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SEPARATION AND ESTIMATION OF TOCOPHEROLS IN VEGETABLE OILS BY THIN-LAYER CHROMATOGRAPHY

By M. K. GOVIND RAO, S. VENKOB RAO and K. T. ACHAYA

A method for separation of α-, β-, γ- and δ-tocopherols by thin-layer chromatography, and their subsequent colorimetric estimation, is described. Recovery is 97–98%, and no pretreatment of the unsaponifiable matter is necessary. The contents of individual tocopherols in a number of oils are recorded.

Introduction

Various chromatographic procedures for the separation and estimation of the four major tocopherols have been developed. Hickman,[1] Kofler[2] and Quaife[3] used column chromatography to resolve the tocopherols, the difficult β- and γ-isomers being separated as their nitroso derivatives. The procedure is tedious and the separations not too satisfactory. Potentiometric[4, 5] and polarographic[6] methods have been used for estimation; here again the β- and γ-isomers are measured together. Brown[7] and Russell Eggitt & Ward[8] successfully used reversed-phase paper chromatography to separate the tocopherols, but the material had first to be rigorously freed of glycerides, carotenoids and sterols by long chromatographic runs, with some danger of tocopherol destruction. Green and coworkers[9, 10] obtained good separations on zinc carbonate-impregnated paper, often without extensive pre-treatment. The well-standardised two-dimensional quantitative paper chromatographic method of the British Analytical Methods Committee[11] is in wide use at present. Even this procedure does not separate β- and γ-tocopherols until these are converted to nitroso derivatives.

Recently thin-layer chromatography (TLC) with a five-component solvent system was successfully applied[12] to the direct qualitative separation of *all* the four tocopherols, which all earlier methods[13, 14] including even gas–liquid chromatography,[15] had failed to effect. A procedure for quantitative estimation of individual tocopherols following such TLC separation is now reported. Thereafter it has been employed to estimate α-, β-, γ- and δ-tocopherols in seven vegetable oils. Obviously the presence or otherwise of other forms of tocopherol in these oils is however not excluded.

Experimental

Materials

Tocopherols.—The following was the stated purity of materials kindly given by Distillation Products Industries, Rochester, N.Y. (U.S.A.):

d-α-tocopherol	:	Pure
d-β- ,,	:	Contains 73% β-, 14% α-, and 13% non-tocopherol
d-γ- ,,	:	Contains 3–5% α-tocopherol
d-δ- ,,	:	98% δ-tocopherol

Each tocopherol concentrate was dissolved in benzene to give a concentration of about 1 μg./μl. An artificial mixture of approximately the same total concentration was also made, and the contents of individual tocopherols calculated from the stated purities of the components.

Solvents.—Light petroleum (b.p. 60–80°), acetic acid and acetone were AnalaR grade, and isopropyl ether and ethyl ether reagent grade.

Oils.—Castor, sesame and safflower seeds (all of Indian origin) and soya-beans (of American origin) were extracted with a solvent to yield the oils. Groundnut and cottonseed oils were laboratory-expelled products. Neem oil was a commercial Indian sample of characteristic odour and colour.

Method

The TLC procedure was essentially that of Mangold.[16] Glass plates (20 × 20 cm.) were coated with a 275-μ layer of silica gel G (E. Merck, Darmstadt) containing 1 ml. of 0·1% sodium fluorescein. Tocopherols show up as purple spots under ultra-violet light. The solvent

system used by Stowe[12] was employed, viz., light petroleum (b.p. 60–80°), ethyl ether, isopropyl ether, acetone and acetic acid in the volume ratio 127 : 1·5 : 16 : 6 : 1·5. The benzene solution of the tocopherol (10–150 μg.) was applied with a micro-pipette. After development the spots were circled under ultra-violet light and loosened using a thin spatula. The loose material was sucked using vacuum into a small sintered funnel. The tocopherols were extracted on the funnel with 3 × 1 ml. portions of absolute alcohol. A similar area of silica gel within the solvent front was also taken as a blank. The tocopherol content of the alcoholic extract was determined by the procedure of Emmerie & Engel.[17] The colour was developed in the dark with 3·5 ml. of 0·5% αα′-dipyridyl and 0·5 ml. of 0·2% ferric chloride. After 2 min., the optical density was measured at 520 mμ on a Unicam SP 600 spectrophotometer in a 1-cm. cell.

Standard graphs were drawn up for each of the four tocopherols correlating optical density of the colour complex with the quantity of tocopherol taken. Since each tocopherol responds differently to the Emmerie–Engel reagent, a separate calibration curve for each tocopherol was necessary.

Recovery of tocopherols from the artificial mixture

Six aliquots of the artificial mixture in amounts ranging from 25 to 150 μg. were spotted, developed and estimated.

Tocopherol contents of vegetable oils

Safflower, neem, castor and soya-bean oils (15 g. each) were saponified according to the procedure of the vitamin-E panel[11] by refluxing for 5 min. with potassium hydroxide (1·6 g./g. of oil) and pyrogallol (60 ml. of 5% w/v ethanolic solution). The unsaponifiable matter was extracted with peroxide-free ethyl ether, and the ether extract washed free of alkali and evaporated under reduced pressure.

Results and discussion

As shown in Table I, an average recovery of about 97–98% of individual tocopherols is obtained from an artificial mixture. If the quantity of each tocopherol exceeds 50 μg., diffused spots are obtained. At the lower limit, between 1 and 3 μg. is revealed. About 10 μg. is adequate for quantitative estimation. In the range up to 150 μg. the plots for the tocopherols are all linear, but distinct for the four forms. A typical TLC separation of tocopherols is shown in Fig. 1. A decided advantage of the procedure is that the total unsaponifiable matter can be directly spotted without further beneficiation, minimising attendant losses. Recovery of added tocopherols from safflower oil ranged from 94 to 96%.

Table II summarises the results of tocopherol estimation of seven vegetable oils by the present procedure. Agreement with earlier values shown alongside is excellent. Vegetable oils are apparently generally devoid of β-tocopherol. The tocopherol content seems to show a wider variation in castor oil than in other oils, recorded values ranging from 0·78 mg./g.[18] to 0·31 mg./g.,[19] with several estimates of about 0·50 mg./g.,[20] similar to the present figure.

Table I

Recovery of tocopherols from an artificial mixture

(Solution spotted contains α–0·3766, β–0·2160, γ–0·2526, δ–0·3244 μg./μl.)

Solution spotted, μl.	Tocopherol recovery, %			
	α–	β–	γ–	δ–
25	98·16	95·94	96·00	94·80
50	93·20	95·94	96·04	96·00
75	103·20	101·30	96·00	100·70
100	96·24	100·00	100·00	95·84
125	98·90	99·12	99·30	98·96
150	98·60	97·82	98·70	96·64
Average	98·1	98·3	97·7	97·2

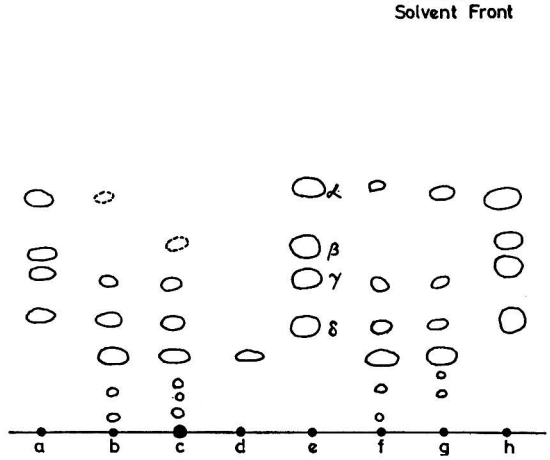

FIG. 1.—*Thin-layer chromatography on silica-gel G of tocopherols, β-sitosterol and the unsaponifiable matter from four vegetable oils*

a, e, h: Artificial mixtures of α-, β-, γ- and δ-tocopherols
d: β-Sitosterol
b: Unsaponifiable matter from castor oil
c: ,, ,, ,, neem oil
f: ,, ,, ,, soya-bean oil
g: ,, ,, ,, safflower oil
(For chromatographic conditions, see text)

Both the present totals and individual tocopherol contents for cottonseed, groundnut and soya-bean oils are remarkably close to those of Green *et al.*[10] A distinctly higher δ-tocopherol content is now found for safflower oil than that reported by Täufel & Serzisko.[18]

Table II

Individual tocopherol contents of vegetable oils (three estimations each)

Oil	Present work					Literature values				Reference
	Total tocopherol, mg./g. of oil	Individual tocopherols, % total α-	β-	γ-	δ-	Total tocopherol, mg./g. of oil	Individual tocopherols, % Total α-	β- + γ-	δ-	
Castor	0·43–0·46 (0·45)	Trace	Nil	47·1–56·5 (51·2)	43·5–52·9 (48·8)	0·78	Trace	48·7	47·8	18
Cottonseed	0·91–0·92 (0·92)	52·6–59·3 (55·4)	Nil	40·4–48·0 (44·6)	Nil	0·81	58·0	42·0[a]	Nil	10
Groundnut	0·92–0·94 (0·93)	35·4–38·6 (36·4)	Nil	62·9–65·4 (64·1)	Nil	0·95	35·5	64·5[a]	Nil	10
Neem	1·08–1·23 (1·17)	Nil	Trace	48·3–52·9 (49·5)	47·1–52·7 (50·5)	—	—	—	—	—
Safflower	0·87–0·91 (0·89)	45·6–55·9 (51·5)	Nil	18·1–26·3 (21·9)	22·6–29·1 (26·6)	0·75	62·0	23·5	6·7	18
Sesame	0·65–0·67 (0·66)	37·6–40·8 (38·9)	Nil	60·9–62·7 (61·5)	Trace	0·60	—	—	—	21
Soya-bean	0·91–0·99 (0·96)	11·3–12·6 (12·0)	Nil	59·9–63·0 (61·8)	25·6–27·6 (26·2)	1·18	13·5	59·0[a]	27·5	10

[a] No β-, all γ-tocopherol (figures in brackets are averages of 3 results)

The tocopherol patterns of two oils have been determined for the first time. Sesame oil, in good agreement with an earlier determination,[21] contains 0·66 mg./g. of total tocopherol. This consists of the α- and γ-forms in the ratio of 2 : 3. Neem oil has a high total tocopherol content (1·17 mg./g.), made up almost equally of γ- and δ-forms with just a trace of the β-form.

Acknowledgment

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NITROGEN FRACTIONS AND SOLUBLE CARBOHYDRATES IN ITALIAN RYEGRASS. I.—Effects of Soil Temperature, Form and Level of Nitrogen

By T. Z. NOWAKOWSKI, R. K. CUNNINGHAM and K. F. NIELSEN*

In a glasshouse experiment Italian ryegrass S 22 grown for 9 weeks during October–December in clay loam at three soil temperatures 11°, 19·5° and 28° was given 6 levels of N (0–500 p.p.m.) as NH_4^+-N or NO_3^--N. Tops grew best at 19·5° and with NO_3^--N yields were greatest at 100 p.p.m. and with NH_4^+-N at 200 p.p.m. Increasing NO_3^--N above 200 p.p.m. greatly decreased growth but increasing NH_4^+-N did not. At 100 p.p.m., NO_3^--N gave better yields than NH_4^+-N but at 200 or more p.p.m. the reverse was true. Total-N, total soluble-N and nitrate-N were much more, and protein-N, amide-N (particularly asparagine) and α-amino-N much less in grass given NO_3^--N than in grass supplied with NH_4^+-N. These differences increased with increasing amounts of applied N. Increasing soil temperature considerably increased the total soluble-N and decreased the protein-N irrespective of the form of N added. With all treatments, plants contained very little carbohydrate; at 11° soluble carbohydrates decreased with increasing level of N, more with NH_4^+-N than NO_3^--N. The causes of nitrate-N accumulation in grass are discussed.

Introduction

The geographical distribution of grass species is determined by various climatic factors of which soil temperature is of major importance. The effect of soil temperature on plant growth was reviewed in detail by Richards *et al.*[1] Temperature not only affects growth but also the chemical composition of plants. Several papers have been published on the influence of temperature on the uptake of inorganic cations and anions by plants,[2–8] but the nutritional value of the herbage depends largely on its organic composition, such as nitrogen fractions and soluble carbohydrates; information on the effect of soil temperature on these is scarce. Sullivan & Sprague[9] studied the effect of temperatures ranging from 10° to 32° on the chemical composition of perennial ryegrass (*Lolium perenne* L.) in growth chambers. High temperatures increased the respiration rate and the top growth was then low in soluble carbohydrates, but rich in amides and ammonium salts; the accumulation of these nitrogenous substances was

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a characteristic of plants grown at high temperature. They referred also to the work of Altergott[10] who ascribed the death of plants at high temperature to the accumulation of ammonium-N. Alberda,[11] studying the effects of cutting, light intensity and night temperature on growth and soluble carbohydrate content of *Lolium perenne* L., showed that at low light intensity the number of tillers and the amount of carbohydrates were decreased. He found also that, although a night temperature of 20° was optimal for growth, the carbohydrate content was inversely related to night temperature within the range 10–30°. This led to exhaustion of carbohydrate reserves and after repeated defoliation the highest yield was obtained at the lowest temperature.

The work described here investigated the effect of changing soil temperature and the level and form of nitrogen on the soluble nitrogen fractions and soluble carbohydrates in grass. Such knowledge may contribute to the more efficient use of nitrogen fertilisers by showing the best time to apply them and also may help to improve the nutritional value of grass. Parks & Fisher[12] showed that the growth and N-uptake by Italian ryegrass were higher at 20° than at 10° or 30°. In this work the grass was grown at 11°, 19·5° or 28°, so that temperatures above and below optimal were used.

Experimental

Italian ryegrass (*Lolium multiflorum*) S 22 was grown for 9 weeks during October to December in transparent plastic pots in a greenhouse. Each pot contained 1·5 kg. of air-dried Rothamsted clay-loam derived from Clay-with-Flints, mixed with 500 g. of quartz. Nitrate-N (NO_3^--N) and ammonium-N (NH_4^+-N) were added at six levels (0–500 p.p.m. of N), and all pots received a uniform dressing of P, S, K, Ca and Mg. The fertilisers were mixed with the soil. There were three replicates. Nitrification of NH_4^+-N was inhibited by treating the quartz with 10 p.p.m. of 6-trichloromethyl-2-chloropyridine) ('N-Serve', Goring[13]) dissolved in acetone; after the acetone had evaporated, the quartz was mixed with the soil. The soils were re-wetted to field capacity when about half the available moisture was gone.

After growing for 15 days from seed at 19 ± 4° the ryegrass was clipped 2 in. above the soil surface and placed in water-baths at 11°, 19·5° and 28° (all ±1°); temperature treatments were not replicated. Fans circulated the air above the pots, keeping the temperature uniform within the range 19 ± 4°. Daylight was supplemented by 400-W HPMV fluorescent lamps above the tanks to give an 18-h. day and an average radiation of approximately 200 cal. cm^{-2} day^{-1}.

After 47 days at the three soil temperatures, the grass was cut at soil level and fresh and dry weights of tops and roots were measured. The NO_3^--N and NH_4^+-N contents and pH (in water) were measured in soil samples taken from all pots at the end of the experiment.

Analytical methods

The grass samples were immediately freeze-dried, ground to pass a 0·5-mm. mesh sieve and stored in a cold room at 5°. All analyses were done in duplicate on freeze-dried material, which had a residual moisture content of about 6%. The results for total-N were calculated as percentage of dry matter but all nitrogen fractions (except total soluble organic-N) were expressed as percentage of total-N.

Dry matter was determined by drying the freeze-dried samples (1·0 g.) for 16 h. at 105°.

Total-N was determined by the Kjeldahl method; the salicylic acid–thiosulphate modification was used to include nitrate-N. The ammonia was distilled in a Hoskins apparatus[14] into 2% boric acid containing Conway & O'Malley's[15] indicator. The distilled ammonia was titrated with 0·01N-sulphuric acid.

Extraction of non-protein-nitrogen

Non-protein-nitrogen was extracted from freeze-dried material by 80% (v/v) ethanol as described by Nowakowski.[16] The total nitrogen of this fraction was designated 'soluble nitrogen'.

Soluble nitrogen was determined by the micro-Kjeldahl method as described by Nowakowski.[16]

Protein-N was calculated as the difference between total nitrogen and total soluble nitrogen.

Ammonia nitrogen was determined in 20-ml. samples of protein-free extract, using a semi-micro-distillation apparatus (Bremner[17]), by steam-distillation with 1·0 g. of heavy magnesium oxide into 3 ml. of 1% boric acid aqueous solution containing Conway & O'Malley's indicator. The distilled ammonia was titrated with 0·07143N-sulphuric acid. It is assumed that the calculated value for ammonium-N includes part of glutamine decomposed during steam distillation.

Nitrate nitrogen was determined in 2–5-ml. samples of protein-free extract by a new method (Bremner[17]) ; nitrate-N was reduced to ammonia by adding 2 ml. of titanous sulphate solution (Bremner & Shaw[18]). [Titanous sulphate 15% (w/v) solution supplied at present by the British Drug Houses Ltd. does not reduce nitrate-N to ammonia quantitatively.] Gasser[19] found that, when ferrous sulphate was added to titanous sulphate, nitrate-N was recovered quantitatively as ammonia. The modified reagent is prepared by adding 15 ml. of ferrous sulphate solution (200 g. of $FeSO_4,7H_2O$/litre in 0·5N-sulphuric acid) and 10 ml. of water to 25 ml. of titanous sulphate. Two ml. of this reagent, prepared daily, are used for each determination.

Amide nitrogen was measured as the increase in ammonia nitrogen on hydrolysis of 15 ml. of de-proteinised extract with 15 ml. of 2N-sulphuric acid at 100° for 3 h. After hydrolysis, the acidity of the hydrolysate was decreased by adding 15 ml. of 1·8N-sodium hydroxide before distilling a portion of the solution with 1·0 g. of heavy magnesium oxide. The increase in ammonia represents the nitrogen of the acid–amide group of asparagine and glutamine.

Glutamine nitrogen was determined by the method of Vickery *et al.*[20] Five ml. of de-proteinised extract were hydrolysed with 10 ml. of phosphate–borate buffer (pH 6·5). The hydrolysate was distilled with 3 ml. of borax–sodium hydroxide buffer (pH 9·6). The increase in ammonia represents glutamine amide-N.

Asparagine nitrogen was estimated by the difference between amide-N and glutamine-N.

Amino-acid nitrogen.—α-Amino-nitrogen was determined by the micro-method of Van Slyke *et al.*[21] In this method the most difficult and critical part is to deliver 1 ml. of 0·125N-barium hydroxide solution without contaminating it by CO_2 from the air. Most satisfactory results were obtained by use of a 1-ml. burette with interkey interchangeable stopcock fitted with a FLOUN–P.T.F.E. key with fine control device (supplied by C. Springham & Co.). The burette was directly connected with a reservoir containing the barium hydroxide solution.

Total soluble carbohydrates were determined by extracting 1·0 g. of grass with 100 ml. of water on a water-bath for 2 h. under a reflux condenser. The contents of the vessel were filtered, washed with boiling water, and the filtrate and washings made up to 500 ml. Soluble sugars were determined by the anthrone method of Murphy[22] but 10 ml. of anthrone reagent and 2 ml. of plant extract were used instead of 5 and 1 ml. as recommended.

The results are not discussed statistically because the temperature treatments were not replicated and because the amount of plant material was such that replicates of nitrogen treatments had to be bulked.

Results

The results dealing with yields have already been reported by Nielsen & Cunningham[8] and are not discussed here except in so far as they are relevant to this paper.

The grass in soil supplied with NH_4^+-N absorbed N mainly but not entirely as NH_4^+. Tests showed that ' N-Serve ' inhibited nitrification completely only after 10 days. At the end of the experiment the soil in all pots given NH_4^+-N had less than 2 p.p.m. of NO_3^--N.

The soil which did not receive nitrogen had pH 6·4. Both soil temperature and NH_4^+-N had little effect on pH but increasing NO_3^--N from 0 to 500 p.p.m. lowered the pH to 5·7.

Percentage of total-N in grass (Fig. 1a)

Addition of nitrogen up to 200 p.p.m. sharply increased the % of total-N in grass and increasing the N level above 200 p.p.m. produced a further gradual increase. The % N in the grass was always more with NO_3^--N than with NH_4^+-N, irrespective of soil temperature. In grass grown in soil without added N the % N was least at 11° and most at 28°. With 100 p.p.m. of N, as NO_3^-- or NH_4^+-N, the difference in the % N between grass grown at 11° and grass

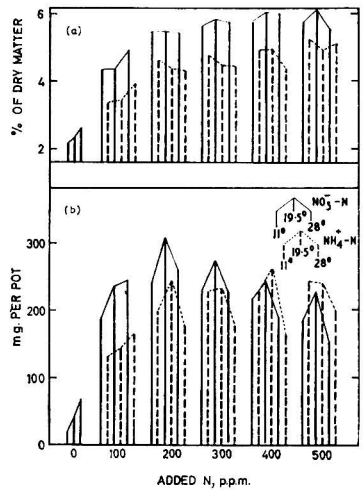

FIG. 1.—*Effects of soil temperature, form and level of N on (a) % total-N in grass and (b) the amount of N in grass*

grown at 19·5° was small, but increasing the soil temperature to 28° considerably increased % total-N in the grass. Above 100 p.p.m. N, soil temperature had little effect on % total-N in grass.

Amount of N in grass (Fig. 1b)

Fig. 1b shows that grass from the control pots without added nitrogen took up 16 mg. of N per pot (equivalent to 8 p.p.m. of soil N) at 11° and the N-uptake increased to 66 mg. of N per pot (equivalent to 33 p.p.m. of soil N) at 28°. This was due to an increase in the rate of mineralisation of soil nitrogen. Presumably temperature had a similar effect on mineralisation in all the other N treatments, but the additional N mineralised did not show in the uptake figures (Fig. 1b) when more than 100 p.p.m. of fertiliser-N was added. At N levels above 100 p.p.m. factors other than the amount of mineral-N in the soil appear to govern uptake of N. With NO_3^--N, the N-uptake increased with increasing level of N up to 200 p.p.m. and then decreased. With NH_4^+-N, the N-uptake increased with increasing supply of N up to 300 p.p.m. and then remained constant. Up to 300 p.p.m. the grass took up more N when supplied with NO_3^--N than when given NH_4^+-N, but with more than 300 p.p.m. the reverse was true.

Total soluble nitrogen and protein-N (Figs. 2 and 3)

The total soluble-N as % of total N (Fig. 2) increased with increasing amounts of N and was much greater in grass receiving NO_3^--N than in grass given NH_4^+-N at all N levels. Total soluble-N in grass increased with soil temperature irrespective of the form of N added. The form of N affected soluble-N more than did the soil temperature. The changes in protein-N were the reverse of those in soluble-N (Fig. 3).

Ammonium-N content (Fig. 4)

At soil temperature of 11° increasing N had very little effect on accumulation of NH_4^+-N in grass, irrespective of the form of N given. As soil temperature increased, the NH_4^+-N content increased at all levels of applied N and with both forms of N. This increase was greater with NH_4^+-N than with NO_3^--N. The effect of temperature increased with increased addition of NH_4^+-N.

Nitrate-N (Fig. 5)

Grass receiving NO_3^--N had more NO_3^--N than grass given NH_4^+-N. Nitrate-N in the grass increased greatly with increases in the amount of NO_3^--N given, but with NH_4^+-N, NO_3^--N

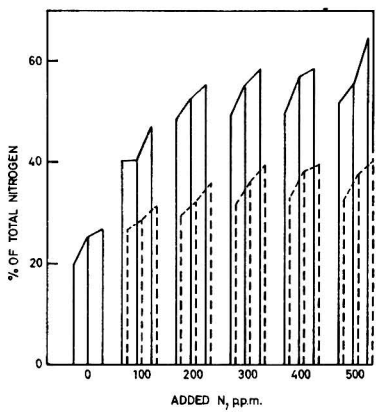

FIG. 2.—*Effects of soil temperature, form and level of N on total soluble-N in grass expressed as % of total-N*
(Key as in Fig. 1)

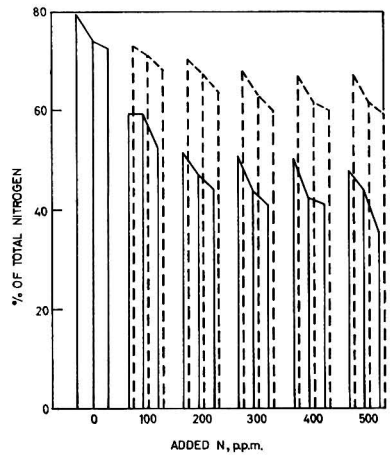

FIG. 3.—*Effects of soil temperature, form and level of N on protein-N in grass expressed as % of total-N*
(Key as in Fig. 1)

increased little. Also NO_3^--N in grass grown both with and without added N increased greatly when soil temperature was raised. Increasing soil temperature from 11° to 28° affected accumulation of NO_3^--N in grass more than increasing the amount of added N above 100 p.p.m. The form of the applied N had a greater effect on accumulation of NO_3^--N than did the soil temperature. However, in this experiment there was still a considerable amount of NO_3^--N in grass given NH_4^+-N, probably because nitrification was not inhibited completely in the first 10 days by 'N-Serve'.

Total soluble organic nitrogen (Fig. 6)

This fraction, calculated as the difference between total soluble nitrogen and mineral nitrogen (ammonium-N + nitrate-N), was much greater in grass given NH_4^+-N than in grass supplied with NO_3^--N. Increasing both level of N and soil temperature did not affect the soluble organic nitrogen, expressed as percentage of total-N, but, expressed as percentage of total soluble-N, it decreased when level of N and soil temperature increased.

Total amide-N (Fig. 7)

In grass grown without added N, amide-N was most at 19·5° and least at 28°. Grass given NH_4^+-N had more amide-N than grass given NO_3^--N at all N levels; amide-N was greatest

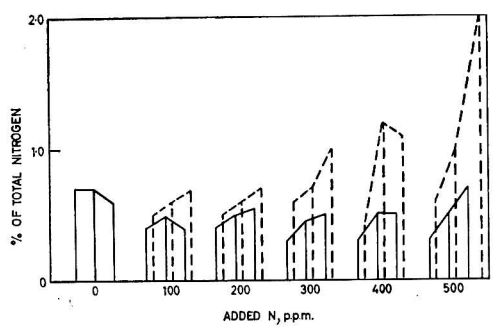

FIG. 4.—*Effects of soil temperature, form and level of N on ammonium-N in grass expressed as % of total-N*
(Key as in Fig. 1)

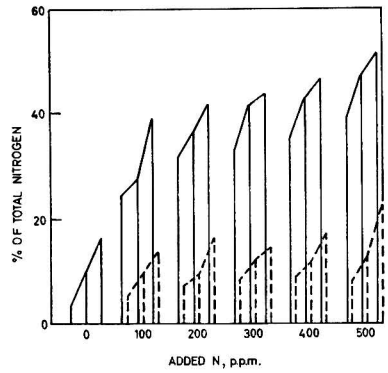

FIG. 5.—*Effects of soil temperature, form and level of N on nitrate-N in grass expressed as % of total-N*
(Key as in Fig. 1)

at 400 p.p.m. of NH_4^+-N and then decreased. Except with 300 p.p.m. N, amide-N increased with increasing soil temperature from 11° to 19·5°. Further increase in soil temperature decreased amide-N at all levels of applied NH_4^+-N. In grass given NO_3^--N, amide-N decreased as level of N or soil temperature increased. The amide-N content of grass was affected more by the form of N than by soil temperature.

Glutamine-N (Fig. 8)

The form of N applied had very little effect on glutamine-N at 100 p.p.m. of N, but at 200 p.p.m. of N and above, glutamine-N was greater in grass receiving NH_4^+-N. An increase of NH_4^+-N from 100 to 200 p.p.m. increased glutamine-N but further increases in N level had no consistent effect. With 100–200 p.p.m. of NH_4^+-N, increasing soil temperature increased glutamine-N but decreased it at higher N-levels. With NO_3^--N, there was so little glutamine

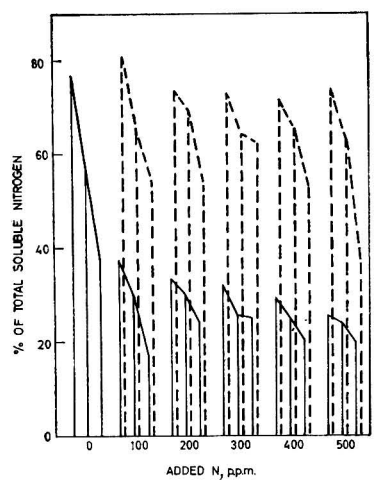

FIG. 6.—*Effects of soil temperature, form and level of N on total soluble organic-N expressed as % of total soluble-N*
(Key as in Fig. 1)

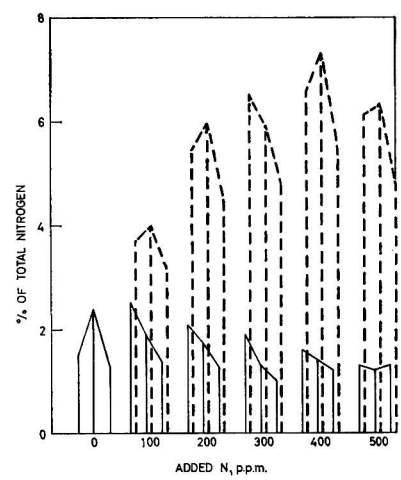

FIG. 7.—*Effects of soil temperature, form and level of N on total amide-N expressed as % of total-N*
(Key as in Fig. 1)

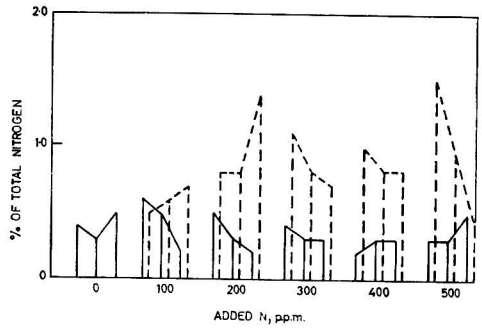

FIG. 8.—*Effects of soil temperature, form and level of N on glutamine-N expressed as % of total-N*

that the analytical error in its determination was probably greater than the effects of N level and soil temperature.

Asparagine-N (Fig. 9)

The form and level of N and soil temperature changed asparagine-N similarly to total amide-N (Fig. 9).

α-Amino-N (Fig. 10)

Grass given NH_4^+-N had more α-amino-N than grass given NO_3^--N. Increasing NH_4^+-N increased α-amino-N whereas increasing NO_3^--N decreased it slightly. With NO_3^--N, increasing soil temperature decreased α-amino-N but with NH_4^+-N the effect was not consistent.

Unaccounted soluble organic-N

The unaccounted soluble organic-N (total soluble organic-N less amide- and α-amino-N), which includes also all the errors of determination of the other fractions, ranged from 3 to 11% of total-N, but there was no clear pattern of the effects of soil temperature, level or form of N. This nitrogen fraction was not identified.

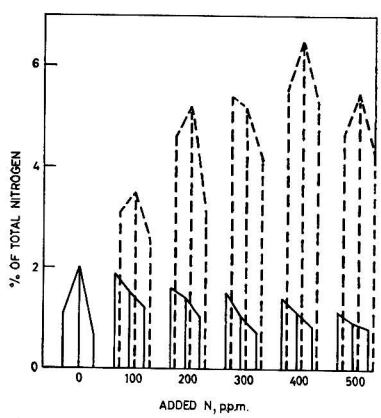

FIG. 9.—*Effects of soil temperature, form and level of N on asparagine-N expressed as % of total-N*
(Key as in Fig. 1)

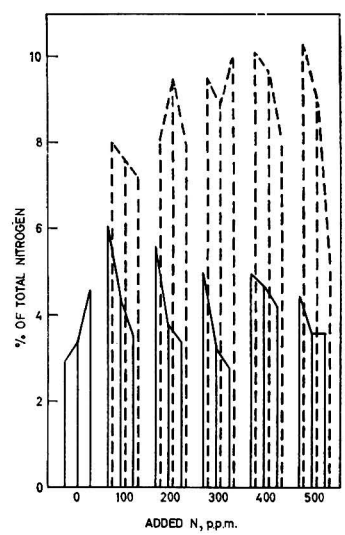

FIG. 10.—*Effects of soil temperature, form and level of N on α-amino-N expressed as % of total-N*
(Key as in Fig. 1)

Total soluble carbohydrate content in grass (Table I)

Samples from six treatments were too small for carbohydrates to be determined. The carbohydrate content was very low in all the samples analysed. This could be caused by the long, 18-h. day (leading to increased leaf production), the high temperature and the high rate of N application as well as by the low light intensity (Alberda[11, 23]). However, even in the control, when N supply was very small, carbohydrate was also low suggesting that the light factor rather than the high N supply accounted for low carbohydrate. At a soil temperature of 11°, increasing level of N decreased soluble carbohydrates in grass and this decrease was greater in grass given NH_4^+-N. At all levels of applied NH_4^+-N, soluble carbohydrates decreased when soil temperature increased from 11° to 19·5°. A further increase of soil temperature to 28° decreased carbohydrates only at the 100 p.p.m. level. With NO_3^--N, increasing soil temperature decreased carbohydrates only with the smallest amount of applied N.

Table I

Effects of soil temperature, form and level of nitrogen on the total soluble carbohydrate content in grass

(Total soluble carbohydrate content as % of dry matter)

Soil temperature	Without added N	NO_3^--N added, p.p.m.					NH_4^+-N added, p.p.m.				
		100	200	300	400	500	100	200	300	400	500
11°	6·7	5·9	4·2	4·3	n.d.	4·2	5·1	3·5	3·1	2·8	3·0
19·5°	4·3	5·6	4·3	5·0	n.d.	4·9	4·0	2·2	2·6	n.d.	2·5
28°	4·6	4·5	4·1	4·8	n.d.	4·9	3·5	2·5	2·8	n.d.	n.d.

Discussion

The discussion will be limited to a few of the more important results.

The effect of soil temperature on total soluble-N and growth

Raising soil temperature from 19·5° to 28° diminished yields[8] (Fig. 11), probably because respiratory losses were higher at 28°. When respiration is excessive and carbohydrate reserves are low, protein may also be respired and soluble nitrogen compounds accumulate. Alternatively, the low carbohydrate level may restrict protein synthesis, again resulting in an accumulation of soluble nitrogen. This accumulation of total soluble nitrogen as yields decreased agrees with the results of Sullivan & Sprague.[9]

The effect of ammonium-N accumulation on growth

When soil temperature was raised from 19·5° to 28° the accumulation of NH_4^+-N in grass receiving NH_4^+-N was accompanied by a decrease in asparagine-N. Thus at 28° asparagine synthesis from NH_4^+-N was retarded. Asparagine synthesis appears to involve aspartate, adenosine triphosphate (ATP) and ammonia.[24] The energy required for synthesis of both

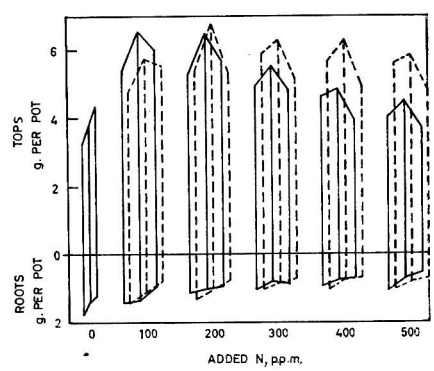

FIG. 11.—*Effects of soil temperature, form and level of N on yields of tops and roots of Italian ryegrass*
(Key as in Fig. 1)

aspartate and ATP, is ultimately derived from carbohydrates. Thus shortage of carbohydrates could lead to an accumulation of ammonia. However, even when NH_4^+-N concentration in grass was extremely large, 2% of total-N (0·1% of dry matter) the growth of the plants was checked, although no plants died.

Effect of nitrate accumulation on growth

Grass grown at 28° with 500 p.p.m. of NO_3^--N had 51·3% of total-N as nitrate, which corresponds to 2·79% of nitrate-N in dry matter. In spite of this extremely large nitrate content, grass did not die and this confirms Bonner's[25] view that plants tolerate large accumulations of nitrate. However, the accumulation of nitrate was related to growth. The top growth was greatest with 100 p.p.m. of NO_3^--N at 19·5° (Fig. 11) ; when NO_3^--N was 28·7% of the total-N in grass (corresponding to 1·13% of nitrate-N in dry matter) and above this growth decreased ; Hageman & Flesher[26] found that maize growth and protein synthesis were not suppressed when nitrate concentration was 0·154% fresh weight, corresponding to 0·8% dry weight, assuming 20% dry matter in fresh material.

Cause of nitrate accumulation in grass

The most striking result in this experiment was the large accumulation of NO_3^--N in the grass. This could be attributed to interactions between several possible factors, such as : (1) increased uptake of NO_3^- ; (2) decreased activity of nitrate reductase resulting from differences either (*a*) in soil temperature or (*b*) in NO_3^- concentration in the grass ; (3) lack of carbohydrates for nitrate reduction and (4) decreased rate of photochemical reduction of nitrate. In connexion with these factors :

(1) Williams & Vlamis[5] found that raising the soil temperature affected the absorption of NO_3^- by barley plants more than that of any other ion ; changing the soil temperature from 13° to 25° increased NO_3^- absorption tenfold.

(2*a*) Anderson[27] found that in October less nitrate was reduced in *Solanum dulcamara* than in June when the temperature was higher ; he concluded that the nitrate-reducing substance is thermolabile. The question arises whether there is a change in nitrate-reductase activity when the temperature is raised and more NO_3^- is absorbed. Nightingale,[28] working with apple trees, found that reductase activity was greatest at 18·3° and least at 7·2° and 32·2°. While his results with apple trees may not hold for grass, they do indicate that accumulation of nitrate in grass grown at 19·5° is not necessarily due to a reduction in the activity of nitrate reductase.

(2*b*) When investigating the effect of nitrate concentration on the activity of nitrate reductase isolated from soya-bean leaves, Evans & Nason[29] found that most activity occurred when the nitrate concentration in the substrate was 0·02M, the activity remaining constant with increased concentration of nitrate. In contrast, Hageman & Flesher[26] found that nitrate reductase activity in extracts of maize seedlings or leaves increased as nitrate concentration in the nutrient solution and in the plant tissue increased ; the concentration of nitrate (0·8% of dry matter) which they found did not suppress growth and protein synthesis corresponds to 0·14M. In our experiment, at 19·5° the nitrate content of grass given 100 or 200 p.p.m. of NO_3^--N was between 1·2 and 2·0% of dry matter or 0·24 and 0·40% of fresh material when production of total organic-N (total-N less inorganic-N) by the grass reached a maximum and remained constant (Table II). Assuming uniform distribution of nitrate in the tissue this would

Table II

Relationship between total organic-N and nitrate-N in grass grown at 19·5°

Amount of NO_3^--N given, p.p.m.	Nitrate-N	Total organic-N
	% of dry matter	
0	0·26	2·10
100	1·15	2·93
200	2·04	3·50
300	2·43	3·40
400	2·61	3·48
500	2·61	3·52

correspond to a molarity range of 0·22 to 0·36. In our experiment, done over the long period of 9 weeks, changes in nitrate-reductase activity were not detected, but when nitrate in tissue exceeded 0·22–0·40M no more organic-N was formed and nitrate accumulated. The discrepancy between the findings of Evans & Nason,[29] and both ours and those of Hageman & Flesher,[26] probably arises because enzymes within cells operate in different conditions from those which are convenient for studies *in vitro*. However, Fig. 5 shows that increasing soil temperature increased nitrate accumulation in grass similarly with NO_3^--N or NH_4^+-N, despite the greater nitrate content when NO_3^--N was given. This suggests that the concentration of nitrate was not limiting nitrate activity in our experiment.

(3) The energy required by enzyme systems which produce triphosphopyridine nucleotide (TPNH) and diphosphopyridine nucleotide (DPNH) is derived from the intermediates of carbohydrate metabolism. In our experiment nitrate may have accumulated in grass at a soil temperature of 19·5° because nitrate-reductase activity diminished from lack of energy needed to produce reduced pyridine nucleotides.

(4) Evans & Nason[29] also showed that TPNH could be produced photochemically and Turner *et al.*[30] found that the rate at which triphosphopyridine (TPN) is reduced in spinach chloroplasts increased linearly with increasing light intensity.

Candela *et al.*[31] found that darkness inhibited nitrate-reductase activity. Lack of light is a limiting factor either when energy for producing reduced pyridine nucleotides is derived from the intermediates of carbohydrate metabolism or when pyridine nucleotides are reduced photochemically. In our experiment, light intensity was low and this was probably the main reason for nitrates accumulating in grass at a soil temperature of 19·5°. The further increase in nitrate content of grass when soil temperature was raised to 28° probably reflects the combined effect of two factors, a soil temperature above the optimum[28] (~18°) and limited production of TPNH, each of which decreased the activity of nitrate reductase.

