it spread itself over a space of about 213 square centimetres, and seeing that, in the case of blotting paper we have two exposed surfaces, this figure requires to be doubled, making 426 square centimetres, and still there remains to be taken into

account, the bibulous spongy structure of the paper, which of necessity furnishes a further very large extension, not easy to compute.

I find the actual density of the paper = .485, hardly a third of the sp. gr. of cellulose, which is reckoned at 1.5; moreover by measurement I have ascertained that the ream of 480 sheets is  $5\frac{1}{2}$  inches thick, giving a thickness for each sheet of .01148; the separate paper fibres average .000888 of an inch in diameter, so that it takes about 13 fibres to constitute the thickness of the paper, from which I conclude that at least we must have an available surface of about 5538 square centimetres, or let us say 450 times more than when a platinum capsule, as described by Wanklyn, is used. Similar calculations show the thickness of the milk film not to be more than .0089 of a millimetre on the paper, whereas on the glass it is .515, or in the platinum capsule 4.0 millimetres, that is to say a trifle more than 450 times thicker than on the paper.

It may well be then that if we can deposit our milk solids with this extreme degree of tenuity there must cease to be any difficulty for the solvent to reach the fat, and this has proved itself to be the case.

At first this was all I proposed, or even hoped for. I was very doubtful of the use of paper for anything that involved weighing, only intending it as a framework on which to spread out the solids; however, experience soon convinced me that the solids-not-fat could also be estimated with the greatest accuracy upon the paper after the fat had been removed, and with due and proper precautions, even the total solids, could be got at with very fair precision. I am not however, disposed, to place too much reliance upon the direct estimation of the total solids, in my opinion it is preferable to add the fat and solids-not-fat together, and so get the total solids, for the reason that I find it is practically impossible to hit with certainty the moment when the total solids are quite dry, and before the fat begins to oxidise, for the latter event follows immediately on the former. I am accustomed, therefore, first to rough dry the total solids, without attempting, or even wishing, to get rid of all the water, then to extract the fat, and finally thoroughly dry the solids-not-fat, which give all the necessary data, except the ash and the specific gravity.

It is not to be supposed that we got to a completely satisfactory issue in this matter all at once; naturally enough we have had failures, and had to feel our way through difficulties, till now we feel ourselves able to offer you a process, which, if conscientiously carried out, may be trusted with the greatest confidence, and which I now proceed to describe in detail.

No special apparatus whatever, is really necessary; I have however found it very convenient to adopt some simple things, which greatly facilitate the manipulation, and they are here exhibited. As for material, the only extra article is some stout white blotting paper, known in the trade as "*white demy blotting*," "*mill 428*" weighing 38lbs. per ream; this should be in unfolded sheets, machine cut into strips,  $2\frac{1}{2}$  inches wide, and 22 inches long, each sheet in this manner cuts into 7 strips.

I have tried other papers, but none have answered so well as this, it is very porous and just thick enough. Each of these strips is carefully rolled into a helical coil, for which purpose I use a little machine, made by myself, consisting of a stout double wire,

cranked twice at right angles, and mounted in a simple frame, one end of the strip being thrust between the two wires, the handle is turned, and the coil made with great facility. This may be done, for the nonce, on a glass rod, the size of a cedar pencil. Two points have to be carefully attended to—the paper must not be broken, and the coil must be somewhat loose, the finished diameter being a little under an inch. I am in the habit of rolling up a considerable number at a time, and placing each within a brass ring as it is rolled, inscribing on one corner with a lead pencil its own proper number.

These coils are next thoroughly dried, and I need hardly say, the accuracy of the process depends upon this drying. This can be satisfactorily done in an ordinary air-bath at 100°C., providing the bath be heated properly, and the papers kept in it long enough. I found the common way of heating the thin bottom of the bath with a single jet, not to answer; my bath is placed upon a stout iron surface, which is heated by a large ring of jets; in this way the heat is evenly distributed over the whole of the bottom of the bath, and the papers which are put in a cage frame of tinned iron wire  $5 \times 2\frac{1}{2}$  inches, and divided into 8 partitions, get evenly and completely dried, if allowed to remain in the bath all night, and weighed in a weighing tube next morning, and their weights having been registered, according to their numbers, stored away ready for use as follows:

The milk to be examined is shaken, and with a pipette 5c.c. are discharged into a small beaker, 2 inches high by  $1\frac{1}{4}$  diameter, of a capacity of about 30c.c. weighing about 12 grams. This charged beaker is first weighed, and then a paper coil gently thrust into the milk very nearly to the bottom, in a few minutes the paper sucks up nearly the whole of the milk. The paper is then carefully withdrawn by the dry extremity of the coil, and gently reversed, and stood, dry end downwards, on a clean sheet of glass, with a little dexterity all but the last fraction of a drop can be removed from the beaker and got on the paper. The beaker is again weighed, and the milk taken got by difference. It is of importance to take up the whole of the milk from the beaker, as I am disposed to consider the paper has a selective action, removing the watery constituents of the milk by preference over the fat.

The charged paper is next placed in the water oven on the glass plate, milk-end upwards, and rough dried, mismanagement may possibly cause a drop to pass down through the coil on to the glass; this accident ought never to occur, but if it does it is revealed in a moment by inspection of the surface of the glass, and the experiment is thereby lost.

In about an hour it is rough dried, and in a suitable condition for the extraction of the fat, I employ a Soxhlet extractor, using a wide mouthed flask weighing 15 to 25 grams., standing  $4\frac{1}{2}$  inches high, and having a capacity of 150 to 180c.c.; a dozen syphonings off of the solvent (I use ether or light petroleum indiscriminately) completely washes the fat out of the paper. The paper is now removed from the extractor and set to dry in the wire cage in the air bath; meanwhile the solvent is distilled off from the fat, the latter dried in the flask, air being blown through occasionally, then weighed: The solids-not-fat must be thoroughly dried in the air bath, the fat having been removed there is no longer any fear of oxidation, so that it may be got to a constant weight, in a properly arranged air bath kept at 100°C., in 3 hours at the most.

When it is desired to get the total solids direct, my experience has taught me to expect good and reliable results in an ordinary bath with 3 hours drying; less than that is not enough, and very soon after 3 hours the solids begin to increase rapidly in weight, so that I recommend this time to be strictly adhered to, and door of the bath on no account to be disturbed during that interval. This however is a matter in regard to time, which is controlled by the particular bath, and depends upon the air current, and the position of the paper with relation to the air current; so that it is necessary to regulate the time, and position of the paper to suit the peculiarities of the particular bath, and it is these circumstances which make the direct estimation of the total solids so touchy. I have, however, experiments in hand, which, I feel confident, will soon dispose of this difficulty.

As already said, it is, at present, better to compute the total solids by the addition of the figures obtained by the direct determinations of the solids-not-fat and the fat.

We will now take a few examples, and compare the results obtained by my method with those got by other methods.

To begin with, let us take one of our earliest experiments, it refers to a sample distributed among 13 members of the Milk Committee, November 26th, and was analysed in three several ways; more or less by all of us, and also by myself in my own way.

The average of the *total solids* by the ordinary method of drying in platinum capsule=13.66 with max. 13.82 and min. 13.51.

By a special *method of Carter Bell's* the mean=13.78, max. 13.94 and min. 13.63.

By my method in six consecutive experiments

13.24	13.26	}	Mean=13.263
13.23	13.21		Max.=13.36 and Min. 13.21
13.28	13.36		

These figures were got by the direct weighing of the total solids on the paper, and you will observe they agree well among themselves, and if we exclude the last, which, I believe, was oxidised, remarkably well, and are .397 below the mean of the ordinary method, and .417 below Carter Bell's.

Now for the *fat*.

By the *ordinary method* the mean=4.01, with a max. 4.22 and min. 3.74.

By *Carter Bell's* process the mean=3.94 with max. 4.25 and min. 3.50.

By *Plaster of Paris* in Soxhlet the mean=4.25 with max. 4.63 and min. 3.71.

By my method

4.62	}	Mean=4.62
4.64		
4.60		

These figures, I think it must be admitted, agree very well, and they show an excess of fat over the old method of .61 and .67 above Carter Bell's, and .37 above the Plaster of Paris.

The *solids-not-fat*.

By the *ordinary process*=9.63 with max. 9.77, min. 9.50.

By *Carter Bell's* method=9.80 with max. 10.10, min. 9.47.

By my method

8.88	}	Mean=8.85.
8.79		
8.88		

that is .78 below the ordinary, and .95 below Carter Bell's.

Reckoned by summation of the fat, and the solids-not-fat, the total solids amounted to

13.50	}	Mean=13.47.
13.43		
13.48		

or as will be observed

.26	}	Mean=.22
.20		
.20		

above the amount by direct experiment.

I may as well here remark that the total solids, always come out by direct experiment about .2 per cent. lower than when calculated by the addition of the fat and the solids-not-fat, and this occurrence is in no way specially connected with the new process, it is common to other methods as well. I do not, however, propose to deal with this point on the present occasion, I have my own views on the subject, and if I may be favoured with a further hearing at a future time, it will give me much pleasure to submit those views to the Society, together with other analogous points which have presented themselves in the course of this investigation, for the present we are only concerned with a consideration of this process as a new method of manipulation.

We will now, if you please; take some examples of *skim milk* which, in common with Dr. Vieth, and partly also with Mr. Baynes, have been analysed in four different ways, Dr. Vieth extracting the fat by means of Plaster of Paris in a Soxhlet, Mr. Baynes, by a modification of his own, and I by the old method in platinum capsules, calculating the fat indirectly by the difference between the solids before and after extraction with petroleum spirit, and also by the new method.

No. 1

	Total Solids.	Solids-not-fat.	Fat.
Vieth.	10.024	9.485	.539
Adams. {	Old.	9.91	.12
	New.	9.67	.71
Baynes.	—	—	.77

## No. 2

	Total Solids.	Solids-not-fat.	Fat.
Vieth.	10·318	9·595	·723
Adams. { Old.	10·21	10·08	·13
{ New.	10·39	9·37	1·02
Baynes.	—	—	—

## No. 3

	Total Solids.	Solids-not-fat.	Fat.
Vieth.	9·803	9·414	·389
Adams. { Old.	9·665	9·55	·11
{ New.	9·28	8·76	·58
Baynes.	9·79	9·27	·52

## No. 4

	Total Solids.	Solids-not-fat.	Fat.
Vieth.	9·894	9·558	·336
Adams. { Old.	9·63	9·50	·13
{ New.	9·24	8·70	·53
Baynes.	9·75	9·20	·55

## No. 5

	Total Solids.	Solids-not-fat.	Fat.
Vieth.	9·73	9·451	·279
Adams. { Old.	9·63	9·57	·05
{ New.	9·49	8·96	·52
Baynes.	9·67	9·17	·50

Now comes a milk extra rich in fat.

## No. 6

	Total Solids.	Solids-not-fat.	Fat.
Vieth.	18·711	8·704	10·007
Adams. { Old.	18·470	8·81	9·65
{ New.	17·860	7·74	10·12
Baynes.	18·48	8·48	10·00



As might be expected, the old method in every case gives *fat* much below the truth, the average deficiency = .582 per cent. Dr. Vieth, with the plaster, is on an average .214 per cent. short, and Mr. Baynes gets result, very closely agreeing with the new process, being only .018 per cent. less.

As also might be expected, the new method reduces the solids-not-fat in greater proportion than it augments the fat, for the solids-not-fat, besides suffering a reduction due to the extra fat removed, give up the last trace of water, which has always been retained in the horny proteids in the same way that the fat is, and hitherto none of the expedients resorted to for setting free the locked up fat, such as the use of Plaster of Paris, or sand, or glass; is available in the estimation of the solids-not-fat, whereas the paper method lends itself with equal readiness to the perfecting this part of the process, by getting rid of the last trace of water. In this way it comes about that the new process gives on an average .52 per cent. less than Mr. Baynes, .62 per cent. less than Dr. Vieth, and .81 per cent. less than I myself got in the platinum dish.

As a matter of course, it follows pretty much the same with the Total Solids, which are in every case lower from .215 to .414 per cent. than the amount got by the old method. These seven examples, one of ordinary whole milk, five of skim milk, and one of milk enriched by the addition of cream, fairly illustrate the application of the method to the several varieties likely to be met with in practice, and you will no doubt deem it superfluous to multiply these examples, which have not been chosen with a view of casting a favourable light upon this new method of manipulation, but because by the kind assistance of Dr. Vieth and Mr. Baynes, I am able to bring to bear trustworthy checks upon the results.

Moreover, the conditions have been kept, as far as possible, of an ordinary every day character; no more time or trouble has been spent upon these particular test analyses than would be given in the ordinary way by any careful manipulator.

To gather up the points brought forward, I venture to submit, gentlemen, that hitherto milk analysis has fallen short of desirable and necessary accuracy, owing to the occlusion of fat and water within the dried proteids, and out of reach of solvents or removal by heat at 100 °C.

That the retention of the fat by the old processes varies with the proportion of fat originally present, also with the condition of the residue when the solvent is applied, a residuum of fat remaining in almost every case, whichever of the old processes is employed.

As regards the solids-not-fat, that neither of the older processes is capable of removing the last, which proves to be a very considerable, trace of water, and consequently that this datum has hitherto always been returned much too high. And, as a consequence, the data fat and solids-not-fat, as hitherto estimated, have been given in terms relative and not absolute, varying with the processes employed, and the different personal methods of working.

That the new process may fairly lay claim to the following advantages:—

1. The complete extraction of the fat is made easy and certain.
2. Likewise the perfect removal of the last trace of water from the solids-not-fat is also made equally easy and certain.

3. These data being in terms absolute, there ought, and it is reasonable to expect there will be, perfect concordance among different workers using this process.
4. In point of accuracy my later experience leads me confidently to look for concordance within the second place of decimals, the accuracy depending chiefly upon the efficiency of the drying apparatus, about which I hope to have something to say on a future occasion.
5. Another point of no little value is, that a large number of analyses may be set going together, and the charged papers at any time after rough drying put on one side, so that the subsequent stages of the analysis may be resumed at any convenient moment, and eventually the paper with the solids-not-fat stored for any length of time for future reference.
6. As regards time, cost of material, and personal attention, no method can be done quicker, cheaper, or with less looking after; it can be worked without any special apparatus, or material, beyond the paper, although I strongly recommend the winder for the paper and the wire cage for drying the papers, against which trifles it will be remembered that all expensive platinum dishes are dispensed with, except for the ash, which is best done in a capsule of from 70 to 100 c.c. capacity, such as is usually employed in water analysis.

I have now said all I have to say for the present on this subject. The process as regards its general acceptance, will, of course, stand or fall upon its own merits, but rest assured, gentlemen, that I feel the responsibility of presenting for your consideration a new mode of procedure in milk analysis; enough trouble and to spare has already arisen by reason of the variety of methods in use, and a new method, unless fit from all points to supplant its predecessors, can only be reckoned more or less of a nuisance; if I had any fear of this I should not have troubled you with this paper.

Allow me, in conclusion, to acknowledge the assistance I have received from Dr. Vieth, Mr. Baynes and my assistant, Mr. L. Stansell, in the course of these experiments.

#### VALUATION OF MILK SOLIDS INSTEAD OF A LIMIT OR STANDARD.

By C. ESTCOURT, F.I.C., F.C.S.

In the paper read in December last by Mr. Heish, it is rendered perfectly clear that he did not understand the object of my paper read 16th November, 1883.

The question I dealt with was the commercial, and not the dietetic value of milk. There exist, and will no doubt continue to exist, various opinions with regard to the dietetic value of the fat compared with the solids-not-fat of milk. Thus Mr. Heish would prefer an increase in the solids-not-fat, even at the expense of the quantity of fat; but this view is largely affected by the condition of health of the consumer of the milk.

In some special diseases, no doubt, a fat diet is far from desirable, but taking the large number of milk consumers (that is the million) I do not hesitate to say they would much prefer a milk from which it is possible to obtain a fair quantity of cream, to one which, with an increase of non fatty solids, gave a decreased amount of cream.

Taking this view, therefore, I still consider it of great importance to the complete settlement of the question that some such method as the one brought forward by me

should be adopted. Probably some modification of this method might commend itself to the general body of Public Analysts, and indeed, with a uniform method of analysis which, it is hoped, will be the result of the labours of the milk Committee, some definite method of valuing milk will have to be adopted. With regard to Mr. Heish's suggestion, I confess I fail to see how it is to be applied. We shall be no nearer the settlement of the vexed question by adopting his views than we now are. By adopting, however, some method of valuation depending upon the commercial value of the milk, we shall have some fixed point to start from. With this view I commend my own suggestion to the more careful consideration of the Society, until it is superseded by a better one.

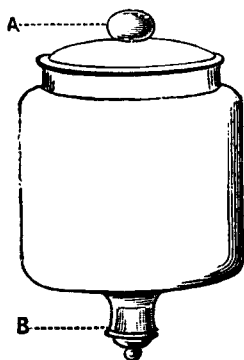
CONCLUSION OF THE PROCEEDINGS OF THE SOCIETY.

### ON A SIMPLE APPARATUS FOR THE ESTIMATION OF FAT IN MILK.

BY ALFRED C. WILSON, F.C.S.

THE piece of apparatus which I am about to describe, is intended for use in the Soxhlet Tube, and consists of a tube about two inches long, and about one and a quarter in diameter (according to the size of the soxhlet tube) each end being closed by means of a ground glass stopper, the lower opening being only a quarter, or three sixteenth of an inch in diameter. It is especially adapted for the abstraction of fat in milk.

For instance, let us suppose that the weight of the tube has been ascertained, a measured quantity of milk is introduced through the wide neck A, the small opening being closed, the stopper is inserted, and the weight taken. Total weight—weight of tube = weight of milk taken.



The stopper A is again removed, and the tube next placed on a water bath of suitable form, and the water allowed to evaporate off. (This usually takes place in about two hours) the tube is then cooled, the stopper A inserted, and the weight again taken. Total weight—weight of tube = weight of solids in the measured quantity.

The *two stoppers* are now removed, the *small one* being placed *inside* the tube, and the whole introduced into the soxhlet tube, and proceeded with as usual.

## COCAIN—THE NEW LOCAL ANÆSTHETIC.

BY A. PERCY SMITH, F.I.C., F.C.S.

COCAIN ( $C_{17}H_{24}NO_4$ , "Lossen") is the alkaloid contained in the coca leaves (*Erythroxylon Coca*, Lam.), which was first isolated by Niemann. In 1862, Lossen discovered a second volatile principle, viz., hygrin, which has been but little investigated. The other components of coca are ecgonin, tannin, and a peculiar wax.

The crystals, both of cocain and its hydrochlorate, are similar in form and belong to the monoclinic system. They form, from solution in alcohol or ether, in feathery sprays, with strong depolarizing power. From chloroform no crystals are deposited.

Cocain melts at  $98^\circ$ , is easily soluble in chloroform, ether, and alcohol, but requires 704 parts of water. The salts are freely soluble in water.

The habit of chewing coca leaves, practised by the natives of South America, has been known since the sixteenth century. It was said to have the power of counteracting the effect of physical exertion.

When cocain was first isolated, it was believed to be similar in property to caffeine, theine, and theobromine, which check the process of waste in the human body; but no facts have been observed conclusive of this supposition. In small doses, cocain has an exhilarating effect on the nervous system; in large doses, it has a fatal effect, by causing a cessation of respiration.

The toxic effects of cocain appear, however, to be mild, and its action not cumulative.

The lethal dose is not known. In a case of attempted suicide, 1.5 grammes had no serious effect; but the purity of the alkaloid taken is open to some doubt.

Coca leaves contain from .02 to .2 per cent. of cocain; but the present scarcity of the raw material in Europe has prevented much experiment.

The usual dose for a human being is .05 gramme cocain hydrochlorate.

When an aqueous solution of the alkaloid is injected subcutaneously, it produces a feeling of warmth, followed by an insensibility to pain in the immediate neighbourhood, and finally a reddening of the skin. The effect ceases in about 30 minutes.

Cocain placed on the tongue, or any exposed surface of mucous membrane, deadens the action of the nerves of the same.

From this cause it has proved valuable in operations upon the eye and teeth, and is recommended as of possible use in lithotrity, by injecting it into the urethra. In fact, if further experiment shows that the anæsthetic power may be fully relied on, cocain has a great future before it. At present the supply is small, and costs about 1s. 6d. per grain.

It has lately been ascertained that cocain has an excellent effect upon human frames which have been exhausted by diarrhoea; and as an injection is valuable in preventing the craving for morphia.

Cocain is a stimulant which has the property of quickly increasing and sustaining, in a harmless manner, the physical powers of the body, and it has a more strengthening effect than alcohol. Its use in cases of bodily exertion may be readily comprehended.

Whether, in cases of mental exertion, the powers of the mind can be increased or sustained, is an open question.

As a stomachic it quickly restores normal appetite after a debauch.

The most important uses of the alkaloid are, however, for the production of local anæsthesia, and for the cure of the craving for morphia.

Owing to the kindness of Herr Merck, of Darmstadt, who sent me a small quantity of coc. mur., I have been able to make a few experiments, the quantity at my disposal being but small.

My chief aim was to study the form of crystals from various solvents, by the aid of polarised light, as I have found that method of great use in many cases, both for identification, and especially as a test for purity.

Cocain does not crystalize very readily; any hastened evaporation of the solvent is fatal; spontaneous evaporation is necessary, and alcohol the best solvent. In a few hours in a warm room, the film will have dried completely, and show feathery sprays of strong depolarising power. It remains to be seen what effect adulteration will have upon the form of crystals. I have found, in some cases, that mixtures of alkaloids refuse to crystallize at all, but dry up to a gummy mass.

Neither  $H_2SO_4$  nor  $HNO_3$ , with or without  $K_2Cr_2O_7$ , produce any colouration.

Phosphotungstic acid = gelatinous white ppt. sol. in AmHo.

Phosphomolybdic acid = yellowish wh. ppt. sol. in AmHo, and in hot  $HNO_3$ .

Aniline sulphate +  $H_2SO_4$  (test for nitrate) = brown ring.

## ON THE ANALYSIS OF CONCENTRATED MEAT PREPARATIONS.

By DR. A. STÜTZER.

Director der landwirth Schaftlich-Chemischen Versuchstation für Rheinpreussen.

(*Specially written for the ANALYST and translated by Mr. Frederick H. Hatch.*)

I commenced testing, some years ago, with special reference to their physiologically important constituents, a certain number of articles of food introduced into Germany for the use of invalids and children. I stated that my investigation led to highly interesting results as regards the comparative nutritive value of the various substances so tested.\*

Several of the specimens then examined belonged to the ordinary class of preserved foods, such as, for instance, condensed milk, &c. I also further tested such articles of food as beef, fowl, eggs, and oysters, the two latter being usually greatly overrated in point of nutritive value as compared with meat. Then, again, I analysed caviare, extract of beef, and other preserved food of animal origin. It is strikingly obvious, of course, that, in regard to the various descriptions of food as a means of nourishment, there prevails, not amongst the general public only, but amongst chemists also, a number of erroneous conceptions, which can only be refuted by careful and exhaustive chemical analyses of such articles of food.

\* *Centralblatt für allgemeine Gesundheitspflege*, 1882: 179-188 und. *Repertorium, für analyt. Chemie*, 1882 T 161-169.

I have latterly taken up the consideration of the various special concentrated meat preparations, some of which are being sold in England, and some in the United States of North America.

In the tests applied to these meat-preparations I firstly established the percentage of organic ingredients, mineral ingredients, and water; and secondly I tried, as far as feasible, to decompose the organic principles in them. I determined, for instance, what proportion of the nitrogen contained in these preparations must be credited to the easily digested albumen and to peptone; and thus estimated, in the usual way, by multiplying by 6.25 (assuming that the constituent principles contained on an average 16 per cent. of nitrogen), the quantity of albumen and peptones. I believe that it would be right to put upon the latter a very special value, because upon them depends pre-eminently the physiological nutritive value of all articles of food of animal origin. Some specimens contained no albuminous principles soluble in water; and with these it became necessary to ascertain, by means of an additional digesting experiment, how much of the albuminous principles can be digested. In the next place I take into consideration the quantity of nitrogen present in the form of meat-bases (such as creatine, carnine, &c.), because these meat-bases, together with potash and phosphoric acid, possess a very high importance as relishing and stimulating means for the nervous system. The analysis of the mineral components went to the determination of phosphoric acid, potash, and chlorine.

The analytical methods used in the investigation of the preparations are the following:—

1. *Determination of the Water*.—Two grammes of the powdered substance are dried, until it ceases to lose weight. For extracts or liquid substances, a few grms. are weighed exactly, dissolved in as little water as possible, the solution evaporated over the water-bath in a platinum dish, containing 10-15 grms. previously ignited sand, and dried at least four hours at 95-100° C until the contents of the dish cease to lose weight. The loss of weight is calculated as water.

2. *Mineral Substances*.—The residue, obtained in the determination of the water, is ignited. As soon as the contents of the dish have turned grey, it is allowed to cool; ammonium nitrate is now mixed in with a platinum wire, and the mixture heated again for half an hour. The addition of sand to liquid aliments and food extracts has the advantage that, in the determination of the water, the moisture more quickly evaporates; further, that, in the estimation of the mineral substances, the air has more opportunity for oxidising the carbonaceous matter, the ignition being thereby somewhat accelerated. On the other hand, the melting ammonium nitrate rapidly burns the carbonaceous residue, and decomposes the carbonates formed in the combustion. The ash thus obtained is free from carbon and carbonic acid. In the half-hour ignition, after the addition of the ammonium nitrate, the nitrates formed are so completely decomposed that on dissolving the residue no trace of nitric acid is to be detected.

3. *The Organic Matter* is calculated from the difference by subtracting the water and the mineral substance from the weight of the original substance.

The following table shows the respective amounts of water, organic substance, and mineral matter found in the nine specimens of concentrated food analysed, in alphabetical order:—

Name.	Water.	Organic Substances.	Mineral Substances.	Total.
Benger's Peptonised Beef Jelly ..	89.68	9.43	.89	100
Brand's Essence of Beef ..	89.19	9.50	1.31	100
Carnrick's Beef Peptonoids ..	6.75	87.75	5.50	100
Johnston's Fluid Beef ..	49.49	45.32	5.19	100
Kemmerich's Extract of Beef ..	20.95	60.81	18.24	100
Liebig's Extract of Beef ..	19.33	57.52	23.15	100
Murdoch's Liquid Food ..	83.61	15.83	.56	100
Savory and Moore's Fluid Meat ..	27.01	60.89	12.10	100
Valentine's Meat Juice ..	59.07	29.41	11.52	100

4. *Potash*.—2–5 grms. of the dry or extracted food-substance (if a liquid, at least 10 grms.) are weighed out exactly, dried, and gently ignited, so that a gradual combustion takes place. As soon as no more fumes are given off, and the whole mass appears black, it is allowed to cool, extracted repeatedly with hot, very dilute hydrochloric acid, the residue brought upon a large filter, and well washed; the filter is then spread out upon a glass plate, and, partly by means of a spatula, partly with the help of a wash-bottle, the washed carbonaceous substance brought back into the platinum dish. After evaporating to dryness on the water-bath, the residue is heated till it turns white; on cooling, dilute hydrochloric acid is added, the liquid mixed with the first extract, brought into a porcelain dish, and evaporated to dryness on the water-bath. The residue is washed with water into a beaker and warmed; barium chloride, ferric chloride, and ammonia are then added successively, the latter in such a quantity that the liquid remains just alkaline. The precipitate (consisting of  $\text{BaSO}_4$ ,  $\text{FePO}_4$ , and  $\text{Fe}_2\text{O}_3$ ) is filtered, washed, the filtrate, after the addition of ammonium carbonate and ammonium oxalate, warmed for some time on the water-bath, and filtered, the precipitate (consisting of calcium oxalate and barium carbonate) being well washed; the filtrate is then evaporated to dryness in a platinum dish, and gently ignited to get rid of the ammonium salts. The residue is dissolved in water, the small quantity of barium carbonate filtered, and the filtrate, after the addition of platinum chloride, evaporated nearly to dryness. The residue is treated with alcohol, filtered, and washed with alcohol. The  $\text{K}_2\text{PtCl}_6$  on the filter is dissolved in hot water, the solution evaporated to dryness in a glass dish, the residue dried for  $\frac{1}{2}$  hour at  $100^\circ\text{C}$  in the air-bath, and weighed.

(To be continued in our next.)

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

**DISTINCTION BETWEEN RICE-MEAL AND BUCKWHEAT-MEAL.**—The sample is made into paste with strong potash-lye, heated on the water bath, and treated with hydrochloric acid. In case of rice, the paste is yellow, and after treatment with acid, white. Buckwheat gives a dark-green paste which is turned red by hydrochloric acid—*A. Lehn* in *Zeitschr. f. Anal. Chem.*

OWING to the press of original matter on this subject, the usual abstracts are postponed.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

IN a paper read before the Pharmaceutical Society, Messrs. Cripps and Dymond give the following test for the presence of aloes :—Place 1 grain of the suspected drug in a glass mortar, standing on white paper ; add now 16 drops of strong sulphuric acid, and triturate till the whole is dissolved ; then add 4 drops of nitric acid, sp. gr. 1.42 ; and, lastly, one ounce of distilled water. If aloes be present, a colour varying from deep orange to crimson will be produced, according to the kind of aloes employed. To confirm, add liquor ammoniæ, when the colour is deepened, usually to a deep claret. The authors append a table of the exact shades of colour produced by the use of this test, with all the B. P. preparations containing aloes, and with others, which, while similar in appearance, do not contain this drug.

---

## MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

OWING to the press of other matter, we are obliged to hold over the usual abstracts under this heading.

---

## TO CORRESPONDENTS.

OWING to the press of other matter previously in type, we regret to have to omit several interesting communications this month, for which, however, room will be found in our next.

MR. A. C. ABRAHAM, of Bold Street, Liverpool, writes to point out that the ether mentioned on page 226 of THE ANALYST, as being commonly sold in England for anæsthetic purposes, sp. gr. .717, is really a *methylated* ether, and only used for purposes of *local* anæsthesia. He further mentions that it is difficult in practice to raise ether from pure spirit even to the .720 required by the British Pharmacopœia.

---

## BOOKS, &amp;c., RECEIVED.

AIDS to the Analysis of Food and Drugs, by Aubrey Husband ; Fresenius' Quantitative Analysis, Vol. II., Part I., translated by Chas. E. Grove, F.R.S. ; American Druggist ; American Grocer ; British and Colonial Druggist ; Brewers Guardian ; Canadian Pharmaceutical Journal ; Chemist and Druggist ; Country Brewers Guardian ; Cowkeeper and Dairymans' Journal ; Illustrated Science Monthly ; Independent Journal ; Invention ; Journal of Chemical Industry ; Journal of Microscopy and Natural Science ; Medical Press ; Medical Record ; The Miller ; Monthly Magazine of Pharmacy and Chemistry ; New York Analyst ; Pharmaceutical Journal ; Pharmaceutical Record ; The Polyclinic ; Popular Science News ; Publishers' Circular ; The Sanitarian ; San Francisco News Letter ; Science Scientific American ; Scientific Californian ; Society of Arts Journal ; Hospital Gazette.

---

## NOTICE TO CONTRIBUTORS.

All Letters and MSS. intended for the literary part of this Journal should be addressed to the Editorial Department, 325, Kennington Road. London, S.E.



# THE ANALYST.

APRIL, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—	
(a) REPORT OF MEETING .. .. .	61	(a) EXAMINATION OF PEPPER, C. NEUSS ..	76
(b) DETERMINATION OF MIXTURES OF MILK-SUGAR AND CANE-SUGAR, By A. W. STOKES, F.C.S., AND R. BODMER, F.C.S. .. .. .	62	(b) DETERMINATION OF SALICYLIC ACID IN WINE. G. HEINZELMANN .. .. .	77
(c) DISCUSSION ON THE ABOVE .. .. .	66	(c) DETECTION OF CU AND Pb IN WINE, H. HAGER .. .. .	77
(d) NOTES ON THE EXAMINATION AND COMPOSITION OF MILK AND MILK PRODUCTS, By DR. P. VIETH .. .. .	67	MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	77
(e) NOTE ON AN ADULTERATED SAMPLE OF TEA, By DR. A. BOSTOCK HILL .. .. .	71	(a) DETERMINATION OF MORPHINE IN OPIUM, V. PERGER .. .. .	77
(f) NOTE ON THE OPTICAL ESTIMATION OF MILK-SUGAR, By A. H. ALLEN, F.C.S. .. .. .	72	(b) REACTION FOR CHLORAL HYDRATE, M. HIRSCHFIELD .. .. .	78
ORIGINAL ARTICLE:—		MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
ON THE ANALYSIS OF CONCENTRATED MEAT PREPARATIONS, By PROF. STUTZEE .. .. .	73	(a) ANALYSIS OF INDIGO, H. M. RAU .. ..	78
		(b) LAKMOID AS AN INDICATOR, M. C. TRAUB ..	79
		(c) DETECTION OF MONOCARBONATE IN SODIUM BICARBONATE, A. KREMEL .. ..	79
		(d) SEPARATION OF COPPER FROM CADMIUM, P. GUCCI .. .. .	79
		LETTER TO THE EDITOR:—	
		ON MILK STANDARDS FROM C. HEISCH, F.I.C. ..	80
		BOOKS RECEIVED—NOTICES, &c. .. .. .	80

## SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting of this Society was held at Burlington House, on Wednesday, the 11th March, the President, Dr. Hill, in the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election as members:—Mr. J. A. Bond, Colonial Laboratory, Auckland; Mr. H. Sugden Evans, Chief Public Analyst to Dominion of Canada, Montreal. As associate:—Mr. H. C. Williams, Assistant to Mr. C. T. Kingzett.

Mr. Kingzett and Mr. Baynes were appointed scrutineers to open the voting papers, and reported that the following had been elected:—

As members:—R. Bodmer, F.C.S., Analytical Chemist, London; J. Ruffe, M.R.A.C., F.C.S., Analytical Chemist, London; C. G. Stewart, Analytical Chemist, London.

As Associates:—J. C. Bredin, Assistant to Dr. Bernays; W. Hogben, Assistant to Dr. Drinkwater.

Mr. W. Johnstone read a paper "On an Improved Apparatus for Milk Analysis," and exhibited a specimen to the members present. This paper will be published in our next issue.

The discussion on Mr. Adam's paper on "Milk Analysis," read at the January meeting was then resumed, and again adjourned for Mr. Adam's reply. A communication from Mr. Abrahams, on the subject of Mr. Adam's paper, was also read.

The complete discussion, together with the author's reply, will also appear in our next issue.

The next meeting of the Society will be held at Burlington House, on Wednesday, 8th April.

## THE DETERMINATION OF MIXTURES OF MILK-SUGAR AND CANE-SUGAR.

BY A. W. STOKES, F.C.S., AND R. BODMER, F.C.S.

*Read at the Meeting, on 11th February, 1885.*

AMONGST several beautiful and delicate methods introduced by Dr. F. Pavy, F.R.S., for estimating sugar, there is one in which a modified Fehling solution is used. In this process the oxide of copper, instead of being precipitated, is kept in solution by strong ammonia. So that, on slowly adding the sugar solution, the point where the Fehling solution becomes colourless is distinctly seen, without being masked by the usual dirty-red precipitate.

This method is recorded in some of the chemical text-books, but for some unknown reason has not yet received the general attention that it deserves. For delicacy and accuracy, combined with ease of working, it is far superior to any other method yet in use.

We have for some months past been engaged in a series of experiments to adapt this method to the requirements of those dealing with mixed sugars, especially in the case of condensed milks. In our hands the process works almost perfectly.

Dr. Pavy has found that if a mixture of cane-sugar and milk-sugar be boiled for seven to ten minutes, with a two per cent. solution of citric acid, the cane sugar will be inverted, but the milk-sugar will remain unacted upon.

Hence, given a mixture of the two sugars, it is only necessary to determine the milk sugar direct by means of the modified Fehling solution in one part of the mixture; then to boil another part with citric acid, and determine the added reducing power, to obtain at once the percentages of the two sugars.

The Modified Fehling Solution consists of:—

34.65	grammes of Crystallized Sulphate of Copper.
170	,, ,, Rochelle Salt.
170	,, ,, Caustic Potash.

made up to 1 litre.

*buret =*  
 4.158  
 20.4  
 20.4  
 + 4.000 gm to 1 lit }

120 c.c. of this fluid, with 400 c. c. of ammonia (sp. gr. .88) are made up to 1 litre.

This ammoniated cupric fluid constitutes the Pavy solution.

10 c.c. of this = 0.005 glucose.

It is advisable to standardise the solution with a weighed quantity of cane-sugar (inverted). Exact quantities of the solution (we prefer 40 c.c.) are placed in a small flask; this fits tightly to a cork suspended by a short piece of india-rubber from a burette. The cork has a side tube to allow of the escape of ammonia. The india-rubber tube is compressed by a screw (devised by Dr. Pavy) which will allow a rapid stream or a very slow succession of drops to pass into the flask.

The burette is filled with the solution of mixed sugars, well diluted, the flask containing the 40 c.c. of ammoniated solution is attached, and is heated till the fluid boils. A slow succession of drops is now allowed to run in from the burette till the blue colour disappears. The reading of the burette is taken. The milk should have been diluted, so that from 6 to 12 c.c. may be used to decolorize the copper solution. The action of milk-sugar on the copper solution is very slow, hence it must not be run in rapidly. On

the other hand, it must be run in within a limited time, else oxide of copper will begin to deposit, owing to the evaporation of the ammonia. Should the oxide of copper begin to deposit before the liquid becomes colourless, the screw-tap must be closed, a fresh quantity of the ammoniated solution be taken in another flask, and the quantity of sugar solution shown by this partial titration must be added at once. This is boiled for a short time, and then the sugar solution is run in from the burette, a few drops at a time, till the liquid is decoloured.

It is exceedingly easy to over-run the proper quantity. Therefore it is advisable to take a third quantity of the ammoniated solution, and add to it at once 0.2 c.c. less than the quantity of sugar solution found necessary above, and then boil till the oxide of copper begins to deposit. If the blue colour does not disappear before this takes place, it is certain that the quantity before added is within 0.1 c.c. of the true quantity required.

Should the blue colour, however, disappear, it will be necessary to work backwards, adding each time 0.2 c.c. less than before, till the exact point is reached. After a little practice it will not be found necessary thus to retrace one's step more than once. Thus the milk-sugar is determined.

Meanwhile, another measured portion of the mixed sugar solution is boiled with two per cent. of citric acid for seven to ten minutes. This is cooled, neutralized, or rendered slightly alkaline with ammonia, and either made up to the same bulk, or preferably made up to twice the original volume. This is titrated like the previous liquid.

We have now two readings of the burette; the first due solely to the action of the milk-sugar; the second due to the combined action of the milk-sugar and inverted cane-sugar; the milk-sugar has not been inverted.

A simple calculation will at once give the percentages of the two sugars present, or they may be found from an inspection of the table accompanying this paper.

There is great diversity amongst chemical authors as to the reducing power of inverted milk-sugar upon the ordinary Fehling solution.

It was essential to determine the reducing power of milk-sugar upon this modified Fehling solution. We found great difficulty in doing this, owing to the want of purity of most of the samples of milk-sugar. But at last, obtaining specimens found on the determination of their carbon and hydrogen to be pure crystallized samples, we have come to the conclusion that 100 parts of milk-sugar equal 52 parts of glucose or 49.4 parts of cane-sugar in reducing power upon this liquid.

The process has been tried upon aqueous solutions containing known quantities of sugars. For instance, a solution containing:—

1 per cent. of cane-sugar and 0.75 per cent. of milk-sugar, gave  
 0.95 ,, ,, 0.76 ,, ,, by this method.

With ordinary milks its working is almost perfect. The analyses of three samples is shown below. In these each constituent was separately determined; the totals sum up fairly near to 100.

	No. 1.	No. 2.	No. 3.
Water ..	87.700	88.58	87.69
Fat ..	3.240	1.98	2.92
Casein ..	4.000	4.77	4.19
Milk-sugar..	4.348	4.16	5.36
Ash ..	0.720	0.76	0.74
	<hr/>	<hr/>	<hr/>
	100.008	100.25	100.90

In No. 1 the casein was determined by Wanklyn's ammonia process. In Nos. 2 and 3 the casein was estimated by precipitation; this is a far less exact process than the former.

Next we took milks to which known quantities of cane-sugar had been added. As an illustration of the results obtained, as well as of the method of working, we give this example—

To a milk 3.25 per cent. of cane sugar was added.

Twenty c.c. of this was coagulated with citric acid, made up to 200 c.c. and filtered (solution A). (This coagulation, to get rid of the casein, is not necessary, though it renders the end of the reaction more easy to be seen).

Forty c.c. of Pavy's solution took 8.5 c.c. for decolouration. Since 40 c.c. Pavy's solution = .02 glucose.

The solution contains  $\frac{.02 \times 100 \times 10}{8.5} = 2.35$  per cent. glucose.

To convert this into milk-sugar—

$$\frac{2.35 \times 100}{52} = 4.51 \text{ per cent. of milk-sugar in the original milk.}$$

Boiled 50 c.c. (solution A) with one grm. citric acid for ten minutes, made up to 100 c.c. after neutralization with ammonia.

Forty c.c. Pavy's solution took 7 c.c. of this for decolouration :—

$$\frac{.02 \times 100 \times 20}{7} = 5.71 \text{ per cent. glucose.}$$

Subtracting from this the glucose found above as the equivalent of the milk-sugar—

5.71

2.35

—

We get 3.36 per cent. of glucose due to the cane-sugar.

Converting this into cane-sugar—

$$\frac{3.36 \times 95}{100} = 3.19 \text{ per cent. of cane-sugar found,}$$

While 3.25 „ „ was actually added.

Other examples taken at random from our books show :—

Cane Sugar	..	Added.	—Found.
		2.90	2.82
		3.20	3.05
		5.50	5.68
		10.50	10.80

We have tried the effect of boiling mixed sugars with acetic, oxalic, and tartaric acids, but find that citric acid is to be preferred. We are aware that the use of this solution has been severely criticized by a member of the Society of Public Analysts, but we think this may have been from want of a sufficient trial of the fluid. Of course, if the proportions of potash and ammonia are varied, the results will not be the same. This is equally true of the constituents of the ordinary Fehling solution, as the same critic has pointed out.

The usual method of determining the milk-sugar direct with Fehling solution, and then inverting both sugars with a mineral acid, and again determining the reducing power of the mixed inverted sugars, does not seem to give good results. It has been pointed out by Dr. Muter that to determine milk-sugar alone with the ordinary Fehling solution, an invariable procedure must be followed, and especial precautions be taken, otherwise different results will be obtained by each operator.

The fact that few writers agree as to the actual reducing power of inverted milk-sugar, seems to indicate that mineral acids have a varying action upon milk-sugar, according to the strength or dilution of the sugar solution.

By this method, the milk-sugar never being inverted, constant results can be obtained by all competent operators.

By the usual Fehling solution method, the casein must be precipitated from a milk, else ammonia will be formed by the action of the potash, and some oxide of copper may be kept in solution. In this process the milk may be used direct after dilution.

The ordinary Fehling solution will not keep; the ammoniated solution retains its strength indefinitely.

There is no muddy mixture of red particles and a bluish liquid to have to deal with. It is essential that the Pavy fluid be perfectly clear during the whole time.

We append a table to facilitate the calculation of results.

Table showing percentage values in glucose, crystallized milk-sugar, and cane-sugar, for 40 c.c. of Pavy's solution decolourized:—

c.c. Solution.	Glucose.	Milk-Sugar.	Cane-Sugar.	c.c. Solution.	Glucose.	Milk-Sugar.	Cane-Sugar.
4·0	0·500	0·961	0·475	7·0	0·285	0·549	0·271
·1	·488	·938	·463	·1	·281	·541	·267
·2	·476	·915	·452	·2	·277	·534	·264
·3	·465	·894	·442	·3	·274	·526	·260
·4	·454	·874	·431	·4	·270	·519	·256
·5	·444	·854	·422	·5	·266	·512	·253
·6	·435	·836	·413	·6	·263	·506	·250
·7	·425	·818	·404	·7	·259	·499	·246
·8	·416	·801	·396	·8	·256	·493	·243
·9	·408	·785	·387	·9	·253	·486	·240
5·0	·400	·769	·380	8·0	·250	·480	·237
·1	·392	·754	·372	·1	·247	·474	·234
·2	·384	·739	·365	·2	·244	·469	·231
·3	·377	·725	·358	·3	·241	·463	·229
·4	·370	·712	·351	·4	·238	·458	·226
·5	·363	·699	·345	·5	·235	·452	·223
·6	·357	·686	·339	·6	·232	·447	·221
·7	·350	·674	·333	·7	·229	·442	·218
·8	·344	·663	·327	·8	·227	·437	·216
·9	·339	·652	·322	·9	·224	·432	·213
6·0	·333	·641	·316	9·0	·222	·427	·211
·1	·327	·630	·311	·1	·219	·422	·208
·2	·322	·620	·306	·2	·217	·418	·206
·3	·317	·610	·301	·3	·215	·413	·204
·4	·312	·600	·296	·4	·212	·409	·201
·5	·307	·591	·292	·5	·210	·404	·199
·6	·303	·582	·287	·6	·208	·400	·197
·7	·298	·574	·283	·7	·206	·396	·195
·8	·294	·565	·279	·8	·204	·392	·193
·9	·289	·557	·275	·9	·202	·388	·191

## DISCUSSION.

DR. MUTER said that he had previously published a process on the same subject, its essential point being that it was based upon actual weighing of the two sugars—estimating the milk-sugar and ascertaining the cane by difference. Since then, he believed, many analysts had given up those methods to a great extent, and they now used the polariscope instead, and found they could apply it to this question. In the process then before them the only thing he perceived that was somewhat in want of discussion was whether the method of inversion was perfectly reliable or not. Mr. Stokes, he understood to admit that it was a question of the length of time of inversion, whether he inverted all the cane-sugar or not. With regard to these inversion processes he knew that it was entirely a question of the mode of working; all sorts of fractional inversions might be obtained by difference in working. It seemed to him essentially necessary that for the success of the process certain conditions should be laid down and exactly followed, or comparative results would not be obtained.

MR. HEINER said he would like to have heard some experiments as to the action of citric acid upon pure milk-sugar, to see whether it underwent any change, or was absolutely unaffected. The process used by the Excise Chemists for the detection of cane-sugar in malt worts, viz., boiling for a very short time with a certain amount of acid, in which the cane-sugar was said to be inverted and nothing else, appeared to him to be analogous to this method, but after all it was only a kind of a fluke how deeply the inversion proceeded. He should like to know whether it was not similarly a matter of chance in this case. As far as he could see the chief claim to novelty was the employment of citric acid for the inversion of cane-sugar, and the actual process was independent of the use of ammoniacal Fehling solution. He believed that any other process of sugar determination would do quite as well. Mr. Stokes was mistaken in supposing that he originally looked unfavourably at Pavy's process. At first he got most satisfactory results, and was charmed with the process, but when he came to examine it more deeply he found that every alteration in the conditions, made a tremendous difference in the result, and that a variation, which was quite within the limits of a chance experiment, entirely altered the result obtained. He thought that Soxhlet's experiments had established the right way of estimating, not only milk-sugar, but many other sugars, and in the new edition of Fresenius the method was very accurately described. By both the volumetric and gravimetric methods very excellent results could be obtained without using the Pavy solution.

DR. DUPRE said that with much of the criticism he agreed. The value of estimating cane-sugar was, of course, only in regard to condensed milks. Did not ordinary condensed milk contain cane-sugar inverted? if so, the method just described would not be applicable.

MR. BODMER, in reply, said that as to boiling they had made experiments on the action of citric acid, and found that if dilute solution were used, the milk-sugar could be boiled for 35 minutes, and the reducing power of that milk-sugar would be perfectly unchanged. It was evident that 35 minutes was ample time to invert the cane-sugar, but they had generally found 10 minutes to be sufficient—seven

minutes being the minimum. As to the behaviour of the blue liquid under different conditions, they standardized it against cane-sugar, and after months it was unchanged. He had had liquids containing various quantities of sugar of various strengths, and did not find different results. If a certain amount of cane-sugar were present, the results were almost exactly identical. As to hurrying being necessary, if the work were done according to the method laid down there was no occasion for hurry—having got an approximate test, then, if the colour had not disappeared, they added a little more, generally 2 c. c., when the colour would disappear, and there would still be time to boil without the oxide of copper coming down.

### NOTES ON THE EXAMINATION AND COMPOSITION OF MILK AND MILK PRODUCTS.

By DR. P. VIETH, F.C.S.

*Read at the Meeting 11th February, 1885.*

THE paper which I am going to bring before you, includes what may be called a "Report on the work done in the Aylesbury Dairy Company's Laboratory during the year 1884." At a time when so much attention is paid to the composition and examination of that most important, much used, and much abused article of diet, called milk, notes and figures like those which I am in the position to put before you cannot be regarded valueless. It is for this reason that I thought it worth while to give you, as I have done in previous years, a brief account of the work done, and the results obtained in the laboratory under my charge during the past year. Some additional remarks will, I hope, tend to increase, rather than diminish, your interest in the paper.

Besides a vast number of specific gravity determinations, 15,773 analytical and microscopic examinations were executed during the year 1884, comprising:—

Samples of milk	..	..	..	..	..	14,235
"    cream	..	..	..	..	..	1,290
"    skim milk	..	..	..	..	..	85
"    buttermilk	..	..	..	..	..	50
"    butter	..	..	..	..	..	15
Sundries	..	..	..	..	..	98
						15,773
				Total	..	..

I wish to repeat here what I have said already on different occasions, viz., that I think the specific gravity a very valuable item for judging a milk. To take the specific gravity as long as the milk is sweet is a very simple thing, in whatever way it is done, whether by means of a specific gravity bottle, or a Sprengel tube, or a suitable hydrometer. Whenever and wherever a milk sample is examined, the specific gravity should be taken. This is, unfortunately, not done, and in some cases which came under my notice, where it had been done, I have reason to believe that not even that very small amount of skill and trouble which is wanted was bestowed upon the determination, and the results obtained were wrong.

The specific gravity of the mixed milk of several—say, at least, four—cows fluctuates in very narrow limits, and a standard could be fixed much easier than in the

case of any of the chemical constituents. The determination of the specific gravity helps to detect the addition of water in the simplest way. Moreover, it has been proved that, basing upon the figures for specific gravity and total solids, the amount of fat can be calculated to a high degree of accuracy, and thus the specific gravity affords the means of simplifying milk analysis, or checking the results.

In my laboratory the specific gravity is taken by means of a hydrometer, specially made for the purpose, and called lactometer, an instrument which, provided its scale is right, shows the specific gravity without any trouble, and in the shortest time. The temperature of the milk must be taken in consideration; lactometers show the actual specific gravity at 15° C. A table for correcting the specific gravity according to temperature, is appended to this paper. We find the specific gravity to vary from 1.030 to 1.034, with very few exceptions. Very rich Jersey milk, containing about 15 per cent. of total solids, and 5 per cent of fat, for instance, has a specific gravity of 1.034 to 1.035. The specific gravity of a mixed milk should never fall below 1.029, unless an excess of cream be present.

Fat is in every case of importance, or special interest, determined by exhausting in a Soxhlet apparatus the dry powder obtained by evaporating about 10 grams. of milk with twice or three times its weight of plaster of Paris. For the bulk of the samples, analysed simply for controlling purposes, you will remember that I was in the habit of using the lacto-butyrometer for determining the fat. This instrument answers the purpose very well. In order to find out how far it can be trusted in each separate case, a very close investigation, extending over a period of nine months, and comprising 1,439 samples, was carried out in this way, that the results gained by means of the lacto-butyrometer were compared with those obtained by using more exact methods. This investigation led to the following results:—When milk, containing from 3.0 to 3.6 per cent. of fat, was tested, the results obtained by using the lactobutyrometer were correct, or differed within .2 per cent. in 84 cases out of 100, and in 9 cases out of 100 the difference was .3 per cent. With an increase in the percentage of fat, the differences were also increasing in number, as well as in extent. In far the larger number of cases, the results obtained fell short of the truth, and very large differences occurred in some cases referring to exceptionally rich milks. On the other hand, in testing poor milk, containing less than 3 per cent. of fat, the results indicated sometimes exceeded the actual percentage of fat present. The extreme differences of the whole series were + .4 and — 1.2 per cent.; but these extremes were very scarce indeed. The average difference of the 1,439 determinations was— .18 per cent. The lactobutyrometer cannot be used for scientific researches, and the instrument is not meant to be used in such cases. But it may be used with great advantage in cases where the object in view is to get a fair idea as to the richness of milk, or to find out whether a milk contains at least a certain amount of fat. In the case of milk with from 3.0 to 3.6 per cent. of fat the results are all what can be expected.

When speaking about the importance of the specific gravity, I mentioned the possibility of calculating the fat in milk from specific gravity and total solids. About three years ago, Mr. Hehner gave us an exceedingly interesting paper on this subject. His



conclusions and formula were based upon results arrived at by employing Wanklyn's method of milk analysis, which was then still in general use, but which I sincerely hope, nobody will think of using any more ere the present year has gone by. Fleischmann and Morgen have worked out two formulæ, the one for calculating the total solids from specific gravity and fat, and the other for calculating the fat from specific gravity and total solids. They used analytical methods, which I think the most reliable ones, and which I am in the habit of using myself, viz., drying the solids to constant weight, and extracting the fat, after having dried up the milk with plaster of Paris. The formulæ are as follows:—

$$\text{I.}—t = a \times 1.173 + 2.71 \left(100 - \frac{100}{s}\right).$$

$$\text{II.}—a = t \times .852 - 2.31 \left(100 - \frac{100}{s}\right).$$

t = percentage of total solids.

a = „ fat.

s = specific gravity (water = 1).

I have made 65 comparative experiments, and found the figures for fat extracted and calculated generally agree entirely, or differ not more than .1 per cent. In a very few instances the difference was .2; whilst the average difference was .01 per cent. Having thus satisfied myself of the applicability of the formulæ, I commenced in July last to calculate the fat of all the samples taken on the arrival of the milk. For the samples taken by the Company's inspectors, the lactobutyrometer test was continued for several reasons.

Among the 14,235 milk samples analysed, there are 10,399 taken from the railway churns, when arriving at the Company's premises at Bayswater. The rule is, that at least *one* sample of the milk of each farmer supplying the Company is analysed daily, samples of morning and evening milk being taken alternately. The monthly averages of these analyses are given in the following table:—

1884	Sp. gr.	Tot. Slds.	Fat.	Slds.-not-fat.
January ..	1.0325	12.89	3.55	9.34
February..	1.0325	12.77	3.53	9.24
March ..	1.0323	12.72	3.50	9.22
April ..	1.0323	12.65	3.43	9.22
May ..	1.0324	12.64	3.34	9.30
June ..	1.0323	12.50	3.31	9.19
July ..	1.0319	12.60	3.47	9.13
August ..	1.0318	12.95	3.87	9.08
September	1.0321	13.28	4.11	9.17
October ..	1.0324	13.53	4.26	9.27
November	1.0324	13.65	4.36	9.29
December..	1.0326	13.39	4.10	9.29
Yearly average	1.0323	12.96	3.74	9.22

This extensive control is, of course, of the greatest consequence to the business, as it not only guards against frauds on the part of the farmers supplying milk, or their servants, but also allows to make a selection when the question arises to enter into new contracts. But, at the same time, the strict control is a great benefit to the public, who have the best possible security for a supply of pure and rich milk. How grateful the public are for such a benefit may be gathered from an opinion expressed a short time ago to this effect, that the Aylesbury Dairy Company employs an analyst in order to

be able to bring the milk down to the limits of the Society of Public Analysts—or, in other words, to water the milk without fear of prosecution.

We have seen what the composition of the milk is when it arrives; now I shall have to tell you its composition when handed over to the customers. To show this is easy enough. The Company employs a staff of inspectors, whose business it is to control the men when working their rounds. The inspectors have to take samples from the men, and hand them over to the laboratory, where they are analysed and compared with samples taken before the milk was sent out. During the past year, 3,573 such samples have been taken, and the total solids of them averaged as follows:—

1884.	Tot Slds.	1884.	Tot. Slds.
January .. .. .	12·8	July .. .. .	12·6
February .. .. .	12·7	August .. .. .	12·9
March .. .. .	12·7	September .. .. .	13·2
April .. .. .	12·6	October .. .. .	13·5
May .. .. .	12·5	November .. .. .	13·4
June .. .. .	12·5	December .. .. .	13·3
Yearly average .. .. .	12·6	12·9 total solids.	

A comparison of these figures with those previously mentioned, clearly shows that there are, practically speaking, no differences; but that the milk supplied to the customers is of the same average composition as when received from the country. In some instances there are differences, owing to cream rising in the churns; but considerable differences are exceedingly rare exceptions. I have brought two such rare cases before you on former occasions.

The cream dealt with by the Company is partly separated from milk, by centrifugal force on the premises, partly received from the country. The value of cream rests almost exclusively with the amount of fat present. As there does not exist a ready means to determine the fat in cream except extraction, the ordinary cream samples were treated in this way, that the total solids were determined, and the fat calculated, with the aid of a table I have drawn up, and communicated to our Society a year ago. The monthly average composition of cream was found to be as follows:—

1884.	Tot. slds.	Fat.	1884.	Tot. slds.	Fat.
January .. .. .	39·6	32·6	July .. .. .	45·4	39·0
February .. .. .	40·6	33·6	August .. .. .	44·3	37·7
March .. .. .	41·1	34·2	September .. .. .	43·3	36·3
April .. .. .	40·6	33·7	October .. .. .	42·4	35·6
May .. .. .	43·1	36·4	November .. .. .	40·6	33·6
June .. .. .	43·7	37·0	December .. .. .	40·8	33·9
Yearly average .. .. .	42·1 total solids		35·3 fat.		

Less concentrated sorts of cream are churned into butter, so that the ordinary cream, as supplied to the customers, contained from 35 to 40 per cent. of fat. Samples taken by the inspectors from the men on the rounds, gave, when analysed, practically identical results.

If, for the purpose of separating cream from milk, the latter is submitted to centrifugal force, in those ingenious machines called “milk centrifuges,” or “cream separators,” a very poor skim milk is obtained. In about 80 samples of skim milk analysed during the year 1884, the amount of fat varied from ·2 to ·6 per cent.

In buttermilk, less than 1 per cent. of fat should be left, provided the process of churning is properly conducted. But, owing to the richness of the cream, and, more-

# TABLE FOR CORRECTING THE SPECIFIC GRAVITY OF MILK ACCORDING TO TEMPERATURE.

By Dr. P. VIETH, F.C.S., &c.

Degrees of lactometer.		DEGREES OF THERMOMETER ( <i>Fahrenheit</i> ).																		Degrees of lactometer.											
45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	
20	19.0	19.0	19.1	19.2	19.2	19.3	19.4	19.4	19.5	19.6	19.7	19.8	19.9	19.9	20.0	20.1	20.2	20.2	20.3	20.4	20.5	20.6	20.7	20.9	21.0	21.1	21.2	21.3	21.5	21.6	20
21	19.9	20.0	20.0	20.1	20.2	20.3	20.3	20.4	20.5	20.6	20.7	20.8	20.9	20.9	21.0	21.1	21.2	21.3	21.4	21.5	21.6	21.7	21.8	22.0	22.1	22.2	22.3	22.4	22.5	22.6	21
22	20.9	21.0	21.0	21.1	21.2	21.3	21.3	21.4	21.5	21.6	21.7	21.8	21.9	21.9	22.0	22.1	22.2	22.3	22.4	22.5	22.6	22.7	22.8	23.0	23.1	23.2	23.3	23.4	23.5	23.7	22
23	21.9	22.0	22.0	22.1	22.2	22.3	22.3	22.4	22.5	22.6	22.7	22.8	22.8	22.9	23.0	23.1	23.2	23.3	23.4	23.5	23.6	23.7	23.8	24.0	24.1	24.2	24.3	24.4	24.6	24.7	23
24	22.9	23.0	23.0	23.1	23.2	23.3	23.3	23.4	23.5	23.6	23.7	23.8	23.9	24.0	24.1	24.2	24.3	24.4	24.5	24.6	24.7	24.8	24.9	25.0	25.1	25.2	25.3	25.4	25.6	25.7	24
25	23.8	23.9	24.0	24.0	24.1	24.1	24.2	24.3	24.4	24.5	24.6	24.7	24.8	24.9	25.0	25.1	25.2	25.3	25.4	25.5	25.6	25.7	25.8	26.0	26.1	26.2	26.3	26.4	26.6	26.8	25
26	24.8	24.9	24.9	25.0	25.1	25.1	25.2	25.3	25.4	25.5	25.6	25.7	25.8	25.9	26.0	26.1	26.2	26.3	26.5	26.6	26.7	26.8	27.0	27.1	27.2	27.3	27.4	27.5	27.7	27.8	26
27	25.8	25.9	26.0	26.1	26.1	26.2	26.2	26.3	26.4	26.5	26.6	26.7	26.8	26.9	27.0	27.1	27.3	27.4	27.5	27.6	27.7	27.8	28.0	28.1	28.2	28.3	28.4	28.6	28.7	28.9	27
28	26.7	26.8	26.8	26.9	27.0	27.1	27.2	27.3	27.4	27.5	27.6	27.7	27.8	27.9	28.0	28.1	28.3	28.4	28.5	28.6	28.7	28.8	29.0	29.1	29.2	29.4	29.5	29.7	29.8	29.9	28
29	27.7	27.8	27.8	27.9	28.0	28.1	28.2	28.3	28.4	28.5	28.6	28.7	28.8	28.9	29.0	29.1	29.3	29.4	29.5	29.6	29.8	29.9	30.1	30.2	30.3	30.4	30.5	30.7	30.8	31.0	29
30	28.6	28.7	28.7	28.8	28.9	29.0	29.1	29.2	29.3	29.4	29.6	29.7	29.8	29.9	30.0	30.1	30.3	30.4	30.5	30.7	30.8	30.9	31.1	31.2	31.3	31.5	31.6	31.8	31.9	32.1	30
31	29.5	29.6	29.6	29.7	29.8	29.9	30.0	30.1	30.2	30.3	30.4	30.5	30.6	30.8	30.9	31.0	31.2	31.3	31.4	31.5	31.7	31.8	32.0	32.2	32.4	32.5	32.6	32.8	33.0	33.1	31
32	30.4	30.5	30.5	30.6	30.7	30.9	31.0	31.1	31.2	31.3	31.4	31.5	31.6	31.7	31.9	32.0	32.2	32.3	32.6	32.7	32.9	33.0	33.2	33.3	33.4	33.6	33.7	33.9	34.0	34.2	32
33	31.3	31.4	31.4	31.5	31.6	31.8	31.9	32.0	32.1	32.3	32.4	32.5	32.6	32.7	32.9	33.0	33.2	33.3	33.6	33.8	33.9	34.0	34.2	34.3	34.5	34.6	34.7	34.9	35.1	35.2	33
34	32.2	32.3	32.3	32.4	32.5	32.7	32.9	33.0	33.1	33.3	33.5	33.6	33.7	33.9	34.0	34.2	34.3	34.5	34.6	34.8	34.9	35.0	35.2	35.3	35.5	35.6	35.8	36.0	36.1	36.3	34
35	33.0	33.1	33.2	33.4	33.5	33.6	33.8	33.9	34.0	34.2	34.3	34.5	34.6	34.7	34.9	35.0	35.2	35.3	35.5	35.6	35.8	35.9	36.1	36.2	36.5	36.6	36.8	37.0	37.3	35	

**DIRECTIONS FOR USE.**

Find the Temperature of the Milk in the uppermost horizontal line, and the Specific Gravity in the first or last vertical column. In the same line with the latter, under the Temperature, is given the Corrected Specific Gravity.  
 For Example: Supposing the Temperature to be 51°, and the Specific Gravity 34°, the Specific Gravity, corrected to 60° Fahrenheit, is 32.9; or if the Temperature is 66°, and the Specific Gravity 29, the Corrected Specific Gravity is 29.8.

over, to the great variations from day to day as to quantity, source, age, and degree of acidity, churning was not, in every case, as perfect as could be desired.

The composition of 15 samples of butter, of different origin, varied within the following limits:—

Fat .. .. .	84.1 to 87.6
Water .. .. .	9.6 ,, 14.2
Curd, &c. .. .. .	.6 ,, 1.5
Ash .. .. .	.2 ,, 1.7
Insoluble fatty acids .. .. .	86.5 to 88.4

For the sake of comparison, a sample of butterine was procured, and analysed, with the following results:—

Fat .. .. .	85.8
Water .. .. .	10.0
Curd, &c. .. .. .	3.1
Ash .. .. .	1.1
Insoluble fatty acids .. .. .	95.4

It is impossible, in this paper, to enter into details with regard to those samples which come under the head of sundries. I hope, however, to make some observations on some of them in a future paper.

#### NOTE ON AN ADULTERATED SAMPLE OF TEA.

By A. BOSTOCK HILL, M.D., F.I.C.

*Read at the Meeting 11th February, 1885.*

I am induced to bring the following results of analysis before the Society, not because I have discovered anything new, as regards the adulteration of Tea, but because, now all tea is examined in bond, an adulterated sample is a great rarity.

The sample in question was bought in one of the rural districts of the County of Warwick, and submitted to me by the inspector in December last. It was a green tea, but very slightly faced. The following were the results obtained:—

Total Ash	= 12.10	} per cent.
Soluble Ash	= 1.29	
Alkalinity as Potash	= 0.13	
Sandy matter	= 6.00	
Extract	= 37.00	

The extract was obtained by boiling 1 gram. for an hour under an upright condenser, it was light in colour, but had a somewhat bitter astringent taste. On precipitating, an infusion, by neutral acetate of lead, and testing with dilute solution of ferric chloride, a green colour was produced, and a precipitate formed on standing; this, I believe, indicates that, at least, several per cent. of catechu had been added to it. A microscopical examination of the leaves showed that the great majority of them were from the tea plant, but there were several small ones, as to whose origin I have some doubt. These results show that the tea was adulterated with 6 per cent. of sand, which was not magnetic. The low amount of ash soluble in water, and the small degree of alkalinity of the ash, clearly prove the presence of exhausted leaves.

I may add that the sandy matter appeared to be all in the centre of the leaf, and not spread generally over the surface.

## NOTE ON THE OPTICAL ESTIMATION OF MILK-SUGAR.

BY ALFRED H. ALLEN, F.C.S., F.I.C.

*Read at the Meeting, 11th February, 1885.*

At the December meeting of the Society of Public Analysts, I made mention of a process of optically estimating milk-sugar, which is largely employed in America, and was communicated to me recently by Professor S. P. Sharples, a description of which appears in the February number of the ANALYST. To the description I appended a footnote, signed with my initials "A. H. A.," but which is attributed by the printer to "A. Hill."

Within the last few days, I have received a copy of a paper by H. W. Wiley (reprinted from the *American Chemical Journal*, Vol. vi, No. 5) in which he shows, by a number of experiments that, as ordinarily employed, the optical estimation of milk-sugar is below the truth, owing to the solubility of the lævo-rotatory albuminous bodies in an excess of the solution of basic lead-acetate. He proposes, therefore, to limit the amount of lead solution used. He prepares it by boiling a saturated solution of lead-acetate with excess of litharge, and diluting the solution to a specific gravity of 1.97. 1 c.c. of this re-agent, will precipitate the albuminoids from 50 to 60 c.c. of milk. Professor Wiley, however, strongly recommends the replacement of the lead solution by an acid solution of mercuric nitrate, prepared by dissolving mercury in twice its weight of nitric acid of 1.42 specific gravity, and diluting the solution so obtained with an equal measure of water; 1 c.c. of this re-agent is added to the standard quantity of milk, the solution made up with water to 102.4 c.c., agitated, and at once filtered. It will be observed that an allowance is made 2.4 c.c. for the volume occupied by the albuminous precipitate. Through the courtesy of Mr. Bodmer, I have had the opportunity of perusing an abstract of his paper "On the determination of cane-sugar in the presence of milk-sugar." I think the point established, that cane-sugar is readily inverted by citric acid, while milk-sugar undergoes no change, a very important one.

I am indebted to Mr. H. T. Brown for calling my attention to the use of invertin, the soluble ferment of yeast, as a reagent for inverting cane-sugar, but its employment seems to have been first proposed by Kjeldahl. For effecting the inversion of cane-sugar by invertin, Kjeldahl (*Zeits. Anal. Chem.*, xxii. 588) treats 50 c.c. of the sugar solution with a little concentrated alcoholic solution of thymol, and adds a little yeast previously washed and ground up with water. The thymol completely prevents fermentation without interfering with the action of the invertin. The mixture is allowed to remain for twenty-four hours at a temperature of about 50° to 52°C. It is then diluted to 100 c.c., filtered, and the cupric oxide, reducing power and optical activity estimated. From the increase in the former, and the change in the latter, the amount of cane-sugar present in the original solution can be determined.

H. T. Brown modifies the foregoing process by grinding well washed yeast in a mortar, with a little water and ether, or chloroform, and adding a small quantity of the product to the sugar solution, previously saturated with ether or chloroform. The liquid is then kept at a temperature of 30°C. for half an hour, when it is filtered and examined as before. If chloroform has been employed it must be got rid of by heating the liquid before adding Fehling's solution.

(CONCLUSION OF THE SOCIETY'S PROCEEDINGS.)

## ON THE ANALYSIS OF CONCENTRATED MEAT PREPARATIONS.

BY DR. A. STUTZER.

*(Concluded from March Number.)*

5. *Phosphoric Acid and Chlorine.*—A weighed quantity of the substance is placed in a platinum dish, and about 10 c.c. of a concentrated solution of sodium carbonate added. The whole is evaporated to dryness, ignited, the residue extracted with nitric acid, and the solution divided into two parts: in the one the chlorine is precipitated with silver nitrate, and in the other, the phosphoric acid with molybdenum. The addition of sodium carbonate is necessary to prevent the formation of pyrophosphoric acid, and the volatilization of small quantities of chlorine during the ignition.

Table showing the phosphoric acid, chlorine, and potash in the nine articles above described in the same order:—

	Phosphoric Acid.	Chlorine.	Potash.
Benger's Peptonised Beef Jelly .. ..	·30	·16	·53
Brand's Essence of Beef .. ..	·19	·06	·20
Carnrick's Beef Peptonoids .. ..	1·27	1·41	1·33
Johnston's Fluid Beef .. ..	1·91	·84	1·72
Kemmerich's Extract of Beef .. ..	6·56	·85	8·30
Liebig's Extract of Beef . . . .	7·83	·84	10·18
Murdoch's Liquid Food .. ..	·10	·05	4·17
Savory and Moore's Fluid Meat .. ..	1·49	2·67	4·20
Valentine's Meat Juice .. ..	3·76	·05	5·11

6. *Fat* is determined by extracting with ether in the well-known way. A simple and good extraction apparatus has been described by me in "Böckmann's chemisch-technische Untersuchungsmethoden, p. 579, where a drawing of the same is also to be found. The only one out of the nine examined that was found to have any fat was Carnrick's Beef Peptonoids, which yielded 10·67 per cent.

7. *The Determination of the Nitrogen* (total quantity) was conducted by me in the following way:—The dry substance, or if liquid, after evaporation with calcium oxalate in small dishes of thin tin, is ignited in an iron tube with soda-lime, and the ammonia given off absorbed by titrated sulphuric acid. Space does not allow of my describing in this article the great advantages obtained by using an iron tube, instead of the usual glass one, in the ignition of nitrogenous substances. Notwithstanding these advantages, however, I have lately seldom used the iron tubes, as I prefer, in many cases, to estimate the total nitrogen in animal foods by Kjeldahl's (*Zeitschrift für Analytische Chemie* 1883, s. 366–382) method, as this method also gives very good results, and obviates the troublesome evaporation to dryness of the substance to be examined.

Nitrogen occurs in the examined animal food substances in four forms: as digestible albumen, indigestible albumen, pepsin, and in form of meat bases (creatine, carnine, etc.)

8. *Insoluble Albumen.*—Albuminoids are resolved by the acid juices of the stomach (Pepsin) into a digestible and an indigestible part. This decomposition takes place in such a manner that it is possible to separate the digestible from the indigestible nitrogen compounds with the greatest accuracy, and to determine them. The soluble part of the albuminoids, calculated from the nitrogen dissolved by artificial digestion,

by multiplying with the factor, 6.25 (making the usual assumption that the albuminoids contain on an average 16 per cent. nitrogen), I call *Digestible Albumen*:—the indigestible part, containing, besides C H O and N, also S and P, in the form of organic compounds, *Indigestible Albumen*.

The digesting liquid (pepsin solution) is prepared in the following way:—The mucous membrane of the fresh stomach of a pig is cut into small pieces with the scissors, and placed in a bottle with a large mouth, together with 5 litres of water, 2.5 grms. salicylic acid, and 75 c.c. hydrochloric acid (in 100 c.c., 10 grms. HCl). The mixture is allowed to stand two or three days, and is frequently shaken. It is then poured through flannel, without pressing, and filtered through paper. The digesting experiments are made by me in a water-bath, made of strong sheet metal, 75 c.m. long 50 c.m. wide, and 15 c.m. high. This size allows of 10–12 experiments being carried on at the same time. The water in the water-bath is kept at about the same level as the liquid in the glass tubes, and can be so regulated, that, after heating early in the morning, with a large flame, to 40° C., the temperature, even when the gas pressure changes, does not alter more than 1–2°.

The examination is conducted as follows:—2 grms. of Carnrick's beef peptonoids, or 4 grms. of Johnston's fluid beef (the other meat preparations containing no insoluble albuminoids), are brought each into three different beakers, covered with  $\frac{1}{4}$  litre of the Pepsin solution, containing 0.15 per cent. HCl (as described above), and warmed one hour on the water-bath, at 40° C.; at intervals of 1–2 hours more, 0.1 per cent. hydrochloric acid is added, until, after being kept at 40° C. for twelve hours, the 250 c.c. Pepsin solution have received 2.5 grms. HCl. The hydrochloric acid is used in the form of a 10 per cent. solution (100 c.c. = 10 grms. HCl). The insoluble part is filtered through a good filter-paper (the nitrogen of which is known), and washed with water, until the filtrate gives no further turbidity with silver nitrate. It is then washed twice with alcohol, to remove the water as far as possible, and to accelerate the drying, dried at 100–110° C., and the indigestible nitrogenous substance estimated by ignition with soda-lime. The filter-paper is cut in small pieces, and ignited with the substance (its nitrogen being afterwards subtracted from the total quantity), as it is impossible to free the precipitate with quantitative accuracy from the paper. A good filter-paper contains not more than 0.00001–0.00002 gm. N., and in such papers the nitrogen may be neglected. The determination of the nitrogen in the presence of filter-paper, according to Kjeldahl's method, is not to be recommended.

In quoting analyses, I am accustomed only to give the quantity of nitrogen present as indigestible albumen; I do not make the calculation with the factor 6.25, as the composition of the indigestible has not yet been determined.

9. *Digestible Albumen*.—The digestible albumen differs, as I have mentioned above, from the indigestible albumen by being soluble in the acid juices of the stomach. It differs from the other nitrogenous substances, pepsin and the meat bases, by the property the part soluble in water possesses of forming with  $\text{CuO}_2\text{H}_2$ , a compound insoluble

in neutral liquids; and it is possible to use this property for a separation method. I will first describe the way in which the  $\text{CuO}_2\text{H}_2$  is prepared:—

100 grms. cupric sulphate are dissolved in 5 litres of water, and 2.5 grms. glycerine added. The cupric-hydrate ( $\text{Cu O}_2 \text{H}_2$ ) is then precipitated by adding sodium hydrate until the solution is alkaline, the precipitate filtered, and rubbed up in a dish, with water containing 5 gm. glycerine per litre  $\text{H}_2\text{O}$ . The last traces of alkali are removed by repeated decantation and filtration. After again filtering, the precipitate is finally rubbed up with water, containing 10 per cent. glycerine, so as to form a homogeneous mass capable of being sucked up with a pipette. The amount of  $\text{CuO}_2\text{H}_2$ , which should be about 0.4 gm., is quantitatively estimated in 10 c.c., and the whole preserved in well-stoppered bottles. The application of glycerine, in the preparation of the cupric hydrate, is necessary to prevent  $\text{CuO}_2\text{H}_2$  splitting up into  $\text{CuO} + \text{H}_2\text{O}$ .

For the analysis, 1 gm. of the dry food is placed in a beaker, 100 c.c. water added, and the whole boiled. In the case of fluid, or extract foods, a greater quantity is weighed out, dissolved in water, and diluted to a certain volume. From this solution a quantity is taken, by means of a pipette, equivalent to about 1 gm. of the dry substance. This is diluted to about 100 c.c., and brought to boiling.

As soon as the liquid is cool down to 40–60°C, 10 c.c. of the cupric hydrate is added, the mixture well stirred, and filtered. The residue is washed on to the filter with water, the filter filled twice with alcohol to remove, as far as possible, the water from the filter, dried at 100°C, and ignited with soda-lime, as described above. From the nitrogen thus found is subtracted the nitrogen determined as indigestible albumen, and the remainder taken as *digestible albumen*. Following the usual custom, I have calculated this nitrogen as albumen, by multiplying with the factor 6.25.

10. *Peptone*.—The peptones, which, from a physiological point of view, are equivalent to the digestible albumen, possess the property of forming soluble compounds with  $\text{CuO}_2\text{H}_2$ , and of being precipitated with phospho-tungstic acid, while the meat bases can be precipitated neither with cupric hydrate, nor with phospho-tungstic acid.

A few grams of the substance under examination are exactly weighed out, warmed with 100 c.c. water in a beaker, the requisite quantity  $\text{CuO}_2\text{H}_2$  added, filtered, and the residue well washed. The filtrate is evaporated to about 30 c.c., mixed with 15 c.c. of a 20 per cent. hydrochloric acid solution, and the peptones precipitated with a concentrated solution of sodium phospho-tungstate. The precipitate is filtered, washed with dilute sulphuric acid (50 gm.; 1 liter  $\text{H}_2\text{O}$ ) and the sulphuric acid removed by washing several times with absolute alcohol. The filter and contents are then dried at 100°C, and the nitrogen determined by ignition with soda-lime, in the way already described. Peptones have almost exactly the same composition as albumen, and the peptone may be calculated from the peptone-nitrogen, in the same way as the albumen was calculated from the albumen-nitrogen, by multiplying by the factor 6.25. I do not maintain that this factor is right under all circumstances; we shall have to use it until we have more facts concerning the chemical composition of the albuminoids and peptones, and by a convention of analytical chemists have replaced the number 6.25 by more accurate factors.



11.—*The Nitrogen in the form of meat bases* can be found by collecting carefully all the filtrates after the removal of the peptones, evaporating and estimating the nitrogen in the residue. In most cases this troublesome process can be replaced by an indirect method, which consists in subtracting from the total nitrogen, the nitrogen in the form of indigestible albumen, digestible albumen, and peptone, and taking the difference as nitrogen in the form of meat bases.

Table showing the distribution of the total nitrogen found in the nine articles examined:—

	As Albumen easily digested.	As Peptone easily digested.	As Meat Bases (Creatine, &c.)		Total.
			As Albumen not digestible.	and other nitrogenous compounds.	
Benger's Peptonised Beef Jelly ..	0.386	0.741	—	0.422	1.549
Brands' Essence of Beef .....	0.360	0.969	—	0.151	1.483
Carnrick's Beef Peptonoids .....	9.060	1.110	0.220	0.100	10.490
Johnston's Fluid Beef .....	2.824	2.837	0.148	1.394	7.203
Kemmerich's Extract of Beef .....	1.258	2.308	—	6.167	9.733
Liebig's Extract of Beef .....	0.848	0.248	—	7.782	8.914
Murdoch's Liquid Food .. .....	2.066	0.037	—	0.187	2.290
Savory and Moore's Fluid Meat....	0.869	0.427	—	7.472	8.768
Valentine's Meat Juice .....	0.292	0.760	—	1.448	2.500

Taking these amounts as calculated on the original substance, we get what I consider to be an accurate estimate of the actual relative values of these concentrated foods. To save trouble they are this time placed in their relative position of value, according to the views hereinbefore expressed, as to the high value of the readily digestible peptones and albumen they respectively contain, as distinguished from the merely stimulating action of the meat bases, such as creatine, carnine, and other similar nitrogenous bodies:—

	Albumen.	Peptones.	Total per
	Easily digestible	Easily digestible	centage value.
Carnrick's Beef Peptonoids .....	56.625	6.937	63.562
Johnston's Fluid Beef .....	17.650	17.730	35.380
Kemmerich's Extract of Beef .....	7.862	14.24	22.287
Murdoch's Liquid Food .....	12.912	0.231	13.143

The remaining five being under ten per cent. are not classified.

The article which appeared to be most valuable as food, namely, Carnrick's Beef Peptonoids, was a fine dry powder made of meat, wheat gluten, and evaporated milk. In another paper I have more minutely described the characteristics of each of the articles examined, but I do not do so here, as being out of place in a purely chemical paper, designed to show what I consider should be the true basis of the analysis of concentrated foods.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

THE EXAMINATION OF PEPPER.\*—By CHR. NEUSS.—Pharm. Ztg. [1885]. 30–26.

—The author recommends the following simple method. The powder under examination is covered with concentrated hydrochloric acid. The particles of pepper then assume an intense yellow colour, so that all foreign substances at once become visible. If the powder be not very fine, the small grains may be separated and determined approximately.

F. II. II.

**THE DETERMINATION OF SALICYLIC ACID IN WINE.\***—By G. HEINZELMANN, *Zeitschr. f. Spiritusindust.* [1884] 7.996.—The author has tested the different methods for the determination of small quantities of salicylic acid in fermented liquids. His results, however, were too low. This is probably due to the salicylic acid being carried over with the alcoholic vapours; and the author, therefore, recommends the following modification of Pellet-Grobert's method:—50 c.c. of the liquid acidulated with sulphuric acid are shaken up with ether, and the ethereal extracts, after fixing the salicylic acid with soda, distilled on the water-bath. The residue is evaporated to dryness, acidulated with sulphuric acid, and extracted with benzol. To the benzol-extract is added ferric chloride; the amount of salicylic acid is then calculated from the intensity of the violet-coloration by comparison with solutions containing a known amount.

To determine whether the salicylic acid is fixed or free, the liquid is distilled in a current of steam. The steam carries the free salicylic acid over, which can then be detected in the distillate with ferric chloride. F. H. H.

**A NEW METHOD FOR THE DETECTION OF COPPER AND LEAD IN WINE.**—By H. HAGER, *Pharmaceut. Centralhalle.* No. 8, p. 78 [1885]. The wine to be tested is mixed with half its volume of a clear solution of caustic soda, which may contain carbonate. The liquid should remain clear, even when boiled. A turbidity with coloration points to the presence of copper. About six c.c. of this solution are poured into a test-tube, a piece of tin-foil introduced, and the whole heated for half-an-hour on the water bath. If lead and copper be absent, the foil remains perfectly bright, whereas, in the presence of a trace of one of these metals, it becomes dull and gray, with larger traces dark gray—greyish-black for lead, and brown or brown-black for copper.

This re-action may be applied to all cases when other metals are not present, and the solution remains clear after the addition of alkali. F. H. H.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

**DETERMINATION OF MORPHINE IN OPIUM.**—On comparing the methods of E. Merk, Hager-Godeffroy, the *Pharmacopœa Austriaca*, and the one proposed by himself, V. Perger (*Jour. f. prakt. Chem.*), finds most surprising differences in results obtained. The author's method is as follows: The opium is heated with barium hydrate and water, filtered, and washed with hot water. The filtrate containing all morphine, is saturated with carbon dioxide, and the whole rapidly evaporated to dryness on a water-bath. The dried mass is extracted with absolute alcohol, and the alcoholic filtrate freed from alcohol by distillation. The residue is allowed to stand some time with ammonia water, brought upon a filter, washed with water containing ammonia, then with chloroform, then dried and weighed. This crude morphine may be purified by dissolving in acetic acid, adding a few drops of potassium ferrocyanide, filtering, neutralizing the filtrate with ammonia, and allowing the morphine to crystallize out, collecting the crystals upon a filter, drying and weighing. W. H. D.

\* Chem. Zeitung.

A REACTION FOR CHLORAL HYDRATE.—By M. HIRSCHFELD.—Archiv der Pharm. [1885]. 223. Hft. 1, p. 26.—If to a solution of chloral hydrate, calcium sulphhydrate, Ca (SH)<sup>2</sup>(Rhusma) be added, a red colour is produced on standing half-a-minute, which afterwards turns purple-red. The filtered solution contains the colour. A weaker, but still visible reaction is obtained, when first sulphuretted hydrogen and then lime-water is added. After about a minute, the liquid becomes reddish pink.

F. H. H.

---

### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ANALYSIS OF INDIGO.—Dr. H. M. Rau, in the Journal of the American Chemical Society, gives the following method—a modification of Fritsche's:—An 8 oz. Erlenmeyer flask is provided with a doubly perforated rubber stopper. Through one perforation passes a syphon tube, the lower end of which (inside the flask) is enlarged to funnel form, in which is placed a plug of glass wool; through the other perforation passes a tube terminating just below the cork, bent at right angles, and provided with a stop-cock. This flask is first weighed empty, and then there are introduced 1½ to 2 grammes of the sample of indigo, 3 to 4 grammes of grape-sugar, 15 to 20 c.c. of a 40 per cent. caustic soda solution, and about 120 c.c. of 90 per cent. alcohol—sufficient to bring the bulk up to about 7 fl. oz.

The apparatus thus charged is then weighed, the syphon tube closed with a rubber tube and pinch cock, the stop-cock closed, and the flask heated on the water bath for 25 to 30 minutes, with frequent shaking. At intervals the stop-cock is momentarily open and closed, to relieve the pressure of the alcohol vapours. The indigo readily dissolves, the impurities settling to the bottom. After solution is complete, the flask is allowed to stand for an hour, and so much as possible of the solution run off into a beaker through the syphon tube, the glass-wool acting as a filter to restrain any insoluble impurities. Another weighing of the flask gives the necessary data for the calculation of the proportion of the sample contained in the solution run off. Through this clear solution a current of CO<sub>2</sub> is passed for fifteen minutes; finally, air is passed through to complete the oxidation. The indigotine and indinibine precipitate in copper-coloured flaky crystals, which are collected on a weighed filter, washed with boiling water, then with hot dilute HCl, and finally with water, and then dried and weighed.

Dr. Rau believes that all oxidation processes for the valuation of indigo give unreliable results, since a large proportion of the impurities absorb oxygen, and are reckoned as indigo.

E. W.

---

LAKMOID AS AN INDICATOR. BY M. C. TRAUB. *Archiv der Pharm.* [1885], 223, Hft. 1, p. 26.—The author describes a substitute for litmus as an indicator. Lakmoid is a substance resembling litmus, obtained by the action of sodium nitrite upon resorcin. This reaction has already been communicated by the author (*Ber. d.d. Chem. Gesell.* XVII. 2,616.)

The substance is prepared in the following way :—A mixture of 10 parts resorcin, 1 pt. sodium nitrite, and 1 pt. distilled water are heated at a temperature which must not exceed 120° C, until ammonia ceases to be evolved. The fused mass is dissolved in 10 pts. distilled water, and excess of hydrochloric or acetic acid added. The precipitate formed is collected on the filter, washed, and dissolved, after drying at 100° C, in absolute alcohol. The filtered solution is then allowed to evaporate in the exicator. Lakmoid crystallises in red-brown shining plates.

For the indicator, 0.5 grms. are dissolved in about 100 c.c. of a solution consisting of equal parts of alcohol (96 per cent.) and water. This solution is very delicate ; ammonia solution of strengths  $\frac{N}{100}$ ,  $\frac{N}{200}$ ,  $\frac{N}{400}$ ,  $\frac{N}{800}$ ,  $\frac{N}{1000}$  are distinctly coloured blue by two drops ; with a solution  $\frac{N}{5000}$  the indicator gives, just perceptible colouration.

The author maintains that his indicator is more delicate than litmus, and more applicable than phenolphthalein.

Lakmoid-paper may also be prepared by adding 5 drops of potash to a solution of lakmoid in 45 per cent. alcohol (1 pt. lakmoid : 1,000 pts. alcohol), and soaking filter-paper in the solution thus prepared.

F. H. H.

DETECTION OF MONOCARBONATE IN SODIUM BICARBONATE. *Pharm. Blatt.* Bd. 17, p. 849.—Monocarbonate is easily to be detected in sodium bicarbonate according to A. Kremel, by testing with phenolphthalein solution, which gives a red colouration when a trace of monocarbonate is present.

F. H. H.

A NEW METHOD FOR THE SEPARATION OF COPPER FROM CADMIUM. BY P. GUCCI. *Ber. d.d. Chem. Ges.* 17, 2,659.—Bismuth having been removed by precipitation with excess of ammonia, nitric, hydrochloric, or sulphuric acid is added in sufficient quantity to dissolve the precipitate at first formed. A small excess of a 10 per cent. solution of ammonium benzoate is then added, which precipitates the copper as cupric benzoate. The cadmium is detected in the filtrate with ammonia and ammonium sulphide. By using a small excess of ammonium benzoate great exactitude may be obtained in the separation, and the method admits of application in quantitative analysis.

F. H. H.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

TO THE EDITOR OF "THE ANALYST."

DEAR SIR,—I was unfortunately prevented from being present at the meeting of the Society of Public Analysts, at which Mr. Estcourt's paper, printed in your last number, was read, so could make no remarks on it. Will you give me space for a few words on the subject in your next? Mr. Estcourt says I have misunderstood the purpose of his paper, which is to give a money value to milk. If this be his purport I certainly mistook it. I thought he meant to bring before Public Analysts a mode of valuation which would enable them to fill up their certificates more accurately and fairly than they do at present, and a re-perusal of his paper leaves the same impression on my mind. When he says "I propose that a milk which contains such a percentage of non-fatty solids and fat solids as multiplied by their respective factors will together produce 100 shall be considered milk of full value," I concluded he meant that a Public Analyst was to certify it as "genuine milk," and against this I protested. There may be a difference of opinion as to whether butter-fat and water are of more money value than milk, but I should hope there is no one who would seriously assert that it is genuine milk, and this is what the Public Analyst must do in some cases if Mr. Estcourt's system, as I understood it, were adopted. A Public Analyst must say either that a milk is genuine, or it contains so much added water, or it is skimmed, no other course is open to him. I am very far from undervaluing fat, but I cannot say that an excess of fat on a clearly watered milk makes it into "genuine milk." It was to meet cases of this kind that I proposed the mode of valuation contained in my paper read on December 17th.

Let us take the case of the milk I then mentioned.

Non-fatty solids	$6.01 \times 7.85 = 47.17$
Fat	$8.39 \times 11.10 = 93.12$

Mr. Estcourt's value	140.29
----------------------	--------

Now this milk I certified watered to the extent of 20 per cent. and in the observation column said "The large quantity of fat in a milk so manifestly watered leads to the belief either that it is purposely made up to deceive, or that it is dipped off the top of a churn of watered milk that has been standing some time." The latter was proved to be the case. Can anyone think that the regular customers of the vendor of this milk (in this particular instance the Infirmary) got milk with over 8 per cent. of fat? I think not. The top dip was specially intended for the Inspector, the milk would be well mixed before the patients got it. Now if any valuation in which fat is allowed to pass in the place of non-fatty solids be adopted, such a case would pass undetected and we should be landed in this absurdity—the man who simply watered his milk would be convicted, while the man who added to the crime of watering his milk, the further offence of doing his best to jockey the Inspector and deceive the Analyst, would be certified to have sold genuine milk, if indeed it were not called extra rich.

Curiously enough the very week in which I read my paper I had another sample of a dip off the top of a churn, but this time it was a churn of milk, not milk and water, the result of Analysis T S 17-85 S N F 9-14 F 8-71, very nearly the same fat as the previous one. The solids-not-fat in the milk deprived of fat were therefore 10.09 per cent. The contrast between these two samples very well illustrates the respective merits of the two modes of valuation.

I remain, Dear Sir,

Yours faithfully,  
CHAS. HEISCH.

Laboratory, 79, Mark Lane, March 24th 1886.

## BOOKS, &amp;c., RECEIVED.

Acetic Acid and Vinegar, Ammonia and Alum, edited by John Gardner, F.I.C., F.C.S.; The Medical Annual and Practitioner's Index, 1884-5; The Modulus of Hydrometers, by E. Waller and N. Hathaway; Report of the Committee of Public Health of New York; Report on the Health of the Rural District of Ashton, by A. Bostock Hill, M.D.; Report to the New Jersey Board of Health on Butter Analysis, &c., by H. B. Cornwall, Ph. D., F.C.S.; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; British and Colonial Druggist; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Illustrated Science Monthly; Independent Journal; Invention; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; New York Analyst; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Publishers' Circular; Reportorium der Analytischen Chemie; San Francisco News Letter; Science; Scientific American; Society of Arts Journal; Hospital Gazette.

# THE ANALYST.

MAY, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
(a) REPORT OF ORDINARY MEETING.. ..	81	(a) DETECTION OF PETROLEUM ETHER IN ETHER, D. VITALI .. ..	95
(b) AN IMPROVED APPARATUS FOR MILK ANALYSIS, By W. JOHNSTONE, F.I.C.	81	(b) QUANTITATIVE DETERMINATION OF ALOES IN MIXTURES, H. HAGER .. ..	95
(c) DISCUSSION ON MR. ADAMS'S MILK PROCESS .. ..	85	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
(d) MR. ADAMS'S REPLY .. ..	90	(a) DETERMINATION OF TITANTIC ACID IN IRON AND IRON ORES, A. LEDEBUR	96
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—		(b) DISTINGUISHING BETWEEN CHLORIDES, BROMIDES, AND IODIDES, H. HAGER	97
(a) NEW APPARATUS FOR THE DETERMINATION OF STARCH, R. REMPEL .. ..	94	REVIEWS:—	
(b) DETERMINATION OF STARCH IN GRAIN, M. MARKEE .. ..	94	(a) FRESSENIUS'S QUANTITATIVE ANALYSIS, VOL. II. .. ..	97
(c) DETERMINATION OF CASEIN IN COW'S MILK, FRENZEL AND WEYL .. ..	95	(b) ACETIC ACID, &c. (CHURCHILL'S TECHNOLOGICAL HANDBOOKS, No. 3).. ..	98
		LAW REPORTS—IMPORTANT DECISION .. ..	98
		BOOKS RECEIVED—NOTICES, &c.	

### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The ordinary meeting of the Society was held in the Rooms of the Chemical Society, Burlington House, on Wednesday, 29th April, having been specially deferred from the regular date. The President, Dr. A. Hill, was in the chair.

The following gentlemen were elected members of the Society, viz.:—Mr. H. Sugden Evans, Chief Public Analyst to the Dominion of Canada, Montreal; Mr. J. A. Bond, Analyst, Auckland, New Zealand; Mr. C. G. Stewart, Analytical Chemist, St. Thomas' Hospital.

Mr. H. C. Williams, Assistant to Mr. Kingzett, was elected an Associate.

Mr. M. A. Adams, F.R.C.S., read his reply to the criticisms passed upon his milk process, and after some further business the meeting was adjourned.

### AN IMPROVED APPARATUS FOR MILK ANALYSIS.

By WILLIAM JOHNSTONE, F.I.C., F.C.S., &c.

(Read at the Meeting on 11th March, 1885.)

The apparatus I have to describe is one based upon the same principle as that of Soxhlets, and like it, may be used for the continuous extraction of a substance by means of a volatile fluid, and, when once started, it requires no further attention until the extraction is completed.

As you will observe, it is composed of an upright Liebig's condenser, made of copper, nickel plated, and having the internal or condensing tube of a comparatively wide bore, so as to expose a large condensing surface. To the lower projecting portion of the condensing tube, and at a distance of about four inches from the end, a collar of brass, 3½ inches in diameter, is attached, having what is called a bayonet catch. To this a

cylindrical glass vessel, about seven inches in length, being also fitted with a brass collar, can be attached, and made air-tight by means of an india-rubber washer. Passing through the top portion of the brass collar attached to the condensing tube, there are two small tubes, which are inserted or terminate in the condensing tube above.

Having thus explained the construction of the apparatus, I will now endeavour to describe the process of extraction.

The platinum basin, containing the perfectly dry residue, is placed into a cylindrical funnel, having a plug of cotton or glass wool, the end of which is inserted into a beaker, previously weighed.

The beaker and funnel is then inserted into the large glass vessel of the apparatus, and properly attached to the condenser.

The position or height of the platinum basin must be such, that the end of the condensing tube within the glass vessel must be inside of the platinum basin, and reaching to within about an eighth of an inch of the milk residue, or bottom of the platinum basin. When all is in proper position, ether is then cautiously poured through the upper part of the condenser, in quantity more than sufficient to fill the platinum basin.

The outside vessel is then heated by means of a water bath, when distillation commences. Upon the addition of the ether the end of the condensing tube becomes immersed in ether, thereby nominally closing the tube, and causing the ether-vapour to ascend through the lateral tubes into the upper and cool part of the condenser, and being there condensed, falls back into the basin below, displacing an equal amount of ether containing fat, which, in its turn, falls into the weighed beaker below.

It is evident, therefore, that so long as heat is applied to the outer vessel, there will be a continual evaporation and condensation in turn, and therefore a constant overflow of the platinum basin, and that after a sufficient time has been allowed to elapse, the milk residue will have been thoroughly exhausted of all its fat.

The use of the cotton or glass wool is obvious, namely, to allow of the fat being estimated directly by weighing the beaker, or indirectly by weighing the platinum basin after extraction.

The last method is generally applicable, except when rendered inaccurate by the ether carrying over some of the solids-not-fat. To be always able to estimate the fat and the solids-not-fat, the wool (or wool + funnel) must be previously weighed.

The particles of residue (if any) carried over by the ether, are retained by the wool, and by drying and weighing the same the amount is thereby ascertained, the gain in weight added to the weight of residue in the basin gives the amount of solids-not-fat contained in the milk.

Having thus far described the apparatus and process of extraction, I wish now to draw your attention to the following series of results, and also to mention a new process of milk analysis.

The results that I have tabulated are the average results of duplicate analyses of six samples of milk, analysed by four different processes of analysis.

Before describing the different methods employed, I may as well mention that in every instance the residues were dried to constancy, and out of the forty-eight analyses

represented in the table, in no instance was there ever observed an appreciable increase in the weight of the total solids, owing to a supposed oxidation of the fat upon prolonged drying, a theory which I hope will not gain so much notoriety as the milk-sugar one did. The processes employed were as follows:—

W. The ordinary Wanklyn extraction.

S. Extraction by means of Soxhlet's apparatus.

J. By my blotting-paper process immediately to be described, and in the apparatus I have just described.

A. By coagulating the milk with acetic acid, and then extracting in apparatus above described.

What I call my new process is extremely simple, and consists in the employment of very thick blotting-paper in conjunction with flat bottomed platinum basins.

The basins I have been using are  $2\frac{1}{2}$  inches in diameter, and the blotting-paper is cut in circles of such a size that they go easily into the basin, absorb the milk, and lie perfectly flat on the bottom.

When the residue is perfectly dried, that is when it ceases to lose weight, it is weighed, and the extraction of the fat then commenced in the apparatus before you.

When sufficiently extracted the basin is removed, and again dried and weighed, when we obtain the solids-not-fat, or if desired, the fat can be recovered and also weighed.

The results on the diagram, although not altogether satisfactory, are of themselves of sufficient interest as to justify me in laying them before you; however, if it had not been for the paper read by Mr. Adams on 28th January, I should not have brought these results so soon before the Society, as I have a much larger number of experiments on hand, the results of which I intend to lay before you on some future occasion.

As mentioned, I have made a large and extensive series of experiments in the direction indicated by Mr. Adams in his paper, namely, in trying to adopt blotting-paper mechanically, so to speak, in milk analysis; my object in so doing being precisely similar to that of Mr. Adams's, namely, to overcome, if possible, the nature of the horny residue so often obtained when a milk residue has been completely dried; that is, when constant weighings have been obtained, and at the same time to allow the fat to be easily extracted from the residue so obtained.

With this end in view I was led to adopt, as I have mentioned, circular dishes of thick blotting-paper, using flat bottomed platinum basins, similar to the ones I have just exhibited, and from an examination of the tabulated results it will be seen, with a certain amount of success. There is, however, one particular peculiarity between the results obtained by Mr. Adams and myself when using the blotting-paper, and that peculiarity is, that Mr. Adams always obtains by his process total solids *lower* than those obtained by the whole method (except in one instance) and that I always obtain results higher; the difference, however, I think, will be again accounted for by our old friend, the milk-sugar, so under those circumstances I refrain from making any further remarks at present, but, in conclusion, I have to express a hope that the Milk Committee will not overlook the coagulation of the milk by acetic acid.



## AVERAGE OF DUPLICATE ANALYSES.

		W.	S.	J.	A.
I.	Total Solids	8.47	8.43	8.71	8.60
	Solids-not-Fat	6.37	6.17	6.26	6.08
	Fat	2.10	2.26	2.45	2.52
	Variation in the Duplicate.				
	Total Solids	0.07	0.01	0.01	0.09
	Solids-not-Fat	0.08	0.03	0.03	0.05
II.	Total Solids	12.63	12.56	12.78	12.85
	Solids-not-Fat	9.59	9.48	8.92	8.87
	Fat	3.04	3.08	3.86	3.98
	Variation in the Duplicate.				
	Total Solids	0.01	0.09	0.07	0.05
	Solids-not-Fat	0.01	0.09	0.13	0.11
III.	Total Solids	12.82	12.61	13.38	13.02
	Solids-not-Fat	9.67	8.90	9.21	9.05
	Fat	3.15	3.71	4.17	3.97
	Variation in the Duplicate.				
	Total Solids	0.01	0.05	0.03	0.05
	Solids-not-Fat	0.16	0.08	0.14	0.13
IV.	Total Solids	8.92	8.86	9.22	9.06
	Solids not-Fat	6.91	6.77	6.93	6.71
	Fat	2.01	2.09	2.29	2.35
	Variation in the Duplicate.				
	Total Solids	0.01	0.05	0.02	0.00
	Solids-not-Fat	0.01	0.13	0.13	0.09
V.	Total Solids	12.13	12.26	12.25	12.37
	Solids-not-Fat	9.52	9.26	9.31	9.22
	Fat	2.61	3.00	2.94	3.15
	Variation in the Duplicate.				
	Total Solids	0.01	0.09	0.10	0.06
	Solids-not-Fat	0.06	0.14	0.03	0.15
VI.	Total Solids	14.00	14.07	14.30	14.22
	Solids-not-Fat	10.27	10.00	9.27	9.11
	Fat	3.73	4.07	5.03	5.11
	Variation in Duplicate.				
	Total Solids	0.00	0.05	0.03	0.06
	Solids-in-Fat	0.01	0.10	0.01	0.12
	The above sample taking Total Solids throughout at 14 per cent.				
	Total Solids	14.00	—	—	—
	Solids-not-Fat	10.27	10.00	9.27	9.11
	Fat	3.73	4.00	4.73	4.89
	Variation in the Duplicate.				
	Total Solids	0.00	—	—	—
	Solids-not-fat	0.01	0.10	0.01	0.12

## History of Samples.

I. Delivered on the 27th October, 1884, at my private address.

II. Delivered on the 1st November, 1884, after having been accused of watering the milk delivered on the 27th.

- III. Delivered on the 8th November, at my private address.
- IV. Milk delivered to house in Great Winchester Street, City, on 11th November.
- V. Delivered on 17th November, at my private address.
- VI. Delivered on 15th December, at my private address.

### DISCUSSION ON MR. ADAMS' NEW MILK PROCESS.

(For original paper see *Analyst*, vol. ix., No. 108, March 1884.)

Dr. Vieth said:—If you consider, that I have devoted the last nine years almost exclusively to analytical work relating to milk, you will, I think, admit, that nobody can be more interested than I am in the new method for the analysis of milk suggested by Dr. Adams. If the paper method stands the test; if none of the methods hitherto used allows to extract all the fat, nor to expel all the water; if the new method gives more exact results; nay, if it is the only one which gives exact result at all; then we can do nothing better, but do away with our present methods and adopt the paper method without delay. But with condemning our old methods, we also condemn all the results, which have hitherto been arrived at and published in connection with milk analysis, for there does not seem to be a hard and fast relation between the results obtained when following the methods hitherto used, or when employing the new paper method. Until now the question has chiefly, not to say exclusively, been, whether the extraction of the fat was done accurately and exhaustively, or not; of course, the non-fatty solids were effected in an equal degree. The paper method opens a new question, which everybody used to look at as settled, viz., how to get exact results as to total solids.

In my opinion every analyst, who takes a real interest in milk analysis, should set to work at once and test the paper method. In order to do my share of this work I have made some experiments, which I am going to communicate to you, and in reference to which I wish to say most emphatically, that they have been carried out with the greatest accuracy and with no other object but the sincere desire to arrive at the truth.

When I in this room urged you, more than once, to give up the old way of determining the fat by boiling the milk solids contained in a small platinum capsule with successive quantities of ether, and adopt a more exact method, pointing to the much higher results obtained by employing the plaster of Paris method, I was repeatedly asked: do you extract the milk fat only, or do you extract something besides? It was exactly this question, which I put to myself, when seeing the larger figures for fat, found by using the paper method. To clear up this point I made the following experiments:—

1. In order to satisfy myself, that the ether employed for the extractions was pure, 80 cc.,—the quantity commonly used—were distilled from a flask; it left behind no residue whatever.

2. A Soxhlet apparatus was kept going empty, that is to say, containing nothing except ether, for four hours. When the flask was dried and weighed, its weight was found to have increased by .0007 and a coating was visible inside. I believe this to be wax or resin washed out from the corks.

3. Experiments with filter paper cartridges, as used for the plaster of Paris method.

Extracted from—

1. Cartridge '0036
2. " '0038 containing 20 gr. plaster of Paris.
3. " '0047 " " " " "
4. " '0059 " " " " " wetted with 5 cc. of water and dried.
5. " '0048 " " " " " " " solution of milk-sugar and dried.
6. " '0058 " " " " " " " " " " "

4. Experiments with blotting paper, rolled up in coils, received from Dr. Adams.

Extracted from—

1. Coil '0120
2. " '0138
3. " '0180 wetted with 5 cc. solution of milk-sugar and dried.
4. " '0200 " " " " "
5. " '0185 " " " " "
6. " '0172 " " " " "
7. " '0157 " " " " "
8. " '0164
9. " '0151
10. " '0145
11. " '0148
12. " '0123
13. " '0126
14. " '0144

Coil No. 7 had been kept covered with ether over night, and was then extracted for three hours, yielding '0157 grm. ether extract. When treated a second time in precisely the same way, another yield of '0072 grm. ether extract was obtained, proving that it is rather difficult and timetaking to free this kind of paper entirely of matter soluble in ether.

The coils, No. 1 to 6, were evidently not of the same material as those numbered from 7 to 14. The ether extract of the former had a distinct smell of vanilla, proving, I believe, that coniferous wood had been employed in the manufacture of the paper in question. This smell was not noticed with the latter coils, which further distinguished themselves by sucking up milk or other liquids less readily.

The duration of extraction was generally three hours, but in several cases was preceded by keeping the object covered with ether over night.

There is some greasy matter extracted by ether from the filter paper which I use for my plaster cartridges, as well as from the blotting paper used in the paper method. The filter paper cartridge, weighing about 1.5 grms. gives about '005 ether extract; the blotting paper coil, weighing about 4.5 grms. gives from '015 to '020 ether extract. As for the plaster method 10 grms. of milk are taken, the error amounts to '05 per cent., whilst in the case of the paper method, for which 5 grms. are taken, the error is multiplied by twenty, and equals to a plus of fat of '3 to '4 per cent.

An excess of fat or rather ether extract found will, of course, depress the non-fatty solids, but the total-solids should come out the same by any method of drying, provided that all the water has been expelled and no appreciable oxidation has taken place. The paper method seems to give, in the majority of cases, total solids much below the usual drying in a platinum capsule. My next endeavour therefore was to find out, if any

and which of the dissolved constituents of milk retains water, when dried in the ordinary way.

I used for the experiments two solutions of milk-sugar, a solution of casein and one of albumen, and determined the solids in the usual way, as well as by means of paper coils. The results were as follows:—

	<i>Platinum capsule.</i>	<i>Paper coil.</i>
Solution of Milk-sugar .....	4·685 per cent.	4·770 per cent.
„ „ .....	9·425 „	9·360 „
„ Casein .....	3·229 „	3·334 „
„ Albumen .....	3·439 „	3·448 „

There is, practically speaking, no difference between the corresponding determinations.

I further made comparative analyses of two samples of milk, when the following results were obtained:—

<i>1. Sample.</i>	<i>Usual Method.</i>		<i>Paper Method.</i>	
Fat .....	3·501 per cent.		3·939 per cent.	4·056 per cent.
Solids-not-fat..			8·279 „	7·911 „
Total-solids....	12·480		12·218 „	11·967 „
<i>2. Sample.</i>				
Fat .....	3·741 per cent.	3·712 per cent.	4·084 per cent.	4·089 per cent.
Solids-not-fat .			8·855 „	8·843 „
Total-solids....	12·892 „	12·923 „	12·939 „	12·932 „

In the analysis of the latter sample paper coils were used, which had been previously extracted, and yielded ·0164 and ·0151 ether extract respectively. As 4·9442 and 4·8718 milk had been sucked up by the coils, the ether extract would, when returned as fat, be equal to an additional quantity of fat of ·332 and ·310, and the figures for the analyses would then stand as follows:—

Fat .. .. .	4·416 per cent.	4·399 per cent.
Solids-not-fat .. .. .	8·523 „	8·533 „
Total-Solids.. .. .	12·939 „	12·932 „

The coils used were similar to that one which gave ·0072 ether extract when treated with ether a second time.

There is a striking point in the comparative analyses of the two milk samples, viz. :—In the first sample there is a difference of ·3 per cent. in the total-solids; while, in the second sample, the total-solids agree. Limited as the number of comparative analyses, made up to the present, naturally is, there nevertheless exists a parallel case among those communicated to the Society by Dr. Adams. This will be best shown by the following Table, to which I now wish to draw your attention:—

You will observe, that the figures for the percentage of total-solids found by the paper method vary from those obtained by the usual method from + ·07 to — ·85; whilst the differences, when following the usual method, and calculating the solids, using Fleischmann and Morgen's formula, vary from + ·14 to — ·23. They keep within the limits of ·14 per cent. in seven cases out of the eight given, and this eighth case does not refer to a normal milk, but to a mixture of milk and cream.

1	2	3	4	5	6	7	8	9	10
No.	USUAL METHOD.			Tot. Sol. calcul. F. & M.	Differences. Columns 4 & 5.	PAPER METHOD.			Differences. Cols. 4 & 10.
	Spec. Grav.	Fat.	Tot. Sol.			Fat.	Sol. n. f.	Tot. Sol.	
1	1.0360	.54	10.02	10.06	+ .04	.71	8.96	9.67	— .35
2	1.0360	.72	10.32	10.27	— .05	1.02	9.37	10.39	+ .07
3	1.0362	.39	9.80	9.89	+ .09	.58	8.73	9.31	— .49
4	1.0362	.34	9.89	9.83	— .06	.53	8.70	9.23	— .66
5	1.0364	.28	9.73	9.87	+ .14	.52	8.97	9.49	— .24
6	1.0255	10.01	18.71	18.48	— .23	10.12	7.74	17.86	— .85
7	1.0322	3.50	12.48	12.56	+ .08	4.00	8.10	12.10	— .38
8	1.0330	3.73	12.91	13.05	+ .14	4.09	8.85	12.94	+ .03

It is only four weeks ago that I took occasion to express to you my very favourable opinion on Fleischmann and Morgen's formula. It is based upon dozens of most carefully executed analyses, and tested against hundreds made by different chemists and at different places, always giving highly satisfactory results. How, then, is it that these results, arrived at in a purely mathematical way, in some instances do agree with the results obtained by employing the new method, and in others do not? Basing on so limited a series of experiments, I hesitate to give a decided answer to this question; but cannot help to think that, with a piece of blotting-paper, 55 square inches large, and weighing 4.5 g., an item is introduced into milk analysis which is open to grave objections. One objection of considerable moment is, the separation which takes place whilst the milk is being sucked up by the paper. If you examine a paper coil after the process has been finished, you will find quite distinct layers in the paper.

I am of opinion that a great many more comparative experiments will have to be made, and thereby a great deal more experience gained, before we shall be able to decide ultimately on the merits of the paper method for the analysis of milk. I hope the question will be taken up by the members of this Society, particularly with a view to clear up the rather mysterious point with regard to the differences in the total-solids.

DR. DUPRE said he was very sorry that Dr. Vieth, having gone so far, had not taken a little more trouble and gone a little farther. It seemed to him that Dr. Vieth had missed the essential point which had to be considered in the process. It was not a question whether these coils contained fat or not, because no careful Analyst would think of using them without first extracting them with ether. The main point to consider was whether such a paper could be repeatedly dried again to the same weight. He had not been able to make any experiment himself, having been very busy otherwise, but suggested that perhaps Dr. Vieth might be able to do so. Say, take one or two coils dry, and weigh them, then moisten them, and dry and weigh again. Can the paper be re-dried again to the same weight, or to anything like the same weight? He himself had no doubt that it could not be dried again to exactly the same weight, but what would be the variation? It seemed to him that Mr. Adams' method would stand or fall essentially on that point, and therefore he should have been very glad had Dr. Vieth been able to clear it up.

Had Dr. Vieth made an experiment of mixing all the different milk constituents together, and making an artificial milk, subjecting that to analysis? If he had followed the figures correctly, Dr. Vieth had invariably found more solids when using the milk constituents separately, while in the case of actual milk he always found less, and it was just possible that the three substances mixed, might have some influence on the result. That seemed to him another essential point in the consideration of Mr. Adams' process. He must confess that, individually, he was remarkably well pleased with the methods—he was astonished how well it worked, how easy the manipulation was, and how many analyses could be carried out at a time. He had often urged upon the younger members of the Society, who had time at their disposal, to do work, and bring it before the Society; let them take this opportunity, and bring light upon the subject.

MR. ADAMS said that he had to leave town by an early train, and merely wished to say that at the next or the following meeting of the Society he should like to bring forward another paper with reference to the application of the paper method to the treatment of sour milk, and he hoped then to be in a better position to bring further experiments before them.

MR. HEHNER said that he kept a coil for a number of days in the water-oven, and it was then practically the same weight as when he weighed it first; it had not appreciably diminished. He had himself a suspicion that the paper would lose, but from that experiment he had not found it so. He was convinced that the extract came from the paper, and not from the cork; he got a residue exactly like Dr. Vieth's, with a vanilla odour, and readily forming a soap when heated with alcoholic potash.

MR. KINGZETT said that every chemist working with such a crude paper would, in the ordinary course of his business, naturally extract them with ether, because it was well-known that resinous soaps were used in the preparation of those papers, which were soluble in ether. His opinion was that the validity, so to speak, of the process must stand or fall, not by any one detail, but by the uniformity of results obtained by a number of men working on the same milk in the same way.

DR. VIETH, in reply, said that he made the extractions because Mr. Adams, when he read his paper, said nothing whatever about it, and in the instructions which he issued to the members of the Milk Committee, after he (Dr. Vieth) had drawn attention to this source of error, merely mentioned that it might be a precaution to extract the paper coils, but did not make a point of it, and he wished to insist that it was absolutely essential to extract the paper coils, or exact results would not be obtained.

DR. HILL, in concluding the discussion, said that he had tried an experiment with the papers, but his first lot were spoilt, for, although he took the precaution of digesting the new indiarubber bung, he did not extract all the sulphur, consequently the paper, instead of being colourless, was a bright yellow. The next time the paper came the right colour, and the result was very satisfactory, but of course it would be unfair to jump to any conclusion on the result of one or two experiments. They must bear in mind that the process was perfectly new to all of them, and a process, however good, always failed more or less at first; and, under these circumstances, the results already

obtained were encouraging, and ought to induce them to go further into the merits of the process. Whatever objections, real or theoretical, they might have to the process, they must not lose sight of the fact that, by it, the maximum fat ever obtained was got out, and therefore, if this was fat got out of the milk, they must be nearer the truth, and that seemed to him to be a great point which the process had in its favour.

The following letter from Mr. A. C. Abraham, to the President of the Society was then read :—

87, Bold Street, Liverpool,  
April 10th, 1885.

DEAR SIR,—I have taken the liberty of sending to you, by this post, a small piece of apparatus, such as I described in the latter part of a paper printed in the ANALYST of February, 1884, p. 20, and recommended for use in the analysis of milk.

I have done so because the essential points of my process have been re-produced by Mr. Adams, in his recent paper, and as I have thought that some points of difference are to the advantage of my process, it occurred to me that perhaps some members of your Society might like to see the apparatus employed by me.

I should, therefore, be much obliged if you would show the apparatus, and, if you think fit, explain the method of using it, which I give below.

I have also sent some of "Parker's paper fibre lint," which was introduced some years since for surgical purposes, but has found but little favour in that direction.

"A piece of Parker's paper fibre lint, 4 inches by 2, is made into a roll, a piece of thin wire is passed through the centre, wound once or twice round the roll, and fixed into the stopper of a suitable weighing bottle, in such a manner that the roll may be sufficiently far from the sides to enable it to be lifted in or out without any fear of touching the sides. The roll is then taken out of the bottle and dried in a water-oven, with the bottle, until its weight is constant; 5 c.c. of milk are then dropped upon it from a pipette, when the stopper, with the roll attached, is re-inserted in the bottle, and the whole weighed. The stopper is then removed, and with its attachment placed in a drying-oven with the bottle, and kept there until it ceases to lose weight. The excess of weight over the original weight gives the total-solids. The stopper and roll are now removed, and placed in another similar bottle—preferably ground to fit the same stopper as the first—sufficient ether added so that the roll may be covered (about 50 c.c. is a convenient quantity) and allowed to macerate some hours; it is then transferred to another similar bottle, and again to a third, after which the fat will be found to have been entirely extracted. It is now removed, and again weighed as before; the loss is fat."

I am, dear Sir,  
Faithfully yours,

A. Hill, Esq., M.D., &c.,  
President of the Society of Public Analysts.

A. C. ABRAHAM.

P.S.—The roll enclosed is rather too tight. The enclosed reprints are from *Pharmaceutical Journal*. The ANALYST contains one or two corrections.

The discussion having been at this point adjourned, was resumed at the meeting on 29th April, when Mr. Adams replied as follows :—

In the first place, Mr. President, allow me to say a word regarding Mr. Abraham's claim to the original notion which lies at the bottom of my process. It is interesting to know that some one else has been working independently, and entirely unknown to me, in the same direction, though so far as it appears in a very different manner, and only in an experimental and tentative way.

Turning to the important work of Dr. Vieth, I think the Society is indebted to that gentleman, as indeed I feel myself to be, for the elaborate pains he has bestowed upon what we may regard as valuable experiments in themselves, independent of their bearing upon my process; and personally I fully appreciate Dr. Dupré's cordial, though cautious encouragement.

Before replying to the points raised by these two gentlemen, allow me to re-state the claims which I make for the new process; omitting minor advantages, as set forth in the original paper, and cut as short as possible, they are two, viz., that by means of the papers coils, we can so spread out the deposited milk-solids as to be enabled

1. To extract the whole of the fat, and
2. To dissipate the whole of the water.

These are the fundamental claims, and so far as I have heard, they not only remain unassailed, but, in point of fact, they are now firmly established by the independent testimony of Dr. Vieth, for working in his own way, with his own precautions he obtained .36 more fat by the paper, than by his usual process with plaster, and he admits that the total solids got by the paper are much below those got by the usual drying in platinum, in other words are more completely dried. So far then, Dr. Vieth agrees, and there are none to contravene these premises, but then he goes on to say the paper itself contains a fatty substance and he gives us a series of experiments with 15 or 20 coils, in which he shows an ether extract, amounting in each case to from .012 to .016, or a mean of .014 of what he considers to be a fatty substance natural to the paper; this does not quite accord with my own experiments, however, for the moment let that pass.

He finds also the same thing, and to precisely the same degree, with the filter paper which he employs in the formation of the cartridge to contain the plaster of Paris as used for the fat extraction by his usual method, but, says he, I use so much less weight of paper with the plaster, only 1.5 against your 4.5 in the paper process, therefore my error of .005 is magnified twenty times by yours; surely this is an error in arithmetic, a miscalculation altogether. Even by his own showing, the two papers, weight for weight, yield identically the same amount of extract, and where he uses 1.5 grams. of paper, with 10 grams. of milk, I use 4.5 grams. with 5 grams. of milk, or say 9 grams. with 10 grams. of milk, the difference therefore is but sixfold and this, supposing it is true, which remember, I am by no means admitting, is capable of being reduced to the vanishing point, for has he not shown us that, the extract of these paper coils is very uniform, the extreme variation from the mean being only .002 which, of course, represents only  $\frac{4}{1000}$  % on 5 grams. of milk, so that as with a filter ash an allowance could easily be made, which would bring the error, by reason of this extract, a long way within the range of ordinary experimental error, and, indeed, not the half of the amount of the error, .0036 to .0059, which he has habitually neglected, and which he now charges upon the filter papers used by him in the construction of his cartridges for the plaster process; so that, even on his own grounds, the advantage remains with the paper process.

But I am unwilling to concede even this much to him. My own experiments have shown that the paper coils, fairly treated, yield nothing like so much extract as Dr. Vieth states. I say, fairly treated, because, of course, paper is paper, which it is not at all fair and quite unnecessary to treat as though it were a substantial material like platinum or porcelain. A filter-paper may be tortured and bullied into a pulp; but we don't treat it so, and our coil deserves equal consideration. I find, if the paper is



handled roughly, the fibres are displaced and washed away, and might be counted as "extract;" but if the plain papers are extracted carefully, for three or four hours, in the Soxhlet, just as though they were charged with milk-solids, they yield an extract of a practically uniform weight of about .006, but my own impression and belief is that when charged with milk-solids, the extract due to the paper itself does not amount to more than a fraction of this. I believe the milk-solids, acting as a varnish, hold back loose fibres, which, if the paper be merely wetted with a fluid incapable of leaving a residue on drying, are liable to be shed. Perhaps, in this way, we may account for Dr. Vieth's second yield of "ether extract," and, possibly, he might have got a third, or a fourth, if he had patience to go on, and which he supposes to indicate an extreme tenacity of fat-holding property in the paper; but which I believe to be, in part, at all events, disintegrated fibres, got by harsh and unfair treatment.

In passing, allow me to point out how completely Dr. Vieth's stricture under this head is falsified by the actual practical facts of our joint competitive experiments. According to his contention, my results should show an excess of fat equal to something like three or four-tenths per cent. But do we find this was so? Certainly not. Mr. Baynes, who extracts with powdered glass, was, on an average, only .018 behind me, and, in one instance, got .06 more than I, and, on another occasion, .02 more. So that, unless it can be shown that his glass yielded fat, it can't be shown that my paper did. But, in any case, as Dr. Duprè has remarked, there can be no sound objection to the process because that the paper can be made to yield an extract, seeing that this can be provided against, either by the ordinary precaution of previous extraction, or, as I would suggest, by making a reasonable allowance, based, of course, upon experiment, with each batch of paper.

I am not surprised at Dr. Duprè's anxiety on account of the supposed difficulty of drying the paper coils to a constant weight. As already mentioned I was originally beset by the same anxiety, and it was only after repeated experiment that I satisfied myself there is no ground for this objection. I allow that, in an ordinary bath, it may require extra care in the drying, but that is more the fault of the bath than the paper. The ordinary drying oven is a most unscientific affair, there is little or no current through it, and such as there is, exposes the object to unequal draught, so that the thing is liable to be scorched on one side, while its temperature is unduly depressed on the other. Of course, with the unequal distribution of temperature, there is also unequal drying. However, I hope we shall soon have a bath free from these objections.

Notwithstanding the difficulty of the bath, this question as to the getting a constant weight has been *practically* settled. We can do it, and Dr. Vieth's experiments, abundantly confirm the fact that it can be done, as witness his duplicate experiments with the extracted papers where the fat agrees within .005 per cent., and the solids-not-fat within .012 per cent., which, working upon 5 grams. of milk, must mean an agreement in the weights within  $\frac{1}{4}$  to  $\frac{6}{10}$  of a milligram. at the outside.

In relation to this point there is another consideration in our favour, which is worth notice, viz.. that the hygroscopic quality of the paper is greatly modified by the absorp-

tion of the milk-solids ; naked raw paper, like cellulose in all forms undoubtedly is very attractive to and very retentive of moisture, but when charged with the milk-solids is much less so, anyhow I do not at all despair of converting Dr. Duprè to the belief that drying of the paper coils in a proper bath can easily be made quite reliable.

There remains very little else calling for reply. Dr. Vieth said something about the introduction of 4 or 5 grams. of weight as an element of difficulty. I am astonished at his saying that when we are discarding platinum capsules or other such vessels at least two or three times the weight, and then his own, we may say, exquisitely concordant results, already quoted, with the paper method, show that there is absolutely nothing in such a criticism ; the mere putting forwards such an objection betrays, as I suspect, a conscious weakness of his own case.

I cannot see that it is worth while entering into a comparison between the total-solids got experimentally, and those calculated by purely mathematical formulæ, since the working factors of those formulæ are the product of reasoning based upon the very same experimental data now in question.

Mathematical methods may, and doubtless will, be devised, capable of accurate application, but until the fundamental data, upon which they are based, are put beyond dispute, they cannot be used as weapons in the argument.

In bringing this discussion to a close, Mr. President, allow me to thank you, and the members of the Milk Committee, for the pains and interest you have taken, and, as I hope, will continue to take in the suggestions I have had the honour of laying before you. The tenour of those suggestions is in full accord with the growing knowledge which accumulated experience has forced upon us. I need not remind you that the Somerset House Staff have all along pursued a course which is capable of exhausting the solid residue more completely of its fat than the ordinary Wanklyn process, and among our best and most active workers for some time past, the tendency has been to seek improved methods whereby the yield of fat has been increased, and the solids-not-fat reduced, and the necessity for a modification of practice and limits has found abundant recognition.

As far back as March, 1882, Mr. Hehner, in an admirable paper, calls attention to the serious necessity "of altering both the official method of analysis and the official limits." Dr. Vieth also in February, 1883, and subsequently, has again and again expressed a similar opinion, so that it astonishes me to find him exhibit what I may call a nervous alarm, lest the older methods being found wanting, the accumulated results derived therefrom shall be rendered obsolete. No doubt it is natural for Dr. Vieth, with his large special experience, to be very jealous of any disturbance of the traditional and established modes of procedure, and extra cautious in accepting new ones, but he will, I am sure, agree with me, that it would be as reasonable to discountenance geographical exploration, lest our maps become disorganized, as to reject this new method of milk analysis simply because "it opens up a new question which everybody used to look upon as settled."

CONCLUSION OF THE SOCIETY'S PROCEEDINGS.

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

NEW APPARATUS FOR USE IN THE DETERMINATION OF STARCH.—*Berichte der Deutsch. Chem. Gesell.* 5, p. 621.—R. Rempell describes some new apparatus for use in the detection of starch, which have stood the test of six months' work with him.

Starch in corn, potatoes, etc., is usually determined by heating the finely powdered substance with a 0.25–0.5 per cent. tartaric acid solution in the well-known Linter's pressure flasks for 3–4 hours on a paraffin or oil-bath, inverting the filtered solution with hydrochloric acid, and determining the grape-sugar formed with Fehling's solution.

The new pieces of apparatus are explained with the help of drawings, which naturally cannot be reproduced here. The author has improved the Lintner's flask by inventing a new hermetical stopper. The neck is surrounded by a metal ring, which is connected on both sides with an iron stirrup or hoop; through the horizontal part of the latter works a vertical screw, which presses a metal and glass plate, to the latter of which is attached an india-rubber washer, on to the ground glass edge of the bottle, and closes it hermetically.

Instead of the inconvenient paraffin or oil-bath, the author has constructed an air-bath of sheet copper. The bath can be used with four flasks of the described construction. The four openings are closed with covers consisting of two parts, and provided with a hole in the middle for the neck of the flasks. The bottom of the air-bath is covered with an asbestos-plate to prevent the flasks becoming unequally heated. A little distance above this is a false bottom, with an opening in the middle, and this is followed by a perforated bottom. The circulation in a so constructed bath is regular, and all parts become equally heated; the path taken by the heated air is shown by means of arrows in a sectional drawing.

The author's apparatus permits of clean and accurate work. The flask and the air-bath may be obtained from the firm, Rohrbeck's Nachfolger, in Vienna.

F. H. H.

---

THE DETECTION OF STARCH IN GRAIN AND POTATOES. By M. MARKER.\*—Three grms. of the finely powdered substance are heated with 50 c.c. of water to 90°C, allowed to cool to 65°C, and finally heated with 5 c.c. of a cold infusion of malt, obtained by heating 50 grms. malt with 1 litre water.

After half an hour, tartaric acid (10 per cent. sol.) is added, the mixture heated for half an hour at a pressure of 3–4 atmospheres, and allowed to cool down to 65°C. Another 5 c.c. of the malt infusion are now added, and, after half an hour, the liquid inverted, after the addition of 15 c.c. hydrochloric acid and 150 c.c. water, by heating at 100° for 2½ hours on the water-bath. The sugar produced is determined, after neutralisation with soda, according to Allihn's method.

F. H. H.

---

\* *Repert. der Anal. Chem.*, No. 7 [1885] p. 116.

ON THE DETERMINATION OF CASEIN IN COW'S MILK BY PRECIPITATION WITH SULPHURIC ACID.\*—By FRENZEL AND WEYL.—20 c.c. of the well shaken milk are mixed with 60 c.c. distilled water in a beaker of 150 c.c. capacity. The mixture being well stirred, 30 c.c. of sulphuric acid (1%<sub>v/v</sub>) are run in. The beaker is then covered, and allowed to stand in a cool place. The supernatant liquid is decanted through a weighed filter. Finally, the precipitate is brought on to the filter, and washed twice with water. The washing is continued with alcohol, at first 90 per cent., and then absolute, and finally with ether, 10 times if a skimmed, 15 times if an ordinary milk. The filter is dried at 110°C and weighed.

The analyses lead to the following conclusions on the part of the author:—

1. It is sufficient to dilute with four times the volume, without essentially influencing the exactitude of the casein determination.
2. The use of carbonic acid is superfluous.
3. The method is quicker than the one in use (Hoppe-Seyler's); first, because it is not necessary to pass carbonic acid; secondly, because the quantity of liquid to filter is considerably smaller.

F. H. H.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

ON THE DETECTION OF PETROLEUM ETHER IN ETHER.—Pharm. Centralhalle xxvi. 15, p. 169.—D. Vitali (L'Orosi, 1885-8) communicates that an adulteration of ether with petroleum ether has been observed by Stroppa. This cannot be detected by the smell, and not always by mixing with alcohol. The petroleum ether can, however, be isolated by mixing in the cold with sulphuric acid. Sulphuric ether is thus absorbed and the petroleum ether remains unattacked. The operation is best conducted in a graduated test-tube by adding sulphuric acid, while stirring and cooling, till the top layer no longer diminishes. By comparing this layer with the volume of the original ether, the approximate quantity of petroleum ether present can be determined.

For identification it is sufficient to bring the ethereal solution, by means of a pipette, into another test-tube, and to shake with iodine; ether is coloured yellow, petroleum ether, pink.

F. H. H.

THE QUANTITATIVE DETERMINATION OF ALOES IN MIXTURES.—By H. HAGER, Pharm. Centralhalle, N.F. [1885] 6. 130.—Aloes are completely soluble in 80 per cent. alcohol, and only partially in 90 per cent. and absolute alcohol. On the other hand they are completely insoluble in a mixture of two vols. chloroform, three vols. benzol, and one vol. absolute alcohol.