Changes in soluble organic-N

There was little soluble organic nitrogen in grass grown with NO_3^--N but, as usual (Steward *et al.*[32]), this fraction, with asparagine as a main constituent, increased when NH_4^+-N was given. Increasing soil temperature decreased total soluble organic-N probably because carbohydrates were depleted. There was much soluble organic-N unidentified and this agrees with the results of Brady[33] with fresh ryegrass and clover.

Conclusions

Both soil temperature and form of N greatly influence the composition of grass and therefore its nutritional value. Our experiment shows that when much N (above 300 p.p.m.) is given to grass grown at high soil-temperature and low light-intensity, NH_4^+-N gives better growth than nitrate. At these high rates of N the uptake of NH_4^+-N is also higher than that of NO_3^--N. Most of both forms of N was taken up at 19·5°. Yields of grass at 11° and 19·5° were smaller when nitrate content exceeded 1·1% of dry matter and at 28° were still smaller at 1·9% of dry matter. In spite of this apparent relationship there was no evidence that the accumulation of nitrate was solely responsible for the smaller growth, which was more probably caused by inadequate light intensity. The small amount of carbohydrate associated with low light intensity was no doubt a main cause of the accumulation of nitrate. Grass fertilised with NH_4^+-N had a larger percentage of total-N as protein-N than grass given NO_3^--N, which shows that NH_4^+-N was used more efficiently for protein synthesis. Grass grown at 28° synthesised protein less efficiently than at 11°.

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PHYSICO-CHEMICAL STUDIES ON AGRICULTURAL SPRAYS. VI.*—Survey of Methods for Measuring the Wetting Ability of Spray Formulations

By C. G. L. FURMIDGE†

Several methods have been suggested for the measurement of the wetting efficiency of high-volume sprays and some of these have been examined to see how far they will predict the wetting ability of a range of wetting agents applied to several leaf surfaces.

It is concluded that a satisfactory wetting test must involve the appropriate spray target surface and also take into account the effects of spray droplet impaction ; these requirements are satisfied by a test based on the visual assessment of wetting under practical spraying conditions. Of the other tests examined, the measurement of the receding contact angle is of value in recognising the extreme case of complete wetting of a surface and the tape-sinking test may be used, with anionic and non-ionic wetters, as a rough guide to the overall efficiency of wetters on leaf surfaces.

Introduction

The preceding paper[1] in this series emphasised that the concentration of any wetting agent added to a high-volume spray formulation should be kept to the minimum that will provide the required coverage of the target surface. The extent of the coverage that is required

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varies with the purpose of the spray and with the mode of action of the toxicant. Where the target surface is readily wetted or where the emulsifying or dispersing agents used in the spray formulation also possess some wetting properties, the addition of extra wetting agents may be unnecessary. The question whether wetters should be used and, if so, what concentration should be employed, can be answered only by testing the wetting properties of the formulations. Several wetting tests for agricultural sprays have been proposed. The emphasis has been placed on developing laboratory tests, using artificial conditions in the hope that the results obtained would predict the wetting properties of sprays in the field.

A satisfactory wetting test for use with high-volume sprays should be capable of assessing the wetting performance of any spray liquid whatever the conditions under which it is used. Ideally, the test should provide a measure of all degrees of wetting, from zero to complete wetting on any type of foliage surface. In the present work, some of the wetting tests have been surveyed, to see whether they will predict the wetting ability of a range of wetting agents on several types of leaf surfaces.

Experimental

Materials

Surface-active agents.—Sixty-five surface-active agents were used in these tests; they included anionic, cationic, non-ionic and ampholytic types and are listed in Table I of Part V of this series.[1] A range of concentrations of each of these materials was prepared in distilled water, the concentration being based on the percentage weight of active ingredient in solution.

Solid surfaces.—The surfaces included the four leaf types described in Part V,[1] apple (M2 rootstock), plum (Victoria), blackcurrant (Baldwin) and banana (Lacatan). The measurements on each leaf type were carried out on leaves of similar age and from similar positions on the plants.

An artificial surface of white beeswax was also used, prepared by dipping clean, glass microscope slides into molten beeswax kept at 90°. After being dipped, the slides were allowed to drain and cool and the wax to solidify; they were used within 24 h. of preparation.

Wetting test methods

(a) *Visual assessment*

The most commonly used wetting test is the visual assessment of the proportion of a solid surface that remains covered with a liquid film after excess of the liquid has drained off. This visual assessment may be carried out after dipping the surfaces into the spray liquid or after spraying the surfaces to beyond run-off; the surfaces used are usually leaves but artificial surfaces have also been tried. This method suffers from the disadvantage that it is difficult to distinguish accurately between those areas of the surface that remain covered by a film of liquid and those that are not so covered.

Ashworth & Lloyd[2] suggested the use of this type of test as a standard for the field evaluation of wetting agents. The difficulty of assessing the leaf area wetted was minimised by progressively increasing the concentration of wetter until the leaf samples were 100% wetted, a condition that can be assessed much more easily than intermediate degrees of wetting. The efficiency of the wetters was compared in terms of their concentration required to promote 100% wetting.

In the present work this method has been used to measure intermediate as well as complete wetting. The whole leaves (in the case of banana, portions of leaf) or beeswax slides were immersed in the wetter solution for a few seconds, then removed, allowed to drain for 10 sec. and examined. The practical difficulties and inaccuracies in this assessment were overcome by using experienced observers and by classifying the extent to which the surface remained covered by the liquid in the ranges 5, 10, 25, 50, 75, 90, 95 or 100% of the total surface. This wetting classification was assisted by suitable charts and observer bias was checked by using solutions containing fluorescent dyes, the areas wetted being examined under ultra-violet light. By these means, the visual assessment method can be made, with care and practice, both accurate and reproducible, as will be shown in a later paper.

(b) *Methods based on the surface properties of spray liquids*

Fig. 1 shows some typical curves of wetting (as determined visually) plotted against concentration for several wetters on leaf surfaces. The variation of wetting with concentration of wetter is similar to the variation of many other physical properties of wetters with concentration and several surface properties have been used as measures of wetting ability. These include the area of spread of constant-sized drops on solid surfaces, the surface and interfacial tensions of the spray liquid, the contact angles of the liquid on various solid surfaces and the spreading coefficient. Many early workers in this field appear to have been confused between the physical requirements for the wetting of surfaces by sprays and those for the spreading of individual spray droplets over the surface. The difference between these two effects was stressed by Evans & Martin[3] who defined wetting as the ability of the liquid to form a persistent liquid-solid interface when excess of the liquid has drained off, and spreading as the ability of the liquid to form a liquid–solid interface by surface activity over the plane surface of a solid.

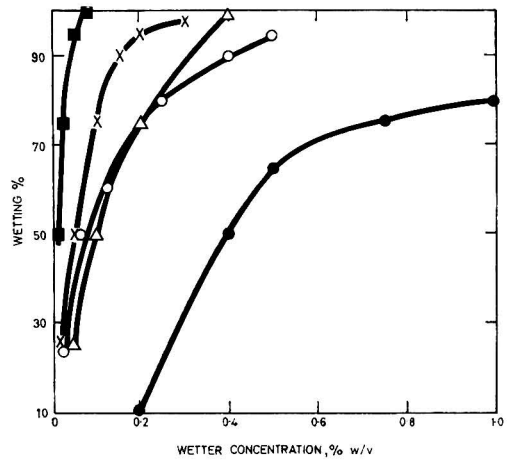

FIG. 1.—*Variation of leaf wetting with concentration of wetting agent*

■ cetylbetaine on blackcurrant leaf (upper surface)
○ sodium dodecyl sulphate on blackcurrant leaf (under surface)
× potassium oleate on plum leaf (under surface)
△ dodecylpyridinium bromide on apple leaf (upper surface)
● ,, ,, ,, banana leaf (under surface)

Area of spread.—This was used by O'Kane *et al.*[4] as a measure of the 'contact quality' of a spray liquid. In the present work, the measurements were carried out by forming 0·05 ml. drops of the wetter solutions from a hypodermic needle and carefully placing them on a horizontal solid surface. When the drop had finished spreading, its plan was photographed and its area measured from the projected negatives. As reported by Evans & Martin,[3] it was found extremely difficult to get reproducible areas of spread even on the uniform surface of beeswax, and virtually impossible on leaf surfaces. Therefore, this method was abandoned.

Surface tension.—This property has been used for the comparison of the efficiency of wetting agents.[5, 6] However, Cooper & Nuttall[7] pointed out that wetting was based on three interfacial tensions: the air–liquid surface tension, the air–solid and solid–liquid interfacial tensions; and that it was unwise to base conclusions on the value of one of these interfacial tensions only. Hamilton,[8] and more recently Thompson,[9] have shown that in fact surface tensions give no reliable guide to wetting behaviour.

Contact angles.—The contact angle of a liquid on a solid surface is a function of all three interfacial tensions and it has been used by many workers[3, 10–15] as a measure of wetting and spreading behaviour. The hysteresis exhibited by the contact angle of most liquid–solid systems

complicates the use of this surface property and some workers have used the equilibrium value (θ_E) given by

$$\cos \theta_E = (\cos \theta_A + \cos \theta_R)/2$$

where θ_A and θ_R are the advancing and receding values of the contact angle. Evans & Martin[3] pointed out that the advancing and receding angles should be considered separately when studying the behaviour of liquids on solid surfaces and that the advancing angle was likely to be important in governing the spreading ability of the liquid whilst the receding angle would govern the wetting ability.

The advancing and receding contact angles have been recorded on beeswax and leaf surfaces by photographing the profile of moving drops.[16] The angles were measured with a protractor from the projected photographic negatives. Receding angles of less than 10° were found difficult to measure accurately in this way but they were classified according to the trail of liquid left behind the moving drop. No trail was left when the receding angle was greater than 5°; a patchy trail whose width measured across the line of motion was less than that of the drop itself, was left when the angle was in the range 1–5° and a completely wetted trail, of the same width as the drop, was left when the receding angle was zero.

The contact angles were measured for various concentrations of each wetter, the concentration being progressively increased until the receding contact angle was reduced to zero on each surface.

Spreading coefficient.—This is given by the expression

$$W = \gamma_{LA}(\cos \theta_A - 1)$$

where γ_{LA} is the liquid–air surface tension, and Martin[17] has shown that this can be used to predict the spreading ability of spray liquids. If θ_R is used in place of θ_A, the expression may be useful in predicting the wetting ability.

In calculating this coefficient, the contact angles were measured as described above and the surface tensions were measured by the drop weight method, using the correction factors listed by Harkins & Brown.[18]

(c) *Air displacement methods*

Several methods of this type are used in the textile industry, the most important being the Draves test[19] and the Shapiro test.[20] The *Draves test*, in which the wetting efficiency of a solution is related to the time taken for a standard hank of cotton to sink in the solution, was used to assess leaf wetting by Thompson.[9] He found that this test provided a better correlation with leaf wetting under high-volume spraying conditions than did tests based solely on the measurement of surface or interfacial tensions. The *Shapiro test*, a modification of the Draves test in which a length of standard cotton tape is used in place of the cotton hank, has been proposed by Ashworth & Lloyd[2] as a standard laboratory test for the screening of wetters for agricultural sprays. A total of nine wetters (anionic and non-ionic) were tested by this method and the results showed good agreement with visual tests of the wetting of cabbage leaves; no cationic wetters were examined.

In the present work the test was used as described by Ashworth & Lloyd.[2] A 22-cm. length of cotton tape (1 in. wide, unbleached and conforming to B.S. 1625 : 1950) with a 0·500-g. weight attached to its lower end, was completely immersed in the wetter solution. Initially the tape was buoyant due to the entrapped air but as this escaped the tape sank. The time taken between immersion of the tape and the moment it began to sink was recorded as the sinking time. This was measured in various concentrations of each wetter and the sinking times were plotted against concentration on a log/log scale. Most wetters gave a straight line plot from which the concentration of wetter required to cause the tape to sink in a standard time could be deduced.

Results

In the first place, the methods were examined to see how accurately they could predict the extreme case of complete wetting. It has been assumed that any test method that did not

predict this extreme condition reasonably accurately was unlikely to be of value in predicting intermediate degrees of wetting.

The visual assessment method has been used to list the 65 wetters in order of their efficiency at promoting 100% wetting on each of the surfaces used and also to provide the minimum concentration of each wetter required to promote 100% wetting of each surface (the minimum wetting concentration). The list is shown in full in Table I of Part V of this series[1] and an abridged list of the best 15 wetters on each surface is given here (Table I, sections 1, 2, 3, 5 and 6). These results have been used as standards for the evaluation of other methods.

A range of solutions, each containing the minimum wetting concentration of different wetters on a particular surface according to visual assessment, were examined by the various wetting test methods. In terms of wetting, all these solutions were equivalent, and any satisfactory method should have given approximately constant results with the whole range of solutions. Where the method showed any promise in this test it was used to list the full range of 65 wetters in order of their efficiency and this order of ranking was compared with the corresponding order of ranking given by the visual tests using Kendall's coefficient of rank correlation.[21] This is given by the expression:

$$\text{Coefficient of rank correlation } \tau = \left(\frac{2P}{\frac{1}{2}n(n-1)}\right) - 1$$

where P is the number of greater rank values appearing below any specific rank value in the correlated column, and n is the number of ranks.

The maximum value for τ is unity, representing perfect correlation; as the correlation becomes worse, τ becomes smaller. As a practical guide it has been assumed that a minimum value of $\tau = 0{\cdot}80$ is required for reasonable correlation and even with this value, the ranking position of some wetters may be considerably misplaced. Good correlation throughout the whole list required a value of $\tau = 0{\cdot}90$.

The behaviour of cationic surface-active agents on leaf surfaces often differs considerably from that of anionic and non-ionic materials.[22] Because most formulated sprays contain only anionic or non-ionic surface-active agents and some methods may not cope adequately with cationic materials, the value of τ has been calculated separately for the 39 anionic and non-ionic wetters in the full list. The coefficients are referred to as τ_T for the total of 65 wetters and τ_{AN} for the anionic and non-ionic wetters.

Finally, any test method that gave reasonable correlation with either the full range or with the anionic and non-ionic wetters only were evaluated against the visual assessment method for predicting intermediate degrees of wetting.

(a) *Visual assessment*

Some useful conclusions can be drawn from correlation of the visual assessment results among themselves. The efficiency of wetters on any one surface has been correlated with their efficiency on each of the other surfaces and the values of τ obtained are listed in Table II.

The correlation found between the four leaf surfaces is generally poor; plum and blackcurrant leaves show the closest correlation for all wetters ($\tau_T = 0{\cdot}79$), whilst plum and apple show reasonable correlation for anionic and non-ionic wetters only ($\tau_{AN} = 0{\cdot}82$). The results on the beeswax surface do not correlate with any of these four leaf types.

The fact that the various leaf types differ so markedly in the ease with which they are wetted by particular wetters means that it is impossible to classify wetters in order of their general wetting efficiency, only their specific efficiency related to a particular surface. It also means that wetting results obtained on one solid surface cannot be used to predict the wetting efficiency of the same solutions on a different solid surface. Further, it would seem unlikely that any test employing artificial surfaces can be related generally to the wetting of all types of leaves.

(b) *Methods based on surface properties*

Table III shows the values of the surface tension, the advancing contact angle and the spreading coefficient (based on θ_A) for the best 15 wetters on banana and beeswax surfaces, measured using solutions containing the minimum wetting concentration of each wetter. The

Table I

Order of efficiency of the best 15 wetters, from the total of 65 used, as given by the various test methods

The wetter numbers refer to the order of efficiency of these materials at wetting apple leaves (see Table I, Part V, this series[1]). The wetters used here are listed below

(concentration as wt.-%)

1 Apple leaf wetting (Visual assessment)		2 Blackcurrant leaf wetting (Visual assessment)		3 Banana leaf wetting (Visual assessment)		4 θ_R = 0 on banana leaf		5 Plum leaf wetting (Visual assessment)		6 Beeswax wetting (Visual assessment)		7 θ_R = 0 on beeswax		8 Value of θ_R on beeswax at concn. of 0·01%		9 Tape sinking in 15 sec.		10 Tape sinking in 30 sec.	
Wetter number	Min. wetting concn.	Wetter number	Min. wetting concn.	Wetter number	Min. wetting concn.	Wetter number	Concn. required	Wetter number	Min. wetting concn.	Wetter number	Min. wetting concn.	Wetter number	Concn. required	Wetter number	θ_R	Wetter number	Concn. required	Wetter number	Concn required
1	0·018	5	0·030	4	0·010	4	0·010	5	0·029	6	0·012	6	0·012	6	2°	1	0·018	8	0·013
2	0·021	3	0·031	24	0·012	14	0·012	3	0·032	3	0·016	3	0·018	3	7°	8	0·019	1	0·014
3	0·024	1	0·035	14	0·013	37	0·012	6	0·033	17	0·018	5	0·020	37	8°	3	0·024	3	0·015
4	0·024	4	0·040	37	0·015	3	0·013	1	0·034	5	0·021	17	0·024	14	11°	7	0·025	7	0·016
5	0·026	14	0·045	3	0·018	6	0·014	14	0·035	1	0·030	41	0·034	4	11°	6	0·032	4	0·017
6	0·030	11	0·050	15	0·022	24	0·015	4	0·040	41	0·032	1	0·052	24	11°	4	0·035	6	0·017
7	0·032	6	0·050	6	0·026	15	0·019	8	0·050	32	0·042	32	0·055	15	12°	13	0·042	13	0·024
8	0·035	10	0·055	5	0·027	5	0·020	7	0·055	8	0·050	8	0·072	11	12°	14	0·054	11	0·029
9	0·035	7	0·065	8	0·032	11	0·026	10	0·060	14	0·066	14	0·074	5	13°	11	0·056	14	0·035
10	0·035	12	0·070	16	0·035	25	0·030	11	0·060	18	0·070	40	0·075	2	14°	16	0·057	17	0·036
11	0·035	17	0·070	11	0·035	17	0·030	15	0·060	7	0·075	7	0·075	49	20°	17	0·061	15	0·036
12	0·040	15	0·075	10	0·038	10	0·035	17	0·065	37	0·080	18	0·085	47	21°	18	0·062	12	0·036
13	0·040	16	0·090	7	0·040	7	0·040	18	0·070	48	0·090	48	0·092	12	23°	5	0·063	16	0·037
14	0·045	13	0·100	35	0·040	16	0·042	2	0·070	12	0·095	37	0·093	17	24°	15	0·064	37	0·040
15	0·045	20	0·100	1	0·042	35	0·045	13	0·075	2	0·100	12	0·094	22	26°	12	0·075	18	0·042

Wetters used:

1. Sodium 4-ethyloctyldiethylpropyl sulphate
2. Hexadecylbetaine
3. Sodium dinonyl sulphosuccinate
4. Octylphenol-polyethylene glycol (8½E)
5. Didecyldimethylammonium bromide
6. Sodium di-n-octyl sulphosuccinate
7. Sodium di-(2-ethylhexyl) sulphosuccinate
8. Sodium dinonyl phosphate
9. Oleylamine polyethylene glycol (9E)
10. Dodecylaminopropionic acid
11. Iso-octyl-*o*-cresol polyethylene glycol (10E)
12. Sodium dodecylaminopropionate
13. Sodium dodecylbenzenesulphonate
14. Nonylphenol-polyethylene glycol (8E)
15. Nonylphenol-polyethylene glycol (10E)
16. Nonyphenol-polyethylene glycol (11E)
17. Tridecyl-polyethylene glycol (12E)
18. Cyclohexylamine dodecyl sulphate
20. Dodecylammonium (chloride) propionic acid
22. Oleylamine polyethylene glycol (6E)
24. Dodecyldiethanolamide
25. Iso-octyl-*o*-cresol-polyethylene glycol (8E)
32. Monoethanolamine oleate
35. Hexadecyldimethylethylammonium ethyl sulphate
37. Nonylphenol-polyethylene glycol (6E)
40. Sodium oleate
41. Morpholine oleate
47. Oleylamine-polyethylene glycol (5E)
48. Potassium oleate
49. 1-Hydroxyethyl-2-heptadecenylimidazolidine

Table II

Coefficients of rank correlation of the order of efficiency of wetters on various surfaces as given by visual assessment

	τ_T	τ_{AN}	τ_T	τ_{AN}	τ_T	τ_{AN}	τ_T	τ_{AN}
Apple leaf	0·62	0·66	0·51	0·60	0·72	0·82	0·43	0·40
	Blackcurrant leaf		0·53	0·58	0·79	0·79	0·51	0·45
			Banana leaf		0·60	0·62	0·52	0·50
					Plum leaf		0·52	0·47
							Beeswax	

results in each column are far from constant and it may be concluded that these properties bear no direct relation to the wetting of the surfaces.

Evans & Martin[3] have shown that wetting should be related to θ_R rather than to θ_A and it was noted that the value of θ_R was zero for each of the solutions used for the results in Table III. Therefore, the values of θ_R were measured for the full range of 65 wetters on banana and beeswax surfaces and the order of efficiency of the wetters was listed in terms of the minimum concentration required to reduce θ_R to zero on the appropriate surface. The first 15 wetters in these lists are shown in Table I, columns 4 and 7.

Table III

Values of the surface tensions (γ), advancing contact angles (θ_A), and spreading coefficients (W) of the wetter solutions listed in Table I, each containing the minimum wetting concentration for banana leaves and beeswax surfaces

	Banana leaves				Beeswax		
Wetter number	γ, dynes/cm.	θ_A	W, dynes/cm.	Wetter number	γ, dynes/cm.	θ_A	W, dynes/cm.
4	37	61°	−19	6	36	33°	−6
24	44	91°	−46	3	42	53°	−17
14	38	40°	−9	17	37	62°	−20
37	33	61°	−17	5	35	61°	−18
3	29	62°	−15	1	41	73°	−29
15	32	44°	−9	41	39	69°	−25
6	31	19°	−2	32	43	68°	−27
5	32	45°	−9	8	31	69°	−20
8	32	83°	−28	14	41	37°	−8
16	32	44°	−9	18	35	64°	−20
11	31	10°	−1	7	28	46°	−9
10	29	31°	−4	37	30	54°	−12
7	30	45°	−9	48	34	57°	−16
35	37	85°	−34	12	28	47°	−9
1	36	55°	−15	2	31	68°	−19

Comparison of columns 3 with 4 and 6 with 7 in Table I shows that the wetters appear in a similar order and that similar concentrations are required to produce complete wetting and to reduce θ_R to zero. In fact, good correlation between complete wetting and $\theta_R = 0$ was found with all 65 wetters, as shown by the high τ values in the first line of Table IV.

Other possible ways of listing the efficiency of wetters based on values of θ_R have been tried using the beeswax surface. These have included a classification based on the concentration of wetter required to reduce θ_R to 20°, a classification based on the value of θ_R at wetter concentrations of 0·01% (section 8, Table I) and 0·05%. None of these methods shows good correlation with actual beeswax wetting (see Table IV).

Because the complete wetting of surfaces is found when $\theta_R = 0$, the value of θ_E will be given by $\cos\theta_E = (1 + \cos\theta_A)/2$. Thus the values of θ_E show similar variations to those of θ_A listed in Table III, and they are of no use for the assessment of complete wetting.

Table IV

Correlation of methods based on θ_R with visual wetting assessment

Method of listing wetters	Beeswax surface		Banana leaf	
	τ_T	τ_{AN}	τ_T	τ_{AN}
Concn. for $\theta_R = 0$	0·96	0·96	0·89	0·89
Concn. for $\theta_R = 20°$	0·55	0·51		
Value of θ_R at concn. 0·01%	0·41	0·48		
Value of θ_R at concn. 0·05%	0·60	0·56		

The spreading coefficient based on θ_R will be zero when $\theta_R = 0$. Thus the correlation of this property with visual wetting is the same as that calculated for $\theta_R = 0$ in Table IV. There is, however, little point in using the spreading coefficient when the value of θ_R alone is sufficient.

Although the extreme condition of complete wetting is governed by the condition that θ_R should be zero, the value of θ_R has been found to be of no use as a guide to the extent of the incomplete wetting of surfaces; the reasons for this are discussed later.

(c) *Tape sinking method*

This method was used to list the 65 wetters in order of their concentration required to sink the standard tape in 15 sec. and in 30 sec. The first 15 wetters in these lists are given in Table I, sections 9 and 10; the complete lists have been correlated with the lists from the visual tests and the coefficients are shown in Table V.

Table V

Correlation coefficients of the tape-sinking method with the visual assessment of wetting

Visual wetting list	List based on the wetter concentration required for tape-sinking time of			
	15 sec.		30 sec.	
	τ_T	τ_{AN}	τ_T	τ_{AN}
Apple leaf	0·59	0·80	0·68	0·86
Blackcurrant leaf	0·55	0·58	0·62	0·65
Banana leaf	0·57	0·53	0·68	0·55
Plum leaf	0·68	0·75	0·72	0·79
Beeswax	0·51	0·39	0·52	0·38

τ_T at a sinking time of 15 sec. shows that this test gives poor correlation with the actual wetting of these surfaces; some improvement was shown in τ_T with sinking time of 30 sec. Ashworth & Lloyd[2] suggested that this method might not be suitable for cationic wetters and, in fact, when the cationic materials were ignored, τ_{AN} showed an improvement on certain surfaces, notably apple and plum. The 30-sec. test agreed well with the wetting of apple leaves but, as shown in Table I, this correlation is limited to predicting approximately the correct order of wetting ability; the actual wetter concentration required to sink the tape in 30 sec. was considerably lower than that required to wet apple leaves. The tape-sinking test was of little value in predicting the correct order of efficiency of wetters on blackcurrant and banana leaves and it was not improved by any further variation of the sinking time; the values of τ_{AN} for sinking times of 10 and 60 sec. compared with banana leaf wetting are 0·55 and 0·65 respectively.

When the tape-sinking times for a range of concentrations of individual wetters were plotted on a log normal scale against the wetting produced by these solutions on various leaf surfaces, a straight-line relationship was found (Fig. 2). Such a relationship does not necessarily mean that intermediate degrees of wetting are related to the tape-sinking time but may indicate only that each property is varying in a similar way with the wetter concentration. If this is the case, the plots produced by different wetters on the same surface will not be superimposed and this is shown in Fig. 3 where the tape-sinking times for various concentrations of several materials have been plotted against the wetting produced on the under surfaces of banana and apple leaves. The tape-sinking times show no correlation with banana leaf wetting (Fig. 3a), but they do show a rough correlation with apple leaf wetting (Fig. 3b) in that most of the points lie within the pair of dotted lines, and the results lying outside these lines are mainly those obtained with cationic wetters. These correlations are similar to those found in the tests for complete wetting.

Table I shows that most of the best 15 wetters given by the 15-sec. tape-sinking test are also included amongst the best 15 wetters on all four leaf types. Thus the tape-sinking test does provide a rough classification of those wetters that are likely to be 'good' on many leaf surfaces and it will predict roughly the order of efficiency of anionic and non-ionic wetters on certain leaf surfaces.

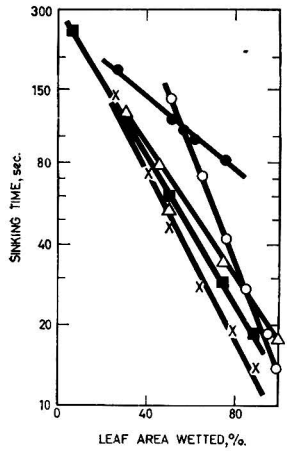

FIG. 2.—*Relationship between tape-sinking time and leaf wetting for various wetting agents*

○ iso-octyl-*o*-cresol-polyethylene glycol (5E)—apple leaf under surface
● sorbitan mono-oleate-polyethylene glycol (20E)—apple leaf under surface
× iso-octyl-*o*-cresol-polyethylene glycol (5E)—blackcurrant leaf under surface
△ dodecyl betaine—plum leaf, under surface
■ di(decylaceto) dimethylammonium bromide—banana leaf, under surface

Theory of wetting

The upper limit of wetting is found when the receding angle is zero and, theoretically, once the receding angle is finite, the liquid should drain completely from the surface, resulting in zero wetting. In practice zero wetting does not occur under these circumstances for two reasons. Firstly, a leaf is irregular in surface contour so that liquid may collect in the depressions and, secondly, a solid surface is seldom perfectly uniform in surface properties so that liquid retracts more readily from certain areas than from other areas. This results in the liquid film breaking up into discrete droplets which may be too small to run down the surface. The condition for a liquid drop to remain stationary on a solid surface is given by :[16]

$$mg \sin \alpha < w\gamma_{AL}(\cos \theta_R - \cos \theta_A) \quad . \quad . \quad . \quad . \quad (1)$$

where m is the mass of the drop,

g is the acceleration due to gravity,

α is the angle of inclination to the horizontal of the surface,

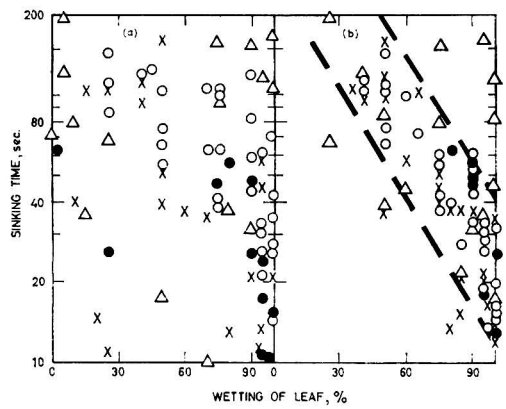

FIG. 3.—*Relationship between tape-sinking time and wetting of the under surface of (a) banana leaves, (b) apple leaves, by various concentrations of wetting agents*

● anionic wetters
○ non-ionic wetters
△ cationic wetters
(25 wetters in all)

w is the width of the drop measured in the plane of the surface, at right angles to the line of most probable movement,

γ_{AL} is the air/liquid surface tension.

This equation cannot be used to predict wetting because the value of m is unknown and variable. However, the equation does fix the lower limit of wetting which will occur when $\theta_A = \theta_R > 0$; under this condition m must be zero if α is finite and no liquid can remain on the surface. (The special case of $\theta_A = \theta_R = 0$ represents the extreme case of wetting and spreading when the whole surface is covered by a very thin film of liquid having negligible mass.)

A further complication in the measurement of intermediate degrees of wetting is introduced by the variation of w in Equation (1) with the impaction velocity of the spray droplets.[16] This effect has been illustrated by Ashworth & Lloyd[2] by comparing the wetting of cabbage leaves produced by dipping them in wetter solution with that produced by spraying the leaves under standard conditions with the same wetter solutions to beyond run-off. The dipping test, with no impaction complications, showed consistently lower degrees of wetting than were found in the spraying test until the wetter concentration was increased sufficiently to cause complete wetting of the surface.

Thus complete wetting of a surface by high-volume sprays occurs when θ_R is zero and complete non-wetting when θ_R and θ_A are equal and finite. Between these limits the wetting is governed by a function of both the advancing and receding angles. This function is not a constant but varies with the spray impaction velocity and with the actual values of the contact angles, becoming more dependent on the receding angle as that angle approaches zero. This effect is similar to that described in connexion with the retention of sprays.[16]

Conclusions

It is impossible to devise a fully satisfactory test for the wetting efficiency of sprays that does not involve the appropriate spray target surface and also take into account the effects of spray droplet impaction. These requirements are satisfied by a test based on the visual assessment of wetting under practical spraying conditions. However, a subjective test of this type is open to criticism in terms of observer bias and inaccuracy; these aspects will be considered in the next paper in this series.

None of the other wetting tests considered in this work can be accepted as fully satisfactory for the measurement of the wetting efficiency of high-volume sprays, except in certain limited cases.

The limiting case of complete wetting of the surface, which is not affected by spray impaction velocity, may be recognised when the receding contact angle is zero.

The tape-sinking test can be used as a guide to the wetting of certain leaf types by anionic and non-ionic systems, and it also provides a rough classification of the overall efficiency of wetters on leaf surfaces. Such a classification is far from perfect but it may provide a useful starting point in the selection of wetters for any specific spray application.

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PHYSICO-CHEMICAL STUDIES ON AGRICULTURAL SPRAYS. VII.*—The Visual Assessment of Spray Coverage

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The wetting ability of spray formulations is best assessed by a visual examination of the sprayed surfaces, but such a method is open to criticism in terms of observer bias and inaccuracy. By use of a panel of observers, the method has been examined to determine the magnitude of such errors and to find means of minimising them.

Inexperienced observers showed considerable underestimation in determining the proportional areas of leaf surfaces wetted, particularly over the centre of the wetting range (30–80% of surface area wetted). As expected, the accuracy was improved by the use of charts and of a system of wetting classification that widened the assessment steps over the centre of the range.

By including fluorescent tracers in the wetter solutions and photographing the wetted leaves under ultra-violet light, the wetting was assessed also in terms of black areas on a white background. This proved to be much more accurate than direct leaf assessment and, although it cannot be recommended as a general method for wetting assessment, it is valuable for training observers by illustrating their errors.

Introduction

Visual assessment is the most commonly used method of examining spray coverage and, as shown in the preceding paper,[1] it is the most suitable method for measuring the wetting behaviour of sprays. The main drawback in the method is in obtaining consistent and reliable results over the whole wetting range. The possible errors arise from two main causes. Firstly, the difficulty in distinguishing those areas of the surface that are covered with a thin film of liquid from those areas that are not so covered. Secondly, the observer bias which causes some observers to give consistently high or low assessments, even when the wetted area is readily distinguishable. The purpose of the work described in this paper was to examine the relative importance of these errors and to determine how they could be minimised.

Experimental

The wetting assessments were made on the under surface of laurel leaves. Solutions were prepared containing various concentrations of wetters to provide a range of wetting from 10% to 100% on these leaves.

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The wetters used were oleylamine condensed with approximately 6 moles of ethylene oxide, and sodium dodecyl sulphate. All the solutions also contained a fluorescent dye, 0·1% wt. Primuline A150 combined with 0·1% wt. polyvinyl alcohol (grade Alcotex 99/H). This combination has been suggested by Staniland[2] for tracing spray liquids.

A panel of nine observers having little or no experience in this type of assessment was divided into three groups of three people. Between 40 and 50 leaves were taken for each group and each leaf was dipped into one of the wetter solutions. The wetter solutions were chosen at random and the observers had no idea which leaf was dipped into which wetter solution. After dipping, the leaves were allowed to drain for a few seconds and then passed to the three observers of one group who assessed individually the proportional area of the leaf surface that was covered by solution. The leaves were then placed under an ultra-violet lamp and photographed, the fluorescent dye in solution showing clearly the whole area of the leaf surface that was covered by liquid. After processing the film, the negatives were projected on to a suitably ruled screen, where the area of leaf wetted could be measured accurately; these figures were then compared with the results as assessed by the group. The process was repeated for each of the three groups comprising the complete panel and repeated again for each of three assessment methods that were tried.

The methods used for assessing the wetted area of the leaves were as follows:

Method I.L: estimating to the nearest 10% of the total leaf area, i.e., listing the result as the nearest figure in the range 0–100% wetted.

Method II.L: estimating to the nearest figure in the range 0, 5, 10, 25, 50, 75, 90, 95 or 100% wetted.

Method III.L: estimating according to the same range as Method II but with the assistance of a chart illustrating the limits of these wetting ranges (Fig. 1).

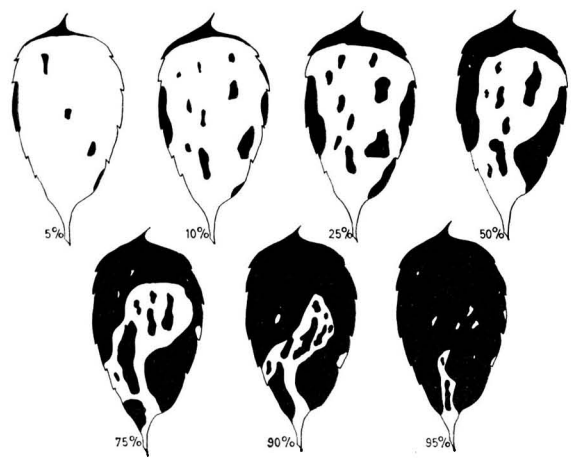

FIG. 1.—*Standard wetting chart for laurel leaves*

Once the leaf assessments were complete, the nine observers were asked individually to assess the wetting from the negatives when projected on to a plain screen, using the same three methods (designated as methods I.N, II.N and III.N).

Results and discussion

Figs. 2a and 2b show the average assessment error plotted against degree of wetting for two of the observers using Method I, and the curves illustrate the two main types of error found with all the observers. In fact, six observers produced error curves similar to those shown in Fig. 2a, whilst the remaining three produced error curves similar to those in Fig. 2b. In both

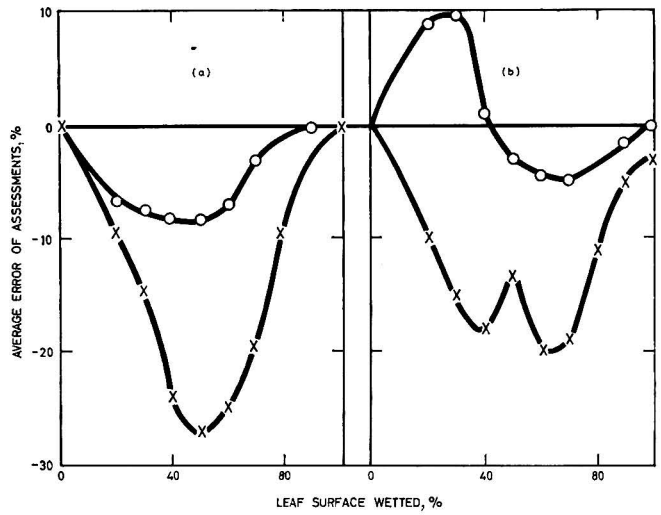

FIG. 2.—*Variation of average assessment error with degree of wetting*
(a) Observer No. 2 (b) Observer No. 6
○ assessments by method I.N
× „ „ I.L

cases the error varies considerably with the degree of wetting and this is further illustrated in Table I where the combined results of all nine observers are collated to show the extent of the over- and under-estimation found by methods I.L and I.N.

Table I

Variation of assessment error with degree of wetting

Method I.L

Correct wetting (%)	Percentage number of results estimated as : 10%	20%	30%	40%	50%	60%	70%	80%	90%	100%	No. of estimations
20	72	*28*									21
30	48	32	*16*	4							48
40	18	45	18	*8*	10						69
50	6	33	26	26	*4*	4	2				60
60		17	22	28	13	*20*					39
70			7	27	33	19	*7*	*7*			27
80					5	14	43	*33*	5		21
90								25	*67*	8	24
100									48	*52*	27

Total number 336

20·5% of total number were estimated correctly, 74·4% were underestimated, 5·1% were overestimated

Method I.N

20	52	*40*	—	4	4						27
30	3	32	*38*	14	11	2					63
40	1	11	32	*38*	17	1					90
50		1	18	38	*32*	11					99
60				19	48	*33*					27
70					4	8	*44*	44			27
80						5	20	*45*	30		18
90								11	*78*	11	9
100									16	*84*	36

Total number 396

42·1% of total number were estimated correctly, 41·4% were underestimated, 16·5% were overestimated

The results in Fig. 2 and Table I show clearly that it is much easier to estimate proportional areas from the black-and-white picture as given by the photographs than it is to estimate the wet and dry areas direct from the leaf. The latter may be done only by the light reflected from the wet surface and so the observation must be carried out with the light source, the object and the eye in a certain position relative to one another. With an irregular surface such as a leaf, one viewing position only shows light reflected from those areas of the surface that are correctly orientated to the incident light and the leaf has to be viewed from several positions to enable light to be reflected in turn from all points on the leaf surface. This means that the observer never sees the complete picture of the wetting at any one time and consequently the leaf results tend to be seriously underestimated. The photographs provide a complete picture of the wetting and the underestimation is not so pronounced.

Most of the observers showed a maximum error over the centre of the wetting range (see Fig. 2a) when estimating direct from leaves. The same effect was found to a lesser extent on the negative assessments except for three observers who produced error curves of the type shown in Fig. 2b where the low wetting is overestimated and the high results are underestimated. As expected, both these types of error are minimised by using the assessment range in Methods II and III where the 50% value is retained but the overall number of assessment values in the centre of the wetting range is reduced. This is shown in Table II which lists the results of all nine observers using Methods II and III.

The results show that Method II gives a considerably greater proportion of correct assessments than Method I, and the use of the wetting chart in Method III produces a further improvement in accuracy.

Tables I and II show the variation between the methods in terms of the overall results of all the observers, but equally important is the variation in any one method between the different observers. This is shown in Table III where the results for each observer are listed to show their arithmetic mean error and the variance over all the assessments using each method.

As expected, a very considerable difference in the results was found between observers using Method I. The mean error varied widely in both the leaf and the negative assessments but many of the observers were remarkably consistent as shown by the low variance results. This was particularly noticeable with Method I.N where some observers, notably Nos. 2, 4, 7 and 9, were seriously underestimating the wetting but their bias was consistent. Observers 1, 6 and 8, who showed least consistency in their results, produced assessment curves of the type illustrated in Fig. 2b.

Despite the fact that Tables I and II show that method II.L was more accurate than Method I.L, a majority of the observers found method II.L to be less satisfactory than Method I.L in terms of both mean error and variance (Table III). The increase in mean error shown by many observers using Method II.L is due to the 25% assessment steps in the centre of the wetting range in Method II, where a mistake of one place in the assessment produces a greater error than a mistake of two places with the 10% steps used in Method I. This effect is not so noticeable on the assessments from the negatives and most observers gave a smaller mean error with Method II.N than with Method I.N.

The use of the chart in Method III.L produced a reduction in the mean error with almost all the observers, and the results obtained with Method III.N were extremely accurate.

To illustrate the extent to which training and experience in this type of assessment can affect the accuracy of the results, six of the observers were put through a repeat test using Method III. Before carrying out the assessments, they were shown their previous results and with the aid of the photographs used in the N series of tests, their individual errors were illustrated. The results of this repeat test are given in the last section of Table III. By comparison with the results of the previous tests, their assessments were extremely accurate and reasonably consistent. The assessments from the negatives were almost faultless, and experience would further improve the leaf assessments.

Conclusions

The visual assessment of the wetting of leaves is difficult and can be extremely inaccurate. The errors are enhanced by the difficulty in distinguishing the wetted areas from the non-wetted

Table II

Variation of assessment error with degree of wetting

Method II.L

Correct wetting (%)	Percentage number of results estimated as: 5%	10%	25%	50%	75%	90%	95%	100%	No. of estimations
25	18	44	*38*						81
50	3	15	48	*33*	1				135
75			6	67	*27*				33
90				13	12	*25*	41	9	39
95						34	*60*	6	33
100						25	33	*42*	27
								Total number	348

36·2% of total number were estimated correctly, 57·5% were underestimated, 6·3% were overestimated

Method II.N

25		14	*73*	13					114
50			15	*62*	23				162
75				28	*72*				48
90					16	*84*			24
95					6	61	*33*		33
100							33	*67*	33
								Total number	414

65·3% of total number were estimated correctly, 21·7% were underestimated, 13·0% were overestimated

Method III.L

25	3	40	*54*	3					78
50		4	51	*40*	5				129
75				62	*38*				39
90				4	33	*56*	7		42
95						*33*	*67*		30
100						7	67	*26*	27
								Total number	345

46·4% of total number were estimated correctly, 50·7% were underestimated, 2·9% were overestimated

Method III.N

10		*56*	44						18
25		21	*65*	14					78
50			10	*76*	14				117
75				26	*71*	3			66
90					6	*94*			42
95						18	*82*		30
100							33	*67*	36
								Total number	387

73·6% of total number were estimated correctly, 16·8% were underestimated, 9·6% were overestimated

areas of the leaf and they are worst over the centre of the wetting range. As expected, the overall accuracy is improved by using fewer assessment steps over the centre of the range than are used at the extremes of wetting and by using a chart to illustrate the various wetting assessment steps. The difficulty in distinguishing the wetted from the non-wetted areas can be removed by incorporating fluorescent materials in the wetting liquid and making the assessments under ultra-violet light or by photographing the leaves under ultra-violet light and making the assessments from the negatives. This method has serious limitations because the addition of fluorescent tracers to a spray liquid will modify its wetting properties. However, the fluorescent tracer method, used in conjunction with direct leaf assessments, is valuable for training observers by illustrating their errors. After such a training, and with further experience, the visual assessments of all degrees of leaf wetting can be reasonably accurate.

Acknowledgments

The authors would like to thank Prof. Kearns for his support of this work and the members

Table III

Variation in error between observers

Observer	Method I.L			Method I.N		
	No. of assessments	Arith. mean error, %	Variance	No. of assessments	Arith. mean error, %	Variance
1	44	−19·12	87·08	44	−6·18	84·94
2	44	−21·76	79·58	44	−7·35	38·24
3	44	−10·00	78·79	44	+3·53	53·83
4	34	−11·25	194·02	44	−11·18	59·18
5	34	−2·50	106·52	44	+2·65	56·42
6	34	−13·75	111·41	44	+1·76	130·12
7	34	−16·25	207·07	44	−12·94	45·63
8	34	−12·92	186·78	44	+0·29	93·85
9	34	−16·25	189·67	44	−6·18	48·57
Complete panel	336	−14·15		396	−3·96	
	Method II.L			Method II.N		
1	46	−10·97	240·71	46	−3·06	190·40
2	46	−9·58	163·39	46	−2·92	87·68
3	46	−7·50	150·71	46	+3·89	128·73
4	36	−16·15	274·62	46	+1·11	107·30
5	36	−8·46	153·54	46	+2·22	109·21
6	36	−18·46	231·54	46	+4·31	108·79
7	34	−19·17	179·71	46	−8·75	217·68
8	34	−17·29	208·65	46	+8·19	194·50
9	34	−16·46	194·52	46	−1·25	166·25
Complete panel	348	−13·18		414	+0·42	
	Method III.L			Method III.N		
1	43	−10·45	128·69	43	−5·91	136·65
2	43	−5·76	140·81	43	−2·42	75·19
3	43	−8·03	178·03	43	+3·18	101·28
4	38	−8·75	238·19	43	−0·76	139·25
5	38	−11·07	265·21	43	+4·24	139·25
6	38	−11·25	134·49	43	+1·21	151·61
7	34	−8·96	197·78	43	−2·73	112·64
8	34	−10·00	204·35	43	−0·15	111·70
9	34	−10·63	226·77	43	+0·61	73·06
Complete panel	345	−9·33		387	−0·30	
	Method III.L (repeat)			Method III.N (repeat)		
1	40	−6·45	108·23	40	−1·84	104·31
3	40	−6·10	118·64	40	+1·42	89·32
4	40	−4·21	148·51	40	−0·94	74·68
5	38	−7·25	114·38	38	+2·82	82·30
7	38	−9·12	134·32	38	−2·94	101·68
8	38	−7·38	144·20	38	+1·12	84·71
Complete panel	234	−6·75		234	−0·06	

of the panel for their co-operation. The panel consisted of: Miss G. Browning, Miss D. Greenhow, Miss C. Hales, Miss M. Holgate, Miss A. Knight, Miss M. Mealing, Mr. A. Baker, Mr. G. M. Clarke and Mr. T. Cox.

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STORAGE LIFE OF VACUUM-PACKED ICED TROUT. I.—Influence of Packing Material

By POUL HANSEN and B. V. JØRGENSEN

The storage life of gutted trout vacuum-packed individually in polyamide or polyethylene bags and kept in wet ice exceeded 2 weeks. The trout packed in polyethylene showed signs of slight fat oxidation at the end of the second week, while that in polyamide showed no fat oxidation throughout the experimental storage period of 3 weeks.

Trout in both types of package suffered microbial spoilage during the third week. Viable aerobic counts throughout the storage period were higher in polyethylene- than in polyamide-packed trout.

Introduction

Most of the trout produced in Danish trout farms are packed in ice or are frozen for refrigerated transport and distribution abroad. In both cases serious degradation in quality may occur during the refrigeration period if the trout are gutted before being packed and the package allows access of air to the belly wall surfaces of the fish. Vacuum-packing of the gutted trout before freezing and frozen storage will delay the fat oxidation considerably and thus increase the storage life as shown in recent experiments.[1]

During storage of gutted trout in ice both fat oxidation and microbial deterioration are possible causes of quality degradations. Recent experiments[2, 3] with gutted trout ice-packed with access of air to the fish have shown that oxidation of fat usually will be the limiting factor of keeping quality. Under these conditions the induction period of peroxide value will rarely exceed 4 days and the subsequent fat oxidation may proceed rapidly and in some cases limit the storage life to as little as 1 week.

The present experiment is designed to test the microbial deterioration and the fat oxidation of vacuum-packed gutted trout packed in wet ice and to determine the limiting factor of keeping quality.

The vacuum-packing was carried out on a commercial type packing machine which evacuates and seals plastic bags in which the fish are laid one in each bag. The plastic bags were either of polyamide or polyethylene, both of a foil thickness of 0·04 mm.

Experimental

The fish tested were rainbow trout (*Salmo irideus*) of live weight about 200 g. which had been raised at a commercial trout farm on Sealand. During the last 6 days before the experiment they were kept without food in fresh water at 15°. They were taken out of the water on 4 February, 1964. When dead they were gutted and the gills and kidney blood were removed. The fish were then washed in fresh water and within 2 h. of death vacuum-packed and buried in wet flake ice.

A sample of six fish was taken from each group after 1, 7, 10, 14, 17 and 21 days of storage. When unpacked the edible part including the skin of each fish was divided into three parts, the tail part of one fillet being used for microbiological examination without any surface wipe or cleaning, the remainder of this fillet for organoleptic tests and the other entire fillet for determination of peroxide value.

Organoleptic tests

The six samples per group were cooked in a casserole by immersion for 15 min. in warm water at 90° containing 2% of salt and 0·02% of acetic acid. The warm samples were served under code to a panel of six tasters, each of whom tasted one sample from each group. The tasters were asked to pay particular attention to the meats of the middle belly wall, and to give an eating quality score on each sample and indicate any of the following defects if found : discoloration, stale or rancid taste, other off-flavour, soft or sloppy texture, dry or tough texture. On the 21st day the samples were judged on appearance and odour only, the quality score of this day presenting odour only.

Peroxide values

The above mentioned 6 fillets per group were divided into two sub-samples each of 3 fillets, which were minced together and extracted according to the method suggested by Tollenaar.[4] The peroxide values were determined on these extracts by Lea's method.[5]

Microbiological examination

After homogenisation of 10 g. of fillet with 90 ml. of physiological salt solution in a Waring Blendor, decimal dilutions were made from the suspension. Inoculations were carried out simultaneously in plate count agar (Difco) and sulphite–iron media,[6] respectively, for determining the total aerobic count and the presence of sulphite-reducing clostridia. The total aerobic count was determined after incubation at 25° for 4 days, and the sulphite-reducing clostridia after incubation at 30° for 2 days.

Results

Measurable peroxide values were not found until the 21st day, when the polyamide subgroups showed 0 and 2·2 and the polyethylene subgroups 8·7 and 12·2 mequiv./kg. of fat respectively. Peroxide determinations of the polyethylene-packed fish showed traces, however, on the 14th and 17th day of storage.

The total viable aerobic counts were a few hundreds per g. for both groups after 1 day of storage and increased to more than 10×10^6 in the polyethylene group and to about 1×10^6 in the polyamide group on the 21st day. The average logarithms of counts of both groups increased nearly linearly with time as shown in Fig. 1. The difference between groups was significant during the third week of storage. The counts of sulphite-reducing clostridia in all samples were below 100 per g.

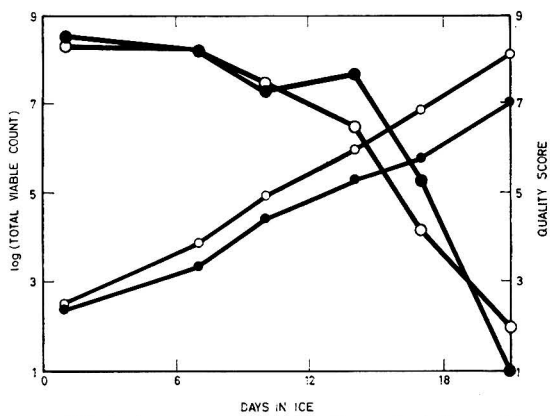

FIG. 1.—*Effect of storage on eating quality and viable aerobic count*
● Polyamide-packed trout
○ Polyethylene-packed trout

Fig. 1 also shows the averages of eating quality scores of the groups. The polyamide-packed fish retained good quality during the first 2 weeks of storage and showed a drastic fall in quality during the third week, the fish being inedible on the 21st day. On the 14th and 17th days of storage the polyethylene-packed fish showed a lower quality than the polyamide-packed fish, the difference presumably being associated with the occurrence of slight rancidity in the former. On the 17th day all six samples of the former fish and only one sample of the latter fish were noted to have rancid or stale taste. Differences in eating quality of the groups, however, did not exceed about 1 score unit and were not significant.

It should be noted that all samples on removal from the bags on the 17th and 21st day had a definite spoilt odour and at the same time the appearance of the fish was unappetising due to the milkiness of the slime.

Discussion

All samples showed low counts of sulphite-reducing clostridia throughout the 3 weeks of storage. The experiment thus does not indicate any risk of botulism from the package types involved.

The vacuum-packed trout were fully edible during the first 2 weeks of storage in ice and showed definite spoilage during the third week, although cooked samples were still acceptable on the 17th day. Microbial activity caused spoilage in both types of package during the third week of storage while with the polyethylene packing fat oxidation also seemed to be involved.

Differences in bacterial growth and fat oxidation of groups noted in the third week of storage may be associated with the difference in gas permeability of the two types of package material used. The trout packed in the more permeable polyethylene bags showed an about tenfold higher total aerobic count than the polyamide-packed trout at the end of the experiment. At the same time the former trout showed an average peroxide value of about 10 and the latter an average of about 1 mequiv./kg. of fat.

It should be noted that on the 17th day when the polyethylene-packed fish showed only traces of peroxides, all tasters found slight rancid or stale taste, in most cases accompanied by slight yellow-brownish discolorations of the belly flap. This indicates that the peroxide value may not be useful as an indicator of initial fat oxidation of such products.

The experiment indicates that polyethylene and polyamide bags are about equally suitable for ice storage of vacuum-packed trout up to about 10 days. For longer storage the latter material should be preferred.

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OXYGEN DIFFUSION AND AEROBIC RESPIRATION IN COLUMNS OF FINE SOIL CRUMBS

By D. J. GREENWOOD and D. GOODMAN

Experiments are described which support the validity of an equation defining the extent to which microbial aerobic respiration in columns of fine soil crumbs is restricted by diffusion in the gas phase between the crumbs.

Columns, exposed at one of their ends to air, but closed at the other, were prepared from sieved fractions of soil crumbs. Different aeration treatments were imposed by water-saturation followed by draining to different extents. Since the respiration rate of soil micro-organisms is unaffected by lowering the oxygen concentration down to approx. 3 μM, the respiration rates in such columns are proportional to the lengths of the zones having oxygen concentrations greater than 3μM and are thus aerobic. The equation was tested, therefore, by determining the lengths of these aerobic zones by two different methods (one based on the equation and one not) described in detail. The mean lengths determined by these methods were 4·3 cm. and 3·7 cm., respectively.

Introduction

Previous work[1] has shown that, despite the complex nature of microbial activities in soil, a reasonable approximation to the effect of oxygen concentration on aerobic respiration can be obtained by considering that the respiration rate is unaffected by oxygen concentrations greater than the very low value of 3μM and does not take place at all at lower oxygen concentrations. Thus the aeration status of a soil, as far as respiration is concerned, can be defined approximately in terms of the proportion of soil having an oxygen concentration greater than 3 μM and is thus, effectively, an aerobic zone.

Low oxygen concentrations in moist soil crumbs contained in columns which are open to the atmosphere at one end but closed at the other, can result either from inadequate diffusion in the gas space between crumbs or from inadequate diffusion in the aqueous phase within crumbs. If the crumbs are minute, the oxygen concentration at their surfaces will be only slightly greater than that at their centres and hence low oxygen concentrations in columns of minute crumbs could only result from inadequate diffusion in the gas phase between the crumbs.

On this basis, by considering oxygen diffusion in the gas phase, and by letting x be the depth from the surface of the column, it can be assumed that

$$D_x.\partial^2 C/\partial x^2 = Q \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

$$E_x = E_0(1 - ax) \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

$$D_x/D' = (E_x/E')^4 \quad \text{(reference [2])} \quad . \quad . \quad . \quad . \quad (3)$$

with boundary conditions

$$C = C_0 \quad \text{at} \quad x = 0$$
$$C = 0 \quad \text{and} \quad \partial C/\partial x = 0 \quad \text{at} \quad x = l$$

The symbols are as follows : C is the oxygen concentration (ml. of oxygen per ml. of gas phase) ; D_x is the diffusion coefficient of oxygen through soil at depth x ; E_x is the volume of gas space per unit volume of soil also at depth x ; l is the length of the aerobic zone ; a is a constant ; Q is the oxygen uptake in ml. per sec. per ml. of soil when oxygen is not limiting ; and E' and D' are the gas-filled pore space of, and the coefficient of diffusion of oxygen through, columns which have been water-saturated and then drained until they contain no inter-crumb water. It is assumed that D' and E' are independent of x.

From Equations (1), (2) and (3) and the stated boundary conditions it may be deduced that

$$\frac{3a^2l^2 - a^3l^3}{(1 - al)^3} = \frac{6D'C_0a^2(E_0)^4}{Q(E')^4} \quad . \quad . \quad . \quad . \quad . \quad (4)$$

which may be conveniently solved numerically by setting $u = al$ and writing

$$\frac{3u^2 - u^3}{(1 - u)^3} = \frac{6D'C_0a^2(E_0)^4}{Q(E')^4} \quad . \quad . \quad . \quad . \quad . \quad (5)$$

The purpose of the work described in this paper was to test the validity of Equation (4). Experiments are described in which the lengths of aerobic zones in columns of fine crumbs were obtained by two entirely different methods ; one was based on the equation and the other was not.

Experimental

Soil

Soil A described previously[1] was used throughout. Before use it was air-dried and sieved between the limits of : 0·18 and 0·42 mm., 0·42 and 0·59 mm., 0·59 and 0·84 mm., 0·84 and 1·68 mm., and 1·68 and 3·36 mm.

Water content

This was determined as the loss in weight of soil on being dried in an oven at 100° for 24 h. All values are expressed as percentage of oven-dry weight.

Volumes of water and solid material in soil columns

These were calculated from the loss in weight when the moist soil and air-dry soil were heated

at 100° for 24 h. and from the specific gravity of the air-dry soil. The specific gravity was determined, by pycnometer, as described previously on soil samples which had been wetted *in vacuo*.[1] In the calculations it was assumed that the loss in weight on heating was caused by loss of water only, and that the volume of moist soil was equal to the total volume of water plus that of the oven-dry soil.

Water content of soil crumbs held under a suction of 50 cm. of water

Soil crumbs (5 g.) were spread evenly over the muslin base of a cylinder (7 cm. dia. and 0·5 cm. high). The soils were wetted *in vacuo*, the cylinders were placed on a tension plate, subjected to a suction of 50 cm. of water, covered with a beaker to prevent evaporation and allowed to equilibrate for 14 h. before the water content of the soil was determined.

Diffusion coefficient of carbon tetrachloride through air and through soil crumbs

These were determined by the method of Penman[3] which was based on measuring the rate of evaporation of carbon tetrachloride through air and through columns of soil crumbs. The columns were of the same diameter and were prepared in the same way as those used for respiration rate measurements. Great care was taken to ensure that steady-state conditions were established before the rates of evaporation of carbon tetrachloride were recorded. Values of the diffusion coefficients (D_s) at S.T.P. were assumed to be related to the diffusion coefficient (D_{tp}) at temperature t and pressure p by the relation[4]

$$D_{tp} = D_s.(t/273)^{1.75}.76/p \quad . \quad . \quad . \quad . \quad . \quad (6)$$

The mean value obtained in this way for the diffusion coefficient of carbon tetrachloride through air corrected to S.T.P. was $6{\cdot}47 \times 10^{-2}$ cm.^{2} sec.$^{-1} \pm 0{\cdot}21$ (16 d.f.) compared with $6{\cdot}32 \times 10^{-2}$ cm.^{2} sec.$^{-1}$ for the diffusion coefficient through oxygen cited in the literature.[5]

Respiration rates of columns of soil crumbs

The apparatus used for these measurements was a modification of the electrolytic rocking percolator[6] which permitted the continuous measurement of oxygen uptake whilst maintaining a constant oxygen partial pressure. The main difference between the new apparatus and the electrolytic rocking percolator was that the percolator containing soil in the latter apparatus was replaced by a vertical tube about 10 cm. long and 2·3 cm. i.d. The tube at its upper end was sealed by a B29 cone, and about 1 cm. from this end a side-arm was attached, to connect the soil tube to the manometer. The lower end of the soil tube was closed by a rubber bung with a narrow glass tube passing through it. One end of the narrow tube was flush with the inner surface of the bung where it was covered with a fine mesh and the other end was connected to a tap.

Air-dry soil crumbs (30 g.) were added to each of seven tubes, which were then tapped until the bulk density of the soil in each tube was the same. The tubes were evacuated and water added slowly via the taps until the soil samples were completely saturated with water, and then the pressure in the tubes was restored to atmospheric. Some of the water was removed from two of the tubes by applying slight suctions at the taps to give the soils gas-filled pore spaces which, according to Equation (4), were large enough to prevent respiration from being inhibited by lack of oxygen. In the same way different amounts of water were removed from the other five tubes so that the soils had widely different water contents, and thus different gas-filled spaces. After this adjustment of water content, the tubes were connected by side-arms to manometers and closed by means of caps (volume 85 ml.) with B29 sockets. To absorb CO_2 evolved by the soil, a small cup containing 10% potassium hydroxide and a filter paper was inserted in each cap. (In experiments to test the efficiency of CO_2 absorption it was found that when 4 ml. of CO_2 were generated in the apparatus, 90% was absorbed in 40 min.) Water was added to each volume-compensating percolator until the volume of gas phase was the same as in the corresponding soil tube plus its cap. The pressure inside the apparatus was then reduced to about 72 cm. Hg, the manometer filled with copper sulphate solution (reference [6], Fig. 1) and measurements of oxygen uptake recorded at intervals over a period of about 7 days.

The respiration rates, when expressed in the form of ml. of O_2/ml. of soil/sec. of the two aerobic tubes were assumed to be equal to Q.

Respiration rates of soil crumbs held under a suction of 50 cm. of water

Soil crumbs (1·68–3·36 mm.) were saturated with water and brought to a suction of 50 cm. of water as previously described. Eight 1·5-g. samples of such crumbs were carefully spread over the bottom of Warburg flasks and to four of the flasks an additional 0·5 ml. or 1 ml. of water was added. Respiration rate measurements were made in the usual way[1] with stationary flasks.

The moisture content of the crumbs was taken as the mean of the values before and after transfer to Warburg flasks.

Relation between gas-filled pore space per unit volume and distance from the surface of the column

At the completion of respiration rate measurements, the lengths of the seven columns were each determined as the mean of six measurements per column. The distribution of soil and water down each column was then determined while it was kept closed at its base and in a vertical position. For each column the soil was removed in successive sections of known and approximately equal but measured length (about 1 cm.) and their weight and water contents determined. These weights were less than the true weights of soil in the subsections as some adhered to the side of the tube. The weights were therefore corrected for this loss by multiplying by the ratio of the total weight (oven-dry basis) added to the column, to the sum of the weights (same basis) recovered in all the subsections of the column. The water contents were not corrected in any way, the assumption being that the degree of incomplete recovery would be similar for both soil and water.

It was assumed that a linear relation existed between the weight of soil per unit volume of subsection, M_x, and distance x from the surface of the column. The gradient of this relation was calculated from the weights of oven-dry soil in each subsection of the column. The mean gradient was calculated from the six or seven columns in each experiment and the standard errors were obtained from the different columns. To facilitate calculation it was assumed that the subsections in each column were of equal length.

In those experiments where the mean gradient was not significantly different from zero (i.e., all experiments except those with 0·42–0·59 mm. crumbs), M_x was taken to be independent of x, and was calculated for each column from the volume of the column and the total dry weight of soil it contained. For those columns where the mean gradient significantly differed from zero (i.e., those containing 0·42–0·59 mm. crumbs) M_x was calculated in a similar way except that the mean gradient was used to correct for the compression of soil down the column.

Gas-filled pore spaces, E_x, in each of the subsections was calculated as

$$E_x = 1 - M_x/d - M_x W_x \quad . \quad . \quad . \quad . \quad . \quad (7)$$

where W_x is the weight of water per unit of weight of soil in the subsection at depth x, and d is specific gravity.

Values of E_x were plotted against depth x from the surface of the column and the best line of the type $E_x = E_0(1 - ax)$, i.e., Equation (2), was fitted by eye through them. Estimates of E_0 and $E_0 a$, were made from inspection of this line.

Determination of the lengths of aerobic zones in columns of soil crumbs

Method 1

In this method the lengths were obtained by solving Equation (5) graphically. Values for the Q, E_0 and a terms of the equation were determined as described above. The estimation of the remaining terms was based on the fact that water-saturated columns of each of the different fractions of crumbs were drained of most of their inter-crumb water but little of their intra-crumb water by a suction of 50 cm. of water. Thus E', the gas-filled pore space of a column when it contained water-saturated crumbs but no inter-crumb water, was calculated from the water content of crumbs held under a suction of 50 cm. of water, the weight of oven-dry crumbs per unit volume of column and the specific gravity of the oven-dry soil. The diffusion coefficient of oxygen through such a column was given by

$$(D'/D'')_{O_2} = (D'/D'')_{CCl_4} \quad . \quad . \quad . \quad . \quad . \quad (8)$$

where $(D'/D'')_{O_2}$ is the ratio of the diffusion coefficient through soil to that through air for

oxygen and $(D'/D'')_{CCl_4}$ is the same ratio for carbon tetrachloride. It was assumed that all columns had the same values of D' and E' as those of the columns used in the determination of diffusion coefficients (Table II).

Method 2

In this method the lengths, l, were calculated from the equation

$$l = (K/Q) \times L \quad . \quad . \quad . \quad . \quad . \quad . \quad (9)$$

where K is the respiration rate of the column in ml. of oxygen/ml. of column/sec., Q is the rate when respiration is uninhibited by lack of oxygen, and L is the length of the column.

Results

Measurements used to calculate lengths of aerobic zones

(a) *Gas-filled pore space of columns of crumbs containing inter-crumb water.*—The measurement of gas-filled pore space depended on the weight of soil per unit volume of column at points down the column. In the earlier experiments there was no significant compression of soil with increase of depth so that measurements were not made on each column. The measurements which were made, and which are summarised in Table I, showed a tendency for the weight of soil per unit volume to increase with depth down the column, but this compression was only significant in columns of 0·42–0·59 mm. crumbs. It was only in columns of these crumbs that compression was taken into account in assessing the gas-filled pore space.

Table I

Increase in oven-dry weight per unit volume down columns of fine crumbs

(The % increase in weight per cm. increase in depth is the increase in oven-dry weight per unit volume per cm. length multiplied by 100 and divided by the mean oven-dry weight per unit volume)

Crumbs sieved between the indicated limits, mm.	Mean % increase in weight per cm. increase in depth	S.e. of diff. (d.f.) for mean % increase in weight per cm. increase in depth
0·18–0·42	0·48	±1·79 (5)
0·18–0·42	0·44	±0·63 (6)
0·42–0·59	2·06	±0·35 (6)
0·42–0·59	1·75	±0·15 (5)
0·59–0·84	1·25	±0·62 (6)
0·84–1·68	1·31	±0·67 (6)

The gas-filled pore space E_x at a depth x from the surface of the column approximately fitted an equation of the type $E_x = E_0(1 - ax)$, i.e., Equation (2), where E_0 and a are constants. The standard error calculated from a random sample of lines was ±0·037 ml./ml. (56 d.f.) and each line was fitted on the basis of eight points. Those columns which, according to both Methods 1 and 2, contained anaerobic zones had values for E_0a between zero and 0·07 and for E_0 between 0·05 and 0·36. For this group, values for E_0a tended to increase with increases in the values for E_0 but in all other columns E_0a never exceeded 0·03 and E_0 varied between 0·18 and 0·39.

(b) *Diffusion coefficients and gas-filled pore spaces of columns of water-saturated soil crumbs which contained no inter-crumb water.*—The ratios of the diffusion coefficient of carbon tetrachloride though soil to that through air $(D'/D'')_{CCl_4}$ were similar to those calculated from Penman's expression[3] ($D'/D'' = 0{\cdot}66E$ where E is the gas-filled pore space) except for columns of the smallest crumbs (0·18–0·42 mm.) where they were lower (Table II). The gas-filled pore space of the columns of crumbs was smaller, the smaller the crumb size (Table II).

(c) *Aerobic respiration rates of columns of soil crumbs.*—The oxygen uptakes of all the columns of soil crumbs were related to time by exponential curves. Fig. 1 gives the oxygen uptakes of columns of soil crumbs having the high and the low gas-filled pore spaces shown in Fig. 2. Over limited periods of time, in these experiments between 15 and 40 h., 40 and 100 h., and 100 and 150 h., the rates of respiration were approximately constant, and were used in the calculation of the lengths of aerobic zones. Rates over similar periods were used in the other calculations.

Table II

Physical data on columns of small crumbs

Crumbs sieved between the indicated limits (mm.)	0·18–0·42	0·42–0·59	0·59–0·84	0·84–1·68	S.e. of diff. (d.f.)
Columns used for respiration rate measurements					
1. Mean oven-dry weight per unit volume	0·94	0·93	0·81	0·81	±0·018 (59) Minimum ±0·022 (59) Maximum
Columns used for diffusion rate measurements					
2. Mean oven-dry weight per unit volume	0·97	1·00	0·80	0·76	±0·032 (7)
3.* Weight of water per 100 g. of crumbs under a suction of 50 cm. of water	33	27	41	39	±1·2 (11)
4. Gas-filled pore space (E') of columns water-saturated and then equilibrated with a suction of 50 cm. of water	0·31	0·33	0·37	0·41	±0·026 (7)
5. Actual gas-filled pore space of the columns used for CCl_4 diffusion measurements	0·33	0·24	0·38	0·42	±0·031 (7)
6. D'/D'' calculated from Penman's expression,[3] $D'/D'' = 0{\cdot}66E$	0·22	0·22†	0·25	0·27	±0·022 (7)
7. D'/D'' calculated from CCl_4 diffusion measurements	0·15	0·23†	0·19	0·24	±0·019 (7)

* The same results were obtained with samples used for respiration rate measurements.
† Values were corrected to what they would have been if they had the gas-filled pore space in column 4 by using Equation (3).

Values for Q (the respiration rate of the soil when respiration was uninhibited by lack of oxygen) were measured on columns of crumbs which were first water-saturated and then partially dried. Therefore the possibility existed that the determined values of Q were less than the true values owing to inadequate aeration or to lack of water.

Each determination of Q was the mean of measurements made on two columns of soils containing different amounts of water. On average the moister soil had a water content of 47% and a respiration rate 6% lower than the drier soil with water content 39% (Table III).

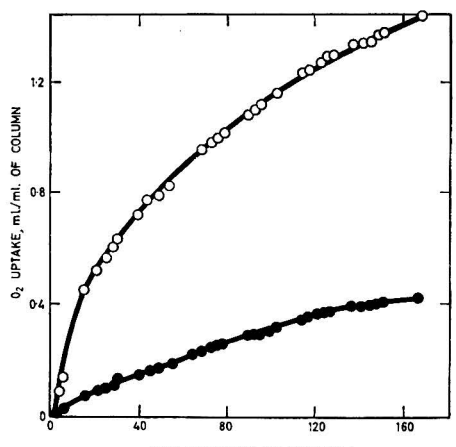

FIG. 1.—*Rates of oxygen uptakes of columns of 0·42–0·59 mm. crumbs, water-saturated and then drained of inter-crumb water to different extents*

Uptakes are expressed as ml. of O_2/ml. of soil column. The higher rate of uptake (○——○) is for the column with gas-filled pore space distribution shown in the upper graph of Fig. 2, and the lower rate of uptake (●——●) is for the column having the gas-filled pore space distribution shown in the lower graph of Fig. 2.

Although according to Equation (1) none of the columns used for the determination of Q contained any anaerobic zones, it would appear that a small error resulted from inadequate aeration, possibly because of localised inhomogeneity of the soil.

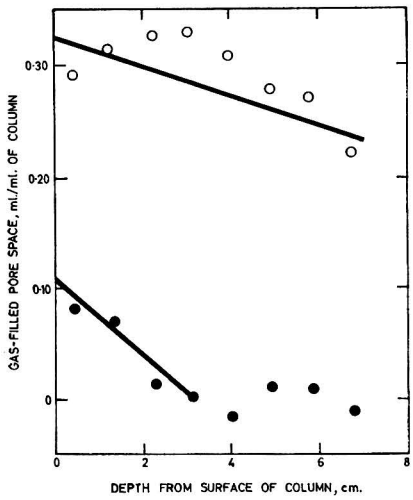

FIG. 2.—*Volume of gas-filled pore space per unit volume of column at different depths from the surface of columns of 0·49–0·59 mm. soil crumbs, first water-saturated* in vacuo *and then drained of inter-crumb water to different extents*

Respiration rate measurements were made on 1·68–3·36 mm. soil crumbs that had been water-saturated and then equilibrated overnight with a suction of 50 cm. of water. The mean respiration rate of such crumbs was 0·423 μl./g./sec. compared with 0·475 μl./g./sec. after the crumbs had been resaturated with 0·5 or 1 ml. of water so that their respiration rates would not be inhibited by lack of oxygen. The s.e. of diff. was $\pm$0·011 μl./g./sec. (12 d.f.). Since the water content of the soils in the columns (Table III) was in all cases greater than their water contents when subjected to a suction of 50 cm. of water (Table II), only a small error could have resulted from restriction of respiration from lack of moisture.

Comparison of the lengths of aerobic zones calculated from the foregoing measurements by Methods 1 and 2

The lengths are summarised in Table IV. When both methods gave lengths that were less than the length of the column, the mean length obtained by Method 1 was 4·3 compared with 3·7 by Method 2. The difference between the lengths obtained by the two methods was, on a percentage basis, less for short aerobic zones than for long ones, and was less for columns with low than with high values of Q.

Table III

Determination of Q, the respiration rate of soil when it is uninhibited by lack of oxygen

[Measurements of Q were always made in duplicate; water-saturated columns of crumbs were partially dried until according to Equation (4) they contained no anaerobic zones, but had different water contents. The table gives a comparison of the values of Q obtained at the higher water contents with those at the lower water contents]

Group of results that were meaned	Mean respiration rate $\times 10^6$ (ml./ml./sec.)		S.e. of diff. (d.f.)	Mean % water content	
	Higher-water content	Lower-water content		Higher-water content	Lower-water content
$Q > 2{\cdot}5 \times 10^{-6}$	5·2	5·5	$\pm$0·170 (9)	47	39
$Q > 2{\cdot}5 \times 10^{-6}$	1·8	2·0	$\pm$0·059 (14)	48	40
Crumbs sieved between the indicated limits (mm.)					
0·18–0·42	3·3	3·6	$\pm$0·204 (9)	46	33
0·42–0·59	3·5	3·6	$\pm$0·058 (5)	46	40
0·59–0·84	3·1	3·2	$\pm$0·103 (3)	48	43
0·84–1·68	3·1	3·3	$\pm$0·053 (5)	52	44
All samples	3·2	3·4	$\pm$0·074 (24)	47	39

In addition to the data summarised in Table IV the following results were obtained. There were 25 instances where the mean lengths obtained by Method 1 was equal to, and the mean length obtained by Method 2 was less than, the length of the column, and only three

Table IV

Mean lengths of aerobic zones in columns of small crumbs

Group of results that were meaned	Mean length (Method 1)	Mean length (Method 2)	S.e. of diff. (d.f.)
Crumbs sieved between the indicated limits, mm.			
0·18–0·42	4·6	3·7	±0·38 (24)
0·42–0·59	2·8	3·3	±0·19 (24)
0·59–0·84	6·3	4·0	±0·39 (12)
0·84–1·68	4·7	4·5	±0·33 (10)
Respiration rate × 10^6 (ml./ml./sec.)			
>2·5	4·6	3·5	±0·29 (32)
<2·5	4·1	3·9	±0·27 (40)
Length of aerobic zone (Method 2)			
>4 cm.	6·0	5·4	±0·30 (32)
<4 cm.	2·9	2·4	±0·27 (40)
All experiments except those with dia. 0·42–0·59 mm.	2·8	3·3	±0·19 (24)
All experiments	4·3	3·7	±0·21 (73)

Note. No account was taken of the compression of soil down the columns in determinations of the theoretical lengths except when the crumb fraction was 0·42–0·59 mm. in diameter.

instances where the reverse occurred. The mean lengths of all 28 columns were 11·7 cm. (Method 1) and 7·3 cm. (Method 2). There were also six columns, other than those used for determinations of Q, where both methods showed that the columns were fully aerated.

Discussion

The mean lengths of the aerobic zones obtained by Methods 1 and 2 were approximately the same, although there were very large errors in the differences between the means; these differences require explanation. In Method 2 the lengths were determined from measurements of respiration rates and lengths of the columns, both of which can be determined accurately. Therefore the errors between the mean lengths must have arisen from errors in the determination of the lengths by Method 1 and thus may have arisen from any of the following factors:

(1) Inadequate correction for the compression of soil down the columns.
(2) The approximation (required for the assessment of E' and D') that columns in which the aerobic zones were measured had the same values for the mean oven-dry weight per unit volume as the columns used in the measurement of diffusion coefficients (Table II).
(3) Uneven distribution of gas-filled pore space, for example, by the formation of through-channels.
(4) Absence of steady-state conditions.
(5) Values of Q may have been reduced by inadequate supplies of oxygen (Table III).
(6) The approximation that the respiration rate of soil crumbs was not inhibited unless the oxygen concentration at the surfaces of the crumbs was zero. Many of the crumbs were as large as 2 mm. in diameter when dry and, because of reduction of their surface areas by contacts between adjacent crumbs, may have had their respiration rates appreciably reduced even at fairly high oxygen concentrations.
(7) The diffusion coefficient of oxygen was taken as that into air whereas the oxygen contents at points towards the base of the column were much lower than that of air.
(8) Diffusion of organic compounds produced in the anaerobic zones into the aerobic zones where they would be metabolised and thus would enhance the respiration rates.

The theoretical length of the aerobic zones is more sensitive to changes in gas-filled pore space than to similar proportionate changes in any of the other determinations necessary for

the solution of Equation (4). Moreover, determinations of the gas-filled pore space are themselves very sensitive to factors (1) and (3) above. For example, a 2% increase in dry weight per cm. depth down a column of soil, 8 cm. in length and containing 57% of water uniformly distributed down the column would result in the gas-filled pore space decreasing from 0·18 ml./ml. at the surface of the column to 0·06 ml./ml. at its base. It would seem, therefore, that the small compression of soil down the columns (Table I) may be of major importance in explaining the errors. Factor (4) is also probably of importance because steady-state conditions are almost certainly not established in the early stages of respiration rate measurements. Some experimental evidence in support of this view is that there is a smaller difference between lengths of aerobic zones as ascertained by Methods 1 and 2 at the lower than at the higher respiration rates. Since respiration rates decreased with time from the start of making measurements, it appears that the differences between the lengths obtained in the two methods would be less at the end than at the beginning of each experiment.

Despite the errors, the equations described in this and previous papers[1, 7] undoubtedly provide a means of determining the approximate extents of anaerobic zones in columns of soil crumbs. Furthermore, because field soils can be simulated by columns containing mixtures of different sized crumbs, it may be possible to apply either these or similar equations with sufficient accuracy to determine whether or not a field soil contains any anaerobic zones.

It seems probable that many soils never have any anaerobic zones under any climatic conditions. On the other hand there are many soils which may have anaerobic zones in some climatic conditions but not in others. What is required, therefore, is a way of assessing the probability of a field soil having anaerobic zones over a period of, for example, a year's duration. Any direct method, at best, would be able to show only the extent of anaerobic zones at the precise instant of measurement and would give no idea of what would happen if the weather conditions changed. By contrast, the methods based on the equations would enable predictions to be made of the probable occurrence of anaerobic zones under different weather conditions. This is because the equations are in terms of parameters, such as gas-filled pore space, which alter in a definable way with changes in climatic conditions. In addition, methods based on the equations would have the advantage that they would only require measurements of easily measurable soil properties. It would seem, therefore, that methods based on the proposed equation may offer a better, but yet imperfect, means of assessing the aeration status of field soils than would any direct method.