To determine aloes in a mixture, the author proceeds thus:—The liquid is evaporated to dryness; the residue, on cooling, pulverized and macerated at 18-25° C. with the alcohol-chloroform—benzol mixture. This mixture dissolves the resins—guaiacum, jalapin, scammonium, myrrha, senna, etc. After extracting sufficiently, the mass is dried, pulverized, and treated at 50° C. with 80 per cent. alcohol. The

\* *Repert der Anal. Chem.*, No. 7 [1885], p. 117.

alcoholic extract, containing the aloes in solution, is evaporated to dryness in a weighed dish, and extracted with a two per cent. ammonia solution (to every grm. residue 12-15 c.c. ammonia solution). To this solution is added lead acetate in excess, and a few drops of ammonia until alkaline. This precipitates the aloes completely as a lead compound. The precipitate is collected on the filter and washed with distilled water. The aloes are finally dissolved by mixing with ammonium sulphate, and heating with 80 per cent. alcohol, evaporated to dryness and weighed.

F. H. H.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ON THE DETERMINATION OF TITANIC ACID IN IRON AND IRON ORES.—BY A. LEDEBUR. —*Chemiker Zeitung*. [1885]. IX., 27, p. 483.—The methods in use for the separation of titanic acid from iron, are neither easy, nor very accurate. The complete fusion with potassium bisulphate, is a difficult matter, and the subsequent solution in cold water requires a considerable amount of patience and time. Further, the titanic acid, precipitated by boiling, is never completely pure; it nearly always contains small quantities of iron, and always phosphoric acid.

To obviate these difficulties, the author proposes the following method:—2-5 grms. of the ore are treated with concentrated hydrochloric acid, evaporated on the water-bath to dryness, the residue warmed with a little hydrochloric acid, diluted with water, filtered and washed with cold water. The residue on the filter is dried, fused with potassium and sodium carbonates, treated with water, excess of hydrochloric acid added, and the whole evaporated to dryness on the water-bath. The residue is gently warmed with concentrated hydrochloric acid, diluted with cold water, filtered and washed with cold water. The two filtrates are brought together, and contain generally all the titanic acid. It is, however, advisable to test the silica by boiling with sodium carbonate, in which it should completely dissolve.

To the titanic acid solution, concentrated sulphuric acid is now added in sufficient quantity to convert the chlorides into sulphates. The hydrochloric acid is driven off by evaporation. On cooling, the residue is diluted with a large quantity of water, sulphurous acid added, and the mixture warmed gently, in order to reduce the iron present. The liquid is then partially neutralised with sodium carbonate (care being taken that not sufficient carbonate is added to produce a permanent precipitate), and heated for a couple of hours in a flask, the mouth of which is covered with a watch glass. From time to time, fresh water is added, to replace that evaporated, and to prevent the oxidation of the iron, sodium bisulphate is added, as soon as the liquid no longer smells of sulphurous acid. The whole of the titanic acid is thus precipitated, together with a small quantity of phosphoric acid and iron. The precipitate is allowed to settle, the filtrate tested by prolonged boiling, and the precipitate washed with water. It is then dried, fused with sodium and potassium carbonate and the fusion treated with water. The whole of the phosphoric acid goes into solution, while sodium titanate and oxide of iron remain undissolved. The

residue, after filtration, is then warmed with strong sulphuric acid, until completely dissolved, the solution diluted with a large quantity of water, again reduced with sulphurous acid, neutralised with sodium carbonate and the titanous acid precipitated by a two hour's boiling. The precipitate, which should be quite white, is then filtered, washed and weighed.

To determine the titanous acid in pig-iron, 15 grms. are dissolved in nitric acid, evaporated, and ignited, to destroy the carbon compound, and to expel the nitric acid. The residue is dissolved in concentrated hydrochloric acid, diluted with water and filtered. The residue on the filter, after washing and drying, is fused with sodium carbonate and potassium nitrate, the silica is then removed by evaporating the fusion with hydrochloric acid, and the filtrate proceeded with as above.

F. H. H.

A NEW REACTION FOR DISTINGUISHING BETWEEN THE CHLORIDES BROMIDES AND IODIDES OF SODIUM AND POTASSIUM, ALSO FOR DETERMINING BROMINE AND IODINE IN SODIUM CHLORIDE AND IODINE IN SODIUM OR POTASSIUM BROMIDE. BY H. HAGER.—Pharm. Centralhalle, XXVI., p. 141.—This reaction depends upon the different behaviour of potassium permanganate with the haloid salts of the alkalis.

If a piece of the haloid salt, the size of a pea, be dissolved in 3-4 c.c. dilute sulphuric acid and 1 c.c. of a solution of potassium permanganate be added, with the chlorides no reaction takes place, whereas the bromides give a light yellow, and the iodides an intense yellow or gold solution.

In the case of pure sodium chloride the permanganate colour disappears entirely after an hour, and the solution remains perfectly colourless. On the other hand a direct yellow colouration is obtained on the addition of permanganate solution, even if only traces of bromine or iodine are present in the sodium chloride.

F. H. H.

#### REVIEWS.

FRESENIUS QUANTITATIVE ANALYSIS, VOL. II. Translated by *Charles E. Groves, F.R.S.*

Part I. London: J. and A. Churchill.

LONG expected, and waited for by all chemists, we now have the pleasure of greeting the new edition of this standard book. It is a matter of congratulation that Messrs. Churchill have not delayed till the entire work had left the author's hands, but have commenced its issue in parts. The present one is practically limited to organic ultimate analysis, and, as a work of reference and minute detail, it is, of course, simply beyond criticism. The translator has done his work well, and so faithfully that he has not even attempted to modernise, or rather equalise, the nomenclature in any way, and we, therefore, meet with "potash," "hydrate of potassa," and "chloride of potassium" all together. This is, however, a matter of no real moment compared with the practical contents of the book; but we submit that, for the sake of symmetry, it would be advisable to employ, say, the regular fixed nomenclature in use in the *Chemical Society's Journal*, when producing the next English edition of the work. Doubtless the sale will be so large that another issue will be rapidly called for, and then we hope that our modest suggestion will meet with attention.

ACETIC ACID AND VINEGAR, AMMONIA AND ALUM (being No. 3 of Churchill's Technological Handbooks). Edited by *J. Gardner, F.I.C.* London: J. and A. Churchill.

THIS is the third number of the excellent series of little handbooks on technology, issued by Messrs. Churchill. It will be found quite as useful as its predecessors, which have already received favourable notice in our columns. Thoroughly practical and complete, and yet without a line of unnecessary verbiage, these books should be greatly appreciated by the class of readers for whom they are intended, and their issue should prove a remunerative, as well as a happy thought to the proprietors.

#### LAW REPORTS—IMPORTANT DECISION.

In the Queen's Bench Division, on March 31st, before Mr. Justice Mathew and Mr. Justice A. L. Smith, the case of *Knight v. Bowers* raised a curious and important question under the Sale of Drugs Act, 1875 (35 and 39 Vic., c. 63, section 6), the question being whether the Act applies to a case in which the tradesman sells something quite different from what is asked for. It had arisen in this way:—A woman went into the shop of a herbalist and asked for some saffron; he gave her savin. Both saffron and savin are in the British Pharmacopœia, but they are very different things, and savin is in the list of poisons. The woman gave the savin to the public analyst, who found it to be savin, and thereupon she summoned the herbalist, under section 6 of the statute, which says that any person who sells an article to the prejudice of the purchaser, which is not of the nature, substance, or quality of the article demanded by the purchaser, shall be guilty of the offence. The magistrate, however, declined to convict, thinking that the Act is confined to cases of mixing and adulteration, but he stated a case to raise the question.—Mr. Bosanquet, Q.C., appeared on behalf of the inspector, who appealed against the decision, and contended that the case clearly came within the 6th section, and that the Act was not confined to cases of adulteration. That section was introduced to meet the very objection that the Act was only confined to cases of adulteration. That section applies wherever one article is given for another. [Mr. Justice Mathew.—If a person asks for coffee and gets chicory, the statute is not to apply.] Just so, although if coffee is sold mixed with chicory, the statute affords a remedy. So, if a person purchases butter and gets butter mixed with lard, he has a remedy, and if he gets only lard, then he has no remedy. [Mr. Justice A. L. Smith.—Does not the maxim *caveat emptor* apply in such cases? Where there is an adulteration the purchaser may not be able to discover it; but surely he must know butter from lard, or coffee from chicory.] Educated persons may be able to distinguish coffee from chicory, or savin from saffron; but ignorant persons might not be able to do so, and the Act is for the protection of the poor and the ignorant. [Mr. Justice A. L. Smith.—Suppose it occurs by mistake.] Then that would not really be an offence within the Act, for the seller would not really and knowingly deliver a different article, and a magistrate, of course, would not convict, for no one can be criminally liable for a mere mistake. If this section does not apply to such cases as the present, what is the use of it, and why was it introduced? [Mr. Justice Mathew.—Suppose a housekeeper purchases tea, and gets "exhausted" tea, or tea leaves which have already been boiled (a laugh), would not that be within the Act?] Surely it would be so, and under section 6. [Mr. Justice A. L. Smith.—There the purchaser does not get an article of the substance, nature or quality of that demanded.] Nor here; the cases are surely strictly parallel; indeed, more so than the case put, for the tea once was tea, at all events, but savin never was saffron. The contrary construction not only makes nothing of the enactment, but makes it worse than nothing, and quite mischievous; for the result will be that if the tradesman only mixes the article, he commits an offence, but if he substitutes something quite different, he escapes.—No counsel appeared on the other side, and the Court came to the conclusion that the magistrate was wrong, and ought not to have dismissed the case on the ground assigned.—Mr. Justice Mathew said the case clearly came within the 6th section. The thing asked for here was saffron; the thing given was savin, which was something quite different. If this were not an offence, many offenders would escape; as, for instance, those who sell lard for butter, or chicory for coffee, which clearly were offences within the statute.—Mr. Justice A. L. Smith agreed, as he could not, he said, see his way to any other construction of the enactment, though he was not sure that it was intended by the Legislature in all cases in which one drug or other article was sold for another. The previous sections, however, dealt with mixing, and the 6th section distinctly said that no one should sell an article not of the nature of that demanded, and certainly savin was not saffron; it was clearly within the terms of the enactment, and, although he doubted whether it was intended by the Legislature, yet they had certainly enacted it.—The case, therefore, was sent back to the magistrate.

—*Times*.

# THE ANALYST.

JUNE, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
(a) REPORT OF MEETING .. .. .	99	(a) DETERMINATION OF TANNIN, F. BECKER	112
(b) ON THE TREATMENT OF SOUR MILK INTENDED FOR ANALYSIS, By M. A. ADAMS..	99	(b) DETERMINATION OF TANNIN, E. JOHANSON	113
(c) ON REICHERT'S METHOD OF EXAMINING BUTTER-FAT, By A. H. ALLEN ..	103	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS:—		(a) ESTIMATION OF ZINC IN TUTTY POWDER, M. LIEBSCHUTZ .. .. .	113
REPORT OF APRIL MEETING, By DR. WALLER .. .. .	105	(b) DETERMINATION OF GRAPHITE IN MINERALS .. .. .	113
ORIGINAL ARTICLES:—		(c) SILVER IODIDE AS A BLOW-PIPE REAGENT, P. CASAMAJOR .. .. .	114
(a) A SIMPLE AND EFFECTIVE FAT EXTRACTION APPARATUS, By F. WALLIS STODDART	108	(d) SEPARATION OF TITANIUM FROM NIOBIUM AND ZIRCONIUM, E. DEMARCAV ..	114
(b) ON A NEW VARIETY OF PYRITES FOR SULPHURIC ACID MANUFACTURE, By DR. DRINKWATER .. .. .	109	(e) METHOD FOR THE DETECTION OF ARSENIC, H. HAGER .. .. .	114
MONTHLY RECORD OF RESEARCHES IN FOOD ANALYSIS:—		REVIEWS:—	
(a) EXAMINATION OF BUTTER AND OLEOMARGARINE, M. LIEBSCHUTZ.. ..	111	AIDS TO THE ANALYSIS OF FOOD AND DRUGS, By H. AUBREY HUSBAND, M. B.	115
(b) DIRECT DETERMINATION OF INVERT SUGAR, H. BODENBENDER .. ..	112	SPECIAL NOTES .. .. .	115
		CORRESPONDENCE .. .. .	116

### PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An ordinary meeting was held at Burlington House, on Wednesday, the 13th inst.

In the absence of the President through indisposition, the chair was taken by Dr. Muter, Vice-President.

The minutes of the previous meeting were read and confirmed.

Mr. A. C. Wilson, Analytical Chemist, Stockton-on-Tees, was proposed as a member.

The following papers were read and discussed:—

“Observations on the Permanganate Test in Water Analysis,” by Dr. Dupré, F.R.S.

“On Reichert’s Method of Examining Butter-fat,” by A. H. Allen, F.C.S.

The two papers by Dr. Alfred Hill and Dr. Bostock Hill were unavoidably postponed. Dr. Dupré’s paper will appear in our next issue.

The next meeting of the Society of Public Analysts will be held at Burlington House, on Wednesday, the 10th June.

### ON THE TREATMENT OF SOUR MILK INTENDED FOR ANALYSIS.

By MATTHEW A. ADAMS, F.R.C.S., F.C.S.

*Being a contribution to the transactions of the Society of Public Analysts at their meeting April 29th, 1885.*

I IMAGINE all will agree that the curdled condition of sour milk, presents a mechanical difficulty by no means trifling, to the food analyst, whatever be the method of analysis employed, which adds not a little to the uncertainty of the results, because of the



impossibility to obtain an uniform distribution of the milk constituents throughout the sample, and this condition obviously becomes an obstacle amounting to an absolute barrier to the paper method of analysis, which I have lately brought to your notice. It was, in these circumstances, essential to discover some innocent expedient, whereby curdled milk might be restored to its natural limped emulsified condition. In as much as coagulation has often set in before the sample intended for analysis reaches the hands of the analyst, it is out of the question attempting anything of the nature of an antiseptic treatment to anticipate or arrest the changes which fermentation brings about; there is no help for us, unless we can restore the already curdled milk, to a fluid condition, and this is what we set ourselves to do.

Of course it is well known that precipitated casein is soluble in potash or soda, but it does not appear to have been remarked that ammonia added to sour milk, not only re-emulsifies the coagulum, but also maintains it in the re-emulsified state for a considerable time, apparently arresting fermentive change, and so preserving the milk in a physical condition suitable for our purposes of analysis, and so far as I can find, in no way interfering with the analysis. In proof of which I beg to offer for your consideration the following experiments.

I first set myself to ascertain what, if any effect, ammonia had in the ordinary course of analysis of a sample of fresh milk; for this purpose I divided a specimen of fresh milk into two portions, to one of which I added ammonium, and examined the two by the paper, and also the usual methods, with the following results per cent.

Solids-not-fat	8.31	8.38	Average.	
Fat	4.50	4.44	8.345	} Without Ammonia.
Total solids	12.81	12.82	4.47	
			12.815	
Solids-not-fat	8.16	8.26	Average.	
Fat	4.41	4.44	8.210	} With 9 per cent. of Ammonia.
Total solids	12.57	12.70	4.425	
			12.635	

From which it appears that there is a certain small loss on the total solids, caused by the ammonia treatment, which loss is chiefly borne by the solids-not-fat, the difference being

Solids-not-fat	0.135
Fat	0.045
Total solids	0.18

with the same sample, on drying 5c.c. in platinum capsules, the difference on the total solids was 0.31 per cent.

In another sample of fresh milk, treated with 7.1 per cent. of ammonia we got

Solids-not-fat	8.76	8.62	Average.	
Fat	2.76	2.77	8.69	} Without Ammonia
Total solids	11.52	11.39	2.765	
			11.455	
Solids-not-fat	8.47	8.46	Average.	
Fat	lost		8.465	} With 7.1 per cent. of Ammonia

The loss on the solids-not-fat being 0.225 per cent., and the same dried in platinum gave a loss on the total solids = 0.33 per cent.

It was noticed that the dried residues of the ammoniated samples were much darker than the others; they had become brown like partially caramelized sugar, and the thought struck me at once that perhaps the ammonia has the effect of causing the milk-sugar to give up more readily its water of crystallization, and with this idea I next experimented with a simple solution of milk-sugar, with the following results:—

				Average.
Sugar Solution only	3.74 p.c.	3.68 p.c.	=	3.71
Ditto + 4½ p.c. of Ammonia	3.49	3.47	=	3.48
Ditto + 12 p.c. of Ammonia	3.54	3.45	=	3.49

This was with paper coils, and there is again a loss=0.23 % on the ammoniated sample. The same thing done in porcelain dishes gave:—

				Average.
Sugar Solution only	3.50	3.50	=	3.50
Ditto	3.53	3.53	=	3.53
Ditto with Ammonia	3.44	3.41	=	3.425
Ditto	3.42	3.35	=	3.385

Difference 0.11 per cent.

I cannot believe that in this later case the drying in the porcelain dishes can have been complete, it being so very difficult to get rid of the last trace of moisture from the sticky residue when in mass in the dishes, though it is accomplished easily enough on the paper coil, and I beg to be allowed to remark that incidentally this is a corroboration in part of my contention in regard to the paper method. But to return to our present subject, there was again observed the same darkening effect produced by the ammonia, although the experiments were done side by side under precisely the same conditions. Those that had the ammonia became sooner and more dark than those that had not, and, as you will have observed, the loss on the paper coils viz., 0.23 % is practically the same loss as we find in the milk.

So far then as these few experiments go, it appears that the addition of ammonia has no great effect upon the milk, and such as it has is confined to its action on the sugar, and further, I think we may safely conclude that it only causes the sugar to give up its molecule of water more readily than it otherwise would do.

We now pass to the consideration of the details of two sets of experiments on very old and decomposed milks. The first relates to a sample received from Dr. Vieth, January 7th, and examined jointly with him and Mr. Baynes (No. 5, page 52 in the March ANALYST). The other sample is one received from Dr. Vieth, December 19th, 1884 (No. 1, page 51). On the first milk by the paper process, my results were as follows:—

					Average.
Solids-not-Fat	9.05	9.01	8.91	8.89	= 8.96
Fat	0.52	0.48	0.54	0.55	= 0.52
Total Solids	9.57	9.49	9.45	9.44	= 9.48

On March 26th, when 78 days old, this milk was decomposed, having a thick felted growth of fungus on its surface, the addition of 11.3 per cent. of ammonia, with a

vigorous shake broke up the clot and brought the whole into a limpid condition, except for a few flakes of the mycelium of the fungus, and on analysis we got :

					Average.
Solids-not-fat	8.76	8.58	=		8.67
Fat	0.42	0.51	=		0.46
Total solids	9.18	9.09	=		9.13

Showing a loss on the original analysis when in the fresh state of

Solids-not-fat	0.29
Fat	0.06
Total solids	0.35

and with the second, originally when fresh, the following results were obtained :—

					Average.
Solids-not-fat	8.97	8.88	8.91	9.07	= 8.96
Fat	0.70	0.68	0.78	0.68	= 0.71
Total solids	9.67	9.56	9.69	9.75	= 9.67

On March 26th, when 98 days old, and very much decomposed, it was treated with 12.3 per cent. of ammonia with the following results :—

					Average.
Solids-not-fat	6.09	6.12	=		6.105
Fat	0.67	0.64	=		0.655
Total solids	6.76	6.76	=		6.76

Showing a difference of

Solids-not-fat	2.86
Fat	0.06
Total	2.92

To these I add particulars respecting a sample only a week old—which, when in the fresh state, gave

			Average.
Solids-not-fat	8.89	8.87	8.88
Fat	3.64	3.72	3.68
Total Solids	12.53	12.59	12.56

And with 8.1 per cent. of ammonia when a week old—

Solids-not-fat	8.77	—	0.11
Fat	3.77	Difference	+ 0.09
Total solids	12.54		— 0.02

In the first of the two very old samples you observe the loss is only 0.35 per cent., whereas in the second the loss is 2.91 per cent.

I can't pretend to explain this irregular difference of loss, the sample 98 days old, certainly in outward appearance alone, was even to the unassisted senses much more decomposed than the one 78 days old. So far as they go, the facts only confirm the opinion of those who hold that no regular time allowance can be made in calculating the effect of changes which occur during the decomposition of milk.

As regards the last experiment, the results are anomalous, and for the want of time I could not repeat the experiment—indeed, the experiments altogether are neither so numerous or elaborate as I could wish, they were conducted under the pressure of other important engagements, and would not have been brought forward at the present time, except that it appeared very desirable to show with as little delay as possible that

the mere coagulation of milk is no barrier to the adoption of the paper method of milk analysis, and the only object sought for, so far as the present communication is concerned, is to point out a simple and innocent way whereby the restoration of coagulated milk to a condition suitable for analysis can be accomplished, and I submit that the addition of ammonia answers this purpose very satisfactorily.

#### ON REICHERT'S METHOD OF EXAMINING BUTTER-FAT.

BY ALFRED H. ALLEN,

(*Read at the Meeting on 13th May, 1885*)

I HAVE recently been in the habit of using Reichert's process as a means of sorting samples of butter for further examination, and with such satisfactory results, that I think it desirable to call the attention of the Society to the advantages possessed by the process, which consists in distilling the saponified fat with a dilute acid, and estimating the volatile acids in the distillate, by titration with standard alkali.

Hehner and Angell seem to have been the first to attempt to determine the volatile acids in butter by a process of this sort, but, as is well known, they subsequently abandoned the method in favour of that with which their names are more specially associated. Heintz attempted to titrate the soluble acids without separating them from the insoluble, but was unsuccessful. Lechartier distilled off the volatile acids, and converted them into barium salts, of which he found pure butter-fat to yield at least 12 per cent., while butterine did not yield more than a twentieth of this quantity.

The first successful application of the principle involved in these processes is due to Reichert, who, by always using the same quantity of butter, and of alcoholic potash for saponification, adding the same quantity of acid for decomposing the soap, and collecting a definite quantity of distillate, found that he obtained very constant results, an average of 14 c.c. of decinormal alkali being required for the neutralization of the distillate from  $2\frac{1}{2}$  grammes of butter-fat. Medicus and Scherer have tested this method with good results, and Meissl also speaks highly of the process. He uses five grammes of butter, and a weaker alcohol, for dissolving the alkali requisite for saponification, in order to avoid the loss of volatile acids by etherization during saponification. He tested 84 samples of butter by this method, and concluded that if 110 c.c. of the distillate from 5 grammes of butter required less than 26 c.c. of decinormal alkali for neutralization, the butter should be regarded as adulterated. Ambuhl states that Reichert's process possesses every advantage over Hehner's, which is becoming replaced by the former. He finds the volatile acids from  $2\frac{1}{2}$  grammes of butter to require an average of 14.7 c.c. of decinormal alkali.

The process has also been employed extensively by Dr. G. C. Caldwell, from whose report to the New York State Board of Health I have taken the foregoing statements as to the results of previous observers. He found the volatile acids from 27 samples of butter-fat to neutralise from 12.7 to 15.5 c.c. of alkali.

Dr. L. W. McCay (*Chem. News*, l. 151) has also employed the process with very satisfactory results, and has pointed out the ambiguity attending the description of the method employed by other experimenters.

C. E. Schmitt uses phosphoric acid, instead of sulphuric acid, for decomposing the soap, and finds the amount of decinormal alkali to vary from 13.0 to 14.3 c.c. for 2½ grammes of butter. He further found that goat's milk butter required 13.58 c.c. and ewe's milk butter 13.65 c.c. of decinormal alkali.

Lastly, Mr. R. W. Moore (*Chem. News*, l. 268) has employed the process, and recommends it as giving more information respecting butter than either Hehner's or Kœttstorfer's method. He has also applied the method to a number of other oils, all of which gave trifling quantities of volatile acids, with the exception of cacao-butter and cocoa-nut oil, which gave distillates which neutralised 1.6 and 3.7 c.c. of decinormal alkali respectively.

Here, then, we have a general consensus of opinion in favour of the process, and my own experience fully bears out that of other observers. In carrying out the method, some of the operators do not seem to have been very particular respecting the volume of the acidulated liquid before distillation, but it is evident that this is a point of importance, as the same amount of volatile acid cannot be expected to be found in the first 50 c.c. distilled, if the original liquid measured 70 c.c., as would pass over if the original liquid measured 100 c.c. The following are the conditions I have found best in performing the operation:—

Weigh out 2.5 grammes of the clarified butter-fat, and saponify in a closed flask by heating it with 25 c.c. of approximately seminormal caustic potash.\* Transfer the product to a porcelain basin, and evaporate off the alcohol at a steam heat. Dissolve the residue in water, add dilute sulphuric acid in slight excess, make up the liquid to 75 c.c. with water, add some fragments] of pumice coiled round with platinum wire, and distil gently till 50 c.c. have passed over. Titrate the distillate with decinormal caustic alkali, using phenol-phthaleïn as an indicator.

The distillate from a genuine butter-fat requires *not less than* 12.5 c.c. of alkali for its neutralization. This corresponds to 3.9 per cent. of butyric acid distilled over, so that somewhat over 4 per cent. of volatile acids, in terms of butyric acid, may be considered to be yielded by the process. A further amount could probably be obtained by continuing the distillation, but not so much more as might be expected, for Duclaux has shewn that, if an aqueous solution of butyric acid be distilled to one third, from 75 to 80 per cent. of the total acid will be found in the distillate. In the case of the higher homologues of butyric acid a still larger proportion passes over during the first part of the distillation.

The following table is an epitome of the results obtained by various chemists, who have placed on record the results of their experience of Reichert's process. The figures represent the percentage of volatile acids, in terms of butyric acid, found in the distillate under the conditions described. The figures are calculated from the volume of decinormal alkali required to neutralise the distillate from 2.5 grammes of fat, by multiplying the number of c.c. taken by the factor 0.312.

---

\* I prefer to conduct the saponification in a closed flask, so as to render impossible any loss from the volatilization of butyric ether; but I believe the precaution to be superfluous, as the somewhat large amount of alcohol used has a tendency to retain in solution any ether which may be formed, and its saponification speedily ensues.

	Reichert.	Meissl.	Ambuhl.	Caldwell.	Schmitt.	Moore.	Allen.
Butter-fat: Highest	4.51	..	..	4.96	4.46	..	4.71
Lowest	4.23	..	..	3.96	4.06	..	3.87
Average	4.37	..	4.59	4.59	4.24	4.09	4.21
Limit	3.90	4.06	..	3.90	..	..	..
Ewe's-butter ..	..	..	..	..	4.26	..	..
Goat's-butter ..	..	..	..	..	4.24	..	..
Cocoa-nut oil ..	..	..	..	..	..	1.15	1.09
Palm-nut oil ..	..	..	..	..	..	..	0.75
Palm oil ..	..	..	..	..	..	0.25	..
Cacao-butter ..	..	..	..	..	..	0.50	..
Olive oil ..	..	..	..	..	..	..	..
Cotton-seed oil ..	..	..	..	..	..	0.09	..
Butterine } ..	..	..	..	0.06 to 0.50	..	0.12 to 0.18	0.33
Oleomargarine }	..	..	..	..	..	..	..

A consideration of these results shows that there is a much sharper distinction between butter and butter substitutes when examined by Reichert's process than is indicated by Hehner's, Kœttstorfer's, or Bell's method; and, as Mr. Moore has pointed out, even cocoa-nut oil presents a marked difference from butter-fat. Hence, as a means of *sorting* butters, I am strongly in favour of Reichert's process, which is applied with but little more trouble than Kœttstorfer's, and gives more enlightening results.

It is stated by more than one chemist that butyric acid is added to butter-substitutes. It would probably be used in the form of a metallic butyrate or as artificial tributyrin. It would probably be possible to detect such a sophistication by treating the sample with a limited quantity of alcohol and examining an aliquot part of the alcoholic solution by Reichert's method. This suggestion is based on the observation of Dr. James Bell that the butyric radical of natural butter is not readily dissolved out of the fat by alcohol, and hence, probably, forms part of a complex molecule, whereas added butyrin would not be similarly circumstanced.

### DISCUSSION.

Mr. Fox said the process appeared to be quite as elaborate as Mr. Hehner's, and more complicated than that introduced by Mr. Wanklyn and himself.

Mr. HEHNER said it was unintelligible to him why Mr. Allen should state that the method was the best hitherto published, and yet should use it only as a preliminary test, showing thereby a characteristic want of confidence in the method which he recommended. It also seemed remarkable that the previous workers—who, as Mr. Allen had pointed out, disregarded the initial volume of the fluid to be distilled—should have obtained results practically identical with those of Mr. Allen. Whatever comparative results Reichert's process was capable of furnishing, if always performed in the same manner, yet Analysts should not, on principle, tolerate a process by which only a fraction of the substance to be estimated was obtained. In the case of the estimation of organic matter in water, where we were not in possession of a method for estimating its totality, we might be temporarily satisfied with a process like the albuminoid ammonia one; but, in all cases where a real and accurate estimation was possible, such rough and ready methods should not be admitted by careful Analysts.

Dr. MUTER, in concluding the discussion, said that the subject of Butter Analysis was a well-worn one. He thought they could not do better than to use, as a preliminary test, the specific gravity; and then, if necessary, Mr. Hehner's process and its various modifications—including estimation of insoluble and soluble fatty acids.

### CONCLUSION OF THE PROCEEDINGS OF THE SOCIETY.

### PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS.

COMMUNICATED BY DR. E. WALLER, NEW YORK.

At the meeting of the American Society of Public Analysts, held April 13th, 1885, Dr. Bartley spoke of the conference recently held by the Brooklyn Health Board with the manufacturers of wall paper. The result was an agreement on the part of the manu-

facturers to avoid the use of arsenical pigments in their goods, and to take back any lots of goods of their manufacture now in the market, which might be proved to contain arsenic.

MR. MARTIN, Secretary, read a number of replies from various analysts in different parts of the country regarding the methods for the analysis of milk. Out of 20 analysts, 16 expressed a preference for Wanklyn's method, more or less modified, the modification proposed by Dr. Waller being usually preferred. Four preferred the use of some such form of apparatus as that of Soxhlet for extraction of the fat, the milk having been dried on asbestos or some absorbent material. Ethylic ether was generally preferred to petroleum ether for extracting the fat. The minimum standard composition for milk approved by the majority was essentially that of the British Society of Public Analysts, as regards percentage of solids-not-fat, though most believed in requiring three per cent. or more of fat.

A report on the comparison of different methods of analysis by Drs. Kent and Grothe, of the Brooklyn Health Depot, was then read. The methods tried, were:

1. Waller's modification of Wanklyn's process.
2. Soxhlet's areometric method, the gravity being taken by means of a Westphal balance.
3. Babcock's asbestos method, (absorption of the milk in asbestos, drying and extracting in a Soxhlet.)

4. Adams' paper method. The results for fat were:—

No. of Sample.	Wanklyn.	Soxhlet (Areo.)	Babcock (Asbestos).	Adams (Paper).
1		3.52	3.48	
2	3.22	3.68	3.41	
3	3.41	3.98	3.82	4.43
4	3.58	4.18	4.01	4.67
5	3.16	3.40	3.28	3.46
6	3.39	3.82	3.73	4.07
7	3.61	3.83		
8		4.06		4.40

A coarse filtering paper was used in the Adams' method.

T. S. GLADDING read a paper, giving his experience, with a comparison of the modified Wanklyn method and that of Adams.

No. 1.	Total Solids	Wanklyn.	Adams.
	Fat	12.85	12.60 12.50
		3.58	4.14 4.13
No. 2.	Total Solids	12.47	12.03 12.07 12.05
	Fat	3.06	3.66 3.66 3.61
No. 3.	Total Solids	5.33	5.44
	Fat	0.53	0.90

In the Adams' method, Schleicher and Schull's filter paper was used. The fat was extracted in a Soxhlet apparatus, the ether being run through it nine or ten times.

In drying by the Adams' method, three hours were found to be ample, and no danger of oxidation from complete drying, seemed to exist. Of four filter papers saturated with milk, and dried, on heating for 2½ to 5 hours, in two cases there was no change in weight, in another, a loss of 1 milligram., and in the fourth a gain of 0.5 milligram.

After determining the ash of the amount of paper used, an attempt was made to determine ash, by incinerating the paper from Adams' process; the results, after deducting ash of the paper, were:—

Wanklyn	0.62	0.68	0.69
Adams	0.57	0.58	0.52

MR. GLADDING suggested that possibly some chlorides had been volatilized in the Adams' method.

DR. KENT related experiments made by himself, to determine the solubility of milk-sugar in petroleum ether. The petroleum ether was distilled, those portions distilling between 25° and 65° C being selected. A quantity of this was shaken in a flask with pulverised milk-sugar, allowed to stand for 24 hours, and then 25 c.c. were evaporated allowed to stand 24 hours more and 25 c.c. more evaporated. This was repeated a third time. No residue was obtained in any case.

The question was raised as to whether lactic acid was not frequently a constituent, extracted by ether, and made to count as fat. Reference was made to the publication of some results obtained at Somerset House, England, where the percentage of fat extracted by ether, was apparently high in the case of soured milks.

DR. BARTLEY remarked on the concordant results obtained by the modified Wanklyn method, showing that at least the same amount was extracted in every case.

DR. KENT spoke of difficulties experienced in detaching the residue from the bottom and sides of the dish, in order to insure thorough action of the ether. Mr. Martin suggested that in evaporating, the most of the fat comes to the top, thus obviating the necessity for such complete dislodging of the milk residue. Dr. Bartley stated that he usually weighed a small rod with his dish, and stirred the milk solids, just as they approached dryness, thus obtaining a powdery residue readily acted upon by the solvent.

MR. MARTIN stated that in using a wider dish than usual, side by side with the ordinary size, though the milk-solids were more thinly spread, the difference of results on extraction were practically nil.

MR. GLADDING observed that in using the Adams process, he got 0.2 per cent. difference when the milk was dropped upon the paper, from that obtained when the milk was soaked up into the paper as prescribed by Mr. Adams.

The question of legislative enactments was referred to. In some of our States, the laws prescribe a certain minimum percentage of solids, of fat, &c., that shall exist in milk offered for sale. As these requirements have been based on results by the Wanklyn method, the question of the method of analysis, or of proposed modifications of the standard, are of grave importance. Dr. Newton spoke of difficulties in enforcing the law in New Jersey in the case of milks evidently adulterated, since the State Agricultural Experiment Station had, by use of the method of drying with sand, obtained lower figures for solids on genuine milks than had been obtained by other methods.

DR. BARTLEY suggested that in controlling a milk supply, speed was often of great importance, and that it might sometimes be necessary to sacrifice absolute scientific accuracy, for the sake of rapidity, with fairly accurate results comparable among themselves.

DR. KENT thought that the asbestos method was as rapid as any other, acknowledging, however, that where the ash has to be determined, a separate portion must be taken.



DR. BARTLEY said that after trying evaporation with sand, &c., and different forms of extraction apparatus, he had thrown them all aside, with the fragile and costly apparatus, and had adopted the modified Wanklyn method as the most rapid and serviceable.

DR. GROTHE expressed his preference for the Soxhlet areometric method, both for speed and accuracy.

A committee, consisting of Drs. Newton and Waller, was appointed, with power to add to their number, for the purpose of investigating and reporting on the comparative value, &c., of methods of milk analysis.

### A SIMPLE AND EFFECTIVE FAT EXTRACTION APPARATUS FOR USE IN MILK ANALYSIS.

BY F. WALLIS STODDART.

THERE have been so many descriptions of improvements in milk analysis published of late that I am tempted to send you some account of an extractor which I have had in use now for a year, and which, I believe, cannot be equalled for simplicity and effectiveness.

This piece of apparatus somewhat resembles one described in the last number of the ANALYST, but is far simpler. It is made entirely of glass, in two pieces, as follows:—The upper part consists of a yard of  $\frac{3}{4}$  in. glass tube, preferably fitted as an upright condenser, blown at the lower extremity on to a cylindrical glass cup of 3 in. diameter. The lower piece consists of a glass cup similar to that just described, having stoppered into its centre about 8 inches of glass tube, provided at its junction with the cup with the little arrangement first described. I believe, in the Shenstone vertical condenser for enabling condensed liquid to flow downwards without checking the upward current of vapour. This shorter tube is connected with a flask of petroleum ether. Finally, the upper cup (inverted) is accurately ground into the lower.

The mode of using is as follows:—The flat dish containing the residue, obtained by simple evaporation without stirring or addition of any kind, is placed, residue downwards, on a filter paper, which is then turned over the edge of the dish so as to keep its place, and the whole put, still upside down, into the lower cup: the upper part of the apparatus is then fitted on, and the flask containing ether boiled on the water bath.

By this means a copious current of hot vapour plays almost directly upon a mere film of milk residue which is completely exhausted in an hour or less. This apparatus has been made by Cetti in two sizes, one adapted for a single dish in cases where the fat is to be weighed, and the other capable of taking several dishes at the same time; in the latter case the dishes may stand upon their edges, or be placed one above the other. The ground surfaces are so accurate that there is no appreciable loss of ether after several hours use.

Whilst upon this subject permit me to add, that in my opinion, porcelain dishes are in all respects far preferable to platinum, provided they are of the proper shape, viz., about  $2\frac{3}{4}$  in. by  $\frac{1}{2}$  in., with flat bottom and vertical sides. Such dishes, as far as I know, are only to be obtained of one London firm, but could no doubt be produced by others if there were a sufficient demand.

---

 ON A NEW VARIETY OF PYRITES FOR SULPHURIC ACID MANUFACTURE.

BY DR. DRINKWATER, F.C.S., ED. SCHOOL OF MEDICINE.

IN using the term "*New*" for the form of Pyrites I am about to describe, it must be understood to apply only in a commercial sense. Pyrites are, as is well known, largely used for the manufacture of sulphuric acid, the advantage being cheapness, and the disadvantage (often a great one), the presence of arsenic and other impurities, which interfere with the use of the acid for many manufacturing purposes—prohibiting its use, in fact, in many cases. The use of "Sulphur Acid" instead of "Stone Acid" entails a considerable addition to cost of manufacture; and hence several methods have been devised to remove the arsenic (only one impurity of many) from stone acid, so as to enable it to take the place of the more expensive sulphur product.

A "Stone"—to use the technical term for Pyrites—which is absolutely free from arsenic, and, at the same time, possessing physical properties of value to a manufacturer, is a "find" of some importance to a vitriol-maker.

Such a specimen has recently passed through my hands; and the results of the examination, I think, are of sufficient interest to lay before the readers of THE ANALYST.

During the last ten years, I have examined some hundreds of samples of various kinds of pyrites used in sulphuric acid manufacture, and I have found arsenic in all. I do not say that this experience includes all known varieties of pyrites, but it certainly includes a number which are mentioned in several works as being free from this objectionable and troublesome impurity.

The amounts varied, even in the same description of ore. The largest amount I have found in average samples of a cargo is 2 per cent.

Two ores only have I found to be absolutely free. One was an Algerian; the other a Welsh ore. I carefully examined the former for arsenic myself, and the following is a complete analysis made by my assistant:—

Sulphur	..	..	..	..	..	44·8
Insoluble	..	..	..	..	..	5·2
Iron	..	..	..	..	..	46·6
Lead	..	..	..	..	..	·02
MnO <sub>2</sub>	..	..	..	..	..	·22
Calcic Carbonate	..	..	..	..	..	3·1
						99·94

It also showed traces of nickel and cobalt.

This Algerian ore was of a greenish colour, of a soft character, making a large quantity of smalls, and difficult to burn in the kiln. It made very good acid; but, with the greatest care, at least 4·5 per cent. of sulphur remained in the burned ore.

The Welsh ore is of a different character. It is very hard, and difficult to break. In appearance it resembles the white pyrites of Saxony. It makes very little smalls; burns well and completely in the burners.

The following is a complete analysis :—

Insoluble matter	..	..	..	..	5.8
Sulphur..	..	..	..	..	48.3
Iron	..	..	..	..	42.1
Arsenic ..	..	..	..	..	<i>nil.</i>
Alumina	..	..	..	..	1.4
Carbonate Calcium	..	..	..	..	2.5
					100.1

Another specimen, containing some quartz, and not, perhaps, a fair average sample, showed only 45 per cent. of sulphur.

The burned ore I sampled from a large bing with great care, and it only contained 3 per cent. of unburned sulphur. The great hardness would lead one to suspect that more than 3 per cent. would be wasted ; but a considerable practical experience has shown this to be otherwise.

There is no difficulty whatever, with this ore, to keep the burners up to a full red heat.

The acid produced is certainly of a superior quality for pyrites acid. It is of a good colour, and entirely free (as would be expected) from arsenic. In fact, it could not be distinguished, either by physical or chemical tests, from the best sulphur acid.

I give an analysis of the acid made from this ore, and I may state that I have partially analysed several samples of acid made by another firm who are using the same mineral.

ANALYSIS OF ACID. GRAVITY 1.841.

Hydrated Sulphuric Acid	..	..	..	..	97.14
Water not required for Oil of Vitrol strength	..			..	28.26
Sulphate of Lead	..	..	..	..	.003
Saline Matter..	..	..	..	..	.031
Arsenic	..	..	..	..	<i>Nil.</i>
Nitrogen Compounds	..	..	..	..	<i>Nil.</i>
					100.000

These acids are as good, for any commercial purpose, as the best sulphur acid ; and, of course, can be procured at a much cheaper rate.

The Algerian ore has apparently disappeared from the market ; but this Welsh ore, raised from the Cae Coch Mine, is superior in many respects, and will, I have no doubt, when fully known, be largely used for the production of an acid which might be sold as sulphur acid. It has been examined by a well-known Swansea firm, and also by Messrs. Johnson, Matthey, and Co., who both certify it free from arsenic.

It must be remembered that our supplies of sulphur are obtained from only two localities, although I believe that a third has recently been discovered.

The sulphur which is known as recovered sulphur is not by any means free from arsenic—at least, that is my experience—and, therefore, can hardly be reckoned on as a

source of pure acid. Taking all these facts into consideration, it will at once be seen that the matter is an important one to manufacturers of sulphuric acid, and, I think, a sufficient excuse for my taking up the pages of THE ANALYST.

For the sake of comparison, I append the results of my examination of a number of well-known varieties for arsenic; and, while on the subject, may state that, after a number of trials, I find the fusion method for estimating arsenic gives the best results. It is quick and easy to work, and not so liable to error as the process of oxidation with nitric acid. After fusion, I dissolve in water, and precipitate with washed  $H_2S$ , redissolving the precipitate in carbonate of ammonia, and again precipitating with acid—weighing on tared filter as  $As_2S_3$ .

## ARSENIC IN PYRITES, PARTS PER CENT.

	1	2	3
Cornish Pyrites ..	.93	.56	.5
Norwegian „ ..	.32	.20	—
Belgian „ ..	.40	1.03	.22
Spanish „ ..	1.81	1.65	.89
(Most Spanish ores contain selenium also.)			
Irish „ ..	2.00	—	—
Recovered Sulphur..	.68	—	—

I have to thank Mr. Veitch, of Crief, and Mr. MacArthur, Leith Chemical Works, for supplying me with a number of specimens, especially of the Welsh ore; and also for allowing me full opportunities for observing the behaviour of the ores in manufacturing operations. Also Mr. Marshall for assistance in the analysis of a number of pyrites samples.

## MONTHLY RECORD OF RESEARCHES IN FOOD ANALYSIS.

At the last meeting of the American Chemical Society, M. Liebschütz described the following method for—

THE EXAMINATION OF BUTTER AND OLEOMARGARINE, being a modification of the method of J. David (Comptes Rendus, 1882, xciv., 1427). Like M. David, he makes a baryta soap with the fatty acids, by treating the fat with crystallised barium hydrate and alcohol, evaporating off the alcohol, and washing out the glycerine; then removing the excess of baryta by sulphuric acid, and filtering, using litmus as an indicator of the exact point of neutralisation of the baryta. The residue after concentration, he finds, however, is by no means a mixture of glycerine and water only. The addition of alcohol in excess throws down a considerable amount of salts which have remained in solution; by evaporation after that treatment, the glycerine is obtained, and after drying (finally in the air bath), the product is weighed. Pure butter yields about 13.7 per cent. of glycerine in this way, while oleomargarine yields only 7 per cent. The product when ignited, affords when derived from butter fat 5 per cent. of ash (barytic), but when from oleomargarine from 0.3 to 0.6 per cent. of ash. In reply to a question, M.

Liebschütz said that he supposed that the glycerine derived from butter fat contained barium salts of some of the volatile fatty acids of the butter, or possibly sulpho salts of the same, but he had not examined them.

The 5 per cent., or 0·3 to 0·6 per cent., of (barytic) ash is reckoned not on the amount of the original butter fat, but on the weight of impure glycerine obtained. That point was not made clear in the paper, but was brought out in the discussion.

E. W.

ON THE DIRECT DETERMINATION OF INVERT SUGAR WITH REFERENCE TO THE SUGAR TRADE.\*—By H. BODENBENDER.—*Deutsche Zuckerindustrie*, 1884, p. 1253.—The author has already drawn attention to the fact that many sugar beets contain a substance which possesses the property of converting cupric oxide in alkaline solution, into yellow cuprous hydrate. As this substance is not destroyed by boiling with alkalies, it must be present, with its power of reducing cupric oxide, in all raw products. But considerable trust has been placed lately in the determination of the invert sugar in trade-products; and, as the latter is also recognised by its property of reducing oxide of copper, it is of importance to be able to distinguish between the two substances. Invert sugar is destroyed by boiling with alkali, whereas the above-mentioned substance remains unaltered. Sugar, molasses, etc., which are to be tested for invert sugar, must be titrated directly, and after boiling with potash. The invert sugar is the difference, and not, as is at present assumed, the direct determination.

Commercial sugars examined in this way, show always a small quantity of this substance and, on the other hand, little or no invert sugar.

F. H. H.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

THE QUANTITATIVE DETERMINATION OF TANNIN.—By DR. FR. BECKER†, Ber. d. Osterr. Ges. Z. Förderung d. Chem. Ind., 1884, 107.—The method adopted by the author is the following:—About five grms. methyl violet are dissolved in a litre of hot water and filtered, on cooling, into a well-stoppered flask.

Ten grms. of pure well dried tannin (Schering's Tannin levissimum) are dissolved in a litre of water; further ten grms. of the tannin, under examination (of which the water is determined separately), are dissolved in water and made up to a litre.

Fifty c.c. of the methyl violet solution are run into a beaker, and heated with 450 c.c. of water to about 50°C. Into this solution is run the pure tannin solution, the mixture being constantly stirred, until all the colouring matter is precipitated, which may be ascertained by filtering a small quantity. If the filtrate is still coloured, it is poured back and more tannin run in. A good filter-paper must be used, or the filtrate runs through turbid.

Having determined how much pure tannin solution is required to decolourise 50 c.c. of the methyl violet solution, the tannin solution under examination is treated in

\* *Chemische Industrie*, Jan. 1885, p. 22.

† *Chem. Zeit.* 30 [1885], p. 534.

the same way, and its value in comparison with pure tannin calculated from the two results.

This method may also be applied to the raw materials (containing tannin) used in dyeing, for instance, sumach, knopfern, etc., in which case 20-40 grms. to the litre are used.

F. H. H.

E. JOHANSON finds that the precipitation of tannin by a solution of gelatine is effected more completely, and in a better condition for filtration, if, besides ammonium chloride, as proposed by Schulze and Lehmann, there is also added a small quantity of chromium sulphate or of chrome-alum. The author proceeds in the same manner as Lehmann, but he adds to 100 c.c. of the solution containing sal-ammoniac from 5 to 8 drops of a solution containing 1 part chromium sulphate in 25 parts of water. In order to ascertain the end of the reaction, he filters small quantities into two test glasses of equal width, adds to the one a few drops of a solution of gelatine, observing if the two liquids, when held up against a sheet of black glazed paper, appear opaque or transparent. As long as a precipitate is formed, these portions and the washings of the little filters are poured back to the main quantity. If acetic or tartaric acid is present, the liquid should be neutralized before proceeding to the determination. Johanson points out that, though this method gives good results with the tannin of galls and of oak-bark, an extract of coffee gives no precipitate with solution of gelatine, so that caffeo-tannic acid cannot be determined in this manner. This shows that only quantities of tannin of one and the same kind can be compared with each other.

W. H. D.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

A METHOD FOR THE ESTIMATION OF ZINC IN TUTTY POWDER.—(Zinc dust) was described by M. Liebschütz at the last meeting of the American Chemical Society held May 1st, 1885. It consists in treating one gramme of the powder (after removing metallic iron by means of the magnet) with a 10 per cent. solution of copper sulphate, stirring with a platinum spatula. The precipitated copper, after thorough washing with water and dilute sulphuric acid, is dissolved in nitric acid and titrated with potassium cyanide. The amount of copper thus found multiplied by 1.03 gives the amount of zinc present. Lead, if present, should be removed by evaporating the nitric acid solution down with addition of sulphuric acid, dilution and filtering, the results are only approximate (within 2 to 3 per cent).

E. W.

DETERMINATION OF GRAPHITE IN MINERALS.—In the School of Mines Quarterly (Vol. vi., No. 2) (N.Y.), J. B. Mackintosh proposes to estimate graphite, by fusing the coarsely pulverised mineral with caustic potash in a silver crucible at a low heat, stirring with a silver wire. When no more gritty particles are perceptible, the crucible is cooled, the melt soaked out with water, and the solution filtered through a weighed Gooch crucible. After washing with water and hydrochloric acid, the graphite alone is left. The results, so far as tested, seem to be all that can be desired.

E. W.

SILVER IODIDE AS A BLOWPIPE RE-AGENT—was the subject of a paper by Mr. P. Casamajor at the May meeting of the American Chemical Society. AgI. was found to give the same results as the tincture of iodine, proposed by Messrs. Wheeler and Ludesking (*Chemical News* LI. 88) and in some cases might, perhaps, replace it with advantage. It was noted that with certain coatings the tint did not keep well on the plaster of Paris tablets, mentioned by those gentlemen.

E. W.

ON THE SEPARATION OF TITANIUM FROM NIOBIUM AND ZIRCONIUM.\*—By E. DEMARCAÿ. —*Comptes. rendus.* [1885]. 100-740.—The separation of titanium from niobium and zirconium, according to the methods in use, is troublesome and long. A quicker and easier separation is the one proposed by the author, in which the solution of the oxides in hydrofluoric acid is fractionally precipitated at the boiling temperature by ammonium hydrate.

If titanium is to be separated from niobium, very dilute potassium hydrate is added to the boiling, very dilute solution of the oxides as long as it gives an acid reaction; if the boiling be continuous, niobic acid separates out. As soon as litmus-paper is coloured lilac by the solution, the potassium hydrate is replaced by very dilute ammonium hydrate, which is added until the solution is just neutral. The precipitate formed contains scarcely a trace of titanous acid. The solution is filtered hot, and the precipitate washed with hot water. The filtrate is heated to boiling, and ammonium hydrate added until strongly alkaline. The liquid then contains only titanous acid which, after filtering, separates out. The intermediate precipitate consists of a mixture of titanous and niobic acids, and must be submitted to the same process.

Zirconium is not precipitated by ammonium hydrate from the boiling hydrofluoric acid solution, provided the liquid already contains ammonium fluoride. A very dilute solution is therefore fractionally precipitated with dilute ammonium hydrate, first at a boiling and afterwards at a lower temperature. The separation takes place just as quick as with niobium. Finally, the zirconia is precipitated by potash, as it is only partially precipitated by ammonium hydrate even in the cold.

F. H. H.

A METHOD FOR THE DETECTION OF ARSENIC. BY H. HAGER. *Pharmaceutische Centralhalle* [1885], No. 5, p. 45.—This method possesses the advantage that it can be carried out in solutions strongly acid with hydrochloric or sulphuric acid, and gives good results with a dilution of 1 : 40,000.

The method is the following:—The solution in hydrochloric acid is brought into a test-tube of about 1 cm. in diameter, and so that the liquid stands about 3 cms, high. A piece of tin-foil about the size of half-a-crown is then folded together and introduced into the liquid. On gently warming the solution to 40°-50° C, it turns yellow, yellowish-brown or brown-yellow, according to the quantity of arsenic present, at the same time generally becoming turbid. After some time metallic arsenic separates out. In the absence of arsenic, the solution remains colourless, even when a metallic separation takes place.

\**Chem. Zeit.*, 26, 470.

In solutions containing metals which combine with the arsenic during the separation (*e.g.* antimony) the reaction does not take place. Chlorine, nitric acid and nitrates in small quantities do not interfere.

F. H. H.

### REVIEWS.

AIDS TO THE ANALYSIS OF FOOD AND DRUGS, by *H. Aubrey Husband, M.B.* London: Bailliere, Tindall, & Cox.

THE object of this addition to Messrs. Bailliere's "Students' Aid Series," is stated in the preface to consist in the supplying of "a want often expressed to him by candidates for sanitary degrees for a collection of short processes for the detection of the commoner adulterations in Food and Drugs." Taking the book, therefore, in this light, as simply intended to give the superficial amount of knowledge required under such conditions, it will be doubtless found to perfectly answer its purpose. We do not intend to engage in the amusement of "breaking a butterfly," which any real criticism of this little work would involve, but cannot help pointing out the actual danger and absurdity of examinations which can be passed on the information that a student would absorb from such books alone. Local authorities might, deceived by a man's possession of a "degree in Sanitary Science," appoint him public analyst as well as a medical officer; then would come a state of affairs disastrous to both traders and the public. Supposing this were so, and the neophyte received a sample of some of the best prepared cocoas in the market, and acted upon the dictum on page 15 that "if the fat be found below 20 per cent., the cocoa should be returned as adulterated;" or, again, if he got some bread, and following his "examination" knowledge, he estimated the alumina as instructed, and then simply allowed "8 grains of alum in the 4lb. loaf for accidental additions" (page 53) without taking any ratio to the silica found, what would Somerset House say to him? The processes have evidently been chiefly condensed from Mr. Blyth's book, for we find many peculiarities of that work, without, however, having the saving explanations given us at the same time.

### SPECIAL NOTES.

UNIFORMITY OF CHEMICAL ANALYSIS.—This question has again been considered by the Chemical Trade Section of the London Chamber of Commerce. The following resolution, passed at the meeting in March, was confirmed on 23rd April, 1885:—"That (referring to the Report of the Analytical Chemists' Sub-Committee of 1884) the margin for divergence be one per cent. In case the difference should be found to be greater, the results should be communicated to both chemists, and, in the event of their not being able to agree within the limit of one per cent., the sample should be referred to another chemist, in accordance with present custom."

THE Directors of the Gas Light and Coke Company are circulating the following suggested amendment to the late Dr. Voelcker's well-known specification for coal tar creosote. The author is Dr. Tidy, who proposes:—1. That the creosote shall be completely liquid, at a temperature of 100° F., no deposit afterwards taking place until the oil registers a temperature of 95° F. 2. That the creosote shall contain at least 25 per cent. of constituents that do not distil over at a temperature of 600° F. 3. That, tested by the process hereafter described, the creosote shall yield a total of 8 per cent. of tar acids. 4. That it shall contain no admixture of bone oil, shale oil, or of any substance not obtained from the distillation of coal tar; and that the first 25 per cent. of the distillate shall have a specific gravity



greater than that of water. *Process to be adopted for determining the coal tar acids*:—1. 100 c.c. of the well-mixed creosote are to be distilled at a temperature of 600° F., until no further distillate comes over. The distillate so obtained is to be mixed and well shaken in a stoppered flask, with 30 c.c. of a solution of caustic soda, having a specific gravity of 1·200, water being 1·000. The mixture is then to be heated. This done, the stopper is to be replaced in the flask, and the hot mixture again shaken vigorously for at least a minute. The contents of the flask are now to be poured into a separating funnel, and the soda solution drawn off. The creosote is to be heated a second and a third time in a similar manner with the caustic soda solution, except that only 20 c.c. of the soda solution shall be used for the second and third extractions, instead of 30 c.c. as in the first extraction. 2. The three soda solutions are now to be mixed together. When cold, any particles of creosote are to be got rid of by means of a separating funnel. This done, the solution is to be thoroughly boiled, in order to expel the last traces of creosote present in the solution. The mixture is then to be allowed to cool. When cold, dilute sulphuric acid (1 of acid to 3 of water) is to be added (about 35 c.c. will be required), until the solution becomes slightly acid to litmus. The whole is then to be poured into a separating funnel, and allowed to stand until perfectly cold, and the tar acids well separated. 3. The tar acids are now to be dissolved in 20 c.c. of the caustic soda solution (sp. g. 1·200) and 10 c.c. of water. The mixture is then to be boiled, and filtered through a funnel with a plug of asbestos. The asbestos plug is to be washed with not more than 5 c.c. of boiling water. The solution is to be allowed to cool perfectly in a 100 c.c. measure. It is then to be rendered slightly acid with dilute sulphuric acid (1 to 3), (10 c.c. will probably be found sufficient for this purpose). The whole is again allowed to stand for *two hours* until perfectly cold, when the percentage (by volume) of the tar acids is to be read off. *Process to be adopted in estimating the quantity of Distillate*:—The operation is to be conducted in a retort, fitted with a thermometer, and heated with the naked flame of a Bunsen burner. The heat applied is to be gradually raised to 600° F., and continued at that temperature until no further matters distil over. The distillation of the 100 c.c. should be completed within half an hour.

### C O R R E S P O N D E N C E .

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

TO THE EDITOR OF "THE ANALYST."

DEAR SIR,—At the March meeting of the Society of Public Analysts, I brought forward some experiments and observations referring to Mr. Adams' new method for the analysis of milk, which were criticised by Mr. Adams at the subsequent meeting in April. As I had no opportunity to reply to Mr. Adams, I should feel much obliged if you could find space in the next number of THE ANALYST for the following remarks:—

1. I did not admit that the total solids got by the paper method are, in every instance, much below those got by the usual drying, but pointed out that, in some instances, both methods—even in the hands of Mr. Adams—as well as the calculation by a mathematical formula, give agreeing results.

2. When referring to the error arising from the presence in filter paper and in blotting paper of a substance soluble in ether, I did not contend that the error in the paper method is twenty times as large as that in the Soxhlet method, but simply stated that the error in the latter method is multiplied by ten, while the three times larger error in the paper method is multiplied by twenty.

3. I did not say something about the introduction of 4 or 5 grams. of weight as an element of difficulty, but spoke of a piece of blotting paper, 55 square inches large, and weighing 4·5 grams. as an item which might be open to grave objections.

4. It is rather astonishing to find Mr. Adams speak of valuable experiments upon which elaborate pains had been bestowed, and at the same time suspect rough handling of the paper coils—perhaps even with greasy fingers. I must refuse either the compliment or the reproach.

5. Leaving it to Mr. Adams to allude to geographical explorations, I content myself with exploring as much as I can that field on which I am working, and this I do without giving anybody reason to say that I am nervously alarmed. No doubt there are some points in Mr. Adams' method which want further investigation and explanation. I tried to do my share of the work, and urged other analysts interested in the question to do theirs, so that we might get at the truth. Mr. Adams took much pains to criticise my experiments, but did not bring forward a single new fact which might clear up those dubious points, to which I took occasion to draw attention.

Faithfully yours,

Bayswater, May, 1885.

P. VIETH.

# THE ANALYST.

JULY, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
(a) REPORT OF MEETING .. .. .	117	(a) DETECTION OF CITRIC ACID, G. MANN ..	126
(b) SOME OBSERVATIONS ON THE PERMANGANATE TEST IN WATER ANALYSIS, BY DR. DUPRE, F.R.S. .. .. .	118	(b) EUCLYPTOL, C. JAHNS .. .. .	126
(c) DISCUSSION UPON DR. DUPRE'S PAPER	121	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
(d) NOTE ON A SAMPLE OF ADULTERATED PEPPER, BY DR. BOSTOCK HILL, F.I.C.	122	(a) DETERMINATION OF PHOSPHORIC ACID, C. GLASER .. .. .	127
(e) DISCUSSION ON DR. HILL'S PAPER ..	123	(b) KJELDAHL'S METHOD FOR DETERMINATION OF NITROGEN, E. BOSSHARD .. .. .	127
PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS:—		(c) ANALYSIS OF TUNGSTEN STEEL, L. SCHNEIDER AND F. LIPP .. .. .	128
REPORT OF MEETING, BY DR. WALLER .. .. .	123	(d) NEW INDICATORS FOR USE IN DETERMINATION OF CAUSTIC ALKALIES IN THE PRESENCE OF CARBONATES, R. ENGEL AND J. VILLE .. .. .	128
ORIGINAL ARTICLE:—		(e) TITRATION OF IRON ORES, W. HEMPEL	129
SUBSTITUTES FOR TARTARIC ACID IN BAKING POWDER, BY J. NAPIER, F.C.S. .. .. .	124	(f) SEPARATION OF COPPER AND CADMIUM, A. BEHAL .. .. .	129
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—		(g) NEW METHOD FOR THE DETERMINATION OF PHOSPHORIC ACID BY MOLYBDENUM, MEINEKE .. .. .	129
(a) DETERMINATION OF CANE-SUGAR, GRAPE-SUGAR, AND DEXTRIN IN THE SAME SOLUTION. BIARD AND PELLET ..	125	(h) QUANTITATIVE DETERMINATION OF URIC ACID, E. LUDWIG .. .. .	130
(b) DETECTION OF LIQUORICE IN BEER, R. KAYSER .. .. .	125	(j) ANALYSIS OF VULCANISED CAOUTCHOUC, B. UNGER .. .. .	131
(c) DETERMINATION OF FREE SULPHURIC ACID IN VINEGAR, B. KOHNSTEIN ..	126	REVIEWS:—	
		(a) SIMON'S MANUAL OF CHEMISTRY ..	132
		(b) ALLEN'S COMMERCIAL ORGANIC ANALYSIS .. .. .	133
		CORRESPONDENCE, &C. .. .. .	134

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

A General Meeting of this Society was held at Burlington House, on Wednesday, the 10th June. Dr. Alfred Hill, President, in the chair.

The minutes of the previous meeting were read and confirmed.

Mr. Clayton and Mr. Fox having examined the voting papers reported that the following gentleman had been elected as a member:—Mr. A. C. Wilson, Analytical Chemist of Stockton.

The following papers were then read and discussed:—

“Remarks on Sewage-farm Milk and Butter,” by Dr. Alfred Hill, F.I.C.

“Note on a Sample of Adulterated Pepper,” by Dr. Bostock Hill, F.I.C., F.C.S.

“On Admixtures of Malt Extract, with certain Fixed Oils,” by E. G. Clayton, F.C.S.

“On the Determination of Dissolved Oxygen in Water,” by Dr. Dupré, F.R.S.

The next meeting of the Society will be held at Dublin on August 1st., when ladies are invited to attend.

## SOME OBSERVATIONS ON THE PERMANGANATE TEST IN WATER ANALYSIS.

BY DR. DUPRE, F.R.S.

*(Read at the Meeting, May 13th, 1885.)*

The permanganate test, as is well known, was first proposed for use in water analysis by Professor G. Forchhammer, of Copenhagen, in the year 1850, but only got into pretty general use after it had been taken up and improved by Prof. Miller. The test, however, even as improved by Prof. Miller, still laboured under several rather serious defects. In the first place, no definite temperature was given at which the experiments should be conducted, whereas, as I have shown, variations of temperature, even of no very great amount, very seriously affect the result, particularly in the case of bad waters. Secondly, the operation was carried out in open flasks or beakers, in consequence of which the presence of various gases or dust in the air affected the result. Dr. Tidy advised the making of a check experiment with pure water, simultaneously with the experiment on the water under examination, and to deduct the oxygen absorbed by the pure water from that absorbed by the water under examination. It is obvious that no great accuracy is thus obtainable, and the question of fixity of temperature is left untouched.

To obviate these uncertainties I proposed, some years ago, the use of a constant temperature, and of a closed flask or bottle. This suggestion has been adopted by the Water Committee of our Society, and is described in the directions for the analysis of water issued by the Society. The temperature adopted is, as all here will recollect, 80° F, and this has been done so that the analyst can, at all times of the year, obtain a constant temperature of that degree, by heating. When I first adopted the closed vessel, I did so solely with a view to obviate the necessity of a parallel experiment with pure water, and to render the test not only simpler, but at the same time also more accurate. After I had adopted the plan I found, however, that it carried with it another considerable advantage in the fact that it enables us to apply the test for the comparative estimation of organic matter, to waters to which the open test is absolutely inapplicable. The open test becomes perfectly useless in a water containing appreciable quantities of chlorides; but the presence of chlorides is no hindrance to the test when a closed vessel is employed. The test fails in an open vessel, as every chemist will understand, on account of the mutual action of permanganate and hydrochloric acid, whereby the one becomes reduced and the other oxydized into water and chlorine, part of this latter escaping. When, however, the experiment is carried on in a closed vessel, the chlorine, which is of course also produced, is retained in solution, and when at the end of the experiment iodide of potassium is added, this free chlorine liberates exactly the same amount of iodine as would have been liberated by the permanganate from which it was produced, and the effect is the same as if no permanganate had been destroyed by the presence of chlorides. It thus becomes possible to estimate the amount of oxydizable organic matter, even in sea water containing upwards of 2,000 grains of chlorides per gallon. In illustration of the advantage thus derived, I cannot perhaps do better than refer to the series of analyses of Thames water, made on behalf of various authorities, to be

laid before the Royal Commission on Metropolitan Sewage Discharge. Those that used the permanganate test and closed vessel had no difficulty in tracing first the gradual increase in the amount of oxydizable organic matters in the water of the river proceeding from Teddington towards Barking, and next its gradual diminution when proceeding from Barking and Crossness towards the sea. Thus, taking the mean of the analysis at low water made during the months of January, March, and May, 1882, we had

Teddington Weir	..	..	..	0·109	grs.	oxy.	abs.	per	gall.
Chelsea	..	..	..	0·165	„	„	„	„	„
Greenwich	..	..	..	0·204	„	„	„	„	„
Barking	..	..	..	0·272	„	„	„	„	„
Northfleet	..	..	..	0·151	„	„	„	„	„
Thames Haven	..	..	..	0·106	„	„	„	„	„
Yantlet Creek	..	..	..	0·070	„	„	„	„	„

at the latter point, which is nearly opposite Southend, the water is almost all sea water, and contained less oxydizable organic matter than the river water did at Teddington.