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SEPARATION AND COMPOSITION OF 'POLAR' WHEAT-FLOUR LIPIDS

By J. J. WREN and ANNA D. SZCZEPANOWSKA

The lipids of an untreated white flour, which had been milled commercially from Canadian wheat, were extracted at −23° with chloroform–methanol and freed from contaminating non-lipids (including lipophilic protein) by means of aqueous potassium citrate. They were then eluted on a preparative scale from columns of silicic acid, using continuous, concave gradients of methanol in chloroform and also pure methanol. The fractions obtained were examined by infra-red and chemical methods, and by chromatography on aminoethylated paper. Eleven known classes of polar lipids were identified, each class being estimated to account for between 1% and 15% of the flour lipids. Other classes were detected but remain unidentified.

Introduction

Although the lipids of wheat flour normally constitute less than 2% of the weight of the flour, they are a complex mixture, including glycerides, glycolipids, phospholipids, sterols and free fatty acids, and they have important functions in bakery products.[1–5] Understanding their functions must ultimately depend upon knowledge of their chemical structures, physical properties, and amounts. In large measure by means of chromatography on silicic acid, much has recently been learnt about the classes of lipids present in flour; gas chromatography has also yielded much information on the fatty acid composition of whole extracts and some separate classes.

Roughly half of the flour lipids are so-called 'polar' lipids—that is, those more polar than free long-chain fatty acids—which are considered to be of more functional importance than the others because they have more potential in surface phenomena and in interactions with starch and gluten. The aims of the studies to be described were, firstly, fractionation of the polar lipids on a relatively large scale (for future physical and functional studies) and, secondly, their characterisation by chemical and other means.

Extraction was carried out at low temperature and non-lipid contaminants, including lipophilic protein, were removed from the extracted lipid by a new washing technique. The lipid was fractionated on silicic acid columns by elution with a continuous, concave gradient,[6, 7] which gave better resolution than that achieved with continuous, convex gradients[8–11] or discontinuous gradients.[3, 12–16] Infra-red monitoring of the effluents[17] minimised the work required to locate peaks in repeated experiments. Because an antioxidant was incorporated in the solvents used[18] the effluents were stable, and the lipids could be safely stored in solution at low temperature. The lipids were characterised chiefly by infra-red spectrophotometry, chromatography on aminoethylated paper,[19] and deacylation.[20]

Experimental

Isolation of lipid

A 350-g. portion of flour was extracted with chloroform–methanol (2 : 1, v/v) at −23°, as described previously,[6] but with potassium citrate in place of sodium chloride in the washing procedure. The crude extract (2·1 l.) was shaken thoroughly with 2% aqueous tripotassium citrate monohydrate (350 ml.) and left at −23° overnight. The clear lower layer that separated was then washed twice, in similar fashion, with 350-ml. portions of chloroform–methanol–water (3 : 48 : 47, v/v/v) containing 1% citrate, and evaporated at <30° in a rotary evaporator. The residue was redissolved in 100 ml. of chloroform–methanol (2 : 1, v/v), recovered by evaporation, and then washed into a polypropylene tube with chloroform (25 ml.). This gave a cloudy solution which was mixed with a few drops of water and centrifuged. The tube was carefully punctured to release the clear chloroform solution, leaving behind a wet solid. The solution was evaporated, and the residue obtained was dissolved in fresh chloroform and stored at −23°. If more solid appeared during storage it was removed as before.

The yield was 1·30% (of undried flour).

Column chromatography

Silicic acid (450 g.) (Mallinckrodt, A.R., through 100 mesh) was mixed with 1·5 l. of methanol (A.R.), and after ½ h. the supernatant methanol and fine particles were poured off. The residue was transferred to a column (dia. 7 cm.) with chloroform (A.R.), and washed with chloroform until it became uniformly transparent. Lipid (16·5 g.) was then run on in 100 ml. of chloroform, and washed in with a further 500 ml. ; this and the other eluents used contained 0·005% BHT.[18] A gradient elution device[21] was connected, in which a Winchester bottle (dia. 13 cm.), containing 2·75 l. of 40% (v/v) methanol in chloroform, was used as reservoir, and a 10-l. bottle, containing 6·2 l. of chloroform, as mixing chamber. The effluent was collected in 20-ml. fractions. Every third fraction was evaporated in a tared beaker at 40° *in vacuo*, and the residue weighed. The other fractions were examined directly in an infra-red spectrophotometer,[17] then pooled in appropriate groups, partly evaporated, and stored at −23°.

The description above applies strictly to the experiment from which Fig. 1 was obtained, in which only peaks A to F were eluted by the gradient. In other similar experiments two more major peaks, G and H (see reference [7]), were obtained by continued elution with methanol (2·5 l.) or with a gradient of 40% to 100% (v/v) methanol in chloroform.

Infra-red analysis

After evaporation, selected fractions were examined on sodium chloride plates by casting films from chloroform solutions.

Chromatography on aminoethylated paper

Ten disks of Whatman AE30 paper (K.C.T. pattern, 14·5 cm. in diameter) were placed centrally on five sheets of Whatman No. 1 paper (23 cm. square), which in turn rested on a glass plate (20 cm. square). Another glass plate was placed so that a small hole at its centre rested at the centre of the paper disks ; it was then loaded evenly with weights (4 kg.). A PTFE tube was fitted firmly into the hole and the following solvents were introduced in turn : 200 ml. of 8N-aqueous ammonia ; 400 ml. of ethanol–water (4 : 1, v/v) ; 500 ml. of 2,6-dimethylheptan-4-one–acetic acid–water (4 : 5 : 1, v/v/v). After being washed the disks were separated, dried for 1 h. at 80°, and stored in a polyethylene bag.

A solution of 100–200 μg. of lipids in 20 μl. of t-amyl alcohol–benzene (1 : 1, v/v) was applied in each 72° segment on a starting line 2 cm. from the centre. The chromatograms were developed for 4 cm. with 2,6-dimethylheptan-4-one–acetic acid–water (8 : 5 : 1, v/v/v)[19] through a paper wick cut so that development took ~45 min. They were left overnight to dry and cut into 36° segments for the following tests :

Lipids.—With Rhodamine 6G,[22] protoporphyrin,[23] Malachite green.[24]

Amino groups.—Sprayed with 0·2% (w/v) ninhydrin and 10% (v/v) 2,6-lutidine in n-butanol and heated at 105° for 5 min.

Phosphates.—Sprayed with the reagent of Hahn & Luckhaus[25] and left at room temperature for 1 h. ; blue zones.

Sugars (naphthoresorcinol[26]).—Sprayed with 0·2% (w/v) naphthoresorcinol and 5% (v/v) phosphoric acid in moist n-butanol, heated at 105° for 10 min., and left at room temperature overnight ; blue-green zones on a pink background.

Sugars (diphenylamine–urea[27]).—Sprayed with a reagent containing 2% diphenylamine, heated at 100° for 5 min., and left at room temperature overnight ; yellow or pink zones on a blue-grey background. In both tests for sugars, detection was improved by observing fluorescence under ultra-violet light.

Deacylation

Before use Zeo-Karb 225 ion-exchange resin (as beads, 14–52 mesh) was washed successively with 2N-aqueous hydrochloric acid, 6N-aqueous pyridinium chloride, and water (until the effluent was neutral). Water was distilled in an all-glass apparatus, and ethanol was distilled through a fractionating column after refluxing with sodium hydroxide and silver nitrate. Chloroform was distilled after refluxing with dicyclohexylamine and then after refluxing with phosphorus pentoxide and 2,4-dinitrophenylhydrazine.

At room temperature, 0·7 ml. of 0·15N-lithium hydroxide in ethanol–water (6 : 1 v/v) was mixed with a solution containing 4–12 mg. of lipid in 0·2 ml. of chloroform and 0·1 ml. of ethanol.[28] After 15 min., the mixture was shaken with 3 ml. of water, 2 ml. of ethanol and 4 ml. of chloroform, and the emulsion formed was broken by centrifuging (10 min.). The upper layer thus separated was passed through a column (3 × 1 cm.) of Zeo-Karb 225 which was developed with 7 ml. of ethanol–water (1 : 1, v/v). The effluent was collected, washed twice with 3-ml. portions of ether, and evaporated at $<30°$ in a rotary evaporator. (A few drops of acetone were added to inhibit foaming.) The residue was dissolved in 1 ml. of t-butanol–water (4 : 1, v/v) and stored at $-23°$ until applied to chromatograms.

Chromatography of deacylation products

System X.—Layers of cellulose (Macherey-Nagel, without binder), 0·5 mm. in thickness, were prepared by the usual thin-layer chromatography (TLC) technique and washed by development with water-saturated phenol. They were then left overnight to dry, washed three times by immersion in ether, and again left to dry before use. The chromatograms were developed[29–31] (15 cm.) with water-saturated phenol, left overnight to dry, and washed twice with ether before spraying.

System Y.—Before use, Whatman No. 1 paper was washed (8–16 h.) with 0·1N-aqueous hydrochloric acid by descending development, and left overnight to dry. Chromatograms were developed[29, 30] with n-butanol–acetic acid–water (5 : 3 : 1, v/v/v) by ascending technique (20 cm.) and left to dry.

System Z.—Unwashed paper and n-propanol–ammonia (sp. gr. 0·880)–water (6 : 3 : 1, v/v/v)[20, 32] were used in the same way.

Detection methods.—Periodate–Schiff,[33] silver nitrate dip (system Z only),[34] Dragendorff (Merck[35] reagent no. 30a), ninhydrin (as above), ultra-violet method[36] for phosphates.

Results and discussion

The results presented are for a single flour of 70% extraction, containing no improver or supplement, which was milled commercially from Canadian wheat and stored in a can at $-23°$. Another flour of similar origin, which was also examined in detail, afforded somewhat less lipid in the washed extract; it gave the same results for glycolipids but was markedly deficient in phospholipids.

Chloroform–methanol extracts flour lipid at least as efficiently as other solvents commonly used for this purpose, and provides a solution that can be subjected to a washing technique like that of Folch *et al.*[37] without prior evaporation (cf. Fisher *et al.*[16]). However, it extracts large amounts of lipophilic protein,[38] even when used at low temperature. Part of the protein is precipitated and removed during washing, but part remains, some being slowly precipitated during storage, some eluted from silicic acid in lipid fractions, and some retained on silicic acid so that recoveries after chromatography are low.[6] Lipophilic protein is significant practically because it cements silicic acid particles together and so blocks columns.[7] Washing by the established technique[37] with sodium chloride sometimes removed most of the protein, but not always, and evaporating in different ways[39, 40] seldom helped. Washing with potassium citrate[41] gave much better results.

The yield of washed lipid was reproducible but appeared to be only about 80%. Infrared analysis revealed very little lipid in the protein removed by centrifuging, and this was not bound, since it could easily be washed off with chloroform. However, the aqueous layers and interfacial precipitates rejected during washing probably contained some lipid. Using a new hydrolytic method,[42] it was found in other experiments that 10–20% of flour lipid cannot be extracted under normal conditions. This is probably the bound lipid of the starch granules;[43, 44] granules carefully purified from a similar flour contained 0·26% of hydrolysate lipids.

The column method differs from that previously described[6] in several ways, but most notably in that the ratios, eluent volume/weight of adsorbent/weight of lipid applied, are changed from 6 l. : 45 g. : 1·7 g. to ~12 l. : <450 g. : 16·5 g. Better resolution was obtained despite the large relative saving in eluents.

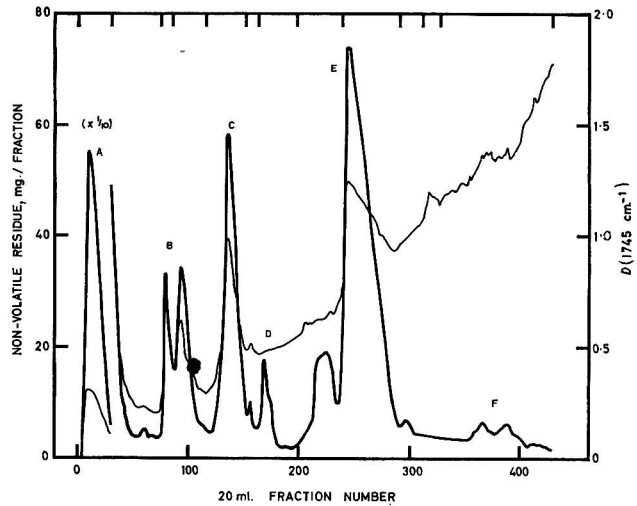

FIG. 1.—*Elution curves of washed flour lipid from silicic acid*
Thick line: gravimetric
Thin line: infra-red spectrophotometric
Peak divisions listed in Table I are indicated at the top

The elution curves for a typical column are shown in Fig. 1. Regions of the curves are designated by letters which, to avoid confusion, are those used in previous publications[6, 7] for the main peaks. Although several new peaks can be identified, these are referred to as parts or satellites of the main peaks (Table I).

The citrate-washed lipid contained 5100 p.p.m. of potassium, only 40 p.p.m. of which were recovered in peaks A to F and 350 p.p.m. in peaks G and H. Slightly smaller quantities of sodium were also recovered; these were probably exchanged[45] for potassium by the silicic acid (which contained about 400 p.p.m. of sodium). No calcium or magnesium (limit of detection, 25 p.p.m.) was found in citrate-washed or sodium chloride-washed lipid. This indicates the

Table I

Correlation of results of column and paper chromatography and deacylation

Peak	Zones on amino-ethylated paper	Deacylation products	Weight of lipids (g. per 100 g. applied to column)
A (main)	i*, ii	d_1*, d_2	54·0
(late)	i, iii*, iv	d_1, d_2*, d_3	
B (first)	iii, iv, v*, vi, vii, viii	d_1, d_2, d_3, d_4*, d_5	5·0
(second)	vii, viii*, ix	d_1*, d_2, d_4, d_5	
C (main)	viii, ix, x*, xi	d_2*, d_5, d_8	5·7
(late)	viii, x, xii*, xiii	d_2*, d_5, d_6	
D	x, xii*	d_2, d_6*	1·2
E (early)	xiv*, xv, xvi	d_6	18·0
(main)	xv, xvi*	d_2, d_5, d_6*	
(late)	xv, xvi*, xvii	d_4, d_5, d_6, d_7*	
F (early)	xv, xvi, xvii, xviii*	d_5, d_6*, d_7, d_9	3·5
(main)	xvi, xvii, xviii, xix*	d_1, d_4, d_5, d_6, d_8*	
G (early)	xvi, xx*, xxi*	d_8*, d_{10}*, d_{11}	5·2
(main)	xxi, xxii*	d_4, d_6, d_8, d_{11}*	
H	xxiii, xxiv*, xxv	d_2, d_4, d_6, d_{11}*	1·8
		(Total	94·4)

* Major constituent

absence of chelated aggregates such as those found in extracts of flax seed.[46, 47] Most fractions from sodium chloride-washed lipid gave on evaporation protein residues which were difficult to redissolve. In contrast, only fractions of peak E (late) from citrate-washed lipid gave such residues, and these were small.

Lipids were partly identified by their elution behaviour[45] and infra-red spectra (Fig. 2). The spectra were correlated with data published for flour lipids[15, 48, 49] and well-characterised lipids from other sources (e.g., in references [50, 51]).

Aminoethylated paper was chosen for analytical chromatography of eluted fractions because the order of mobility on it should differ from that on silicic acid, and because Mumma & Benson[19] had used it with some success. However, to achieve reproducible results and good resolution, it proved necessary to develop a fairly elaborate technique. Radial development gave better resolution than linear. Numerous detection reagents were useless due to background coloration. Lipid dyes proved useful and so, somewhat surprisingly, did ninhydrin; it gave only a pale mauve background, on which amino compounds appeared as dark mauve zones and some other compounds white. Most phosphate reagents coloured the background but one,[25] which was used previously[52, 53] on thin-layer chromatograms, gave excellent results. Naphthoresorcinol and diphenylamine–urea reagents were useful for glycolipids, but not specific.

Each fraction from silicic acid gave at least two zones on paper. The zones were given code numbers in the order in which they were obtained from successive fractions (Table I). Data pertaining to their identification are given in Table II. Any zone may obviously be formed by more than one class of lipids.

Of several deacylation methods tested, only Brockerhoff's[28] liberated digalactosyl glycerol quantitatively without extensively converting phospholipids to glycerol cyclic phosphate[54] or alcohol derivatives.[55] To avoid spurious products it was essential to use pure reagents. The water-soluble deacylation products were chromatographed in three systems, detected by the periodate-Schiff method, and identified with the help of several reagents. Ten of the eleven products distinguished (Table III) were identified from data in the literature, and by direct comparison with authentic compounds or their deacylation products; product d_3, detected (like d_2 and d_6) only by the periodate-Schiff and silver nitrate methods, was not identified. Since an unknown compound might conceivably form a zone coincident with that of a known compound, giving the same colour tests, in each of the three chromatographic systems used, the identifications are not completely rigorous. Product d_5 was probably a series of lysolipids which (since infra-red analysis showed that deacylation was virtually complete) were probably of the glycerol ether type (see Norton & Brotz[56]).

After acid hydrolysis the following constituents were detected: glycerol, in all peaks (Fig. 1); ethanolamine, in peaks B (early) to G (early); inositol, in peak G (early); choline, in peaks G (main) and H; galactose, in peaks B (early) and C (main) to H; glucose, in peaks B (late), D, E (early) and F (main); mannose (tentative) and other sugars, in F (main). Peaks A, B (late) and D gave strongly positive Liebermann–Burchard tests.

Conclusions

Peak A

Triglycerides, fatty acids, diglycerides and sterols are the major constituents, which were partly resolved into peaks by some columns similar to the one described. The minor constituents include pigments, and two unidentified lipids from which d_2 and d_3 were derived. Apparently d_2 is identical with the deacylation product of galactosyl diglycerides, which are eluted in peak C. (Carter *et al.*[15] also obtained this product from flour lipids eluted by chloroform.)

Peak B

The twin peaks are formed by lipids of various classes, including at least two tailing from peak A. As indicated by elution behaviour,[57] by the positive phosphate reaction of zone v, and by the identification of d_4, phosphatidic acids constitute most of the first peak. Monoglycerides, a sterol glucoside,[11, 15] and an unidentified phospholipid (zone viii) are constituents of the second peak.

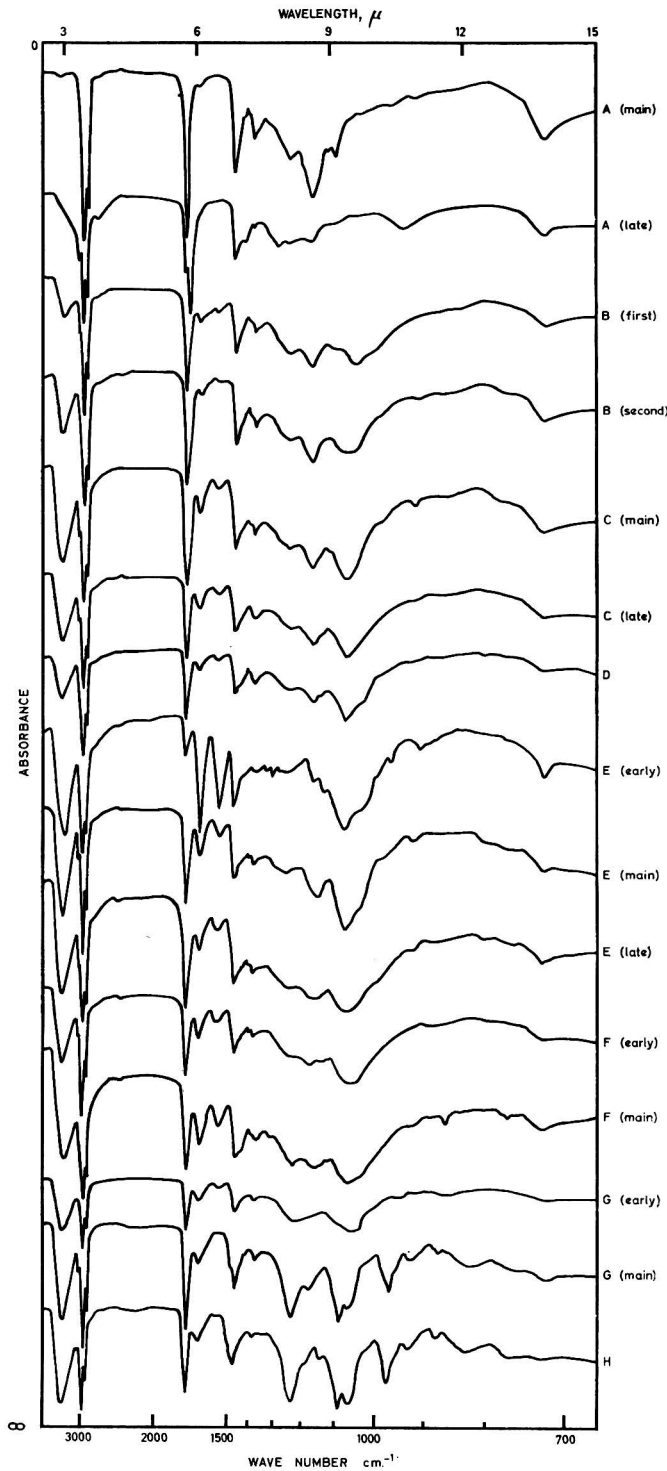

FIG. 2.—*Infra-red spectra*

Table II

Chromatography on aminoethylated paper

Zone	Rhodamine 6G	Protoporphyrin	Malachite green	Ninhydrin	Phosphate	Naphthoresorcinol	Diphenylamine–urea	100 × R_F	Authentic compound(s) forming indistinguishable zone
i	O	+	L	L		+	+	96–100	Triglycerides
ii	B	+	L					89–96	Palmitic, oleic acids
iii	P							93–100	Sterols
iv	Y	+					+	86–93	
v	O	+	D	L	+		+	65–72	
vi	Y	+	D					35–43	
vii	O	+	D					94–100	Monostearin
viii	O	+	D	L	+	+	+	48–55	
ix	Y	+	D	L			+	76–82	
x	Y	+	D	L		+	+	64–70	(lit. :[19] galactosyl diglycerides)
xi	Y			D				13–19	
xii	O	+	D	L			+	42–52	
xiii	Y	+	D					33–40	
xiv	Y	+	D			+	+	35–45	Glucocerebrosides
xv	O	+	D			+	+	41–51	
xvi	Y	+	D	L		+	+	30–40	Digalactosyl diglycerides
xvii	O	+	D		+			4–9	
xviii	O	+	D	D	+	+	+	26–32	(lit. :[19] phosphatidyl glycerols)
xix	O	+	D	D	+	+	+	41–50	DL-α-(dipalmitoyl)-phosphatidyl ethanolamine
xx	O	+	D	D	+	+	+	26–34	
xxi	Y	+	D	P	+			16–21	Phosphatidyl inositols
xxii	Y	+	D	L	+	+	+	52–62	Phosphatidyl cholines (from egg)
xxiii	Y	+						49–54	
xxiv	Y	+		L	+			32–40	Lysophosphatidyl cholines
xxv	Y					+	+	26–31	
(Not detected								0	Phytoglycolipid)

B, blue O, orange P, pink Y, yellow L, lighter than background D, darker than background

Peak C

Contrary to the report of Mason & Johnson,[58] galactosyl diglycerides,[48] forming most of peak C, are major constituents of the lipid of unbleached flour. When re-chromatographed they were eluted selectively[15, 59] with 2% methanol in A.R. chloroform; minor lipids with absorption peaks near 1550 and 1650 cm.$^{-1}$ (Fig. 2), which were presumably cerebrosides, were then eluted later. Two minor deacylation products, d_6 and d_8, seem to be derived from novel types of glycolipid and phospholipid, respectively, since their known precursors (digalactosyl diglycerides and phosphatidyl ethanolamines, respectively) were eluted in peaks E and F.

Table III

Water-soluble deacylation products

Product	100 × R_F in system			Authentic compound forming indistinguishable zones
	X	Y	Z	
d_1	76	57	73	Glycerol
d_2	68	23	57	(lit. :[29–31] galactosyl glycerol)
d_3	43	41	48	
d_4	31	50	27	Glycerol phosphate
d_5	~90	~90	~90	Lysolipids
d_6	54	07	39	Digalactosyl glycerol
d_7	48	48	50	(lit. :[29–31] di-(glycerol) phosphate)
d_8	67	12	49	Glycerol ethanolamine phosphate
d_9	18	3?	7	(lit. :[29, 31] sulphoquinovosyl glycerol ?)
d_{10}	7	4	30	Glycerol inositol phosphate
d_{11}	92	28	42	Glycerol choline phosphate
(Not detected	56	50	55	Glycerol cyclic phosphate)

Peak D

The infra-red elution curve (Fig. 1) shows that this peak contains relatively little ester. The full spectrum (Fig. 2),[60] the Liebermann-Burchard test, and the finding of glucose indicate that the chief constituent may be a sterol glucoside.

Peak E

The early part, which was fairly well resolved as a separate peak, was formed by glucocerebrosides,[14, 15, 49] which after re-chromatography in 6% methanol gave a spectrum identical with that of an authentic specimen. The main part was formed by digalactosyl diglycerides.[15, 48] Both parts contained an unidentified constituent (zone xv) which, after isolation from aminoethylated paper, gave characteristic absorption peaks near 1715 and 1360 $cm.^{-1}$

Peak F

The early part appears to be formed chiefly by phosphatidyl glycerols (which have been eluted previously[57] just before phosphatidyl ethanolamines), and the main part by phosphatidyl ethanolamines. Deacylation, and also colorimetric assay,[61] provided some evidence that sulphoquinovosyl diglycerides[62] were present in the early part; both parts contained several minor, unidentified lipids.

Peak G

Phosphatidyl inositols (for similar elution behaviour see Chang & Sweeley[63]) and lysophosphatidyl ethanolamines formed the early part, and phosphatidyl cholines the main part. Since d_4 and d_6 were minor deacylation products of the main part it seems probable that lyso derivatives of phosphatidic acids and digalactosyl diglycerides were present.

Peak H

This peak was formed chiefly by lysophosphatidyl cholines. The most abundant classes of polar lipids are digalactosyl diglycerides, phosphatidyl cholines, galactosyl diglycerides, glucocerebrosides, monoglycerides, phosphatidic acids, sterol glucosides, lysophosphatidyl cholines, phosphatidyl ethanolamines, phosphatidyl inositols, and lysophosphatidyl ethanolamines. This is the approximate order of abundance in the flour studied, the concentrations ranging from about 15% to about 1% of the lipid. The results given serve to identify these classes adequately, and also to reveal perhaps a dozen minor classes which are incompletely characterised.

No evidence was obtained for the presence of a phytoglycolipid;[64] Carter *et al.*[65] reported its occurrence in wheat germ oil but Mason & Johnson[58] found little in the endosperm.

The lipids studied were presumably[10, 11, 66] derived from germ as well as endosperm. Quantitative comparison of the polar lipid classes in different varieties of grain, and in parts of the grain of single varieties, will ultimately require more powerful separation methods than those used hitherto. It seems important to note that estimations by a deacylation method alone[16, 20, 67, 68] would be invalid because in these studies several classes of lipids (differentiated by chromatographic behaviour) gave apparently identical deacylation products.

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THE COMPOSITION OF *BOMBACOPSIS GLABRA* SEED OIL

By J. A. CORNELIUS, T. W. HAMMONDS and G. G. SHONE*

The fatty acid composition of the oil has been examined and found to contain 34·5% of the cyclopropenoid acid sterculic acid, 43% of palmitic acid and small quantities of stearic, oleic and linoleic acids.

Introduction

Bombacopsis glabra (Pasq.) A. Robyns [formerly known as *Bombax oleagineum* (Decne) A. Robyns][1] is a member of the Bombacaceae family, and is to be found in tropical Africa and South America. The seeds are light brown in colour with whitish striations, and 5–12 seeds are found enclosed in an ovoid capsule.

Pieraerts *et al.*[2] stated that *Pachira aquatica*, Aubl. seeds (these are related to *B. glabra*,[1] but from the published photographs[2] they could be *B. glabra* seeds) were eaten in Brazil in their raw state. It would be extremely unwise to ingest these seeds in view of the fact that this work (and that of de Bruin *et al.*[3] on *P. aquatica* oil) shows that these seeds contain an appreciable quantity of sterculic acid, which is known to be physiologically active[4] and toxic to chickens and rats.[5]

Experimental and results

Preliminary examination

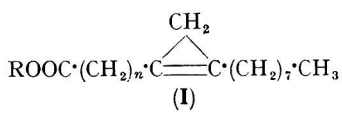
Oil was extracted from seeds of Northern Rhodesian origin with light petroleum (b.p. 40–60°) at ambient temperature using a top-drive macerator, and was found to have the characteristics given in Table I.

Table I

Constants for B. glabra *seed and oil* (*B.S.* 684 : 1958)

Kernel in seed, %	78·3
Oil in kernel, % (moisture-free basis)	45·2
Oil	
Refractive index n_D^{26}	1·4654
Acidity, % (as oleic acid)	1·6
Saponification value, mg. KOH/g. oil	196
Iodine value (Wijs, 30 min.)	54
Unsaponifiable content, %	0·76

The oil gave an intense Halphen colour, indicating the presence of a cyclopropene group. An assessment of the cyclopropenoid content, by spectrophotometric estimation of the Halphen colour at 505 mμ,[6] gave a result of 34 ± 4% [as sterculic acid (**I**, $n = 7$, R = H)]. Titration of the oil, and the fatty acid methyl esters, with hydrobromic acid[7] at 60° gave a cyclopropenoid content of 33% as sterculic acid (34·5% as methyl sterculate).



(I)

The infra-red absorption spectrum of the oil was typical for a largely saturated fat except for a band at 9·9 μ (indicating -CH_2- cyclopropane/ene, and confirmed by a weak near-infra-red absorption at 1·65 μ). The only absorption maximum in the ultra-violet region occurred at 210 mμ.

Preparation of fatty acid methyl esters

Methyl esters were prepared from the oil by trans-esterification using potassium methoxide

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in methanol[8] (0·5 h. reflux or 16 h. room temperature). The cyclopropenoid content of the esters was found to equal that of the oil by hydrobromic acid titration.

Gas–liquid chromatography

Fatty acid methyl esters were separated by gas–liquid chromatography using:

(1) The 'Panchromatograph' (W. G. Pye & Co.) in conjunction with 150 × 0·4 cm. columns packed with either 10% polyethyleneglycol adipate (PEGA) (160°) or 15% diethyleneglycol succinate (DEGS) (170°) on 80–100 mesh Celite or Chromosorb W (impregnated with hexamethyldisilazane) and using a flame ionisation detector (400 v.).

(2) A Gas Chromatography Ltd. (GCL) instrument with 183 × 0·4 cm. columns packed with either 10% polyethyleneglycol adipate (160°) or 5% Apiezon L (200°) on 80–100 mesh Celite, and using an argon ionisation detector (1000 v., tritium source).

Argon was used throughout as carrier gas.

Table II

Equivalent chain lengths found for various fatty acid methyl esters

Instrument	Pye and G.C.L.	Pye	G.C.L.
Stationary phase	PEGA	DEGS	Apiezon L
Oleate	18·2	18·4	17·7
Linoleate	18·8	19·2	17·6
Linolenate	19·5	20·2	17·6
Sterculate	19·8	20·3	18·5
'X'	19·2	19·5	—
Dihydrosterculate	19·25	19·4	18·7
9(10)-Methyloctadecanoate	18·3	18·2	18·3

Isolation of cyclopropenoid material

This material was isolated by the urea-complex technique, using fatty acid methyl esters and allowing the complexes of the unwanted esters to crystallise first at room temperature,[9] and then at −20°; this was followed by crystallisation of the cyclopropenoid containing complexes at −45°, and finally of the cyclopropenoid ester at −45°.[4] Gas–liquid chromatography showed the isolated material to be composed of methyl sterculate (equivalent chain length Table II) but no malvalate (**I**, $n = 6$, $R = CH_3$); a small sterculate decomposition peak was obtained ('X', Table II). This isolate was obtained with a purity of >98% (as methyl sterculate) as determined by hydrobromic acid titration.

Hydrogenation of the sterculate isolate and the original oil

Hydrogenation was carried out in ethanol over a platinum oxide catalyst up to 60°. Dihydrosterculate, nonadecanoate and 9(10)-methyloctadecanoate were produced, but no corresponding products for malvalate were detected. The equivalent chain lengths of these products are given in Table II.

Oxidation of the unsaturated methyl esters

Fatty acid methyl esters, obtained from Bombacopsis oil, were oxidised[10] and methylated using boron trifluoride/methanol[11] to yield mono- and di-esters from the original unsaturated materials present in the oil. Gas–liquid chromatography of the reaction products gave a very small peak in the suberic ester position on polyethyleneglycol adipate, but not on Apiezon L, indicating the absence of methyl malvalate in the original esters.

Quantitative analysis of methyl esters of B. glabra

The quantity of palmitate present in the ester mixture was estimated by gas–liquid chromatography of methyl esters derived from the oil before and after hydrogenation, after the addition of a known quantity of methyl myristate. The total quantity of cyclopropenoid material present was known from the hydrobromic acid titration results. The remaining

material consisted of the esters of the C_{18} acids, stearic, oleic and linoleic (linolenic being absent). The contribution of each of these esters was assessed from the gas–liquid chromatograms of the total methyl esters prepared from the oil. Table III gives the fatty acid composition of the oil.

Table III

Fatty acid composition of Bombacopsis glabra *seed oil* (%)*

Palmitic	43·0	Linoleic	7·8
Stearic	2·8	Sterculic	34·5
Oleic	12·0		

* Calculated iodine value for this composition—53

Discussion

Cyclopropenoid fatty acids have been found in a small number of seed oils of the natural order *Malvales*[12] and are most abundant in *Sterculia foetida* seeds, the fatty acids of which contain 61%[13] cyclopropenoid fatty acids mainly as sterculic acid.

The fatty acid composition of *B. glabra* oil, given in a preliminary publication,[14] was incorrect for the following reasons. The presence of acidic catalysts during the methylation of cyclopropenoid acids results in rapid decomposition of the cyclopropene ring ;[15] thus, the boron trifluoride–methanol reagent used previously[14] gave many spurious peaks on gas–liquid chromatography up to an equivalent chain length of 23·3. Only one decomposition peak occurs when cold extracted oil is transesterified with potassium methoxide in methanol, and this seems to be a result of gas chromatography rather than esterification. Also, rapid decomposition of most of the cyclopropenoid material occurs when the material is brought into contact with silver nitrate,[15, 16] and chromatography on silicic acid impregnated with silver nitrate is not possible.

It has not been found possible to estimate the sterculic acid content of the oil from the area of the sterculate peak produced on gas–liquid chromatography as some of the sterculate is decomposed or polymerised during chromatography, the extent depending upon chromatographic conditions.[15]

Acknowledgment

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JOURNAL OF THE SCIENCE OF FOOD AND AGRICULTURE

ABSTRACTS

MARCH, 1965

The general arrangement of the abstracts is as follows: 1.—AGRICULTURE AND HORTICULTURE. 2.—FOOD; also appropriate Microbiological Processes; Essential Oils. 3.—SANITATION, including Water; Sewage; Atmospheric Pollution, etc. 4.—APPARATUS AND UNCLASSIFIED.

INDEX OF AUTHORS' NAMES

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JOURNAL OF THE SCIENCE OF FOOD AND AGRICULTURE

ABSTRACTS

MARCH, 1965

I.—AGRICULTURE AND HORTICULTURE

General: Soils and Fertilisers

Fertility of soils and elements of rural sociology in Africa south of the Sahara. J. Lebrun and P. P. Lefèvre (*Enquêtes bibliograph., CEDESA*, 1964, 182 pp.).—A bibliography with 1413 references from many publications in alphabetical list of authors with a geographical index. Topics dealt with include systems of exploitation of the soil, various pedological aspects, productivity factors in soil, methods of culture, sylviculture, agricultural education of the indigenous population, etc. H. S. R.

Alluvial black earth soils. E. Ostendorff and F. Beinroth (*Z. PflErnähr. Düng.*, 1964, **106,** 97—107).—Alluvial black earths (**I**) are described and the climatic, geological and hydrological conditions giving rise to their formation in Donauried are detailed. As the water table falls in marsh and bog areas **I** form. Where woodland penetrates, brown-earth-like forms of **I** appear and eventually the soil is decalcified and degraded. M. Long.

Some physical and chemical properties of pumice soils in Oregon. C. T. Youngberg and C. T. Dyrness (*Soil Sci.*, 1964, **97,** 391—399).—Analyses, water relationships and fertility data of a no. of pumice soils are recorded. The average particle density in these soils was 2·61 and bulk densities range from 0·5 to 0·9 g/cc. The high internal porosity of pumice particles influences chemical as well as physical properties of the soils. In dispersing samples for mechanical analysis, >30 min. shaking is advised to avoid breakdown of individual particles, and the first hydrometer reading is preferably taken at 70 rather than at 40 sec. Unsaturated flow in the soils is very slow; the '15 atm. %' is consistently below the wilting point (sunflower method). The moisture-holding capacity is generally high, much of the available moisture being within the 'readily available' range. The level of fertility of pumice soils is satisfactory if expressed in conventional terms, but is low if calculated as the amounts of the nutrients per acre in the profile.
A. G. Pollard.

An infiltration method of measuring the hydraulic conductivity of unsaturated porous materials. E. G. Youngs (*Soil Sci.*, 1964, **97,** 307—311).—Two methods were used for obtaining the necessary unsaturated conditions in a column of the porous material; one maintained, at the surface, a suction level through a porous membrane, the other maintained the air pressure in the dry porous material at a pressure greater than atmospheric with the surface kept saturated. The porous material used was slate dust and the columns were subsequently broken up into sections so that a moisture profile could be determined. After a sufficient lapse of time both infiltration methods gave satisfactory results. T. G. Morris.

Soil core water permeameter for field use. R. D. Flannery and D. Kirkham. (*Soil Sci.*, 1964, **97,** 233—241).—The construction of the permeameter is described. Glass jars supported in a wooden rack carry the funnels in which the core of soil in its sampler is placed. Water is added to the soil under certain specified conditions and the volume collected in the jar in known time is measured. The hydraulic conductivity formulae from which results may be calculated are given with full details for making the measurements.
T. G. Morris.

Influence of installation depth on infiltration from unbuffered cylinder infiltrometers. H. Shull (*Soil Sci.*, 1964, **97,** 279—280).—It was shown that depth of installation caused a highly significant difference in measured cylinder infiltration. This effect appeared soon after infiltration started and it continued for at least 1000 min.
T. G. Morris.

Subdrainage in heavy soils: theoretical considerations. H. Fukuda (*Soil Sci.*, 1964, **97,** 281—285).—A discussion.
T. G. Morris.

Surface area measurements of soils. II. Surface area from a single point on the water isotherm. B. R. Puri and K. Murari (*Soil Sci.*, 1964, **97,** 341—343).—It is shown that the specific surface area of a soil is related to the amount of water absorbed per g. by the soil when in equilibrium with 53% R.H. by the equation: Specific surface area = (amount of water $\times$ N $\times$ A) 18 m.²/g., where N is Avogadro's number, $10^{23} \times 6{\cdot}025$, and A is the area occupied by 1 mol. of water (10·8Å²). T. G. Morris.

Relative erodibility of three loess-derived soils in South-western Iowa. B. L. Schmidt, W. D. Schrader and W. C. Moldenhauer (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 570—574).—Field measurements and org. C. analyses indicated a significant decrease in surface soil depth with slope at three sites (increasing profile development and clay content) in both virgin and cultivated soils. At one site in which long-term erosion was least simulated rainfall studies showed higher infiltration and lower rates of soil loss.
A. H. Cornfield.

Effects of ridges on erosion of soil by wind. D. V. Armbrust, W. S. Chepil and F. H. Siddoway (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 557—560).—Smooth surfaces and ridges 1·3—20·3 cm high in sand and simulated cultivated soil were exposed to wind velocities ranging from 47 to 83 m.p.h. under laboratory conditions. Ridges 5·1 to 10·2 cm. high showed little erosion since soil particles were trapped between the ridges and average wind velocity was reduced. Ridges less than 5·1 cm. high were less effective in reducing erosion. Extensive erosion on ridges higher than 10·2 cm. occurred due to increased wind velocity at the ridge crests and increased wind eddying. A. H. Cornfield.

Soil moisture relationship in two Flint Hills range sites under different management practices. V. V. Mohda (*Dissert. Abstr.*, 1964, **25,** 1451—1452).—In an upland area (*U*) Al tubes were installed to give access to 5 ft. below the surface; similar tubes were set up in a clay-pan area (*P*) to a depth of 3 ft. Soil moisture contents were measured (neutron technique) at 1 ft. intervals of depth over a period of 2 years. Comparison was made between light and heavy stocking and also of early, mid- and late spring burning in respect of the amount and distribution of water in the soils. Moisture contents diminished with advance of the growing season fluctuations being greatest in the surface 1—2 ft. In *U* soil moisture contents diminished with increase in stocking density and with early spring burning. With moderate stocking differences in moisture content did not follow the same trend. On *P* no significant differences in soil moisture or org. matter contents could be associated with the different treatments. Average moisture contents in the first ft. depth were similar in *U* and *P*; in the second ft. that of *U* exceeded that of *P* by an average of 0·4 in.; in the third ft. values for *U* averaged 0·68 above those for *P*. In *P* water penetrated to the third ft. only after heavy rain, e.g., as in autumn. A. G. Pollard.

Effect of soil compaction on plant growth and nutrient uptake; a technique to study its mechanism. G. S. Murty (*Dissert. Abstr.*, 1964, **25,** 1452—1453).—Sunflower plants were grown in pots of a silt loam compacted to different degrees; growth and uptakes of N, P, K and Ca were examined at intervals. Root activity, as shown by use of ^{86}Rb, diminished with rise in soil compaction but increased with soil aeration. Fertiliser treatment increased root activity only in the most heavily compacted soil. Top growth was severely restricted by heavy compaction (to bulk density of 1·60 g./c.c.) but was little affected by more moderate compaction. Aeration of the soil increased top growth significantly only in the later growth period and particularly, in the highly compacted soil. Ill effects of compaction may be compensated in part by forced aeration and may be more than compensated by fertiliser use. The % N in the plants decreased with soil compaction in the early stages but increased later, notably after aeration of soil or application of fertiliser. The % P in the plants was similarly affected by soil compaction but was increased by aeration when P fertiliser was given but decreased when this was withheld. Soil compaction lowered the K uptake in early growth but increased it later under high compaction when no fertiliser was given and to a smaller extent when fertiliser was applied. A. G. Pollard.

Effect of sanding non-irrigated clay soil on sugar cane yield. L. H. Lee and Y. S. Chan (*Rep. Taiwan Sug. Exp. Sta.*, 1964, No. 34, 31—55).—The ploughing-in of sand into the top layer of non-irrigated clay soil is regarded as more practical and economical than liming, chalking, adding org. matter or using artificial soil conditioners to improve the condition of certain clay soils under sugar cane in Formosa. (From English summary.) W. Elstow.

Combining surface mulches and periodic water applications for reclaiming saline soils. D. L. Carter and C. D. Fanning (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 564—567).—Water applied by periodic sprinkling of surface-mulched soil resulted in greater salt removal and higher leaching efficiency than did either flooding or periodic sprinkling of bare soil. Cotton bur and chopped shrubbery mulches were equally effective. Salts were leached down to 5 ft. when mulches were applied and only to 3 ft. when they were not applied. The effect of the mulches is probably due to reduced evaporative loss of water, resulting in less upward movement of water and salts. A. H. CORNFIELD.

Salt accumulation and salt distribution as an indicator of evaporation from fallow soils. E. J. Doering, R. C. Reeve and K. R. Stockinger (*Soil Sci.*, 1964, **97**, 312—319).—Two plots on a silty clay loam were used. One was leached with 152 cm. of irrigation water and the other was untreated. Subsequently the soil in the plots was sampled in increments to a depth of 150 cm. The samples were analysed for moisture, Cl^- concn., electrical conductivity and total salt concn. The initial concn. of Cl^- were greater in the unleached than in the leached plots. Near the surface the concn. increased with time for both plots. Below 30 cm. the concn. increased with time for the leached plot and decreased with time for the unleached, indicating that on the leached plot salts diffuse downwards but on the unleached salts are removed from the deeper layers faster than they are being diffused downwards. Evaporation rates were calculated from the salt accumulation measurements on the top 30 cm. using an equation derived theoretically. These rates were compared with those obtained using an evaporimeter. The capacity of the soil to transmit water upwards and not the evaporative conditions at the surface, controlled the rate of evaporation. T. G. MORRIS.

Microrelief influences in a saline area of ancient glacial Lake Agassiz. II. On shallow ground water. L. C. Benz, F. M. Sandoval, R. H. Mickelson and E. J. George (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 567—570).—The ridge–depression microrelief in a saline area caused impounding of precipitation in the depressions. Ground water in the depressions was relatively non-saline to a depth of 20 ft. compared with adjacent ridges, which had highly saline ground water. Hydraulic conductivity was much higher in the depressions than in the ridges. A. H. CORNFIELD.

Effect of discrete ped density on maize root penetration in a Planosol. W. M. Edwards, J. B. Fehrenbacher and J. P. Vavra (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 560—564).—Large maize roots in a Planosol silt loam were confined to the larger spaces between peds, but many medium and small roots penetrated the discrete peds in the claypan B horizon directly under a maize hill. Discrete peds were penetrated only when their bulk density was less than about 1·8 g./c.c. Mixing the A and B horizons reduced the root restrictive effect of the A2 horizon. A. H. CORNFIELD.

Diffusion of alkali chlorides in clay–water systems: A discussion of a Report by G. F. Dutt and P. F. Low. I. Comment on the Dutt–Low Report. G. H. Bolt and F. A. M. de Haan (*Soil Sci.*, 1964, **97**, 344—346). **II. Response to Bolt–de Haan comment.** P. F. Low and G. R. Dutt (*Soil Sci.*, 1964, **97**, 346—349). T. G. MORRIS.

Extraction of clay–water systems. J. V. Lagerwerff (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 502—506).—Theoretically predicted extract concn., assuming a constant salt-sieving factor, agreed with experimental results obtained by pressure extraction of Na-montmorillonite suspensions. A greater pore size of the membrane caused a stronger salt-sieving effect. Results with Na–Ca systems showed a gradual increase of the concn. ratio $(Na/Ca^{-0·5})$ of the extract. A. H. CORNFIELD.

Rapid method for estimating exchangeable hydrogen and exchange capacity in soils of the moist tropics. K. R. Middleton and D. R. Westgarth (*Soil Sci.*, 1964, **97**, 221—228).—Air-dried soil (2 mm.) is dried in a vac. desiccator overnight. An aliquot (1—5 g. depending on the expected exchange capacity) is shaken for 20 min. with 50 ml. of aq. 0·451% $Ba(NO_3)_2$, and a similar aliquot is shaken with aq. 0·441% Ba acetate in centrifuge tubes which are centrifuged and Ba is precipitated as chromate from a 10-ml. aliquot. in presence of NH_4NO_3. After 3 h. the contents of the tubes are filtered into a 50-ml. flask containing 2·5 ml. of 25% H_3PO_4. The ppt. is washed and filtrate and washings and diluted to 50 ml. and the optical density (O.D.) determined using a violet filter. The O.D. is a measure of the Ba absorbed by the soil, and may be calculated from a calibration curve. The pH (glass electrode) of the supernatant solution after the initial Ba treatments is also measured and then the supernatants are titrated with 0·02N-NaOH to measure the *HS* released from the soil by the Ba treatment. The results are extrapolated to pH 7. A linear relationship exists between pH and exchange capcity and therefore between pH and exchangeable H. The reproducibility of this method for exchange capacity is of the same order as that of the standard method. T. G. MORRIS.

Cationic activities in clay suspensions and equilibrium dialysates. E. O. McLean and R. E. Franklin, jun. (*Soil Sci.*, 1964, **97**, 260—267).—Bentonite and illite suspensions were prepared with various saturations of Ca and K by adding the metal hydroxides to the hydrogen clays. Equal vol. of suspensions and water were separated by a cellulose membrane (average pore diameter 48 Å). The various systems were equilibrated for $\leqslant$17 days with shaking at intervals and then the activities of both Ca and K in the suspensions and dialysates were determined by the clay membrane electrode technique. Concn. of Ca and K were measured by flame photometer. In some tests the suspensions and dialysates were vigorously aerated with air of high, medium or low-level CO_2 content. The effect of CO_2 aeration was marked. With low-level CO_2 (essentially nil) the concn. of Ca in the dialysate was 0·4% of that absorbed on the clay. Normal air increased this 4-fold and air enriched with CO_2 (10%) increased this to 8 times that of the low-level CO_2. T. G. MORRIS.

Potassium exchange as affected by cation size, pH and mineral structure. C. I. Rich and W. R. Black (*Soil Sci.*, 1964, **97**, 384—390).—In 14 acid soils examined neutral N-NH_4OAc displaced K more effectively than did N-$Mg(OAc)_2$; $MgCl_2$ was as effective as N-NH_4OAc. An explanation of the data is based on the hypothesis that exchangeable K is not distributed over the entire exchange material, but that a portion is held at wedge-shaped interlayer spaces of partly 'opened' micas and of partly 'closed' vermiculite and montmorillonite. K thus held is exchangeable quickly only by small ions, e.g. NH_4^+ or H^+ by a process of proton or H-bond transfer. In such soils fixation of K^+ and NH_4^+ is inhibited by hydroxy-Al and -Fe interlayer 'props' (cf. C. I. Rich, *Proc. Soil Sci. Soc. Amer.*, 1960, **24**, 26). A. G. POLLARD.

Potassium–calcium exchange equilibria in soils: specific absorption sites for potassium. P. Beckett (*Soil Sci.*, 1964, **97**, 376—383).—The exchange reaction was examined in a loamy sand soil in which the small clay fraction consisted largely of montmorillonite. Equilibrium between K^+ and Ca^{2+} held on the exchange surfaces in field soils accords with equations established by Gapon (*J. gen. Chem., USSR*, 1933, **3**, 144) and by Eriksson (*Soil Sci.*, 1952, **74**, 103), only when the soil contains a certain amount of exchangeable K already. The equilibrium involving this initial quantity of exchangeable K is best described by an equation of the type of the Langmuir adsorption isotherm. These and other considerations together with some experimental data indicate that a portion of the exchangeable K in soil is held on sites having a specific binding power for K. A. G. POLLARD.

Effects of wash solvents on cation-exchange capacity measurements. C. R. Frink (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 506—511).—The cation-exchange capacity (CEC) of a cation-exchange resin was similar irrespective of the cation (Na, Ca or Al in N solution) of the saturating solution or whether water or 8 different org. solvents were used to remove excess saturating solution. The CEC of montmorillonite varied with the solvent used; low results with solvents of high dielectric constant could be avoided by using high-speed centrifugation; high results with acetone, t-butanol and dioxan were due to salt retention even after long washing. The CEC of a muck soil saturated with NH_4OAc increased with decreasing dielectric constant of the solvent used to remove excess salt. MeOH and ethylene glycol, in particular, removed more org. matter than did the other solvents. The use of 0·001N solution of the saturating salt, instead of an org. solvent, with a correction for occluded salt gave satisfactory results for the CEC of clays. A. H. CORNFIELD.

Fate of exchangeable iron in acid clay systems. G. W. Thomas and N. T. Coleman (*Soil Sci.*, 1964, **97**, 229—232).—The hydrolysis of Fe^{3+} sorbed on to various clays and soils has been studied using N-chloride salts as replacing agents. Clays used were montmorillorite, hectorite, nontrorite and vermiculite and several clay soils. All materials were saturated with Fe^{3+} and the cation-exchange capacities were determined using N-$MgCl_2$ followed by washing and then displacing the Mg with Na and measuring the Mg. Exchangeable cations were replaced by either N-NaCl or N-$CaCl_2$ and the amount of Fe in solution estimated colorimetrically. Total replaced acidity was determined by titrating the salt leachate with NaOH to pH 8. When the potentiometric curve was plotted the acidity due to Fe^{3+} could be identified. In montmorillonite approximately 15% of the exchangeable acidity was present as H^+ and Al^{3+}. In hectorite appreciable amounts of H and Mg were displaced from Fe–clay. No Al was found. Vermiculite was impossible to saturate fully with Fe and loss of exchangeable Fe and the appearance of Mg was very rapid. Hydrolysis of Fe was thought to occur readily at normal temp. T. G. MORRIS.

Influence of sorbed hydroxyl and sulphate on neutralisation of soil acidity. A. Mehlich (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 492—496).—Soils were treated with anion-exchange resin to contain sorbed OH^- or SO_4^{2-} and then equilibrated with $Ca(OH)_2$ in increasing amounts. The permanent charge, due mainly to Al^{3+} in mineral soils, was first neutralised, and this was followed by simultaneous neutralisation of variable charge acidity and anion-exchange acidity. Variable charge probably arises from covalently bonded H of aquo groups in the co-ordination sphere of hydrated Al and Fe^{3+}. Anion-exchange acidity derives from acid radicals substituted for OH^- in the co-ordination sphere of Al and Fe^{3+} associated with clay surfaces or in hydrous oxide crystals. Anion-exchange acidity may constitute an appreciable part of the total acidity of Red-Yellow Podsolic and Reddish-Brown Lateritic soils.
A. H. CORNFIELD.

Mineralogical examination of arid-land soils. B. L. McNeal and T. Sansoterra (*Soil Sci.*, 1964, **97**, 367—375).—The investigation aims to establish criteria for determining the approximate layer-silicate composition, primarily of the fine ($< 2\mu$) soil fraction and to correlate mineralogical composition with soil properties, particularly in arid soils. The scheme of analysis involves determination of mica by means of its K content, of quartz + feldspar as residue after pyrosulphate fusion, of amorphous constituents from the SiO_2 and Al_2O_3 dissolved by aq. NaOH after heating at 100°, and of Kaolinite from the SiO_2 and Al_2O_3 sol. in NaOH after heating at 500°. Montmorillonite, vermiculite and chlorite are calculated from simultaneous equations involving measurements of surface area, cation-exchange capacity, and hydroxyl-water loss. Details of the methods and of the interpretation of results obtained with a no. of arid soils are recorded together with *X*-ray diffraction patterns of the $< 2\mu$ fractions of the soils for comparative purposes.
A. G. POLLARD.

Krilium adsorption by clay minerals and their derivatives. S. K. De and R. K. Jain (*J. Indian Chem. Soc.*, 1964, **41**, 379—383).—The absorption of Krilium (hydrolysed polyacrylonitrile) (**I**) by a number of clays in their H^+ and Ca^{2+} forms is examined. The adsorptive capacities of the clays and their H- and Ca-forms decreased in the order: montmorillonite, vermiculite, attapulgite, biotite, kaolinite and pyrophyllite. The Ca deriv. showed the greatest, and the H deriv. the least, adsorptive capacity. The adsorption values did not obey the Freundlich or Langmuir equations which suggests that the association of **I** with the clays is not adsorptive, but involves other factors. Possibly the CN groups of **I** form complexes with metals in the clay minerals; thus **I** will be more beneficial as a soil conditioner in the presence of Ca salts.
J. I. M. JONES.

pH–% base saturation relationships of soils. J. S. Clark and R. G. Hill (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 490—492).—The curves obtained by plotting pH − 0·5 p(Ca + Mg) against % base saturation, calculated from the ratio of exchangeable $Ca^{2+} + Mg^{2+}$ to the sum of Ca^{2+}, Mg^{2+}, Al^{3+} and H^+ extracted from soils by N-KCl had the characteristic shapes of clay titration curves. Values for some montmorillonitic soils agreed closely with the theoretical curve for Al-bentonite. Podsolic and glei soils had lime potential values higher than those for montmorillonite soils at the same % base saturation.
A. H. CORNFIELD.

Influence of low temperature and various concentrations of ammonium nitrate on nitrification in acid soils. O. E. Anderson and F. C. Boswell (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 525—529).—Nitrification of NH_4^+-N 100 p.p.m. added to acid soils (pH having been increased to 6—6·4 by liming), was completely suppressed or delayed for several weeks in the case of sandy soils incubated at 5·6°. In a clay loam nitrification was severely retarded at 2·8°, but was rapid at 8·3° after a 6-week delay period. Nitrification in the clay loam at 8·3° was more rapid than in a loamy sand at 32·2°. When NH_4^+-N, 200—400 p.p.m. was added nitrification was completely suppressed or delayed in all soils at low temp., and in the sandy soil nitrification was delayed even at 32·2°. The depressive effect of low temp. and addition of NH_4^+ was most apparent in the loamy sand, intermediate in the sandy loams, and least in the clay loam.
A. H. CORNFIELD.

Soil absorption of atmospheric ammonia. B. A. Malo and E. R. Purvis (*Soil Sci.*, 1964, **97**, 242—247).—Rainwater samples were collected and NH_3 was determined by Nesslerisation. Rate of soil absorption of NH_3 was also measured by exposing air-dried 2 mm. soil in dishes for 24 hours. Absorbed NH_3 was extracted from the soils with 0·5% HCl; and NH_3 in the extract was estimated. Absorption of NH_3 by Whatman No. 2 filter paper and by soil from atm. of known, controlled NH_3 content was also determined. In New Jersey the NH_3 content of rainwater was significantly higher in June and Nov., which were relatively dry months. Daily rates of NH_3 absorption from the air varied from 0·05 lb. of N in a sand to 0·2 lb. per acre in a sandy loam. N from the atm. is believed to have been a factor in the production of high maize yields in the absence of fertilisation.
T. G. MORRIS.

Effect of period of air-dry storage of soils on the subsequent accumulation of mineral nitrogen during incubation. A. H. Cornfield (*Plant & Soil*, 1964, **20**, 260—264).—The accumulation of NH_4^+- and NO_3^--N during incubation, under standard conditions, of 9 mineral soils (N 0·099—0·393%) was determined following 1 week and 12 weeks of air-dry storage. NH_4^+-N accumulation was usually slightly greater whilst NO_3^--N accumulation was usually less during incubation after 12 weeks than after 1 week of air-dry storage. However, for the soils as a whole, there were only small differences in either constituent accumulating during incubation due to period of air-dry storage. Total mineral N accumulation (NH_4^+- + NO_3^--N) was very similar for 4 of the soils, was lower for 3 of the soils, and was higher for 2 of the soils during incubation following 12 weeks than following 1 week of air-dry storage. The average values for the soils as a whole were only slightly different between the two periods of air-dry storage.
A. H. CORNFIELD.

Effects of ionising radiation on soils and subsequent crop growth. H. J. M. Bowen and P. A. Cawse (*Soil Sci.*, 1964, **97**, 252—259).—Samples of two loam soils were irradiated at a range of doses from 0·001 to 60 Mrads, and then planted to lettuce, barley or flax. After harvest the crops were analysed. Irradiated soils dried out more slowly and had a reduced percolation rate, compared with controls. Other physical properties were little affected but exchangeable N increased. This N was present in different forms according to whether the soil had received a sterilising dose (2 Mrad) or not. With < 2 Mrads NH_3 release was small and NO_3^--N was high but if the dose was > 2 Mrads NH_3 release increased 8—10-fold. The amount of N produced on irradiation depended on the moisture content during the post-irradiation period. In general NO_3^--N increased with moisture content for the same level of irradiation but NH_3-N was little affected. Saturation of the soils with CO_2, and irradiation under CO_2, inhibited the production of NO_3^--N and NH_3-N; boiling the soil with EtOH immediately before irradiation largely inhibited the production of both forms of N. Growth of crops was stimulated by irradiation of the soil, probably because of the increased N levels.
T. G. MORRIS.

Forms of phosphate in Lakeland fine sand after six years of heavy phosphate and lime applications. J. G. A. Fiskell and W. F. Spencer (*Soil Sci.*, 1964, **97**, 320—327).—Previously unfertilised Lakeland fine sand was treated annually since 1951 with superphosphate and lime. In 1957 soil samples at different depths were analysed for total P, and various forms of extractable P. It was found that the added P was retained in the profile, the amount present increasing with the rate of application. Lime applications did not affect the native P level but it did affect the movement of P in the soil. At high rates of application some P passed out of the 0—48-in. levels. The results obtained by selective dissolution using acid NH_4OAc, alkaline-fluoride, aq. NaOH and dil. H_2SO_4 are discussed with respect of the forms of P present.
T. G. MORRIS

Isotopic dilution as a method of relating phosphorus retention to availability of phosphorus in soils. D. E. Baker (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 511—517).—Active solid phase P (ASP) as determined by isotopic dilution was an index of the amount of 'available P' contained in a soil. A constant proportion of the ASP was not in solution or removed from all soils by any of the extracting reagents. The % ASP removed by all extractants was affected by the rate of added P. Providing native ASP was in equilibrium with added ^{32}P, the % ASP removed by Bray's No. 1 extractant (0·025N-HCl–0·03N-NH_4F) from each soil type was an index of its retention characteristics.
A. H. CORNFIELD.

Partially acidulated rock phosphate as a source of phosphorus to plants. I. Growth chamber studies. E. O. McLean and R. W. Wheeler (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 545—550).—Millet and lucerne gave as good yields and absorbed as much P when the rock phosphate applied had been treated with 10% as compared with 100% of the amount of H_3PO_4 required to convert it to triple superphosphate. In sand culture maize seedlings absorbed amounts of P in proportion to the water solubility of added rock phosphate and a no. of PO_4^{3-} compounds, but in soil P uptake was similar regardless of the P source. Results are discussed in relation to PO_4^{3-} reaction products occurring in soils when PO_4^{3-} materials are added.
A. H. CORNFIELD.

Phosphorus movement in a calcareous soil. I. Predominance of organic forms of phosphorus in phosphorus movement. R. J. Hannapel, W. H. Fuller, S. Bosma and J. S. Bullock (*Soil Sci.*, 1964, **97**, 350—357).—Columns of 7 in. of an air-dry sandy loam were covered with a 1-in. layer of the same soil which had been mixed with plant residues (bean and barley) labelled with ^{32}P and incubated at 37° for 7 days after moistening to the moisture equiv.

Deionised water was added to the top of the soil columns and the displaced soil solution collected in HCl of pH < 1. Total, inorg. P and Ca content of the solutions were determined. Additions of sucrose and phosphate were made to other similarly treated columns. All the org. matter treatments increased the amount of total P movement, because of movement of org. P in the soil, additions of inorg. P (unless accompanied by additions of org. matter) did not increase the movement of P through the soil. By using the ^{32}P-labelled material it was also demonstrated that a large proportion of the org. P came from the mobilisation by microbial action of the native soil P, the org. matter added as sucrose or plant residue being important as a substrate for the soil organisms. T. G. MORRIS.

Exchangeable aluminium, acidity and water-soluble phosphate in some acid Minnesota soils as affected by added potassium monobasic phosphate and calcium hydroxide. Samuel Kwaku Takyi (*Dissert. Abstr.*, 1964, **25,** 1455).—Soils under examination were characterised by: pH (N-KCl) 4·0—5·6; water-sol. P, low; NH_4F-sol. P, medium-high; exchangeable K, high; extractable Fe, low; exchangeable Al, varying over a wide range. 'Valence values' for Al ions indicated its presence as Al^{3+} and $Al(OH)^{2+}$. Incubation of the more acid soils with $Ca(OH)_2$ (**I**) greatly reduced exchangeable acidity and exchangeable Al and increased water-sol. P. Treatment of soils with KH_2PO_4 (**II**) slightly increased pH and water-sol. P, and lowered exchange acidity and exchangeable Al. When **II** and **I** were added simultaneously to soil the action of **II** was generally overshadowed by that of **I**. Probably, **I** did not affect the action of **II** directly but pptd. the exchangeable Al which might otherwise have 'fixed' part of the added **II**. A. G. POLLARD.

Phosphate potentials of soils. II. Microbial effects. R. E. White (*Plant & Soil*, 1964, **20,** 184—193).—When samples of Upper Greensand soil were shaken with dil. aq. $CaCl_2$ containing PO_4^{3-}, microbial activity was sufficiently stimulated to alter the measured value of Schofield's phosphate potential. The effect was more rapid and more marked for air-dried than for field-moist soils. However, the effect on the potential was not significant during the first 2—4 h. of shaking a moist sample and probably not significant during the first 0·5—1 h. of shaking an air-dried sample. A. H. CORNFIELD.

Self-diffusion of calcium-45 into certain carbonates. N. Lahav and G. H. Bolt (*Soil Sci.*, 1964, **97,** 293—299).—An equation describing the self-diffusion process involved was derived and compared with results obtained. Finely ground calcite, dolomite, pptd. $CaCO_3$ and a commercial lime were used. A suspension of the material was treated with a known amount of labelled Ca solution and shaken. The ^{45}Ca activity was measured immediately and then after known times the activity of the supernatant solution obtained by centrifuging was measured. The amount of ^{45}Ca isotopically exchangeable was found from a modification of the equation. The experimental results obtained with all but the commercial lime were in agreement with the theoretical diffusion equation. Grinding the material did not affect the results to any degree. There was a lack of self-diffusion in the commercial lime. Probably the particles were coated with silicate; in experimental tests silicate added to $CaCO_3$ inhibited self-diffusion. T. G. MORRIS.

Boron in saline and non-saline soils in South-eastern Saskatchewan. C. H. E. Werkhoven (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 542—545).—The hot-water sol. B content (*Soil Sci.*, 1944, **57,** 25) of salt-affected chernozemic soils ranged from 0·1 to 2·0 p.p.m. The B % in wheat grown in six soils receiving B (1·1—2·2 p.p.m.) as H_3BO_3 increased with level of applied B. The B% in lucerne was not affected under similar conditions. The lower level of applied B increased yields slightly in some cases. The dry-matter yields of both crops decreased linearly with application of Mg (200—400 p.p.m.). Na^+ applications decreased B % in wheat and yields of lucerne, whilst added Ca and Mg had no effect on B% in either species. For a specific soil type there was a highly significant correlation ($r = 0{\cdot}689$) between B % in wheat and hot-water-sol. B, but no such significant correlation occurred for lucerne. A. H. CORNFIELD.

Status and transformation of sulphur in Mississippi soils. L. E. Nelson (*Soil Sci.*, 1964, **97,** 300—306).—The org. S status of some soils has been examined. pH, total S, combined org. S, oxidisable inorg. S and sol. SO_4^{2-}-S were determined as well as total org. N. S ranged from 64 to 353 lb./acre and values were highly correlated with org. C, total N and total S. The average org. C : N : S ratio was 126 : 10 : 1·1. When incubated in a moist state in closed polythene bags at room temp. org. S was mineralised to SO_4^{2-}-S, the total amount mineralised being related to the level of org. S in the soil, in most cases. T. G. MORRIS.

Anionic effects on sulphate absorption by soils. T. T. Chao (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 581—583).—Of the inorg. anions studied PO_4^{3-}, MoO_4^{2-} and F^- had the greatest effect in reducing adsorption of SO_4^{2-} by soils. Of the org. anions oxalate, tartrate, gluconate, malate and thiocyanate showed the greatest effect. Adsorption of SO_4^{2-} was decreased by increasing concn. of OH^- and HCO_3^-, but this was attributable mainly to increasing pH. The extent of depression of SO_4^{2-} adsorption by the org. anions was not related to length of C chain or no. of functional -OH or -COOH groups present. A. H. CORNFIELD.

Profile of natural radioactivity in Hartsells loam. J. D. Burton, W. B. Guenther and H. M. Owen (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 500—501).—Radioactivity of samples of a loam profile under forest was higher one week than one day after collection. Distribution of K and initial radioactivity were more closely related to clay + silt than to clay content. A. H. CORNFIELD.

Wheat stubble management. I. Influence on physical properties of a Chernozem soil. R. E. Ramig and A. P. Mazurak (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 554—557).—Twelve years of subsurface tillage or one-way disking of a Chernozem under a wheat–fallow system increased water-stable aggregates to a greater extent than did ploughing or burning and ploughing. On wheat plots the ratio of water entry after 10 and 120 min. was higher under subsurface tillage and ploughing treatments than under the other treatments. A. H. CORNFIELD.

Infra-red and differential analysis of humic acid samples from various soil types, worm casts and streptomycetes. H. W. Scharpenseel, E. König and E. Menthe (*Z. PflErnähr. Düng.*, 1964, **106,** 134—160).—Humic and fulvic acid prep. from different sources can be separated on the systematic basis of an extensive spectra characterisation. Only the separation of the same monomeric structural units into particle size fractions takes place in radio-column chromatography. Differential thermal analysis of humic acid prep. from various sources, of hydrolysis residues and of prep. carried out in O_2 show the same peaks during the course of pyrolysis. Interpretation of the heating curves appears uncertain with regard to pedological type, grey or brown humic acids, mull, moder or raw humus. M. LONG.

Pyrolysis of soil organic matter. M. Schnitzer and I. Hoffman (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 520—525).—Organic matter extracted from the O-2 and Bh horizons of a podsol were heated to temp. ranging from 150 to 540°. The C content of the materials increased and the O content decreased with rising temp.; at 540° both materials contained the same levels of C and H and no O. Some of the N and S in the materials were present even after heating to 540°. Phenolic OH groups were more stable than COOH groups but both were eliminated between 250° and 400°. Both types of groups were more stable in the org. matter from the Bh than in that from the O-2 horizon. Differential thermogravimetric curves showed that functional groups were eliminated at low temp., whilst the nuclei decomposed at high temp. The main reactions governing the pyrolysis of O-2 horizon org. matter were dehydrogenation up to 200°, decarboxylation and dehydration up to 250° and continuous dehydration up to 540°. Dehydration was the main reaction governing the pyrolysis of Bh org. matter. A. H. CORNFIELD.

Contribution of organic matter and clay to soil cation-exchange capacity as affected by the pH of the saturating solution. C. S. Helling, G. Chesters and R. B. Corey (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 517—520).—A buffered Ba^{2+} saturating solution ranging in pH from 2·5 to 8·0 was used to determine the cation-exchange capacity (CEC) of 60 soils of varying org. C and clay contents. The average CEC of org. matter increased from 36 to 213 mequiv. and of clay from 38 to 64 mequiv. per 100 g. as pH of the saturating solution increased. The mean contribution of org. matter to total soil CEC increased from 19% at pH 2·5 to 45% at pH 8·0. A. H. CORNFIELD.

Isolation and characterisation of cellulose from soil organic matter. U. C. Gupta and F. J. Sowden (*Soil Sci.*, 1964, **97,** 328—333).—The amount of cellulose that could be extracted from a variety of soils by Schweitzer's reagent has been determined and compared with amounts isolated by other methods. It was found that the cellulose values (glucose obtained by hydrolysis) by the Waksman proximate analysis were about four times higher than the values obtained by Schweitzer's reagent in the org. horizon of three of the soils, but nine times higher in a brown forest soil. The infra-red spectrum of the cellulose from a podsolic soil was very similar to that of filter paper but the cellulose isolated from a peat and a brown forest soil had a less similar spectrum. The isolated cellulose represented a very small proportion of the total org. matter. T. G. MORRIS.

Growth and activity of soil micro-organisms in glass micro-beads. I. Carbon dioxide evolution. J. F. Parr and A. G. Norman (*Soil Sci.*, 1964, **97,** 361—366).—Two sizes of glass beads (149 and 37 μ, dia.), from which surface alkali and extraneous colloidal matter had been removed, were used as media for studies of the growth and activities of soil micro-organisms. A technique is described for adding sub-

strate and inoculum to the sterilised beads and for the determination of CO_2 produced by the organisms. In comparative tests of the decomposition of glucose by a mixed culture of soil organisms and by a pure culture of *Bacillus subtilis*, CO_2 production was more rapid and more complete when the smaller beads were used. In corresponding trials using *Aspergillus terreus*, production of CO_2 was much more rapid and the total recovery of CO_2 was greater in the larger bead medium. A. G. POLLARD.

***Azotobacter* species in soil. III. Effects of artificial inoculation on crop yields.** M. E. Brown, S. K. Burlingham and R. M. Jackson (*Plant & Soil*, 1964, 1964, **20**, 194—214).—Although yields of many species in pot tests were increased by inoculation of seeds, roots or soil with *Azotobacter* cultures, these increases were not significant even at $P < 0{\cdot}1$, indicating large replicate variance. Significant yield increases often occurred when soils known to produce diseased crops were inoculated with *Azotobacter*. Many field tests on normal soils produced only two significant yield increases, due mainly to failure to establish the *Azotobacter* inoculum in the rhizosphere. Addition of mineral fertilisers or carbohydrates, varying soil moisture level and size and age of the *Azotobacter* inoculum did not influence the effects of inoculation. A. H. CORNFIELD.

Micro-organisms able to grow on certain aromatic compounds in normal and treated soils. D. J. S. Branson (*Dissert. Abstr.*, 1964, **25**, 1489—1490).—Counts of micro-organisms present in normal soils and in abnormal types, e.g., coal shale, clay soaked in crude oil or refined oil or containing creosote, are recorded. Of organisms examined, the no. able to utilise hydrocarbons and simple phenols was in the (decreasing) order; toluene (**I**), benzene (**II**), dodecane (**III**) (all at 0·1%), *o*-cresol (**IV**), (0·01), anthracene (**V**) 0·1), phenol (**VI**) (0·05), naphthalene (**VII**), (0·1), resorcinol (**VIII**), (0·1%). Rates of utilisation, as indicated by time to max. count, were in the (descending) order **V, II, VIII, IV** and **III, I, VII, VI.** With nearly all soils and hydrocarbons examined counts were in the order: actinomycetes > bacteria > moulds. Morphological variations in colonies caused by hydrocarbons and also some effects of prior treatment with hydrocarbons on the utilisation of others are examined. A. G. POLLARD.

Respiration studies on soil treated with hydrocarbons. A. L. Dobson and H. A. Wilson (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 536—539).—The respiration rate (O_2 uptake) of composite samples of oil-impregnated soils near oil wells was greater than that of similar oil-free soils, but the reverse was true when undisturbed cores were used. Addition of 2% crude oil, paraffin oil or pharmaceutical mineral oil to soil increased respiration rate to a greater extent than did addition of 10—20% of the oils. A. H. CORNFIELD.

Effect of shape on oxygen diffusion and aerobic respiration in soil aggregates. D. J. Greenwood and D. Goodman (Appendix: J. A. Nelder) (*J. Sci. Fd Agric.*, 1964, **15**, 781—790).—An equation that related the shape of irregular soil aggregates to their respiration rates and the O_2 concn. at their surfaces was verified experimentally by use of models of soil aggregates cut from agar containing yeast and glucose, and surrounded by different O_2 partial pressures. The respiration rates approx. fitted the equation when shape factors [surface area/(vol.)$^{\frac{1}{3}}$] were between 5·5 and 6·8. Sieved soil aggregates had shape factors independent of size and had values between 5·5 and 6·1. A method was developed for determining the surface area-to-vol. ratios of soil aggregates from measurements of the mean lengths of random chords through the aggregates. Experimental verification of two equations derived was obtained. E. M. J.

Mechanisms involved in soil aggregate stabilisation by fungi and bacteria. R. F. Harris, G. Chesters, O. N. Allen and O. J. Attoe (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 529—532).—Aggregate stabilisation was a function more of microbial synthesis of soil-binding substances than of no. of micro-organisms. Extensive production of mycelium and spores in soil aggregates by a fungus known to be capable of aggregate stabilisation did not necessarily result in an increase in the stability of the aggregates. Incubation with *Mucor silvaticus* under aerobic conditions resulted in the formation of water-stable aggregates >2 mm. at all stages of incubation. Aggregates stabilised under anaerobic conditions had a high proportion of aggregates 0·5—1 mm. and 1—2 mm. early in the incubation period. A relatively high proportion of the total water-stability of sucrose-amended aggregates, possessing an indigenous mixed microflora, was due to 0·5—2 mm. aggregates in the early stages of incubation, indicating that bacteria were more important than fungi in the initial stabilisation of these aggregates. A. H. CORNFIELD.

Determination of available plant nutrients in soils using chelating agents. E. Rauterberg (*Z. PflErnähr. Düng.*, 1964, **106**, 128—134).—Air-dried soil is extracted with 0·05M-NH_4-EDTA and the extract clarified with activated charcoal. P, Na, K, Ca, Mg, Fe and Al can be determined in this extract. P, Fe and Al concn. fall markedly whilst that of Ca rises as the pH of the solution rises from 4·0 to 6·0. M. LONG.

Segregation studies of dry blended fertiliser. L. J. Pircon, M. de la Puente and M. Sheth (*J. agric. Fd Chem.*, 1964, **12**, 357—362).—Blended fertilisers on handling often show marked non-homogeneity caused by segregation of the constituents. This is shown to be due to a 'friction factor', calculated from the angle of repose of conical piles of the individual ingredients. If the friction factor of all the individual ingredients is tailored to meet certain tolerances the tendency to segregation is markedly reduced. W. ELSTOW.

Minor element release from organic-nitrogen fertiliser materials in laboratory and field studies. J. G. Fiskell, P. H. Everett and S. J. Locascio (*J. agric. Fd Chem.*, 1964, **12**, 363—367).—In experiments with water melons and cucumbers on sandy soil very low in minor elements it would appear that the org. N fertilisers, e.g., activated sludge, N tankage, Peruvian guano and castor pomace, can act as significant sources of Cu, Zn and Mn. (11 references.) W. ELSTOW.

Ammoniation of triple superphosphate fertilisers with gaseous ammonia and with nitrogen solutions. R. F. Nunn, S. I. Srinivasan and L. F. Albright (*J. agric. Fd Chem.*, 1964, **12**, 351—357).—Factors affecting the ammoniation of triple superphosphate fertiliser in a rotary-drum reactor were studied. W. ELSTOW.

Ammoniation of fluidised triple superphosphate fertilisers. E. G. Shook, P. T. Shannon and L. F. Albright (*J. agric. Fd Chem.*, 1964, **12**, 347—351).—The ammoniation of triple superphosphate fertiliser on a fluidised bed reactor was studied. Triple superphosphate may ammoniate to between 7 and 8·5% NH_3 in 10 to 15 min. under easily controllable conditions. W. ELSTOW.

Phosphate rock solubilisation by repeated extractions with citrate solutions. W. M. Hoffman and H. J. Breen (*J. agric. Fd Chem.*, 1964, **12**, 344—346).—The potential value of phosphate rock may be estimated by any of the three solvents, neutral NH_4 citrate, 2% citric acid or alkaline NH_4 citrate. The ranking of the rocks was about the same whether based on a single extraction or the total of four consecutive extractions. The newer rock discoveries of Peruvian, North Carolina and sea bottom rocks compare favourably with the highly sol. rocks from Curacao and North Africa. W. ELSTOW.

Colorimetric determination of calcium in soil extracts. D. E. Peaslee (*Soil Sci.*, 1964, **97**, 248—251).—Glyoxal bis-(2 hydroxyanil) forms selectively a red complex with Ca in alkaline, alcoholic media, and this reaction is applied for determining Ca in soil extracts. Full details of the procedure are given. The absorbance of the colour is measured at 535 mμ with water as blank. The colour is stable for at least 20 min. and Beer's law is obeyed in the range 0—40 μg. of Ca. The optimum pH for the colour is 12. Virtually no interference is given in the determination of 0—2·6 p.p.m. of Ca by the presence of Mg, Al, Fe, Mn, Zn, Cu, Sr, PO_4^{3-} and SO_4^{2-}. T. G. MORRIS.

Modifications in the curcumin procedure for the determination of boron in soil extracts. A. S. Baker (*J. agric. Fd Chem.*, 1964, **12**, 367—370).—Some modifications, including a new extracting solvent, of the Dible *et al.* method (*Analyt. Chem.*, 1954, **26**, 418; J.S.F.A. Abstr., 1954, ii, 4) for the determination of trace B in soil are described. W. ELSTOW.

Making soil pastes for salinity analysis: a reproducible capillary procedure. D. E. Longenecker and P. J. Lyerly (*Soil Sci.*, 1964, **97**, 268—275).—Dry sieved soil is put into a Whatman No. 52 paper cup (high wet strength paper) and placed on blotting paper which covers a layer of fine sand contained in a trough. A water table is maintained at a depth of 5 (preferable) or 10 cm. below the sand surface. The soil in the cups is wetted by capillary absorption and essentially complete saturation takes 6—9 h. Regardless of soil texture water absorption patterns were very similar. The saturation extract from soils wetted by this method was compared with that obtained by the usual method. Differences in pH and electrical conductivity were slight. The level of exchangeable Na could be related to delays in wetting, those soils with < 15% wetted normally; those with >15% required longer. Particle size also had an effect. T. G. MORRIS.

Antibiotics in soils. VI. Determination of micro-quantities of antibiotics in soils. D. A. Soulides (*Soil Sci.*, 1964, **97**, 286—289).—Two methods of determination were used, the direct assay method (*Soil Sci.*, 1961, **91**, 94—99) and the extraction-isolation technique. In this the large sample of the soil is treated with buffer solution, the liquid separated and evaporated. The antibiotic is then dissolved out of the dry residue and bio-assayed. The technique was successful in isolating and estimating 0·3 μg. of carbomycin, 0·08 μg. of Terramycin, 0·02 μg. of Aureomycin and 0·15 units of bacitracin. T. G. MORRIS.