On the other hand, those that used the permanganate test in an open vessel could not use it at all at points of the river at or below Greenwich. This result is all the more interesting, seeing that the organic carbon and nitrogen process also in some measure broke down. Sea water contains not only appreciable proportions of organic carbon and nitrogen, but shows a ratio between these two elements worse even than is found in pure sewage.

The difference in the results yielded by the permanganate test in a closed and open vessel respectively, will, of course, increase with the amount of chlorides present in the water, and also with the temperature at which it is carried out. Now, 80° F. is, even for a closed vessel, but more particularly if the vessel has to be opened during the duration of the experiment in order to add more permanganate, rather high, and yet if the test is to be made at a temperature which requires a slight amount of heating, even in summer, a lower temperature cannot be well chosen. In order to get over this difficulty, I have lately adopted a plan of making the experiment at or near 0° C. One quarter—or half—litre of water is placed into a perfectly clean well-stoppered glass bottle, of about 350 c. c. or 600 c. c. capacity respectively. The water is cooled by putting the bottle in iced water, 10 c. c. of a solution of tribasic phosphoric acid, and 10 c. c. permanganate solution are added, and the iced water containing the bottle is put for twenty-four hours into an ordinary refrigerator, containing some ice. At the end of that time, the permanganate remaining is estimated as usual. The substitution of phosphoric for sulphuric acid renders the titration of the iodine somewhat sharper, and there is less tendency of the blue colour coming back again when once it has been discharged. The experiment should, of course, be started in the morning, so that there is time to notice the effect produced by several hours standing, so as to be able to decide whether or no any more permanganate ought to be added before letting it stand over night (10 c. c. permanganate to  $\frac{1}{4}$  litre water is quite sufficient in the case of pure sea water). I find that the oxygen absorbed from permanganate at 0° C. in presence of phosphoric acid is about the same as

that absorbed in four hours at 80° F. in presence of sulphuric acid, although the two experiments do not always yield the same relative results when applied to different waters.

At this low temperature less chlorine is produced than at 80° F., and what there is shows far less tendency to escape. By adopting this low temperature, the advantage is gained that all kinds of water can be tested on a uniform plan, and comparative results arrived at.

In connection with this subject, I would mention that the plan advocated by many chemists, particularly in Germany—viz., boiling the water for ten or more minutes with sulphuric acid and permanganate, is, of course, totally inapplicable to waters containing an appreciable proportion of chlorine. If the water contains about 100 grs. of common salt per gallon, the whole of the permanganate, from 10 c. c. of the standard permanganate solution, becomes destroyed, and an amount of chlorine nearly equivalent to it is expelled; and with far less than that proportion of chlorides, the test is so seriously affected, as to become illusory as a measure of organic matter oxydizable by permanganate. (See table at end.)

As it is, as stated above, desirable to have a given test arranged, if possible, in such a manner that it can be applied to all, or at least most, varieties of water, in the same way, so as to insure fairly comparative results, this process of boiling the water with acid permanganate, which, strictly speaking, can only be used in the absence of chlorides, cannot be recommended for general adoption. At the end of this paper will be found a few analyses, illustrating the points here brought forward.

The phosphoric acid was made by dissolving 250 grams. of pure glacial phosphoric acid in water, boiling till it was all converted into tribasic acid, and, when cold, adding a dilute solution of permanganate until a faint tint remained perceptible after standing over night. The whole is then made up to 1 litre. The permanganate I always use is of the strength recommended by Professor Miller—viz., so that 1 c. c. contains 0.0001 grm. of available oxygen.

A few words as to the value of the permanganate test may not perhaps be out of place here.

No one will, of course, contend that this permanganate test will give us an absolute measure of the amount of organic matter in a water under examination. This, however, is no more than can be said against every process as yet proposed; even the organic carbon and nitrogen process gives two only out of at least four, and perhaps more, elements contained in the organic matter; and is, therefore, not an absolute measure of the actual amount of organic matter present. But this, I think, may be said in favour of the permanganate test:—A water (I mean a water naturally met with, not one artificially made with substances never found in water,) which does not absorb any appreciable proportion of oxygen from permanganate, may be pronounced a pure water; and no other, purely chemical, test so far known will then indicate anything else.

On the other hand, a water absorbing much oxygen must be pronounced impure. There is, of course, the well-known case of peaty waters; but these, if regard be paid

merely to analytical details, would be a stumbling-block in the case of every method of analysis.

For the purpose of tracing the gradually increasing, and again, gradually diminishing, degree of impurity of a river subjected, in part of its course, to sewage pollution, the permanganate test, as here proposed, is, I believe, by far the best.

One more point in favour of the permanganate test I may be permitted to bring forward. It is urged by adherents of the carbon and nitrogen process, as necessarily in its favour, that it at least gives the total amount of organic carbon and nitrogen contained in a water; and this is—in a great measure, at least—true as regards organic carbon. But is this necessarily an advantage? The process, as before stated, takes no notice of the other elements; and the original compounds may have undergone profound changes by oxydation, and yet contain the same amount of organic carbon and nitrogen; and, to carry this argument to its legitimate conclusion, the ammonia, nitric acid, carbonic acid, and water, into which the organic matter is ultimately changed, ought to be looked upon as in every respect as hurtful as the organic matter from which they were derived.

There is some reason to believe that as the organic matter is oxydized, it becomes less hurtful, and this change the permanganate process, in some measure, enables us to trace.

In conclusion, I would urge everyone not to rely on any one analytical process, but submit every sample of water brought before him to as many different tests as possible, not omitting the microscope, and to draw his conclusions from all available data. In coming to a conclusion, let him bear in mind the recommendation laid before this Society by Mr. Hehner and myself some years ago—a recommendation which, I am happy to say, has been adopted by the German Society of Analytical Chemists, namely, to take as your standard the unpolluted waters of the district from which the particular water is derived, and not any so-called standard of purity chosen more or less arbitrarily.

Oxygen absorbed from permanganate:—

Nature of Water.	In 10 min. boiling.	In 4h. at 80° F.	In 24h. at 0° C.
Trafalgar Square Water, (11.5 grs. Chlorine per gal.)	0.098*	0.0134	0.0160
Trafalgar Square Water $\frac{1}{2}$ grain Isinglass per gal.	0.182	0.0188	0.0233
Chelsea Co., Water (1.05 grs. Chlorine per gal.)	0.1995†	0.0568	0.0449
Pure Water	.. .. .	.. .. .	0.0032

#### DISCUSSION.

DR. DUPRÉ, before reading his paper, stated, that as he proposed to speak on Dr. Tidy's work, he was rather anxious that Dr. Tidy should be present, and consequently wrote to him on the 9th May, asking him to be kind enough to attend that meeting, but he was sorry to see that he was not there, and therefore his remarks must be made in Dr. Tidy's absence, through no fault of his (Dr. Dupré's.)

\* During the 10 minutes' boiling, the chlorine evolved was equivalent to 0.8 c.c. permanganate solution, or representing an absorption of 0.056 grains oxygen per gallon.

† During the boiling, an amount of chlorine was evolved equivalent to 0.15 c.c. permanganate solution, or representing 0.0105 grains of oxygen per gallon.

Dr. Muter said they must all thank Dr. Dupré for the very able remarks he had made on the subject of water analysis. It brought them back again to the old days when they used to fight their battles in the Water Committee, and to the very remarks made at that time. They discussed them all very fully, and came to the conclusion that if a man wanted a chemical opinion of a water, the best way was to employ the two processes, viz.—the albuminoid ammonia and the permanganate—and take the indications of both. They did not adopt the organic carbon process, as they did not think its results were to be compared with those obtained by the joint use of the others. As regards the phosphoric acid, he was very strongly with Dr. Dupré then, but he thought the majority of the committee had an opinion as to the greater convenience of the sulphuric acid. They, however, ordered the sulphuric acid to be carefully treated with permanganate, and to stand a certain time before it was used. In drawing the instructions he thought they not only had the closed bottle and fixed temperature, but the comparison experiment as well, and it was always his custom to do that. As regarded chlorine, there was no doubt that Dr. Dupré's change of temperature would be a very good thing, although the ordinary process did very well with saline waters—he could testify to that from one particular water which he knew of, and in which, although very saline, the permanganate results were a mere nothing. The great thing was to put in all the permanganate at once, and not open the bottle again. He thought they would all agree with Dr. Dupré, that any analyst who came forward and said the permanganate process was useless, merely because there was salt in the water, had tried the process in a prejudiced way.

Dr. Dupré, in reply to questions, said that about 10 c.c. of permanganate was generally sufficient, and he had always assumed that it was necessary that the water should remain decidedly pink. 10 c.c.'s were easily decolourised in water containing 100 grains of salt per gallon at the boiling temperature.

With regard to closing the bottle, there was the chance of a great loss by leakage, and a very large amount of permanganate would be decolourized, but at 80°, or still more at 0°, the action between the hydrochloric acid and the permanganate was trifling.

As to the extraordinary proportion of organic nitrogen to carbon in sea water, his opinion was that it might be due in part at least, to experimental error caused by the presence of chlorides.

There could be no doubt that a water containing absorbed oxygen oxidised less permanganate than water containing none, and if a water is boiled it always absorbs more permanganate than before it was boiled, and he believed that was due to the fact of the oxygen having been boiled out.

Mr. Cheshire said he had always found it most useful to use the microscope, and he thought it likely to give more reliable results than anything else.

---

#### NOTE ON A SAMPLE OF ADULTERATED PEPPER.

BY DR. BOSTOCK HILL, F.I.C.

(*Read at the Meeting, June 10th, 1885.*)

All those who have had much experience in the examination of pepper, know that adulteration of two kinds is practised. The addition of starch, chiefly rice starch, and the addition of sandy matter.

As a result of examination of a large number of samples of both white and black pepper, I find the ash to vary in the white from 0·8 to 2·0 per cent., while in the black it is generally from 3–5. In black peppers, however, of low quality, the ash may prove to be as high as 7 or 8 per cent., owing to grit or sand being introduced, I believe, in the shape of shop sweepings. I have come to this conclusion, owing to the fact that on more than one occasion I have discovered starch granules of different kinds, and in one instance a piece of chicory root. In the sample I desire to call your attention to now, we have quite another kind of adulteration. Under the microscope can be seen pepper cells, and a large quantity of matter of very doubtful origin. But on incineration an ash is left of a reddish colour, equal to 24·2 per cent. of the weight of the pepper taken. At first I thought this was sand, but on further examination I find it to be smooth, and scarcely at all gritty, and it proves to be, on analysis, finely-ground clay or

brick-dust, and by the intimate way it seems mixed, is undoubtedly added by the manufacturer.

I may add that last week a sample of pepper was submitted to me by an Inspector containing 11·08 per cent. of ash, which, although slightly gritty, appears to be greatly like that found in the previous sample.

I have examined peppers constantly for a period of nine years, but have never before seen an ash like those described above.

DISCUSSION.

MR. HEHNER said that he was informed that ordinary dirty pepper was being whitened with nitrate of bismuth, in order that a higher price may be obtained for it.

MR. BERNARD DYER said that he had had samples of pepper which had been sold in Mincing Lane as white, but which were in reality black, and had been beautifully coated over with a kind of whitish brick clay, which all washed off in water. It had been done so skilfully, that the peppercorns might be rubbed in the hands without its being noticed that they were sophisticated.

CONCLUSION OF THE PROCEEDINGS OF THE SOCIETY.

PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS.

COMMUNICATED BY DR. E. WALLER, NEW YORK.

AT the meeting of the American Society of Public Analysts held May 11th, 1885. Dr. Bartley, the president, remarked that, to his surprise, he had found that well waters contaminated with drainage from cow stables, in several instances had shown little or no free and albuminoid ammonia, though large amounts of nitrites and nitrates were detected.

A paper was read by W. Möller, Ph.B. on the Hübl method, (see ANALYST, vol. x page 9,) for the testing of oils and fats. After describing the theoretical considerations on which the process is based, and the method of analysis, a list of the results of numerous tests was given as follows :--

Shark liver oil.. .. .	268·2	Oleic Acid .. .. .	86·2
Manhaden oil .. .. .	170·8	"Olenic" acid .. .. .	85·7
Porpoise oil .. .. .	131·2	Butyric acids .. .. .	—
Seal oil .. .. .	103·4	Olein .. .. .	82·3
Sea-elephant oil .. .. .	88·5	Commercial Stearin .. .. .	1·7
Lard oil .. .. .	47·2		
Linseed oil .. .. .	175·7	Bees wax .. .. .	5·3
Corn oil .. .. .	119·2	Japanese wax .. .. .	5·61
Cotton seed oil .. .. .	107·9	Bayberry tallow .. .. .	1·38
Benne oil .. .. .	107·2		
Sweet Almond oil .. .. .	101·7	Butter .. .. .	36·8
Rape seed oil .. .. .	99·4	Oleomargarine .. .. .	53·5
English Mustard oil .. .. .	94·6	"Butter," a .. .. .	42·8
American Mustard oil .. .. .	85·5	"Butter," b .. .. .	43·0
Castor oil .. .. .	84·6	"Butter," c .. .. .	43·6
Olive oil, a, .. .. .	81·3	43·71 % Oleomargarine } .. .. .	43·52
Olive oil, b, .. .. .	102·9	56·29 % Butter } .. .. .	28·07
Palm oil .. .. .	48·6	19·54 % Oleomargarine } .. .. .	88·54
Cocoa Butter .. .. .	34·4	31·94 % Butyric acid } .. .. .	54·36
Muskat Butter.. .. .	31·6	48·52 % Butter } .. .. .	
Cocoanut oil .. .. .	6·8	68·24 % Olive oil } .. .. .	
		31·76 % Cotton seed oil } .. .. .	
Mutton fat .. .. .	57·3	47·88 % Butter } .. .. .	
Lard .. .. .	55·0	33·29 % Oleomargarine } .. .. .	
Beef suet .. .. .	38·4	18·82 % Cotton seed } .. .. .	

Butters "a" "b" and "c" were analysed by Dr. Waller by the method of washing



the insoluble fatty acids, &c., and found to contain 90 to 91 per cent. insoluble fat acids and 4.2 to 4.6 per cent. soluble fat acids. "Olive oil, *b*" though sold for genuine olive oil, was apparently (by this test at least) almost entirely, if not entirely cotton seed oil.

DR. MARTIN remarked that this process would seem to be good as an absolute method, and not only as an adjunct. About everything on the list which might be used to adulterate or be substituted for butter, gave iodine numbers so far removed from that for genuine butter, that no difficulty would occur. As to beef fat, that could not be used without a partial separation of the stearine, and the result would be an increase in the iodine number.

---

### SUBSTITUTES FOR TARTARIC ACID IN BAKING POWDERS.

BY J. NAPIER, F.C.S.

TARTARIC ACID and cream of tartar are largely used nowadays by bakers and others for raising bread and pastry, along with bicarbonate of soda; and no objections, that I am aware of, have ever been made to the use of these ingredients, as long as they are free from any injurious impurity. I would, however, draw attention to the fact that these "genuine" baking powders are apt to contain lead derived from the acids used. Tartaric acid, cream of tartar, and citric acid often contain notable quantities of lead; indeed, from the results of many experiments on different samples, I have come to the conclusion that neither tartaric nor citric acid can, commercially, be obtained free from lead.

Again, nearly all mineral waters contain perceptible quantities of lead—part of which, I have no doubt, being derived from the citric or tartaric acid used for flavouring.

Considering the prices of tartaric acid and cream of tartar, and the prices at which some baking powders are sold, the amount of active ingredients must be very small indeed, or some very much cheaper substitute is used instead. That cheap substitutes are used we have evidenced often enough—one of the first being alum, which is now universally considered inadmissible, although one may yet find a few even of the medical profession who still take an opposite view.

In an editorial in THE ANALYST for June, 1884, sulphate of alumina and bisulphate of potash are justly condemned as substitutes for tartaric acid—the latter, as there stated, being offered under "Royal Letters Patent" by a name similar to tartaric acid. Although bisulphate of potash is a good producer of carbonic acid, there can be no doubt its use is open to grave objection—for, even in doses of from 10 to 15 grains it is a purgative. The dangerous nature of sulphate of potash is well known to the French Government; and it is years ago since they condemned the sale of wines containing two grammes of sulphate of potash per litre, or less than one grain per ounce. Is it not possible that one of the objections to the use of alum may be the strong purgative action of the sulphate of potash which it contains? The quantity of bisulphate of potash recommended to be used for raising flour is from 1 to 1½ per cent.; so, reckoning a sack of flour to yield 100 four-pound loaves, one pound of bread will contain

5,000 grains of flour—which, at 1 per cent., equals 50 grains of bisulphate. Any one, therefore, eating half-a-pound (a very moderate quantity) of such bread is taking a dose of a powerful purgative, equivalent to 25 grains bisulphate of potash.

In last September's ANALYST, I drew attention to phosphoric acid as a substitute for tartaric and citric acids in mineral waters. This is now, I believe, extensively used with great success—not only on account of its cheapness as compared with tartaric and citric acids, but because it is sold free from lead and other objectionable impurities. As substitutes for tartaric acid and cream of tartar in baking powders and bread raisers, phosphoric acid, the acid phosphates of ammonia, potash, and lime, in the liquid and solid form, are at present offered; and I have examined samples of these, and found them free from lead and other metallic contaminations. With bicarbonate of soda, they appear to be excellent producers of carbonic acid—the resulting phosphates formed in the bread and pastry not having the purgative objections that sulphate of potash and soda have. Indeed, the Medical Works describe phosphate of soda as being comparatively mild in its effects, and tasteless, and well suited, on that account, for administration to children and invalids.

---

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

ON THE DETERMINATION OF CANE-SUGAR, GRAPE-SUGAR AND DEXTRIN IN THE SAME SOLUTION, BY BIARD AND PELLET. *Zeitsch. f. Anal. Chem.* 24, 2 p. 275.—The solution is first titrated with Fehling's solution and the grape-sugar determined. A second part is boiled with acetic acid, (which only inverts the cane-sugar) and titrated. Finally, a third portion is completely inverted with sulphuric acid and also titrated.

From the difference of the first and second titrations is calculated the amount of cane-sugar, and from the difference of the second and third, the dextrin.

F. H. H.

---

ON THE DETECTION OF LIQUORICE IN BEER.\*—BY DR. R. KAYSER.—Corresp. a *bayer Vert. d. Angew. Chem.* 1885, 17.—The author proposes the following method, which, according to him, allows of the detection of less than 1 grm. liquorice in 1 litre beer:—1 litre beer is evaporated on the water-bath to half the volume, and on cooling, precipitated with a slight excess of concentrated plumbic acetate. After 12-24 hours, the precipitate is filtered, well washed, and rinsed into a flask, so that the whole makes up to about 300-400 c.c. The liquid is then heated for an hour, on the water-bath, and sulphuretted hydrogen passed into it, while still warm, until the lead compounds are decomposed. After well shaking, the cold liquid is filtered through a folded filter, and the sulphuretted hydrogen washed out.

The lead sulphide on the filter retains the glycyrrhizic acid of the liquorice. It is washed with 150-200 c.c. alcohol (50%) into a flask, heated to boiling and filtered. The filtrate is evaporated to a few c.c., and a few drops of ammonia added, which turns

---

\* *Chem. Zeit.* 44. 781.

the pale yellow liquid brown-yellow; the latter is then evaporated to dryness, the residue dissolved in 2-3 c.c. water and filtered. The filtrate possesses the characteristic liquorice taste, and separates out a flocculent resinous mass (glycyrretin) on heating with a few drops of hydrochloric acid on the water-bath. The residue from a beer free from liquorice possesses no taste or a slightly bitter one, and gives at the most only a whitish turbidity.

F. H. H.

**THE DETERMINATION OF FREE SULPHURIC ACID IN VINEGAR\*.**—By B. KOHNSTEIN.—Dingl. polyt. Journ. [85] 256. 128.—The author proposes the following method similar to the one used by him and F. Simand for the determination of the acids in Tanners' liquids. 100 c.c. of the vinegar are shaken up with freshly ignited magnesia, until the liquid gives a neutral reaction, and filtered. 25—30 c.c. of the filtrate are evaporated to dryness in a platinum dish, and the residue ignited at not too high a temperature. This converts the magnesium acetate into carbonate, the magnesium sulphate remaining unaltered. The residue is now evaporated with water containing carbonic acid, digested with hot water and filtered. The precipitate is washed until free from sulphuric acid, the filtrate freed from calcium (if present) by precipitation with oxalic acid, the magnesia precipitated with sodium phosphate, and weighed as pyrophosphate. From the latter weight, the amount of free sulphuric acid can be easily calculated.

Vinegar sometimes contains a small quantity of magnesium salts, which might influence the results. To get over this difficulty, 100 c.c. of the vinegar are ignited, and the ash dissolved in hydrochloric acid. In this solution the magnesia is determined, after removing the calcium as pyrophosphate. The amount found is then subtracted from the first result.

F. H. H.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

**ON THE DETECTION OF CITRIC ACID.**—By G. MANN.—Zeitschrift f. Anal. Chem. 24., 2, p. 201.—This is a colour reaction, which is characteristic for citric acid, and serves to distinguish the latter from malic and tartaric acids.

About 1 grm. citric acid and 0.6—0.8 grm. thick glycerine are carefully melted together over a small flame until the mass becomes just solid. This glycerine compound is then brought into solution, by boiling with aqueous ammonia, and evaporated till faintly alkaline. If to this solution 1.3 drops of diluted fuming nitric acid (1 acid : 5 water), or a few drops of an ordinary 8—10 per cent. solution of peroxide of hydrogen be added, an intense green colour is produced, which, on warming, changes to blue.

F. H. H.

**EUCALYPTOL**, from *Eucalyptus globulus*, according to C. Jahns, has the formula,  $C_{10}H_{18}O$ , boils between 176—177°C., has the spec. grav. .923 at 16°C., is without optic action, and is identical with cajuputol, as it shows the same chemical and physical properties. He corroborates the observation of Faust and Homayer (1874), that the eucalyptol of Cloëz was a mixture, and shows that *Eucalyptolum purissimum* of commerce is merely rectified oil of eucalyptus.—*Arch. d. Pharm.*

W. H. D.

## MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ON THE DETERMINATION OF PHOSPHORIC ACID.—By C. GLASER, *Zeitschrift f. Anal. Chem.* 242, p. 178.—The following method has been used for some years by the author for the determination of phosphoric acid in manures. It is based upon the fact that phosphoric acid in the presence of calcium salts is precipitated directly by magnesia-mixture and ammonium citrate, provided sufficient sulphuric acid be present to convert all the calcium salts into sulphates, and not more ammonium citrate be used than is necessary to keep the calcium salts in alkaline solution.

The following re-agents are necessary :—

(1). *Magnesia Mixture*—140 grms. magnesium sulphate, 150 grms. ammonium sulphate, and 30 grms. ammonium chloride are dissolved in 350 c.c. ammonia (16 per cent.) and 1,650 c.c. water, and, after standing a few days, filtered. (2). *Citric Acid Solution* (50 per cent.) (3). *Ammoniacal Wash-Water*—250 c.c. ammonium hydrate (16 per cent.) made up to 1 litre with water. (4). *Dilute Sulphuric Acid*—100 c.c. sulphuric acid (66°) diluted to 1 litre. The solution containing the phosphoric acid is brought into a beaker, and ammonia added until a slight turbidity is produced. Sufficient citric acid solution is then added to dissolve the precipitate, care being taken that the clear solution remains slightly alkaline. The solution is now allowed to cool, and magnesia mixture dropped in while stirring; finally ammonium hydrate is added in excess. After six or eight hours standing, the solution is filtered, the precipitate washed with dilute ammonium hydrate, dissolved in dilute sulphuric acid, and the phosphoric acid precipitated in the filtrate by adding ammonium hydrate and a little magnesia mixture. As soon as the beautifully crystalline precipitate has settled (after about an hour) the liquid is filtered through an asbestos filter with the help of a filter-pump, washed, ignited, and weighed.

This method requires less time and is cheaper and easier than the molybdenum method; it is also absolutely accurate, as the following analyses show :—

	Molybdenum.	Magnesia Mixture.
South Carolina Rock.. ..	27.62 per cent.	27.71 per cent.
Navassa Guano .. ..	23.95 „	23.84 „
Mona Island, Guano .. ..	27.84 „	27.79 „

F. H. H.

ON KJELDAHL'S METHOD FOR THE DETERMINATION OF NITROGEN.—By E. BOSSHARD, *Zeitschrift f. Anal. Chemie.* 24, 2, p. 199.—The author has submitted Kjeldahl's method to a strict examination, and has obtained excellent results with it, as the analyses accompanying his paper show.

He points out, however, one source of error :—Kjeldahl recommends the addition of zinc to prevent bumping during the distillation of the ammoniacal liquid. If this be done, care must be taken that the sodium hydrate used be free from nitrates, which get reduced and form ammonia. Farther, it is advisable to use only a small excess of sodium hydrate and as little zinc as possible, for a rapid evolution of gas is apt to carry over some of the alkaline liquid into the distillate.

F. H. H.

[As some of our readers may have missed seeing the method referred to in the above abstract, the following are the details of the process as originally given by the author.

The substance, whether solid or liquid—and, if solid, it need not even be powdered—is weighed or measured into a small flask, in which the subsequent treatment is executed. In the case of substances containing about 10 per cent of nitrogen, about 0.1 to 0.2 grm. are sufficient; if less is present, up to one grm. may be taken. Ten c. c. of strong sulphuric acid, known to be free from ammonia, are now introduced by means of a pipette, the flask, in an inclined position, placed upon wire gauze over a gas flame, and the contents heated for one or two hours to near the boiling point of the acid, which may be known from the occasional slight bumping. If the substances retain their nitrogen very tenaciously, such as quinine, morphine, etc., a little fuming sulphuric or anhydrous phosphoric acid may be added to the contents of the flask in the beginning. When the action of the acid is completed, which usually results in a solution of the solid, the flame is removed, and a fine spray of finely powdered permanganate of potassium, contained in a tube closed with a fine wire gauze and inserted in the neck of the flask, is made to rain down upon the acid in the flask until the latter assumes a green colour. The flask is then cooled, the contents diluted with water, 40 c.c. of solution of soda, sp. gr. 1.300 added, and the flask immediately connected with a well-cooled condenser, previously arranged, and provided with a receiver containing standard volumetric acid. If the original flask is too small, the contents may be transferred to another, the water being used in portions to wash the original flask carefully. To prevent bumping during the distillation, a few pieces of zinc may be placed in the flask. The estimation of the ammonia, by finding the amount of unsaturated acid, is a simple operation, and accomplished with great sharpness. It may be performed in several ways. The author prefers to add a mixture of iodide and iodate of potassium (which give up a quantity of free iodine equivalent to the free acid present), and so estimate the amount of free iodine by volumetric solution of hypophite of sodium.—ED. ANALYST.]

---

ON THE ANALYSIS OF TUNGSTEN STEEL (WOLFRAMSTAHL) AND TUNGSTEN IRON (WOLFRAMEISEN).—BY L. SCHNEIDER AND F. LIPP, *Zeitsch. f. Anal. Chem.* 24. 2, p. 292. *Berg. u. Hüttenmann, Jahrbuch*, 1884, p. 34.—To the substance under water is added gradually twice the quantity of bromine, and the whole gently warmed. Nitric acid is then added, and the mixture evaporated to dryness. After repeating this operation, the substance is dissolved in dilute nitric acid, tungstic acid and silica remaining insoluble. To separate from these the still undissolved oxide of iron, the residue is melted with sodium hydrate, lixiviated with water, the solution evaporated to dryness with nitric acid, and the mixture of tungstic acid and silica weighed. To separate the two acids, the powder is melted with potassium bisulphate (5 of mixture : 1KHSO<sub>4</sub>) until a clear fusion is obtained. The cold fusion is treated with a dilute solution of ammonium carbonate, which dissolves the tungstic acid, the insoluble silica remaining behind. The weight of the latter, subtracted from the total weight, gives the tungstic acid.

To determine the carbon in tungsten-steel, the substance is treated with cupric chloride, and the residue combusted in a stream of oxygen. Tungsten-iron is not attacked by cupric chloride, and the carbon must, therefore, be determined by direct combustion in oxygen.

F. H. H.

---

ON TWO NEW INDICATORS FOR USE IN THE VOLUMETRIC DETERMINATION OF CAUSTIC ALKALIES IN THE PRESENCE OF CARBONATES.—BY R. ENGEL AND J. VILLE.—*Comptes rend.* [1885]

100. 1074.—The authors use sulphindigotic acid or Poirrier's soluble blue (c.4.B.), both of which give much more delicate reactions than either phenacetoline or phenolphthalein. The sulphindigotic acid is prepared by neutralising a solution of indigo in fuming sulphuric acid with calcium carbonate, adding ten times the volume of water and filtering. Its colour is not affected by alkaline carbonates, whereas caustic potash and soda change it to yellow. If, for example, caustic potash is to be titrated in the presence of potassium carbonate, two or three drops of the indicator are added to the mixture. The liquid assumes a yellow colour, which as soon as the potash has been neutralised, turns blue. Between the yellow and the blue an intermediate green is produced; but by placing the glass on a white ground, it is easy to observe that every drop of the liquid produces a blue zone. The titration is complete when the blue zone no longer makes its appearance, and the liquid has assumed a blue tint. The soluble blue (Poirrier's c.4.B.) in aqueous solution is a still more delicate reagent. The solution is turned red by caustic alkalis and not changed by carbonates. One to two drops of the indicator colour a mixture of the two pink. If now standard sulphuric acid be run in, a blue zone is produced, which disappears when the alkali is neutralised. F. H. H.

---

ON THE TITRATION OF IRON ORES.\*—BY WALTHER HEMPEL.—D. Chem. Ges. Ber. [85] 18. 1130.—The author recommends the fusion of the ores with a mixture of calcium and sodium carbonates. 0.3 grm. of the finely powdered ore are intimately mixed with 0.4 grm. pure sodium carbonates and about 2 grms. of precipitated calcium carbonate (free from iron), and heated in a platinum crucible for an hour in an oxydation-furnace.

The mass is heated with concentrated hydrochloric acid, which dissolves out the iron in a few minutes. The liquid is boiled for about five minutes longer in order to reduce the higher oxides of manganese which may be present, diluted and titrated with stannous chloride in the ordinary way.

The method is easy and gives good results.

F. H. H.

---

THE SEPARATION OF COPPER AND CADMIUM.—BY A. BEHAL.—Journ. Pharm. Chim. [85] 5 Ser. 11, 553.—A strongly alkaline solution of sodium tartrate produces no precipitate in the solution of a mixture of copper and cadmium salts. A slight turbidity is sometimes produced, and this vanishes on stirring. If the solution now be boiled, the whole of the cadmium is precipitated as oxide, the copper remaining in solution. Ammonium salts retard the precipitation, and must therefore be destroyed by boiling with a slight excess of alkali. F. H. H.

---

A NEW METHOD FOR THE DETERMINATION OF PHOSPHORIC ACID BY PRECIPITATION, WITH MOLYBDENUM.—BY MEINEKE.—Repert der Anal. Chem. [85] 10. 153.—This method is founded on the fact that the yellow precipitate, produced by the action of ammonium molybdate on phosphoric acid, when heated to 400-500° C, loses water and

---

\*Chem. Zeit., 44. 779.

ammonia, and undergoes partial decomposition, giving molybdic-molybdenum oxide—a dark nearly black residue. This compound is very stable, only slightly hygroscopic, and admits of accurate weighing. The author's researches show that it contains

4·018 %  $P_2O_5$

65·195 % Mo

30·787 % O

corresponding to the formula  $P_2O_5 \cdot 4 (Mo_6O_{17})$ .

The method is carried out in the following manner:—The solution of the phosphate, containing nitric acid and about 20—25 % ammonium nitrate, is precipitated at 50°-60° with molybdenum solution, and allowed, *without further warming*, to stand for a few hours. The precipitate is then collected on a filter, washed with a 20 % ammonium nitrate solution (slightly acid with nitric acid) till ten drops give no precipitate with sulphuretted hydrogen, and then a few times with water, or, in order to facilitate the drying, with small quantities of cold water, alcohol and ether.

The dried precipitate is removed from the filter, and brought into a flat platinum dish. The filter paper is ignited in a separate platinum dish, the ash added to the precipitate, and the covered dish heated to a temperature sufficient to cause a slow decomposition of the precipitate, which shows itself in a blackening of the latter. With larger quantities of precipitate, the author removes the dish from the flame, and crushes the mass with a glass rod in order to expose the undecomposed yellow portions to the higher temperature. After about  $\frac{1}{2}$  hour the precipitate is generally black throughout, and can, after cooling in the dessicator, be weighed. It contains 4·018 %  $P_2O_5$ . We select the following from the authors results:—(1.) 50 c.c. of a solution of sodium phosphate evaporated to dryness gave 0·10921 grm. (mean)  $P_2O_5$ . (2.) 50 c.c. same sol. precipitated with magnesia mixture, 0·10959 grm. (mean)  $P_2O_5$ . (3.) 50 c.c. + 100 c.c.  $HNO_3$  + 100 c.c.  $NH_3$  + 100 c.c.  $H_2O$ , cooled to 55°C precipitated with 130 c.c.  $MoO_3$  gave 0·10930 grm. (mean)  $P_2O_5$ . A + error of 0·00009 grm.

F. H. H.

---

ON THE QUANTITATIVE DETERMINATION OF URIC ACID.\*—By E. LUDWIG.—Wien. med. Jahrb. 1884.—To a measured volume of the urine is added simultaneously, magnesia mixture and ammoniacal silver solution—the uric acid precipitates as magnesium silver salt, and the phosphoric acid as ammonium-magnesium phosphate. The precipitate is collected in the filter, exhausted with the Bunsen filter-pump, and washed with water containing ammonia. The precipitate is then washed with a solution of potassium sulphide, soluble potassium urate being produced. After filtering from the silver sulphide and ammonium-magnesium phosphate the clear filtrate is acidulated and evaporated to a small volume, the uric acid separating out crystalline, the latter is washed on to a filter, dried, freed from sulphur with bisulphide of carbon and after drying weighed.

F. H. H.

---

\* Chem. Zeit. 40. 716.

ON THE ANALYSIS OF VULCANIZED CAOUTCHOUC, WITH SPECIAL REFERENCE TO THE DETECTION OF THE SULPHUR.—By B. UNGER, *Zeitschrift f. Anal. Chem.* 24. 2. p. 168.—The author states that all continental manufacturers follow the English method, and use the English golden sulphide of antimony. The latter is obtained by the decomposition of a mixture of calcium sulph-antimonate, calcium hyposulphite, and calcium polysulphide. The product of this reaction is calcium sulphate, antimony pentasulphide, and free sulphur. In the vulcanization (*i.e.*, the transformation to an organic compound at a high temperature), only the two latter compounds can have any influence.

To find out, therefore, how much available sulphur the sample contained during its preparation, it is necessary to know the amount of sulphur, antimony, and calcium present.

1. *Determination of the Sulphur.*—0.5–0.55 grms. of the sample are weighed out, and cut into about 100 equal pieces. 12 grms. of cupric oxide are also weighed out, and intimately mixed with 2 grms. of sodium carbonate (of which the sulphur has been accurately estimated). A layer of this mixture is then placed in a porcelain crucible, containing a little cupric oxide, and about 8 pieces of the caoutchouc pressed carefully down into the mass, taking care that they do not touch the sides of the crucible. Over this another layer of the mixture, and about 10 pieces of the caoutchouc are added, this alternation being continued until all the caoutchouc is in the crucible. The remainder of the mixture is shaken on to the top, and pressed tightly down with a little pure cupric oxide. The crucible is then placed in a larger platinum one, and heated very gently over the flame, taking care that the temperature does not rise high enough to cause volatilisation of the sulphur. After about half an hour the temperature is raised to a good red heat, and the crucible then allowed to cool.

The fusion is dissolved in hydrochloric and nitric acids, and evaporated to dryness on the water-bath. The residue is stirred up with a good quantity of water and allowed to settle. The liquid is then filtered, the residue washed, and the filtrate made up to about 600 c.c. The liquid is then precipitated warm with barium chloride and allowed to stand overnight. The precipitate is finally filtered, washed, ignited gently, and weighed. This gives the sulphur.

For example:—

0.501 grm. caoutchouc treated with 12 grms. CuO and 2 grms. Na<sub>2</sub>CO<sub>3</sub> (containing S equal to 0.01 grm. BaSO<sub>4</sub>) gave 0.2445—0.01 grm. BaSO<sub>4</sub> (impure). This melted with 1.2 grm. Na<sub>2</sub>CO<sub>3</sub> (= 0.006 BaSO<sub>4</sub>) dissolved and precipitated with BaCl<sub>2</sub> gave 0.222—0.016 = 0.206 grms. BaSO<sub>4</sub> = 5.72% S.

2. *Determination of the Antimony.*—About 1.5 grm. of the sample are cut into about 50 pieces, and placed with 10 grms. crystallised sodium sulphide in a porcelain crucible. The mixture is at first heated gently to drive off the water, afterwards slowly to a dull red heat, the melted mass being now and then stirred with an iron wire.

As soon as a phosphorescence becomes visible, the mass is allowed to cool, dissolved in water, filtered from separated carbon, the filtrate warmed and saturated with hydrochloric acid. The next day the liquid is filtered, the mixture of sulphur and antimony sulphide being brought on to a double weighed filter, washed with



water, finally with alcohol, dried in the water-bath, allowed to stand an hour in the ordinary atmosphere, and the two filters weighed (the alteration in weight being assumed to be the same in both filters).

The mixture of antimony sulphide and sulphur is mixed with 1.5 gm. flowers of sulphur in a weighed porcelain crucible. The whole is then weighed, and the weight of the crucible subtracted. This crucible is then placed in a much larger one, upon the bottom of which 1 gm. flowers of sulphur has been placed, and covered with a platinum lid. The whole is then heated until the sulphur is completely burnt off. The residue in the smaller crucible then consists of  $Sb_2 S_3$ , and on its cover is a small sublimate of the same.

1.616 gm. caoutchouc treated with 10 grms.  $Na_2 S$  gave 0.5345 gm.  $Sb_2 S_3 + S$ ; 0.5235 gm. of this heated with  $S$  gave a residue of 0.151 gm.  $Sb_2 S_3$ —in all 0.1542 gm.  $Sb_2 S_3 = 6.813\%$  Sb.

(3). *Determination of the Calcium.*—About 2.5 grms. caoutchouc are burnt in a small porcelain crucible. A white ash is obtained, which still contains a good deal of antimony oxide. The edge of the crucible is then smeared with fat, half-filled with strong hydrochloric acid, and allowed to stand for 24 hours. The whole is now brought into a beaker, and the milky liquid warmed on the sand-bath; a heavy powder separates out. The liquid is saturated with ammonia, which precipitates the remainder of the antimony oxide, filtered, and precipitated hot with ammonium oxalate. The precipitate is washed, dried, and burnt to calcium carbonate.

2.495 grms. sample gave 0.019 gm.  $Ca CO_3 = 0.3046$  per cent. Ca.

Calculating the sulphur to the Sb. and Ca found, and taking the balance as free sulphur, we have:—

Sb <sub>2</sub> S <sub>3</sub> .. .. .	11.353
Ca So <sub>4</sub> 2H <sub>2</sub> O .. .. .	1.310
Free Sulphur.. .. .	.934
Caoutchouc (balance) .. .. .	86.603
	100.000

According to this calculation, for every 100 grms. pure caoutchouc, 15.74 grms. golden sulphide of antimony of the following composition were used in the preparation:—

83.50 Antimony pentasulphide.  
9.63 Gyps.  
6.87 free Sulphur.

100.00

F. H. H.

#### REVIEWS.

MANUAL OF CHEMISTRY. *A Guide to Lectures and Laboratory Work for Beginners in Chemistry. A Text-Book specially adapted for Students of Pharmacy and Medicine. By W. Simon, Ph.D., M.D., Maryland College of Pharmacy, Baltimore. London: Baillière, Tindall, and Cox.*

THIS is a somewhat difficult work for a British critic to review, because, although from the author's experience, it may, doubtless, be taken as quite sufficient for its purpose in

America, it is not exactly the class of book at present required by students of pharmacy in this country in several respects. Commencing with the usual chapters on chemical theories, extending to sixty-three pages, and including a description of the processes and instruments employed by chemists, it passes to the non-metals, and, thereafter, to the metals, without any definite arrangement of the former other than a conventional one, and the salts of the pharmacopœia are treated as customary under their respective metals. Then follows a chapter upon analytical chemistry, and here we meet with the really original part of the work in the shape of coloured slips, painted by hand, by the author's wife, illustrative of the precipitates produced. The eye for colour possessed by this lady is marvellously correct, and the resemblances to the substances represented are really excellent. Especially ingenious and wonderful are the representations of the successive play of colours produced in testing for alkaloids, exactly reproduced in the most dexterous and happiest manner. Following this we have chapters devoted to such articles of organic chemistry as are included in the pharmacopœia, and the whole winds up with a section on physiological chemistry. Having thus given well merited praise to the excellent illustrations, we think that the author will find it necessary to modify some portions of his work in the next edition. For example, we find (p. 25) the following description of the making of a thermometer:—

“This instrument may be easily constructed by filling with mercury a glass tube, having a bulb at the lower end, and placing it in boiling water. The point to which the mercury rises is marked B. P. (boiling point), and the tube sealed by fusion of the glass. It is then placed in melting ice,” &c.

It is to be feared that the author, if he tries, will find it necessary to fill and seal the tube, and thus get the vacuum *before* graduating or too great reliance could not be placed in the instruments he would produce. One more extract and we have done (taken this time from the organic portion), which has struck us as the perfection of beautiful simplicity, were it only practical.

“GENERAL MODE OF OBTAINING ALKALOIDS.—The disintegrated vegetable substance (bark, seeds, &c.), is extracted with acidified water, which dissolves the alkaloids. When the alkaloid is volatile, it is obtained from this solution by distillation, after having been liberated by an alkali. Non-volatile alkaloids are precipitated from the acid solution by the addition of an alkali, and the impure alkaloid thus obtained is purified by again dissolving in an acid and reprecipitating, or by dissolving in alcohol, and evaporating the solution.”

Experts in cinchona and opium valuation have reason to be thankful that the real extraction of alkaloids is not such a simple matter, or they would soon find themselves in the position of Othello. In conclusion, we fear this work will not, in its present condition, make its way in this country, where the ground it seeks to cover is already occupied by several old standing manuals of pharmaceutical chemistry, which, having passed through many issues, have had the inevitable defects of a first edition eliminated.

---

COMMERCIAL ORGANIC ANALYSIS. *A Treatise on the Properties, Proximate Analytical Examination, and Modes of assaying the various Organic Chemicals and Products employed in the Arts, Manufacture, Medicine, &c.* By Alfred H. Allen, F.I.C., F.C.S. Second Edition, Revised and Enlarged. London: J. and A. Churchill. Vol. I.

WHEN Mr. Allen's book originally appeared it was felt by all analysts that it supplied

a distinct want, although it was not, in all respects, quite free from errata, and the processes given were here and there somewhat open to adverse criticism, which was freely indulged in, especially in pharmaceutical circles. Although the edition has been rapidly exhausted, the author has yet had time to revise and consider, with the result that the present issue is a distinct advance in every respect on the former. The new work will be in three volumes, of which the first is now out, and includes the alcohols, ethers, carbohydrates, and vegetable acids. We reserve any detailed criticism until the whole work is before us, but from a hasty perusal of the present volume, we have formed the opinion that the advance is so marked that no analyst will be able to do without discarding his old edition in favour of the present one, as everything is brought down to the latest date, and the actual matter enormously increased.

### CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

TO THE EDITOR OF "THE ANALYST."

SIR.—As I was not present during the reading and discussion of my paper on "Reichert's Process of Examining Butter Fat," I shall be glad if you will allow me space to reply to the remarks made during the discussion, as printed on page 105 of the ANALYST.

Mr. Fox is reported to have said that Reichert's process appeared as elaborate as Hehner's, and more complicated than that introduced by Mr. Wanklyn and himself. Now that he has had the opportunity of seeing the description of the method in print, he will probably have found reason to modify his opinion respecting the elaborate nature of the process. The length of time occupied in estimating the soluble and insoluble acids of butter-fat by Hehner's method—a process which I have used with satisfaction for a number of years—depends much upon the details of the manipulation adopted, but I am not aware that it can, in any case, compare, in point of speed, with Reichert's process, by means of which an assay of butter-fat can be made in about 80 minutes. As to the process of Messrs. Wanklyn and Fox, I have yet to learn that it aims to be anything better than a qualitative test for the presence of butter-fat. Certainly the authors of the method do not appear to have laid stress on the value of its quantitative indications. Mr. Hehner's chief cause of complaint appears to be that I have not recommended the adoption of Reichert's process to the exclusion of other methods of examining butter-fat, which he calls, somewhat uncomplimentarily, "a characteristic want of confidence on my part;" but my contention is that, before giving a certificate of adulteration, an analyst should use all available means of checking the indications of a particular method; and, hence, I should, on principle, supplement the results of any method I have habitually employed for sorting butters by other tests which might furnish corroboration. Mr. Hehner's objection to Reichert's process, as an arbitrary method, has some plausibility; but an arbitrary method, by which fully 80 per cent. of a substance is determined, is very different from one by which only a small fraction is shown. Really the practical point is whether Reichert's process is capable of giving us information such as cannot be obtained with equal ease and certainty by either Bell's, Kettstorfer's, or Hehner's process. As a matter of fact, I believe it is capable of this.

Dr. Muter seems to think that the last word has been said upon butter analysis, and that the specific gravity should be relied on as a preliminary test. But he himself has shown that the indications thus obtained are fallacious, and that cotton-seed stearin, among other possible butter-substitutes, would remain unrecognised by this test. Kettstorfer's test again fails in the case of cocoa-nut and palm-nut oils, and even Hehner's process gives a less sharp distinction between these fats and butter-fat than is shown by Reichert's process. As I stated in my paper, every probable sophistication of butter-fat, except, perhaps, added butyrin, can be recognised by Reichert's process. Can the same be said for any other? If not, Reichert's method is a valuable addition to our tools. Those who use it for sorting butters will have the advantage of a rapidly-worked process, the indications of which can be supplemented in all doubtful cases, and they will be led to detect sophistication in cases where analysts, employing other methods, would not even suspect it.

In conclusion, I can assure Mr. Hehner that Reichert's process is something better than a "rough and ready method," and, even in his hands, will be found a useful adjunct, and, in many cases, a convenient substitute for the method of Angell and Hehner.

Yours, &c.,

ALFRED H. ALLEN.

Surrey Street, Sheffield, June 10th, 1885.

P.S.—I have just received a letter from Professor H. B. Cornwall, who writes:—"I am glad to learn that you use Reichert's test. I depend upon it for my own butter analyses, and our New Jersey Committee of Public Analysts are now engaged in a systematic examination of genuine butters by it with a view to ascertaining its reliability at all seasons, and under all conditions as to breed of cow, food, time of calving, &c. Thus far (four months successive trial) it has stood perfectly all tests."

A. H. A.

# THE ANALYST.

AUGUST, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
(a) REMARKS ON SEWAGE-FARM MILK AND BUTTER, By DR. HILL, PRESIDENT..	135	(a) DETERMINATION OF THE VALUE OF PEPSIN, O. SCHLICKUM ..	147
(b) ON ADMIXTURES OF MALT-EXTRACT WITH CERTAIN FIXED OILS, By E. GODWIN CLAYTON, F.C.S. . . . .	140	(b) SPECTROSCOPIC BEHAVIOUR OF FATTY OILS, DOUMER ..	148
PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS:—		(c) SAPONIN, M. C. SCHIAPARELLI ..	148
DISCUSSION ON WATER ANALYSES MADE BY DR. WALLER .. . . . .	143	(d) QUANTITATIVE DETERMINATION OF CINCHONIDINE IN QUININE SULPHATE, DE VRIJ ..	148
ORIGINAL ARTICLE:—		(e) REACTIONS OF ALKALOIDS AND GLUCOSIDES, SERENA ..	149
ON THE PROCESSES AND STANDARDS OF FOOD ANALYSIS IN USE AT THE MUNICIPAL LABORATORY OF THE CITY OF PARIS, By DR. MUTER .. . . . .	143	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—		(a) GRAVIMETRIC DETERMINATION OF MANGANESE, H. VON JUPITNER ..	149
(a) DETECTION OF ALKANET IN WINE, H. HERZ .. . . . .	146	(b) ESTIMATION OF UREA, A. B. LYONS ..	150
(b) WATER IN POTATOE STARCH, SAARE ..	146	(c) ESTIMATION OF UREA, H. J. HAMBURGER	150
(c) DETECTION OF WATER IN MILK, UFFLEMAN .. . . . .	146	(d) DETERMINATION OF ALUMINA AND IRON IN THE PRESENCE OF PHOSPHATES, M. KRETZSCHMAR ..	151
(d) ARTIFICIAL HONEY, H. HAGER..	147	(e) SEPARATION OF ZINC, W. HAUPE ..	152
(e) DETERMINATION OF ORGANIC MATTER IN WATER, M. PETIT .. . . . .	147	(f) DIMETHYLAMIDOAZOBENZOL AS AN INDICATOR, FISCHER..	152
		(g) NEW EXTRACTION APPARATUS, T. WEYL	153
		(h) VALUATION OF OZOKERIT, B. LACH ..	153
		(j) DIPHENYLAMINE AS A REAGENT FOR CHLORINE, H. HAGER ..	154
		(k) QUANTITATIVE ANALYSIS BY ELECTROLYSIS, A. CLASSEN AND R. LUDWIG	154
		BOOKS RECEIVED AND NOTICES TO CORRESPONDENTS .. . . . .	154

### REMARKS ON SEWAGE-FARM MILK AND BUTTER.

By ALFRED HILL, M.D., F.I.C., F.C.S.

*President of the Society of Public Analysts.*

*(Read at the Meeting, 10th June, 1885.)*

The products in question have frequently been the objects of suspicion and attack by persons who knew, or thought they knew, something about the subject.

With regard to the milk, it has been said to be poor and watery, to possess, when 24 hours old, a rancid odour, and to produce typhoid fever.

The butter made from the milk of cows fed on a small proportion of sewage-grass has also been stated to be so offensive, that it could not be borne upon the table. Such statements as these seeming to me *prima facie* incredible, I determined, as far as Birmingham is concerned, to test their accuracy; for it seemed, and still seems to me a matter of the greatest importance that the truth upon this question should be known, especially at a time when the disposal of the sewage of towns upon land is on

the increase, and considering that it is likely to be more and more adopted to the exclusion of artificial methods, for the reason that it is the most satisfactory of all systems.

I obtained, therefore, samples of milk direct from the Birmingham Sewage-Farm, and submitted them to both organoleptic examination and chemical analysis, the result being that the milk proved to be of as good a quality as, if not better than, ordinary milk, and that its *keeping* properties were in no way inferior, although fresh sewage-grown grass formed a large part of the cows' food.

In order to test the quality of the butter made from it, I requested the wife of the farm manager, who thoroughly understands butter making (although no butter is ordinarily made on the sewage-farm) to make a churning for me, which she was kind enough to do.

The resulting butter was excellent in quality, and retained its sweetness and other properties as well as other fresh butter, although the weather at the time was excessively hot; so that the conditions of the experiment were as unfavourable as possible.

Subsequently, I had 48 cows at the sewage-farm milked, in the presence of one of our Assistant Inspectors, and I analysed the samples by the ordinary (Bell's) method. *The results are given below in Table A.*

As, however, the samples above were yielded by cows at a time when they were eating certain sewage-grown products, but were not eating sewage-grown grass, I have thought it desirable to submit to analysis another series of milks from cows of whose fodder sewage-grown grass formed a considerable part, in order to see whether the constitution of the milk became thereby changed. *The results are given below in Table B.*

The ages of the cows vary from three to nine years. The time since calving ranged from three weeks to eleven months. Altogether there are 144 milking cows on the farm, and they yield 400 gallons of milk daily, which gives an average to each cow per diem of a little over 2 $\frac{3}{4}$  gallons. The breed of cows is the "Cross Short-horn,"

Besides sewage-grown grass, part long and part cut, the food of the cows includes sewage-grown cabbage, ensilage, brewers' grains, some cake, and a little mixed flour, which latter is given with the cut grass.

Some years ago, viz, in 1872, 1875, 1876, I examined the milks of a number of cows in different dairies, as well as some purchased by the Inspector, the results of which are recorded at page 44 of the first volume of THE ANALYST. None of these cows were fed on sewage-grown products, and it is interesting to observe how little are the differences in the three series of analyses, as seen below:—

	Average of Milks from 46 Cows in ordinary Dairies in 1872, 5, & 6.		Average of 48 samples of Sewage-Farm Milk, 1882.		Average of 48 samples of Sewage-Farm Milk, 1885.	
Fat	..	3·22	..	3·69	..	3·95
S. N. F.	..	9·74	..	9·45	..	9·14
Total Solids	..	12·98	..	13·11	..	13·10
Ash	..	·73	..	·77	..	·77

An inspection of the tables will show that the average quality of the milk is decidedly high, that the Specific Gravity is indicative of good quality, and varies very equally with the fluctuation of chemical composition, that in nearly all the few samples in which the fat is low the solids-not-fat are higher than usual, and *vice versa*, but that in many instances the quantities of both fat and solids-not-fat, *i.e.*, total solids, are high. Thus the average figures of the different results are as follows:—

Specific gravity	.. ..	1031.9	Max.	1036.0	Min.	1027.5
Percentage measures of cream		12.1	Max.	20.5	Min.	5.0
Fat	.. ..	3.69	Max.	7.59	Min.	2.1
Solids-not-Fat	.. ..	9.45	Max.	10.64	Min.	8.40
Total Solids	.. ..	13.11	Max.	17.01	Min.	11.38
Ash	.. ..	0.77	Max.	0.84	Min.	0.70

From No. 1 to No. 40, inclusive, the food of the cows consisted of cut hay, turnips, mangel-roots, with some brewer's grains and flour.

From No. 40 to 45, inclusive, the food was cut hay, rabi roots, some linseed and a little bean flour.

From No. 46 to No. 48 it was hay, turnips, mangels, with brewers' grains and a little flour.

Everything was grown upon the Sewage-Farm except the linseed cake, flour, and brewers' grains.

These facts clearly disprove the allegation that sewage-farm milk is of poor quality, and that it acquires a rancid odour in 24 hours, and, even if it did, this would be no proof of the other contention that its consumption produces typhoid fever. There are sewage-farms in considerable number in the country, notably at Edinburgh, where the Craigtintny meadows, irrigated with sewage, have been used for growing grass for feeding cattle, for two hundred years, and where, as I can testify from my own observation, the sewage is applied in excessive quantities; at Croydon, Warwick, Rugby, Merthyr Tydfil, and other places, but in no case has it ever been proved that disease was propagated by the milk produced on such farms, on account of its origin.

Such an assumption appears to be about on a par with another made some years ago, on the strength of a suggestion by Dr. Cobbold, that sewage-grown grass was calculated to introduce parasites into the system of the cow, and to prove injurious to it and to the consumers of its milk and flesh.

In order to test this assumption a special and crucial experiment was made; a cow was reared and fattened exclusively on the agricultural products of sewage irrigation at Breton's farm, under the superintendence of Mr. W. Hope, V.C. On being killed and thoroughly examined by Dr. Corfield it was found "that the animal in question was entirely free from parasites of any kind."

The practical verdict of the retail vendors of milk in Birmingham confirms the analytical results, for that produced on our sewage-farm always commands a higher price by a half-penny per gallon than milk produced by private farmers and cow-keepers.

TABLE A.  
Milks from the Sewage Farm at Saltley (Birmingham).

No. of Sample.	Date. 1882.	Sp. Gr.	Percentage Volumes of Cream.	T. S. M.	S. N. F.	Fat.	Ash.
1	Jan. 23rd	1033·5	15·0	13·84	9·86	3·96	·84
2	"	1030·5	16·0	14·55	9·53	5·07	·77
3	"	1032·0	7·0	12·25	9·21	3·00	·75
4	"	1027·5	7·5	15·45	8·40	7·02	·71
5	"	1033·0	11·0	11·87	9·58	2·37	·78
6	"	1029·0	16·0	17·01	9·42	7·59	·78
7	"	1031·5	13·0	13·66	9·54	4·11	·82
8	"	1034·0	13·0	13·50	10·01	3·57	·77
9	Jan. 25th.	1032·0	14·0	13·58	9·68	3·98	·82
10	"	1030·0	12·5	11·58	8·63	2·98	·80
11	"	1031·0	13·0	12·93	9·03	3·97	·70
12	"	1033·5	11·0	13·45	9·93	3·57	·80
13	"	1034·5	10·5	13·39	10·11	3·28	·81
14	"	1033·0	12·0	13·50	9·59	3·94	·76
15	"	1032·5	7·5	13·19	9·34	3·89	·76
16	"	1032·0	9·0	13·38	9·37	3·94	·74
17	Jan. 30th	1028·0	15·5	12·72	8·96	3·83	·71
18	"	1032·0	14·5	13·35	9·65	3·71	·73
19	"	1033·0	16·0	13·82	9·71	4·15	·77
20	"	1031·0	14·0	13·09	9·16	3·89	·78
21	"	1033·0	18·0	13·38	9·50	3·89	·76
22	"	1032·0	12·0	13·22	9·50	3·80	·81
23	"	1032·5	6·0	12·34	9·52	2·88	·74
24	"	1030·0	11·5	12·78	9·06	3·74	·77
25	Feb. 1st	1030·0	15·0	11·87	8·87	3·07	·70
26	"	1031·0	11·0	11·52	8·76	2·83	·72
27	"	1032·0	14·0	12·65	9·42	3·32	·72
28	"	1036·0	12·5	12·80	10·24	2·53	·78
29	"	1034·0	13·0	11·97	9·82	2·10	·76
30	"	1033·0	9·0	12·16	9·87	2·37	·84
31	"	1031·0	13·0	13·15	9·14	4·06	·75
32	"	1034·0	8·5	12·13	9·94	2·17	·80
33	Feb. 6th	1030·5	9·5	11·93	8·53	3·42	·81
34	"	1032·0	15·0	12·21	9·17	3·12	·81
35	"	1031·5	20·5	13·71	9·17	4·53	·81
36	"	1031·5	5·0	13·46	9·32	4·13	·75
37	"	1030·5	15·5	12·61	9·34	3·33	·76
38	"	1031·0	8·0	11·38	9·00	2·44	·73
39	"	1030·5	10·5	14·17	9·73	4·49	·79
40	"	1033·5	9·0	14·52	10·64	3·92	·81
41	Feb. 8th	1029·0	12·0	11·76	8·49	3·36	·72
42	"	1032·5	10·0	13·18	9·71	3·44	·80
43	"	1031·0	12·5	12·58	9·27	3·30	·76
44	"	1032·0	16·5	13·36	9·60	3·82	·80
45	"	1031·5	15·0	13·38	9·34	4·12	·77
46	"	1033·5	8·5	12·46	10·17	2·30	·82
47	"	1034·0	10·5	14·74	10·04	4·78	·83
48	"	1033·0	13·5	13·70	9·56	4·19	·80
	Average -	1031·9	12·1	13·11	9·45	3·69	·77

TABLE B.  
Milks from the Sewage Farm at Saltley (Birmingham).

No. of Sample.	Date. 1885.	Sp. Gr.	Percentage Volumes of Cream.	T. S. M.	S. N. F.	Fat.	Ash.
49	May 27th	1031·0	12·0	12·57	9·51	3·11	·78
50	"	1030·5	7·0	13·38	9·34	4·07	·79
51	"	1032·0	17·0	12·59	9·51	3·11	·79
52	"	1030·0	12·0	12·87	9·26	3·65	·78
53.	"	1031·5	8·0	12·28	9·43	2·92	·76
54	"	1032·0	9·0	13·68	9·55	4·19	·81
55	"	1031·5	11·0	12·14	9·11	3·07	·75
56	"	1032·0	11·0	13·32	9·70	3·65	·79
57	May 29th	1029·0	36·0	13·57	8·90	4·73	·78
58	"	1031·5	11·0	13·36	9·39	4·02	·79
59	"	1032·0	11·0	12·02	9·30	2·79	·81
60	"	1033·0	8·0	11·38	9·70	1·74	·75
61	"	1030·5	10·0	12·24	9·02	3·28	·77
62	"	1032·0	11·0	12·75	9·95	2·86	·80
63	"	1031·5	14·0	13·63	9·99	3·67	·79
64	"	1029·5	13·0	12·64	8·88	3·79	·75
65	May 30th	1029·5	13·0	12·63	8·96	3·62	·78
66	"	1031·5	12·0	12·34	9·28	3·01	·77
67	"	1031·0	9·0	12·50	9·38	3·07	·77
68	"	1031·5	10·0	12·80	9·50	3·21	·79
69	"	1031·0	12·0	11·91	9·01	2·98	·74
70	"	1031·5	14·0	13·29	9·46	2·80	·81
71	"	1030·5	16·0	12·78	9·01	3·71	·80
72	"	1031·0	12·0	12·81	9·28	3·48	·79
73	June 3rd	1029·5	13·0	12·84	9·10	3·80	·74
74	"	1031·5	12·0	13·81	9·70	4·01	·80
75	"	1031·0	9·0	14·06	9·65	4·36	·81
76	"	1031·5	10·0	14·66	9·68	4·90	·79
77	"	1031·0	12·0	13·12	9·30	3·80	·77
78	"	1031·5	14·0	13·48	9·45	3·98	·77
79	"	1030·5	16·0	12·96	9·28	3·59	·78
80	"	1031·0	12·0	13·32	9·51	3·74	·79
81	June 4th	1030·5	10·0	13·58	9·37	4·15	·79
82	"	1025·0	15·0	14·94	8·10	6·76	·77
83	"	1024·0	18·0	16·28	8·16	8·10	·77
84	"	1029·0	10·0	12·54	8·95	3·54	·79
85	"	1028·0	9·0	12·53	8·67	3·80	·75
86	"	1025·5	12·0	14·15	8·29	5·88	·76
87	"	1025·0	15·0	13·61	8·18	5·38	·79
88	"	1027·5	16·0	14·79	8·71	6·00	·79
89	June 5th	Curdled	Curdled	12·49	9·01	3·42	·75
90	"	"	"	14·91	8·97	6·01	·73
91	"	"	"	13·75	9·11	4·56	·75
92	"	1027·0	11·0	11·55	8·11	3·40	·72
93	"	Curdled	Curdled	11·52	8·29	3·20	·71
94	"	1031·0	24·0	12·95	8·91	3·99	·78
95	"	1030·0	12·0	12·88	9·00	3·81	·76
96	"	Curdled	Curdled	12·78	8·91	3·83	·76
	Average - -	1030·2	12·8	13·10	9·14	3·95	·77



## ON ADMIXTURES OF MALT-EXTRACT WITH CERTAIN FIXED OILS.

By E. GODWIN CLAYTON, F.C.S.

*(Read at the Meeting, June 10th, 1885.)*

Extract of Malt, which at the present time is manufactured and sold in large quantities, is prepared by digesting crushed barley-malt in water for some hours, and concentrating the strained infusion *in vacuo* until a viscid extract is obtained, possessing a consistency greater than that of honey, and containing not more than about 25 per cent. of water. The extract so prepared is sub-translucent, of a yellowish-brown colour, and so tenacious that it only escapes very slowly indeed from an inverted wide-mouthed vessel. It is very sweet, and has also the taste and odour characteristic of malt, but greatly intensified.

Less concentrated forms of this article are also manufactured, but on the present occasion I wish to speak only of the kind of malt-extract just described.

A good malt-extract should be rich in active diastase, while it must contain considerable proportions of albuminoids, maltose, dextrine, and phosphates; it should, moreover, be free from alcohol. According to a published analysis by Dr. Geissler, of Dresden, one of the best malt-extracts now in the market possesses the following percentage composition:—

Water .. .. .	24.3
Maltose .. .. .	36.5
Dextrine and other extractive matter .. .. .	30.5
Albuminoids (containing 1.05 per cent. of N) .. .. .	7.4
Mineral matter (containing 0.56 per cent. of P <sub>2</sub> O <sub>5</sub> ) .. .. .	1.3
	100.0

Inasmuch as diastase, when heated in aqueous solution to a temperature above 68° Centigrade, loses in great part its power of converting starch into dextrine and maltose, and is entirely destroyed at 100° Centigrade, precautions must be taken, throughout the whole process of manufacturing extract of malt, that the first-named temperature shall not be exceeded; indeed, in order to obtain a really good product, lower temperatures should be employed. I am informed that the malt-extract "Kepler," with which my experiments have been made, is concentrated in vacuum-pans at temperatures ranging between 32° and 38° Centigrade.

Not only is malt-extract of considerable nutritive value, by reason of the proteids, carbohydrates, and phosphates present, but, by diastatic action, it greatly assists in the assimilation of starchy foods. It is also said to have been found useful as a remedy for chest affections, and in cases of indigestion and general debility.

A knowledge of these facts has naturally led to the manufacture of various preparations, consisting of admixtures of malt-extract with certain drugs and remedial agents; as, for instance, with citrate of iron and quinine, cod liver oil, hypophosphite of calcium, &c. Among these preparations, *Oleum Morrhuæ cum Extractum Malti* (Kepler), or, in other words, Cod Liver Oil with Kepler Malt-Extract, holds a prominent place. Quite recently, the manufacturers of this article requested me to examine samples of it, and to express an opinion as to the real nature of the state of admixture of the ingredients.

The question to be answered was as follows:—*Is the preparation an emulsion, or would it be more correct to call it a solution? That is to say, are the particles of oil merely held in suspension throughout the mass of the extract of malt, or are the ingredients, in the proportions used, truly miscible with one another?*

It is but fair to state that, as regards the conclusion arrived at, I can make no claim to originality. The observations about to be detailed were merely made in the course of an investigation carried out for Messrs. Burroughs, Wellcome and Co. (the manufacturers of the Kepler Malt-Extract, already referred to), who, although desirous of having an independent expression of opinion, had already, with their chemical manager, Dr. O. Witté, observed how intimately associated were the two ingredients of the *Oleum Morrhuæ cum Extractum Malti*. It was Dr. Witté himself, I believe, who first noticed this fact.

My observations have, however, been somewhat extended, and the general results appeared of sufficient interest to be embodied in a short paper, which I now venture to bring before the Society of Public Analysts, with—I may mention—the full knowledge and consent of the gentlemen alluded to.

The following facts appeared worthy of attention, in connexion with this enquiry:—

1. Viewed with the naked eye, the mixture appears completely homogeneous, the most narrow examination failing to detect the presence of oil-globules. Oily emulsions are usually far from possessing a homogeneity of appearance, and in them suspended oil-globules can, as a rule, be discerned without difficulty.

2. The preparation possesses a translucency approaching to transparency, while thin layers are almost perfectly transparent, with only a faint opalescence. All emulsions which I have hitherto met with have been quite opaque, and more or less milky in appearance.

3. The components of the mixture do not exhibit the smallest tendency to separate from one another, even after long-standing. The opposite is the case with many emulsions, a layer or globules of the lighter constituents rising to the surface of the heavier.

4. The preparation is, in general, indistinguishable under the microscope from the simple extract of malt, not the faintest indications of oil-globules being visible. In some samples, however, the outlines of numerous minute and immobile round bodies *can* be discerned; but so intimately associated are these spherules with the menstruum surrounding them, and so faint and indistinct are the indications of their presence, that their figures can only be made out with considerable difficulty. No emulsion with which I am acquainted presents a similar appearance under the microscope.

If, now, a very small quantity of water be added to the drop on the slide, and the latter be again viewed under the microscope, a remarkable change is observed. The field is now seen to be crowded with separated oil-globules, moving about freely in all directions, possessing sharply-defined and strongly-marked outlines, high refractive power, and all the distinctive characters of free spherules of oil. The drop *now* has all the appearance, under the microscope, of an oily emulsion; and it is no longer

transparent, but is milky, in consequence of the presence of innumerable precipitated oil-globules.

When a very little water is allowed to flow under the thin square of glass, covering a drop of the malt-extract and oil placed upon a fresh slide, capillary attraction causes the drop to become quickly surrounded by a ring of water. The junction between the two liquids is almost instantaneously marked by a turbid band, visible to the unaided eye, and seen, when magnified, to be chiefly made up of separated and freely-moving oil-globules; while the centre of the drop remains, as before, nearly transparent, and devoid of globules. In this way, the remarkable contrast between the microscopical appearance of the preparation alone, and the same after the addition of water, can be observed upon one and the same field.

5. If this contrast is well-marked under the microscope, it is no less noticeable without the aid of that instrument. For, when mixed with a little water, the translucent liquid becomes opaque, milky in appearance, and, but for the fact that the globules of oil are smaller, resembles in all respects an ordinary oily emulsion.

6. A similar separation or precipitation takes place on the addition of ether or alcohol; the smaller globules of oil, however, soon coalesce into spheres and patches of comparatively large size.

7. Attempts to form similarly intimate admixtures of cod liver oil with various liquids, other than malt-extract, such as cane-sugar syrup, molasses, and concentrated dextrine solution, entirely failed; in one case only, namely, in that of the syrup, could I succeed in producing even an emulsion. In the other cases, complete separation of the oil, in a layer at the surface, took place in a very short time after the liquids had, to all appearance, been thoroughly incorporated with one another. The mixture of syrup and cane-sugar, however, constituted a true emulsion, in which numerous globules of oil were plainly visible.

These observations and results render it impossible for me to come to any other conclusion than that the mixture of cod liver oil and malt-extract is *not* an emulsion, in the ordinarily accepted sense of that word, but that its constituents are in a condition of more intimate admixture, namely, that of solution.

It is clear that the oil is in a different physical condition, *before* the addition of water, from that in which it is seen to be, *after* such addition. What can the former condition be but one of solution, seeing that the oil is instantaneously thrown down as a visible precipitate, precisely as it is separated, on the addition of water, from solution in ether, benzene, and other similar menstrua?

It appears to be established, beyond all doubt, that cod liver oil and extract of malt are truly miscible in certain proportions, or, to make the same statement in another form, that the extract is a solvent for the oil. The proportion of oil present varies somewhat, but, in one of the most satisfactory samples submitted to me, it amounted, I am told, to about 33 per cent. It is probable that those samples, in which faint indications of oil spherules were visible under the microscope before the addition of water, had not been perfectly mixed, or that they contained a quantity of oil rather greater

than malt-extract is capable of taking up. A limit must, of course, be reached in this as in other cases of solution.

The results obtained led to further experiments, made with the view of ascertaining whether other fixed oils are miscible with malt-extract. It is sufficient to say, shortly, that by thoroughly well mixing the respective oils with extract of malt, in a slightly warm mortar, it was found possible to make admixtures of the extract with sperm oil, seal oil, train oil, castor oil, and olive oil, which resembled, to some extent, in their behaviour the preparation previously described. As these experiments were made on quite a small scale however, I do not care to attach much importance to them. I hope to repeat them upon a large scale and with proper mixing apparatus.

The fact that malt-extract and cod liver oil are miscible is certainly curious and interesting. It is also, I venture to think, of some importance from a medical point of view, for, if the nauseous oil can be given in a state of actual solution in so pleasant and nutritive a menstruum as malt-extract, the difficulties attending its administration to some persons, and its assimilation by others, must necessarily be diminished.

A question arises, as to which of the constituents of the malt-extract are especially concerned in this solvent action; but it is one to which I am not at present prepared to hazard an answer. The experiment with dextrine, already referred to, may have some slight significance, as tending to show that this constituent of malt-extract, at all events, is not, when in aqueous solution, miscible with the fixed oils.

CONCLUSION OF THE PROCEEDINGS OF THE SOCIETY.

---

PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS.

COMMUNICATED BY DR. E. WALLER, NEW YORK.

At the meeting of the American Society of Public Analysts, held June 22nd, 1885, the subject for discussion was with regard to the epidemic of typhoid fever at Plymouth Pa. Results of analyses of the water there made under the direction of Dr. Waller were communicated to the meeting. The cause was apparently a case of typhoid fever in a house on the banks of the stream supplying the water company's reservoir. The excreta were thrown on the snow, near the bank of the stream, and, nine days after a thaw, the fever broke out with about 50 or 60 cases within a couple of days, among those using the water. The epidemic was, no doubt, prolonged by contamination of wells in the town.

---

ON THE PROCESSES AND STANDARDS OF FOOD ANALYSIS IN USE AT  
THE MUNICIPAL LABORATORY OF THE CITY OF PARIS.

BY JOHN MUTER, PH.D., M.A., F.I.C.

"They order these things better in France" is a trite saying, and having been courteously furnished with a copy of the report of M. Girard, the eminent *Chef du Laboratoire*, made to the Prefect of Police on this subject, it has seemed to me interesting to the members of our Society and the readers of the ANALYST generally, that a careful critical *resumé* of the same should be laid before them, with the view of enabling them to

form an opinion how far the proverb may be held as applicable to Food Analysis. Before entering upon the chemical portion of the work (which is reserved for communication to the Dublin meeting of the Society), it will be well to state some facts about the constitution and working of the Laboratory, as far as staff and other arrangements are concerned. The establishment consists of a number of rooms on the ground floor and basement. On the former are found three large laboratories, capable of accommodating 35 workers, a private laboratory for the chief analyst, two dark rooms for polarization, &c., together with the offices and reception rooms. On the basement are (1) an engine room, with a gas engine, dynamo, and turbine, driving a system of ventilation; (2) a room for gas analysis and vacuum operations; (3) a dark room for micro-photography and spectrum analysis; (4) a room for organic analysis and dialysis; (5) a distilling room, and (6) the usual offices. The total annual cost is something under £9,000, and includes the chief analyst at £400 per annum, the sub-chief at £300, and 25 assistants, whose salaries vary from £220 to £100, together with a staff of "*Inspecteurs expert*," 20 in number, at salaries from £100 to £150. Here it is a matter of remark, that while no salary of any man entrusted with responsible work is under that usually paid to an ordinary analytical assistant in Great Britain, yet the remuneration of the heads of the departments is low as compared with what chemists of equal standing would require in this country. For example, at Somerset House Dr. Bell gets £1,000, and Mr. Bannister £750, per annum; and it is certain that, with us, really competent men could not be got to give up private practice for less than these amounts. There are two kinds of analyses performed. (1) The so-called qualitative, which is done without charge to any one bringing a sample. This is, of course, often really a quantitative analysis, as in the cases of milk, wine, and butter, but the laboratory has only to place the article in one of four classes, and report that it is either *good*, *passable*, *bad*, but *not injurious*, or *bad and injurious*. (2) The so-called quantitative analysis, in which the laboratory has to provide figures, and can charge fees ranging from 5 to 50 francs. For the convenience of the public, each police station in Paris receives samples of the gratis class only, and they are collected daily and taken to the laboratory by one of the police vans. Each purchaser must give the name and address of the vendor of the article he submits, and if it be found bad, then one of the "expert inspectors" is sent to the shop to inspect the similar articles in stock, and take samples, on which, if condemned, proceedings are taken. The public do not, as a rule, proceed; but if they get an unfavourable report, it is a guide to them not to buy any more at the shop in question, and the check thus obtained is clearly shown in the following paragraph by M. Girard:—

"This manner of exercising a larger and surer measure of sanitary police has commenced to discourage the adulterators, by inflicting on them a double penalty; not only do the ordinary convictions produce a direct action upon the condemned, as well as a restraining action on those who feel inclined to go and do likewise, but also the public, who, in the ordinary course of life, omit to surround themselves, while buying, with the legal conditions necessary to ensure a judicial establishment of the fact that they did not tamper with the article, yet know very well that they are not the authors of the adulteration. Thus, enlightened by the certificate of analysis, they can change the

furnisher, and seek elsewhere such food and drink as will be worth the price they are willing to give, and that will repay the trouble of their search. This just abandonment of the adulterating vendor by his cheated client constitutes for the former a pecuniary punishment not less severe than the fines inflicted in Court." As with the public analysts of this country, the laboratory has nothing to do with any ulterior legal proceedings; it simply examines the article and gives its report, and there its functions end. This limitation has, however, not prevented the similar throwing of personal abuse at the chemists employed, just as the public analysts with us are often treated by those who defend what they call—falsely call—*trade interests*, and under a pretence of being willing that real offenders should be punished, constitute themselves the drag upon the wheels of sanitary progress, by abusing the passive agents of the administration of the law. On this point, M. Girard says: "Those who have been condemned have well abused the law and its auxiliaries—*i.e.*, ourselves; human nature was ever thus, and we could not expect to be exceptions, but, instituted to protect the public, we would be wanting in our mission if we had not denounced fraud wherever we detected it. It is true that we would have been happy to have found fewer wrong-doers, but that was not in our power; we have no right to gloss over any of the results of our analyses or of our researches. Whatever has been the recrimination to which our action has given birth, it is certain that repression of adulteration has become more general and more serious. This amelioration is specially felt in such articles as milk and wine. They have ceased, except in rare occasions, to dilute the milk to the enormous extent found at first on our commencing work, and they less frequently and less largely add to wines those commercial, crude alcohols, of which the dangers have been recently demonstrated by our leading pathologists." Thus, it is evident that what has occurred in this country has been reflected in France, as a similar effect at home was specially noted by more than one professional speaker at the Food Conference, held last year at the Health Exhibition.

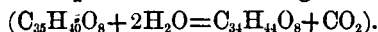
As to the management of samples, each person on leaving the same is received by an appointed officer, who enters all particulars on a book, and detaching a counter-foil, hands the same to the applicant, telling him at the same time when to call for the result. He then attaches to the sample the corresponding number, and sends it in to the laboratory, without any other indication of its source. The samples are first examined (especially in the case of wine) by experts trained to judge by physical appearances, taste, &c., and the results having been entered in a special register, they are then divided among the chemists, who are bound to commence the analysis the same day. The chemists are each specialists in some particular article of food or drink, certain men being charged only with the analysis of milk, others of wine, others of fats and oils, and some with microscopic work. This has been found the only way to obtain sufficient rapidity of work and control of accuracy. While, however, all are specialists, they yet have sufficient general ability to take part in any branch of work, should a special requirement arise.

Having thus briefly sketched the *locus in quo* and *modus operandi*, it is now necessary to turn to the actual work itself.

(To be continued in our next.)

## MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

ON THE DETECTION OF ALKANET IN WINE.—By HERR HERZ.—Report der Anal. Chem. 13, 210.—Wine coloured red with alkanet-root gives a yellow precipitate with ether; but the alkanet can best be extracted with amyl alcohol. To isolate the alkanet from other colouring-matters, oil of almonds, or olive oil is added, and the amyl alcohol driven off in the water-bath. The oil assumes a red colour in the presence of alkanet, which, by saponification with soda, changes to a splendid blue, disappearing on the addition of acid. Wines which have stood six months or so, with alkanet, colour the oil red, the saponified solution is, however, not blue but green. As alkanet-red is converted by nitrogen compounds into alkanet-green



the change probably depends upon some such reaction, although ether extracts a red colouring matter and not green.

F. H. H.

ON THE DETERMINATION OF THE WATER IN POTATOE STARCH, by SAARE. Zeit. f. Anal. Chem. 242, p. 293. Zeitschr. f. Spiritus ind. 7. 550.—The author weighs out 100 grms. of the substance under examination, washes it into a 250 c.c. flask, fills up to the mark (at 17.5 C.), weighs, and subtracts the weight of the dry empty flask. The amount of water is calculated from the difference, with the help of the following table:—

Weight found.	Amount of water.	Weight found.	Amount of water.	Weight found.	Amount of water.	Weight found.	Amount of water.	Weight found.	Amount of water.
grms.	%	grms.	%	grms.	%	grms.	%	grms.	%
289.40	0	284.25	13	279.15	26	274.05	39	268.90	52
289.00	1	283.90	14	278.75	27	273.65	40	268.50	53
288.60	2	283.50	15	278.35	28	273.25	41	268.10	54
288.20	3	283.10	16	278.00	29	272.85	42	267.75	55
287.80	4	282.70	17	277.60	30	272.45	43	267.35	56
287.40	5	282.30	18	277.20	31	272.05	44	266.95	57
287.05	6	281.90	19	276.80	32	271.70	45	266.55	58
286.65	7	281.50	20	276.40	33	271.30	46	266.15	59
286.25	8	281.10	21	276.00	34	270.90	47	265.75	60
285.85	9	280.75	22	275.60	35	270.50	48		
285.45	10	280.35	23	275.20	36	270.10	49		
285.05	11	279.95	24	274.80	37	269.70	50		
284.65	12	279.55	25	274.40	38	269.30	51		

DETECTION OF ADDED WATER IN MILK.—J. Uffelmann has already proposed the estimation of nitrates as a proof of the watering of milk. Writing to the *Milch Zeitung* he repeats his views, and gives the following simple test, which he says will show the slightest trace of nitric acid. Put into a white porcelain capsule an amount of diphenylamine about the size of a split pea, and pour over it about 25 minims of sulphuric acid (absolutely free from a trace of nitric acid), and stir the mixture with a clean glass rod until it is of a pale rose colour. Now let three or four drops of the suspected milk, or other fluid, trickle down the side of the capsule, which should be kept as motionless as possible. If much nitric acid be present in the fluid under examination, there will in-

stantly form, at the point where it meets the test, a blueish stripe, which gradually spreads until it forms a blue cloud throughout the fluid. If the quantity be, on the contrary, very minute, it will be some minutes before the blue colour becomes visible, but so delicate is the test that where there is the slightest trace of nitric acid present, it will, even under these circumstances, spread until it clouds the entire amount of test-fluid with a delicate blue. Two drops of ordinary drinking water are sufficient to show the re-action.

W. H. D.

---

NOTES ON THE MANUFACTURE OF ARTIFICIAL HONEY.—Centralhalle 27, 303.—H. Hager warns analytical chemists against American honeys, which are now being adulterated with a syrup manufactured from maize, the method being kept secret. His own experiments show, however, that if wheat or maize-starch (not potatoe-starch) be treated with oxalic acid or any other powerful organic acid, a syrup is produced, which in a certain concentration, and after standing two or three weeks, exactly resembles, in taste and appearance, an old honey.

F. H. H.

---

ON THE DETERMINATION OF ORGANIC MATTER IN WATER.\*—M. PETIT.—Journ. de Pharm. et de Chimie., Ser. 5, Tome 11, p. 305.—The author points out the necessity of having a uniform method for the determination of the value of potable waters and, with regard to the titrations of the organic matter, recommends that the water, under examination should, in all cases, be acidulated with sulphuric acid and boiled ten minutes. The permanganate of potash solution should contain 633 grm. of the salt to a litre. The author maintains further, that the percentage of organic matter alone, does not suffice for the examination of a potable water, as the utility of the latter is materially altered by a high percentage of ammonia and nitric acid. It would, therefore, be advisable to determine the whole quantity of nitrogen, to calculate it as albumen, and on these data to base a classification of potable waters.

F. H. H.

---

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

DETERMINATION OF THE VALUE OF PEPSIN.—O. Schlickum recommends for this purpose the conversion of albumen into peptone. An egg is placed in boiling water for five minutes, and rapidly cooled by cold water; the albumen will be hard, the yolk semi-liquid. Cut the albumen into very fine pieces, and rub through a fine sieve; of this albumen put 10 gms. into a solution of 0.1 pepsin in 150 water, add 2.5 HCl, and keep the mixture at a temperature of 40°C. by means of a water-bath. On being dissolved, the albumen is transformed first into hemialbumose, and afterwards into peptone. Digest for 12 hours, then filter, and to 10 c.c. of the filtrate add, drop by drop, 1 c.c. HNO<sub>3</sub>, which should not cause more than a slight opalescence.—*Ph. Ztg.* W. H. D.

---

\* archinder Pharm. [1885] 11. 452.



SPECTROSCOPIC BEHAVIOUR OF FATTY OILS.—In accordance with Doumer, the fatty oils may be divided into four classes with reference to their spectroscopic behaviour. First, such oils which show the absorption spectrum of chlorophyll, as Olive, Hemp, and Nut Oil do. Second, such oils which show no absorption spectrum at all, *i.e.*, which allow all rays to pass unabsorbed, to this class belong Castor oil and oil of sweet and bitter almonds. Third, oils which absorb all chemical rays of the spectrum; to this class many oils belong, and their spectrum is very characteristic; it only shows the warmer portion of the spectrum (red, orange, yellow, and about one-half of the green) unchanged, but here it stops. The balance of the spectrum is absorbed. To this class belong Rapeseed, Linseed, and Mustard oils. A fourth-class seems to be a modification of the former; the absorption comprises only bands of the chemical position of the spectrum, instead of being complete, as in the former case. This class comprises Sesame oil, Poppyseed oil, Cottonseed oil, and Peanut oil.—*Am. Chem. Review.*

W. H. D.

SAPONIN FROM SAPONARIA OFFICINALIS.—M. C. SCHIAPARELLI.—The saponaria root having been treated with alcohol, 90 per cent., and the alcoholic fluid allowed to stand in a cool place, a flocculent yellow substance is deposited on the sides of the vessel. This substance is washed with alcohol and digested with a mixture of alcohol and ether, to remove colouring matter.

It is very difficult to obtain saponin perfectly free from mineral substances, but this can be done by treating it successively with alcohol and hydrate of barium, and separating the barium by means of sulphuric acid.

The saponin thus obtained is recovered by solution in water; it is then precipitated by alcohol and ether, and again recovered by a sufficient quantity of alcohol. On evaporation *in vacuo*, the alcoholic solution deposits white flocks of pure saponin. These are washed with ether and dried *in vacuo* over sulphuric acid.

Analysis gives results similar to the formula suggested by Rochelder,  $C_{32}H_{51}O_{18}$ .

Saponin is a very white amorphous powder, which provokes sneezing. It is poisonous; soluble in water, slightly soluble in alcohol (1-100 boiling); insoluble in ether, benzene, and chloroform.

Aqueous solutions of saponin possess the curious property of dissolving salts insoluble in water (PbS, BaSO<sub>4</sub>, etc.) It is precipitated by acetate of lead and ammonio-nitrate of silver. Saponin is levogyrate. Saponite of barium is a white amorphous powder, the formula of which is probably  $(C_{32}H_{51}O_{18})_3Ba_2$ .

Saponin heated on a water bath with dilute acids separates into glucose and a crystalline substance, *Saponetin*,  $C_{50}H_{99}O_{15}$ , insoluble in water and in ether, soluble in alcohol.—*Bull. de la Soc. chimique.*

W. H. D.

ON THE QUANTITATIVE DETERMINATION OF CINCHONIDINE IN QUININE SULPHATE OF COMMERCE.\*—De Vrij recommends, in the *Pharm. Weekblad*, the following method for determination of cinchonidine in quinine sulphate:—Five grms. of the preparation under examination are dissolved in 11 c.c. normal sulphuric acid, by warming to about

\**Archiv der Pharm.* [1885] 11. 452.

60° C. The solution is evaporated in a weighed dish to incipient crystallisation, and the crystalline mass, formed on cooling, mixed with a sufficient quantity of distilled water to bring it up to the original weight. The acid sulphate of cinchonidine goes into solution. The solution is separated from the undissolved acid sulphate of quinine, shaken up with excess of soda and 25 grms. ether, the latter of which brings the separated cinchonidine in crystals to the surface of the liquid, where they can be collected and weighed.

The method is not absolutely accurate, but suffices to detect a mixture of 2 per cent. cinchonidine with quinine sulphate.

F. H. H.

REACTIONS OF ALKALOIDS AND GLUCOSIDES.—Serena in *l'Orsi* gives the following colour-reactions produced on treating certain of these compounds *successively* with a few drops of concentrated sulphuric acid and a very small quantity of a dilute solution of chloride of iron, with the aid of a slight heat:—

Santonin : canary yellow ; violet.

Codeine : light violet-red, deepened by heat ; sky-blue.

Anilin : not changed ; violet-red, becoming wine-red on heating.

Solanine : orange-red, then yellow (with water, light blue ; violet).

Solanidine : same reactions.

Cholesterin : orange-red ; violet (with water, green).

Sabadilline : orange-red ; wine-red.

Colocynthin : orange-red ; blood-red.

Papaverine : purplish-red ; colourless, then violet on heating.

Elaterin : reddish-yellow ; grass-green.

Narceine : coffee brown ; bluish-green.

Opiatine : no colouration ; green, rapidly becoming deep blue.

Nitro-atropine : no colouration ; violet.

Eserine : orange-yellow ; intense brownish-red (with water, cloudy light blue).

Digitalin : brownish-red ; bright brownish-red with a point of violet, and on addition of water greenish-yellow.

Smilacin : deep orange-yellow ; reddish-brown with violet reflection ; violet, then bluish-green, and on heating, light violet.

Apomorphine : not changed ; at point of contact violet streaks, the bluish-green mass becoming light violet on heating.

W. H. D.

### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

ON A NEW GRAVIMETRIC METHOD FOR THE DETERMINATION OF MANGANESE.—By HAMIS VON JUPTNER.—Chem. Zeitung. [85] 39. 692.—This method is based on the conversion of manganous acetate by ignition into red oxide of manganese ( $Mn_2O_3$ ) and has been used by the author for the determination of manganese in iron, steel, iron ores and slags.

The hydrochloric acid solution of the substance under examination (ferrous oxide when present, must first be oxidised with potassium chlorate, and the chlorine removed) is neutralised in a measuring flask, ammonium chloride added, the mixture precipitated in the cold with barium carbonate, and digested for some time with excess of ammonium hydrate. The flask is now filled to the mark, the liquid well mixed, and filtered through a dry filter. From an aliquot part of the filtrate ( $\frac{1}{2}$ ) the barium is removed with sulphuric acid, the solution neutralised with ammonium

hydrate, heated to boiling, and precipitated with ammonium sulphide. The precipitate is collected on a small filter and washed with water containing ammonium sulphide. It is then dissolved in acetic acid, the solution filtered from the insoluble residue, brought into a weighed platinum dish, evaporated, ignited and weighed as  $M_3O_4$ .

F. H. H.

NOTE ON THE ESTIMATION OF UREA BY LABARRAQUE'S SOLUTION.—BY A. B. LYONS, M.D.—(Pharmaceutical Record, May, 1885.)—Unquestionably the simplest and most rapid method of estimating urea in the urine is that known as the hypobromite process. Urea is decomposed in an appropriate apparatus, by means of a solution of bromine in excess of caustic soda. The hypobromite solution is unstable, and hence must be freshly prepared when required for use. Dr. Squibb has endeavoured to remove the difficulty by substituting for the hypobromite solution the U. S. P. liquor sodæ chlorinatæ, and furnishes a simplified form of apparatus in which this may be used. It is quite possible that, with some samples of this solution, satisfactory results can be obtained but the author's experience has been that the quantity of gas evolved has been uniformly less than it should be. A specimen of urine, which showed by the hypobromite process two per cent. of urea, would yield with the hypochlorite solution, gas corresponding with only 1.6 per cent. or less. He has not had the time to investigate the cause of this difference. The gas is evolved much more slowly when the hypochlorite is used, and the reaction does not appear to be complete, even when a large excess of the reagent is employed. He finds, however, that there is a way to overcome the difficulty, and, in fact, to *change the hypochlorite into hypobromite* extemporaneously. Simply add to the solution chlorinated soda, of which 25 c.c. should be sufficient to decompose the urea in 4 c.c. of urine, 5 c.c. of a 20 per cent. solution of potassium bromide, a few minutes before the urine is introduced. With some specimens of the solution of chlorinated soda it is necessary to add also a little caustic soda—2 to 5 c.c. of a ten per cent. solution—to insure absorption of all the carbonic anhydride found in the reaction. The solution of chlorinated soda must answer the U. S. Pharmacopœia requirement of containing at least two per cent. of available chlorine. With this modification of Dr. Squibb's plan, results are obtained identical with those reached by the hypobromite process; and, as the reagents are easily procurable, there need be no longer any reason why the estimation of urea should present any especial difficulties.

W. H. D.

A NEW METHOD OF ESTIMATING UREA BY TITRATION BY H. J. HAMBURGER.—Reagents.—1. *Alkaline solution of bromine.* Dissolve 30 grms. of sodium hydrate in 1 litre of water, and shake the liquid with about 20 cubic centimeters of bromine. The resulting clear yellow liquid will become somewhat cloudy after 15 minutes, and must be filtered through asbestos, after which it remains clear. 2. *Solution of arsenite of sodium.* Weigh 19.8 grms. of arsenious acid, warm on a water bath with a solution of 10.6 grms. of pure carbonate of sodium, and dilute to 1 litre. (Since arsenite of sodium may be had in the market, the re-agent could be also prepared by dissolving 38.4 grms. of this

salt in water to one litre.) 3. *Iodine solution.* Dissolve 12.7 grms. of iodine in water, with the aid of iodide of potassium, to 1 litre. Before the actual analysis, it is necessary to adjust the relation between the arsenite and the iodine solution. For this purpose, 10 c.c. of the former, mixed with 20 c.c. of a saturated solution of carbonate of sodium, and a few drops of clear starch solution are first mixed, and this is titrated with the iodine solution until a blue tint just begins to appear. The relation of the alkaline bromine and of the arsenite solution is ascertained by measuring off 10 c.c. (or more) of the bromine solution, adding an excess of 1.2 to 3 c.c. of arsenite solution, passing carbonic acid gas through the mixture for about 15 minutes, rinsing the gas delivery tube into the liquid, and then adding 20 c.c. of a nearly saturated solution of carbonate of sodium and starch solution. The excess of arsenite solution is now titrated back with the iodine solution. Finally, the bromine solution is compared with a solution of urea of known strength, by cautiously adding the bromine to the urea solution until the latter acquires a yellow tint, then adding an excess of 1.2 to 3 c.c. of bromine solution, and finally titrating back with the arsenite and the iodine solution. [When the liquids are once adjusted, it will be found that the analysis takes a comparatively short time, particularly if a series of them is to be performed.] *Execution of analysis.* Measure off 15 (or 10, or 20) c.c. of urine; add bromine solution cautiously and under agitation until no more gas is given off, and then add 1.2 to 3 c.c. more of bromine solution. After 5 or 10 minutes, add arsenite solution until the colour of the liquid turns lighter yellow, and then test with starch and iodide of potassium paper, to see if the latter is still rendered blue (this is only necessary in the first titration in a series); if not add about 3 c.c. of arsenite solution. Now pass carbonic acid gas (best from a cylinder or reservoir) through the liquid, add about 20 c.c. of sodium solution, and a few drops of starch paste, and determine the excess of the arsenite by the iodine solution. From the data thus obtained, the quantity of urea may be easily calculated.—*Chem. Centralbl.*—

W. H. D.

---

ON THE DETERMINATION OF ALUMINA AND IRON IN THE PRESENCE OF PHOSPHORIC ACID. M. Kretschmar. *Chem. Zeit.* 53, 942.—If a solution, containing aluminic and ferric phosphates, and acidulated with hydrochloric, nitric, or sulphuric acid, be changed into one acidulated with acetic acid, both phosphates separate out. Further, if the solution contains more phosphoric acid than is necessary for the formation of both phosphates, the precipitate contains an amount of phosphoric acid corresponding to the quantity of alumina and iron. It is evident, therefore, that if, on the one hand, the weight of the alumina and iron be known, and, on the other, the amount of phosphoric acid contained therein, the quantity of alumina and iron can be easily calculated. The author has applied this fact to a method which reduces the otherwise troublesome separation of iron and alumina to a simple phosphoric acid determination, which is especially to be recommended when a large number of phosphate determinations have to be carried out daily. The method is illustrated in the following details of an analysis of a native phosphate:—5 grms. of phosphate are dissolved in hydrochloric and nitric acids, and made up to 500 c. c. From this, two quantities of 100 c. c. (= 1 grm.) are measured

off. The liquid is cooled down as far as possible, ammonia added in just sufficient quantity to produce a precipitate, and the solution acidulated with acetic acid. The two precipitates are freed from calcium and magnesia by washing with hot water. One of the precipitates is then weighed; the other dissolved in hydrochloric acid, treated with citric acid and ammonia, and precipitated with magnesia mixture. The formula used in the calculation is the following, where G = the weight of the alumina-iron precipitate:—

$$\begin{aligned} \text{Fe}_2\text{O}_3 &= (0.906 \text{ G} - \text{Mg}_2\text{P}_2\text{O}_7) 3.08. \\ \text{Al}_2\text{O}_3 &= \text{G} - (\text{Fe}_2\text{O}_3 + \text{Mg}_2\text{P}_2\text{O}_7, 0.14). \end{aligned}$$

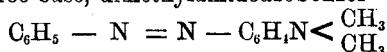
F. H. H.

ON A SIMPLE METHOD FOR THE SEPARATION OF ZINC FROM THE OTHER METALS OF ITS GROUP.\*—BY W. HAUPE. Chem. Zeit. [1885] 543.—As any easy method for the separation of zinc from iron, nickel, cobalt, manganese and aluminium, the author recommends their conversion into formates, and treatment with sulphuretted hydrogen. The zinc is completely precipitated. The precipitate is always free from manganese and aluminium, and also from nickel, cobalt and iron, if the solution contain a sufficient quantity of free formic acid (12–20 c.c. acid of 1.2 Sp. Gr. to 250–500 c.c. liquid) and the metals be in no great excess, otherwise the precipitate is slightly coloured with the sulphides of the metals. In order to get rid of these traces, the filtered and washed precipitate is dissolved in nitric acid, saturated first with ammonia, and then with formic acid, and again precipitated with sulphuretted hydrogen. The precipitate separates out in a granular form if the sulphuretted hydrogen be passed into the hot solution. During the precipitation a thin skin of zinc sulphide generally clings to the sides of the beaker, which cannot be removed by rubbing. This residue is dissolved, after washing out the beaker, in nitric acid, ammonia and ammonium sulphide added, and the mixture acidulated with formic acid. The precipitate produced is added to that already on the filter, which is then dried and weighed.

F. H. H.

DIMETHYLAMIDOAZOBENZOL AS INDICATOR.—BY BERNHARD FISCHER.—Archiv. der Pharm. xii. 223. 11. 434.—The author, in criticising the indicators in use, points out that there are only two which show the final reaction by a change of colour, namely, *litmus* and *methyl-orange*. The use of litmus is attended with difficulties, as it does not give good results in the presence of free carbonic acid, on the other hand methyl is not affected by carbonic acid, but the change of colour (from orange to pink), is not sufficiently striking for many people, and commercial methyl-orange is not always pure.

Instead of methyl-orange (dimethylamidoazobenzolsulphonic acid), the author proposes the use of the free base, dimethylamidoazobenzol



which can easily be prepared pure, and possesses the advantage that the final reaction is shown by a sharp change of colour from lemon-yellow to pink, which is also plainly

\* Report der Anal. Chem. 12, 206.

visible in gaslight. It is prepared as follows:—9.3 parts (1 mol.) aniline are dissolved in 30 parts (2 mols.) hydrochloric acid (25 per cent.) To this mixture are added, after diluting and allowing to cool, a solution of 7 grms. (1 mol.) sodium nitrite in water. The liquid so obtained is poured into a solution of 12 grms. (1 mol.) dimethylaniline in 15 grms. hydrochloric acid, and after allowing the mixture to stand, sodium acetate (about 30 grms.) added, until the liquid smells distinctly of acetic acid. The separated crystals are purified by re-crystallisation in alcohol; their melting point is about 115°C. The indicator is used in alcoholic solution (1 : 200), about 5 drops being taken to every 100 c.c. liquid.

F. H. H.

---

A NEW EXTRACTION APPARATUS FOR LABORATORY PURPOSES\*.—By THEODOR WEYL.—Feitsche f. Instrumenten Runde (1885). 5.126.—The new apparatus resembles the well-known Drechsel's extractor; it deviates somewhat from the latter, however, in the part intended for the reception of the substance to be examined. This consists of two filters turned one against the other, and connected with a wooden ring and mercury joint. An improvement upon Drechsel's apparatus is the substitution of mercury joints for the india-rubber connections. The apparatus closes hermetically, and is not easily breakable, as it consists of several pieces easily moveable with reference to one another. The substance to be extracted is conveniently introduced, and can be replaced by fresh quantities without occasioning loss.

F. H. H.

---

ON THE VALUATION OF OZOKERIT.—DR. B. LACH.—Chem. Zeit. 51, 905.—The author remarks upon the difficulties attending the estimation of the value of a sample of Ozokerit. The following method has been used by him for some time for the determination of the value of mineral wax:—100 grms. of the wax are treated, at 170°–180° C., with 20 grms. fuming sulphuric acid in a weighed dish, the mixture being constantly stirred until sulphurous acid ceases to be given off. The difference in weight gives the loss due to volatilization (petroleum and water.) The residue is mixed with 10% of a decolourising powder (the residues of blood lye salt) previously dried at 140° C, and allowed to cool. A one-tenth part of the mixture is then weighed out and extracted with benzol. The residue is dried at about 180° C; the difference in weight gives the amount of wax.

The investigation takes about four hours, but does not require much attention.

F. H. H.

---

\* Chem. Zeit. 54, 964.

DIPHENYLAMINE AS A RE-AGENT FOR FREE CHLORINE. By H. Hager, Pharm. Centralhalle 25, 277 and 279.—The author gives a short recapitulation of the various methods for applying diphenylamine to the detection of nitrates, nitrites, &c.; he then points out that diphenylamine is a powerful re-agent for free chlorine.

The solution used is one of diphenylamine sulphate, consisting of 1 vol. of a 3·3 per cent. diphenylamine solution in alcohol and 5 vols. of pure cons. sulphuric acid. The liquid to be tested for chlorine is brought into a test tube of about 1 c.m. diameter, so that it stands about 4·5 c.ms. high. The diphenylamine solution is then poured gently down the side of the test-tube; it collects at the bottom forming the lower layer. If the two layers be mixed, the liquid in the presence of even a trace of chlorine is transiently coloured blue. The appearance of the colouration is shown up by holding a piece of white paper behind the test-tube. If no colouration takes place, pure sulphuric acid is added; a minute trace of chlorine produces a blue layer between the two liquids.

Diphenylamine does not act upon traces of pure bromine.

F. H. H.

QUANTITATIVE ANALYSIS BY ELECTROLYSIS.—A. CLASSEN and R. LUDWIG.—Ber. d. d. Chem. Ges. 18, 1104. The authors add to their former communications (see ANALYST, 105, p. 227) that the quantitative separation of antimony and tin, which affords considerable difficulty in the ordinary gravimetric method, can easily and accurately be carried out by the electrolytic method. The antimony is precipitated in a solution of the sulphides of the two metals in sodium sulphide.

On the other hand, the authors' investigations have led to no satisfactory electrolytic separation of arsenic from tin and antimony.

F. H. H.

#### BOOKS, &c., RECEIVED.

Documents sur les Falsifications des Matières Alimentaires et sur les travaux du Laboratoire Municipal (Deuxième Rapport); Proceedings of the California Pharmaceutical Society for the years 1883 and 1884; Report on Milk and its Adulterations, by E. W. Martin, assisted by Walter Moeller, Ph.B. Report to the Magistrates and Council of the City of Glasgow on the Refuse Water of Chemical Works and other Factories, by Dr. William Wallace, F.R.S.E., F.C.S., F.I.C.; American Chemical Journal; American Chemical Review; American Druggist; American Grocer; British and Colonial Druggist; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Guardian; Cowkeeper and Dairyman's Journal; Independent Journal; Invention; Journal of Chemical Industry; Journal of Microscopy and Natural Science; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; The Sanitarian; San Francisco News Letter; School of Mines Quarterly (New York); Science; Scientific American; Scientific Californian; Society of Arts Journal; Students' Journal.

#### NOTICES TO CORRESPONDENTS.

A. PERCY SMITH. Your communication received, and under consideration for next month.

All communications as to literary matters to be addressed to the Editorial Department, 325, Kennington Road, London, S.E.

# THE ANALYST.

SEPTEMBER, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
(a) REPORT OF MEETING .. .. .	155	(a) SECALE CONUTUM—KOSTER & BERNBEEK	169
(b) ESTIMATION OF DISSOLVED OXYGEN IN WATER—DR. DUPRE, F.R.S. . . .	156	(b) DETECTION OF FATTY OIL IN MINERAL OILS—F. LUX .. .. .	169
(c) NOTE ON THE DETECTION OF FLUORESCENT SUBSTANCES IN LARD OIL—PROF. TICHBORNE .. .. .	161	(c) DETERMINATION OF POTASSIUM BITARTRATE IN CRUDE TARTAR AND IN WINE LEES—F. KLEIN .. .. .	170
(d) ON THE USE OF AMMONIUM CITRATE IN THE ANALYSIS OF PRECIPITATED PHOSPHATES—H. H. B. SHEPHERD .. .. .	161	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY—	
(e) METHOD OF SEPARATING AND DETERMINING ARTIFICIAL COLOURS IN BUTTER & C.—E. W. MARTIN .. .. .	163	(a) NEW METHOD FOR THE VOLUMETRIC DETERMINATION OF CHLORINE, E. BOHLIG	171
(f) PROCESSES AND STANDARDS IN USE AT THE MUNICIPAL LABORATORY OF THE CITY OF PARIS ( <i>continued</i> ):—DR. MUTER ..	163	(b) NEW REACTION FOR TITANIC ACID—R. PRESENIUS .. .. .	172
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD:—		(c) NOTE ON KJELDAHL'S NITROGEN METHOD—D. PEIFFER & F. LEHMANN ..	172
(a) NEW REACTION FOR ALBUMEN—D. OXENFELD .. .. .	167	(d) DETECTION OF CHLORIDES IN THE PRESENCE OF BROMIDES AND IODIDES—L. DE KONINCK .. .. .	173
(b) DETERMINATION OF CASEINE IN MILK—FRENZEL & WEYL .. .. .	167	(e) PHENYLHYDRAZINE AS A TEST FOR SUGAR IN URINE—P. GROCCO .. .. .	174
(c) NEW TESTS FOR GRAPE AND MILK-SUGAR—RUBNER .. .. .	168	OBITUARY NOTICE .. .. .	174
(d) DETECTION OF WHEAT IN RYE FLOUR—WITTMACK .. .. .	169	INTENDED INTERNATIONAL FOOD EXHIBITION .. .. .	174
		BOOKS RECEIVED AND NOTICES TO CORRESPONDENTS .. .. .	174

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The annual country meeting was held (by the kind invitation of its President, Sir Charles Cameron, M.D.), at the Royal College of Surgeons, Stephen's Green, Dublin, at 5 p.m., on Saturday, August 1st. The President being absent, the chair was filled by Dr. Muter, Vice-President.

The following papers were read and discussed:—

“On the Results of the Working of the Adulteration Acts in Dublin,” by Sir Charles Cameron, M.D., F.C.S., F.I.C.

“On the Detection of Fluorescent Substances in Lard Oil,” and “On the Specific Gravity of Butter Fats,” by Prof. Tichborne, F.I.C.

“On the Processes and Standards of Food Analysis in use at the Municipal Laboratory of the City of Paris,” by Dr. Muter, F.C.S., F.I.C.

“On the Analysis of Precipitated Phosphates,” by H. H. B. Shepherd, F.C.S., F.I.C.

“Method of Separating and Determining Artificial Colours in Butter, Fats and Oils,” by Edward W. Martin.

After the meeting, the members and their friends dined at the Shelbourne Hotel, in Stephen's Green, on the kind invitation of Sir C. Cameron.

Several ladies attended, and a very genial evening was spent, and highly enjoyed by the members present.



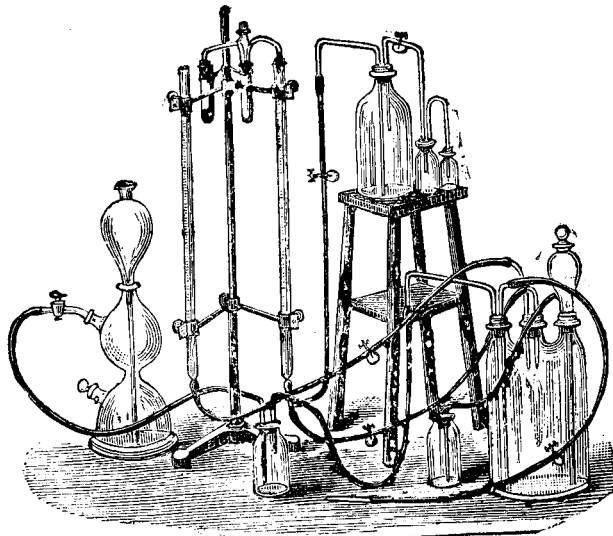
## THE ESTIMATION OF DISSOLVED OXYGEN IN WATER.

By A. DUPRE, PH. D., F.R.S., &amp;c.

*(Read at the Meeting on 10th June, 1885.)*

Up to the year 1872, the estimation of the oxygen dissolved in water had to be accomplished by means of a somewhat troublesome and time-taking process, in consequence of which this feature of water analysis was very greatly neglected. In that year, however, P. Schützenberger and Ch. Rister described a process in which they made use of the remarkable reducing power of an acid discovered by Schützenberger in 1869, and called by him hydrosulphurous acid, now known as hyposulphurous acid or dithionous acid. By the help of this acid, or rather of its sodium salt, the estimation of the oxygen dissolved in water becomes at once easy and rapid, and I have no doubt that everyone once familiar with the process, will very frequently employ it. It is this process, with some modifications introduced by myself and others, which I propose to bring before you this evening.

In the year 1881, while engaged in an extensive series of analyses of Thames water, my attention was, among other features, directed to the varying amounts of dissolved oxygen contained in the river water at different parts of the river, and after trying various processes, I finally adopted the one to be described. The chief difference between it and the original process consists in making use of fully aerated water for standardizing the hyposulphite solution, in place of the ammoniacal sulphate of copper, used by Schützenberger and Rister.



The apparatus employed, which, somewhat complicated at first sight, works very easily, is as follows:—One neck of a three-necked Woulf's bottle, of about one quart capacity, is connected, by means of a doubly-perforated india-rubber stopper; with two burettes for containing indigo and hyposulphite solutions respectively. The hyposulphite burette is connected, at its lower end, in the usual manner, with a supply bottle holding the hyposulphite solution, and both burette and supply bottle are connected

at the top with a small wash-bottle, containing an alkaline pyrogallic acid solution, through which all the air entering, when some of the solution is withdrawn, has to pass and is deprived of most of its oxygen. The second neck carries the vessel for holding the water, usually a pear-shaped vessel of about  $\frac{1}{4}$  litre capacity, with a stop cock below (a separator), by means of which the water can be run into the Woulf's bottle. The delivery tube from this vessel should be carried nearly to the bottom of the bottle. Through the india-rubber stopper of the third neck, three tubes are passed; one, reaching nearly to the bottom, is connected with an apparatus for generating hydrogen; the second, also reaching nearly to the bottom, is used as a syphon for drawing off the contents of the Woulf's bottle without admission of air. The lower, outer end of this syphon tube can be closed by a pinchcock. The third tube passes only just through the stopper, and is connected, outside, with a wash-bottle, through which the hydrogen, passing through the whole apparatus, can escape, while at the same time access of air to the apparatus is cut off by the water contained in the bottle.\*

An experiment is carried out as follows:—The two burettes having been filled, the Woulf's bottle is entirely filled with water, all communications with the air are cut off, the syphon is opened, and the hydrogen turned on. The hydrogen entering the bottle, presses the water out through the syphon, and by the time all the water is expelled, the bottle is filled with hydrogen; after this a gentle current of hydrogen is kept up, as long as the experiment lasts. The syphon is closed, and a small quantity of water is run into the bottle through the separator (20 to 30 c.c., or thereabouts). This water is slightly coloured by running in a small quantity of indigo, and the colour of this indigo cautiously discharged by the careful dropping in of hyposulphite solution. When this is accomplished, the contents of the pear-shaped vessel, which has meanwhile been filled with the water to be examined, are run into the bottle, and the blue colour thereby restored to the contents once more cautiously discharged. The amount of hyposulphite solution required for this is noted, the contents of the Woulf's bottle withdrawn by means of the syphon, and the same process repeated; the mean of the two experiments gives the amount of hyposulphite required by the water under examination. It now only remains to standardise the hyposulphite solution, which is accomplished by repeating the above process, with fully aerated water of known temperature, taking also the mean of the hyposulphite required in two experiments. From the respective quantities of hyposulphite required, the amount of oxygen contained in the water under examination is calculated. At first, I assumed that the proportions of oxygen dissolved in the two waters stood in the same relation as the amount of hyposulphite solutions required to discharge the blue colour in the two, but experiment showed that this was not so. The amount of indigo added to the water is, after its reduction, as a rule, entirely insufficient to take up the whole of the oxygen dissolved in the quantity of water taken for an experiment, and this necessitates a correction. On carrying out the experiment, as described above, it will be noticed that when the blue colour has been discharged, by the cautious addition of hyposulphite, it will very speedily return, and when again discharged, will return again, and this will

\* The apparatus may be obtained at Messrs. Townsend and Mercer, Bishopsgate-street Without.

continue until twice the amount of hyposulphite required to bring about the first decolouration has been added, after which the blue colour does not reappear, if the apparatus contains hydrogen only. This same amount of hyposulphite is required, if instead of taking a small amount of indigo merely, an amount is taken sufficient, after reductions, to take up the whole of the oxygen in the quantity of water used. This proves that the action of the dissolved oxygen on the reduced indigo is not the same as, at any rate, the immediate action taking place between the dissolved oxygen and the hyposulphite, but that in the first case twice the amount of oxygen is taken up at once than in the latter. The amounts of hyposulphite solution required to discharge the blue colour for the first time have therefore to be corrected, according to the amount of indigo employed, before they fairly represent the relative proportions of oxygen contained in the two waters compared. This correction is one half the amount of hyposulphite solution required simply to reduce the indigo added, so long as this is insufficient to take up all the oxygen present in the water, and to divide the amount of hyposulphite required by two whenever the indigo used was sufficient, or more than sufficient, to take up the whole of the oxygen of the water. The following table, giving one series of experiments out of many, will render the point clearer:—

TABLE I.

Amount of water taken.	Amount of indigo taken.	Amount of hyposulphite required.	Corrected amount proportional to the amount of oxygen in the water.
50 c.c.	4 drops*	0·65 c.c.	0·60 c.c. = 0·65 — 0·05
50 „	8 „	0·70 „	0·60 „ = 0·70 — 0·10
50 „	12 „	0·75 „	0·60 „ = 0·75 — 0·15
50 „	60 „ †	1·25 „	0·62 „ = 1·25 — 0·20
100 „	12 „	1·35 „	0·60 „ = 1·35 — 0·15

When the water to be examined is fully aerated, this correction has no influence on the result; its influence is but slight if the water is near its full aeration, but it becomes very noticeable if the water falls considerably short of full aeration. This correction would be unnecessary if the amount of indigo taken were always sufficient to take up, after reduction, the whole of the oxygen present in the water, but in fully aerated water this amount is so great that it is difficult to fix the point at which the last trace of blue has been discharged with any degree of accuracy, since the solution never becomes colourless, but remains more or less yellow. I, therefore, prefer to use much less indigo and to take the point of first disappearance of the blue colour, afterwards applying the correction as above described. The amount of indigo I generally take is such that for its decolouration or reduction about  $\frac{1}{10}$  the amount of hyposulphite will be required than that used in the examination of a water fully aerated at mean temperature.

The actual amount of oxygen contained in the water used for standardizing, is calculated by means of Bunsen's formula, but in order to save trouble, Mr. Dibdin has calculated the amount contained in a gallon of water at various temperatures, so that it can at once be read off.

\* 4 drops indigo required 0·1 cc. hyposulphite for their decolouration, corrections to be applied, therefore 0·05 cc. for every 4 drops indigo taken.

† In this case more indigo was taken than could be oxidised, after its reduction, by the oxygen of the water.

TABLE II.\*

Table shewing the quantity of Oxygen dissolved by water at various temperatures. Corrected to 0° C. at 760<sup>m.m.</sup> pressure.

Temperature Fahrenheit.	Temperature Centigrade.	Cubic Inches of Oxygen per Gallon.
41 °	5·00	2·101
42	5·55	2·074
43	6·11	2·048
44	6·66	2·022
45	7·22	1·997
46	7·77	1·973
47	8·33	1·949
48	8·89	1·927
49	9·44	1·905
50	10·00	1·884
51	10·55	1·864
52	11·11	1·844
53	11·66	1·826
54	12·22	1·808
55	12·77	1·791
56	13·33	1·775
57	13·89	1·760
58	14·44	1·746
59	15·00	1·732
60	15·55	1·719
61	16·11	1·706
62	16·66	1·695
63	17·22	1·683
64	17·77	1·674
65	18·33	1·667
66	18·89	1·660
67	19·44	1·652
68	20·00	1·644†
69	20·55	1·639
70	21·11	1·634

The result of the analysis may be given in different forms. Either the amount of oxygen found in the sample under examination may be given in cubic inches per gallon, or, as I prefer to do, in per cent. of possible amount, or in both. Thus, say a water, having a temperature of 20° C., required 4 c.c. hyposulphite, the fully aerated water, at the same temperature, requiring 6 c.c. of the same solution, while the amount of indigo used took 0·6 c.c. of the same hyposulphite for its reduction. The corrected amounts of hyposulphite are  $4 - 0·3 = 3·7$  and  $6 - 0·3 = 5·7$ . From Table II. we learn that a fully aerated water at 20° C., holds in solution 1·644 cubic inch oxygen per gallon, and we get the proportion  $5·7 : 1·644 = 3·7 : x$ .

$$x = 1·067.$$

$$\text{or, 2nd } 5·7 : 100 :: 3·7 : x; x = 64·9.$$

that is, the water contained 1·067 cubic inches of oxygen dissolved per gallon, and its degree of aeration was 64·9 per cent. of the total possible.

\* The table is calculated for a barometer pressure of 760 m.m. and would require corrections for variations from this, but such correction is mostly within the limits of experimental error.

† 1·644 cubic inch of oxygen at 0° C, and 760 m.m. pressure, weigh 0·594 grains.

One final precaution, sometimes required, should be mentioned. In every case the water to be examined, must, of course, be transferred to the measuring vessel in a manner to avoid æration. This can generally be done by introducing it by means of a syphon passing to the bottom of the vessel, and thus filling it from below. In case, however, the water is but very partially ærated, it will absorb oxygen even while thus filling it into and running it out from the measuring vessel. In order to avoid this, I fill the vessel, in such cases, with carbonic acid, previous to putting in the water, and allow carbonic acid only to enter as the water runs out again. The effect of this is well shown in the following experiment:—A water which, by boiling, had been deprived of most of its dissolved oxygen required, when tested as usual, 0·83 c.c. hyposulphite, whereas, when carbonic acid was used as above directed, it required only 0·15 c.c. of the same; fully ærated water required 10·3 c.c. hyposulphite and the amount of indigo taken required 0·8 c.c. hypo. According to the first experiment, the degree of æration of the boiled water was 4·34 per cent., according to the second, 0·75 per cent. only.

It only remains to describe the preparation of the hyposulphite solution. A concentrated solution of bisulphite of sodium is saturated with sulphurous acid, a bottle of a few ounces capacity is nearly filled with this solution, a small quantity of zinc dust is added, the bottle is closed and shaken for about ten minutes; should it become warm, it must be cooled—easiest under a running tap. The bottle is then allowed to rest, and after the excess of zinc has settled, the clear solution is decanted into a second bottle, of about ten times the capacity of the first. In this bottle thin milk of lime is added to alkaline reaction, after which, the bottle is completely filled with water, stoppered, shaken, and left at rest. The clear liquid, after the precipitate has settled, is the hyposulphite solution required. If too strong, it can easily be reduced by further dilution with pure water. The strength I prefer is such, that  $\frac{1}{4}$  litre of fully ærated water requires about 5 c.c. of the hypo. solution. It may, however, be used both stronger and considerably weaker.

In conclusion, I will give the degrees of æration found at various points of the river Thames on two days (Aug. 9 and Sept. 2, 1882), a comparison between which and the amount of oxygen absorbed from permanganate, as given in my paper printed in the July number of the ANALYST, will be found interesting. Temperature of the water Aug. 9, 65·5° F.; on Sept. 2, 60·5° F.

	Locality.		Percentage of Æration.
Aug. 9	Richmond Bridge	.. ..	100
	Kew Bridge	.. ..	100
	Hammersmith Bridge	.. ..	67·3
	Westminster Bridge	.. ..	54·5
	Tunnel	.. ..	25
Sept. 2	Barking Creek	.. ..	19·7
	Jennington Point	.. ..	23·3
	Erith	.. ..	23·7
	Northfleet	.. ..	38·1
	Coal House Point	.. ..	52·6
	Hole Haven	.. ..	75
	Southend	.. ..	96

It will be seen that the river starts fully aerated at Kow, becomes gradually deprived of its oxygen as it passes the metropolis, and finally re-acquires its proper amount. At first I was under the impression that even at Southend the river had not fully recovered its aeration; but further experiments proved that this apparent deficiency was due to the fact that sea water, such as the water at Southend was, and usually is, at least at high water, is not capable of absorbing so much oxygen as river water, and that, in fact, the 96 per cent. found, by comparison with river water, represents fully aerated sea water.

Much more might be said regarding this process, and its applicability to water analysis, in which I am at present engaged; but I have already detained you too long, and can only express a final hope that other members of our Society will take up this extremely interesting subject, and bring their experience before us.

#### NOTE ON THE DETECTION OF FLUORESCENT SUBSTANCES IN LARD OIL.

By PROF. TICHBORNE, F.I.C., &c., Analyst to the County of Longford.

*(Read at the Meeting on August 1st, 1885.)*

IN 1874, I proposed that fluorescences should be used to detect adulteration. (Proceedings of the Royal Irish Academy, sec. 2, vol. 2.)

That is to say, presuming the adulterant is fluorescent, and the substance under examination is not, we possess a mode of detection in which the delicacy of the test is in inverse ratio to the amount of adulteration present. Or, in other words, fluorescent substances are better perceived in dilute solutions than in strong ones. The most simple and efficient method of determining this fluorescence, when not very evident to the eye, is to put a drop of the pure substance beside a drop of the adulterated specimen, upon a plate of black glass or ebonite, and then to view both by the light obtained from the induction coil spark. Any difference in colour indicates fluorescence.

To give an instance of the application of this method, I received a sample of lard oil lately, which, although sold as "off colour," was guaranteed to be pure lard oil. It clogged the machinery, and my opinion was asked about its purity. I certified that it contained about 10 per cent. of mixed oils of a very miscellaneous character, which included seed oils and petroleum. It was evident that in every ton of this oil, about 2cwt. of miscellaneous "foots" had been introduced for the purpose of disposing of them in an oil, which was avowedly not A. 1. The quantity of petroleum oil, which was easily detected by the above method, must have been very small.

#### ON THE USE OF AMMONIUM CITRATE IN THE ANALYSIS OF PRECIPITATED PHOSPHATE.

By H. H. B. SHEPHERD, F.I.C., F.C.S.

*(Read at the Meeting on 1st August, 1885.)*

My object in writing this paper is not to add another to the many plans which have been proposed for the estimation of "retrograde" or "reverted" phosphate, but to direct further attention to its use as a means of distinguishing between such finely

divided phosphates as precipitated phosphate of lime, guano phosphate, etc., and the harder class represented by the various descriptions of mineral phosphate. It is, I think, admitted by most agricultural chemists that the former are vastly more assimilable, and therefore more valuable, than the latter, and yet analyses made in the customary manner give no indication of the decided difference that exists between the two.

Now, the ammonium citrate method, whatever objections may be raised to its employment as a direct test of assimilability, does certainly furnish some insight into the nature and mechanical condition of a phosphate, and is especially useful as a means of identifying phosphate of lime prepared by precipitation. The various methods involving the use of either ammonium citrate or free citric acid which have been proposed for this purpose, may, I think, be classified under the three following heads, viz., those involving the use of—

- (a) An ammoniacal solution of ammonium citrate, either with or without the application of heat;
- (b) A neutral solution of ammonium citrate;
- (c) A dilute solution of free citric acid.

Of these the employment of neutral ammonium citrate appears to me to have the best claim. Ammoniacal citrate may answer well for the estimation of reverted phosphate in superphosphate, but when applied to precipitated phosphate of lime it dissolves out only a part of the phosphoric acid, giving results very much below the truth. Neutral citrate, however, while nearly as good a solvent for precipitated phosphate as weak citric acid, exerts less solvent action than the latter upon ground mineral phosphates.

Having occasion recently to test some samples of precipitated phosphate of lime prepared from the iron slag obtained in working the Thomas dephosphorising process, I tried the solvent action of ammonium citrate upon them in the following way:—  
2½ grams. precipitated phosphate were digested with 100 c.c. of Joulie's strongly ammoniacal solution, containing 40 per cent. citric acid, for one hour, without the application of heat, then filtered and the residue washed with 150 c.c. of a mixture of equal parts of solution and water. The following were the results:—

	P <sub>2</sub> O <sub>5</sub> sol. in water.	P <sub>2</sub> O <sub>5</sub> sol. in citrate.	P <sub>2</sub> O <sub>5</sub> insol.	Total P <sub>2</sub> O <sub>5</sub> .
A .....	·28 .....	12·67 .....	17·28 .....	30·23
B .....	1·39 .....	15·45 .....	14·58 .....	31·42
C .....	1·34 .....	14·96 .....	15·90 .....	32·20

As will be seen, only about one half the total phosphoric acid dissolved.

I then tried Joulie's ammoniacal solution at a temp. of 80°C upon sample C, and compared it with the neutral solution method used by chemists in Germany for the analysis of Thomas' precipitated phosphate. This method is carried out as follows:—  
250 grams. of ammonium citrate are dissolved in water, accurately neutralised with ammonia (which is best done by means of litmus paper), and the solution diluted to a sp. gr. of 1·09. 2 grams. of the precipitated phosphate are digested with 100 c.c. of this solution for half-an-hour, at a temp. of 50°C. It is then filtered and washed three

times with a mixture of equal parts of solution and water. The residue, while still moist, is transferred to a platinum capsule, covered with a concentrated solution of sodium carbonate, dried, raised to a red heat, dissolved in hydrochloric acid, evaporated to dryness, taken up with nitric acid, filtered, and the phosphoric acid determined in the filtrate by the molybdenum process. The difference between the total phosphoric acid and that insoluble in citrate gives the phosphoric acid soluble in citrate. The following were the results obtained :—

	$P_2O_5$ sol. in water.	$P_2O_5$ sol. in citrate.	$P_2O_5$ insol.	Total $P_2O_5$
By Joulie's strongly ammoniacal solution, at 80°C for 1 hour.	1.34	18.51	12.35	32.20
By the neutral solution method.	1.34	29.06	1.80	32.20

Although the neutral citrate solution gives much higher results than the ammoniacal one, it does not, as will be seen, dissolve out the *whole* of the phosphoric acid present.

The following are some analyses of recently imported parcels of Thomas' precipitated phosphate made in Germany by this process :—

	$P_2O_5$ sol. in citrate.	Total $P_2O_5$
I.	30.06	35.24
II.	29.64	34.53
III.	28.30	31.72

#### METHOD OF SEPARATING AND DETERMINING ARTIFICIAL COLOURS IN BUTTER, FATS AND OILS.

By EDWARD W. MARTIN.

(*Read at the meeting on the 1st August, 1885.*)

To 5 grms. of the dry butter fat, or any dry fat, add 25 c.c. of bisulphide of carbon and shake gently until the solution is complete. Now add 25 c.c. of water, made slightly alkaline with caustic soda or potash, and shake again gently. The alkaline water will dissolve out the colouring matter. This can be separated out and determined, qualitatively by the spectroscope or other means, and quantitatively by making up a standard solution of annato, or whatever the colour is, and applying the colorimetric method.

I have also noticed that "oleo" and some oils emulsify, to a certain extent, in the above method, while butter does not.

If the butter or fat is only slightly coloured, a larger quantity than 5 grms. should be taken. Oleo separates out slowly, butter at once, that is, as far as my few experiments have gone.

#### ON THE PROCESSES AND STANDARDS IN USE AT THE MUNICIPAL LABORATORY OF THE CITY OF PARIS.

By DR. MUTER.

(*Continued from our last.*)

##### (1) WATER.

The first point insisted upon is that with any sample delivered at the Laboratory the fullest information must be given as to its source and surroundings. The mode of analysis for sanitary purposes is something similar to that recommended by the Water



Committee of our Society; the oxygen consumed from permanganate and the albuminoid ammonia being depended on to the exclusion of organic carbon and nitrogen. The latter process is rejected because it "is subject to many causes of error, and is of so extremely delicate a nature as to be almost abandoned at the present time." The permanganate results are stated in terms of oxalic acid equivalent to the permanganate used, and the process is performed by heating the acidulated water to 90° C., and dropping in the reagent until a permanent pink is obtained. According to our views, this temperature is much too high, and there should be a time allowance and a definite indicator employed, so as to obtain results mutually comparable among analysts. What happens at 90° C. in a water, rich in chlorides, would be worth trying; and, again, surely the expression in "oxygen consumed to oxidise the organic matter" is more simple than the oxalic acid idea above referred to. Use is specially made of the hydro timetric method for rapidly ascertaining the nature of the mineral contents of a water. Soap solution is used by titration: (1) On the original sample; (2) After precipitation by ammonium oxalate; (3) After boiling; (4) After boiling and precipitation with ammonium oxalate. Nitrates are estimated by the aluminium or indigo methods, but the very convenient Crum method in the nitrometer appears to have been entirely overlooked. Nitrites are specially estimated by a solution of 5 grammes of metaphenylenediamine in a litre of water slightly acidulated with sulphuric acid. It is used colorimetrically in the presence of sulphuric acid against a solution of pure potassium nitrite, containing .1 millegramme of that salt in each c.c. This solution is prepared by decomposing .406 grammes pure argentic nitrite with an equivalent amount of potassium chloride, and making up to a litre with water. All town waters are specially tested for lead, copper, and zinc. A special feature is also made of the dissolved gases, which are boiled off into a graduated receiver over mercury, and submitted to the action of absorbents, or, more commonly, the oxygen only is estimated by the process of Schutzenberger.

The standards adopted are as follows (*translated for convenience into grains per gallon*):—

- (1) The total residue may range from 35 to 42 grains.
- (2) The calcium sulphate shall not exceed 17.5 grains.
- (3) The chlorine shall not exceed 1.05 grains. If the chlorides be alkaline, no great inconvenience is to be noted, even should they exceed this amount, but if it be a case of calcium chloride, the water is to be looked upon as unhealthy.
- (4) The permanganate indication should not show organic matter equal to more than .35 grains oxalic acid per gallon, and *the organic matter so indicated should not be nitrogenous*. (I have tried the standard, and I find that in ordinary water it somewhat nearly corresponds to the limit of .04 "oxygen consumed in 15 minutes" at 80° F., proposed some years ago by the late Mr. Wigner and myself. The concluding expression is somewhat vague, and one can scarcely imagine that a water is to be condemned unless it be perfectly free from albuminoid ammonia, but no limit is given for the latter indication other than these words.)

- (5) The ammonia should not exceed .07 grains.
- (6) The quantity of metals precipitable by sulphuretted hydrogen should not exceed .07 grains.
- (7) The amount of iron should not exceed .21 grains.
- (8) No sulphuretted hydrogen should be present.
- (9) 100 c.c. of the water should contain 3.25 c.c. of dissolved gases, of which 10 per cent. should be carbonic acid, 33 per cent. should be oxygen, and no inflammable gases, such as carburetted hydrogen, should be present.
- (10) All potable water should contain a trace of silica.