Promoting plant growth. Novacel and S. S. Amor (B.P. 938,170, 14.8.61. It., 12.8., 1.10., and 22.12.60, and 5.5.61).—There is claimed a method of promoting plant growth which consists of providing in the immediate vicinity of a growing seedling or plant or seed a layer or block of foamed org. plastic material (resin based on polyvinyl alcohol, cellulose or water-swelled cellulosic material) through or beneath which the roots may grow—the plastic material being hydrophilic and water-permeable. F. R. BASFORD.

Treatment of artificial fertilisers. Croxton & Garry Ltd. (Inventor: G. A. Rohan) (B.P. 935,999, 24.8.59).—Artificial fertiliser in granular or powder form is rendered non-caking by mixing with another powder (1—10 wt.-%) the particles of which have been coated with a water-repellent substance, e.g., a fatty acid (or a salt thereof), a silicone, and/or an aliphatic amine (or a salt thereof). The powder is preferably china clay. F. R. BASFORD.

Granulation of calcium nitrate melts containing ammonium nitrate of approximately the formula 5 $Ca(NO_3)_2$,$NH_4NO_3$10H_2O. Shell Internationale Research Mij N.V. (B.P. 934,510, 18.10.61. Neth., 20.10.60).—A water and NH_4NO_3 melt containing $Ca(NO_3)_2$, in which the wt.-% of NH_4NO_3 is $\geqslant$ 0·44 times the wt.-% of water, the latter being 12—17 (14—16) wt.-% of the melt (the N content of the melt being $\leqslant$15·5 wt.-%), is granulated by dropping the melt, divided into drops, into a mineral oil containing seed material, in such a way that the drops to be solidified finally pass a zone with a seed material content of 0·01—0·5 wt.-% and a max. temp. of 52° ($\leqslant$30° depending on the η of the oil), the seed material content of the oil being brought to the required value, or maintained at this value, by addition of $Ca(NO_3)_2$,$2H_2O$, $Ca(NO_3)_2$,$3H_2O$ or $Ca(NO_3)_2$,$4H_2O$. J. M. JACOBS.

Plant Physiology, Nutrition and Biochemistry

Effects of light on respiration during photosynthesis. E. B. Tregunna, G. Krotkov and C. D. Nelson (*Canad. J. Bot.*, 1964, **42**, 989—997).—Soya-bean, peperomia and maize leaves were studied. Initial rate of CO_2 production in the dark was affected by previous illumination in proportion to the capacity of the leaves for photosynthesis. Light accelerated CO_2 production in green soya-bean and peperomia leaves, but inhibited it in green maize leaves. There seems to be a close relationship between the photosynthetic apparatus of the leaves and the production of CO_2 both during and immediately following a period of illumination. (12 references.) E. G. BRICKELL.

Chemical adaptation of roots to physiological drought. M. N. A. El-Shourbagy (*Dissert. Abstr.*, 1964, **25**, 1513—1514).—The theory that salt inhibits the metabolic step essential to growth and that salt-tolerance may be induced by supplying the salt-inhibited reaction, was examined experimentally. Tomato plants were grown in culture media containing inhibitory concn. of sucrose or NaCl with and without various concn. of metabolites (amino-acids, vitamins, purines, org. acids, alkaloids). Roots tended to increase growth only when a mixture of 18 amino-acids was added to a medium containing inhibitory concn. of salt. Threonine, phenylalanine, tyrosine, tryptophan, arginine and cystine separately or in combination restored a significant amount of growth of salt-inhibited roots. The mechanism of salt tolerance is probably metabolic, in part, and possibly involves the salt effect on enzymes concerned in the synthesis of amino-acids or on other enzymes containing these amino-acids. A. G. POLLARD.

Biosynthesis of iron-porphyrins in roots in connexion with plant metabolism. B. A. Rubin, V. F. Gavrilenko and N. V. Guzhova (*Dokl. Akad. Nauk SSSR*, 1964, **156**, 961—963).—In studying biosynthesis of Fe-porphyrins, the dynamics of synthesis of protohaematin (**I**) were investigated in whole pea and maize plants and in isolated pea roots. Plants were grown in Knop nutrient mixture of pH 5·6. Root tips (1 cm. long) were cut from peas grown at 25° and each kept in 25 ml. of Bonner nutrient medium in darkness at 25° and examined after each 10 days up to 50 days. **I** was determined by measuring absorption max. of a pyridine–haemo chromogen solution at 420, 525 and 557 mμ. Synthesis of **I** continued and was intensified in isolated roots. Over periods up to 50 days 40—60% of **I** was concentrated in the roots. The formation of large quantities of **I**, which has exceptional catalytic activity, must affect the general metabolism of root systems and their influence in whole plant metabolism. Results show that one direction of influence is active participation of the roots in synthesis of Fe-porphyrin compounds. (13 references.) P. W. B. HARRISON.

Nitrogen nutrition in *Atriplex hastata*, L. R. L. Weston (*Plant & Soil*, 1964, **20**, 251—259).—A study was made to ascertain why *Atriplex hastata*, previously recorded as a nitrophilous plant, can colonise fly ash, which has a low N content. *Sinapsis arvensis*, a weed which does not grow on fly ash, served as a control plant. In solution culture the two species differed in their response to high and low levels of N, the growth rate of *S. arvensis* being much more severely affected at low N levels. *A. hastata* made very efficient use of its N supply. Both species grew better with NO_3^-- than with NH_4^+-N. Uptake of N was not influenced by pH (4—8) when only NO_3^- was present. When both forms of N were present, NO_3^- was absorbed preferentially at low pH and NH_4^+ at high pH. Properties of fly ash which render it a suitable medium for growth of *A. hastata* are discussed. A. H. CORNFIELD.

Interrelationships between potassium and magnesium absorption by oats (*Avena sativa* L.). F. Yoshida (*Versl. landbouwk. Onderz.*, 1964, No. 642, 103 pp.).—Published results and theoretical considerations concerned the influence of cations and anions on the absorption of Mg are reviewed. Water culture experiments are described in which the effects of variations in the relative and absolute concn. of K and Mg, and the pH of the solution were studied. The effects of the concn. of other nutrient ions were also studied. The results are considered in relation to the carrier and the adsorption theory. Evidence was obtained of the operation of two different mechanisms of uptake. (149 references.) P. S. ARUP.

Sulphur nutrition of plants. K. Schmalfuss (*Z. PflErnähr. Düng.*, 1964, **106**, 116—127).—The yield of oats, mustard and horse beans is not affected and that of green maize only slightly by the substitution of SO_4^{2-} for Cl^- fertilisers. The effect on beet, although the tendency is for yield to rise, is variable. Crop water content is lower with SO_4^{2-} than with Cl^- and inorg. S rises with SO_4^{2-} application, although org. S, based on dry matter and protein N, is largely constant. Since the S content of plants is high, it is assumed that S is obtained from the atmosphere. M. LONG.

Influence of foliar leaching on plant nutrition with special reference to root uptake, translocation and loss of calcium. R. A. Mecklenburg (*Dissert. Abstr.*, 1964, **25**, 1472—1473).—Squash plants containing root-absorbed ^{45}Ca, ^{32}P, ^{89}Sr or leaf-absorbed ^{14}C were leached with water and the leachates were fed back to the same or other plants. Such 'recycling' resulted in the redistribution of relatively 'immobile' nutrients, e.g., Ca from mature leaves to younger, rapidly growing plant organs. ^{89}Sr sprayed on potato foliage was leached by a mist of water, absorbed by roots and translocated to the tubers. ^{45}Ca which was leached from leaves and absorbed by roots while leaching was in progress, was more readily leached from the plants than was that which was root-absorbed prior to the leaching period. The sp. activity of ^{45}Ca in leaf leachates was similar to that in the exchangeable form in plants but differed from that in non-exchangeable forms or in the cell sap. Ca was apparently leached from exchange sites in the leaf but not from within cells. The mechanism of these effects is discussed. A. G. POLLARD.

Content of volatile acids in leaves and seeds of ripening oil-bearing plants. L. P. Zhdanova (*Dokl. Akad. Nauk SSSR*, 1964, **156**, 1229—1231).—Total org. acids and individual volatile fatty acids were determined in leaves and seeds (during ripening) of sunflower (**I**), poppy (**II**) and flax (**III**). Tables show (i) increase in oil in dry kernels and decrease in org. acid content of kernel, stem, leaf-stalk and leaf of **I** from 5—10 days before until 50 days after flowering; (ii) variation of moisture in seeds, oil in kernels, formic, AcOH and an unidentified acid (R_F = 0·92) in seeds and leaves of **I**, **II** and **III** at periods from 7 to 44 days after ripening. Ripening is characterised by reduction of moisture content in seeds and of volatile acid content in leaves and seeds and increased oil content of seeds. Leaves of **I** plant were smeared with 16% solution of radioactive sucrose. After 48 h. chromatograms of hydroxamic deriv. of volatile fatty acids present (formic, acetic, propionic, butyric, valeric) were prepared. Radio-autographs of chromatogram showed high specific radioactivity of AcOH. (11 references.) P. W. B. HARRISON.

Effect of pre-planting irradiation of potato tubers with white and monochromatic concentrated sunlight on synthesis and composition of anthocyanins, growth of green mass and yield. S. A. Stanko (*Dokl. Akad. Nauk SSSR*, 1964, **156**, 1232—1235).—Sprouted tubers were irradiated for a short period with conc. (25—40 times normal) sunlight or monochromatic light. Control tubers were exposed to normal sunlight with radiant energy dose 8·8 × 10^5 erg/cm^2. Doses of conc. light were adjusted to give each tuber approx. 330 × 10^5 erg./cm^2, in 'shock' flashes, 120—130 per min. Treated tubers were kept in diffused white light in laboratory for 72 h., during which the content and nature of authocyanins (**I**) were examined. In other tubers, planted in soil, synthesis of **I** was retarded by white and green light, due, possibly, to photo-oxidation of **I** to aurons and flavonols. Blue and red light increased the **I** content. Absorption curves after irradiation show max. moved 5—15μ to right by red

and blue and 5—15μ to left by white and green light. In white and monochrome light, 3-mono- and 3,5-diglucosides of cyanidin were replaced by some deriv. of more hydroxylated and methylated **I**—peonidin, delphinidin, malvidin and hirsutidin. Quantities varied with colour of light. Pre-planting irradiation of mother tubers affected the height, no. of stems, contents of chlorophyll a and b and three carotinoids in leaves, leaf area, wt. of green material, yield and protein, starch and ascorbic acid content of daughter tubers, for white and three monochromatic lights. Blue and red light increased green material 2 to 3 times whilst white and green light gave the same wt. as controls. Red, white and blue light increased tuber yields by 89, 43 and 14%. Green light decreased yield by 12½%. Protein contents were not much affected.
P. W. B. HARRISON.

Amount and chemical form of selenium in vegetable plants. J. W. Hamilton and O. A. Beath (*J. agric. Fd Chem.*, 1964, **12,** 371—374).—Common vegetables (18) grown in soils containing either org. or inorg. bound Se, all absorbed and metabolised Se to varied extents. In all plants a portion of Se absorbed as selenate was converted into org. bound Se. The Se tended to be highest in those parts of the plant generally considered to be inedible. (11 references.)
W. ELSTOW.

Mobilisation of free tocopherols in germinating seeds. A. R. S. Kartha (*J. Sci. Fd Agric.*, 1964, **15,** 759—763).—The rapid and complete disappearance of tocopherols at the earliest stages of germination when the fat stores are still largely intact indicates the probability that they are being mobilised for some unknown essential function in the growth of the embryo which is quite distinct and different from that of an *in vivo* antioxidant of depot fat. (11 references.)
E. M. J.

Mechanism of inhibition of plant viral infections by enzymes. Y. L. Nene (*J. sci. industr. Res.*, 1964, **23,** 245—248).—A review. (23 references.)
E. G. BRICKELL.

Determination of fluorine in plants. J. S. Jacobson, L. H. Weinstein, R. H. Mandl and D. C. McDune (*Abstr. 6th Int. Congr. Biochem.* [*New York*], 1964, 1 p.).—An automated method for routine determination of inorg. F^- is used (no details) for quantities of F^- down to 0·01 μg. per ml. of solution. Conversion of all F to fluoride is very important. For most materials ashing with CaO followed by alkali fusion is satisfactory. For materials containing volatilisable F^-, a H_2SO_4 distillate is suitable. Combustion in O_2 is recommended for org. fluorides. The procedures can determine 10 μg. of F^- in 50 ml. of plant extract or 5 μg./g. of dried plant tissue.
H. S. R.

Detection and semi-quantitative determination of chlorocholine chloride in wheat grain and straw. J. Jung and G. Henjes (*Z. PflErnähr. Düng.*, 1964, **106,** 108—111).—After removal from an alcoholic plant extract using a powerful cation-exchange resin, quaternary ammonium compounds are separated on a thin-layer chromatogram. Chlorocholine chloride, choline and betaine spots are rendered visible with Dragendorf's reagent.
M. LONG.

Gibberellin-like activities of certain auxins and diterpenes. Masayuki Katsumi (*Dissert. Abstr.*, 1964, **25,** 1517).—Of 24 diterpene deriv. tested by the *d*-5 maize bioassay, (−)-kaurene, a kaurenoic acid, a kaurenol and a methyl kaurenate showed gibberellin (**I**)-like activity. The three last-named deriv. and also steviol showed activity in the *an*-1 maize bioassay. Evidence indicated that the exocyclic methylene group of the D ring in this group of compounds is essential for activity, that the 19-methyl group need not be oxidised, the (OH) group attached to C-13 need not be present and that the lactone is not necessary for activity. Several auxins were applied to the leaf sheaths of five dwarf maize mutants; the elongation produced was readily distinguishable from that produced by **I**. Tests on cucumber hypocotyls with **I** and an anti-**I** and with IAA and an anti-auxin suggest that both **I** and auxin are necessary for optimum growth. Grafts of normal with dwarf mutants of maize demonstrated the transference of **I** from normal to the dwarf portions in some but not in all mutants.
A. G. POLLARD.

Rôle of growth substances in flowering, fruit set, development, maturity and storage behaviour of fruits. J. Rodrigues and H. C. Srivastava (*J. sci. industr. Res.*, 1964, **23,** 237—244).—A review. (150 references.)
E. G. BRICKELL.

Effect of plant growth regulators on the infectivity of tobacco mosaic virus. V. Weeraratne and A. E. Rich (*Canad. J. Bot.*, 1964, **42,** 885—889).—Coconut meat extract, a naturally occurring regulator, inhibited infection by 97% if applied before virus inoculation. Three commercially available regulators, IAA, indolebutyric acid, and *p*-chlorophenoxyacetic acid at 100 p.p.m., had only very low inhibiting activities.
E. G. BRICKELL.

Crops and Cropping

Iron and manganese relations in rice and barley. J. Vlamis and D. E. Williams (*Plant & Soil*, 1964, **20,** 221—232).—Optimum growth of both rice and barley in solution cultures occurred with Mn concn, 0·1—0·2 p.p.m. in the nutrient. Below this level of Mn yields of both species fell off rapidly. At higher Mn levels barley yields fell off rapidly, but rice yields were only slightly reduced even with 5 p.p.m. Mn in the nutrient. Mn % in rice leaves increased much more rapidly than that in barley leaves with increasing Mn in the nutrient, but the reverse was true for roots. Fe % in the leaves of both species varied little with Mn level in the nutrient, although it was high where no Mn was supplied. Rice leaves contained about twice as much Fe as did barley leaves. Severe Mn toxicity symptoms appeared on the old leaves of barley with high supply of Mn, but only slight symptoms showed on rice leaves. Neither species developed chlorosis.
A. H. CORNFIELD.

Sodium–potassium relationships in rice nutrition. J. P. Wells (*Dissert. Abstr.*, 1964, **25,** 1524—1525).—Salt toxicity following the use of saline irrigation water is examined in relation to nutritional requirements of rice. For normal growth small amounts of K are essential and small amounts of Na are beneficial. The Ca, K and Mg contents of rice were higher on a soil showing no response to K fertilisers than on one giving such response. The Na contents of the plants on the two soils were in the reverse order. In 'salt spots with high Na contents rice could not be grown by adding balancing amounts of the other nutrients; removal of excess Na was an essential preliminary. Both the Na and K levels and their ratio in soil have important effects on rice growth. At low salt levels, e.g., 10% of cation-exchange capacity (CEC) the Na/K ratio had less effect on rice yields than had that in high-salt soils (40% CEC). In the latter high ratios caused a sharp decline in yield, Na having a greater effect than K. The K content of the plants was not greatly affected by low salt levels in the soil but the Na content was influenced by the Na level and also the Na/K ratio in the soil.
A. G. POLLARD.

Nutrient requirements of maize and sorghum compared. G. S. Dhillon (*Dissert. Abstr.*, 1964, **25,** 1469).—Samples of maize and sorghum plants were taken from long-term manurial trial plots at two locations during early growth (whole plant less roots) and again at maturity (grain, heads without grain or cobs, stalks), N, P, K, Ca and Mg being determined in each sample. In both species the N contents (in excess of that in controls) were functions of amounts of N fertiliser applied. Deficiencies of N or P in mature plants were manifest first in stalks, then in heads or cobs and finally in grain. Of the nutrients examined K was taken up in early growth in the greatest amounts by both species, and was followed by N, Ca, P and Mg in decreasing order. For good yields the N level should exceed that of other nutrients, the desirable order of accumulation being N > P > K > Mg > Ca. Application of P fertilisers increased the uptake of P and in some cases of N also. The Ca and Mg requirements of both crops were relatively low, the Ca intake being a function of the N applied. Mg contents were higher in grain than in other plant parts.
A. G. POLLARD.

Sulphate concentration as an indicator of sulphur status in dryland pasture species. M. B. Jones and W. E. Martin (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 539—541).—Sulphate concn. in subclover and soft chess under high N fertilisation were approx. equal over a wide range of S fertiliser levels. With no fertiliser N the SO_4^{2-} content of annual grasses, including soft chess, were higher than those in rose or subclover. Harding-grass was particularly high in SO_4^{2-}. Where no N was applied SO_4^{2-} in subclover decreased from the early vegetative to the flowering stage, and then decreased when the plants began to wilt at the end of the season. Subclover and rose clover, sampled during the flowering period but before wilting, appeared to be the best plants for determining the S status of dryland pastures in California.
A. H. CORNFIELD.

Effect of supplemental irrigation with saline water on soil composition and on yields and cation content of forage crops. J. Lunin, M. H. Gallatin and A. R. Batchelder (*Proc. Soil Sci. Soc. Amer.*, 1964, **28,** 551—554).—A fine sandy loam growing grasses and clovers was irrigated with synthetic seawater diluted to varying extents (to give electrical conductivity values ranging from 2—12 mmho. per cm.). During the first year all species gave increased dry-matter yields when irrigated with the higher dilutions of seawater in comparison with non-irrigated plots, but not in comparison with plots treated with fresh water. In the following 3 years treatment with saline water had no effect on or reduced crop yields. Ladino clover was more sensitive to salinity than were lucerne, Kentucky fescue and orchardgrass. Soil salinity at depths down to 18 in. increased with the salt level of the water applied. The Na% in all species tended to increase, whilst Ca, Mg and K% were unaffected, with

increasing salinity of the irrigation water. Soil exchangeable Ca decreased, whilst exchangeable Mg, Na and K increased, with increasing salinity of the irrigation water. A. H. CORNFIELD.

Influence of management, nitrogen and potassium on the development and the organic constituents of the vegetative reproductive system of timothy. R. W. Sheard (*Dissert. Abstr.*, 1964, **25**, 1474).—Vegetative reproduction in timothy is examined in relation to the use of N and K fertilisers and to defoliation. Dry-matter yields and the wt., non-polymer and fructan fructose contents of the haplocorms were measured at 10-day intervals. N applications increased dry-matter yields up to 4-fold. Defoliation, which was varied according to the height of the growing point, and the development of axiliary buds modified the distribution of yields over the season but not the total yield. The system of timing defoliation depended on sufficient supplies of N and was limited by the day length requirement of the plants. The development of successive haplocorms from the basal internode of elongating tillers is described, the fructan fructose of the primary haplocorm serving as the nutrient reserve for the developing tillers. The capacity for winter survival and spring re-growth was correlated negatively with the carbohydrate content of the tertiary shoot at the initiation of spring growth, and positively with the insol. protein content. A sufficiency of carbohydrate for energy and a readily available source of protein for synthesis of new protoplasm afford a better basis for re-growth than does a high-carbohydrate–low-protein reserve.
A. G. POLLARD.

Self-heating of esparto grass. H. P. Rothbaum (*J. appl. Chem.*, 1964, **14**, 436—439).—Adiabatic heating experiments on humidified samples of esparto grass (**I**) showed that moist **I** self-heats less easily than hay but microbiological heating leads to further chemical heating at ~96% R.H. This would confirm the importance of R.H. in starting spontaneous combustion since **I** has a much smaller moisture content than hay. C. A. P.

Fertilising experiments on apple and cherry trees grown in pots. R. Fritzsche, B. Krapf and L. Huber (*Schweiz. landw. Forsch.*, 1964, **3**, 121—181).—The effects of varying amounts of N, P, K, Ca, Mg, B, Mn and Zn on varieties of apple and cherry trees grown in pots during 14 years are reported. Deficiency symptoms are noted and photographed and fruit quality and chemical analysis of leaves and wood are recorded. The plants, grown in pots embedded in gravel in a very poor podsol deficient in all mineral nutrients, were then treated with different amounts and ratios of the elements investigated. Deficiencies in N, P, K, Mg and B resulted in characteristic symptoms but the lack of Ca was apparently not important. Excess of P, N and K caused disturbance. Ion competition effects were noted when two major constituents were increased relative to a third ; this was most pronounced in the case of K and Mg and P and Zn. Mg deficiency led to shoots of the same length as normal but much thinner. Effects on fruit quality of any deficiency was very marked, especially with Mg. Typical spots and dead lesions permit positive identification of B deficiency which may be corrected with 0·2% foliar sprays of boric acid. Excess N or P reduced P and Zn respectively in leaves of all trees but the former caused increased Mn in apple wood and leaf. (25 references.) J. B. WOOF.

How to control fruit drop and gummosis in almonds. R. P. Srivastava and D. K. Bose (*Fertil. News*, 1964, **9**, No. 8, 27—30).—Almond trees were sprayed with aq. $CuSO_4$ and boric acid (**I**) in concn. of 0, 1500 and 3000 p.p.m. and 0, 150 and 200 p.p.m respectively one week after fruit set. Gummosis was reduced from 89 to 14% by 200 p.p.m. of **I** while fruit drop was reduced from 98 to 21% by 1500 p.p.m. of $CuSO_4$ plus 200 p.p.m. **I**. S. A. BROOKS.

Effect of magnesium on pineapple growth in an acid lateritic sandy clay. E. Hernandez-Medina (*J. Agric. Puerto Rico*, 1964, **28**, 17—24).—When adequate NPK was present application of Mg to the soil (300 lb. Mg/acre) or as a foliar spray (15 lb. Mg/100 gal.) increased pineapple fruit yields by an average of 2·7 tons/acre on an acid lateritic sandy clay (pH 5·0). Liming and application of Fe, B, Zn, Cu and Mo had no effect. Yields were correlated with leaf Mg %. A. H. CORNFIELD.

Laboratory experiments on systematic breeding for frost resistance in vines. L. Müllner and G. Mayer (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 222—234).—When vines were exposed to temp. at −17° to −23°, slow cooling over several h. was much less injurious than rapid cooling ; the rate of thawing had no effect. The sensitivity of the buds to frost was not related to their position on the vine. A temporary thaw during part of Feb. weakened the resistance of most of the vines under test to subsequent frost. A hybrid vine was much more resistant than ~20 pure European vines. (33 references.) P. S. ARUP.

Effect of zinc deficiency on the amide content of tomato plants. E. Vrachnon, C. Dassion and A. Pomoni (*Naturwissenschaften*, 1964, **51**, 468).—Free amino-acids were extracted by 80% EtOH from leaves of tomato plants with Zn deficiency or with this ameliorated by $ZnSO_4$ applied to roots or leaves. Except for glutamine (**I**), and asparagine (**II**), chromatographically separated amino-acids were similar in normal and Zn-deficient plants. **I** and **II** were very small in normal plants but were 400 and 1018 μg./g. respectively in deficient plants and these figures were increased up to 90 days' growth. Application of Zn to deficient plants brought the values back to normal. (In English.) H. S. R.

Physiological studies relating to tomato fruit cracking. K. Singh (*Dissert. Abstr.*, 1964, **25**, 1475).—In the juice of tomato fruits, sol. solids, dry-matter content and $[H^+]$ increased gradually from the stem end to the blossom end. With advancing maturity, the sol. solids content of the juice increased but there was no change in dry-matter content. The steepness of gradients of the three factors from stem- to blossom-end was associated with radial cracking at the stem-end. Used as a dwarfing compound, 2-chloroethyl trimethylammonium chloride restricted the height and leaf area of the plants and the total water loss by transpiration but increased transpiration rates per unit leaf area. Diminution of water loss by transpiration was associated with greater severity of cracking under greenhouse conditions with no moisture deficiency. Spraying plants twice daily with water or 0·001M-8-hydroxyquinoline sulphate did not affect cracking but bagging the fruits in polyethylene bags or covering the whole with a polyethylene tent increased cracking, probably by increasing temp. or R.H. A. G. POLLARD.

Nature of the resistance of the tomato to some fungal pathogens. R. L. Lowther (*Dissert. Abstr.*, 1964, **25**, 1471).—Relationships between susceptibility or resistance of tomato plants to form pathogens and their contents of amino-acids, amides and sugars are examined. A tomato (Potentate) susceptible to *Cladosporium fulvum* contained more serine, alanine, γ-aminobutyric acid, glutamine and glucose than did resistant or immune varieties. One resistant line contained less aspartic acid, serine, glutamine, fructose, glucose and sucrose ; another resistant line showed a high content of cysteic acid. Similar characteristic differences in metabolite content between healthy and infected tissues and susceptibility to other pathogens are recorded. In some cases, isolates from *C. fulvum* could be differentiated by the colour of the colonies grown on glutamine- or γ-amino-acid-media. A. G. POLLARD.

Fertiliser trials with tomatoes and cucumbers in Puerto Rico. M. R. Ballester, G. Samuels and J. L. Garcia (*J. Agric. Puerto Rico*, 1964, **28**, 49—54).—Tomato on a silty clay loam (pH 5·2) gave the highest yields with application of 50 lb. of N and 100 lb. of each of P_2O_5 and K_2O per acre. Liming had no effect on yields. On a fertile clay (pH 7·5) yields of tomatoes and cucumbers were not increased by N, P or K. On an alkaline silty clay loam cucumber yields were increased by N 100 lb. and P_2O_5 50 lb./acre.
A. H. CORNFIELD.

Variation in the β-carotene and ascorbic acid contents of lettuce and carrot as influenced by seasonal changes in Puerto Rico. M. del C. C. Fernandez (*J. Agric. Puerto Rico*, 1964, **48**, 39—48).—The β-carotene and ascorbic acid contents of lettuce and carrot samples monthly throughout the year were not consistently affected by climatic factors such as temp., no. of rainy days or total rainfall. The ascorbic acid content of lettuce was reduced throughout the year by shading the plants for 10 days before harvest, but its β-carotene content and the ascorbic acid and β-carotene content of carrot were unaffected by shading. A. H. CORNFIELD.

Influence of magnesium and manganese applications to spinach plants on their cation uptake and distribution of magnesium amongst various solubility fractions in their tissue. K. Lehmann (*Z. Pfl-Ernähr. Düng.*, 1964, **106**, 111—116).—Mg depresses the uptake of other ions. Correlations exist between total leaf Mg and the individual Mg fractions, although acetone-sol. Mg. in contrast to water-sol. and -insol. fractions, rises only slightly with increasing total Mg. Mn applications of up to 20 mg Mn per pot have no noticeable affect on Mg fractionation. M. LONG.

Effect of plant density on growth, yield and quality of upland cotton. H. O. Burhan (*Dissert. Abstr.*, 1964, **25**, 1468).—Increase in plant density was associated with fewer flowers, increased shedding and lowered retention and size of bolls, fewer seeds per boll and lower seed and lint indices ; yield per plant diminished whereas yield per unit area increased to an optimum with 1·3 plants/sq. ft. The higher yields per plant at low plant densities probably result from improved photosynthesis. The length, strength and fineness of the cotton fibres were very little affected by differences in plant density. A. G. POLLARD.

Changes in soil productivity induced by pine plantations. S. A. Wilde (*Soil. Sci.*, 1964, **97**, 276—278).—The org. matter content of the soil underneath pine plantations has been shown to increase

with time. Plantations <20 years old were low in org. matter but those >20 years showed increases. The results are discussed.
T. G. MORRIS.

Interrelationship of carbon dioxide narcosis and chilling temperature on the post-harvest physiology of the sweet potato. C. R. Roberts (*Dissert. Abstr.*, 1964, **25**, 1473—1474).—Chilling root tissue of sweet potato at 5° caused an erratic increase in respiratory rate for 5 weeks, after which there was evidence of damage and a sharp decline in respiration. Leakage of K from the tissue was greater from chilled than from non-chilled (15°) tissue during the first day of storage. Possibly chilling affected the permeability of the lipid of the cell membranes or caused disruption of the membrane. An energy relationship between CO_2 evolution, O_2 uptake and K leakage is indicated. CO_2 treatment lowered the loss of K. Electron micrographs of chilled and CO_2-treated tissue showed small coagulated particles in the cells and the formation of a reticulum or a continuous membrane through the parenchyma cells of the root.
A. G. POLLARD.

Pest Control

Evaluation of soil insecticide treatments for control of cyclodiene-resistant southern maize rootworms. G. M. Boush and M. W. Alexander (*J. econ. Ent.*, 1964, **57**, 465—468).—Twenty-six compounds in 65 formulations were evaluated. Of these 9 granular formulations, diazinon, zinophos, G.C. 4072 [diethyl 2-chloro-1-(2,4-dichlorophenyl)vinyl phosphate], phorate, A.C. 43064 [2-(diethoxyphosphinothioylimino)-1,3-dithiolan], Bayer 25141 [*OO*-diethyl *O*-[*p*-(methylsulphinyl)phenyl] phosphorothiolothionate], Bayer 39007 (*o*-isopropoxyphenyl methylcarbamate), U.C. 10854 (*m*-isopropylphenyl methylcarbamate) and U.C. 8305 [*P*-chloro-2,4-dioxa-5-methyl *P*-thiono-3-phosphabicyclo-(4,4,0)-decane], as soil band treatments during mid-July were effective against *Diabrotica undecimpunctata howardi*. Emulsifiable concentrates were ineffective.
C. M. HARDWICK.

Control of southern potato wireworm, *Conoderus falli*, on early-crop potatoes. A. Day, F. P. Cuthbert, jun., and W. J. Reid, jun. (*J. econ. Ent.*, 1964, **57**, 468—470).—Diazinon, phorate and diazinon as a preplanting broadcast application disked in before planting gave adequate control of a moderate infestation. Phorate and parathion in the planting furrow were less effective. Diazinon, parathion and phorate applied in the autumn were promising. Nemacide, Telodrin and Zinophos were also successful, by each method of application.
C. M. HARDWICK.

Zonal accumulation of dieldrin in soil and lucerne residues. D. D. Hardee, W. H. Gutenmann, D. J. Lisk, G. G. Gyrisco and C. M. Edmonds (*J. econ. Ent.*, 1964, **57**, 583—585).—Dieldrin was applied as granules to the soil amongst lucerne in the autumn. Detectable residues were found in the lucerne after 32 months. Residues in the soil were concentrated in the top inch. There was some evidence of an accumulation at 6 in. Dieldrin was still detectable in soil after 44 months. (14 references.)
C. M. HARDWICK.

Control of clover root borer in New York. D. D. Hardee, H. Y. Forsythe, jun., and G. G. Gyrisco (*J. econ. Ent.*, 1964, **57**, 585—586).—Of seven insecticides tested, heptachlor, lindane and Telodrin granules at 0·5 lb./acre were most effective against *Hylastinus obscurus*. Several organophosphorus insecticides were effective at 1 lb./acre.
C. M. HARDWICK.

Effectiveness of various insecticides and miticides against the pear-bud mite. *Eriophyes pyri* (Pgst.) P. J. Kriegler and A. C. Myburgh (*S. Afr. J. agric. Sci.*, 1963, **6**, 625—632).—Previously pear-bud mite had been kept under control incidentally as a side effect of the S-containing sprays applied against pear-scab. Since the advent of the newer synthetic fungicides pear-bud mite infestation has assumed economic importance. Of the pesticides examined Sevin was outstanding in efficacy against the mite whilst Gusathion, Metasystox and parathion were all better than the previously used S compounds. Kelthane and Dimite were not toxic towards the mite. (17 references.)
W. ELSTOW.

Hydrogenation refining vs. efficiencies of spray oils against citrus red mite eggs and California red scale. L. A. Richl, M. J. Garber, J. P. LaDue, J. L. Rodriguez and E. L. Wilson (*J. econ. Ent.*, 1964, **57**, 522—525).—Two naphthenic oils with viscosities equal to those of two paraffinic oils were tested against *Panonychus citri* and *Aonidiella aurantii*. Each was refined by solvent extraction to 92% unsulphonatable residue and another sample subjected to mild hydrogenation. Both were of similar toxicity. Oils formulated with oil-sol. emulsifier are better than the same oils applied by tank mixture method with blood albumin spreader.
C. M. HARDWICK.

***Drosophila* suppression in tomato fields with granular diazinon.** R. C. Riley, J. H. Simpson and G. Winnett (*J. econ. Ent.*, 1964, **57**, 461—465).—Diazinon sprays give good initial control of *Drosophila* but short residual control. 5% granules give at least 5—7 days' control and are as effective as 10% granules. The toxicant volatilises from the granules on to the lower plant surfaces where most flies are found.
C. M. HARDWICK.

Control of *Verticillium* wilt of tomato plants with 'Cyclocel' (2-chloroethyltrimethylammonium chloride). A. K. Sinha and R. K. S. Wood (*Nature, Lond.*, 1964, **202**, 824).—Application of 'Cyclocel' to the soil around the base of tomato plants inoculated with *Verticillium albo-atrum* considerably reduced the mean disease index. It also induced the formation of large no. of tyloses in vessels of both uninoculated and inoculated plants.
S. A. BROOKS.

Factors related to internal browning of tomato fruits. D. R. Tompkins (*Dissert. Abstr.*, 1964, **24**, 4901).—In field trials treatment of the plants with B, sucrose + B, Fe + B, 2-chloroethyltrimethylammonium chloride or (in very wet soil) high ridging of the rows lessened the incidence of internal browning. Gibberellic acid treatment or heavy shading increased the disorder. Affected fruits showed higher activity of polyphenoloxidase, peroxidase and cyclochrose oxidase, lower dry-matter content and % of sol. solids and also higher K contents than did normal fruits.
A. G. POLLARD.

Zectran and Bayer 44646 show most promise in control of cabbage caterpillars. R. G. Prochaska, F. P. Cuthbert, jun., and W. J. Reid, jun. (*J. econ. Ent.*, 1964, **57**, 490—492).—In experiments over 2 years, Bayer 44646 (4-dimethylamino-*m*-tolyl methylcarbamate) amd Zectran were the most effective of 12 compounds (including parathion and toxaphene) against *Trichoplusia ni* on cabbage. They were also effective against light infestations of *Pierris rapae* and *Laphygma frugiperda* on cabbage. Bayer 44646 was moderately phytotoxic.
C. M. HARDWICK.

Oils and surfactants alone, and insecticide–oil combinations for aphid control on turnips and cabbage. D. A. Wolfenbarger (*J. econ. Ent.*, 1964, **57**, 571—574).—The oils tested included fractions of conventional naphthenic and paraffinic oils, alkylate isoparaffinic oils and specific paraffinic oils. The paraffinic oils were most effective against both *Rhapalosiphum pseudo brassicae* and *Brevicoryne brassicae*. Aphid control tended to increase with the rate of application and mol. wt. of the oil. Surfactants were also insecticidal.
C. M. HARDWICK.

Control of garden symphylan, *Scutigerella immaculata*, in mint. C. H. Shanks, jun., and A. J. Howitt (*J. econ. Ent.*, 1964, **57**, 525—527).—Zinophos (5 lb./acre) and Nemacide (10 lb./acre) gave good control of symphalans when applied as sprays or granules in spring or autumn. Zinophos at 2 lb./acre may be sufficient in spring. Parathion and lindane gave little or no control in autumn although parathion was effective in the spring.
C. M. HARDWICK.

Toxicological studies on Egyptian cotton leafworm, *Prodenia litura*. I. Susceptibility of different larval instars of *Prodenia* to insecticides. M. E. Eldefrawi, A. Toppozada, N. Mansour and M. Zeid. II. Reversion of toxaphene resistance in Egyptian cotton leafworm. M. E. Eldefrawi, A. Toppozada, A. Salama and S. A. Elkishen (*J. econ. Ent.*, 1964, **57**, 591—593, 593—594).—**I.** The average wt. of each instar was obtained. LD_{50} values for dichlorvos, toxaphene, Trichlorfon and pyrethrins applied to dorsal surface of larvae were corrected for wt. Except for dichlorvos 4th-instar larvae were slightly more susceptible than the others, but in general the LD_{50} per unit body wt. remained constant.

II. Laboratory-reared strains showed a gradual reduction in resistance to toxaphene as 3rd-instar larvae. This reversion was also found in the field during exposure to trichlorfon and carbamate Carbaryl.
C. M. HARDWICK.

Effect of spider mite populations on yield and quality of cotton. T. D. Canerday and F. S. Arant (*J. econ. Ent.*, 1964, **57**, 553—556).—Plots were artificially infested with *Tetranychus cinnabarinus*. The greatest damage was in those infested in Aug. Heavy rainfall depressed mite no. Yields of seed cotton were 14—44% less in infested plots. Infested crops tended to have smaller bolls and lower quality lint and seed.
C. M. HARDWICK.

Effects of past-season applications of insecticides, defoliants and desiccants on diapausing boll weevils. T. C. Cleveland G. L. Smith (*J. econ. Ent.*, 1964, **57**, 527—529).—Experiments over 3 years in Louisiana showed that all treatments reduced the numbers of diapausing weevils. Guthion and methyl parathion sprays gave 62% fewer overwintering weevils. No treatment eliminated populations.
C. M. HARDWICK.

Control of boll weevil, bollworm spp. and cotton aphid on cotton in 1960—2. A. R. Hopkins and H. M. Taft (*J. econ. Ent.*, 1964,

57, 509—511).—In small plots 13 sprays were evaluated in five experiments. All but two prevented heavy bollworm populations developing. Geigy 30494 [*OO*-dimethyl-*S*-(2,5-dichlorophenyl)thiomethyl phosphorothiolothionate]–DDT, methyl trithion–DDT, Monsanto CP-40294 [*O*-phenyl-*O*-*p*-nitrophenyl methylphosphonothionate]–DDT, toxaphene–DDT and Stauffer R-1504, [*OO*-dimethyl *S*-phthalimidomethyl phosphorothiolothionate]–DDT gave as good control of *Anthonomus grandis* as did Guthion–DDT.
C. M. HARDWICK.

Effect of the bollworm, *Heliothis zea*, on yield and quality of cotton. P. L. Adkisson, C. F. Bailey and R. L. Hanna (*J. econ. Ent.*, 1964, **57,** 448—450).—Significant reductions in yields did not occur until seasonal infestation reached >10 larvae per 100 plants or 2 per 10-ft. row. There was little effect on the quality of lint cotton produced, or on grade and staple. Medium and heavy infestations reduced fibre and yarn strength slightly. C. M. HARDWICK.

Chemical control of subterranean insects in ratoon sugarcane. I. Effect on wire-worm. S. T. Lee (*Rep. Taiwan Sug. Exp. Sta.*, 1964, No. 33, 95—105).—Aldrin, aldrin–BHC and BHC treatments were all effective in controlling wire-worm infestation of sugarcane plantation and resulted in a marked increase in yields when compared with infested plots. Aldrin was most effective and the suggested rate of application is 2·5 lb./ha. E. ELSTOW.

Downy mildew of sugarcane in Taiwan. VI. Further studies on the relation of environments to sporulation. T. Matsumoto, P. C. Chen and S. M. Yang (*Rep. Taiwan Sug. Exp. Sta.*, 1964, No. 33, 53—60).—Conidial formation is affected by (*a*) humidity, (*b*) temp., (*c*) light, (*d*) vigour of the host. Of these (*a*) and (*b*) are very important. The sporulation of the downy mildew is reduced by an increase of light intensity but this effect depends largely on the diseased condition of the host. In infected plants the characteristic downy mildew symptoms are more distinctly shown in test plants well supplied with nutrients than in those grown in undernourished conditions. W. ELSTOW.