Considerable stress is laid upon the microscopic examination. The water is allowed to stand for 12 hours, and the supernatant liquid is examined, first by an objective of 100 diameters, and subsequently by one of 500 diameters. A drop is then evaporated upon a covering glass, the residue stained with methylene blue, mounted in Canada balsam, and examined for microbes by the 500 power. The deposit is also examined by the same two objectives, using successively the following stains—viz., solution of iodine, solution of carmine in glycerine and alcohol, and methyl violet. By these means, starch, vegetable tissues, and bacteria are respectively made manifest. Should the presence of microbes be suspected, resort is had to a cultivation by Koch's, or Angus Smith's methods; the former on a plate covered with sterilised nutrient gelatine, or the latter in tubes containing sterilised solution of gelatine and sodium phosphate, and closed by cotton wool. Mr. Heisch's sugar cultivation is also employed. In criticising these methods, I may say that all yield good results, but in my hands the phosphatised gelatine has given some very striking effects. As some members may not have noticed the process, I give it in some detail. The first point is to obtain some good fresh gelatine in very thin *laminae*, and to thoroughly dessicate the same at the highest possible temperature, and enclose in a carefully cleaned and dessicated bottle. 100 c.c. of redistilled water is well boiled in a clean flask, the mouth is closed by cotton wool, and the whole allowed to cool to 90° F. Four grammes of the gelatine, and 2 centigrammes of sodium phosphate, are now placed in the flask, and the mouth having been again closed, agitation is persisted in until entire solution takes place. A fresh egg is now broken, and a little of the white is added to the contents of the flask, which are then boiled and filtered through a paper sterilised by heat. 25 c.c. of the filtered liquid is then mixed with an equal volume of the suspected water in a tube closed by cotton, and kept at a temperature of 60° to 70° F. A comparison tube is also set up with redistilled and previously boiled water. With really infected water, a cloudy layer of bacteria will form within a comparatively short space of time, and the gelatine will liquify from the surface downwards, and will disengage putrid smelling and inflammable gases.

In bringing to a conclusion this branch of the subject, we may, with justice, note that while the processes employed are very similar to those in use in the laboratories of British analysts, yet the standards are not exactly what we would use in every case, while our methods are so arranged as to give more, according to our idea, mutually comparative results in certain respects. A point is specially noticed with which we all

agree—viz., that in judging any water, regard should be had to the natural constitution of the average waters of the districts.

## (2) WINE.

As might be supposed, this constitutes in France an exceedingly important branch of food analysis, and the report thereon actually occupies 113 pages, and includes the results of nearly 10,000 analyses. To show fully what has been done, I propose to follow the three chief heads of the report.

(a) *The detection of added water, and the general system of analysis pursued.*—In determining the first point, no less than 400 analyses have been made, at the laboratory, of wines of known source and purity. Again, it has been necessary to find a good *commercial* standard for the city of Paris, and for this purpose, nearly 6,000 samples have been examined. The chief factor relied on for the detection of water in wine is a mutual comparison of the extract and alcohol. The latter alone goes for nothing, because when water is added, a little common alcohol, and some colouring matter, are, as a rule, also put in. Here is a case which is given as illustrative of the *modus operandi* of the reducers of wine. A sample submitted by a merchant about to purchase, showed (no foreign colouring matter being present):—

Alcohol	..	..	..	..	..	13·2	° by volume.
Extract	..	..	..	..	..	27·5	grammes
Acidity	..	..	..	..	..	6·3	per
Ash..	..	..	..	..	..	5·0	litre.

On this sample the merchant placed an order, and after receiving the consignment he submitted another sample, which gave:—

Alcohol	..	..	..	..	..	13·1	° by volume.
Extract	..	..	..	..	..	17·5	grammes
Acidity	..	..	..	..	..	4·0	per
Ash..	..	..	..	..	..	4·1	litre.

The intensity of colour was identical with that of the former sample, but the presence of fuschine was proved. The calculation of these results came out as follows:—

100 litres of sample A	=	2750	Extract.
100	„	„	B = 1750
			Difference
			1000

$$\text{then } \frac{2750}{1750} = \frac{100}{x} \text{ therefore } x = 63\cdot63 \text{ of sample A,}$$

$$\text{and } 100 - 63\cdot63 = 36\cdot37 \text{ } ^\circ \text{ of water added.}$$

The calculation of the acidity and ash also agree with this view.

After the above-mentioned experimental research, the following standard was adopted and made legal, viz.:—*The amount of added water in all wine which is not sold as being of any special brand, shall be calculated on the basis of 12 per cent. of alcohol by volume, and 24 grammes of dry extract per litre.*

With reference to the general mode of analysis, the alcohol is estimated by the usual method, but the determination of the extract is special, because it has been found that the amount will vary unless exact conditions are observed. 25 c.c. of the wine are, therefore, introduced into a tared platinum cylinder, 2·8 inches in diameter, and ·9 of an inch deep, and the cylinder is placed on a perforated plate so as to exactly touch the surface of boiling water in a large bath, having its water maintained at a constant

level by means of an automatic feed. After seven hours residues so treated attain a constant weight.

For ascertaining the degree of plastering (*an operation which is held to be illegal, provided the potassium sulphate exceeds 2 grammes per litre*) a quick method is employed. A solution is made containing 5.608 grammes of barium chloride, and 100 c.c. of strong hydrochloric acid per litre. Two tubes (A and B) are taken each with 20 c.c. of the wine, and to A is added 5 c.c., and to B 10 c.c. of the barium chloride solution. After twenty-four hours standing, a little of the clear liquid is poured off from each, and tested with barium chloride. If now A gives no precipitate, the wine is not plastered, and if B gives none, then the plastering is beneath the standard. If a precipitate forms in B, then the usual estimation must be performed. The actual maximum of potassium sulphate found in any genuine unplastered wine by M. Marty, who performed a very large number of experiments, was .583 grammes per litre.

(*To be continued in our next.*)

#### CONCLUSION OF THE SOCIETY'S PROCEEDINGS.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

A NEW REACTION FOR ALBUMEN.—By D. Oxenfeld.\* *Centralblatt für die Medicinischen Wissenschaften*, 1885, p. 209.—If a solution containing albumen be acidulated with formic acid, and a solution of gold chloride (0.1 per cent.) be added, drop by drop, the mixture being warmed, bubbles of gas are formed on the sides of the test-tube; the solution turns first rose-red, and then assumes a beautiful purple-red; on further addition, a blue colour; finally, a blue flocculent precipitate separates out. The rate of change to blue is in proportion to the dilution of the solution of albumen. The blue or violet coloration is also caused by other organic substances, such as grape-sugar, starch, glycerine, leucine, tyrosine, creatine, uric acid, and urea. Pure gelatine gives a dichroic brown or reddish coloration. Impure gelatine (probably containing albumen) gives, when sufficiently concentrated, the purple colour. Gum solutions give also a beautiful purple-red colouration, which differs, however, from the albumen reaction by changing to orange-yellow on the addition of potash or soda. The reaction can be made to succeed with pure albumen solution of a dilution of 1 in 1,000,000. The presence of salt, urea, uric acid, or grape-sugar does not interfere, when not present in excess; otherwise it is only necessary to use more formic acid and gold chloride.

F. H. H.

THE DETERMINATION OF CASEINE IN COW'S MILK.—By G. FRENZEL and T. WEYL.† *Zeitsch. für Physiologisch. Chemic.*, 9, 246.—The authors conduct the determination for technical purposes in the following way:—

30c.c. of the well-shaken milk are measured out with a pipette, and mixed with 60c.c. water; the mixture being well stirred, 30c.c. of a 1 per cent. solution of sulphuric acid are added. The caseine precipitate is brought (after having been allowed to stand for several hours in the cold) on to the filter with water, washed with alcohol and ether, dried, weighed, and ignited.

\* *Zeitsch. für Anal. Chem.* 24, 3, 479.

† *Zeitsch. für Anal. Chem.* 24, 3, 477.

Compared to the usual precipitation with acetic acid and carbonic acid, this method saves one operation (viz., the passing of carbonic acid), and according to the author affords a precipitate, which permits of easy washing, and a clear filtrate. On the other hand, the results obtained, to judge from the analyses accompanying the paper, are always about 2 per cent. less than the total amount of caseine. F. H. H.

NEW TESTS FOR GRAPE AND MILK-SUGAR.—By M. RUBNER.\* *Zeitsch. für Biologie*, 20, 397.—These new reactions are founded on the respective behaviour of the sugars with acetate of lead and ammonia.

If a solution of acetate of lead be added to a dilute solution of grape-sugar and ammonia dropped in the precipitate formed turns, on standing in the cold, gradually (quicker on warming) first yellow, then rose-red or flesh-coloured. It is advisable to take 1—1.5c.c. of acetate of lead solution of the strength generally used in the laboratories to every 20c.c. sugar solution. The author was able to obtain a reddish-yellow colouration with 5c.c. sugar solution of 0.02 per cent. strength.

A second reaction for grape-sugar is the following:—To the sugar solution is added a moderately large quantity of lead acetate, the mixture boiled some time, and ammonia dropped in until a permanent precipitate is obtained; the solution becomes then yellow or red, according to the concentration. After a time a precipitate of the same colour separates out, which, however, soon assumes a yellow colour. The acetate of lead should be for 10c.c. sugar sol. of 2 per cent. strength about 4grm., for the same quantity of a solution of 1 per cent. strength about 2grm.

Both reactions are peculiar to grape-sugar; cane-sugar and dextrine, treated in the same way, give no reaction.

*Milk-Sugar.*—If a solution of milk-sugar be boiled 3—4 minutes with acetate of lead, it turns yellow or brown. If ammonia be dropped in as long as the precipitate first formed dissolves, the liquid assumes at first a deeper yellow, and then an intense brick-red colour; finally it becomes turbid, and a pulverent precipitate of cherry-red to copper-red colour separates out. To make the test successful it is necessary to use a large quantity of lead acetate. The author obtained the best results with 8grms. of lead acetate to every 10c.c. milk-sugar solution of 2 per cent., or 4grms. to 10c.c. of 1 per cent. strength. The delicacy of the reaction extends to a solution of 0.02 per cent. strength.

These reactions can be used for the examination of urine. Phosphates, which would interfere, can be removed by boiling the urine with acetate of iron, or by filtering the precipitate obtained on adding the lead acetate. The test for milk-sugar in urine is interesting, as until now there has been no quick method for its detection. It is conducted thus:—10c.c. of the urine (freed from phosphates) are boiled a few minutes with 3grms. lead acetate, and ammonia added to the boiling hot solution. The author obtained in this way a perceptible colouration with a strength of 0.02 per cent.

F. H. H.

\* *Zeitschr. für Anal. Chem.*, 24, 3, 480.

ON THE DETECTION OF WHEAT-FLOUR IN RYE-FLOUR.—By L. WITTMACK.\*—*Zeitsch. für Anal. Chem.* 24, 3, 463.

About one grm. of the flour to be examined is well rubbed up with 50 c.c. water, and the mixture gradually warmed on the water-bath to exactly 62·5°C; on reaching this temperature, the beaker is removed from the bath, cooled with cold water, and the starch grains examined with the microscope. The rye-starch grains become, at 62·5°C, nearly all swollen up; the most burst, and almost all have lost their original form. The wheat-starch grains, on the other hand, remain mostly quite unaltered, as strongly refractive as normal starch grains, and show, consequently, sharp black borders, while the rye-starch grains, even when they keep their shape, show soft outlines. For the sake of comparison, it is best to make parallel experiments with pure wheat and rye.

By the means of this test, we should be able to detect an addition of 5 per cent. wheat to rye.

F. H. H.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

SECALE CORNUTUM.—*Zeitsch. für Anal. Chem.* 24. 3. 468.—Fresh ergot (Mutterkorn) gives, according to Koster†, a colourless extract on 2 grms. of the powder being shaken with 5 c.c. ether; whereas stale ergot gives a [yellow one. According to Bernbeek‡, the extract of the fresh substance with benzol, carbon bisulphide or ether is neutral, whereas the extract of the older (by decomposition of the fat contained therein) gives an acid reaction.

F. H. H.

ON THE DETECTION OF FATTY OILS IN MINERAL OILS, By Friedrich Lux (*Zeitsch. f. Anal. Chem.* 24. 2. 347).—The author finds his method upon the different behaviour of the fatty and mineral oils, when heated with potash, potassium, soda, or sodium. For instance, rape oil, when heated with potash or soda to a high temperature saponifies—stirring or shaking promoting the reaction. At a temperature of 250° C. rape oil gelatinises with potash or soda in five minutes; after 15 minutes the oil turns brown and solidifies to a buttery mass. On the other hand, mineral oils treated in the same way, become darker, but do not alter their state of aggregation. A. *Preliminary Test*: Detection of larger quantities of fatty oils (about 10 per cent. or more). To about 5 c.c. of the oil under examination, in a test-tube, is added a small piece of sodium hydrate, the liquid heated directly over the flame to boiling, and kept boiling for about one to two minutes. Larger quantities of fatty oil are detected by the peculiar empyreumatic smell given off, and by the solidification of the liquid on slightly cooling. If a negative result is obtained, one proceeds as follows:—B. The detection of smaller quantities of fatty oils (2 per cent. or less). Two middle-sized beakers are taken, of which the one can be so far pushed into the other, as to leave a space of about 1 — 2 cm. between the two. Into the larger beaker is brought enough paraffine to bring its surface half way up between the sides of the two beakers. The inside beaker is also filled with paraffine to the same height. A paraffine-bath, constructed in this way, cannot be over heated. The thermometer, hung in the inner beaker, should be kept at about 200 — 210°C. Two test-

\* Anleitung zur Erkennung organischer und anorganischer Beimengungen im Roggen und Weizenmehl.  
 † *Pharm. Zeit.* 29, 831,  
 ‡ *Pharm. Zeit.* 58, 829.

tubes are now filled with a few c.c. of the oil; to the one is added a few shavings of potassium, to the other a stick of potassium hydrate, so that the latter stands about one cm. above the surface of the oil. The two test-tubes are placed in the bath and the time noted, after 15 minutes they are taken out and allowed to cool. If the mineral oil contain as much as 2 per cent. fatty oil, it solidifies in one of the two test-tubes, generally in both, to a tough jelly.

F. H. H.

ON THE DETERMINATION OF POTASSIC BITARTRATE IN CRUDE TARTAR AND IN WINE LEES. By F. KLEIN.—*Zeitsch. für Anal. Chem.*, 24.3.379.—An interesting paper on the methods in use for the determination of the useful constituents of raw products containing tartaric acid.

The methods in use are:—

- (1.) The titration method.
- (2.) The "*methode à la casserole*" (used in France).
- (3.) The total acid method.
- (4.) The bitartrate analysis.

The first of these methods (used in all countries) is, by itself, practically useless; but, in combination with (3) and (4), it may be used to determine the apparent wine-stone.

The third method is chiefly used in the determination of the value of crude tartar and wine-lees poor in wine-stone but rich in calcium tartrate, such as are used in the manufacture of tartaric acid. There are several different variations in use; the author prefers Warington's, and uses it in the following way:—A sufficient quantity of the substance under examination is weighed out to contain 1.8 — 2.2 grms. potassium bitartrate. This is exactly neutralised, 3 grms. potassium oxalate added, the mixture warmed a quarter of an hour on the water-bath, and, on cooling, filtered and washed. The filtrate and the wash-water are evaporated to about 40 c.c., mixed (after cooling) with 5 grms. potassium chloride and 3 c.c. of a 5 per cent. solution of citric acid, and either stirred 15 minutes, or allowed to stand over night. It is then filtered, washed with a 10 per cent. solution of potassium chloride (which had been previously saturated at the ordinary temperature with pure wine-stone), until a drop of the filtrate imparts the same colour to a strip of litmus-paper as a drop of the wash-liquid itself. The precipitate, together with the filter, are brought into a dish, and titrated with  $\frac{N}{2}$  potash solution. The results are satisfactory. The *bitartrate analysis* determines the amount of real pure potassium bitartrate. For this the author knew of no good method, and has, therefore, worked out the following one for himself:—

1.969 grms. of a pure wine-stone (containing 99.87 per cent. potassium bitartrate) were weighed out, and dissolved in hot distilled water; the solution was evaporated to 44 c.c., 5 grms. potassium chloride added, the whole stirred for a short time, and allowed to stand over night. The next morning, the precipitate was brought on to the filter, which had previously been moistened with the wash-liquid. [The latter is prepared in the following way:—In a litre-flask are mixed 5 grms. finely-powdered pure wine-stone, with 200 c.c. distilled water; after well shaking, 25 grms. potassium chloride

are added, and the flask filled up to the mark with water. After several hours' intermittent shaking, the liquid is filtered, and is then ready for use.] The precipitate of potassium bitartrate on the filter was then washed with 15 c.c. of the wash-liquid, brought, together with the filter, into a dish, heated with distilled water, and titrated with  $\frac{N}{2}$  potash solution. The indicator used was phenol-phthalein. The titration gave 99.60 per cent. potassium bitartrate, instead of 99.87 per cent. According to the author's experiments, the presence of even 25 per cent. calcium tartrate, or an equivalent amount of acid potassium phosphate, does not interfere with the analysis.

The author carries out the analysis of raw products in the following way:—Sufficient substance is taken to contain about 1.8–2.2 grms. potassium bitartrate. The weighed substance is boiled with a sufficient quantity of distilled water, and, after allowing the salt to settle (which generally requires about three minutes), decanted through a moistened filter, this operation being repeated five times. Finally, the salt is rinsed on to the filter with hot water, and washed with boiling water, until a drop no longer reddens litmus-paper. The analysis is then proceeded with as above.

The author adds a system for separating the raw products containing tartaric acid into their essential components, which consists in a judicious application of the three methods described above.

They are:

- a The titration method.
- b The total tartaric acid method.
- c The bitartrate method.

Of these the titration results are expressed in wine-stone percentages. Then  $a - c$  gives the apparent wine-stone, *i.e.*, the sum of all contained acid bodies expressed in wine-stone percentages.

$b - c$  = percentage of dry calcium tartrate (wine-stone and calcium tartrate having the same molecular weight); from this can be calculated the crystallised calcium tartrate.

*Example*—Italian wine lees:—

$a$	=	35.20	per cent. in wine-stone percentages.
$b$	=	36.08	" " "
$c$	=	29.60	" " "

From this we have:—

$a - c$	(35.20 — 29.60)	nominal wine-stone	=	5.60	per cent.
$b - c$	(36.08 — 29.60)	anhydrous calcium tartrate	=	6.48	per cent.
		equiv. crystallised tartrate	=	8.96	"
$c$	=	effective wine-stone	=	29.60	"

F. H. H.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

A NEW METHOD FOR THE VOLUMETRIC DETERMINATION OF CHLORINE—By E. BOHLIG. *Zeit sch. für Anal. Chem.* 24, 3, 408.—This method is based upon the great insolubility of silver oxalate, and its almost instantaneous decomposition in neutral solution, by dissolved metallic chlorides.



A certain volume of the solution, containing the hydrochloric acid or chloride to be determined, is measured off, boiled a few seconds with basic magnesium carbonate, and filtered; on cooling, an aliquot part of the filtrate is shaken up with dry oxalate of silver, and, after standing a short time, filtered. To half the original volume is now added concentrated sulphuric acid, and the whole titrated in the ordinary way with permanganate solution, previously standardised with  $\frac{N}{10}$  oxalic acid (1 c.c. permanganate sol. is then equal to 0.007 grm. chlorine.)

The small amount of the silver oxalate dissolved is, once for all, determined with the permanganate solution, and the titration corrected accordingly,

The author uses this method with great profit in water analysis. In the presence of organic substances, the oxalate formed is first precipitated with calcium chloride and ammonia, and the washed calcium oxalate titrated. F. H. H.

A NEW REACTION FOR TITANIC ACID—R. FRESENIUS. *Zeitsch. für Anal. Chem.* 24. 3. 410.—The reaction is brought about by the yellow liquid, which is obtained by the action of a solution of sulphurous acid on granulated zinc (hyposulphurous acid).

If this reagent be mixed with a moderately concentrated sulphuric or hydrochloric acid solution of titanic acid (for example, one, which in 1 c.c. contains 1.5 mgrm. titanic acid) an intense red colour, approaching a reddish-yellow, is immediately obtained, which soon turns reddish-yellow, then yellow, and finally fades away. The more free acid the solution contains, the quicker the change of colour. On re-addition of the reagent, the reaction is renewed. Shaken up with ether, the ether does not become coloured. If the solution contain in 1 c.c. 0.15 mg. titanic acid, or less, only the yellow colouration is obtained on addition of the reagent. With 0.02 mg. titanic acid in 1 c.c., the colour can only be seen by filling the test-tube nearly to the brim, and looking down through it. The delicacy of the reaction is thus the same as Weller assumes for his test for titanic acid with hydrogen peroxide.

It is to be remarked that the reaction can only then be used when no other acids or oxides are present of which the solutions are reduced with colouration. It is, for example, of no use in the presence of molybdic, tungstic, chromic, niobic, and vanadic acid.

As the reaction is brought about by hyposulphurous acid, and not by sulphurous acid or thio-sulphuric acid, a solution of titanic acid may be used as a reagent for hyposulphurous acid, and especially to distinguish it from sulphurous or thio-sulphuric acid.

F. H. H.

NOTE ON KJELDAHL'S METHOD FOR THE DETECTION OF NITROGEN.—By Dr. Pfeiffer and F. Lehmann, *Zeitsch. f. anal. chem.*, 243-388.

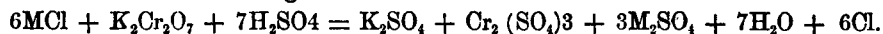
The authors have obtained very satisfactory results with Kjeldahl's method; they were, therefore, the more surprised, by the distillation of ammonium sulphate with 100 c. c. soda (500 c. c. NaHO to the litre) and zinc, to obtain a whole percentage of nitrogen more than was theoretically possible.

By making a number of experiments, they made the discovery that this error is due to the formation of hydrogen, and that its amount is proportional to the intensity of

the latter. This, however, is dependent upon the concentration of the soda (the quantity of zinc is of less importance). The hydrogen formed carries over soda solution in the form of fine spray. The authors' researches show that in half an hour's boiling (with a concentration of 50 c. c. soda solution of the above strength to 150 c. c. water), 12 c. c. hydrogen are given off; in another case (150 c. c. soda solution to 50 c. c. water), 200—300 c. c. hydrogen.

As the soda is generally used moderately dilute, the error usually remains small; but in certain cases it may become considerably larger; and to do away with the possibility of such a case arising, the authors have constructed a "safety tube," which is introduced between the distillation-flask and the condenser. This piece of apparatus frees the hydrogen from the soda spray. Its construction is simple. The tube coming from the flask is attached to a much wider one, the opening of the smaller tube into the wider being covered with an inverted platinum cone, on the top of which are a number of glass beads. By means of a curved tube passing through a cork in the upper mouth of the wider tube, the apparatus is connected with the condenser. F. H. H.

THE DETECTION OF CHLORIDES IN THE PRESENCE OF BROMIDES AND IODIDES.—By Dr. L. L. DE KONINCK. *Zeitsch. für anal. chem.* 24 376.—The author's method is intended to replace the bichromate one, which, according to him, is not to be depended upon, as the reaction does not always result in the formation of chlorochromic acid, but sometimes takes the following course:—



The method has been used by the author for four years; it is characterised by its simplicity, rapidity, and accuracy.

The solution containing the chlorides, bromides, and iodides is precipitated with silver nitrate (the latter in small excess), the precipitate filtered and carefully washed. It is then digested for a few minutes with 4—5 times its volume of a moderately concentrated solution (10—15 per cent.) of ammonium carbonate in the cold. The clear liquid is decanted through a small filter, the filtrate collected in a test-tube, and a drop of potassium bromide solution added. If the original substance contained a chloride, a precipitate of silver bromide is produced. By using potassium bromide instead of nitric acid, one is not liable to be deceived by a small quantity of silver bromide having been dissolved by the ammonium carbonate. If no chloride be present—*i.e.*, if the solution contain pure potassium bromide, only a slight turbidity is produced.

If the method is to be applied to the detection of chlorine in a mixture containing iodates, the silver precipitate must be heated (after filtering and washing) with sulphurous acid, which reduces the silver iodate to iodide, and prevents its influencing the reaction. The presence of cyanogen complicates the method. The silver precipitate must be heated until the silver cyanide is completely decomposed. The residue, reduced with zinc and dilute sulphuric acid, gives a solution containing a chloride, bromide, and iodide, which must be again precipitated with silver nitrate. Sulphur compounds do not act detrimentally. F. H. H.

**PHENYLHYDRAZINE AS A TEST FOR SUGAR IN URINE.**—By P. GROCCO,\* *Annali di Chim. Appl. Alla Farmacia*, 72, 258.—The author applies to the detection of sugar in urine the property possessed by grape-sugar of giving a heavy, almost insoluble, crystalline compound with phenylhydrazine (phenylglukosazon), [discovered by E. Fischer, *Berichte der deutsch, Chem. Gesell*, 17, 579.]

Fifty c.c. of the urine are warmed in the water bath with a solution of two grms. of pure hydrochlorate of phenylhydrazine and  $1\frac{1}{2}$  grms. of sodium acetate in 20 c.c. of water. In the presence of sugar, phenylglukosazon precipitates out, in 10-15 minutes, in stellate aggregations of yellow needles. If the precipitate separates out in the amorphous condition, it may be crystallised out of alcohol. According to the author, the test may be made with as little as 0.01 grms. of sugar in the litre; and it is more to be depended upon than other tests, as no other substance gives a similar re-action.

F. H. II.

---

### OBITUARY.

#### PATRICK WILLIAM EARL, OF DUBLIN.

ONE of the few surviving analytical chemists of the old school has just passed away from our midst in the person of Patrick William Earl, whose decease took place on Tuesday, the 4th inst., at his residence, 40 Westland Row, Dublin. Mr. Earl came of an old Irish family, the Errills (Anglicised Earl), of Carbury, co. Kildare, where a near relative of his, the Rev. Edward Errill, was parish priest in the time of the celebrated Bishop Doyle. At an early age the deceased became the pupil of the late eminent Dr. John Aldridge, Professor of Chemistry in the Cecilia Street School of Medicine, and succeeded Dr. Aldridge in the management of a large manufacturing laboratory attached to the establishment of Messrs. Bewley and Hamilton in Dublin, in which laboratory he spent his health, strength, and talent for a period of forty years, till the firm was about being merged into a limited liability company seven years ago.

Amongst the many chemists who graduated under the deceased, and who now occupy high positions throughout the United Kingdom and America, may be mentioned Sir Charles Cameron, M.D., President of the Royal College of Surgeons of Ireland.

---

### FOOD EXHIBITION.

WE are informed that a great International Cookery and Food Exhibition will be held at the Royal Aquarium, Westminster, in December. The Exhibition will comprise some 150 Classes, and Gold, Silver and Bronze Medals, together with valuable money prizes will be offered in Competition.

Schedules, Prize Lists and Entry Forms, will shortly be issued, and the exhibition is stated by its promoters to be intended to eclipse in magnitude, interest, and completeness, all affairs of a similar kind that have hitherto been held.

---

### BOOKS, &c., RECEIVED.

THE Baumé Hydrometers by C. F. Chandler, Ph.D.; Report of the Waters of the Hudson River together with an Analysis of the same by C.F.Chandler, Ph.D; The Sugar Industry of the United States, by H.W. Wiley; American Chemical Journal; American Chemical Review; American Druggist; American Grocer; British and Colonial Druggist; Brewer's Guardian; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Guardian; Cowkeeper and Dairyman's Journal; Independent Journal; Invention; Journal of the Chemical Society; Journal of Chemical Industry; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; Le Mouvement Hygienique; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; The Sanitarian; San Francisco News Letter; Science; Scientific American; Scientific Californian; Society of Arts Journal; Students' Journal; The Western Medical Reporter.

---

### NOTICES TO CORRESPONDENTS.

All communications as to literary matters to be addressed to the Editorial Department, 325, Kennington Road, London, S.E.

---

\* *Archiv. der Pharm.* 23, 117.

# THE ANALYST.

OCTOBER, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY—	
(a) OPERATION IN DUBLIN OF THE ADULTERATION ACTS—SIR C. CAMERON ..	175	(a) ADULTERANT OF CARBON BISULPHIDE—M. VAN ENGELN ..	189
(b) ON THE PROCESSES AND STANDARDS IN USE AT THE PARIS MUNICIPAL LABORATORY—DR. MUTER ( <i>continued</i> ) ..	179	(b) SEPARATION OF NICKEL AND COBALT—ILINSKI AND KNORRE ..	189
ORIGINAL ARTICLE:—		(c) DETECTION OF NITROGEN IN ORGANIC COMPOUNDS—C. GRAEBE ..	190
MUSTY BREAD—A. PERCY SMITH, F.I.C. ..	181	(d) TESTS FOR BILE IN URINE—C. DEUBNER ..	190
MONTHLY RECORD OF RESEARCHES IN FOOD ANALYSIS:—		(e) QUANTITATIVE DETERMINATION OF NITRIC ACID—WILDT AND SCHEIBE ..	190
(a) ESTIMATION OF FREE SULPHURIC ACID IN VINEGAR—B. KOHNSTEIN ..	184	(f) ESTIMATION OF CYANOGEN IN A MIXTURE OF GASES—G. JAQUEMIN ..	190
(b) BEHAVIOUR OF SERUM ALBUMEN IN PRESENCE OF ACIDS AND NEUTRAL SALTS—E. JOHANNSEN ..	184	(g) SEPARATION OF ALKALOIDS FROM HUMAN REMAINS—H. BECKURTS ..	191
(c) ALBUMINOUS COMPOUNDS IN MILK—J. SEBELIEN ..	184	(h) VOLUMETRIC ANALYSIS OF MANGANESE—W. HAMPE ..	191
(d) RAPID TEST FOR OLEOMARGARINE—PIALLAT ..	185	(j) MOLYBDIC ACID RESIDUES—W. VENATOR ..	191
(e) PETROLEUM ETHER FOR THE EXTRACTION OF WET SUBSTANCES—A. GAWALOSKI ..	185	(k) QUANTITATIVE ANALYSIS BY ELECTROLYSIS—A. CLASSEN AND R. LUDWIG ..	192
(f) DETECTION OF COLOURING MATTERS IN WINE—P. N. ARATA ..	186	(l) DETERMINATION OF ALUMINA AND IRON IN THE PRESENCE OF PHOSPHORIC ACID—R. LUCION ..	192
MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—		(m) DETERMINATION OF TIN IN TIN SCORBLE—R. FRESSENIUS ..	193
(a) ALOIN—J. DIETRICH ..	186	FOOD NOTICES ..	194
(b) DETECTION OF TURPENTINE IN OIL OF LEMON—G. HEPPE ..	187	CORRESPONDENCE ..	194
(c) CUPREOL—O. HESSE ..	187	BOOKS RECEIVED, &c. ..	194
(d) DETECTION AND PROPERTIES OF ANTI-PYRIN—O. SCHWEISSINGER ..	188		
(e) ADULTERATION OF ARGOL—ZIEREK ..	189		

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

### NOTE ON THE OPERATION IN DUBLIN OF THE ACTS RELATING TO ADULTERATION.

By SIR CHARLES A. CAMERON, President of the Royal College of Surgeons, Public Analyst for the City of Dublin.

(*Read at the Meeting, 1st August, 1885.*)

In 1860 the first general act relating to adulteration was passed. It came into operation on the 6th August, and was entitled an Act for preventing the adulteration of articles of food and drink (23 and 24 Viet., c. 84). Dr. Letheby was shortly afterwards appointed public analyst for the city of London, and Dr. Hill was, I think, in 1862 elected public analyst for Birmingham. In 1862 I was elected Public Analyst for Dublin, and I believe that no other appointments were made under the provisions of this Act. The defects in this measure were chiefly as follows:—It did not provide for the appointment of inspectors to obtain articles for analysis; it did not deal with the adulteration of drugs; it vested in the Court of Quarter Sessions the appointment of analysts for districts other than boroughs—a correct procedure so

far as England and Wales were concerned, but an incorrect one so far as it related to Ireland, where the Court of Quarter Sessions have no fiscal business to transact and are merely a tribunal. The purchaser of an article which was to be submitted for analysis was obliged to notify the circumstance to the vendor, in order that the latter might have the opportunity of accompanying the purchaser to the analyst. This provision in the act was an inconvenient one, and rendered the appointment of analysts for remote country districts, useless.

So far as I can learn—having made due enquiry—the Act of 1860 proved to be a dead letter everywhere save in Dublin. It is certain that in no other place was there any conviction obtained under its provision. In Dublin, there are two officials termed deputy clerks of the markets. At my suggestion in 1863 those officers were directed by the Corporation to purchase articles for analysis not in their capacity as officers, but as citizens. They purchased milk, tea, sugar, flour, bread, coffee, mustard and other articles, which were analysed, and a large proportion of the specimens found to be adulterated. The milk was especially found to be largely sophisticated. In this year I adopted for milk a standard of 12 per cent. of solids, but I never advised a prosecution unless the solids were below 11.5 per cent. In stating the amount of adulteration I have always expressed it as so much per cent. added to the pure milk. For example, a gallon of water is added to a gallon of milk. It may in such case be said either that the mixture is milk containing 50 per cent. of added water or that it is milk to which 100 per cent. of its weight of water had been added as an adulterant. I invariably employ the latter method, which is now well understood by the Dublin police magistrates. It conveys a better idea of the extent of the adulteration than the other method. To say that milk contains 66 per cent. of added water does not convey clearly to most minds that two gallons of water have been added to a gallon of milk. More than 100 convictions were obtained in Dublin under the operations of the Act of 1860. They referred in great part to adulterated milk; but the vendors of various other adulterated articles were fined.

In 1872 (10th August) an Act was passed to amend the law for the prevention of the adulteration of food, drink and drugs (35 and 36 Vict., c. 74). It dealt with the adulteration of drugs; it increased the penalty from £5 to £20; it enabled the local authorities to appoint food inspectors; it evidently meant to abolish the necessity for giving notice of intention to have articles analysed, but left the point in a state of ambiguity, the Irish Court of Queen's Bench being equally divided for and against the necessity of notice. It also transferred the power of appointing Public Analysts in Ireland from the Courts of Quarter Sessions to the Grand Juries, who in Ireland are fiscal bodies.

Immediately after the passing of this Act a food inspector was appointed by the Corporation, and in the following year a second inspector was appointed.

On the 27th April, 1874, the House of Commons appointed a committee to enquire into the operations of the laws relating to adulteration. Three days later the Committee commenced to assemble; they held 14 meetings, and examined 58 witnesses, of whom nine only were chemists, namely, Messrs. Hassall, Voelcker, Tidy, Macadam, Wanklyn,

Allen, Bartlett, Sutton, and Cameron. The great majority of the witnesses were persons engaged in the sale of articles liable to adulteration. The evidence of the chemists showed that the Act of 1872 had been productive of good, whilst the testimony of the dealers was chiefly to the effect that it had inflicted injury upon many honest traders, chiefly from want of a proper understanding as to what *does* and what *does not* constitute adulteration, and also from the inexperience of the majority of the analysts. The Committee recommended certain changes in the Act, some of them in the interest of the vendor; and upon the lines recommended by the Committee the present Act, called the Sale of Food and Drugs Act, was laid down (38 and 39 Vict., c. 63, 1875). It enabled the purchasers of articles intended for analysis to send them by registered letter—a change which enabled many of the Grand Juries, to appoint chemists in distant towns to act as analysts for counties. It did not provide for the publication of the name, address, and offence of persons convicted a second time, a clause to that effect having existed in the two previous Acts. This omission is to be regretted. In Dublin the conviction of “old offenders” was on many occasions published at their own expense in the advertising part of the newspapers, and placarded on the walls close to their shops. I cannot understand why this provision was omitted from the Act of 1875.

In my evidence before the committee I insisted upon the necessity of empowering the inspectors to purchase articles compulsory for analysis. The present Act imposes a penalty of £10 on persons who refuse to sell the articles asked for by the inspectors; the penalty ought to be £20, the maximum penalty for selling an adulterated article.

The Sale of Food and Drugs Act has been carried out pretty fully in Dublin, as, indeed, were the two earlier Acts, so far as it was practicable. When they were first put in operation there was a pretty large amount of adulteration practised, as in other towns. Coffee and mustard were rarely met with in a pure state. The former, as a rule, contained chicory, burnt sugar, roasted grain and other impurities; the latter always was more or less composed of wheat or rice flour, and, in a few instances, of sulphate of calcium. Tea was occasionally, but not frequently, adulterated, chiefly with exhausted tea-leaves. Sugar was, except in two instances, always found to be pure. Bread was found to be adulterated with rice-flour, potatoes and alum; and flour with rice and alum.

In 1870 I made an examination of 123 specimens of confections. Those manufactured at three establishments were quite pure. The collections obtained at ten other shops contained poisonous pigments and other impurities in a large proportion of the items. Out of 40 confections coloured yellow only 2 owed their colour to saffron. One was coloured with gamboge, and all the others were coated with chromate of lead, in amounts varying from  $\frac{1}{400\text{th}}$  to less than  $\frac{1}{1000\text{th}}$  of the weight of the confections. The common sugar stick, sold at 1½d. per two ounces, contained the largest proportion of chromate of lead. Twelve articles, chiefly lozenges and “sugar almonds,” had a bright orange hue, due to the presence of a variety of chromate of lead. Thirty-eight of the specimens—comprising “peaches,” sugar almonds, lozenges, comfits, sugarstick, sugar balls, &c—had various shades of red, from

a faint pink to a bright scarlet. Of these, 36 specimens were coloured with cochineal, while 2 contained vermilion in the proportion of 4 grains per ounce of the confection, which was the cheapest kind of sugarstick.

At one time arsenite of copper was frequently used to impart a brilliant green colour to confectionary, but the numerous accidents that occurred from the employment of this poisonous pigment have so alarmed the public that green confectionary is rarely met with\*. Only one of the specimens above referred to was a small figure of a baby with a green frock on it. The colour was composed of a mixture of Prussian blue and chromate of lead. Blue is not a popular colour, only one specimen having streaks of this colour was contained in 123 samples. The pigment employed was ultramarine. Nine specimens were brilliantly coloured with mauve, magenta, &c. As the coal-tar dyes are liable to contain traces of lead, mercury and arsenic, and as their use for the purpose of colouring confectionary has been prohibited by the authorities in Paris, it would appear to be the safer plan not to place these dyes on any substance intended for human food. The quantity used, however, is so small, that no serious consequences are likely to arise from eating confections coloured with these substances.

An article of food containing more than a grain of chromate of lead per ounce may well be regarded as a slow poison. Soft water containing less than .1 grain of lead per gallon has often produced poisonous effects on individuals and families. Chromium is also a poisonous metal. Six grains of a salt of this metal injected into the jugular vein of a dog caused the death of the animal. Workmen engaged in the preparation of chromate of potash often suffer from an ulceration of the throat resembling that of secondary syphilis, and also from slow necrosis of the nasal bones. I have not the slightest doubt but that the use of confectionary coloured with chromate of lead produces a large amount of infantile disease.

Vermilion was at one time an official medicament, but it is no longer administered internally, though still employed in mercurial fumigations. It constitutes the basis of Boerhaave's red pill—a celebrated nostrum of former days. There is a general belief in the inertness of this salt of mercury; but I find that it is capable of producing mercurialism, when given in very large doses. In 1½d. worth (2ozs.) of sugarstick I found 8 grains of this salt. As the dose of calomel—one of the mildest compounds of mercury—for a young child is only a grain, it is evident that vermilion, notwithstanding its great insolubility and comparative inertness, if consumed by a child at the rate of even 2 or 3 grains per diem, would produce some ill result. From experiments which I made at the Dublin Lock Hospital, I found that patients put upon large doses of vermilion became after a time mercurialized. Vermilion is liable to be adulterated. The Chinese vermilion is generally very pure, but cheaper varieties may contain minium. In the vermilion used to colour the confectionary which came into my possession, I found nearly 20 per cent. of red lead—a far more active poison than mercuric sulphide. Lozenges purchased at two of the 13 sugar confectioners' shops contained from 12 to 40 per cent. of an insoluble white clay, known in the trade under the term of *terra alba*. The "peaches," sugar almonds, lozenges and comfits contained

\* Turnip leaf pulp is sometimes used in Dublin to produce green confections.

rice-starch and gum arabic. Cough lozenges and Bath pipe contained gum, sugar and extract of liquorice, a few of them being slightly medicated by the addition of opium and camphor (probably in the form of paregoric elixir). Small quantities (under 3 per cent.) of plaster of Paris were found in the Bath pipe and cough lozenges, but they were probably derived from adulterated liquorice extract.

A figure of a baby in its cradle had the following composition:—The cradle was composed of a mixture of plaster of Paris (calcic sulphate) and sugar; the body of the baby was sugar and rice-starch; its eyes were Prussian blue; its cheeks were tinted with cochineal; and its dress was painted with chromate of lead.

The ten sugar confectioners above-mentioned, whose wares I have now described, were prosecuted before the police magistrates. Five of them, on paying costs (£3), and promising to abandon the use of poisonous pigments, were "let off with a caution." The others were fined respectively £5, and £3 costs; £1, and £1 costs; £1, and £1 costs; 10s., and £3 costs; and 10s.

A great many forms of adulteration have been exposed in Dublin through the agency of the Acts relating to adulteration. Since the year 1870, nearly 700 persons have been convicted in Dublin for selling adulterated food, and the fines imposed upon them have amounted to nearly £3,200. At present very few articles are liable to adulteration save milk and butter. None of the specimens of coffee, tea, sugar, mustard, pepper and liquors, lately collected for analysis, have proved to be adulterated.

I have occasionally taken advantage of the provision of the section of the Dublin Improvement Act of 1849 to reach food adulterators who could not be got at readily under the provisions of the Sale of Food and Drugs Act. The section to which I refer is that which provides a penalty of £1 upon any one who sells any article of food adulterated or fraudulently made. The simplicity of this section I should be glad to see imitated. It does not provide that the samples to be analysed are to be divided into three parts, and that notice of intention to have one of them analysed is to be given to the vendor. When a person purchases an article with the *bonâ fide* intention of consuming it himself, and subsequently is led to suspect its purity, the Dublin Improvement Act enables him to prosecute the vendor, should his suspicions be verified. The fact of the adulteration and the sale of the article have only to be proved to the satisfaction of the magistrate.

---

#### ON THE PROCESSES AND STANDARDS IN USE AT THE MUNICIPAL LABORATORY OF THE CITY OF PARIS.

BY DR. MUTER.

*Continued from our last.*

In expressing the ordinary analysis, no attempt is made to differentiate the sugars, but the total reducing power on Fehling's solution, expressed as glucose per litre, is given. The acidities, both fixed and volatile, are expressed as  $H_2SO_4$  per litre, and the indicator used is phenol-phthalein, 10 c.c. of the wine being first diluted to 200 or 400 c.c. in the case of the former; while the latter is done by saturating with titrated baryta water, boiling off the alcohol, adding an amount of standard acid equivalent to



the baryta used, and distilling to almost absolute dryness. The distillate is then titrated with weak standard alkali, using the phenol-phthalein as before.

The glycerine is estimated by obtaining a suitable residue *in vacuo* over anhydrous phosphoric acid in a platinum boat, weighing it, and then heating in a tube *in vacuo* to 120° C., and again weighing, thus distilling off the glycerine and getting it by difference. All the remaining estimations are made by the usual methods.

(b) *Detection of the addition of commercial glucose, fruit wine, and salicylic acid.*—The industry in fictitious claret is very great in France. In 1881 it reached the amount of 52,000,000 gallons, and has gone on increasing ever since. This liquid, known as *piquette* of raisins and dried fruits, is not sold instead of claret, but is employed to adulterate the genuine article. The process employed for its detection is as follows:—300 c.c. of the sample are mixed with a little well-washed beer yeast, and maintained at a temperature of 85° F., till all fermentation has ceased. The liquid is then filtered and placed in a dialysing apparatus, in which the outer water is constantly renewed automatically. The water is tested from time to time with the polariscope, and when nothing more comes out capable of causing rotation, the dialysis is stopped. The liquid is neutralised by boiling with chalk, and evaporated to dryness on the water bath, stirring towards the end, so that it does not stick to the basin. This residue is treated with 50 c.c. of absolute alcohol, and twice washed with 25 c.c. of the same. The alcoholic fluid having been decolorised with char, and evaporated to dryness, the residue left is dissolved in 30 c.c. of water, and polarised. True claret gives no rotation, or is only very slightly dextrogyrate, while wines mixed with *piquette* of fruit or glucose, are respectively strongly levo or dextrogyrate. The former rotation of fruit wine depends upon the fact that some levulose always remains unfermented, while the latter is due to the non-fermentable dextrorotary bodies always present in commercial glucose. It is to be noted that the detection of fruit wine entirely fails in the higher Rhine wines, which are all naturally prepared partially from sun dried grapes, and the process, therefore, only applies to claret in this respect.

The use of salicylic acid to preserve wine is entirely forbidden in France, being considered injurious to health. It is separated from the wine by shaking up with chloroform, and may be estimated by ferric chloride used colorimetrically (ANALYST Vol. i., p. 193), or crystallised from chloroform and weighed.

(c) *Detection of Artificial Colouring Matters.*—The first point which strikes a British reader is that in France they seem to have missed Dr. Dupre's excellent preliminary test with gelatine cubes (ANALYST Vol. i., p. 186), but they have three tests on which they greatly rely as indications of the direction of the subsequent researches if they prove unfavourable. They are as follows:—

- (1). Ordinary sticks of chalk (like those used for blackboard writing) are steeped in a 10 per cent. aqueous solution of white of egg, and after being allowed to dry spontaneously in the air, are perfectly dessicated at 100° C., and preserved. For use, one surface of a prepared stick is scraped with a knife to remove the excess of albumen, and two drops of the wine are let fall from a pipette on the porous surface thus produced. Ordinary wine gives a grey stain, which in

young and highly coloured wine may verge to blue, but there should be no trace of green, violet or rose.

- (2.) Some wine is mixed with baryta water till the colour becomes greenish, and then shaken up with acetic ether or amylic alcohol and allowed to settle, taking care that the whole is slightly alkaline. Pure wine gives no colour in the upper layer, either immediately or after acidulation with acetic acid, while coal tar derivatives of a *basic* nature give colours indicating that research should be made for such bodies as amidobenzene, fuschine, safranine, chrysoidine, chrysaniline, mauveine, methyl-violet and beibrich red.
- (3.) Ten c.c. of the wine is rendered strongly green by the addition of 2 or more c.c. of 5 per cent. potassium hydrate. To this are added the same number of c.c. of a 20 per cent. solution of mercuric acetate, and the whole, having been shaken up, is filtered. With pure wine the filtrate is colourless both in itself and after acidulating with hydrochloric acid, while that containing any coal tar derivative of an *acid* nature, is coloured red or yellow.

It would seem that the use of the old-fashioned logwood and other vegetable colours is a thing of the past, and the following two mixtures made up and sold for deceiving analysts have been specially discovered in Paris—(1) a mixture of amidobenzene, methyl-violet and the acid sulpho derivative of fuschine; (2) a compound of methylene blue, diphenylamine orange, and the acid sulpho derivative of fuschine. This latter is sold in commerce as *Bordeaux Verdissant*.

(To be continued in our next.)

#### CONCLUSION OF THE SOCIETY'S PROCEEDINGS.

#### MUSTY BREAD.

By A. PERCY SMITH, F.I.C., F.C.S., Rugby.

MUSTINESS is not perceptible in bread until the latter is at least one day old. When the bread is new, no disagreeable taste can be detected; but on the second day, the bread becomes so nauseous as to be uneatable.

The cause of this mustiness appears to have been little, if at all, investigated; the following experiments, therefore, may be not without value.

The following preparations were made:—

- (a) Musty bread, 1 day old, soaked in water, enclosed between watch-glasses.
- (b) Flour, from which the above bread was made, similarly treated.
- (c) Sweet bread, ditto.
- (d) Musty bread, 9 days old, ditto.
- (e) Minute loaf, from good flour, in which a bit of a (after 10 days) was enclosed.
- (f) Thurber's snowflake flour, treated as in b.
- (g) „ „ „ placed in a bag in a damp cellar.

(a) In six days begins to turn yellow, has a powerful disagreeable odour. On the lower glass are white sterile flocci of *Aspergillus glaucus*. The colour gradually deepens to a brilliant orange yellow, and the bread obtains a "cheesy" consistency (looks

exactly like toasted cheese). The odour is very nauseous, something between that of *Russula fetens* and *lactic acid*. Under the microscope, the "cheesy" part is destitute of mycelium, but swarms with minute animalculæ. Some of these are rod-shaped bodies from .00005 to .0002 inch in length, endowed with active motion, which consists of a short rush, then a quivering, and a rapid rotation on the minor axis. (*Vibrio rugula*?) There are also smaller, and apparently spherical bodies, about .00002 inch in diameter, which are not quite so active, but move with a to and fro motion. (*Bacteria termo*?) The magnifying power at my disposal is not high enough to enable me to describe these organisms with greater precision. The reaction is acid to litmus. In ten days, the colour of the mass loses its brilliancy, and subsides into a dull buff. The odour has disappeared. The vibriones can be no longer seen, they are dead. The bacteria are still visible as motionless dots. A portion is inserted in a small loaf *e*.

In this experiment the growth of *A. glaucus* is feeble, the flocci never get beyond the sterile stage. This is due to the bread undergoing putrefaction, subsiding into a pulpy mass which is too damp to permit the growth of the fungus.

(The brilliant colour at first suggested the presence of *icrococcus Maurantiaccus*, but I should not be able to see them with  $\frac{1}{4}$ -inch objective, even if present.)

(*b*) In four days produces "forests" of *Mucor mucedo*. No further change. The flour ultimately dries up.

(*c*.) In four days, sterile flocci of *A. spergillus glaucus* appear. In six days begins to smell after the manner of fungi. A bright red spot is seen (*Palmella prodigiosa*?) which presents nothing characteristic under the microscope and undergoes no further development. A green spot proves to be fertile flocci of *A. glaucus*. In seven days more green spots. Ultimately the bread becomes nearly covered with green *A. glaucus*. There is no *mucor*.

At no period does the bread become "cheesy," but gradually dries up. The odour is entirely different from that of decaying musty bread. The fungus flourishes luxuriantly.

(*d*.) In three days begins to have a nauseous odour, a few colonies *A. glaucus* (sterile) seen on edge where *least moist*. There is a slight tinge of yellow near one edge. Fourth day, the yellow spot has spread, deepened in colour and looks greenish. Another yellow spot showing, near which *M. mucedo* is growing luxuriantly.

Fifth day, the greenish yellow spot is merely change of *A. glaucus* from sterility to fertility. The yellow spot near the *M. mucedo* has spread and is turning "cheesy," but the colour is not so brilliant as in *a*, it will probably deepen with age. Under the microscope the vibriones and bacteria appear as before mentioned.

In this preparation the bread is three times the size of that in *a*, and instead of being laid horizontally in a watch glass, is placed vertically in a wine glass, consequently, the upper portions get comparatively dry and suitable for the growth of the fungi.

(*e*) On breaking open, a cavity, with yellow walls, presents itself where the portion of *a* was inserted. It is moistened and placed in a covered wine glass. The next day active bacteria are found in the yellow spot. No useful information is derived from this experiment.

(f) In seven days dries up completely, without having generated any fungoid growth whatever.

(g) In ten days begins to taste musty, sterile flocci seen. The next day the mustiness is perceptible to the sense of smell. On the 12th day, the flocci having become fertile, are seen to be those of *Mucor mucedo*.

These experiments might perhaps have been extended, but with questionable advantage. We see that ordinary bread turns mouldy, owing to the growth of *A. glaucus*. Musty bread, on the other hand, yields both *A. glaucus* and *M. mucedo*, and then undergoes putrefactive decomposition, becoming the home of vibriones and bacteria. These organisms, of course, can have nothing to do with the mustiness, they only flourish because there is a suitable nidus for their growth. We know how difficult it is to escape from the all pervading germ.

It is, however, curious that the musty bread should decay, and the sweet bread should not, whilst the only *apparent* difference between them is in the growth of *M. mucedo*. The suspected flour produces an abundant crop of *Mucor*, but does not decay. This is, no doubt, due to the fact that starch is not so suitable a nidus, as is dextrin, for bacteria.

Perfectly pure flour failed to decompose when kept between watch glasses, but when placed in a damp cellar readily became musty, and produced a crop of *M. mucedo*.

I think we may fairly conclude that this fungus is the cause of the mustiness, at least, in the two present cases; it by no means follows that it is the agent in *all* musty bread, since other species may possess similar properties.

The history of the musty bread, and flour, with which my baker supplied me, now becomes clear. The flour was stored in a damp place, causing fungoid growth. To arrest decomposition *the flour was then baked*. [This idea occurred to me on noticing that the flour was very dark (brown) in colour, exhibiting evidence of having been charred.] When the bread was baked, the assimilation of moisture regenerated the fungus, thus causing the bread to become musty, for which result it is not necessary for the plant to arrive at maturity; the disagreeable taste is developed as soon as flocci are visible under the microscope.

The reason why the musty bread alone putrefied does not seem clear. It cannot be accidental. Granting, for the sake of argument, that a crowd of bacterial germs was "hunting round" at the particular moment when the specimens were being prepared, it is impossible to conceive that they could have located themselves in one watch-glass to the exclusion of the rest, but a few inches away! Further, no special precautions were taken for their exclusion, and the second portion of musty bread, which was at first kept apart, underwent the same changes in decomposition as did the first portion. It would appear as if the *Mucor* has a specific chemical action upon the bread, that is not possessed by *Aspergillus glaucus*.

## MONTHLY RECORD OF RESEARCHES IN FOOD ANALYSIS.

ESTIMATION OF FREE SULPHURIC ACID IN VINEGAR.—B. Kohnstein, (*Leitmeritzer Rundschau*.) suggests the following as a convenient and practical method for quantitatively determining the amount of free sulphuric acid in vinegar:—100 c.c. of vinegar are shaken up with freshly-burnt pure magnesia in sufficient quantity to effect complete neutralization. The whole is then filtered; 25 to 30 c.c. of the clear filtrate are dried in a porcelain dish, and the residue heated to redness. The magnesium acetate is thereby converted into carbonate, while any sulphate present remains intact. A too high temperature must be avoided in the incineration, so as to prevent any decomposition of sulphate. The incinerated residue is then mixed with water containing carbonic acid, and the whole evaporated down, digested with hot water, and filtered, whereby magnesium sulphate passes into the filtrate, and the carbonate remains on the filter. The filter is washed until a drop of the filtrate on testing ceases to give a reaction for sulphuric acid. The magnesia in the filtrate is then determined as pyrophosphate, any lime present having been previously precipitated. W. H. D.

BEHAVIOUR OF SERUM ALBUMIN IN THE PRESENCE OF ACIDS AND NEUTRAL SALTS.—By E. Johannsen,\* *Zeitschr. für physiol. Chem.* 9, 310. This substance is surprisingly stable in the presence of acids. An addition of acetic acid up to one or two per cent., and of hydrochloric acid up to 0.25 per cent. had no action on it even in a month. Acid albumin was formed by the action of 0.5 per cent. of hydrochloric acid in 16 days, by one per cent. in eight days, by two per cent. in 24 hours, by three per cent. in five hours. If the temperature be raised to 40°C, the change takes place to a much greater extent. In similar experiments on solutions of serum (from white of egg) saturated with magnesium sulphate, the action of the acids took place still more slowly. The precipitate of egg albumin, obtained by adding one per cent. of hydrochloric acid, remained unaltered for at least ten days.

Eichwald was the first to point out that the property of resisting the action of acids might be utilized for precipitating undecomposed albumin by cautiously adding an acid to a solution of white of egg saturated with salt. The author bases on it the following process for obtaining pure albumin. The albumin is precipitated by adding 0.5 to 1.0 per cent of acetic acid to a solution of it saturated with magnesium sulphate; it is then pressed, redissolved, re-precipitated, and finally purified by dialysis. He found that the albumin thus obtained was less clear and soluble than that obtained by Starke's method,† *i.e.*, the introduction of sodium sulphate into a solution of egg albumin saturated with magnesium sulphate. E. E. B.

ALBUMINOUS COMPOUNDS IN MILK.—By J. Sebelien,‡ *Zeitschr. für Physiol. Chem.* 9, 445–464. Owing to the unsatisfactory methods used to separate the albuminous compounds, on the one hand a number of new bodies have been stated to exist in

\* *Zeitschr. für Anal. Chem.* 24, 3, 480.

† *Jahresber. für Thierchem.* for 1881, p. 17.

‡ *Centralblatt für Agrikultur Chemie* 14, 470.

milk, and on the other hand it has been denied that any albuminous substances beside caseine exist in milk. After a very careful investigation of the subject, the author has come to the conclusion that there are at least two albuminoids in milk besides caseine, although one of them is only present in minute quantities.

*Lactoglobulin.*—Probably identical with paraglobulin present in blood, but as only a few milligrammes were obtained from one litre of milk, only its chemical properties and the temperature at which it coagulated could be determined for comparison.

*Lactalbumin.*—This body resembled serum-albumin in every respect except, in its power of rotation, which was considerably less than that of serum albumin, which, like the other constituents of blood, does not pass into the milk unaltered. Lactalbumin cannot be regarded as a modification of caseine, as it contains a much larger percentage of sulphur.

E. F. B.

**RAPID TEST FOR OLEOMARGARINE.**—For this purpose Piallat uses a reagent prepared by putting into a glass 100 grms. of pure copper sulphate, coarsely powdered, adding 320 grms. of distilled water, and then immediately a small quantity of ammonia, stirring with a glass rod, and then adding ammonia, drop by drop, constantly stirring. There is formed a greenish-blue precipitate, whilst cupric sulphate remains in solution. At the end of the experiment, when there is no longer any precipitate formed, the ammonia should only be faintly manifest to the smell. Filter, wash the moist precipitate with distilled water until the washings run through colourless. The precipitate is then dried at a temperature not exceeding 25°, and finely powdered. For the qualitative analysis 2 grms. of butter are taken (which must be made in the laboratory to ensure that it is genuine) spread out on a glass plate, and 2 centigrms. of the reagent are incorporated with it by means of a flexible steel spatula. The mixture takes a light turquoise-blue colour. The butter is then scraped up with the spatula and deposited on another glass plate, spreading it out so as to judge its colour. This is then the standard with which the sample in question is compared. This latter is treated in the same manner, and if it contains margarine, it will take a greener and more intense colour. One-tenth of margarine is thus perceptible to the eye, and the more the butter contains the more the colour will differ from that of the standard. To render this method quantitative several standards are prepared: No. 1 pure, No. 2 mixed with  $\frac{1}{2}$  margarine, No. 3 with  $\frac{1}{3}$ , No. 4 with  $\frac{1}{4}$ , and so on. These are all treated with the reagent as above, and the sample in question being likewise so treated, its proportion of margarine may be found by comparing the colours. Such standards must be prepared fresh daily.

W. H. D.

**PETROLEUM ETHER FOR THE EXTRACTION OF WET SUBSTANCES.**—(*Chem.-Techn. Central Anz.*, No. 29.) A. Gawaloski communicates a useful expedient for causing petroleum ether solutions to pass readily through filters previously moistened with aqueous liquids.

He is often in the habit of making soap analyses, and in such cases decomposes the soap with sulphuric acid, transfers the separated fatty acids upon a filter, and washes

with water. The filtrate and washings are freed from any retained fatty acids by shaking with petroleum ether, and this ether is then used to dissolve the fatty acids upon the filter (the ethereal solution being finally evaporated and dried). In order to be able to pass the petroleum ether solution through the filter, he has hitherto been compelled to dry the filter for many hours before it would permit the ether to run through.

He has now found that by pouring a little absolute alcohol upon the edges of the filter, the latter is so thoroughly freed from the absorbed water, that it will immediately permit the petroleum ether solution to run through, almost in a continuous stream, thereby shortening the time most considerably.

W. H. D.

DETECTION OF COLOURING MATTERS IN WINE.—By Dr. Pedro N. Arata. (*Anales de la Sociedad Científica Argentina XIX.*, 140.) Place from 50 to 100 c.c. of the wine in a porcelain basin, adding from 5 to 10 c.c. of a 10 per cent. solution of potassium bisulphate. Put in 3 or 4 threads of white wool (previously treated with an alkali and well washed). After boiling for 10 minutes, the threads are taken out and placed under a current of water to remove the excess of wine, and if the article be pure, the threads will have a pink colour. This colour changes to green when treated with ammonia, and does not recover its original colour after washing, only a dirty white being left. With wines artificially coloured the colour of the threads after washing is deeper, becoming paler when treated with ammonia, or else changed into yellow. The threads are now boiled with dilute tartaric acid, washed and dried, placed in a test tube, and strong sulphuric acid added drop by drop; this gives characteristic reactions. In many cases the colour is a dirty green, and nothing definite can be observed. In this case more sulphuric acid is added, and the threads pressed with a glass rod for 10 minutes, and made up with distilled water to about 10 c.c. The wool is taken out, and excess of ammonia added. After cooling, from 5 to 10 c.c. of amylic alcohol are added, the whole is shaken, and allowed to stand; the alcoholic layer is blown into a watch glass, evaporated to dryness, and the residue tested with a drop of strong sulphuric acid. The following are some of the colours produced:—

Ponceau R—2R—3R—S and 2S..	..	..	yellowish red with a trace of violet.
Ponceau G and Tropeoline O	..	..	yellow and orange.
Biebrich red	..	..	green.
Bordeaux verdissant and croceine	..	..	blue.
Tropeoline 30 and solid red	..	..	violet.

The action of acids, alkalies, and other reagents, can be tried on the coloured threads or residue.

J. M. V.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

ALOIN.—Dr. Justus Dietrich (*Thesis Dorpat*, 1885) has studied the detection of aloin in animal secretions and excretions, and found the following reactions serviceable for this purpose:—The residue left on evaporating the alcoholic solution was dissolved

in a few drops of nitric acid, the solution evaporated by means of a steam bath, dissolved in alcohol, and the deep red solution treated with a drop of alcoholic solution of potassium cyanide, which produced a rose-colour with five different aloins.

In the following tests the aloin residues were dissolved in a little water:—Chloride of gold produced with barbaloin a raspberry red colour (still recognizable with 0.00006 aloin) after some time changing to violet. Socaloin and cape-aloin gave a rather faint colour, rapidly changing to violet; nataloin red-violet, rapidly turning to violet; Curaçao-aloin bright red. For the detection of aloin the fæces were digested with water acidulated with sulphuric acid, then macerated for 12 hours with three volumes of strong alcohol, the filtrate concentrated, and the residue successively agitated with petroleum benzin and amylic alcohol; on evaporating the latter aloin was left. The treatment of blood and urine was similar. From his results the author concludes that on taking aloes or aloin, the greater portion is excreted with the fæces: a small portion only is absorbed and passes mostly through the kidneys, while the remainder enters the liver, and with the bile is conveyed back into the intestines. W. H. D.

---

DETECTION OF OIL OF TURPENTINE IN OIL OF LEMON.—A test has lately been proposed by G. Heppe (*Chem. Tech. Central Anz.*, III., 371), the reagent being cupric butyrate.

A portion of the oil is gradually heated on a sand-bath in a perfectly dry test-tube, with a small quantity (about as large as the head of a pin) of cupric butyrate, to a temperature of about 172°C. If the oil of lemons be pure, the copper salt dissolves and colours the oil green. If oil of turpentine, however, be present, the oil becomes turbid at the above temperature, turns yellow, and deposits reddish-yellow cuprous oxide. The difference between the pure and the impure oil is so decided, not only after heating, but also after again cooling, that even a person unfamiliar with chemical manipulations can easily recognise it. The adulterated or impure oil is yellow, the pure oil clear and green. If, however, too much of the copper salt had been taken, the oil will be green after cooling, even if oil of turpentine be present. Nevertheless, the yellow precipitate will distinguish it from the pure oil.

The cupric butyrate must be dry and in powder. To raise the temperature over 180°C is not advisable, since the pure oil itself would suffer change in this case. And, although pure oil may even then be still recognized by the test, yet the latter is much less distinctive. W. H. D.

---

CUPREOL is the name given by Dr. O. Hesse to a waxy substance he has obtained from the Cuprea barks. It appears to differ in several respects from cinchol, the corresponding fatty principle of Cinchona bark. Cupreol fuses at 140°C. dissolves easily in ether, chloroform and hot alcohol, crystallizing from the latter on cooling, in colourless scales. It is not very soluble in cold spirit or petroleum ether. The chloroform solution gives a deep red colour with sulphuric acid of Sp. Gr. 1.76. W. H. D.



DETECTION AND PROPERTIES OF ANTIPYRIN.—By Otto Schweissinger. *Archiv. der Pharm.*, 222, 686. This substance (probably dimethyloxychinizine), which has been largely used of late as an antipyretic, is a voluminous crystalline powder, of a reddish-grey to white colour. It dissolves in 50 parts of ether, readily in alcohol and in chloroform, in 0.6 parts of water at 15°C., the solubility increasing at higher temperatures. Antipyrin melts at 113°C., and, on further heating, turns first red and then brown, forming an inflammable residue, smelling like oil of amber: it is soluble in chloroform, alcohol, slightly in ether, insoluble in benzene, oil of turpentine, and potash. Hydrochloric acid has no action on this substance, nor nitric acid (spec. grav. 1.185) in the cold, but on warming, it suddenly becomes red, and on further heating, a reddish-purple oil and a brown resinous substance separate out.

Most of the common reagents for alkaloids give precipitates with antipyrin. Concentrated sulphuric acid is only turned a faint yellow by it. The chief re-agents for detecting antipyrin are ferric chloride, nitrous acid, and fuming nitric acid.

Ferric chloride produces, with dilute solutions of antipyrin (1 in 1,000), an intense reddish-brown colouration, which disappears on adding a drop of concentrated sulphuric acid. The behaviour of antipyrin compared with that of bodies which act in a similar way is given in the following table:—

	On addition of a drop of ferric chloride.	On addition of a drop of concentrated sulphuric acid.
Carbolic acid ..	Blue .. .. .	Dark yellow.
Salicylic acid ..	Violet-blue .. ..	Colourless.
Resorcine .. ..	Blue .. .. .	Yellowish-brown.
Cairine .. .. .	{ First light brown, then dirty dark br'wn	Reddish purple.
Antipyrin .. ..	Reddish-brown .. ..	Colourless.
Quinine .. .. .	Colourless .. .. .	Colourless.

The author gives other tests for distinguishing antipyrin from cairine.