Chemical control of Douglas-fir cone midge, *Contarinia oregonensis*, using a mistblower from a truck-mounted ladder. N. E. Johnson (*J. econ. Ent.*, 1964, **57,** 556—558).—Guthion and Endosulfan (Thiodan) sprays were best in different areas. Cone moths were not found in sprayed cones but *Megastigmus spermatrophus* was not affected. Cone abortion was 49% on controls and 68% on treated branches. Cone midges were reduced by 74% and seed yield increased 2½-fold. C. M. HARDWICK.

New insecticides against adults of two species of *Hippelates* eye gnats. M. S. Mulla and T. S. Adams. (*J. econ. Ent.*, 1964, **57,** 505—509).—Twenty new insecticides were evaluated by the continuous exposure method against *H. collusor* and *H. pusio*; both gave similar reactions. Sumithion, Mevinphos (Phosdrin), Bayer 37289 (*O*-ethyl *O*-2,4,5-trichlorophenyl ethylphosphonothioate), Bayer 37343 [*OO*-diethyl *O*-(3,5-dichloro-4-methylthiophenyl) phosphorothioate] and SD.7587 (dimethyl 1-*p*-chlorophenylthiovinyl phosphate). and Dimetilan were more active than DDT, parathion or malathion. All six carbamates produced rapid knockdown, Isolan being the most rapid (34% within 1 h.).
C. M. HARDWICK.

Acceptability of some fats and oils as food to imported fire ants. L. S. Lofgren, F. J. Bartlett and C. E. Stringer (*J. econ. Ent.*, 1964, **57,** 601—602).—Nineteen of 32 fats and oils gave acceptance ratios good or better than groundnut oil. Crude fats were less acceptable than refined fats. Cottonseed oil was less acceptable when hydrogenated. When the peroxide value for soya-bean oil approached 100 in rancidity, it was not accepted. C. M. HARDWICK.

Toxicity of some pesticide residues to adult *Amblyseius hibisci* with a compilation of effects of pesticides upon phytoseiid mites. B. R. Bartlett (*J. econ. Ent.*, 1964, **57,** 559—563).—*A. hibisci* was exposed to 62 pesticides as contact deposits on paper and the mortality over 4 days listed. No pattern of toxicity could be found. Aramite and Tetradifon were not toxic while CaO–S and S were very toxic. All phosphates except TEPP were very toxic. Chlorinated hydrocarbons had more variable effects. (36 references.)
C. M. HARDWICK.

Chemical control of weeds in crimson clover grown for seed production. W. O. Lee (*U.S. Dep. Agric. agric. Res. Serv. Tech. Bull.*, 1964, 1302, 21 pp.).—Experiments during the period 1957—61 are described. Mid-winter post-emergence treatment with isopropyl *N*-phenylcarbamate gave the best weed control and increased seed yields. (10 references.) E. G. BRICKELL.

Physical data on IPC and CIPC. R. Lubman and K. Sieber (*Chem. Tech., Lpz.*, 1964, **16,** 236—237).—From v.p. measurements were calculated b.p. (which agreed with those determined), vaporisation enthalpy and b.p. at 1 and 5 mm. Hg. The vaporisation losses are determined at 30° and 100°, those for CIPC being smaller than those for IPC. Water-solubility was determined by use of ^{36}Cl-labelled CIPC and found to be 9 mg./100 g. M. GREENAWAY.

Chlorinated derivatives of phenoxy-carbonylic acids and their use in weed control. G. Erfurt, H. Kringer, W. Salzbrenner and M. Pallas (*Chem. Tech., Lpz.*, 1964, **16,** 199—203).—A general article on the method of prep. of the acids, especially 2,4-D, NCPA and 2,4,5-T and their uses, and also their side effects on the flavour of fruits and vegetables (especially vine culture). Effects on the soil are briefly considered and the use of bacterial agents and sex sterilants for insect control is discussed. M. GREENAWAY.

Nut-grass (*Cyperus rotundus* L.) and its control. Tso-Cheng Chang and Wen-biou Sze (*Rep. Taiwan Sug. Exp. Sta.*, 1964, No. 33, 1—16).—The principal reproductive organs of nut-grass are the tubers and basal bulbs, the flowers being mainly sterile and the seeds, when produced, non-germinating. The tuber is very adaptable to environment, being able to withstand extremely adverse conditions. The rhizomes will penetrate 3 cm. of paved road or come up through 50 cm. of sandy loam. Of the herbicides examined the most effective and economical to control nut-grass amongst sugarcane was found to be 2,4-D sprayed 3 or 4 times at the lowest effective rate (1·5 kg./ha.) (12 references.) W. ELSTOW.

Bacterial decomposition of some aromatic and aliphatic herbicides J. B. Bryand, jun. (*Dissert. Abstr.*, 1964, **24,** 4915).—All of the 19 herbicides examined could serve as energy source for soil microorganisms. Cell counts changed very little through seven serial transfers on a herbicide–mineral salts broth. Isolates from the seventh transfer were largely *Pseudomonas*, or *Arthrobacter*; the former group was dominant when the herbicide was a substituted urea (diuron, neburon, Lorox), an acetamide deriv. (Randox, Randox-T), a carbonate (CIPC, Alipur), the endoxyhydrophthalate, Aquathol or the triazine, ipazine. *Arthrobacter* types were dominant when 3-amino-1,2,4-triazole (**I**) or the *s*-triazines, atraton or Ametryne were used. Both groups of organisms occurred in considerable no. when similar tests were made with Hyvar (a substituted uracil), 2,3,6-trichlorophenylacetic acid (**II**) or the *s*-triazines, atrazine, propazine, Prometone and Prometryne. Six *s*-triazines used were of the 6-isopropylamino-series with different substituents in the 4-position. These substituents determined the nature of the microflora developing in enrichment cultures; the ethylamino-group favoured *Arthrobacter*, the diethyl-amino group favoured *Pseudomonas* and the isopropylamino-group permitted growth of both types of organisms. Addition of glucose to media containing **I** or **II** accelerated the disappearance of the herbicides. In percolation tests, **I** (2 p.p.m.) depressed the rate of nitrification of $(NH_4)_2SO_4$, but neither **II** nor 3-amino-2,5-dichlorobenzoic acid at 500 p.p.m. affected nitrification. The action of **I** is on the first stage of the nitrification process since the oxidation $NO_2^- \longrightarrow NO_3^-$ was unaffected by concn. 100 p.p.m. A. G. POLLARD.

Recommended analytical methods for pesticides. XXII. Chlorfenson and chlorfenson formulations. XXIII. Fenson and fenson formulations. Collaborative Pesticides Analytical Commee (F.A.O. Plant Prot. Bull., 1964, **12,** No. 1, 5 pp. reprint; No. 2, 2 pp. reprint).—XXII. Detailed instructions are given for the determination of chlorfenson, 4,4′-dichlorophenyl sulphone (**I**) and 4-chlorophenol (**II**). The method depends on the hydrolysis of chlorfenson with methanolic KOH and the excess alkali titrated with HCl and methyl red indicator, the alkali used up being a measure of the chlorfenson content. **II** is determined as the $CHCl_3$-sol. material from the titration liquid after addition of excess alkali. **II** is obtained by bromometric titration of the NaOH-sol. material in another portion of sample. Chlorfenson atomisable liquids are examined as described above. For water-dispersible powders, the procedure is applied to the acetone-sol. fraction of the material.
XXIII. The methods for fenson and its formulations are similar to those for chlorfenson, the compounds determined being fenson, diphenyl sulphone and *p*-chlorophenol. H. S. R.

Determination of content of γ-hexachlorocyclohexane in technical HCH samples by isotope dilution using ^{36}Cl. K. Sieber, A. Jumar and H. Grosse-Ruyken (*Chem. Tech., Lpz.*, 1964, **16,** 222—225).—Technical HCH contains only 10—15% of the effectively insecticidal γ-isomer. The analysis is performed by adding an accurately determined quantity of ^{36}Cl-labelled γ-HCH (~120 mg.) to the sample, which should contain 100—150 mg. of γ-HCH. The mixture is melted and then cooled, with stirring, and γ-HCH extracted with MeOH and recrystallised from EtOH. The content of γ-HCH is determined by the usual isotope method. ^{36}Cl-labelled γ-HCH is produced by isotope exchange between $Al^{36}Cl_3$ and γ-HCH at 120° for 7 min. Two model samples and three HCH samples were analysed and a relative standard deviation of ±0·43% obtained.
M. GREENAWAY.

Determination of small quantities of some insecticidal esters of dialkyldithiophosphoric acids in factory air. V. Bátora and J. Kováč (*Chem. Tech., Lpz.*, 1964, **16**, 230—232).—A selective method for determining thiometon, Disyton and Trithione is described, based on their inhibitory effect on the enzyme cholinesterase, using a colorimetric method. Sensitivity of determination is increased, and vol. of required air sample reduced, by oxidising the isolated insecticide with peracetic acid. This gives stronger enzyme inhibition, but cannot be used for thiometon, which decomposes on oxidation. Extraction from the air is by suction through an active C adsorbent, and (in the case of thiometon) elution with benzene. (20 references). M. GREENAWAY.

Field technique for oil deposit determination on citrus through colorimetric analysis. H. A. Dean, E. L. Wilson, J. C. Bailey, R. W. White and L. A. Riehl (*J. econ. Ent.*, 1964, **57**, 458—461).—The prep. of the dyed oil is described. It was stripped from leaves and fruit using an automatic, constant-vol. portable pipette. Oil Red A dye was more stable under field conditions than Oil Red O dye. Determination of leaf area by superimposition and planimeter showed <3% differences. The technique of spraying the fruit in the laboratory is discussed. (12 references.)
C. M. HARDWICK.

Organic pesticides. LXXVI. Synthesis of some new carbamic acid derivatives. K. D. Shvetsova-Shilovskaya, N. N. Melnikov, I. M. Borisova and E. G. Novikov (*Zh. obshch. Khim.*, 1964, **34**, 1779—1783).—A new series of pyridylethyl and picolylethyl esters of arylcarbamic acids (aryl is phenyl or Cl- or Me-substituted phenyl) was prepared by reaction (3—4 h.) of equimol. amounts of 2-β-hydroxyethylpyridine and aryl isocyanates (added stepwise). The relation between their structure and biological activity is to be examined. A. L. B.

Effect of Sevin (*N*-methyl-1-naphthylcarbamate) on the carbohydrate and nitrogen metabolism during the germination of cotton seeds. M. I. Naguib (*Indian J. exp. Biol.*, 1964, **2**, 149—152).—Selected cotton seeds were soaked in water or various Sevin solutions for 24 h. before germination for 5 days. The seeds were then ground, extracted with $CHCl_3$ and analysed for carbohydrates, N, total protein and nucleoprotein. Treatment with Sevin increases the rate of nucleotide formation during the early stages of germination. Pure Sevin decreases the rate of respiration and accumulation of nucleoprotein and increases the dry wt., total carbohydrate (especially glucosan) and N content. Commercially formulated Sevin (85% active material + a filler) causes these effects with less intensity and in addition accelerates the rapid deamination of proteins and decreases the rate of galactosan accumulation. (29 references.)
A. T. CARPENTER.

Metabolism of *O* phenyl-*O*-(4-nitrophenyl) methylphosphonothionate (Colep) in plants and animals. G. J. Marco and E. G. Jaworski (*J. agric. Fd Chem.*, 1964, **12**, 305—310).—Colep is metabolised by apple and cotton seedlings to give a variety of metabolites. In general Colep, which is non-polar, is converted into water-sol. polar phenol deriv. With rats, within 24 h. most of an oral dose of Colep was excreted as metabolites in the urine. (20 references.)
W. ELSTOW.

Fate of Amiben in tomato plants. S. R. Colby, G. F. Warren and R. S. Baker (*J. agric. Fd Chem.*, 1964, **12**, 320—321).—Experiments with tomato plants grown in soil treated with Amiben-^{14}C indicate that any Amiben taken up is complexed by the plant with other plant constituents. This results in poor translocation of the Amiben so that the fruit is relatively free of any Amiben residues.
W. ELSTOW.

Metabolism of 3-hydroxy-*NN*-dimethylcrotonamide dimethyl phosphate by cotton plants, insects and rats. D. L. Bull and D. A. Lindquist (*J. agric. Fd Chem.*, 1964, **12**, 310—317).—The metabolism of the experimental systemic insecticide 3-hydroxy-*NN*-dimethylcrotonamide dimethyl phosphate (Bidrin) has been studied by the use of radiometric techniques. Oxidative demethylation to the equally toxic *N*-methyl deriv. occurred in all the biological systems studied but all toxic products decomposed rapidly. Nine P-containing metabolites were detected eight of which are tentatively identified. (17 references.) W. ELSTOW.

Nature of certain carbamate metabolites of the insecticide Sevin. H. W. Dorough and J. E. Casida (*J. agric. Fd Chem.*, 1964, **12**, 294—304).—Eight metabolites of Sevin formed by rat liver microsomes and by cockroaches and houseflies were separated, three of these were tentatively identified. Certain of these metabolites were found in the milk of a goat treated orally with Sevin. The metabolites had reduced biological activity. (25 references.)
W. ELSTOW.

Comparison of some granular carriers for chlordan and heptachlor against the imported fire ant. W. F. Barthel and C. S. Lofgren (*J. agric. Fd Chem.*, 1964, **12**, 339—342).—The comparative efficacy of montmorillonite, vermiculite, bentonite and attapulgite as granular carriers for heptachlor and chlordan used for the eradication of imported fire ant in the U.S.A. were studied. Montmorillonite formulations are considered to be better than the Federal specified attapulgite formulations. W. ELSTOW.

Review of adsorption and desorption of organic pesticides by soil colloids with implications concerning pesticide bioactivity. G. W. Bailey and J. L. White (*J. agric. Fd Chem.*, 1964, **12**, 324—331).—A review of the literature with 161 references covering 30 different org. pesticides. W. ELSTOW.

Conversion of 4-(2,4-DB) to 2,4-dichlorophenoxycrotonic acid (2,4-DC) and production of 2,4-D from 2,4-DC in soil. W. H. Gutenmann and D. J. Lisk (*J. agric. Fd Chem.*, 1964, 322—323).—Evidence is given showing that 4-(2,4-DB) is converted into 2,4-dichlorophenoxycrotonic acid in soil and that this in turn is converted into 2,4-D thus offering further support for the existence of an operative β-oxidation reaction in soil. W. ELSTOW.

γ-Radiation compared with steam and methyl bromide as soil-sterilising agents. C. F. Eno and H. Popenoe (*Proc. Soil Sci. Soc. Amer.*, 1964, **28**, 533—535).—None of the sterilising treatments altered the cation-exchange capacity of a loamy sand or muck soil. Steaming increased extractable N, P and S to a greater extent than did γ-ray and methyl bromide sterilisation. Extractable N was increased more by γ-rays than by methyl bromide. The extent of changes due to the sterilising treatments was greater in the muck than in the loamy fine sand. A. H. CORNFIELD.

Residues of organic mercury compounds in apple. J. D. van Geluwe, W. H. Gutenmann, W. D. Mills and K. G. Parker (*Plant Dis. Reptr*, 1964, **48**, 525—527).—Seven applications of each of four water-sol. org. Hg compounds [di-(*N*-phenylmercuri)-ammonium propionate, diphenylmercuriammonium oxyquinolinate, phenyl mercuric laurylammonium acetate and phenylmercuric oxyquinolate], the last application being approx. 2 weeks before petal fall, resulted in an Hg residue in the fruit of about 25% of that produced by PhHg acetate. A. H. CORNFIELD.

Residual effect of fungicide on larval feeding. K. R. S. Ascher and G. Rones (*Int. Pest Control*, 1964, March/April, Repr., 3 pp.).—Brestan (a formulation of triphenyltin acetate), used for protection against several fungal infections on plants, has toxic effects on a no. of larvae, especially *Prodenia litura* at concn. of 0·1%, with persistence of 1 week after treatment. The product was also effective against *Agrotis upsilon* Rott. Bretan has some phytotoxicity to lucerne. H. S. R.

Pre-emergence herbicides for weed control around young coffee seedlings. J. G. Ibanez (*J. Agric. Puerto Rico*, 1964, **48**, 25—31).—Of a no. of pre-emergence herbicides tested for weed control in coffee seedling plantings the best results were obtained with diuron (2 lb.), neburon (16 lb.), simazine (2 lb.) and Amizine (2—8 lb./acre).
A. H. CORNFIELD.

Some effects of weed competition and herbicide residues on growth and development of bluegrass (*Poa pratensis*). R. K. N. Singh (*Dissert. Abstr.*, 1964, **25**, 1454).—Bluegrass was sown with crab grass (*Digitaria ischaemum* and *D. sanguinalis*). The effects are examined of some herbicides, commonly used on bluegrass turf, on the proportions of the grasses developing and on the rate of uptake of O_2 by leaves of the two grasses. Of 12 herbicides examined 10 persisted in the surface 2 in. of soil for a year in amounts sufficient to be determined by three species of test plants; the two exceptions were DCPA and DMPA. A no. of the herbicides inhibited and others increased the O_2 uptake by grass leaves; species differences in this respect suggest that herbicidal action is, in part, due to modification of respiratory processes. A. G. POLLARD.

Influence of soil properties and microbial activity on the phytotoxicity of linuron and Diphenamid. H. D. Dubrey and J. F. Freeman (*Soil Sci.*, 1964, **97**, 334—340).—Surface samples of 11 types of soil were air dried and treated with herbicides linuron (**I**) [1-methoxy-1-methyl-3-(3,4-dichlorophenyl)urea] and Diphenamid (**II**) (*NN*-dimethyl-2,2-diphenylacetamide) at different rates. The treated soil was watered to field capacity and left overnight. The soil treated with **I** was then seeded with ryegrass and that treated with **II** with oats. After 12 days germination counts were made and after 30 days the plants were harvested and weighed. In another test the soil was sterilised and after treatment with the chemicals was planted as before. The correlation of the ED_{50} values of **I** and **II** with 11 soil properties showed that the ED_{50} of **I** was positively correlated with org. matter content, cation-exchange capacity and exchangeable Mg and K. The ED_{50} of **II** was positively correlated with all but exchangeable K. The highest correlation was obtained with ED_{50} values and the org. matter. When the soils were sterilised **I** was three times and **II** twice as toxic indicating that the herbicides are decomposed by microbial action. T. G. MORRIS.

Fungicidal properties [of complexes of pyridine bases and quinoline with cupric chloride and] of some 1-substituted pyridines. E. S. Cherkasskiĭ, N. N. Selochnik and A. K. Sheĭnkman (*Dokl. Akad. Nauk SSSR*, 1964, **156**, 1197—1200).—Fungicidal properties of complexes of coal tar pyridine bases with $CuCl_2$ and also of five quaternary salts of 4-*p*-dialkylaminophenylpyridines (**I**) were studied. Bases (2 mol.) [pyridine, α-, β- and γ-picoline, 2-methyl-5-vinyl- and 4-ethyl-pyridine, unpurified β-picoline fractions and quinoline (**II**)] were complexed with 1 mol. of $CuCl_2.2H_2O$. All products proved fungicidal against *Fusarium oxysporum* (**III**), *Alternaria humicola* (**IV**) and *Botrytis cinerea* in concn. 0·5% except α- and β-picoline. The **II** complex was effective at 0·1%; it proved more effective than $CuSO_4$ against fungi; in spite of its lower Cu content. Five quaternary salts of **I** were effective Cu-free fungicides, viz., 4-*p*-dimethyl (and diethyl)-amino phenylpyridiniumbenzylobromide, *N*-decyl-4-*p*-dimethyl- (and diethyl)aminophenylpyridinium chloride and *N*-β-(hydroxyethyl)-4-(*p*-diethylaminophenyl)-pyridinium chloride, in concn. 0·036—0·05%, completely killed spores of **III** and **IV**. Roses and chrysanthemums were almost freed from powdery mildew by three sprayings with 0·1—0·25% aq. **II** complex or 0·04% aq. V. 1-Substituted pyridines can be regarded as prospective new Cu-free fungicides. P. W. B. HARRISON.

Effect of nickel salts on development of flax rust *Malampsora lini*. R. K. Kakkar (*Naturwissenschaften*, 1964, 51, 468—469).—It is shown that Ni salts (nitrate slightly better than chloride) had good fungicidal effect of the rust on *Linum usitatissimum* in 0·1—0·3% concn. (spray). No adverse effect in the plants was observed. The leaves had ~6 mg.-% of Ni (dry wt.). H. S. R.

Transmission of common lettuce mosaic virus through the gametes of the lettuce plant. E. J. Ryder (*Plant Dis. Reptr*, 1964, **48**, 522—523).—Lettuce mosaic virus was transmitted in the lettuce plant through the pollen and the ovule. Less than 0·5% infection resulted from pollen infection and over 5% from that through the ovules. A. H. CORNFIELD.

Control of downy mildew on broccoli heads. R. L. Gabrielson (*Plant Dis. Reptr*, 1964, **48**, 593—596).—Bordeaux mixture and basic $CuSO_4$ were the most effective materials for controlling broccoli downy mildew, due to *Peronospora parasitica*. A. H. CORNFIELD.

Control of Sigatoka disease, due to *Mycosphaerella musicola*, of bananas by oil-sprays schedules based on rainfall data. L. Calpouzos, C. Colberg, A. Riollano, C. Ramos and T. Theis (*J. Agric. Puerto Rico*, 1964, **48**, 32—38).—Spraying banana plants with Orchard Spray Oil C only when more than 3 in. of rain fell during the previous 3 weeks (19 applications over the year) gave as good control of the disease as did spraying every 2 weeks, regardless of rainfall, from Jan. to Aug., but not for the rest of the year. A. H. CORNFIELD.

New combinations of nematocides for control of reniform nematode of cotton. W. Birchfield and J. A. Pinckard (*Phytopathology*, 1964, **54**, 393—394).—A combination of pentachloronitrobenzene **I** (2 lb./acre as a 75% emulsifiable formulation) and 1,2-dibromo-3-chloropropane (**II**) (6·45 lb./acre), applied in the row, lowered the infection of cotton seedlings by the nematode more effectively than did **II** alone. Seed dressing with NDT (**III**) (**I**, 7·5; **II**, 15; dieldrin 7·5; granular clay 52·5%) with a methylcellulose sticker, was unexpectedly efficient. Yields of seed cotton were increased by treatment with **II** or **III**. **I** + **II** was the most effective in reducing infection but did not increase yields. A. G. POLLARD.

Recommended analytical methods for pesticides. XXIV. Demeton-ethyl and -methyl. Collaborative Pesticides Analyt. Commee (*FAO Plant Prot. Bull.*, 1964, **12**, No. 3, 2 pp. Repr.).—The products consist of a mixture of *OO*-dialkyl *O*-(2-ethylthio)ethyl phosphorothionate and *S*-(2-ethylthioethyl) phosphorothiolate. The content of mixed isomers is determined by saponification with NaOH in excess and back-titration of the excess, correction being made for free acidity. The *S*-alkyl component is determined by alkaline saponification to mercaptodialkyl-thioether which forms a stable Pb salt sol. in $CHCl_3$ and is titrated iodometrically in the $CHCl_3$ solution. Full details are given. H. S. R.

Gas chromatography retention times and sensitivity data for insecticides and herbicides. E. J. Bonelli, H. Hartmann and K. P. Dimick (*J. agric. Fd Chem.*, 1964, **12**, 333—336).—Some of the basic data of the electron-capture gas chromatography of many of the common pesticides are presented. W. ELSTOW.

Laboratory method for evaluating chemicals as bird repellents. R. I. Starr, J. F. Besser and R. B. Brunton (*J. agric. Fd Chem.*, 1964, **12**, 342—344).—A technique is described for determining an R_{50} value, which is the concn. of a chemical required to repel 50% of the test birds, and from this an estimating equation from which R_1 to R_{99} may be determined. New birds give the most reliable results though previously tested birds may be used for comparative tests. W. ELSTOW.

Laboratory technique for testing bark beetle attractants. R. A. Kliefoth, J. P. Vité and G. B. Pitman (*Contr. Boyce Thompson Inst.*, 1964, **22**, 283—290).—The technique utilises an arena of three layers of ¼-in. masonite pegboard with the $\frac{3}{16}$-in. holes perfectly aligned at 1-in. intervals. A cheese-cloth membrane on the surface provides traction for the beetles and prevents accidental entry; likewise a wire screen between two of the boards prevents escape through ventilation holes. Vials containing test substances are attached below the holes and light, temp. and humidity can be controlled. When recently emerged or trapped beetles of *Ips confusus* Lec or *I. ponderosae* Sw. were released in the centre of the arena they moved very consistently to the pheromones and each species preferred the attractant of its own species. Response was favoured by moist atm. E. G. BRICKELL.

Pesticidal compositions. Chemagro Corp. (B.P. 938,850, 27.4.62. U.S., 27.4.61).—Pesticidal compositions comprising a thiophosphate active agent and a siliceous mineral carrier are stabilised against deterioration by addition of diacetone alcohol (0·5—30 wt.-% on the carrier). F. R. BASFORD.

Insecticidal compositions. Monsanto Chemical Co. (B.P. 937,762, 23.3.60. U.S., 23.3.59).—The storage stability of insecticidal concentrates containing thiophosphate or thiophosphonate as active agent is improved by incorporation of a compound of the general formula R·X·R′ (wherein X is O or S; R and R′ are alkyl of ⩽4 C, cycloalkyl of 4—8 C, or etherified hydroxyalkyl of 4—18 C, or R is H) (1—100) and ester of the general formula R″·Y·R‴ (Y is SO_3, SO_4 or sulphonate group; R″ is H, alkyl of 1—18 C, monocyclic aryl, or alkaryl of up to 16 C in the alkyl portion; R‴ is alkyl of 1—18 C) (0·5—50 wt.-% on active agent). Thus, an insecticidal dust comprising 20% of methyl parathion on attapulgus clay, after storage at 50° during 6 weeks, retains respectively 86, 62 and 98% of active agent in absence of additives, in presence of Et_2SO_4 (5), and in presence of a mixture of Et_2SO_4 (5) and lauryl alcohol (25%). F. R. BASFORD.

Insecticidal compositions. Fisons Pest Control Ltd. (Inventor: C. H. Barker) (B.P. 938,400, 19.3.60).—An improved insecticidal composition of low phytotoxicity, especially suitable for control of insects on greenhouse plants, consists of dimethoate (2·5—40), DDT (5—40), org. solvent (100 pt.) and a surface-active agent of the alkylphenol–ethylene oxide or polyhydric alcohol–fatty acid ester type. F. R. BASFORD.

Insecticidal compositions containing phosphate esters. Fisons Pest Control Ltd. (Inventors: J. F. Harris and H. G. Haynes) (B.P. 937,411, 18.3.59).—The corrosive character of insecticidal compositions containing insecticidal agents of the general formula $OR^{II}(OR^{II})\cdot PX^{I}\cdot X^{II}\cdot CH_2\cdot CO\cdot NR^{III}R^{IV}$ (R^{I} is Me; R^{II} is alkyl of 1—4 C; X^{I} and X^{II} is O or S, but at least X^{I} or X^{II} is S; R^{III} and R^{IV} are H, alkyl of 1—4 C or $NR^{III}R^{IV}$ is heterocyclyl) is minimised by incorporation of a ketone (aliphatic or cycloaliphatic ketone) or ⩽4C (0·1—10 pt.). Formulations described contain *OO*-Me_2 *S*-(*N*-methylcarbamoyl)methyl phosphorodithioate with cyclohexamine or methyl isobutyl ketone. F. R. BASFORD.

Azidoacetic acid ester of pentachlorophenol (I). Chemische Werke Witten G.m.b.H. (B.P. 935,991, 9.4.62. Ger., 2.5.61).—The title compound (m.p. 101°), prepared from the phenol and the acid chloride, has equal bactericidal, fungicidal and insecticidal activities to those of **I** combined with freedom from the main disadvantage of **I** viz., high volatility, irritating action on the mucous membrane, low resistance to washing and leaching, and sensitivity to u.v. rays. H. L. WHITEHEAD.

Seed-treating compositions. Chemische Werke Albert (B.P. 936,584, 22.8.60. Ger., 3.9.59).—An improved seed-treating agent (for affording protection against pests) comprises a granular core (of sand, fine gravel, coke, synthetic fertiliser in granular form, or a soil-improving agent); an inner coating consisting of an adhesive which is capable of adhering firmly to the core but capable of disintegrating in the soil (e.g., dry residue of sulphite waste liquor, casein, glue or cellulose ether adhesive); and an outer, adhesive coating, being different from the inner adhesive and adhering less strongly to the inner coating than the latter adheres to the core, and containing an active material (e.g., natural and/or synthetic resin), so that it is capable of becoming at least partly transferred to seed with which it is admixed. Preferably the core material is of particle size 0·5—5 mm. and contains ⩾20 wt.-% of moisture. F. R. BASFORD.

Imidazole derivatives. Benger Laboratories Ltd. (Inventor: C. Fitzmaurice) (B.P. 939,083, 7.11.60).—A mixture of 4(5)-nitro-

imidazole and Et_2SO_4 is heated near the b.p. for 3 h., then excess of Et_2SO_4 is removed in a vac. The residue is worked up to give *1-ethyl-5-nitroimidazole hydrochloride*, m.p. 189°. The product is active against protozoal infections, e.g., *Trichomonas vaginalis*, *Entamoeba histolytica* and *Histomonas meleagridis*, and poultry feed containing the free base or its salts is especially claimed.
F. R. BASFORD.

Phosphorus-containing esters. Farbenfabriken Bayer A.-G. (Inventors: G. Schrader and W. Lorenz) (B.P. 936,791, 18.5.62. Ger., 19.5.61).—Insecticidal compounds of the general formula $OR^I(OR^{II})\cdot PO\cdot CH(CCl_3)\cdot O\cdot P(X)R^{III}R^{IV}$ are claimed and are obtained in good yield by interaction of $OR^I(OR^{II})\cdot PO\cdot CH(CCl_3)\cdot OH$ with $R^{III}R^{IV}\cdot PXZ$ [R^I and R^{II} are alkyl of 1—4 C; R^{III} and R^{IV} are saturated or unsaturated aliphatic, cycloaliphatic, araliphatic or aromatic hydrocarbon radicals, or R^{IV} may be substituted or unsubstituted alkoxy or cycloalkoxy, or $O\cdot CH(CCl_3)\cdot PO(OR)\cdot OR^{II}$; X is O or S; Z is halogen]. Directions are given for the prep. of *Et 1-dimethoxyphosphinyl-2,2,2-trichloroethyl methylphosphonate* (91% yield). The product, in 0·01% solution, is 100% lethal to caterpillars and aphids.
F. R. BASFORD.

Phosphorothiolic esters and pesticidal compositions comprising them. Murphy Chemical Co. Ltd. (Inventor: M. Pianka) (B.P. 937,361, 20.1.59).—The compounds, which have high acaricidal activity and low toxicity (while some have fungicical and aphicidal activity) are represented by the general formula $OR(OR^I)\cdot PO\cdot S\cdot Z\cdot R^{II}$, wherein R and R^I are alkyl of 1—4 C; Z is S or SO_2; and R^{II} is $CS\cdot NR^{III}R^{IV}$, $CS\cdot OR^V$, alkylthioalkyl or furfuryl, or aryl optionally substituted, but when Z is SO_2 then R^{II} may not be $CS\cdot NR^{III}R^{IV}$ or $CS\cdot OR^V$ (R^{III}—R^V are alkyl or cycloalkyl, or R^{III} is H). One example is *OO-Et₂ S-dimethyldithiocarbamoyl phosphorothiolate*, in the form of a mixture of an oil and a solid. The compound is a very active pesticide and is especially effective against fungi, e.g., spores of *Venturia inaequalis* and *Cercospora melonis*.
F. R. BASFORD.

Insecticides containing 1-(4-halogenophenyl)-1-(4-nitrophenyl)-halogenobutanes and their preparation. Udina S.A. (Inventor: J. E. Biro) (B.P. 936,084, 22.12.59).—There is claimed an insecticide containing as active ingredient a 1-(*p*-halogenophenyl)-1-(*p*-nitrophenyl)butane in which the butyl radical carries at least two halogen atoms, preferably Cl. The insecticide may also incorporate benzil, dioxan, diphenylmethane or xanthone (to inhibit acquired immunity in vermin). The butane compounds may be obtained by condensing a mixture of 1 mol. of a halogenobenzene and 1 mol. of $PhNO_2$ with 1 mol. of a butyl compound containing at least 2 halogen, e.g., αα-dichlorobutanal.
F. R. BASFORD.

Halogenoalkylphenyl carbamates. Upjohn Co. (B.P. 934,576, 28.8.61. U.S., 3.10.60).—Compounds which have insecticidal properties and are much less toxic to man than parathion but are equally potent against citrus fruit mites comprise carbamates of the general formula 2-X-3-R′-4-Y-5-R-C_6H-O·COZ, wherein X is halogen; Y is halogen or alkyl of 1—4 C; R and R′ are alkyl of 1—4 C or R′ is H; Z contains ≯10 C and is selected from alkyl-, alkenyl-, dialkyl-, or dialkenyl-amino, or NZ′; Z′ is alkyl, or oxaalkyl or thia-alkylene, and NZ′ contains 5—9 nuclear atoms. As an example, the method of prep. of *5-ethyl-3-methyl-2,4-dichlorophenyl methylcarbamate*, m.p. 118·5—119·5, is detailed.
F. R. BASFORD.

Phenyl carbamates. Farbenfabriken Bayer A.-G. (Inventors: R. Heiss, E. Böcker, H. Jung and N. Chuo-Ku) (B.P. 937,897, 12.3.62. Ger., 22.3.61).—The carbamates, which have pesticidal properties of superior initial and residual effect (and are especially active against *Aedes aegyptii*) have the general formula $NRMe\cdot CO_2\cdot C_6H_3R^{II}\cdot OR^I$, wherein R is H or Me; R^I is allyl, methallyl or propargyl; R^{II} is H, halogen, alkoxy, alkylthio or dialkylamino; but when R and R^{II} are H then R^I is propargyl. In an example, a conventional method of the prep. is described for *o-propargyloxyphenyl methylcarbamate*, m.p. 75—76°, in 88% yield. Its activity against *Plutella moculipennis*, *Drosophila*, etc., is tabulated.
F. R. BASFORD.

Fungicidal carbamic acid salts. Farbenfabriken Bayer A.-G. (Inventors: H. Lehmann, F. Grewe and W. Lautenschlager) (B.P. 935,981, 19.12.61. Ger., 28.12.60).—There are claimed the *Mn salt* (**I**) of *isopropylenebis-dithiocarbamic acid* (prep. described) and fungicidal compositions containing it. The **I** is especially active against *Phytophthora infestans*, *Cladosporium fulvum*, *Alternaria solani* (on tomato and potato plants), *Peronospora* (*Plasmopara viticola*) on grape vines, *Venturia inaequalis* on fruit with pips, etc.
F. R. BASFORD.

Finely divided cuprous oxide. Imperial Chemical Industries Ltd. Inventors: A. Campbell and A. E. Taylor) (B.P. 936,922, 22.12.60).—The oxide, suitable for use as fungicide, is produced in finely divided form by adding alkali to a Cu_2Cl_2 solution at 30—90° and at such a rate as to maintain pH 6—9.
F. R. BASFORD.

Fungicidal compositions and compounds. Boots Pure Drug Co. Ltd. (Inventors: H. A. Stevenson, J. R. Marshall and A. F. Hams) (B.P. 938,890, 3.2.61).—There is claimed a fungicidal composition in which the active ingredient is a compound of the general formula $2,4,1\text{-}C_6H_3(NO_2)_2\cdot SO_nR$, wherein *n* is 1 or 2; when *n* is 1, then R is alkyl of 5—7 C, cycloalkyl, phenylalkyl, phenylalkyl substituted by halogen or NO_2, or Ph optionally containing halogen, alkyl, alkoxy or carbalkoxy; when *n* is 2, then R is alkyl, halogenomethyl, halogenopropyl, cycloalkyl, phenylalkyl or phenylalkyl substituted by halogen or NO_2. Method of preparing such compounds comprises oxidising the corresponding sulphide with H_2O_2. One compound prepared is *p-chlorophenyl 2,4-dinitrophenyl sulphoxide*, m.p. 144·5—145·5°.
F. R. BASFORD.

Dithiocarbamic acid derivatives and compositions containing them. J. R. Geigy A.-G. (B.P. 938,907, 17.11.61. Switz., 21.11.60).—Compounds $NHMe\cdot CS_3\cdot C_6H_3R'R''$ (R and R′ are H, halogen or alkyl of 1—4 C, or R is NO_2), useful as fungicides, herbicides, insecticides and nematocides, are obtained by interaction of a salt of $NHMe\cdot CS_2H$ with $C_6H_3RR'\cdot SX$ (X is halogen). A representative compound is *S-phenylthio methyldithiocarbamate* (*N-methyl-thiocarbamoylphenyl disulphide*), m.p. 63—64°.
F. R. BASFORD.

Amino-thiocyanatotriazines. Deutsche Gold- u. Silber-Scheideanstalt (Inventor: W. Schwarze) (B.P. 938,900, 17.4.61).—The compounds of the invention, which are useful as fungicides and herbicides comprise 2-thiocyanato-4-NR′R″-6-NHR‴-*s*-triazines, obtained by reacting a corresponding 2-mercapto analogue with a cyanogen halide in fluid medium at −50° to +10° in presence of H halide acceptor (R′—R‴ are alkyl of 1—5 C). Thus, a mixture of 4-ethylamino-6-diethylamino-2-mercapto-*s*-triazine, acetone and N-NaOH is heated for a short time, then after cooling to −5° CNCl is added. The mixture is stirred at −5° for 10 min., and pptd. solid is filtered off and dried, to give *4-ethylamino-6-diethylamino-6-thiocyanato-s-triazine*, m.p. 86°. It can be recrystallised from aq. MeOH. Its fungicidal activity against cucumber powdery mildew is described.
F. R. BASFORD.

Nematodicidal agents. Schering A.-G. (B.P. 936,918, 4.4.60. Ger., 9.4.59).—A composition for combating nematodes contains as active agent the Zn salt of methyldithiocarbamic acid.
F. R. BASFORD.

Animal Husbandry

Magnesium in ruminant nutrition. V. Indirect determination of the intake of magnesium, calcium and potassium by the grazing cow. A. C. Field (*Brit. J. Nutr.*, 1964, **18**, 357—368).—All faeces and urine were collected from three old cows for three days; they were fed on hay and then cut herbage. Each cow was given a standard dose of Cr_2O_3 twice daily and the concn. and amounts of Ca, K, Mg were determined in the excreta: Cr_2O_3 was determined in the faeces and creatinine (**I**) in the urine. In general the diurnal variation in the ratios of Ca, Mg and K to **I**, were in order of increasing coefficient of variation: K : **I**, Mg : **I** and Ca : **I**. None of the ratios showed well-developed diurnal rhythms. Neither was this found in the Ca, Mg and K ratios of concn. to Cr_2O_3 but there was a tendency for the Ca : Cr_2O_3 to be lower in the evenings and higher in daylight. Other aspects are discussed. (23 references.)
C. V.

Influence of feeding above the [Netherlands] starch equivalent standards on live weight and production of dairy cows. N. D. Dijkstra and A. M. Frens (*Versl. landbouwk. Onderz.*, 1963, 69.18, 79 pp.).—In three winter-feeding experiments, the daily increases in milk per cow attained per 1 kg. of surplus starch equiv. were 1·02—1·35 kg. of standard milk. The surplus feeding had some effect on maintaining the live wt., no effect on the fat % of the milk, and caused an increase of 0·1—0·2% in the solids-not-fat. (13 references.)
P. S. ARUP.

Pasture rearing and milk replacer feeding of dairy calves. A. D. L. Gorrill (*Canad. J. Anim. Sci.*, 1964, **44**, 235—247).—In trials with bull calves comparisons are made of indoor with pasture feeding, of whole milk with milk-replacer diets and with different amounts of starter rations, from weaning to 15 weeks of age. The average daily gain in wt. from one week to weaning (130 lb.) was 1·05 lb. with little difference between treatments. Indoor feeding and/or use of milk replacers considerably increased feed consumption. The gain from weaning to 15 weeks was greater on pasture when the pre-weaning diet was whole milk than when it was milk replacer.
A. G. POLLARD.

Relation between birth weight, subsequent weights, body weight gain and feed consumption of Holstein-Friesian steers. R. J. Forrest (*Canad. J. Anim. Sci.*, 1964, **44**, 187—194).—The birth wt. of a bull calf was related positively to the gestation period of the dam, an increase of one day in the gestation period representing

1 lb. increase in the birth wt. of the calf. Birth wt. was also correlated with the body wt. of the steer up to 7 months but the relationship had disappeared at 14 months of age. With calves of 87—112 lb birth wt. the correlation was significant only to 4 months. No correlation between birth wt. and rate of gain in wt. was apparent after the live wt. reached 200 lb. The performance of the animal in the early growth stages cannot be used to predict later development beyond the calf stage. A. G. POLLARD.