The most characteristic test for antipyrin, which, in fact, distinguishes it from all other substances in commerce, is the green colour produced by the action of fuming nitric acid on it. If one drop of the latter be added to one c.c. of the dilute solution of antipyrin (1 in 1,000), this green colour is formed, and remains (in the cold) for several days. If this liquid be warmed, and a second drop of fuming nitric acid be then added, the solution becomes first bright red, then blood-red, and on heating still more, a purple oil separates, which is soluble in chloroform, but insoluble in carbon bisulphide and benzene.

Antipyrin can be detected in urine direct by ferric chloride; but if it be dark, the urine must be concentrated and decolourised with animal charcoal before being tested.

To distinguish urine containing ethyldiacetic acid from urine containing antipyrin, the samples must be distilled; the distillate from the former is coloured by ferric chloride, owing to the volatility of the ethyldiacetic acid; the distillate from the latter is unaffected by the ferric chloride.

E. E. B.

**ADULTERATION OF ARGOL.**—By Zierek. Rep. der anal., Chem., vol. 5, p. 245. The author comments on a recent statement that argol is sometimes adulterated with an acid sulphate, and thinks that alum was mistaken for it. To detect alum, it must be remembered that alumina is not precipitated by ammonia in the presence of tartaric acid, and that this must consequently be first expelled by ignition. Argol adulterated with alum, is usually sold as a powder, which is always lighter in colour than the unadulterated substance. If as much as 20 per cent. of alum be present, it can be detected by its taste. Before determining the amount of acid tartrate of potash in a sample of argol, it should always be tested for sulphates; with a fairly concentrated solution of unadulterated argol, barium chloride should only produce a slight turbidity. The loss of weight of the sample, dried at 100°, should also be ascertained; unadulterated argol only loses about 2 per cent., but if it contains alum, it loses much more, usually 8 to 10 per cent.

E. E. B.

---

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

**ADULTERANT OF CARBON BISULPHIDE.**—M. Van Engelen, (*Journal de Pharmacologie de Bruxelles*,) reports a new adulterant of carbon bisulphide. In testing a commercial sample, he evaporated a portion on a watch-glass at a low temperature, when a whitish oily body was left, having the characteristic odour of turpentine. In order to estimate the quantity of the adulterant present, the liquid was submitted to fractional distillation. Notwithstanding the difference in the boiling points of the two liquids, this method of procedure failed to separate them. When an equal volume of water was added, however, the fractional distillation was easily conducted, and 10 per cent. of essential oil separated.

W. H. D.

---

**SEPARATION OF NICKEL AND COBALT.**—Ilinski and Knorre.—If a solution containing nickel and cobalt and free hydrochloric acid be treated with nitroso- $\beta$ -naphthol, all the cobalt will be precipitated as cobalt nitroso- $\beta$ -naphthol, while the nickel will remain in solution.

To the solution containing a mixture of nickel and cobalt sulphates or chlorides add a few cubic centimetres of hydrochloric acid. To the warm solution add a hot solution of nitroso- $\beta$ -naphthol in 50 per cent. acetic acid and allow the precipitate to settle. When a further addition of nitrosonephthol solution no longer produces a precipitate, the solution is filtered and the precipitate washed with a 12 per cent solution of hydrochloric acid. To the dry precipitate a little pure crystallised oxalic acid is added. It is then ignited in a Rosco's crucible in a current of hydrogen. The cobalt is weighed in the metallic state. In the filtrate the nickel is precipitated with potassium hydroxide, after the acetic acid has been removed by boiling, and finally weighed as metallic nickel.

W. H. D.

ON THE DETECTION OF NITROGEN IN ORGANIC COMPOUNDS. C. Graebe.—The author has found that the Lassaigne method for detecting nitrogen in organic compounds is not universally applicable. The method consists in heating the compound with metallic potassium, and then treating a solution of the cyanide thus formed with a mixture of ferrous and ferric compounds. The formation of Prussian blue indicates the presence of nitrogen in the compound tested. Graebe has found that the diazo-compounds in general do not give this reaction, and he thinks it probable that the nitrogen escapes as a gas before any cyanide is formed.

W. H. D.

TESTS FOR BILE IN URINE. Carl Deubner (*Thesis, Dorpat* 1884,) has obtained the best results with the methods proposed by Hilger and by Rosenbach. According to Hilger ("Archiv d. Phar.," vol. 206, p. 385) the urine is moderately heated and rendered alkaline by barium hydrate; a small portion of the washed precipitate treated carefully with a few drops of concentrated nitric acid gives the well-known colour reactions, green, violet, blue.

Rosenbach ("Chem. Centralblatt," 1876, p. 150) recommends filtering the urine through white filter paper; this acquires a yellow or brown colour, which with a drop of nitric acid changes to yellowish-red, the margin of the spot becoming violet and deep blue, while towards the centre the colour gradually changes to emerald-green. The modification of this test recommended by Deubner consists in placing a few drops of the urine upon a porous plate of white clay, when the spot remaining will show the reaction plainly and for some time. The advantage of this modification is that very little of the material is sufficient for applying the test, and that errors arising from the decomposition of the paper by the acid are excluded.

W. H. D.

A SIMPLE METHOD FOR THE QUANTITATIVE DETERMINATION OF NITRIC ACID.—Wildt and Scheibe.—The Tiemann-Schulze method for determining nitric acid has several sources of error, which may produce unreliable results. The nitric oxide, which is measured over water, is not entirely insoluble; and, unless the water has been previously boiled, it contains oxygen, which will convert a portion of the nitric oxide into nitric and nitrous acids. The consequence of these facts is that the method tends to give too low results. The authors have modified the process by passing the nitric oxide through a solution of sodium hydroxide, to remove all traces of hydrochloric acid from the gas, and then into a flask containing air and water. The nitric oxide is thus oxidised and dissolves in the water present. At the end of the operation the acid liquid is titrated with a standard alkali. The quantity of nitric acid is found at once, thereby avoiding the readings of temperature and barometric pressure as well as the calculations which are necessary when the nitric oxide is measured.

W. H. D.

ESTIMATION OF CYANOGEN IN A MIXTURE OF GASES.—By G. Jaquemin.\* *Reper-toire de Physique*, 1885, 256. Gmelin proposed in 1818 to obtain cyanogen by the action of concentrated solutions of copper sulphate and potassium cyanide on one

\*Rep. der anal. Chem., 5, 247

another, and the author has found that if double the quantity of cyanide proposed by Gmelin be used, that the whole of the cyanogen in the potassium cyanide is liberated.

The cyanogen thus obtained contains, if the potassium cyanide was not pure, carbonic acid as well as some oxygen and nitrogen (from the air). The best absorbent for cyanogen is aniline, which is even better than glacial acetic acid (proposed by Beketeff), which absorbs 80 times its volume of cyanogen. Aniline when shaken up with cyanogen, at once forms cyaniline, whereas it does not appreciably affect carbonic acid, or air, even if left in contact with them for 24 hours.

If it is required to determine the amount of each of these four gases when mixed together, the cyanogen is first removed by aniline, then the carbonic acid by concentrated potash, then the oxygen by pyrogallic acid, the nitrogen being as usual, determined by difference. The analysis can be easily made with Orsat's apparatus.

E. E. B.

---

SEPARATION OF ALKALOIDS FROM HUMAN REMAINS.—By H. Beckurts. Chem. Centralbl. [3], 15, 905. The remains are digested with a solution of oxalic acid in alcohol, and the mixed extracts treated as usual. After shaking up the acid aqueous solution with ether it is just neutralised with lime or baryta water, and again shaken up with ether; this dissolves all the alkaloids except morphine, narceine, and curarine. The aqueous liquid, containing the precipitate of oxalate of lime or barium, and also the morphine and narceine suspended in it, is filtered and the morphine and narceine extracted from the dry precipitate by boiling alcohol, which is said to yield, on evaporation, the alkaloids in a much purer condition than the best amyl alcohol.

E. E. B.

---

VOLUMETRIC ANALYSIS OF MANGANESE IN ALLOYS, MINERALS, ETC.—By Prof. W. Hampe, Chem. Zeit. 9, 1083 to 1088. In this very interesting article, the author gives a very full account of improvements made in this process for estimating manganese by potassium chlorate, which he first published in 1882. The process consists in precipitating the manganese from a strong nitric acid solution by potassium chlorate, and subsequently titrating the hydrated oxide of manganese thus obtained with ammonio-ferrous sulphate and potassium permanganate. The chief precautions to be taken are to use the nitric acid as strong as possible, and to add the chlorate of potash gradually.

Chlorides, if present, must be expelled; copper and nickel do not affect the accuracy of the process; cobalt, lead, and bismuth are slightly injurious if present in much larger quantities than the manganese itself, otherwise they are quite harmless.

E. E. B.

---

MOLYBDIC ACID RESIDUES.—By W. Venator. Chem. Zeit., 9, 1068. The molybdic acid in the residues from the analyses of phosphates, etc., may be recovered in the following way. The liquid is tested for iron, and if none be found, ferric chloride is added until the liquid is dark yellow; ammonia is then added in excess, when all the

phosphoric acid is thrown down in combination with the iron, the excess of iron and some magnesia being also precipitated. The liquid is filtered and barium chloride added, when barium molybdate and sulphate (if present) are thrown down. The precipitate is well washed, dried, and then boiled for a long time with an equivalent weight of ammonium sulphate, when the barium molybdate is completely decomposed; the insoluble barium sulphate is filtered off and the ammonium molybdate obtained by crystallisation, the crystals being found to be very pure.

E. E. B.

QUANTITATIVE ANALYSIS BY ELECTROLYSIS—(*continued*). By A. Classen and R. Ludwig, Ber. der deutsch. Chem. Ges. 18, 1787 (see ANALYST 10, 154).

*Separation of Iron from Manganese.*—If a large excess of ammonium oxalate be mixed with the solution of iron and manganese (which may also contain nickel, cobalt or zinc), these two metals can be most accurately separated by electrolysis. If the liquid be kept at 70° to 80°C., the separation takes place twice as quickly as in the cold. In mixtures of iron and manganese containing 28·81 % , 28·67 % , 24·17 % , and 17·13 % of Fe<sub>2</sub>O<sub>3</sub>, the authors found 28·72 % , 28·60 % , 24·10 % , and 17·01 % of Fe<sub>2</sub>O<sub>3</sub>.

*Separation of Iron from Alumina.*—On electrolysing a salt of aluminium, in the presence of a large excess of ammonium oxalate, aluminium hydroxide is deposited, if the amount of ammonium carbonate, formed by the current, exceeds the amount of ammonium oxalate; hence, in making the separation, care must be taken to decompose the ammonium oxalate as little as possible; this is effected by keeping the liquid cold, and by using a weak current, when accurate results are obtained. In mixtures of iron and alumina, containing 49·006 % , 48·513 % , and 26·240 % of Fe<sub>2</sub>O<sub>3</sub>, 48·85 % , 48·489 % , and 26·197 % of Fe<sub>2</sub>O<sub>3</sub> were found.

*Determination of Copper.*—The time required for their previous process may be shortened to 4–5 hours by keeping the liquid at 70° to 80°C. Only one Bunsen cell is required.

E. E. B.

ON THE DETERMINATION OF ALUMINA AND IRON IN THE PRESENCE OF PHOSPHORIC ACID. By R. Lucion. Chem. Zeit. 9, 1050.

The author considers his process, somewhat similar to that of Kretzschmar (see ANALYST for August), both quick and accurate. After separating the silica, he makes his acid solution of the phosphates up to a definite volume, and divides it into three equal parts. In (1) he determined the total amount of phosphoric acid by ammonium molybdate. To (2) he adds an excess of ammonia, washes well, and weighs the precipitate, which consists of the phosphates of iron, alumina and lime. He also determines, with an oxalate, the amount of lime in the liquid, as the phosphates usually contain carbonate of lime. (3.) The third portion of the phosphate solution is made alkaline with ammonia, the precipitate washed, and then treated with acetic acid, when the phosphates of iron and alumina remain undissolved; the lime is afterwards precipitated as oxalate, and weighed. Then 2 — (1 + 3) = iron + ammonia.

To save time the author prefers in (3) to add the acetic acid to the ammoniacal liquid *before* filtering; as the acid liquid then contains *all* the lime, he has to deduct from the amount of it found the amount of lime determined in (2). E. E. B.

DETERMINATION OF TIN IN TIN SCORIE.—By R. Fresenius and E. Hinz.—*Zeitschr. für anal. Chem.* 24, 3, 412.

The authors give the following process for analysing tin scorie, which in addition to tin, contain antimony, arsenic, molybdenum, tungsten, lead, iron, aluminium, &c.

About three grammes of the sample, very finely powdered, is treated with warm aqua regia; the residue is then filtered off, after diluting the solution, and well washed with water containing ammonium nitrate. In case this residue still contains tin it must be dried, removed from the filter, and fused with liver of sulphur; the mass is then treated with water, and, by filtration, a solution (A) of sulphur compounds is obtained, which will be referred to directly.

The aqua regia solution is then made alkaline with soda, and digested for a long time with warm sodium sulphide. After filtering off the black precipitate which forms, and washing well with water containing sodium sulphide, the filtrate and washings are mixed with the above solution (A), and the whole acidulated with hydrochloric acid. The precipitate thus formed contains, beside tin and much free sulphur, arsenic, antimony, tungsten, and molybdenum, and is then treated with bromic acid. Most of the tungstic acid remains undissolved, and, after removing this by filtration, the liquid is concentrated to a small bulk, but to prevent any loss of tin by volatilization as tin chloride, a few grammes of potassium chloride are first added. As more tungstic acid is deposited during evaporation, the liquid must be again filtered before precipitating the tin in it with ammonium nitrate in the usual way.

To extract the small quantity of tin from the above tungstic acid it is fused with five times its weight of potassium cyanide, when, on treating with water, a little metallic tin (B) is obtained, the tungstic acid being dissolved.

The precipitate obtained with the ammonium nitrate is further purified from tungstic and molybdic acids by fusion with potassium cyanide. On treating with water the mass yields more metallic tin (C) and a grey powder (D).

(B) and (D) are again fused with liver of sulphur, treated with water, filtered, and the solution acidified with sulphuric acid; the sulphides precipitated are dried and heated in a current of hydrogen, when sulphur and some of the sulphide of arsenic volatilize. The residue is heated in air, fused with potassium cyanide, and, on treatment with water a small quantity of metallic tin is obtained, which is added to (C) and the whole dried at 100°C., and weighed.

This weighed metallic tin still contains traces of silica, antimony, and arsenic, and to remove them the metal must be dissolved in hydrochloric acid, when the impurities either remain undissolved or volatilize as hydrogen compounds, which are caught in a solution of silver nitrate, and determined in the ordinary way. By deducting these impurities from the weight of (B) and (C) together, the amount of pure tin in the sample is found. E. E. B.

## FOOD NOTICES.

At the request of our publishers we have purchased and analysed several samples of Mellin's food for infants and invalids, with the view of ascertaining whether the article, as at present manufactured, equals in quality the sample of which the analysis, by Dr. Fresenius is exhibited monthly on our title page. We have pleasure in stating that our results approximated sufficiently closely to those of Dr. Fresenius to enable us to assert that this article keeps up all its well-known reputation of being one of the best things of the kind in the market, and is in every respect a wholesome and reliable food for young and delicate persons.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

*To the Editor of THE ANALYST.*

SIR,—Allow me to call the attention of analysts to a convenient arrangement to do away with the use of a pinchcock for regulating the flow of liquids from a burette.

The use of a metallic pinchcock has several drawbacks, as is well known; not the greatest of which is the tendency to cut through the rubber tubing; any corrosion of the metal roughening the surfaces of the working parts makes the delivery of a single drop extremely difficult. My plan is as follows:—

A B and C are respectively the ordinary burette rubber tube and jet tube. In place of a pinchcock I insert a piece of glass rod D about half-an-inch in length, and just fitting the rubber tube.

It will be found that the burette is perfectly sealed, and by pinching the rubber tube upon the glass rod between the thumb and forefinger a very small channel is formed between the rod and tube allowing the passage of one drop or a continuous stream of the liquid at will.

A few moments practice may be necessary at first, but the convenience and comfort of the arrangement will, I am sure be readily appreciated.

Yours truly,

A. LEBRASSEUR.

59, Warwick Gardens, Kensington, W.

## BOOKS, &amp;c., RECEIVED.

ADULTERATION OF BEER, by Professor H. B. Cornwall; The Biological Examination of Water, by C. J. H. Warden; Chemical Equilibrium, the result of the Dissipation of Energy, by G. D. Liveing, M.A. F.C.S.; Contributions from the Department of Pharmacy of the University of Wisconsin; Contributions to our Knowledge of Sewage, by W. R. Nichols and A. R. Allen; Metodo para la investigacion de Algunos, Derivados del Alquitrán en los Vinos, &c.; Proceedings of the California Pharmaceutical Society for the years 1883 and 1884; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; British and Colonial Druggist; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Illustrated Science Monthly; Independent Journal; Invention; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; New York Analyst; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Reportorium der Analytischen Chemie; San Francisco News Letter; Science; Scientific American; Society of Arts Journal; Hospital Gazette.

## NOTICES TO CORRESPONDENTS.

All communications as to literary matters to be addressed to the Editorial Department, 325, Kensington Road, London, S.E.

Dr. B. F. Davenport, Boston.—The report referred to in your communication, dated September 1st, 1885, has evidently miscarried. If there be anything fresh in it as to the methods of milk analysis we will be pleased to have a copy and will abstract it. If it be a simple statistical analyst's report, you will have noticed that we do not now summarise such documents in our columns.

# THE ANALYST.

NOVEMBER, 1885.

## CONTENTS.

	PAGE		PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—		MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—	
ON THE PROCESSES AND STANDARDS IN USE AT THE MUNICIPAL LABORATORY OF THE CITY OF PARIS—DR. MUTER ( <i>concluded</i> )	195	(a) THE ACTIVE PRINCIPLE OF GOA POWDER—PETIT .. .. .	207
ORIGINAL ARTICLES:—		(b) QUANTITATIVE DETERMINATION OF CINCHONA ALKALOIDS—SHIMOYAMA .. .	207
(a) NEW TEST FOR NITRATES IN WATER—A. PERCY SMITH, F.I.C. .. .	199	(c) A NEW COLOUR TEST FOR CODEINE AND ESCULINE—RABY .. . . .	208
(b) ON THE ADULTERATIONS OF SAFFRON—PROFESSOR J. M. MAISCH .. .	200	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY:—	
MONTHLY RECORD OF RESEARCHES IN FOOD ANALYSIS:—		(a) QUANTITATIVE VALUATION OF RESIN IN SOAP—HEINER .. . . .	209
(a) COLOURING MATTER IN GRANULATED SUGAR—HEWITT .. . . .	203	(b) DETERMINATION OF SMALL QUANTITIES OF SODIUM CHLORIDE IN POTASSIUM CHLORIDE—ROTTGER AND PRECHT .. .	209
(b) THE MELTING POINTS OF FATS—BEUSERMANN .. . . .	204	(c) VOLUMETRIC DETERMINATION OF POTASSIUM—DUBENARD .. . . .	210
(c) ESTIMATION OF ACIDITY IN SOUR MILK OR KOUMISS—H. W. WILEY .. . . .	204	(d) A NEW METHOD OF SEPARATING GLOBULINE FROM ALBUMEN—WICHAILOW .. .	211
(d) ESTIMATION OF WATER IN HONEY—WILEY AND BROADBENT .. . . .	204	(e) VOLUMETRIC DETERMINATION OF UREA—PFLEFFER .. . . .	211
(e) APPARATUS FOR FILTERING SMALL QUANTITIES OF LIQUID—WILEY .. . . .	204	(f) DETERMINATION OF CARBON IN IRON—M. CLERE .. . . .	212
(f) ACTION OF TANNIN ON FEHLING'S SOLUTION—SONNENSCHN .. . . .	205	(g) DETERMINATION OF NITROGEN IN MANURES CONTAINING NITRATES—O. REITMAR .. .	212
(g) ADULTERATION OF OLIVE OIL—HIEPE .. . . .	205	(h) PYROGALLIC ACID AS A REAGENT FOR DETECTING MINUTE QUANTITIES OF NITRIC ACID—CURTMAN .. . . .	212
(h) DETERMINATION OF GLYCERINE IN DILUTE AQUEOUS SOLUTIONS AND IN FATS—BENEDIKT AND ZSIGMONDY .. . . .	205	(i) DETERMINATION OF PHOSPHORIC ACID IN PHOSPHATIC SLAGS—C. BRUNNEMANN .. .	212
		(j) DETERMINATION OF SILICON IN IRON—L. BLUM .. . . .	213
		A CHEMICAL CANDIDATE FOR PARLIAMENT .. .	213
		CORRESPONDENCE .. . . .	214
		BOOKS RECEIVED, &c. .. . . .	214

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

### ON THE PROCESSES AND STANDARDS IN USE AT THE MUNICIPAL LABORATORY OF THE CITY OF PARIS.

By DR. MUTER.

(Continued from our last; commenced in the August issue.)

WHEN the basic nature of the coal tar colouring matter has been made evident by the preliminary test No. 2 above referred to, its exact nature is rendered certain by separating the acetic acid or amylic alcoholic layer, filtering it and dividing into two parts. The one portion is then rapidly evaporated with a thread of pure wool, and the other with a few filaments of natural silk. When the threads have taken the dye, they are rinsed in distilled water and examined on the following principles:—

*Rosaniline* or *safranine* give red threads, but on treating with concentrated hydrochloric acid, the former is discolored to a tint resembling that of a dead leaf, and the subsequent addition of water restores the primitive colour. As a rule, *safranine* will only fix on the silk, and not on the wool.



*Soluble aniline violets* give dyed threads which, when treated with hydrochloric acid, become blueish-green, changing to yellow, and water in excess restores the violet.

*Mauvaniline* gives threads which first turn indigo-blue with the acid, then behave like rosaniline, but on afterwards adding the water a reddish-violet is produced.

*Chrysotoluidine* gives threads scarcely affected by hydrochloric acid, but decolorised by boiling with powdered zinc, the colour returning on exposure to the air.

*Amidonitrobenzol* and *Chrysoidine* both give yellow threads, but the former is straw-yellow, and becomes poppy red with strong sulphuric acid, while the latter is orange-yellow, and becomes scarlet with the acid.

Finally, all tar dyes analagous to rosaniline are instantly decolorised by a few drops of sodium bisulphite, while ordinary colours such as cochennille, &c., are not affected, or only very slowly.

When the preliminary test No. 3 gives indication of a tar dye of an acid nature, the following course is to be followed:—

- (1) The wine is strongly acidulated with hydrochloric acid, and shaken up with acetic ether or amylic alcohol.
- (2) The wine is saturated by a slight excess of ammonia, and shaken up with acetic ether or amylic alcohol.

The ethereal fluids are mixed, evaporated to dryness, and the residue tested with a drop of strong sulphuric acid, when the following effects will be observed:—

*Roccelline* gives a *Parma violet* coloration.

*Foundation Red* gives a *maroon* colour.

*Bordeaux R.* and *Bordeaux B.* give a *blue*.

*Ponceau R.*—*RR* and *RRR* give a *scarlet*.

*Ponceau B.* gives a *red*.

*Biebrich Reds.*—These give, according to their nature, colours varying from green to violet. If the main nucleus of the compound be the benzol group they give *deep green*. If both the nuclei of the benzol and naphthol groups be present, the colour is *blue*. If the main nucleus be the naphthol group the colour is *violet*.

*Tropeoline OOO*, 1 and 2, give a *fuschine red*.

*Tropeoline O* and *Chrysoine* give *orange-yellow*, and on slightly diluting with water a poppy red shade comes temporarily.

*Tropeoline Y* gives an *orange-yellow* changed darker by water.

*Tropeoline OO* gives a *violet-red*.

*Helianthine* gives a *brownish-yellow*.

*Eosine B*, *Eosine JJ*, *Safrosine*, and *Ethyleosine* all give a yellow colour. The latter is precipitated by a soluble mercuric salt and exhibits (together with all of its derivatives), a remarkable dichroism.

*Red Coraline* does not come out in presence of ammonia to amylic alcohol, but on adding the ammonia to the original wine it becomes *violet*.

In concluding the reading of this section of the report, one cannot help being struck with the singular ability displayed by M. Gerard, and we must the more con-

gratulate ourselves upon his work, seeing that he is a member of the Society of Public Analysts.

## (3.) BEER.

The methods of analysis followed for this article are in no way different from those in use by us, and there is nothing in this section worth special notice.

## (4.) SPIRITS.

The amount of matter under this head is small, and there do not seem to be any legal limits for the alcoholic strength such as we have in this country.

## (5.) MILK.

The main difference between the method followed in the Laboratory and that used by us is that the judgment is not formed upon "fat" and "non-fatty solids" only, but upon a sort of rough full analysis. The total solids are taken in one portion by evaporating 10 c.c. of the milk in an air bath at 95° C to constant weight. Twenty c.c. of the sample are then mixed with 80 c.c. of water, coagulated by dropping in acetic acid, and after standing for some time, the whole is filtered through a tared filter, and the coagulum is washed. The filter with the coagulum is rough dried and extracted in a "Soxlet" with ether. The fat thus got out is weighed, and the remaining casein is dried and weighed, and then ignited and the ash also weighed. The filtrate from the curd is boiled to coagulate albumen, filtered, and the milk-sugar estimated in the filtrate by Fehling, or by the polariscope.

The standards fixed are as follows:—

## (a) Medium milk for calculating adulteration.

Fat - - - -	4.00
Milk-Sugar - - - -	5.00
Casein and albumen - - - -	3.40
Ash - - - -	.60
Total solids - - - -	13.00

(Corresponding to 9 per cent non-fatty solids.)

## (b) Minimum limit for condemnation:—

Fat - - - -	2.70
Milk-Sugar - - - -	4.50
Casein, albumen and ash - - - -	4.30
Total solids - - - -	11.50

(Corresponding to 2.7 per cent. of fat and 8.8 per cent. non-fatty solids.)

It is, nevertheless, declared that this minimum limit cannot be considered as an absolute one for fixing the point from which the fraud always commences. It is not to be held that because a milk contains 11.5 per cent. of total solids, or 2.7 per cent of fat, or 4.5 per cent of milk-sugar, it is, therefore, exempt from a charge of fraud. The conclusion of the official analyst charged with the analysis of milk is to be a result of the comparative consideration of all points of his analysis. Thus, for example, he may consider as fraudulent, not only every sample the sum of the total solids of which does not reach 11.5 per cent. but also any sample which, although containing more than 11.5 per cent. of total solids, yet shows less than either 2.7 per cent. of fat or 4.5 per cent. of milk-sugar. It is also ordered that, wherever it is possible, the analysis of any milk found

bad by these standards is to be controlled, and the adulteration calculated upon an analysis of the milk of the cows supposed to have yielded it, such milk being compulsorily drawn from them in presence of the expert-inspector.

In large cities like London such a procedure would, as a rule, be impossible, but the general system above noted bears out a suggestion made by myself, now over ten years ago, both in the matter of milk and butter, viz., that while we may have a limit for condemnation based upon the poorest possible natural product, yet given a fall below this limit, then the standard for calculating the added water or fat should be that of a fair average article.

#### (6.) BUTTER.

The processes employed are Hehner's and Koettstorfer's, and the preliminary test is either the latter or the solidifying point of the fatty acids, which is not to be over 38°C. No mention is made of the specific gravity process, and the authorities of the Laboratory appear to have missed this very simple preliminary test, which they would do well to adopt. The standard is 87.5 per cent. of insoluble fatty acids both for condemnation and calculation. The standard for the condemnation in a preliminary Koettstorfer test is 221 milligrammes of KHO for one gramme of butter fat.

Artificial colouring matters are detected by shaking up some of the butter with very dilute spirit, evaporating the liquid, and treating the residue with sulphuric acid for annato (*blue*), with ammonia for turmeric (*brown*), and with subacetate of lead (after re-resolution in dilute spirit) for saffron (*orange precipitate*). On this point it is worthy of notice that if the very dilute spirit be warmed to the melting point of the butter, the extraction is more complete than in the cold.

#### (7.) OILS.

The tests employed for olive oil are the usual ones, viz., saponification and taking the solidifying point of the fatty acids, heating power with sulphuric acid, Pinchon's areometer, and Cailletet's colour test, with acid mercuric nitrate. The two adulterants met with are cotton and ground nut oils, and the following are the standards given:—

	Olive.	Cotton.	Ground nut.
Heating power with sulphuric acid (1.834) ..	55.5°C ..	69.5°C ..	62.0°C
Solidifying point of fatty acids .. ..	22°C ..	35°C ..	31°C
Pinchon's Areometer {	Thermometer degree ..	18 to 22 ..	18.7 .. 18.9
	Scale degree ..	„ ..	15.8 .. 10.5
Cailletet's colour test .. .. ..	green ..	dirty yellow ..	dirty yellow.

#### (8.) BREAD.

Here we also notice that there is no mention of the well-known Logwood test, but instead of it we find one involving burning to ash, extracting with dilute nitric acid, precipitating with ammonia, washing the precipitate, and treating it on platinum foil before the blowpipe with chloride of cobalt. The process given for the estimation of alum is the old potash one, now long since condemned in this country, and in the matter of bread it may be said generally that we are considerably in advance of French methods.

#### (9.) COFFEE.

Once more we are struck in reading this section with the small use they make of that useful article a specific gravity bottle. There is no more certain estimation within

reasonable limits than that of chicory in coffee by the specific gravity of the 10 per cent. decoction; but our Parisian friends quite ignore it, and depend on taking the chlorine in the ash. The standard is .04 per cent. for coffee and .2 per cent. for chicory, and if the chlorine exceeds .04 and the microscope reveals no chicory, then the coffee is to be considered as damaged by sea water. I think, on reflection, our Parisian friends will see the advantage of following British methods in the matter of coffee.

(10.) SWEETS.

The question of the colouring matter of sweets is certainly on a better footing in France than with us. The tests for coal tar colours having been carefully worked out in connection with wines, the Government lay down a list of articles which are forbidden, and also one of such colouring matters as are allowed. Thus it is not a question as with us whether the amount of colour found is dangerous to health, but simply what is the colouring matter and is it in the admitted list or not. It is to be hoped that in any amended Act we may be provided with a similar schedule. I may mention that all colours containing copper, lead, barium, arsenic or mercury, together with all chromates, fuchsin and its derivatives, eosine, naphthol yellow, victoria yellow, tropeolines, xylidine reds, and practically all coal tar derivatives are forbidden.

(11.) SUNDRIES.

Besides the work actually relating to food, the Laboratory has to examine children's toys for forbidden colouring matters, which are similar to those above noted in sweets. In one set of 96 samples, 64 were condemned by the laboratory. A similar duty is performed upon paper used to wrap up articles of food, and of 64 samples which have been suspected and brought to the Laboratory only eight passed as innocuous. Here again are two very important points which should be included in the duties of the Public Analyst, under the guidance of an approved schedule of allowable colours for such things.

In conclusion, it seems as if the Public Analysts of each country excel in what is specially brought under their daily notice, and therefore we would do well to study the French ideas as to wine, while, on the other hand, they would benefit in some other matters by the perusal of our proceedings to a greater extent.

CONCLUSION OF THE SOCIETY'S PROCEEDINGS.

---

NEW TEST FOR NITRATES IN WATER.

By A. PERCY SMITH, RUGBY.

GRANDVAL and Lajoux (*Compt. Rend.* 101) propose the use of Phenolsulphonic acid as a test for minute quantities of nitric acid. I have experimented with it in determining the nitrates in potable water, and can recommend it as a rapid and delicate test.

It would seem to be a matter of little importance whether we determine nitrates or nitrites after ascertaining the total nitrogen, but since the metaphenylene diamine method for nitrites is open to several objections, I consider the estimation of nitrates by the subjoined method as in every way preferable. The objections to the M.P.D. process are:—The trouble of preparing the reagent. The length of time which elapses



the samples examined by me the latter alone were found; in some cases they still had their natural yellow colour, in other cases they were dyed. Bentley was the first one who observed also the presence of corolla tubes attached to the stamens of the saffron flower, and described them in the paper referred to. This adulteration seems to have attracted little attention; but it is evidently very extensively practised at the present time, as will be seen from the samples under examination, one-half of which are thus adulterated. When first noticed by me, and before it had been placed under the microscope, it was supposed to consist of meat fibres, which Duncan, Thomson, J. A. Paris, and others had enumerated among the adulterations of saffron. Some of the more recent German writers have repeated this statement; others merely give directions for the detection of such an admixture, and many, like all the recent French authors consulted by me, omit to mention it, doubtless because it has never been observed by them. Geiger describes these supposed fibres of smoked meat (ham) as being of uniform thickness, odourless, of little taste and not colouring the saliva, or, if dyed, imparting to it a slight yellow colour. This description applies equally well to the corolla tubes. It should be stated, however, that, according to Bentley and Trimen, the admixture of fibres of shredded beef is a common adulterant in Italy. The best means for detecting this adulteration is, without doubt, the microscope, which reveals the transverse striæ of meat fibres. The odour of burning meat differs materially from that of burning vegetable matter, and has been recommended as the principal test by most authors, even as late as 1865 by Marquart; but on experimenting with a few shreds only it may be misleading; it certainly should be regarded merely as a preliminary test, and the most reliable one, the microscope, is very properly insisted upon by careful modern writers. The corolla tubes observed by me were dyed in one case with red saunders, in the others with Brazil wood. The frequency of this adulteration, and its close resemblance to saffron, suggested the probability of its having been overlooked in samples examined before; but those saffrons—many of them adulterated—which I had procured between 1866 and 1876 were entirely free from the tubes; it is possible that this sophistication is only occasionally used, or merely for certain cheap grades. The petals of pomegranate, cut and twisted, were mentioned by Fée, Geiger, and by Dulk, and since their time by others, as being used for adulterating saffron, for which their crimson, and, after drying, brownish-purple colour render them fit to a certain extent; but they contain tannin, giving with ferric salt a blue-black colour, which was in no case observed with the corolla shreds taken from the samples under consideration. The petals of *Saponaria*, mentioned by some authors as adulterants, are almost white, and, to be used as an admixture to saffron, must therefore be dyed; seen under the microscope, they are nearly opaque, or merely translucent, while the corolla shreds—dyed and undyed—noticed by me were almost transparent, and their delicate structure was observed to be identical with shreds still attached to some of the filaments of crocus stamens, so that there can be no doubt of their origin. From the above it would seem as if for some commercial grades of saffron the entire crocus flower was utilized, though not in the condition suggested by Monthus, who recommended to simply dry the flowers, which he regarded as possessing the same medicinal properties as the stigma; he also states that acids

colour the flowers red, and ammonia green. Applied to calendula florets, acids render the yellow colour lighter, but ammonia darkens it. For the inferior grades of saffron the corolla tube is cut into pieces usually about 20 mm. ( $\frac{4}{5}$  in.) long, the stamens are pulled off, and the corolla lobes are torn into shreds; these different parts are then dyed, somewhat rolled and twisted, and mixed with true saffron in sufficient quantity to suit the cupidity of the manufacturer and the price offered by the dealer. Several of the substances used for adulterating saffron, and not derived from the saffron plant, have been mentioned above; in addition to these, a long array of vegetable substances are enumerated by different authors, and it is obvious that any material may be used which is thread-like or slightly clavate, or may readily be twisted into such a shape, and, in addition to this, possesses sufficient flexibility and may be dyed in close imitation of the natural colour of saffron. It would, therefore, seem to be superfluous to mention any of these substances, since, with the aid of the microscope—in most cases even to the naked eye, after a sample has been soaked in water—their different shape is at once revealed. The most important of these sophistications, besides those of mineral origin, consist of florets of composite flowers, more particularly of carthamus, which has been described above, and of the ligulate ray florets of *Calendula officinalis*, *Lin.* The latter attain a length of 20 or 25 mm. ( $\frac{4}{5}$  to 1 in.), and consist of a short tube and a long strap-shaped limb, 2 or 3 mm. ( $\frac{1}{12}$  or  $\frac{1}{8}$  inch) wide, terminating at the apex by three teeth, and marked by four delicate longitudinal veins, of which two are marginal and beneath the teeth united with the two central veins. The limb is smooth, but the outside of the tube is covered with rather long, very thin-walled, colourless hairs, which in the dried condition are more or less twisted and collapsed, so as to present a somewhat moniliform appearance. If not dyed, these florets, on account of their yellow colour, can be used for the adulteration of saffron only to a limited extent, perhaps not exceeding about 2 per cent. in weight, and may then readily be taken as portions of crocus styles. However, the dried florets are more largely used, and, though many red colouring matters may answer for the purpose, the principal ones employed appear to be either red saunders or Brazil wood, both of which will impart a colour similar to that of saffron. If dyed with red saunders, the colouring matter will not be taken up by water, but will dissolve in alcohol with a red, and in ammonia with a purple-red colour. If dyed with Brazil wood, the infusion in water will be tinged red in a few seconds, the colour becoming paler on the addition of an acid, and deeper red on the addition of ammonia; but the infusion is not blackened by salts of iron. A few fragments of a red wood, found in two or three of the samples of saffron, showed the same behaviour, and by microscopic examination and comparison with Brazil wood, were proved to be identical with the latter. In order to make the dyed calendula florets more closely resemble true saffron, they are rolled or twisted in such a manner as to assume an approximately cylindrical shape. In several of the samples examined a number of elongated bodies, curved near one end, and there divided into several nearly cylindrical parts, were observed, bearing some resemblance to the stamens of papilionaceous flowers, deprived of the anthers. But the hairs described before at once revealed their true nature, and after soaking these bodies in water they could be separated into from 6 to

12 calendula florets, which during the manipulation for rendering them saffron-like had been twisted together by the strap-shaped portion of the petals, while the tubular parts remained distinct. It is very likely that the ray florets of *Calendula arvensis*, *Lin.*, as stated by Guibourt, are used in precisely the same manner. The marigold of our gardens is very frequently this species, which is distinguished from *Calendula officinalis*, *Lin.*, by the more lanceolate stem leaves, the pale yellow and usually shorter ray florets, and the erect, instead of incurved, akenes of the *outer* row; the tubular portion is beset with the same kind of hairs. Logwood could not be detected in any one of the samples examined; the crocus tubes, in most cases, also the corolla shreds, and frequently the stamens, were dyed with either red saunders or Brazil wood. The stamens were also generally rolled in such a manner that the anthers were not readily recognised until after immersion in water, when they expanded and revealed their valves, the cells of which, near the margin, are somewhat irregularly quadrangular, and gradually change towards the connective to a transversely elongated form. Several of the earlier writers on saffron mention sand and pieces of lead as mineral adulterants. The use of chalk in this connection appears to have been first noted by Heræus, but was not reported to a scientific journal until 1870, though published in the *Gazette de Marseille* in the fall of 1860. The same adulteration was reported in 1869, in France, by Blacher, and in 1870, in Switzerland, by Rehsteiner, in England by Hanbury, and in the United States by me. Gypsum was also observed as an adulteration in the year 1869, and since that time both calcium salts, occasionally also the corresponding barium salts, have been invariably found as fraudulent admixtures in certain grades of Alicante saffron, rendered somewhat adhesive by means of honey, glucose, or glycerine, which substances, owing to their hygroscopic nature, have the additional advantage of preventing the saffron from getting too dry. The conclusion that may be drawn from these facts and statements is that saffron, in common with every medicinal article, should be carefully examined by the purchaser for all possible admixtures; for unceasing vigilance is the best if not the only guarantee for the purity of drugs.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

COLOURING MATTER IN GRANULATED SUGAR.\*—A sample of the "skimmings" of a syrup made from granulated sugar was sent to the laboratory for examination. The syrup was pale blue, and floating in it, shreds of what looked like albuminous matter of a darker blue. Nearly all the colour separated from the syrup in this way. This coloured matter was tested as follows: Its colour was destroyed by hydrochloric and acetic acids; but not by alkalis (caustic soda and potash); nor by chlorinated lime, nor would it dissolve in hot alcohol. Hence it was inferred to be neither indigo, aniline blue, sulphindigotic acid, nor prussian blue. The colouring matter responded to all the tests of ultramarine. The practical evil of this colour in sugar, used in this way, is that the acid fermentation sets free sulphuretted hydrogen, destroying the value of the article as food. In the small quantity used as "blueing" in sugar, ultramarine is probably harmless, except as above.

W. H. D.

\* From Public Health of Minnesota by Dr. Hewitt.



ACCORDING to R. Beusermann the following may be taken as accurate figures representing the melting and clarifying points respectively of the insoluble fatty acids of butter, and some other fats after saponification with potash, and throwing up by hydrochloric acid, and washing well with hot water:—

	Melt. pt. of the fat deg. C.	Percentage of insol. fatty acids.	Melt. pt. of insol. fatty acids.	End. pt. of insol. fatty acids.
Fat from cow's milk .. ..	34-35	87.77	42-43	45-46
Sesame oil .. ..	liquid	95.86	25-26	29-30
Cottonseed oil .. ..	"	95.75	39-40	42-43
Olive oil .. ..	"	95.43	23-24	26-27
Rapeseed oil .. ..	"	95.14	18-19	21-22
Peanut oil .. ..	"	95.86	31-32	34-35
Cacao fat from Maracaibo beans .. ..	25-26	94.59	48-49	51-52
Caracus beans .. ..	27-28	95.31	48-49	51-52
Trinidad beans .. ..	26-27	95.65	49-50	52-53
Porto Plata beans .. ..	28-29	95.44	49-50	52-53
Machala-Quayaquil beans .. ..	28-29	95.24	49-50	52-53

W. H. D.

ESTIMATION OF ACIDITY IN SOUR MILK OR KOUMISS.—At the meeting of the American Association, Dr. H. W. Wiley presented a method of estimating lactic and acetic acids in sour milk or koumiss. The caseine is precipitated by adding an equal volume of strong alcohol to the milk. After filtering, the acid is determined in the filtrate, using phenyl-phthalein as indicator. The same author spoke of the composition of koumiss made from cow's milk. The analyses show a lower percentage of alcohol and lactic acid, and a higher one of milk-sugar and fat, than are found in European samples, whether made from cow's or mare's milk.

W. H. D.

ESTIMATION OF WATER IN HONEY.—At the same meeting, Messrs. H. W. Wiley and F. V. Broadbent described a new method of estimating water in glucose, honeys, &c. Samples are dissolved in alcohol mixed with a weighed portion of sand, and dried. After cooling to 70° C., they are saturated with absolute alcohol, and dried again to constant weight.

W. H. D.

DR. WILEY has also devised the following apparatus for filtering the small quantities of liquid required in determining the end reaction of Fehling's solution by the use of ferrocyanide of potassium acidified by acetic acid—an operation in which the slow filtration through paper is quite unsatisfactory, since, owing to the redissolving of the suboxide, a clear end reaction is unattainable. The apparatus consists of a tube 5 to 6 inches in length, and  $\frac{1}{16}$  to  $\frac{5}{16}$  in. in interior diameter, one end of which is furnished with a slight rim, which allows the tying on of a strip of washed linen by means of thread; the edges of the other end are fused. Just before testing, the linen-covered end of the tube is introduced into a beaker of water containing finely divided asbestos in suspension, and by a sharp aspiration a firm asbestos felt is made to adhere

to the linen; the water having been poured out by inverting the tube, a partial vacuum is secured by suction, when the tube is introduced into the boiling solution to be tested, and instantly withdrawn, carrying with it a few drops of clear solution, which are immediately turned out into the white porcelain crucible, or other vessels holding the few drops of test-solution required. Finally, the asbestos pulp, with the adhering copper suboxide, is shaken off in a beaker of water acidulated with nitric acid, the tube quickly rinsed with water, and it is ready for another test.

W. H. D.

ACTION OF TANNIN ON FEHLING'S SOLUTION.—A. SONNENSCHNEIN. *Dingler's Polytech. Journ.* 1885, 555\*. Fehling's solution is almost invariably used to determine the amount of sugar in wine. As tannin is present in wine also, the author tested its action on Fehling's solution, and found that the tannin reduced it. .2983 gm. of tannin were dissolved in water, and made up to 500 c.c. 25 c.c. of this solution were boiled for 5 minutes with 25 c.c. of Fehling's solution, the  $\text{Cu}_2\text{O}$  filtered off, ignited, and weighed. From these experiments it was found that 1 gm. of  $\text{Cu}_2\text{O}$  corresponded to .4126 gm. of tannin (and to .4245 of grape-sugar); consequently, if .02 gm. of tannin be present in 100 c.c. of wine, this will be equal to .0206 gm. of grape-sugar, and would be estimated as such. The author has also heard from Prof. J. J. Pohl that glycerine also reduces Fehling's solution. As some of the other constituents of wine—such as succinic acid, &c.—probably act in a similar way, there is always an error in a wine analysis of a few tenths of 1 per cent. in the amount of sugar determined by means of Fehling's solution. The author does not think it probable that this reducing property of tannin can be made use of for the quantitative estimation of commercial tannic acid, as this contains impurities which would also have a reducing action on the copper solution.

E. E. B.

ADULTERATION OF OLIVE OIL.—Dr. Hiepe. *Rep. der anal. Chem.* 5, 326. The author states that olive oil, which is largely exported from Portugal, is frequently adulterated there with a Brazilian oil, which is extracted from the seeds of *Jatropha Curcas* (belonging to Euphorbiaceae). A good test for it is to add some nitric acid and copper to the suspected oil, when its colour, after some time, becomes an intense reddish-brown. No other oil acts in a similar way, and 10 % of this adulterant in an oil can be easily detected by this test.

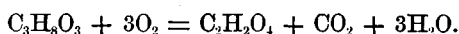
E. E. B.

DETERMINATION OF GLYCERINE IN DILUTE AQUEOUS SOLUTIONS AND IN FATS. By R. BENEDIKT and R. ZSIGMONDY. *Chem. Zeit.*—The results obtained by most of the usual methods—such as evaporating the liquid to dryness, extracting with ether and alcohol and again evaporating, are generally 2 or 3 per cent. lower than the calculated amounts—which are due, without doubt, to the volatilization of some of the glycerine during the evaporations. Zulkowsky's proposal to make use of Köttstorfer's method of analysing fats by titrating them with standard caustic potash, and calculating the

\* *Rep. Anal. Chem.* 5, 260.

amount of glycerine liberated from the amount of alkali required, since it is known that 3 molecules of potash liberate one of glycerine, is often not available—*e.g.*, when the fat contains any of the higher alcohols, myricyl, &c., or wax.

The authors have investigated the process proposed last year by W. Fox for determining the glycerine in linseed oil varnishes, which is based on the oxidation of glycerine to oxalic acid by potassium permanganate in a strongly alkaline solution, according to the equation :—



The oxalate can be precipitated by calcium chloride, and converted into calcium carbonate, 100 parts of which correspond to 92 parts of glycerine. The authors, by their experiments with this process, have shown that :—

(1.) The oxidation of the glycerine to oxalic acid and carbonic acid is not quantitative if a neutral or slightly alkaline solution be employed.

(2.) Less than the theoretical amount of oxalic acid is obtained if the permanganate be allowed to act in a *hot* solution.

(3.) The reaction is quantitative if the oxidation by permanganate take place in a strongly alkaline solution at the atmospheric temperature.

(4.) Oxalic acid may be boiled with permanganate in an alkaline solution without undergoing decomposition.

The authors' process is to take .2 to .5 grm. of pure glycerine (containing 91 per cent.  $\text{C}_3\text{H}_8\text{O}_3$ ), dissolve in 200 to 500 c.c. of water, with 10 grms. of potash (purified by alcohol), and then add a 5 per cent. solution of permanganate at the ordinary temperature, until the liquid is no longer green, but blue or blackish. The solution is boiled, when oxide of manganese is precipitated, and the solution becomes red; sulphurous acid is added till the liquid is colourless; it is then filtered and well washed with boiling water. To the boiling filtrate is added an excess of calcium acetate; the precipitate which forms contains, besides calcium oxalate, silica, and often calcium sulphate as well; the precipitate cannot, consequently, after ignition, be regarded as pure calcium carbonate; and the determination of the amount of oxalate is most accurately effected by titrating it with potassium permanganate in an acid solution, or alkali-metrically after ignition. The authors prefer the latter process, and dissolve the ignited precipitate in seminormal hydrochloric acid, and titrate back with  $\frac{N}{4}$  soda solution, using methyl orange as indicator. The authors found, in 4 analyses, 90.3 per cent., 91.4 per cent., 92.3 per cent., and 92.7 per cent., instead of 91 per cent of glycerine. If the solution of glycerine be very dilute, more than .4 grm. must be taken for analysis—*e.g.*, of a solution containing .05 per cent. of glycerine, 600 grms. were taken, and .0507 per cent. of glycerine found.

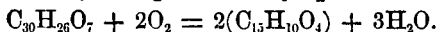
*Determination of the Glycerine in Fats.*—The fat is saponified with potash and pure methyl alcohol, the alcohol evaporated off, the soap dissolved in hot water, and decomposed with hydrochloric acid. The fatty acids are cooled, the solution filtered, neutralised with potash, then mixed with 10 grms. of pure potash, and the glycerine determined as directed above. Methyl is used in place of ethyl-alcohol for the saponification, because the latter, in a certain state of concentration and of alkalinity, is oxidised to

oxalic acid by the potassium permanganate. The complete removal of the alcohol, by repeatedly evaporating with small quantities of water, should be avoided, to prevent loss of glycerine by evaporation. The liquid to be oxidised contains, beside the glycerine, all the soluble fatty acids which were in the fat; by numerous experiments with acetic, butyric, and caproic acids, the authors have found that, under the existing conditions, they are not converted by the permanganate into any compounds which are precipitable by calcium acetate, and, consequently, their presence does not affect the determination of the glycerine.

*Determination of Fats in Wax.*—Beeswax contains no glycerine; and, as fats, on an average, contain about 10 per cent. of it, if the amount of glycerine be determined, the per-centage of fat will be found by multiplying it by 10. In two test mixtures of tallow and wax, containing respectively 50.9 per cent. and 28.4 per cent. of tallow, the authors found 4.93 per cent. and 3.00 per cent. of glycerine, corresponding to 49.3 per cent. and 30 per cent. of tallow. E. E. B.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

THE ACTIVE PRINCIPLE OF GOA-POWDER.—By M. PETIT\*.—According to Liebermann and Seidler the active principle of goa-powder is not chrysophanic acid, as was originally stated by Atfield, but chrysarobin. This substance, of which the formula is  $C_{30}H_{26}O_7$ , is prepared by exhausting goa-powder with boiling benzene; on cooling, the chrysarobin is deposited as a pale yellow powder, which is purified by repeated crystallizations with acetic acid. Chrysarobin dissolved in solution of potash, and treated by a current of air, becomes wholly changed into chrysophanic acid, thus:—



The acid thus formed is precipitated by hydrochloric acid, and the precipitate having been washed and dried, is exhausted in a displacement apparatus with petroleum-ether, from which, on cooling, the acid is deposited in beautiful yellow scales. Chrysophanic acid crystallizes in yellow prisms, fuses at  $152^\circ$ , is insoluble in water, soluble in 224 parts of boiling alcohol at  $86^\circ$ , and in 1125 of alcohol at  $30^\circ$ , soluble in benzene and in acetic acid. It is a feeble acid, soluble in alkalies; these at a temperature of  $195^\circ$  convert it into a substance which is analogous to purpurin, and which colours alum mordants a pomegranate red, and iron mordants a light greenish-blue. Treated with acetic anhydride and acetate of sodium it yields diacetyl-chrysophanic acid; with nitric acid, tetra-nitro-chrysophanic acid; with ammonia, a chrysophanamide. Chrysophanic acid is distinguished from chrysarobin by the following reactions:—

Chrysophanic Acid. Chrysarobin.

Concentrated sulphuric acid, Red colour, Yellow colour.

Fusion with potash, Blue mass, Brown mass.

Soluble only in concentrated potash solution with pelourin green colour.

W. H. D.

QUANTITATIVE DETERMINATION OF CINCHONA ALKALOIDS.—Y. SHIMOYAMA, Arch. d. Pharm. and Ph. Zeit., gives the following directions. At least 0.5 gm. of the

\* *Journal de Pharmacie et Chimie.*

alkaloids are dissolved in a beaker in about 30 or 40 c.c. of water, with the addition of the least possible quantity of very dilute acetic acid, with gentle heat, the solution filtered from any undissolved substances into a tarred beaker, the filter washed, and the united liquid neutralised with very dilute soda solution. If necessary this should be filtered. The filtrate is mixed with a sufficient quantity of solution of sodium oxalate saturated at 18°C., the proper amount being 1 c.c. for each 0.1 gm. of the mixture of alkaloid taken for analysis. The liquid is now concentrated on a water bath to 8 or 10 gms., until a copious precipitate makes its appearance on cooling. Should a slimy mass separate during the concentration, it must be filtered off and well washed with boiling water. To the contents of the beaker there are now added 10 to 15 c.c. of water, and the whole stirred until the slimy mass which separates, together with the precipitated oxalate, is completely redissolved, and the whole is then set aside for three hours at a temperature of 18°C., during which time it should be repeatedly stirred. The contents of the beaker are now weighed, the precipitate is collected on a double filter, and washed a few times (with the aid of a filter-pump) with a solution of oxalate of quinine saturated at 18°C. In order to dissolve any co-precipitated oxalate of cinchonidine, the precipitate is now washed with the above-mentioned saturated solution of oxalate of quinine, into a flask, the latter briskly shaken during fifteen or twenty minutes, and then set aside for two hours at 18°C., the shaking being occasionally repeated. Finally, the precipitate is collected upon a double filter (dried at 110°C., and tarred), well washed with the saturated solution of oxalate of quinine, with the aid of the filter-pump, then dried and weighed. From the weight thus found it is now necessary to deduct the amount of quinine oxalate which had been soaked up during the washing by the filter and by the precipitate. According to Shimoyama's investigations, this amounts to 0.00069 gm. for every gramme of solution of oxalate of quinine. To make the correction, the obtained quantity of oxalate of quinine is first deducted from the total weight of the contents of the beaker. The difference represents the quantity of mother liquid. This is then multiplied with 0.00064, and the product added to the quantity of quinine oxalate found. The amount of pure quinine is found by multiplying the figure obtained for oxalate of quinine with 0.878 (since 1 gm. of oxalate of quinine contains 0.878 gm. of pure quinine).

W. H. D.

In a recent number of the *Journal de Pharmacie* a new colour test for codeine and æsculine is described by M. Raby. To the codeine placed in a watch glass, add two drops of ordinary hypochlorate of soda, dilute, and then add four drops of concentrated sulphuric acid. After mixing with a glass rod, a superb clear blue colouration is produced. In experimenting with thirty of the best known alkaloids, the author found none that produced any colouration which would be confounded with that produced by codeine in the above circumstances. An equally beautiful colouration is, however, produced by æsculine when somewhat differently treated. Bromine cannot be substituted for chlorine in performing this test.

W. H. D.

MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

QUANTITATIVE VALUATION OF RESIN IN SOAP.\*—This method is based upon the behaviour of sodium chloride to separate out the fat soap only, while the resin soap is retained in solution. Dr. Heiner's experiments made with soap containing 20 per cent. of resin, have on an average  $1\frac{1}{2}$  per cent. less of resin. Two samples of equal weight are treated; one is decomposed with  $H_2SO_4$ , and the fatty acids containing the resin are weighed. Dissolve the other sample in water, treat with solution of NaCl, separate the liquid containing the resin and glycerine from the soap mass, wash with solution of NaCl, dissolve again in water, precipitate again, wash repeatedly with the salt solution, and decompose the pure soap with  $H_2SO_4$ , and weigh. The difference in weight of the two results gives the weight of the resin.

W. H. D.

DETERMINATION OF SMALL QUANTITIES OF SODIUM CHLORIDE IN POTASSIUM CHLORIDE.

By F. RÖTTGER and H. PRECHT. Ber. d. Deutsch. Chem. Gesellschaft. 1885. No. 13, p. 2076. When potassium chloride contains as little as .5 per cent. of sodium chloride, it is a very tedious and expensive operation to determine the exact amount of the latter by means of the only accurate process known, *i.e.*, by platinum chloride; in the Stassfurt trade, it is usual to determine all the other constituents of the potash salt, and find the sodium chloride by difference; this is both troublesome and very inaccurate. The authors directed their attention to processes for removing most of the potassium chloride before analysing the mixture; this they found could be effected in the following ways:—

(i). Precipitation of the potassium as acid tartrate of potash, filtering, and using for the analysis the mixture of potassium and sodium chlorides obtained by evaporating the filtrate.

(ii). The conversion of the sodium chloride into sodium hydrate by potash, and removal of the soda with absolute alcohol.

(iii). The direct removal of sodium chloride by washing with dilute alcohol.

The authors consider the third process the best, and tested its accuracy by making a mixture of 99.5 parts by weight of potassium chloride with .5 of sodium chloride; four samples, of 20 grammes each, of this mixture were taken; two were treated with 40 grammes of 90% (by weight) alcohol, and two with 70 grammes of 92.5% alcohol; these were frequently shaken up for half an hour, filtered, and well washed with more alcohol. The filtrates were evaporated to dryness, weighed, and the potassium chloride in them estimated by platinum in the usual way, the sodium chloride being determined by difference. The results of the four analyses varied between .497% and .502% of sodium chloride, the mean being .4985 instead of .5000. Commercial potassium chloride (98% KCl), was analysed in the following way:— 20 grammes of it were treated with 40 grammes of 90% alcohol, and after half an hour, .5 c.c. of a 10% solution of potassium carbonate was added drop by drop. The addition of the potassium carbonate was essential to precipitate the magnesium

\* Monit. Scientif.

chloride present; more of it is not required, as a 98 % potassium chloride does not contain more than .2 % of magnesium chloride. The insoluble residue is washed a few times on the filter, the filtrate evaporated, weighed, and the potassium chloride determined by the platinum solution. If the analysis must be made as rapidly as possible, the authors recommend the following modification:—20 grammes of the sample are placed in a flask, graduated to 110 c.c. (10 c.c. correspond to the volume of the potassium chloride taken), it is then about three-quarters filled with 90 % alcohol, shaken well for half an hour, half a c.c. of the 10 % sodium carbonate solution is then added, and the liquid made up to the mark; when clear, 50 c.c. of the solution is drawn off with a pipette, and treated as above.

E. E. B.

VOLUMETRIC DETERMINATION OF POTASSIUM.—M. Dubenard. Ann. Agron. 11, 326. The potassium salt, acidified with nitric acid, is precipitated by a standard solution of sodium chloroplatinate, the excess of which is reduced by zinc, and then determined by means of a standard solution of silver nitrate. The process is applicable to all the compounds of potassium, *e.g.*, sulphates, chlorides, nitrates, etc. 12 to 15 parts of sodium chloroplatinate are dissolved in 100 of alcohol, and 12-15 parts of silver nitrate in 100 of water. The solutions are standardised as follows:—10 c.c. of the platinum solution are reduced by boiling for a minute with a small quantity of zinc in powder, the whole of the platinum being precipitated, and all the chlorine remains in solution as chloride of zinc and of sodium; the solution is made up to 100 c.c. and filtered. In 50 c.c. of the filtrate the chlorine is determined by titrating with the silver solution, of which, say, 40 c.c. are required. Then  $40 \times 4 = 160 =$  the number of c.c. which corresponds to 20 c.c. of the platinum solution. Half a gramme (0.5 gm.) of potassium nitrate (or sulphate) is then dissolved in a few c.c. of water, acidified with nitric acid, the potassium precipitated with 20 c.c. of the platinum solution, and the volume made up to 100 c.c. with alcohol (95 per cent.); the solution is filtered, and 50 c.c. of it reduced by boiling with a little zinc, again made up to 100 c.c., and filtered; 50 c.c. of this filtrate is titrated with the silver solution, of which, say, 12 c.c. are required; then  $12 \text{ c.c.} \times 4 = 48 \text{ c.c.}$ , subtracted from 160 c.c., represents the amount of chlorine precipitated as potassium chloroplatinate by the half gramme of potassium nitrate; thus  $(160 - 48) \text{ c.c.} = 112 \text{ c.c.}$  corresponds to .5 gm. of potassium nitrate, *i.e.*, to .232 of potash ( $\text{K}_2\text{O}$ ). To determine now the amount of potassium in any salt the process just described is followed, taking 10 c.c. of a solution containing 50 grms. of the salt per litre. The amount of chlorine present in the salt, as a chloride, must be determined by titration, and allowed for before calculating the amount of potassium. Thus, if the 10 c.c. of the solution (*i.e.*, .5 gm. of the sample) required 8 c.c. of the silver solution and 27 c.c. were required of it after the reduction with zinc, the calculation would be  $(27 \times 4 - 8) \text{ c.c.} = 100 \text{ c.c.}$  and  $\frac{100 \times 0.232}{112} = .2076$  gm. of potash in .5 gm., *i.e.*, the sample contained 41.52 per cent. of  $\text{K}_2\text{O}$ .

E. E. B.

A NEW METHOD OF SEPARATING GLOBULINE FROM ALBUMEN. W. WICHAIRLOW.\*  
Journ. of the Russian Chem. Soc., 1885 [1.] 348.—The author has previously shown that albuminoids are completely precipitated by ammonium sulphate, and that this salt is an admirable reagent for detecting them. In this paper the author describes his process for separating the albuminoids of serum. A definite volume of the serum (25 to 30 c.c.) is treated with ammonium sulphate till all the albuminoids have been precipitated; the solution is filtered, and the precipitate washed with a saturated solution of ammonium sulphate until free from chlorides; it is then dissolved in the smallest possible quantity of water, and dialysed for two to three days, the diffused portion being changed every two or three hours. Water is then added to the solution to the amount of about a third the volume of the solution originally taken. It is then filtered, when the globuline remains on the filter, whilst the albumen pass through in the filtrate, which may be proved to obtain no globuline by saturating it with magnesium sulphate, when the liquid remains quite clear. Pure albumen possesses an acid reaction, but it is most probable that the compounds allied to globuline are alkaline. The author considers that globulines and albumens are the products of the decomposition of a complicated group of albuminoids in serum, and that albuminoids ought not consequently to be regarded as very stable organic compounds.

E. E. B.

VOLUMETRIC DETERMINATION OF UREA. By T. PFEIFFER, *Zeitschr. für Biol.* 20 540 to 565.—The author has examined Rautenberg's modification of Liebig's process for titrating urea. 10 c.c. of the urea solution were treated, before the addition of the nitrate of mercury solution, with an excess of pure calcium carbonate, and the end of the titration recognised by the brown ring which a drop gives with sodium bicarbonate, which must be free from sodium carbonate. In this process less of the nitrate of mercury solution is required than in Liebig's or Pflüger's; a solution, of which 1 c.c. corresponds to 10 milligrams of urea, only contains 60.186 grms. of mercury per litre (the acidity of the solution is not of great consequence in this process). Liebig's "dilution co-efficient" (.02) is said by Henneberg, Stokmann, and Rautenberg not to be constant, and must be determined for each solution of mercury. In titrating urine a correction must also be made for the errors introduced by the presence of chloride of sodium. Rautenberg proposes to eliminate the errors by rendering the mixture of urine and baryta slightly acid with nitric acid, and adding the mercury solution till a permanent turbidity is produced. The author finds that this is suitable for the urine of herbivorous animals, which is free from hippuric acid, but that bad results are obtained by it with human urine; in this case it is advisable to precipitate the chlorine with silver nitrate, a slight excess of which does not affect the titration of the urea.

E. E. B.

DETERMINATION OF CARBON IN IRON.†—By M. Clere. *Stahl und Eisen* 5, 259. The carbon is separated from the iron by any suitable process, and converted into carbonic acid, which is allowed to pass through a series of S shaped tubes, connected by india-

\* *Ber. d. deutsch. Chem. Gesellsch.* 1885, No. 13, p. 478.† *Chem. Zeit.* 9, 1356.



rubber rings, each of which contains 1 c.c. of a liquid corresponding to .0005 grm. of carbon. As soon as the first tube is saturated, the carbonic acid passes on to the second, and so on; when all the carbonic acid has been liberated by counting the number of tubes which have been saturated, the amount of carbon (to within .0005 grm.) is at once known. The standard liquid is made by dissolving 4.65 grms. of pure potash in a litre of water, and as an indicator, 0.025 gramme of potassium manganate are added to every 60 c.c. of it. As soon as all the potash in one of the tubes has been carbonated, the current of carbonic acid converts the indicator into permanganate of potash, which is at once seen by the change of colour. The extreme rapidity is one of the chief advantages of this process. The results obtained by it agreed very closely with those found by Boussingault's method of analysis.

E. E. B.

DETERMINATION OF NITROGEN IN MANURES CONTAINING NITRATES.—By O. Reitmar. Rep der Anal. Chem. 5, 261. To avoid making a determination of the nitrogen by Dumas' method in manures which contain nitrogen in the form of nitrogenous organic compounds, as well as of nitrates, the following process is recommended:—1 grm. of the finely divided sample is covered in a shallow dish with 3 c.c. of 50 per cent. sulphuric acid, and the mixture heated in an air bath at 60° to 80° for three to four hours, and then for an hour at a temperature of 120° to 130°, when a moist mass remains, from which the nitric acid has been completely expelled. The nitrogen present in the residue in the form of an organic compound, or as an ammonium salt, can then be determined by Kjeldahl's or Will-Varrentrap's method. The sample must be finely ground, or it is likely to spit when warmed with the acid; also the sulphuric acid should contain about 50 per cent.  $H_2SO_4$ , as if weaker the reaction takes place too slowly, and if more concentrated the action is too violent, especially if any readily reducible organic compounds be present.

E. E. B.

PYROGALLIC ACID AS A REAGENT FOR DETECTING MINUTE QUANTITIES OF NITRIC ACID. By PROF. CURTMAN, Pharm. Rundschau 3, 154.—It has long been known that nitric acid forms a deep brown substance with pyrogallic acid, which possesses an intense colouring power. This property can be made use of for the detection of nitric acid in drinking water, and is better than diphenylamine, as  $\frac{1}{10}$  milligramme of nitric acid can be readily detected by it in a litre of water. To the sample under examination a small quantity of pyrogallic acid is added—less than 1 milligramme to each c.c. of water is sufficient—and then 10 to 12 drops of concentrated sulphuric acid are cautiously dropped into the test-tube so as to form a separate layer at the bottom of the tube. If nitric acid be present a *brown* coloration becomes visible at the junction of the two liquids, or a *yellow* one, if only a very minute quantity of nitric acid be present. E. E. B.

DETERMINATION OF PHOSPHORIC ACID IN PHOSPHATIC SLAGS. By DR. C. BRUNNE-MANN. Chem. Zeit., 9, 1335. The most accurate process for analysing slags, consists in first fusing them with potassium chlorate and sodium carbonate, for if they be merely treated with aqua regia, the amount of phosphoric acid found is always about

one per cent. too low. The fusion takes a long time, and as not more than about three grammes can be taken for the analysis, it is very difficult to obtain a representative sample. To meet these objections the author devised the following process:—10 grammes of the phosphatic slag are boiled for three-quarters of an hour, in a beaker holding about 400 to 500 c.c. with 10 c.c. of sulphuric acid, 50 c.c. of hydrochloric acid, and 20 c.c. of nitric acid (all concentrated). The solution, which is mixed with silica, carbon, and calcium sulphate, is treated with a small quantity of hydrochloric acid, and made up to a litre. 50 or 100 c.c. of the clear solution are then evaporated till most of the nitric and hydrochloric acid has been driven off; the sulphuric acid is very carefully neutralised with dilute ammonia (1:5), the solution evaporated to dryness, and the residue treated in an air bath at 110°C., for half an hour. It is then taken up with nitric acid, filtered, and the phosphoric acid determined in the filtrate by the molybdate method. The author gives the results of a number of comparative analyses made by his process, and by the above fusion method, which agreed very satisfactorily. The author also points out that the addition of concentrated sulphuric acid to a mixture of aqua regia and ignited oxide of iron enables this to dissolve rapidly, and the necessity for heating it for some hours is thus avoided.

E. E. B.

DETERMINATION OF SILICON IN IRON.—By L. Blum. Chem. Zeit. 9, 13. The author's process depends on the decomposition by bromic acid of the compounds of iron with carbon, phosphorus, sulphur, and silicon. Five grammes of the iron are placed in a boiling flask, 100 c.c. of water added, and then 150 c.c. of bromic acid. A violent action ensues, and solution soon takes place; the liquid is then washed into a large evaporating basin, after being boiled for a short time to expel the excess of bromic acid. 10 grms. of ammonium chloride are added, and the solution is then evaporated to dryness. The residue is moistened with concentrated sulphuric acid, taken up with water and filtered. The residue on the filter is washed several times with water, then once with bromic acid, twice with warm water, again with bromic acid, and finally with water till the washings are colourless. The moist precipitate and filter is ignited till all the graphite has been burnt off, when perfectly white silica remains. The ammonium chloride is added to hasten the evaporation of the ferric chloride, and to make the residue dry more readily, and thus render the separation of the silica more complete. This method is both quick and accurate. The author has proved, by numerous experiments that the glass flask is not attacked by the bromic acid, and as the silica obtained is perfectly white, it cannot be contaminated with any compounds of iron; the author calls attention to these two points, because the results obtained by his method are a few thousandths higher than those obtained by other chemists, which he consequently maintains must be too low.

E. E. B.

#### A CHEMICAL CANDIDATE FOR PARLIAMENT.

WE observe among the names of the Metropolitan Candidates at the coming Parliamentary Election, that of Mr. James Bigwood, M.A. Camb.

This gentleman, should the electors give him their confidence, will represent in the next Parliament the Borough of East Finsbury; in which district are situated the works

of Messrs. Champion and Co., the very old-established firm of which Mr. Bigwood is now head. Mr. Bigwood was an early member of the Society of Chemical Industry, and has long interested himself in the anti-adulteration movement. From the first he set his face against the time-honoured practice of adulterating mustard. At one time, and that not long ago, it was commonly maintained that pure mustard was not fitted for use, unless its "pungency were ameliorated" by addition of flour coloured with turmeric. In spite of strong opposition, founded on very obvious grounds, Mr. Bigwood succeeded in introducing to the public the use of pure mustard.

He has also long been an advocate for the manufacture of vinegar direct from pure alcohol, instead of from malt wort, as is now the custom in this country. Some time ago he established a distillery and vinegar factory combined, and there manufactures vinegar by a process of direct oxidation of alcohol without the intervention of *micoderma aceti*, or vinegar plant, an operation which many have considered to be impossible. As is well-known, vinegar produced by this method of acetification possesses absolute and positive keeping powers, greatly superior to those of malt vinegar of corresponding acetic strength.

Should this gentleman obtain his seat, there can be little doubt but that the "Chemical" interest would find in him a worthy "Champion" of its cause.

---

### C O R R E S P O N D E N C E .

---

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

---

#### DISUSE OF BURETTE PINCHCOCKS.

*To the Editor of THE ANALYST.*

DEAR SIR,—The plan advocated in your last issue by M. A. Lebrasseur has been described in the two last editions of my "Volumetric Analysis," and has been in use by me occasionally for the past ten years.

London Street, Norwich,  
October 5th, 1885.

Yours truly,

FRANCES SUTTON.

---

#### BOOKS, &c., RECEIVED.

ANNUAL Report of the Milk Inspector of the City of Boston; Notes on New Drugs and Preparations of the British Pharmacopœia, 1885, by G. T. W. Newsholme; Proceedings of the Californian Pharmaceutical Society and College of Pharmacy for 1883 and 1884; Public Health in Minnesota; Report on the Chemical Examination of the Waters of the Public Wells of Albany, N.Y., by Willis G. Tucker, Ph. D.; American Veterinary Review; American Chemical Review; American Druggist; American Grocer; American Journal of Pharmacy; Brewer's Guardian; British and Colonial Druggist; Canadian Pharmaceutical Journal; Chemist and Druggist; Country Brewer's Gazette; Illustrated Science Monthly; Independent Journal; Invention; Journal of the Society of Chemical Industry; Le Mouvement Hygienique; Medical Press; Medical Record; The Miller; Monthly Magazine of Pharmacy and Chemistry; New York Analyst; Pharmaceutical Journal; Pharmaceutical Record; The Polyclinic; Popular Science News; Reportorium der Analytischen Chemie; San Francisco News Letter; Science; Scientific American; Society of Arts Journal; Hospital Gazette.

---

#### NOTICES TO CORRESPONDENTS.

E. W., New York.—The report of the meeting of the American Society of Public Analysts, arrived just one day too late, and we will therefore publish the same next month, together with the report of the October meeting, should it arrive in time.

All communications to be addressed to 325, Kennington Road, London, S.E.

# THE ANALYST.

DECEMBER, 1885.

## CONTENTS.

PAGE	PAGE
PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS:—	MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS:—
(a) REPORT OF MEETING .. .. . 215	(a) TEST FOR IODOL—VULPIUS .. .. . 229
(b) REPORT OF MILK COMMITTEE .. .. . 216	(b) DETECTION OF SALICYLIC ACID—CUBTMANN .. .. . 229
(c) NOTE ON HONEY—O. HEHNER, F.I.C. . . . 217	(c) EXAMINATION OF GUM ARABIC FOR ARTIFICIALLY PREPARED GUMS—H. HAGER 230
(d) ON THE COMPOSITION OF MARES' MILK AND KOUMISS—DR. VIETH, F.C.S. . . . 218	MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY—
(e) NOTE ON BEEF TEA—O. HEHNER, F.I.C. 221	(a) VOLUMETRIC DETERMINATION OF SOLUBLE PHOSPHORIC ACID IN SUPERPHOSPHATES—A. EMMERLING .. .. . 230
PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS:—	(b) SEPARATION OF THE HALOGENS—CAUSSE 230
REPORT OF MEETING .. .. . 223	(c) DETECTION OF ROSIN OIL IN MINERAL OILS—DEMSKI AND MORAWSKI .. . 231
ORIGINAL ARTICLE:—	(d) SEPARATION OF IRON AND ALUMINA—ILLINSKY AND KNORRE .. .. . 232
THE RELATION OF COCOA-NUT OIL TO THE VARIOUS METHODS OF BUTTER ANALYSIS—R. W. MOORE .. .. . 224	CORRESPONDENCE .. .. . 233
MONTHLY RECORD OF RESEARCHES IN FOOD ANALYSIS:—	LAW REPORT .. .. . 234
(a) ALKALOIDS IN OLD FLOUR—BALLAND .. 228	BOOKS RECEIVED, &c. .. .. . 234
(b) EXAMINATION OF FOOD STUFFS—A. EMMERLING .. .. . 229	NOTICES TO CORRESPONDENTS .. .. . 234

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

### IMPORTANT MEETING.

The first ordinary meeting of the Session 1885-6 was held at the rooms of the Chemical Society, Burlington House, on Wednesday, November 11th, at 8 o'clock. The chair was taken by the President, Dr. Hill.

The minutes of the June meeting were confirmed, and the report of the country meeting at Dublin, as reported in *The Analyst*, was ordered to be entered in the minute book as correct, and confirmed at the next meeting.

A paper by Dr. P. Vieth, "On the Composition of Mare's Milk and Koumiss," was read and discussed.

A paper by Mr. Otto Hehner, F.I.C., "On Beef Tea," was read in abstract by the author, and ordered to be printed in full in *The Analyst*. The same gentleman also contributed a note on the "Analysis of Honey," which was duly discussed.

The President stated that the labors of the Milk Committee had extended over a considerable time, and no doubt the members of the Society were desirous that some

result should be arrived at without delay. He now wished to bring forward the Committee's report, so that the Members might think it over and be prepared for its discussion at the December meeting. He therefore called upon the Secretary to read the following—

#### REPORT OF THE MILK COMMITTEE.

[This Committee was appointed by the Council of the Society of Public Analysts, in December, 1883, in consequence of a resolution passed at the General Meeting of the Society held in November, 1883, and originally consisted of the following gentlemen:—

Mr. Adams, F.C.S., &c.	Public Analyst for Kent.
„ Baynes, F.I.C., &c.	„ „ „ Hull, &c.
„ Carter Bell, F.I.C. &c.	„ „ „ Cheshire.
„ Wynter Blyth, F.C.S., &c.	„ „ „ Marylebone, Devon, &c.
Dr. Dupré, F.R.S., &c.	„ „ „ Westminster.
Mr. Dyer, F.I.C., &c.	Analytical and Agricultural Chemist.
„ Estcourt, F.I.C., &c.	Public Analyst for Manchester.
„ Harland, F.I.C., &c.	„ „ „ Greenwich, &c.
„ Hehner, F.I.C., &c.	„ „ „ Derbyshire, &c.
„ Heisch, F.I.C., &c.	„ „ „ Lewisham, &c.
Dr. A. Hill, F.I.C., &c.	„ „ „ Birmingham.
„ B. Hill, F.I.C., &c.	„ „ „ Warwickshire.
Mr. Kingett, F.I.C., &c.,	Analytical Chemist.
Dr. Muter, F.I.C., &c.,	Public Analyst for Lambeth, &c.
„ Vieth, F.C.S., &c.,	Analyst to the Aylesbury Dairy Co.
Mr. Wigner, F.I.C., &c.,	Public Analyst for Plumstead, &c.

The last-named gentleman having died during the investigation.]

#### TO THE COUNCIL OF THE SOCIETY OF PUBLIC ANALYSTS.

GENTLEMEN,

In accordance with your instructions, we have fully considered the subject referred to us. During the two years that we have been engaged in this matter, we have met very frequently, and have also undertaken the performance of a very large number of analyses of milk, and of the fat extracted therefrom. We have passed in review, and practically studied, all the principal processes of milk analysis that have been proposed within the last fifteen years. Among those specially investigated may be noted (1) Mr. Wanklyn's process, (2) Mr. Carter Bell's process, (3) Dr. James Bell's process, (4) Soxhlet's extraction process, and (5) Mr. Adams' paper extraction process. We have come, after mature consideration, and after receipt of a very able report from a sub-committee, consisting of Messrs. Baynes, Hehner, B. Hill, and Vieth, to the following conclusions:—

#### I. AS TO THE PROCESS OF ANALYSIS.

That in future, the method to be recommended for adoption by the members of the Society of Public Analysts be as under:—

(1.) *Total Solids*.—These to be estimated by evaporating in a platinum dish

about 5 grammes of milk. The residue to be dried to practical constancy, at the temperature of a water oven or water bath.

(2.) *The Process of Fat Extraction.*—Pipette 5 c.c. of milk into a beaker 2 inches deep by  $1\frac{1}{4}$  inch in diameter; weigh, and place into it one of Mr. Adams' coils, viz., rolled-up strip of white demy blotting paper (sharply cut to  $2\frac{1}{2}$  inches wide, and 22 inches long), which must have been previously extracted with ether in a Soxhlet apparatus, and the ether driven off. When as much as possible of the milk has been taken up by the paper, the coil is removed and placed dry end downwards upon a slip of glass, and the beaker (which should be kept covered by a bell-jar during the absorption of the milk) is at once re-weighed. Dry the coil in a water oven for a period of one to two hours, and extract the fat by ether in a "Soxhlet", apparatus, twelve syphonings at least being necessary; the flask in which the solution is collected being as small and light as possible. Boil off the ether, and place the flask in a water oven, in a horizontal position, and dry to constancy; allow to cool for about ten minutes, and weigh.

(3.) *The "Solids-not-fat"* in all cases to be determined by difference.

We strongly recommend that in all cases the specific gravity be taken as a useful control.

## II . AS TO STANDARDS OR LIMITS.

We further recommend that, concurrently with the method above laid down, the following be the limits below which milk should not be passed as genuine, viz:—Total solids, 11·5 per cent., consisting of not less than 3 per cent. of fat, thus leaving not less than 8·5 per cent. of non-fatty solids.

Signed on behalf of the Committee,

ALFRED HILL,  
President.

On the motion of Dr. Muter, the discussion of the above report was adjourned until Wednesday, 9th December next, at 8 p.m.

A hope was expressed that as many members as could conveniently come up to town would be present, and all other persons interested in the matter were also invited to attend the discussion, and take part therein, so that this important question might be thoroughly ventilated, and a perfect agreement arrived at between all parties.

## NOTE ON HONEY.

By OTTO HEHNER.

*Read at the Meeting, November 11th, 1885.*

SINCE laying before the Society some results of my researches on honey, and giving directions for its analysis, an eminent firm of sugar refiners, Messrs. A. Lyle and Co., have brought into commerce a product termed artificial honey, which, in all but flavour and aroma, resembles genuine honey most closely, and consisting as it does essentially of dextrose and levulose, as does honey, the methods previously described are not capable of discriminating between it and pure honey. Whilst there is no pretence at the hands of the manufacturers to pass off their article as pure honey, it may

with certainty be surmised that sooner or later it will be used, by others, fraudulently as substitute for, or admixture with, honey. Analysts may therefore welcome a simple test which allows of its recognition.

On estimating the proportion of phosphoric acid in honeys of undoubted genuineness, I found in four samples the following percentages :—

Light super honey.	Light extracted honey.	Golden honey.	Brown extracted honey.
·014	·013	·016	·035

A sample of aphidic, brown honey dew, gave ·032 p.c.

Messrs. Lyle's artificial honey did not yield a recognisable trace of phosphoric acid when operating on 50 grammes.

Mixtures, on the other hand, of glucose, made from grain, a honey, such as lately flooded the London market, gave ·085, ·108, and ·107 per cent. of  $P_2O_5$ .

It appears, therefore, that pure honey contains from 1 to 3 hundredths of a per cent. of  $P_2O_5$ , grain glucose, as was to be expected, a much larger quantity, whilst honey made from cane-sugar is devoid of it.

The proportion of  $P_2O_5$ , and also of total ash, as far as my experience goes, stands in direct relation to the colour of genuine honey; the lighter it is, the less the quantity of mineral constituents, 50 grms. of light pure honey leaving but an exceedingly slight quantity of ash.

The ash of pure honey, and also of Lyle's artificial preparation, is always strongly alkaline; that from glucose, or glucose mixtures, owing to the mineral acid employed in its manufacture, is *always neutral*. This fact furnishes a most simple qualitative test for starch-glucose in honey.

For the estimation of such small quantities of phosphoric acid as one has to deal with in honey, I can strongly recommend the method described by me in *THE ANALYST*, Vol., IV. 1879. For rapidity, simplicity, and accuracy, I believe it to be unsurpassed.

#### ON THE COMPOSITION OF MARES' MILK AND KOUMISS.

By DR. P. VIETH, F.C.S.

*Read at the Meeting, November 11th, 1885.*

VISITORS to the International Health Exhibition held in London last year will remember the lively scene, which the open space south of the main gallery presented. There was a stud consisting of two stallions, one gelding, and fifteen mares with their foals, brought to London all the way from the steppes of the south-eastern part of European Russia; there was half-a-dozen people, males and females, natives of the same part of the world, living in their bee-hive shaped kubitkas, and taking care of the horses; and there was a Russian log-cottage, in which koumiss was prepared, and where mares' milk could be had in its fresh as well as in the fermented state, in tumblers or shallow bowls turned of wood, the drinking-vessel of the Tartars. All this was exhibited by "Dr. Carrick's Koumiss and Condensed Mares' Milk Factory at Orenbourg," and to me it was one of the most interesting exhibits.

Dr. Carrick had the great kindness to offer me for analysis as many samples of milk and koumiss as I liked to have, and I availed myself of this rare opportunity

with the greatest pleasure, the more so as the number of analyses of mares' milk and koumiss previously published is very limited, and in the majority of the cases there is a great lack of particulars as to the origin of the samples, the conditions under which they had been taken, etc.

The mares were from five to six years old, and had foaled in April and May. During the night the foals were kept in the same enclosure with the mares, and sucked their mothers, while from 8 a.m. to 6 p.m. they were separated, and the mares milked every other hour, five times a day in all. A good milch mare yields about one gallon of milk per day.

Mares' milk is of chalky white colour, of sweet and at the same time somewhat harsh taste, and of aromatic flavour. The re-action of the milk, when quite fresh, was alkaline with very few exceptions, in which the milk was neutral. On some very warm days lactic and alcoholic fermentation set in spontaneously within less than twenty-four hours.

As regards the analytical methods employed for the analysis, the following short remarks may suffice:—Solids (or water), five grammes of milk evaporated and dried to constancy; ash, solids ignited at dark red heat; fat, ten grammes dried with plaster of Paris and extracted in a Soxhlet apparatus with pure ether; proteids, ten grammes precipitated with sulphate of copper, following Ritthausen's method; sugar, not determined but taken by difference.

In order to find out if there was any decided difference in the composition of the different milkings, eleven average samples of the mixed yield of all the fifteen mares were taken and analysed. They were found to agree very closely in their composition, as is shown by Table I.

Another matter of interest was to ascertain the variations occurring in the composition of the milk of individual mares. For this purpose, a sample of the milk of each mare was analysed. Table II. contains the results of these fifteen analyses. It is surprising to find the respective figures for the component parts vary in so very narrow limits:—

Besides these twenty-six analyses, six others were made relating to milk of two mares which were temporarily fed on a "patent feeding bread for horses." The analyses, Table III., show that there was an improvement as to the quality of the milk, but it was accompanied by such a decline in the quantity, that feeding that "bread" was given up as unprofitable.

I should like here to call attention to some analyses of condensed mares' milk, published by me at former occasions, and to the supposed composition of the milk employed, when in its fresh state. (See THE ANALYST, Vol. VIII., page 82, and Vol. IX., page 79.)

It was previously mentioned that in hot weather mares' milk soon gets fermented, lactic and alcoholic fermentation apparently setting in simultaneously. If fresh milk is mixed with some which is well fermented already, the mixture kept under suitable conditions, and treated according to certain rules, alcoholic fermentation will proceed very quickly, and a preparation result, called koumiss.



The mixture of fresh and fermented mares' milk, when twenty-four hours old, is called "fresh koumiss;" it is filled in champagne bottles, corked and wired. In the bottles the alcoholic fermentation advances, and the koumiss, in the course of from twenty-four to forty-eight hours, enters into its second stage. So much of the sugar is then decomposed, and alcohol and carbonic acid formed, that the whole of the contents of the bottle may be drawn off by means of a champagne tap, presenting a highly effervescent liquid. After a fortnight has elapsed, little or no sugar is left undecomposed, and we have now "old koumiss."

In October and November three series of analyses of koumiss were made, each series comprising three samples of the same make, and one, eight, and twenty-two days old respectively when analysed. It was attempted to determine the nitrogenous compounds separately—viz., casein, as precipitated by the lactic acid formed in the koumiss; albumen, precipitated by boiling the filtrate from casein; and peptones, &c., precipitated with tannin. But as the separation was not in every case as exact as could be desired, the total amount of nitrogenous matter is given in the following table. Carbonic acid was not determined.

The analyses, Table IV., clearly show the progress of the alcoholic and lactic fermentation by the gradual increase of alcohol and lactic acid, and the decrease of sugar.

TABLE I.—MIXED MILK OF FIFTEEN MARES.

No.	Sample taken.	Sp. Gr.	Water.	Fat.	Proteids.	Sugar.	Ash.
1	10 Sept., 4 p.m.	1·0350	90·41	0·87	2·11	6·30	0·31
2	16 " 10 a.m.	1·0353	90·30	0·87	1·88	6·64	0·31
3	" " 12noon	1·0352	90·05	0·94	1·85	6·82	0·34
4	" " 2 p.m.	1·0360	90·09	1·13	1·89	6·59	0·30
5	" " 4 p.m.	1·0348	90·25	0·91	1·93	6·59	0·32
6	" " 6 p.m.	1·0351	89·83	1·19	2·03	6·62	0·33
7	22 " 10 a.m.	1·0350	90·08	1·09	1·79	6·75	0·29
8	" " 12noon	1·0349	89·74	1·44	1·89	6·64	0·29
9	" " 2 p.m.	1·0355	89·89	1·21	1·86	6·74	0·30
10	" " 4 p.m.	1·0343	90·22	1·14	1·71	6·63	0·30
11	" " 6 p.m.	1·0349	89·76	1·25	1·86	6·82	0·31
	Average .. ..	1·0349	90·06	1·09	1·89	6·65	0·31

TABLE II.—MILK OF INDIVIDUAL MARES.

No.	Sample taken.	Sp. Gr.	Water.	Fat.	Proteids.	Sugar.	Ash.
12	29 Sept., 10 a.m.	1·0345	90·06	1·12	1·72	6·80	0·30
13	" "	1·0358	90·15	0·78	1·76	6·99	0·32
14	" "	1·0356	90·46	0·83	1·65	6·70	0·36
15	" "	1·0353	90·04	1·00	1·83	6·80	0·33
16	" "	1·0352	90·07	0·95	1·62	7·10	0·26
17	1 Oct., "	1·0356	90·07	0·94	1·65	7·07	0·27
18	" "	1·0350	90·42	0·62	1·62	7·08	0·26
19	" "	1·0345	90·17	0·96	1·58	6·99	0·30
20	" "	1·0351	89·92	0·97	1·62	7·21	0·28
21	" "	1·0354	90·27	0·67	1·76	7·03	0·27
22	3 " "	1·0347	90·28	0·86	1·54	7·04	0·28
23	" "	1·0353	90·17	0·88	1·71	6·95	0·29
24	" "	1·0346	89·88	1·17	1·57	7·07	0·31
25	" "	1·0346	90·11	1·18	1·50	6·91	0·30
26	" "	1·0344	89·92	1·18	1·55	7·05	0·30
	Average .. ..	1·0350	90·13	0·94	1·65	6·98	0·30

TABLE III.—MILK OF MARES SPECIALLY FED.

No.	Sample taken.	Sp. Gr.	Water.	Fat.	Proteids.	Sugar.	Ash.
27	16 Sept., 11 a.m.	1.0355	89.55	1.23	2.07	6.86	0.29
28	22 „ 4 p.m.	1.0339	89.88	1.40	1.80	6.67	0.25
29	24 „ 12 noon	1.0339	89.18	1.28	2.20	7.10	0.24
30	„ „ „	1.0347	89.82	1.18	1.70	7.02	0.28
31	26 „ „	1.0361	88.66	1.67	2.12	7.23	0.32
32	„ „ „	1.0353	88.24	2.14	2.05	7.28	0.29
	Average .. ..	1.0349	89.22	1.48	1.99	7.03	0.28

TABLE IV.—ANALYSES OF KOUMISS.

Age days.	Water.	Alcohol.	Fat.	Nitrogenous Matter.	Lactic Acid.	Sugar.	Ash.
				1. Series.			
1	90.99	2.47	1.08	2.25	0.64	2.21	0.36
8	91.95	2.70	1.13	2.00	1.16	0.69	0.37
22	91.79	2.84	1.27	1.97	1.26	0.51	0.36
				2. Series.			
1	91.87	3.29	1.17	.99	0.96	0.39	0.33
8	92.38	3.26	1.14	1.76	1.03	0.09	0.34
22	92.42	3.29	1.20	1.87	1.00	—	0.35
				3. Series.			
1	91.42	2.25	1.22	1.75	0.70	2.30	0.36
8	92.04	2.84	1.10	1.89	1.06	0.73	0.34
22	91.99	2.81	1.44	1.69	1.54	0.19	0.34

## ON BEEF TEA.

BY OTTO HEHNER.

*(Read before the Meeting, November 11th, 1885.)*

I have lately had, on several occasions, to examine samples of Beef Tea, with a view of deciding whether they were made from meat alone, or unduly charged with gelatinous substances, and in order to be able to arrive at the desired result, I have made a number of analyses of well-known preparations, which, partial though they be, may be of some utility to other analysts. I do not pretend to say one word about the much debated question of the dietetic value of these preparations; the fact that they are largely consumed by invalids, and form an important branch of industry, will, I trust, be taken as sufficient justification for the publication of my figures.

I deviated from the ordinary course of examination in but one particular. For the estimation of the "matters precipitable by alcohol," I dissolved 2 grammes of the substance in 25 c.c. of water, and added 50 c.c. of strong methylated spirit, allowed the precipitate to settle overnight, poured and drained off the clear liquor next morning, and, without washing dissolved it in a little hot water, evaporated in a weighed basin, and dried at 100°. I held this way of proceeding to be more convenient than the extraction with 80% alcohol as usually practised with extracts of meat. In an experiment with Nelson's Gelatine, dried at 100°, I found that thus no less than 93.19% were precipitated, whilst the Liebig Company's Meat Extract gave but a faint precipitate, amounting to 5.1%, my aim being to precipitate as much as possible of the

gelatine, and as little as possible of meat extractives and mineral salts. The phosphoric acid was determined by the process described by me in the ANALYST some years ago, the phosphomolybdate precipitate being dissolved in ammonia, and the solution evaporated, dried at 100°, and the residue divided at 28·5.

	Leibig's Extract.			Nelson's Gelatine.	
Moisture (at 100°)	18·70				
Nitrogen	7·94				
Alcohol precipitate	5·16			93·19	
Ash	23·38			3·25	
Phosphoric acid	6·07			nonc.	
CONCENTRATED BEEF TEA.					
	English No. 1.	English No. 2.	English No. 3.	Russian.	X.
Moisture	36·96	31·00	41·93	24·56	54·31
Nitrogen	8·25	8·36	7·52	9·89	6·79
Alcohol precipitate	27·4	30·3	25·5	35·4	32·3
Ash	4·36	4·13	4·92	6·72	7·57
Phosphoric Acid	1·16	1·00	1·10	·95	2·11
ESSENCE OF BEEF.					
	English No. 1.	English No. 2.	English No. 3.		
Moisture	89·25	89·61	92·32		
Nitrogen	1·36	1·36	·79		
Alcohol Precipitate	3·07	3·74	1·99		
Ash	1·17	1·00	1·30		
Phosphoric acid	·34	·26	·38		

For the sake of comparison, I have calculated these analyses upon the *dry* materials.

The results were as follow :—

		BEEF TEAS.				
Liebig's Extract.		English No. 1.	English No. 2.	English No. 3.	Russian.	X.
Nitrogen	9·7 ..	13·1	12·1	12·8	13·1	14·8
Alcohol precipitate	6·3 ..	43·4	43·9	43·9	46·9	70·7
Ash	28·7 ..	6·9	5·9	8·47	8·9	16·5
Phosphoric acid	7·4 ..	1·8	1·4	1·89	1·26	4·6
Phosphoric acid in ash	25·9 ..	26·1	24·2	22·3	14·1	27·9
		English No. 1.	English No. 2.	English No. 3.		
Nitrogen		12·6	13·1	10·3		
Alcohol precipitate		28·6	36·0	25·9		
Ash		10·7	9·6	16·9		
Phosphoric acid		3·1	2·5	4·9		
Phosphoric acid in ash		29·0	26·0	29·6		

The ashes of all the preparations, excepting of the beef tea marked "Russian," were completely soluble in water, and were practically devoid of lime; whilst the latter samples in question yielded an ash but very partially soluble in water, and consisting to a great extent of calcium carbonate. The ash of Nelson's Gelatine also is insoluble, and mostly calcium carbonate.

In judging of the merits of the samples, as far as their origin—meat or bones—is concerned, I think the analyses show, that the ash should be high, with about one quarter of its weight of phosphoric acid, and that alcohol should not precipitate much more than 25% of the total dry matter of meat essence, nor more than 44% of that of beef tea.

CONCLUSION OF THE SOCIETY'S PROCEEDINGS.

## PROCEEDINGS OF THE AMERICAN SOCIETY OF PUBLIC ANALYSTS.

COMMUNICATED BY DR. E. WALLER, NEW YORK.

THE first meeting for the session 1885-6 of the American Society of Public Analysts was held on October 5th, 1885.

Dr. Edson spoke of the use of gelatine in preserved fruits; some grades of gelatine used contained hydrochloric acid, which was especially objectionable where the preserves were so packed as to come in contact with metal. He also spoke of the use of gelatine in oleo-margarine to give it firmness, and, it was claimed by some, to aid in preserving the article. The method of detecting this addition is receiving the attention of some of the members of the Society. Dr. Grothe suggested that a determination of the nitrogen might be of service.

Dr. Bartley spoke of a case of unmistakable copper poisoning which occurred in Brooklyn last spring from eating pickles coloured with copper. The victim was a child, who was suddenly seized with severe dysentery and inflammation of the colon. A portion of the pickle eaten by the child was found to contain copper. Alum, it was found, is frequently used in pickles to give them crispness.

Dr. Bartley also said that the most interesting part of the work of the Brooklyn Health Department, of Brooklyn, during the past summer, had been the investigation of beer brewing, and of the presence of antimony in fabrics, the metal having apparently been introduced by the use of tartar emetic as a mordant.

A paper was then read by Dr. Otto Grothe on "Beer brewing as practised in New York and Brooklyn." Dr. Grothe had visited different breweries in both cities, and had analysed different beers. As a result of his visits, he stated that bicarbonate of sodium was in extensive use by the brewers for neutralizing the acid, this substance being added in quantities varying from two to nine ounces to the barrel of 32 gallons. Grape-sugar, glucose and corn-meal were extensively used as substitutes for malt, while hop substitutes could not be detected. Other substances found in breweries were juniper berries, isinglass, Iceland moss, cream of tartar, tartaric acid, salicylic acid, bisulphite of calcium and glycerine. Licorice also is often sold to breweries, but it is not in general use. The beer is generally manufactured in much shorter time than in European breweries, and this is made possible by artificial clearing, done by adding a solution of isinglass and cream of tartar in water in the storing, *i.e.*, pressure barrel.

Large breweries effect the cooling of their cellars by ammonia ice machines, and they have done away with the cooling pan altogether. Smaller concerns work with natural ice, which is considered as disadvantageous in every respect. While some breweries are kept scrupulously clean, others are sometimes very dirty, mucor and fungi growing on the walls and tubs.

The beer often contains bacteria and bacilli, and the lactic ferment is very frequently found in the product from breweries where ice is used. *Saccharomyces apiculatus* often occurs to the detriment of the stock. The only means for preventing loss are constant watchfulness and frequent examinations with the microscope. An entire change of the yeast may be necessary in some cases to avoid some hurtful

ferment, which will make the beer "sick." As the salary of an expert master brewer is high, some dispense with such a superintendent, and, as occasion demands, call in a "beer doctor."

In beer, as at present manufactured, Dr. Grothe believes that danger to health may exist in the use of large quantities of sodium bicarbonate, and in the excessive quantities of substances for cleaning the product, and he recommended that some legal limit should be put on the amount to be used.

Dr. Bartley expressed the opinion that excess of bicarbonate of soda, or the presence of such ferments as would cause the beer to be technically called "sick," were the points calling for the special attention of officers of health.

### THE RELATION OF COCOANUT OIL TO THE VARIOUS METHODS OF BUTTER ANALYSIS.

BY RUSSELL W. MOORE, A.B., M.S.

(*Read before the American Chemical Society, Sept. 18th, 1885.*)

THE analytical chemistry of the fats is an undeveloped department of science. The various fats have been discovered, their formulæ determined, and their true nature made known; but analytical processes for separating the various fats and fatty acids from each other with anything like certainty or exactitude, are things for which the student of fatty chemistry is still seeking.

Certain special problems have, however, arisen, which have called for an immediate solution, and the stimulus thus given has resulted in a concentration of thought and investigation upon a single question, to which the discovery of many valuable facts is due. A prominent example in this connection can be seen in the case of butter analysis. Various processes were devised, all with the object of distinguishing true butter fat from other fats. The problem was such a difficult one that at one time it was seriously doubted by eminent chemists whether a satisfactory solution could be obtained, since the number of animal and vegetable fats was so great, their character so similar, and their chemistry so little understood.\*

It is the purpose of this paper to review briefly the various processes which have been used for butter analysis, and at the same time to bring out, more fully and completely than has hitherto been done, the remarkable chemical and physical relations that coconut oil bears to true butter fat.

The melting point of the fat was at one time considered a reliable test, as butter fat melts at an average temperature of  $35.8^{\circ}\text{C}$ , while the ordinary substitutes melt at considerably higher temperatures, viz., ox fat,  $48^{\circ}$ – $53^{\circ}\text{C}$ .; mutton fat,  $50^{\circ}$ – $51.6^{\circ}\text{C}$ .†

It is, however, easy to find palatable oils and fats which, when mixed with that animal product which, for want of a better name is termed oleomargarine, would bring the fusing point of the mixture within the required limits for butter. There is an additional source of uncertainty in the use of this process, due to the fact that butter

\* Hassall, *Food*. London, 1876, p. 435.

† Blyth, *Foods, etc.*, p. 294.

fat becomes harder by age, and the melting point rises considerably in consequence.

In this case coconut oil could be mixed with oleomargarine, or any other substitute for butter, with a higher melting point, since it fuses at a much lower temperature than even pure butter fat, viz :

23·5–24·1° (Muter, ANALYST, I., 7.)

24·2–24·3° (Original Observation.)

The specific gravity of the fat, has also been used as a test, since butter fat has a higher density, viz. : ·91246–·91382 at 37·7°, than what Blyth terms vegetable butterine ·90294, or dripping ·90659. But coconut oil at the same temperature shows a higher specific gravity than pure butter fat, viz. : ·9167,<sup>2</sup> ·9117.<sup>3</sup> Allen gives the specific gravity of coconut oil at the temperature of 100°C, as ·868, and of butter, ·865–·868.<sup>4</sup> Thus mixtures of foreign fats could easily be made, of which the specific gravity would be the same as that of butter.

The process of Hehner<sup>5</sup> was the first one proposed that rested on a purely chemical principle. By this method, as proposed by the author in the original article, the insoluble fatty acids are directly weighed, after washing out the soluble fatty acids with boiling water. The limit fixed by Hehner is between 86·5°/o–87·5°/o of insoluble fatty acids, while ordinary butter substitutes show much higher figures, in most cases as high as 95°/o. The investigations of other chemists<sup>6</sup> have, however, shown that butters are by no means uncommon in which the percentage of insoluble fatty acids will be higher than the limit fixed by Hehner, reaching in some cases as high as 90°/o.

Coconut oil, when examined in strict accordance with Hehner's original directions, yielded 86·43°/o of insoluble fatty acids,<sup>7</sup> which would thus enable it to be mixed with other fats in such a manner as to escape detection by this process. It is true, however, that the subsequent modification of Hehner's process by Dupré,<sup>8</sup> by which the soluble acids are estimated in the washings from the insoluble acids, would probably detect such a mixture by the low percentage of soluble fatty acids found. The low figures by the Hehner process, in the case of coconut oil, are in a measure due to the fact that lauric acid, its principal constituent, is volatilized to some extent at the temperature necessary to dry the insoluble fatty acids, and loss is thus occasioned. Nevertheless, coconut oil is a more suitable fat for adulteration with a view to escape this method than any other fat hitherto examined.

Another method brought out by Koettstorfer<sup>9</sup> depends upon the fact that the comparatively high percentage of glycerides of the lower fatty acids in butter causes it to

<sup>1</sup> Muter, ANALYST, I., 7.

<sup>2</sup> Stillwell, *American Chemist* I., 407 (with correction for temperature as directed).

<sup>3</sup> R. W. Moore, *American Chem. Journal*, VI., No. 6.

<sup>4</sup> Allen, *Commercial Organic Analysis*, II., 136.

<sup>5</sup> Fresenius, *Zeit. für anal. Chem.* XVI., 145.

<sup>6</sup> Fleischman & Vieth, *Fres. Zeit.* 1878, p. 287. Kretschmar, *Ber. Chem. Gesell.*, X., 2091 Kuleschhoff, *Wagner's Jahresbericht*, 1878, p. 999. Jehn, *Archiv. d. Pharm.*, IX., 1878, p. 335. De la Source, *Ibid.*, 1882, p. 929.

<sup>7</sup> R. W. Moore, *Chem. News*, Dec. 5, 1884.

<sup>8</sup> ANALYST, I., 87, 114.

<sup>9</sup> *Fres. Zeit.*, XIII., 199, 431.

require a greater amount of caustic potash for saponification than other fats. Experiment justified this view in a measure for oleomargarine, and beef and mutton tallow required about 195m.g. caustic potash per gm. for saponification, while for pure butter fat 223·5–232·5 m.g. were necessary.

Cocoonut oil in this case also refuses to be classed with the ordinary substitutes for butter, and exhibits figures higher even than pure butter, viz. :

<sup>1</sup> 257·3–258·3

<sup>2</sup> 250·3–246·2 <sup>2</sup>

These high figures are due to the presence of large amounts of lauric acid together with smaller quantities of caproic, capryllic and capric acids.

It is thus possible to mix oleomargarine with cocoonut oil in such a manner as to bring the results within the limits set by Koettstorfer. In proof of this the following mixtures were made with the object of approaching nearly the limits of Koettstorfer: <sup>4</sup>

Cocoonut Oil.	Oleomargarine.	Mgs. K O H per gm.
49·3°/°	50·7°/°	220·0
70·2°/°	29·8°/°	234·9
Washed Oil.		
53·1°/°	46·9	223·6
75·9°/°	24·1	234·9

The oleomargarine used required 193·5 mgs. KHO per gm.

A general method for testing oils and fats has been brought out by Hübl<sup>5</sup> and has been recommended by the author as applicable to the examination of butter for foreign fats. It depends upon the relative capacity for absorbing iodine of the formic, oleic and tetrolic series of acids; the first remaining; under ordinary circumstances, indifferent while each molecule of the oleic series unites with two atoms of iodine, and each molecule of the tetrolic series with four atoms. Thus widely varying figures for various fats and oils are obtained, depending upon the relative amounts of acids of the different series contained in each. Thus, 100 grms. Japanese wax absorb but 4·5 gm. of iodine, while the same quantity of linseed oil, containing 80°/° linoleic acid, absorbs as high as 160 grms. of iodine.

In this list of iodine figures, butter occupies a place midway between cocoonut oil (8·9 gm.) and the ordinary substitutes. Thus mixtures have been made of oleomargarine and cocoonut oil so as to come within the limits of butter, as the following figures will show.<sup>6</sup>

Oleomargarine, 55°/° }  
Cocoonut oil, 45°/° } Iodine figure, 35·5

Lard, 40°/° }  
Cocoonut oil, 60°/° } Iodine figure, 32·2

<sup>1</sup> Valenta, *Dingler's Polyt. Journal*, 249, 270.

<sup>2</sup> R. W. Moore, *Chemical News*, Dec. 5th, 1884.

<sup>3</sup> The oil was thoroughly washed with hot water.

<sup>4</sup> R. W. Moore, *Chemical News*, loc. cit.

<sup>5</sup> *Dingler's Pol. Journal*, 253, 281.

<sup>6</sup> R. W. Moore. *American Chem. Journ.*, VI., No. 6.

In eight samples Hübl found for butter a maximum figure of 35.1, and a minimum of 26.8.

There is, however, a process, the results of which show butter to be an extreme, and cocoanut oil, though considerably in advance of the figures given by the ordinary substitutes for butter, still falls far below the limits set for genuine butter. This method, which was brought out by Reichert,\* consists in distilling off from the sample under examination a definite amount of acid, and estimating the same volumetrically. It is by this method that the fact that butter is comparatively rich in butyric acids, which is considerably more volatile than any acid contained in cocoanut oil, is brought into prominence, for the distillation, as recommended by the author, is stopped at a point when the less volatile acids come over in any quantity.

If the distillation were continued long, and the distillate were not freed by filtration from the acid which condenses in a solid form at the temperature of the condenser, cocoanut oil would give figures very much higher than those of butter, since it is almost entirely composed of acids which can be distilled over with water.† In the relative volatility of the more volatile portion of the acids contained, however, butter stands far ahead of cocoanut oil, the prescribed distillate neutralizing, at least, 13. c.c. of  $\frac{1}{10}$  normal KHO‡ while cocoanut oil, treated in the same manner, requires but 3.7 cb. cm.§ Thus mixtures of the latter with butter would lower the figures considerably :

		Hehner.	Koettstorfer.	Hübl.	Reichert.
Butter,	50 $\frac{\circ}{\circ}$ .	89.50	227.5	35.4	8.7
Oleomargarine	27.5 $\frac{\circ}{\circ}$ .				
Cocoanut oil,	33.5 $\frac{\circ}{\circ}$ .				

Thus it can be seen that cocoanut oil in four processes for testing butter is characterized by properties which render it, for the chemist, a most dangerous adulterant, making possible a large number of mixtures difficult to detect by chemical methods.

The question naturally arises whether cocoanut oil has actually come into use for this purpose of adulteration, and also whether the mixtures contrived to baffle ordinary chemical tests really resemble butter. The first of these questions is most difficult to answer, since the process which is capable of detecting such mixtures has not, by any means, come into general use, and the results obtained by other processes do not bear upon this question.

There is not, however, a complete absence of evidence on this point, since cocoanut oil is mentioned as an adulterant of lard in the ANALYST (VII., 193), and Dietsch || makes note of it as a component of what he terms "Schmalz Butter." The writer also has been informed by an importer of the oil that it has, to his knowledge, been used, with little success however, for the purpose of adulterating both butter and oleomargarine. The mixtures thus produced were, probably, unpalatable, owing to the fact that the odor of the oil had not been removed.

\* *Fres. Zeit.* XVIII., 68.

† Oudemans's *Journ. für prakt. Chem.*, 81, 367.

‡ { Reichert, *loc. cit.*

{ Medicus & Scherer, *Fres. Zeit.* XIX, 159.

§ { Reichert, *loc. cit.*

{ R. W. Moore, *Chem. News*, *loc. cit.*

|| *Nahrungsmittel und Getränke*, 4th Ed., p. 212.



It must, however, be taken into consideration that the oil is produced in a warm climate, where decomposition begins easily; that no means are taken to preserve its freshness, and that when met with in colder countries it is already tolerably old, since the transportation occupies a long time. When first made it is, probably, more agreeable to the taste, for the natives of the countries where it is produced use it for the same purposes as we use butter, and if a demand for a palatable article should arise, it would, probably, be supplied.

The smell and taste of the oil, though disagreeable and unpalatable, can both be removed to a great extent. Careful washing with hot water will accomplish a great deal, and in this way the writer has succeeded in obtaining a tolerably tasteless article. Also, a German patent has been taken out by Jeserich and Meinert\* for rendering vegetable oils, including palm and cocoanut oils, inodorous and edible, so that they can be used in place of butter or in combination with it. The process consists in treating the oil with superheated steam and removing any free fatty acid by saponification with a small amount of calcined magnesia, not exceeding 25%. The patentees claim that in this way a perfectly sweet fat is obtained.

Notwithstanding that cocoanut oil, might be, and possibly is, a most dangerous adulterant for butter, there yet remains a process by which it can be infallibly detected, if present, in any amount—the process of Reichert. This method is also the surest and most reliable for testing butter for any other foreign fats. It is easy and elegant in use, and requires but one standard solution, and that a permanent one, not liable to daily change like the alcoholic potash solution used in the Koettstorfer process. Only one weighing is required of but ordinary exactitude and the transition point of the final titration is as sharp as could be wished. In fact it is a method possessing many merits and few defects, and it is extremely desirable that it should come into general and extended use. Thus far, it has stood all tests in the hands of many chemists. If, then, cocoanut oil should be the means of casting discredit upon all other methods, and thus of bringing that of Reichert into general use, a great and valuable service would be rendered to the subject of butter analysis.

---

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO FOOD.

ON THE PRESENCE OF ALKALOIDS IN OLD FLOUR. BY BALLAND. *J. de Pharm. et de Chim.* [3] 12,341.—If flour, which has been kept for some time in sacks, be exhausted with ether, and the extract evaporated to dryness, some fatty matter is obtained, which is acid, and has a peculiar disagreeable smell and a sharp taste. If this substance be then treated with a small quantity of hot water, and kept in a water-bath for a few minutes, on cooling and decanting the alkaloids can be readily detected on a glass plate with the ordinary reagents (double iodide of mercury and of potassium, ferricyanide of potassium, and chloride of iron).

The reactions are visible in flours which have only been ground for a year to eighteen months; they are very marked in flour which is two or three years old. If the ex-

---

\* *Wagner's Jahresbericht*, 1882, 932.

tracts obtained from these latter be mixed with flour and water and administered to sparrows, death is caused in a few hours, with all the symptoms of poisoning; similar experiments with fresh flour gave negative results.

The author considers that the accidents which have occasionally occurred during wars, and attributed to the use of bad bread, are thus accounted for.

The formation of the alkaloid is due to the transformation of the gluten under the influence of the natural ferment in the flour. E. E. B.

EXAMINATION OF FOOD STUFFS. BY PROF. A. EMMERLING (Chem. Zeit. 9. 1465).—It often happens that in two samples of the same chemical composition, the one poisons the cattle whilst the other is a good food stuff. If chemical examination for metallic and non-metallic poisons and alkaloids gives negative results, the sample must be tested for mould and spores. A full description, with diagram, was given of the author's process for their detection, in the early part of the year.\* As bacilli are very common, it is not advisable to condemn the use of food, even if it contains many of them, except for young animals. From numerous experiments it has been found by the author that cake from *earthenut* (arachis) oil, often contains mould, rarely bacilli; from *cotton seed*, more often bacteria than mould; it also often has an alkaline reaction from fermentation: *Rice meal*, mould common; commonest bacteria occurring in it are micrococci. *Cocoanut oil cake* keeps better, although besides *bacillus subtilis*, it occasionally contains an unrecognised fungus. *Palm cake* very rarely contains moulds or fungi; hence its popularity as a food stuff. E. E. B.

#### MONTHLY RECORD OF ANALYTICAL RESEARCHES INTO DRUGS.

TEST FOR THE NEW ANTISEPTIC, IODOL.—By Dr. Vulpius. Chem. Zeit. 9, 1446. This antiseptic is in use as a substitute for iodoform, as it has no unpleasant smell, and does not produce the same symptoms of intoxication when wounds are dressed with it. The iodol is a micro-crystalline brownish powder, which can be heated to 100° without decomposition; at higher temperatures it gives off iodine vapour, and finally yields a voluminous carbonaceous mass. It is scarcely soluble in water, readily in alcohol, but, on adding water to the alcoholic solution, most of the iodol is re-precipitated. It can, however, be diluted with glycerine. Iodol is also readily soluble in ether and chloroform. It can be recognised by the green colour of a solution of it in sulphuric acid, and by the bright red colour produced when an alcoholic solution is warmed with nitric acid. E. E. B.

DETECTION OF SALICYLIC ACID BY SYNTHESIS OF GAULTHERIA OIL.—By Prof. Curtmann. Pharm. Rundsch. 1885. 3. 155. Most of the present methods for the detection of salicylic acid are based upon the formation of phenol derivatives, and it consequently cannot be readily distinguished from phenol or its compounds. Very few tests (*e.g.*, the green colour with cupric sulphate) detect the acid as such. Salicylic acid can be found with great ease by converting it into methyl salicylate. About 1 c.c. of methyl alcohol is added to the sample, and then about 0.5 c.c. of sul-

\* Chem. Zeit. (9). 263.

phuric acid (concentrated). After boiling for a short time, it is allowed to stand for a few minutes, and then again heated. The smell of Gaultheria oil is now distinctly recognisable, even if as little as 1 mgrm. of salicylate of soda was present in the solution.

E. E. B.

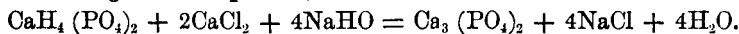
EXAMINATION OF GUM ARABIC FOR ARTIFICIALLY PREPARED GUMS.—By H. Hager. Pharm. Centralhalle 6. 388. A mixture of ferric chloride and potassium ferricyanide in solution is a certain and delicate reagent for artificial gum made from dextrin. The reagent is prepared by mixing 15 drops of the pharmaceutical solution of ferric oxide with 15 drops of the cold and saturated solution of potassic ferricyanide, and 5 drops of dilute hydrochloric acid (spec. gravity 1.165) with 60 c.c. of water. If 6 c.c. of a 20 % solution of the sample of gum be treated with 3 c.c. of this reagent, pure gum yields a clear *yellow* viscous liquid, which remains unaltered from eight to ten hours. If, however, dextrin be present, the yellow colour changes either at once or in the course of an hour, and in two to three hours the mixture has become *blue*.

E. E. B.

#### MONTHLY RECORD OF GENERAL RESEARCHES IN ANALYTICAL CHEMISTRY.

NEW METHOD FOR THE VOLUMETRIC ESTIMATION OF THE SOLUBLE PHOSPHORIC ACID IN SUPERPHOSPHATES.—By Prof. A. Emmerling. Chem. Zeit. 9, 1465. The process is due to the precipitation of monocalcium phosphate as tricalcium phosphate by sodium hydrate in the presence of an excess of calcium chloride. It is materially different from the process described by Mollenda,\* in which the monocalcium phosphate is determined alkalimetrically by sodium hydrate, using phenolphthalein as indicator, after separation of the lime by sodium oxalate. The same indicator is used by this author, who found it advisable to titrate a known volume of the standard sodium hydrate (5 to 20 c.c.) with the mixture of superphosphate (200 c.c.) and calcium chloride solution (50 c.c.), instead of *vice versa*. The latter solution is prepared by dissolving 200 grammes of pure fused calcium chloride in 1,000 of water, and then accurately neutralising the solution. The titration is continued until the sodium hydrate, containing a little phenolphthalein, is completely decolourised. The free acid in the mixture is then determined by titration with sodium hydrate, using methyl orange as an indicator.

The difference gives the amount of soda required to precipitate the tricalcium phosphate according to the equation,



Thus four molecules of sodium hydrate are required to effect the precipitation of one molecule of phosphoric acid ( $\text{P}_2\text{O}_5$ ). The process yields results which differ from those obtained by the uranium process by less than 0.1 per cent. It is also available for phosphates containing iron, except for the double superphosphates ("doppel-superphosphaten"). The mean of seven analyses differed from the results obtained by the molybdate method by + 0.14 per cent.

E. E. B.

\* Chem. Zeit. [1883] 7, 491.

SEPARATION OF THE HALOGENS.—By Causse. *J. de Pharm. et de Chim.* [3] 12,391. Copper turnings are put in a flask containing a solution of sulphurous acid, saturated at a low temperature; the mixture is then left for several weeks, when the solution becomes blue, although the whole of the acid does not necessarily combine with the copper. If some of this reagent be then added, drop by drop, to a mixture of the compounds of sodium or potassium, with chlorine, bromine, and iodine, a white precipitate of cuprous iodide is formed, which can be very easily filtered. When the liquid ceases to yield a precipitate in the cold, it is approximately neutralised, more of the reagent added, and the solution boiled, when cuprous bromide separates and can be estimated. Finally the filtrate, which is now free from iodine and bromine, is tested for chlorine. E. E. B.

DETERMINATION OF ROSIN OIL IN MINERAL OILS.—By H. Demski and T. Morawski. *Dingler's Polytech. Journ.* 258, 82—87. Numerous attempts were made to apply quantitatively the proposed tests for rosin oils (in mineral oils) based on their strong dextrorotatory power, greater solubility in glacial acetic acid and greater affinity for iodine. The authors consider that the first of these methods is much the best as a qualitative test, and that the last is quite untrustworthy. The proposed method of analysis is based on the difference of the solubility of mineral and rosin oils in acetone, rosin oils mixing with it in all proportions, whereas the mineral oils only dissolve in several parts by volume. The solubility in acetone of mixtures containing a definite proportion of rosin oil is, however, dependent on the source of the mineral oil; hence tables of solubility of known mixtures must be made for comparison. The analysis is made in the following way:—50 c.c. of the sample of oil are shaken up several times with 25 c.c. of acetone in a 100 c.c. graduated cylinder, and then left at rest for some time. If the liquid separates into two layers, 10 c.c. of the upper are drawn off and the amount of oil in it determined after removal of the acetone by evaporation. The density of the oily residue must also be determined by adding water to a few drops of it, and then alcohol until the oil just begins to sink. Finally it is necessary to determine the amount of rosin oil which must be added to the sample of oil to enable it to dissolve in half its volume of acetone, which is easily done, for as soon as perfect solution has taken place, the liquid gives a fairly permanent froth when shaken, which will not form as long as any undissolved oil is present. The results of numerous experiments with mixtures of rosin oils (spec. grav. .992 or .990) with several kinds of mineral oil from America, etc., are given, and from them the authors conclude:—(1). That with American and Galician oils an admixture of not less than 35 per cent. of rosin oil is at once detected by its ready solubility in acetone; and with Caucasian and Wallachian oils—with one exception—if 50 per cent. be present. (2). That if the absence of rosin oils has been proved by the polarimeter, the solubility of the oil in acetone indicates its source, Caucasian and Wallachian oils being only about half as soluble as American and Galician oils. (3). That the density of the oily matter in the acetone layer, when either a pure mineral oil or a mixture is taken, is greater than that of the sample. The authors show in detail how, by their method of

analysis and the aid of a polarimeter, the amount of rosin oil in a particular sample of oil is determined, and also the source of the mineral oil in it, but the working cannot be briefly explained without the accompanying table.

E. E. B.

**A NEW METHOD OF SEPARATION OF IRON AND ALUMINA.**—By M. Ilinsky and G. v. Knorre (Ber. d. Deutsch. Chem. Ges., 1885, No. 14, 27282—734). The separation is effected by means of nitroso  $\beta$  naphthol, which was previously employed by these authors for the separation of nickel and cobalt\*. The solution containing the iron and alumina, as sulphates or chlorides, is evaporated to a small bulk, and treated with enough ammonia to produce a slight precipitate, which is redissolved in a few drops of hydrochloric acid. To the cold liquid an equal volume of 50 % acetic acid is added, and then an excess of commercial nitroso  $\beta$  naphthol, dissolved in 50 % acetic acid, is stirred in. After standing from six to eight hours, the ferri nitroso naphthol is filtered off, washed at first with cold 50 % acetic acid, and then with cold water, until a drop of the filtrate evaporated on platinum foil gives no residue. If the solution contain too much water, or a large excess of nitroso naphthol was used, the iron salt is mixed with so much of the latter that the washings are yellow up to the end, but this does not influence the accuracy of the results. After drying the washed precipitate, it is transferred with the filter to a tarred porcelain crucible. A volume equal to the precipitate of pure crystallised oxalic acid is added, the filter folded up, and the whole ignited, carefully raising the temperature very slowly. It is best to place the loosely covered crucible on a piece of asbestos cardboard, and to heat this with a small flame. When no more fumes are evolved, the temperature is raised, and the crucible finally strongly ignited with access of air till all the carbon has been burnt off. In this way the precipitate can be incinerated without the loss from deflagration which would take place if the oxalic acid were not added. To determine the alumina in the filtrate, it is evaporated in a porcelain basin. (if glass vessels are used, the amount of alumina found is too high) to drive off the acetic acid. The solution is then diluted; ammonia added till slightly alkaline, and then gently warmed till the excess of ammonia is expelled. The hydrate of alumina, which is coloured brown by organic matter, is filtered off, washed carefully with hot water, dried, strongly ignited in the blow-pipe flame, and weighed; it should now consist of a snow-white powder. It is not essential that the iron be present as a ferric salt, but as the ferro nitroso naphthol is difficult to wash, and the amount of iron thus found liable to be too high, it is advisable to previously convert the ferrous into a ferric salt. Special attention is called to the following points:—

- (1) Not much free mineral acid must be present.
- (2) There must be plenty of acetic acid, which,
- (3) must not contain more than 50 % of acetic acid.
- (4) The washing must take place in the cold.
- (5) The incineration must be performed very cautiously at the beginning.
- (6) The process is not available when phosphoric acid is present.

Nitroso  $\beta$  naphthol is a very delicate test for ferrous oxide. A natural water, containing 3 mgms. of iron as ferrous carbonate per litre, gave with it a beautiful green colour.

E. E. B.

\* See same Berichte, Vol. 18, p. 699.

## CORRESPONDENCE.

[The Editor is not in any way responsible for opinions expressed by his Correspondents.]

To the Editor of THE ANALYST.

DEAR SIR,—I enclose a sketch of an improved Liebig condenser, which I have used with good success for some time past.

This condenser is much more compact, and is equally as effective as the ordinary form. You will observe much valuable space is saved, which the chemist may use to better advantage.

A is a tube about  $2\frac{1}{2}$  inches in diameter, and 20 to 24 inches long. B is a tube  $1\frac{1}{2}$  inches in diameter, closed at the upper end.

This tube is fitted to the large tube by a thick heavy cork soaked in melted paraffin. The tube E, which reaches nearly to the bottom of the condenser, serves as an inlet for cold water, and F the outlet for the heated water. The tube C, connected with the flask G, carries the hot vapours to the condenser, where they are condensed and delivered by the tube D to any suitable receiver. The tube C, which is connected with a cork to the condenser, should pass up two or three inches beyond the cork to prevent the condensed vapours from passing back into the retort flask.

If properly constructed this condenser is very effective. Very little trouble will be experienced by the vapours condensing in C, and running back; so little surface is exposed to the cold atmosphere and cork connection. The vapours condensing in B run down the walls of the tube, and are completely delivered by the smaller tube D.

This condenser is admirably adapted for the distillation of nearly all liquids, of low boiling points, which do not form explosive vapours, to come in contact with the flame under the flask G. However highly volatile liquids like the ethers may be safely distilled by screening the receiver from the heat of the flame, and by connecting with the receiver a safety tube delivering the vapours escaping out of a window, or through a partition into an adjoining room.

I usually employ as a safety connection, in the distillation of highly volatile and combustible liquids, a tube connected with the receiver, the extreme end of which dips under mercury, covered with a layer, an inch deep, of oil of a suitable character. This arrangement I have found perfectly safe; any escaping vapours are absorbed by the oil.

Very respectfully,

CHAS. B. GIBSON.

Chicago, October 19th, 1885.

To the Editor of THE ANALYST.

DEAR SIR,—The enclosed sketch for a "safety valve" for an extraction apparatus I have used very successfully, when it was desirable to run the extraction for some hours, at the same time the attention being devoted to other work.

The tube A connected with the Liebig as shown, and with the flask B, which is loaded with mercury, and immersed in a vessel of cold water. Another tube passes from B into C, which is partly filled with mercury and oil.

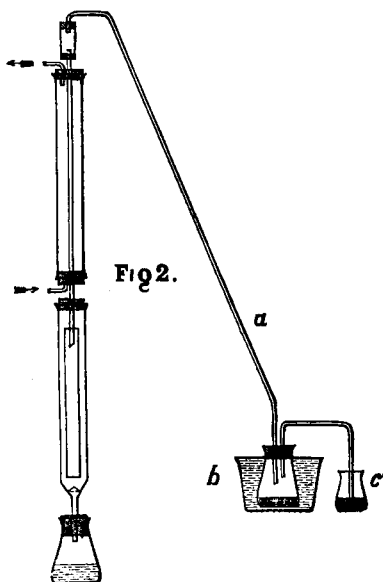
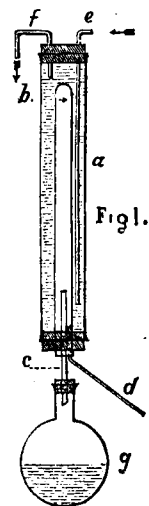
This apparatus is perfectly safe, as any vapours of the ether or benzene, which may not become condensed in the Liebig and flask B, will surely be absorbed by the oil in C.

I have run this apparatus continuously for 24 hours, and have scarcely been able to detect even the smell of ether at C. I have no fear of accidents, even with a high heat from a Bunsen burner when these precautions are taken.

Very respectfully,

CHAS. B. GIBSON.

Chicago, October 19th, 1885.



## LAW REPORTS.

QUEEN'S BENCH DIVISION.—BEFORE MR. JUSTICE MANISTY AND MR. JUSTICE A. L. SMITH.—*KIRK v. COATES*.—This was a case stated for the opinion of this Court by two justices for the borough of Huddersfield, under 20 and 21 Vict., c. 43.—Mr. Moorsom, Q.C., for the appellant, said that the following were the material facts set forth in the special case. On May 27 last, an information was preferred by the appellant, the sanitary inspector for the borough of Huddersfield, against the respondent, a farmer living in the neighbourhood of that town, under section 6 of the Food and Drugs Act, 1875 (38 and 39 Vict., c. 63), charging him with "having unlawfully sold, to the prejudice of the purchaser" (the appellant), a certain quantity of milk which had not been "of the nature, substance, and quality demanded" by him (purchaser). The appellant saw the respondent carrying a small milk-can, which, as he was informed by the latter, contained pure milk. The respondent, Coates, had a cart with him, in which there were one small and three large milk-cans. Two of the large cans were at the back of the cart, and the respondent informed the appellant that the can on the near side contained new milk, while that on the off side contained old milk. The appellant next inquired of the respondent what was contained in the two cans (one large and one small) at the front of the cart, and the latter replied, "New Milk." The appellant then went to his assistant, who was a short distance off, to obtain bottles, for the purpose of taking a sample of milk, but had kept the cart in sight until his return. He then placed his hand upon the large can at the front of the cart (stated by the respondent to contain new milk), and said, "Let me have a pint of milk out of this can, which you have just told me is new." The respondent hesitated, and then said, "That is old milk." The appellant answered, "How is it that you first told me it was new?" and insisted upon being supplied with a pint, for which he paid 1d., the ordinary and proper price in the district for old milk. The several requirements of the Food and Drugs Act, 1875, were then duly complied with by the appellant, and a sample of the milk submitted by him to the borough analyst. From the certificate of the latter, it appeared that the sample had been impoverished by the removal of at least 52 per cent. of the butter fat. The expression old milk was understood in the district to mean milk which had stood for 12 hours, and from which the cream had been removed. It was contended before the justices, on behalf of the appellants that when the respondent had once stated that the milk was new, he could not, on finding out that it was required for purposes of analysis, evade the statute by then disclosing its real character, even though he might have done this before the sale to the appellant had actually taken place. The question for the Court was, whether the respondent was liable to be convicted under the circumstances of having sold milk to the prejudice of the purchaser, which had not been of the nature, substance, and quality demanded. The learned counsel for the appellant said he should contend that the justices ought to have convicted the respondent, on the ground that he had made a representation that the milk offered by him for sale to the appellant had been new milk, whereas it had, in fact, been old milk, and that old milk was not a sufficient description of a class of milk. The question really was, whether a man could evade the statute.—Mr. Justice Manisty said that he thought the question was, whether the respondent had evaded the statute.—Mr. Justice A. L. Smith said that possibly the respondent would have cheated if he had been able to do so, but he had not done so.—Mr. Moorsom said that he should contend that the sale of the milk had been effected when the respondent had said that the milk was new milk. The point was a new one, and one which, in the opinion of the Corporation, was of importance, as if the practice which the respondent had followed was not illegal, there would be very great difficulty in enforcing the statute.—The respondent did not appear.—Mr. Justice Manisty, in giving judgment, said that the justices had rightly decided that there had been no sale of the milk by the respondent except as old milk. On the second question, as to its having been of the quality demanded, the appellant was, by the case stated, out of court.—Mr. Justice A. L. Smith concurred.—Appeal dismissed accordingly. —*Times*.

## BOOKS, &amp;c., RECEIVED.

THE Laws of Massachusetts in Relation to the Sale and Inspection of Butter, Oleomargarine, Cheese &c ; The Laws of Massachusetts in Relation to the Sale and Inspection of Milk ; The Laws of Massachusetts in Relation to the Sale and Inspection of Vinegar ; A New Method for the Determination of Phosphorus in Iron and Steel by J. B. Macintosh ; A Precise Investigation of some Micro-Organisms and Soluble Ferments by C. T. Kingzett ; Project D'Organisation d'un Service de Surveillance des Denrées Alimentaires et Boissons par Jules Wauters ; American Chemical Journal ; American Chemical Review ; American Druggist ; American Grocer ; British and Colonial Druggist ; Brewer's Guardian ; Canadian Pharmaceutical Journal ; Chemist and Druggist ; Country Brewer's Guardian ; Cow-keeper and Dairyman's Journal ; Independent Journal ; Invention ; Journal of the Society of Chemical Industry ; Journal of Microscopy and Natural Science ; Medical Press ; Medical Record ; The Miller ; Monthly Magazine of Pharmacy and Chemistry ; Pharmaceutical Journal ; Pharmaceutical Record ; The Polyclinic ; Popular Science News ; The Sanitarian ; San Francisco News Letter ; Science ; Scientific American ; Scientific Californian ; Society of Arts Journal ; Hospital Gazette.

## NOTICES TO CORRESPONDENTS.

All communications to be addressed to 25, Kennington Road, London, S.E.

A. P. S. (Rugby). Crowded out—next month.