Selective grazing induced by animal excreta. I. Evidence of occurrence and superficial remedy. G. C. Marten and J. D. Donker (*J. Dairy Sci.*, 1964, **47**, 773, 776).—Spraying of sugar or molasses on dung-affected areas of grass rendered such areas acceptable to cattle. M. O'LEARY.

Estimation of forage consumption of dairy cows from measurements of body-weight change, body weight, milk production and concentrate consumption. D. G. Davenport (*Dissert. Abstr.*, 1964, **25**, 1459—1460).—Various mathematical procedures for calculating intakes of estimated net energy are investigated on the basis of data obtained in feeding trials with 67 cows, over 1—10 lactations, observations being made in each of 11 stages of lactation. Estimates based on use of data obtained regardless of stage of lactation or no. of lactations were less satisfactory. A. G. POLLARD.

Comparison of nitrogen-fertilised orchard grass or lucerne at different levels of concentrate feeding for lactating dairy cows. W. P. Apgar (*Dissert. Abstr.*, 1964, **25**, 1456—1457).—The voluntary intakes of N-fertilised orchard grass (*G*) and of lucerne (*L*) as hay or as silage, separately or with concentrates, are examined in relation to milk production. Over a period of 8 months the *G* resulted in lower daily intake, lower milk yields with lower solids-not-fat content but the same butter-fat level and greater utilisation of body reserves as compared with *L*. Supplementary grain feeding lowered forage intakes (dry basis) and increased milk yields to similar extents with both forages. The digestibility of the rations showed no significant difference as between the fifth and thirty-fifth weeks of lactation. Energy digestibility was lower for the grass ration. The crude protein contents of the two forages were similar; the inferiority of *G* is attributable to the lower voluntary intake. A. G. POLLARD.

Coumarin and related compounds of *Anthoxanthum puelii* and *Melilotus alba* and dicoumarol formation in spoilt sweet vernal [grass] and sweet clover hay. E. G. Davies and W. M. Ashton (*J. Sci. Fd Agric.*, 1964, **15**, 733—738).—Chromatographic and spectrophotometric techniques were used. Very small amounts of dicoumarol (the anticoagulant) were found in the hay and larger amounts were present when the hay was inoculated with *Penicillium jenseni* and when formaldehyde was added. During spoilage the coumarin content of the hay decreased and the related compounds increased to a steady level in about 4 weeks. In mixed oatgrass/cocksfoot hay, added *trans*-2-hydroxycinnamic acid and not coumarin gave rise to 4-hydroxycoumarin which with formaldehyde gave dicoumarol. While mouldy sweet vernal hay may contain dicoumarol, unless such hay contains a much higher concn. than was found experimentally, it is unlikely to cause 'sweet clover disease'. (20 references.) E. M. J.

Toxic and anti-vitamin E factors in raw kidney beans (*Phaseolus vulgaris*). H. F. Hintz (*Dissert. Abstr.*, 1964, **25**, 1463).—Constituents of the beans which cause nutritional muscular dystrophy (*NMD*) in lambs are examined with particular reference to a possible anti-vitamin E factor. Rats and guinea pigs fed a ration containing 74% of raw bean and 10% of casein died within 14 days; the toxic effect was not reduced by supplements of vitamin E, Se, ethoxyquin or Ca pantothenate but was eliminated by autoclaving the beans for 30 min. at 15 p.s.i. Soaking the beans in water diminished the toxicity but the beans still lowered the rate of gain in wt. of rats. Diets containing 18·5% of raw beans and 24% of casein produced almost normal growth. The digestion coeff. of raw bean protein was approx. 28% and was unaffected by supplementary trypsin; it was increased to 75% by autoclaving the material. Evidence of the presence of an anti-vitamin E factor was obtained in raw and in water-soaked beans in trials with rats and chicks. With chicks, in contrast to rats, feeding autoclaved beans increased the vitamin E requirement; extraction of beans with ethanol lowered but did not eliminate this effect. Addition of linoleic to ethanol-extracted beans in a chick ration increased the incidence of *NMD*. The lipoxidase activity and the anti-vitamin E activity were apparently unrelated. A. G. POLLARD.

Protein nitrogen in rumen ingesta of steers fed single or mixed sources of nitrogen with and without added grain. M. Akram (*Dissert. Abstr.*, 1964, **25**, 1456).—The protein present in the rumen ingesta of fistulated steers was determined 6 h. after feeding a basal ration of prairie hay supplemented with various N sources. The ability of the N sources to provide protein in the rumen differed considerably. Oilseed meals (soya-bean, cottonseed) were the best of protein sources examined and, fed individually, were equal or superior to combinations of N sources. A. G. POLLARD.

Diurnal excretion of chromium oxide by ruminants when administered in sustained-release pellets. W. J. Pigden, K. A. Winter, G. J. Brisson and G. I. Pritchard (*Canad. J. Anim. Sci.*, 1964, **44**, 207—214).—An extruded pellet containing 10 g. of Cr_2O_3 (dissolution time 4—5 days) and a pressed pellet [Cr_2O_3, 32 g. (for cattle); 20 g. for calves or sheep; dissolution time 16—20 days] are compared as indicators in determinations of faecal output of animals on pasture. The diurnal excretion cycle occurring with conventional dosing with Cr_2O_3 was eliminated by the pellets. Small differences were apparent between grab samples of faeces from cattle grazing under different systems. The pellets are useful in trials with animals in confinement, but have only restricted value for those on pasture. A. G. POLLARD.

Factors related to the utilisation of carbohydrates by ruminants. I. Effect of varying concentrate-to-hay ratios on the efficiency of ration utilisation. II. Effect of dehydrating and pelleting on carbohydrate utilisation. R. G. Hinders (*Dissert. Abstr.*, 1964, **25**, 1462—1463).—I. Fistulated cows were fed rations in which different proportions (30—90%) of the estimated net energy were supplied as concentrates. The requirements of digestible energy and total digestible nutrients (*TDN*) per lb. of fat-corrected milk (*FCM*) diminished with increase in the % of dietary concentrate. The estimated net energy for *FCM* production was similar for all rations With increase in the % of concentrate in the ration, the relative proportions of acetic acid in the rumen-volatile fatty acids (*VFA*) diminished, that of butyric acid increased and that of propionic acid remained unchanged. Relations between wt. of *TDN* utilised per lb. of *FCM* and the concn. of total *VFA* and various ratios of individual acids, are established.

II. The ruminal and total digestion of the carbohydrate of lucerne fed (*a*) long or (*b*) dried and pelleted, were compared in fistulated steers. Less N-free extract was digested in the reticulo-rumen with *b* than with *a*. More of the digestible fibre disappeared in the reticulo-rumen when *a* than when *b* was fed. The proportions of individual acids in the ruminal *VFA* differed with the two types of feed. A. G. POLLARD.

Bermuda-grass hay, citric acid and yeast in steer rations. R. C. Gray, jun. (*Dissert. Abstr.*, 1964, **25**, 1461).—In conventional metabolism trials with steers, digestibilities of high roughage rations containing Bermuda-grass and Pensacola bahia-grass hays, with and without a concentrate or lucerne meal were tested. The supplementary concentrates and, to a small extent, lucerne improved the digestibility of the grasses. In other trials using pelleted rations containing the basal ration with cottonseed meal and ground maize showed improved digestibility on addition of 1% of lucerne meal; 4% of the meal had the reverse effect. A finishing ration (30% hay + concentrates) was supplemented with citric acid (1·63%) and/or yeast 1%. Digestibility was highest in the ration containing no yeast and lowest in that to which yeast and citric acid were added. Yeast lowered protein digestibility. The performance of animals in the growing phase did not influence that in the finishing phase. A. G. POLLARD.

Uptake by plants of diethylstilboestrol and of its glucuronide. B. Gregers-Hansen (*Plant & Soil*, 1964, **20**, 215—220).—The possibility that the oestrogen diethylstilboestrol (used to enhance the fattening of livestock) which, together with its glucuronide, is excreted by the animal may accumulate in soil and be absorbed by plants was studied. In pot tests uptake of the two compounds by a no. of species from soil and solution culture was so small that the possibility of a toxic hazard to humans is unlikely. A. H. CORNFIELD.

Certain effects of tranquillisers, with and without diethylstilboestrol, on ration utilisation by bovine animals. M. M. McCartor (*Dissert. Abstr.*, 1964, **25**, 1463—1464).—In feeding trials and digestibility studies with steers, two tranquillisers (trifluomeprazine and Trans-Q) fed singly or in combination with diethylstilboestrol had no significant effects on rates of gain in wt., N retention, digestibility of the ration or on carcass characteristics. A. G. POLLARD.

Bloat investigations: foam stabilising protein of lucerne. J. M. McArthur, J. E. Miltimore and M. J. Pratt (*Canad. J. Anim. Sci.*, 1964, **44**, 200—206).—Foam formation in the rumen of cattle grazing lucerne is caused by a protein present in the leaves. Fractionation of fresh lucerne protein on columns of agar gel granules and physical examination of the fractions show the active material to be an 18-S protein. A. G. POLLARD.

Relation of rumen mucinolytic bacteria and of rumen protozoa to bloat in cattle. B. Mishra (*Dissert. Abstr.*, 1964, **25**, 1464).—

Lucerne hay (4 lb.) fed to fistulated cattle prior to grazing lucerne or to the feeding of freshly cut lucerne, prevented bloat. The no. of aerobic and anaerobic mucinolytic bacteria in the rumen of bloated animals and of those recently fed fresh lucerne were greater than in those fed lucerne hay. In general, rumen fluids of bloated animals showed greater mucinolytic activity than did those of non-bloated animals receiving the same feed. Bloat was reduced by feeding anti-foaming agents, mucin, starch or dextrose via rumen fistulae and in most cases there was a corresponding reduction in no. of mucinolytic bacteria. Inoculation of the rumen with mucinolytic organisms accentuated bloat conditions in animals fed bloat-inducing rations. Protozoa were less numerous in rumens of bloated than in those of non-bloated animals. Possible effects of a decrease in salivation and thus of mucin in facilitating bloat are indicated.
A. G. POLLARD.

Effects of various low-protein diets on the distribution of ruminal nitrogen and on the nitrogen required for maintenance of African sheep. R. C. Elliot and J. H. Topps (*Anim. Prod.*, 1964, **6**, 345—355).—Total N and NH_3-N content of the rumen are both closely related to dietary N, the diurnal changes in N content being most marked with the higher protein feeds. With a veld grass containing 2·8% of crude protein NH_3 levels are unusually high due to recycling of urea. Coupled with high rumen NH_3 concn. are high apparent requirements of digestible N, although the former is not likely to be the cause of these high requirements. N equilibrium is found only where total digestible nutrient intake is adequate for maintenance.
M. LONG.

Effect of Aureomycin supplementation on urea utilisation in the rations of growing-fattening lambs. D. Cahill and D. M. McAleese (*Sci. Proc. R. Dublin Soc.*, 1964, **1**, 123—130).—Substitution of urea for soya-bean meal in a barley-soya-bean meal diet significantly depressed the growth rate and feed conversion of lambs. Supplementation with Aureomycin (20 p.p.m.) increased the growth rate and N retention due to improved feed conversion.
S. A. BROOKS.

Effects of hormones, buffer salts and protein quality on the feeding of spring lambs. W. C. Woolfitt, W. E. Howell and J. M. Bell (*Canad J. Anim. Sci.*, 1964, **44**, 179—183).—Rates of gain in wt., feed efficiency and carcass grading were not improved by ear implants of oestradiol–progesterone (**I**) or diethylstilboestrol (**II**). Orally administered **II** lowered the rate of gain in wt. and feed efficiency. Lambs receiving $NaHCO_3$ grew slightly faster than did controls and showed somewhat higher feed efficiency but no significant effect on carcass quality. Na citrate (2%), with or without $NaHCO_3$ (2%), in the diet restricted growth rates somewhat and lowered feed efficiency. Fish meal in the ration gave results similar to those of linseed-oil meal.
A. G. POLLARD.

Comparison of the feeding value of maize and sorghum for fattening pigs. F. X. Vanschoubroek, R. L. Spaendonk and W. Nauwynck (*Anim. Prod.*, 1964, **6**, 357—362).—With live wt. gain, food conversion efficiency, slaughter quality and ham composition as criteria no statistical difference is found between 40% maize ration and 40% *Sorghum vulgare subglabrescens* rations.
M. LONG.

Influence of energy and protein level in rations for finishing market pigs on performance and carcass characteristics. D. E. Waldern (*Canad. J. Anim. Sci.* 1964, **44**, 168—173).—Comparison is made of two levels of protein and two levels of energy in self-fed finishing rations for pigs. High-energy rations produced faster growth with lower feed consumption and carcasses having more loin and back fat than did the low-energy ration. Levels of protein had no significant effect on rate of gain in wt., feed consumption or carcass quality. An interaction is shown between the protein and energy contents of the ration such that the adverse effect of the high-energy ration on carcass quality was modified by a high level of dietary protein.
A. G. POLLARD.

Effects of oxytetracyline and oleandomycin separately and together in pig diets. W. C. Smith, J. L. Adam and H. M. Tonks (*Anim. Prod.*, 1964, **6**, 363—368).—Pigs receiving rations containing a mixed supplement of oxytetracyline (**I**) (10 g./ton) and of oleandomycin (**II**) (2·5 g./ton) and **I** (10 g./ton) grow faster than those receiving **II** alone. There is no difference between the mixed supplement and **I** by itself. Visceral wt. are unaffected by antibiotics although pigs receiving **II** have a shorter gut.
M. LONG.

Protein requirement of the laying hen. R. Shapiro (*Dissert. Abstr.*, 1964, **25**, 1466).—A system of feeding protein and protein-free diets separately permitted close control of protein and energy intakes. Whole-egg protein, intact or solvent-extracted, was the sole source of N. On the basis of N retention and egg production the daily protein requirement thus determined was 13—14 g. The efficiency of conversion of retained N into egg-N was probably $\geq 50\%$. The min. level of protein (using egg albumin) needed to provide near-max. N retention (1200 mg. N/head/day) was 16—17 g./head daily. At this level the efficiency of N retention was 44·5%. Of the retained N approx $\frac{2}{3}$ was used for synthesis of egg protein. Use of a mixture of amino-acids simulating egg albumin in a ration resulted in effective N retention and egg protein but 25% replacement of the acids by egg protein was necessary to maintain egg size. The principal limiting N factor in egg protein was the total N rather than any essential amino-acid N.
A. G. POLLARD.

Limiting amino-acids in laying hen diets. D. G. Britzman (*Dissert. Abstr.*, 1964, **25**, 1457—1458).—Supplements of methionine and/or lysine were added to layers' rations (16% protein) for hens kept in (*a*) cold-walled houses with litter floor or (*b*) in individual cages in a warm house. In (*a*) no improvement in egg yield, feed efficiency, body wt., egg wt., Haugh units, fertility or hatchability resulted from the supplements. All these factors showed slight but not significant improvement when an 11%-protein ration containing 0·2% of DL-methionine was supplemented with 0·3% of L-lysine. Egg production with this ration was inferior to that with a 16%-protein ration; body wt. was lost with the former but gained with the latter ration. In (*b*), a methionine supplement to a low-protein–high-energy ration had no beneficial effect but a combination of methionine (0·2%) and L-lysine (0·3%) increased egg production and feed efficiency without affecting body wt., egg wt. or Haugh units. Of a range of amino-acids examined only methionine affected (improved) laying performance. In high-energy–low-protein rations tryptophan in addition to methionine and lysine may become a limiting factor.
A. G. POLLARD.

Processed hatchery by-product as an ingredient of poultry rations. E. L. Wisman (*Poultry Sci.*, 1964, **43**, 871—876).—Hatchery by-product meal (prep. from infertile eggs, dead embryos, unsaleable chicks, etc.) gave satisfactory wt. gains in broilers when added to their diet. Unidentified growth factor(s) activity was present in the material. Combinations of hatchery by-product meal with blood meal, feather meal and meat and bone meal satisfactorily replaced up to 50% of soya-bean meal protein in the diet of birds to 4 weeks of age.
A. H. CORNFIELD.

Utilisation by chickens of energy from faeces. W. J. Pryor and J. K. Connor (*Poultry Sci.*, 1964, **43**, 833—834).—Replacement of 20% of crushed sorghum by faeces in a chick trial showed that the faeces had a metabolisable energy value of 1·10 kcal. per g. of dry matter, which was about 30% of the value of the feed from which it originated.
A. H. CORNFIELD.

Chick feeding value of meals prepared from glandless cottonseed. C. Jonston and A. B. Watts (*Poultry Sci.*, 1964, **43**, 957—963).—Glandless cottonseed meals (produced from cotton bred to contain very few pigment glands), which were extracted with hexane and air-dried, were of lower nutritive value than when they were mildly heated after flaking or extracted with hexane–acetone–water. Lysine and methionine were limiting amino-acids in meals prepared from both glandless and glanded cottonseed, but when the meals were heated lysine was more limiting where glanded than where glandless seed was used. Unheated hexane-extracted glandless meals when fed as the main source of protein in a 21% ration caused slight gumming around the beaks of chicks. This property was eliminated when the unextracted flakes were either mildly heated or extracted with hexane–acetone–water. Treatment with this mixed solvent also improved the feeding value of glanded cottonseed meals. Glandless meals produced by two current commercial processes were of the same nutritional value as soya-bean meal.
A. H. CORNFIELD.

Evaluation of meat meal as a protein supplement for the chick. J. D. Summers, S. J. Slinger and G. C. Ashton (*Canad. J. Anim. Sci.*, 1964, **44**, 228—234).—Feeding trials with growing chickens using meat meal as sole protein, with and without supplements of the essential amino-acids which are deficient in meat, demonstrated that the lowered efficiency of meat meal for chickens results from amino-acid imbalance rather than from inadequate digestibility. Comparison with soya-bean meal indicates that the amino-acids limiting the nutrient value of meat meal are probably (in diminishing order) methionine, tryptophan, isoleucine, cystine, threonine, arginine. In experimental diets containing meat meal as sole protein, excessive proportions of P and Ca may contribute to the poor response to such diets.
A. G. POLLARD.

Influence of solvent-extracted fish meal and stabilised fish oil in broiler rations on performance and on the flavour of broiler meat. J. O. Hardin, J. L. Milligan and V. D. Sidwell (*Poultry Sci.*, 1964, **43**, 858—860).—Growth and feed efficiency of broilers to 9 weeks of age were similar with all levels of added solvent-extracted fish meal (5—15%) plus stabilised fish oil (0—1·5%). Significant off-flavours in the meat occurred only when the highest level of both supplements were added in combination.
A. H. CORNFIELD.

Interactions of fats and fatty acids as energy sources for the chick. N. R. Artman (*Poultry Sci.*, 1964, **43**, 994—1004).—The utilisation by chicks of relatively saturated fats or fatty acids was increased by mixing them with relatively unsaturated fats or fatty acids. Increasing the proportion of unsaturated to saturated fat resulted in successively smaller increases in the utilisation of the saturated fat. Although menhaden fish oil was well utilised and mixtures of it with other fat produced good growth and feed efficiency, at high levels it tended to suppress chick growth. A. H. CORNFIELD.

Phosphorus availability studies with feed grade phosphates. B. C. Dilworth and E. J. Day (*Poultry Sci.*, 1964, **43**, 1039—1044).—The relative availability to chicks of the P in a no. of materials, compared with that in NaH_2PO_4 as 100, was 93 for β-$Ca_3(PO_4)_2$, 54—63 for low-F rock phosphates, 56—84 for defluorinated phosphates, and 38% for soft phosphate. Materials with availabilities >79% did not differ significantly from the standard in bone ash response. A. H. CORNFIELD.

Comparison of three methods of supplying calcium to laying hens. A. L. Mehring, jun. (*Poultry Sci.*, 1964, **43**, 976—981).—The performance of laying hens receiving 2·45% Ca in the feed, 1·25% Ca in the feed with free access to calcite crystals, and 0·20% Ca in the feed with free access to Ca was studied over one year. There were no significant differences due to treatment in egg production, efficiency of feed utilisation and hatchability of eggs. There was a tendency for shells to be stronger where free access to Ca supplement was provided. A. H. CORNFIELD.

Availability of phytic acid phosphorus for chicks. III. Effect of calcium and vitamin D_3 levels on the utilisation of calcium phytate. P. W. Waldroup, C. B. Ammerman and R. H. Harms (*Poultry Sci.*, 1964, **43**, 926—931).—The availability of the P of Ca phytate to chicks increased with decreasing dietary Ca level or Ca : P ratio. At low levels the availability of the P or Ca phytate was similar to that of $CaHPO_4$ as indicated by growth and bone calcification. However, these low levels of Ca were not adequate for optimum performance of the chicks. Increasing the vitamin D_3 level of the diet above 360 I.C.U. per lb. of feed increased the availability of P from both sources. A. H. CORNFIELD.

Calcium and phosphorus requirements of White Leghorn pullets from 8 to 21 weeks of age. L. R. Berg, G. E. Bearse and L. H. Merrill (*Poultry Sci.*, 1964, **43**, 885—896).—The Ca requirement in the diet of pullets over 8—21 weeks of age for optimum growth, bone ash and subsequent laying house performance was $\geqslant$0·4% and the P requirement was $\geqslant$0·3% when the Ca : P ratio of the diet ranged from 1 : 1 to 1 : 2. The entire P requirement could be supplied by P of plant origin. A. H. CORNFIELD.

Acceptance of arsenicals and antibiotics for use in laying mashes. D. K. Andrews, H. R. Bird and M. L. Sunde (*Poultry Sci.*, 1964, **43**, 903—908).—A review. A. H. CORNFIELD.

Influence of dietary selenium and age on the metabolism of selenium-75 by chicks. P. L. Wright and F. R. Mraz (*Poultry Sci.*, 1964, **43**, 947—954).—Growth rate of chicks was severely reduced by the addition of Se (16 p.p.m. as Na_2SeO_3) to the diet, and moderately reduced by the addition of Se at 8 p.p.m. Feed consumption decreased only after the fourth week, and then only when the feed contained Se 16 p.p.m. Of the ingested Se 83% was found in the faecal and urinary excretion. Approx. 0·8% and 0·2% of a single oral dose of ^{75}Se was found 120 h. after dosing in the livers and kidneys when the added Se was 8 and 16 p.p.m. respectively. These values were not greatly influenced by age of birds or the period over which the Se diets were supplied. A. H. CORNFIELD.

Nutritional relationships between vitamin E, selenium and related factors in the domestic fowl. E. D. J. Walter (*Dissert. Abstr.*, 1964, **25**, 1467).—Depletion of Se and vitamin E (**I**) in chicks did not affect the respiration of liver or brain slices. The distribution of intraperitoneally injected ^{75}Se in tissues of 3-week chicks was unaffected by dietary **I**. Ethoxyquin (**II**) (0·05%) fed continuously diminished the occurrence of exudative diathesis, possibly by protecting the reserve **I** in tissues. Turkey poults, on a *Torula* yeast–Cerelose diet, showed a high incidence of muscular dystrophy in the gizzard; this was unaffected by supplementary feeding of cystine (0·15), methionine (0·4), ethoxyquin (**II**) (0·025%) or Se (0·01—0·1 mg./kg.). Larger proportions of **II** (0·3%) gave partial protection; complete protection was afforded by **I** (20 i.u.) or Se (1 mg./kg.). Anaemia was prevented as effectively by Se (0·1 mg./kg.) or **II** (0·3%) as by **I**. Increased serum-glutamic-oxaloacetic transaminase serves as a reliable test for muscular dystrophy in chicks or turkey poults. A. G. POLLARD.

Effect of acetylsalicylic acid and oxytetracycline on the performance of hens and broiler chicks. B. L. Reid, A. A. Kurnick, J. M. Thomas and B. J. Hulett (*Poultry Sci.*, 1964, **43**, 880—884).—Addition of 0·05% of acetylsalicylic acid (**I**) to the diet of laying hens increased egg production 4—6% during both warm and cool weather. Adding 0·005—0·080% of **I** to the diet of broiler chicks to 4—8 weeks of age had no effect on wt. gains or feed efficiency. Addition of oxytetracycline (25 g. per ton of feed) to the diet of laying hens increased egg production 5%. A. H. CORNFIELD.

Effects of oestrogenic hormones, terephthalic acid and calcium level upon oxytetracycline utilisation by chicks. R. H. Harms, H. R. Wilson and P. W. Waldroup (*Poultry Sci.*, 1964, **43**, 970—973).—Reducing the dietary Ca level from 1% to 0·4%, addition of terephthalic acid (8 lb./ton) or dienestrol diacetate (0·352 g./kg. of diet), or injection of diethylstilboetrol (0·015 g. per bird) or estradiol (0·001 g. per bird) all significantly increased the oxytetracycline level in the blood of chicks receiving 400 g. of the antibiotic per ton of feed. A. H. CORNFIELD.

Effect of 17-α-ethyl-17-hydroxynorandrostenone (Nilevar) on rate of lay in the turkey. K. I. Brown (*Poultry Sci.*, 1964, **43**, 966—970).—Injection of Nilevar (0·008—0·016 g. per hen) into laying hens caused cessation of oviposition for 12 days, after which the hens gradually resumed laying at the pre-injection rate. The treatment did not increase total egg production or fertility. A. H. CORNFIELD.

Effects of continuous and intermittent reserpine and different choline treatments on the growth and reproductive performance of turkeys. G. W. Friars, S. J. Slinger and W. F. Pepper (*Poultry Sci.*, 1964, **43**, 941—946).—Addition of reserpine (0·25 p.p.m.) to the diet of poults from 9 to 19 weeks of age reduced wt. gains of turkeys, whilst treatment during the 12th and 15th weeks of age increased wt. gains to 19 weeks of age. Both reserpine treatments reduced feed efficiency. Choline (0·441—0·517 g./lb. of feed) had no consistent effect on wt. gains over the whole period, although growth of males was enhanced and that of females depressed during some parts of the growing period. The treatments had no significant effect on fertility, but the continuous reserpine trestment depressed hatchability of fertile eggs. A. H. CORNFIELD.

Effect of fasting prior to slaughter on yield of broilers. M. J. Smidt, S. D. Formica and J. C. Fritz (*Poultry Sci.*, 1964, **43**, 931—934).—There were no significant losses in dressed wt. of birds which were fasted for up to 16 h., but supplied with water, before slaughter. Dressed wt. decreased significantly when the fasting period before slaughter ranged from 24 to 40 h. A. H. CORNFIELD.

Performance of single and multiple caged White Leghorn layers. R. W. Lowe and B. W. Heywang (*Poultry Sci.*, 1964, **43**, 801—805).—Mortality averaged 34% where five birds were kept in a 24 × 18 in. cage, 16% with two birds in a 12 × 18 in. cage, and <8% with single birds in 8 or 10 × 18 in. cages. A tranquilliser did not reduce mortality in the largest cage. On a hen-housed basis egg production was 24% less in the largest and 6% less in the medium size cages than in the smallest cages. There was little difference due to treatment on total feed consumption or feed efficiency with respect to egg production, but wt. gains were greatest in the multiple cages birds. A. H. CORNFIELD.

Influence of temperature and humidity on broiler performance. J. L. Milligan and P. N. Winn (*Poultry Sci.*, 1964, **43**, 817—824).—Tests in controlled environment chambers showed that constant temp. between 15·5 and 26·7° gave optimum wt. gains, feed conversion, pigmentation and feathering of broilers between 5 and 10 weeks. High R.H. had an adverse effect on performance at 35—37·8° but not at lower temp. Varying R.H. had no consistent effect on performance at temp. lower than 15·5°. A. H. CORNFIELD.

Rearing and maintaining pullets on controlled lighting. R. K. Noles and R. E. Smith (*Poultry Sci.*, 1964, **43**, 848—857).—Birds restricted to 6 h. light per day from 8—21 weeks of age consumed less feed, showed reduced wt. gains and matured 6 days later than did birds reared under natural daylight. Egg production and wt., and mortality of birds over 350 days, were no different between birds receiving a constant 15-h. daylength and those receiving natural daylight plus 15 min. extra daylight on successive weeks. A. H. CORNFIELD.

Microbiological survey of poultry houses and hatcheries; isolation, characterisation and pathogenicity of the more commonly found bacteria. R. P. Singh (*Dissert. Abstr.*, 1964, **25**, 1496—1497).—Counts were made of micro-organisms in the air, trough-water and litter in poultry houses and hatcheries. In general, hatcheries having a common incubator and hatcher room showed rather larger counts. Raising chicks on wire resulted in lower counts than did raising on sawdust litter. Details of species distribution and certain characteristics of the organisms are recorded. A. G. POLLARD.

Effects of air and/or heat on the rate of accumulation of solids in indoor manure digestion tanks (indoor lagoons). A. A. Al-Timimi,

W. J. Owings and J. L. Adams (*Poultry Sci.*, 1964, **43**, 1051—1056).—The dry-matter % of the manure in tanks under hen cages was not significantly affected by heating (35·5°) and/or aerating the liquid in the tanks, although more water was required to maintain the level in the tanks where heating and aeration were practised. A. H. CORNFIELD.

Effect of lowering dietary protein level of laying hens during the production period. W. J. Owings (*Poultry Sci.*, 1964, **43**, 831—833).—Reducing the protein content of the diet from 17·5% to 15·3% or 13·3% after 16 weeks' production did not reduce egg production, body wt., egg size or Haugh unit scores during the following 24 weeks of production, but increased feed efficiency with respect to egg production. A. H. CORNFIELD.

Response of laying hens to dietary oils and purified fatty acids. J. E. Marion and H. M. Edwards, jun. (*Poultry Sci.*, 1964, **43**, 911—918).—Hens which had been reared on low-fat diets were supplied with coconut, maize and menhaden oil and methyl oleate and linoleate. Egg production, size and hatchability were positively associated with the linoleic acid content of the diet. High levels of eicosatrienoic acid were present in the liver and heart tissue of fat-deficient hens. The level of this acid was not reduced by feeding coconut oil and methyl oleate, but the acid disappeared from the tissues when methyl linoleate, maize oil and menhaden oil were supplied. The level of the acid in the tissues was a valid indicator of linoleic acid deficiency except when menhaden oil was included in the diet. A. H. CORNFIELD.

Effect of algae, dried lake weed, lucerne and ethoxyquin on yolk colour. G. Madiedo and M. L. Sunde (*Poultry Sci.*, 1964, **43**, 1056—1061).—The addition of 1% of algae + 0·001% of ethoxyquin (**I**) to a yellow maize basal diet increased egg yolk pigmentation, and a further addition of 0·01% of **I** increased pigmentation further. Safflower oil (5%) had no effect. Lucerne (5%) increased pigmentation, and when added with 0·01—0·10% of **I**, pigmentation was increased further. 0·002% of β-apo-8′-carotenal was as effective as lucerne (5%) + **I** (0·01%). Algae (1%) + **I** (0·1%) was more effective than lucerne (5%) without **I**. Lake weed (5%) was not as effective as the other materials tested. High levels (25,000—125,000 i.u. per kg. of diet) of vitamin A reduced yolk colour in proportion to the amount supplied during the 15th to 25th week after supplementation started. A. H. CORNFIELD.

Influence of calcium in modern laying rations on shell quality and interior quality of eggs. C. V. Reddy (*Dissert. Abstr.*, 1964, **25**, 1540—1541).—Comparison of rations containing different proportions of Ca showed that max. shell quality and optimum egg production were obtained with dietary levels of 3·05—3·85% of Ca (N.R.C., 1960, recommended level was 2·25%). Ca levels >3·85% were detrimental to egg production and were associated with lowered feed efficiency. Serum-Ca levels increased with dietary Ca but blood-P and -protein were not significantly affected. A. G. POLLARD.

Transference of strontium-89 and calcium-45 from egg shell to egg contents and embryo. F. R. Mraz and P. E. Waibel (*Poultry Sci.*, 1964, **43**, 1065—1066).—Newly-laid eggs were dipped in solutions containing labelled Ca, Sr and Ba and then incubated for 20 days. At the end of this period appreciable amounts of the radionuclides were found in the shell, but no measurable quantities were found in the contents of the egg or the embryo. Hens were given intramuscular injections with labelled Ca and Sr 5—15 h. after oviposition and the eggs laid the following day were incubated for 20 days. Appreciable amounts of the radionuclides were found in the shell and small amounts in the embryo, the amounts decreasing with time of dosing after oviposition. A. H. CORNFIELD.

Effect of dietary cholesterol on serum and egg cholesterol levels over a period of time. H. M. Edwards, jun., and V. Jones (*Poultry Sci.*, 1964, **43**, 877—879).—Addition of 1% of cholesterol to the hen's diet increased serum- and egg-cholesterol levels 10 days after supplementation commenced, but the levels usually returned to normal after another 10 days. A. H. CORNFIELD.

Comparison between chemical determination of xanthophylls and yolk pigmentation scores for xanthophyll supplements. G. Madiedo, E. F. Richter and M. L. Sunde (*Poultry Sci.*, 1964, **43**, 990—994).—The xanthophyll levels of lucerne, yellow maize, algae, lake weed and marigold petals as determined by chemical methods were well correlated with the yolk colour of eggs from hens receiving the supplements. A. H. CORNFIELD.

Residues in eggs and tissues of chickens on rations containing low levels of DDT. B. J. Liska, B. E. Langlois, G. C. Mostert and W. J. Stadelman (*Poultry Sci.*, 1964, **43**, 982—984).—Feeding broilers a ration containing DDT (0·1 p.p.m.) for 4 weeks or 1 p.p.m. for 1 week resulted in detectable DDT residues in the skin, meat and particularly in the fat. DDT (0·1 p.p.m.) in the diet of laying hens for 1 month resulted in negligible levels of DDT in body tissues and egg yolks, but 0·5 p.p.m. in the diet resulted in significant levels of DDT in the fat, skin and egg yolk. A. H. CORNFIELD.

Serum glutamic-oxaloacetic transaminase levels, muscular dystrophy and haematological measurements in chicks and poults as influenced by vitamin E, selenium and methionine. E. D. Walter and L. S. Jensen (*Poultry Sci.*, 1964, **43**, 919—926).—Addition of Se (0.0001 g./kg.) and vitamin E (2·5 i.u. per kg. of feed) to a poult diet which was deficient in both had an additive effect in the prevention of muscular dystrophy. Serum levels of glutamic-oxaloacetic transaminase (GOT) were closely related to incidence of muscular dystrophy. With chicks, although rations deficient in Se and vitamin E resulted in slight increases in serum GOT activity, substantial increases were obtained only when the diet was made dystrophy-producing by lowering its methionine content. The anaemia that developed in turkey poults fed a diet deficient in vitamin E and Se was accentuated when additional methionine was supplied. Addition of 0·00005 g. of Se per kg. of diet corrected this condition. The Se requirement for growth increased with the methionine level of the diet. A. H. CORNFIELD.

Effect of dimetridazole on growth and prevention of histomoniasis in poultry. W. C. McGuire, M. W. Moeller and N. F. Morehouse (*Poultry Sci.*, 1964, **43**, 864—871).—Max. improvement in growth rate and feed efficiency of turkeys over 3—24 weeks occurred when 0·01—0·10% dimetridazole was added to the diets. With chickens in growth tests of 4—8 weeks' duration the optimum dietary level of the drug was 0·0125 to 0·0500%. All levels of the drug were effective in reducing histomoniasis. A. H. CORNFIELD.

Relative activity of drugs against experimental histomoniasis in turkeys. J. K. McGregor, A. E. Ferguson, M. C. Connell and W. D. Morrison (*Poultry Sci.*, 1964, **43**, 1026—1030).—Carbo-O-Sep (0·0375% in the feed) and Emtryl (0·0312%) were effective antihistomonal agents in two battery experiments. Enheptin A (0·05%) was superior to Emtryl in the feed, but Emtryl (0·03%) in the drinking water was more effective than Enheptin A or Emtryl in the feed. Furazolidone (0·011%) and Histotat (0·0187%) were less effective than either Carb-O-Sep or Emtryl. A. H. CORNFIELD.

Animal feed supplements. Società Farmaceutici Italia (B.P. 938,916, 19.3.62. It., 22.3.61).—The supplement consists of an alkaline hydrolysate of mycelium of *Nocardia rugosa*. F. R. BASFORD.

Animal feeds. Nopco Chem. Co. (B.P. 939,017, 9.5.62. U.S., 9.5.61).—The growth response and feed efficiency of animals is increased by introducing into animal feed a mixture of a first material A (prepared by fermenting wet wheat bran under aerobic conditions, using *Aspergillus oryzae*) and a second material B (prepared by fermenting wet wheat bran under aerobic conditions, using *Bacillus subtilis*), in a wt. ratio of 1 : 3 to 3 : 1. F. R. BASFORD.

Animal feeding stuffs. Corn Products Co. (Inventor : R. B. Dawson) (B.P. 938,921, 6.8.59).—There is claimed a solid shaped animal feeding stuff (lick) comprising a homogeneous mixture of glucose or other crystallisable sugar, chalk (1—15 wt.-%), and optionally other nutrients and/or prophylactic or therapeutic substances. The chalk should be of particle size 1—25μ. F. R. BASFORD.

2.—FOODS

Carbohydrate Materials

Cereals, flours, starches, baking

Irradiation of rice bran with gamma-rays. H. Díaz Blasco, N. Moundiroff and J. G. Gómez Artero (*Rev. Argent. Grasas Aceites*, 1963, **5**, 57—60).—An attempt has been made to destroy the lipolytic enzyme in rice bran by irradiation with γ-rays (2—12 × 10^6 rads). Immediately after the irradiation the activity, as measured by the acidity, was greater than that of the control, but on storage (200 h.), the rate of development of acidity was slightly reduced. (21 references.) L. A. O'NEILL.

Physicochemical properties of rice in south-east Asia. B. O. Juliano, G. B. Cagampang, Lourdes J. Cruz and Remedios G. Santiago (*Cereal Chem.*, 1964, **41**, 275—286).—Tests developed in the U.S.A. for the evaluation and selection of rice for specific cooking characteristics (Beachell and Stansel, *Intern. Rice Comm. Newsletter*, 1963, spec. issue, 25—40) were applied to 55 milled samples of rough rice from five countries of S.E. Asia. Dimensions and hull %

of the rough rice, 100 grain wt. and protein content of the brown rice and protein and amylose contents, gelatinisation temp., alkali digestion values and pasting characteristics (Amylograph) are reported for each sample and correlations between them are summarised. Correlations between the results obtained and the cooking and eating qualities preferred in the respective countries are discussed. (28 references.) E. C. APLING.

Fat acidity of sound maize by the rapid method. A. Joffe and J. G. C. Small (*Cereal Chem.*, 1964, **41**, 230—242).—Fat acidity was determined by the official rapid method (Official Methods of Analysis, Ass. off. agric. Chem. Wash., 9th edn., 1960) on 100 g. sub-samples of 4 × 20 kg. lots of sound maize, stored individually in air-tight bottles at 21°, selected at random at intervals over 23 days and milled under controlled conditions. Considerable variations in fat acidity were found; in two samples these are ascribed to real fluctuations in the fatty acid levels in the maize during storage, but in the others heterogeneity of the samples was to great for clear interpretation. The variations found did not follow any significant diurnal or periodic cycle, but were sufficient to detract from the use of fat acidity determinations for the prediction of maize storage behaviour or for the quality assessment of stored maize. (23 references.) E. C. APLING.

Comparative swelling characteristics of polished and unpolished wheat and rice during cooking. S. N. Raghavendra Rao and H. S. R. Desikachar (*Cereal Chem.*, 1964, **41**, 316—319).—Measurements of the relative swelling and expansion ratios of polished and unpolished rice (medium grained variety, *Bangara sanna*) and wheat (a hard bread wheat, Punjab variety, and a *Jave* wheat, *Triticum dicoccum*) during cooking are reported. Swelling and expansion was much lower for wheat than for rice and lower for unpolished than for polished samples. Cooking for 45 min. was insufficient to achieve a soft product from wheat in which the outer bran was intact. E. C. APLING.

Fertiliser studies on some wheat varieties. J. S. Schlesinger (*Cereal Sci. Today*, 1964, **8**, 200—202).—Flour quality indices are related to wheat cultural practice. For the eight varieties, adequate N, available to the plants during the spring growing period, with normal moisture, will result in higher quality wheat as measured by % protein, sedimentation, Farinograph peaks and tolerances, loaf vol. and bake quality score. Coupled with proper fertilisation, this will bring max. yields. Definite varietal variations are apparent from a study of the milling data, regardless of fertiliser treatment, in % flour extraction, maltose and ash content. The significant negative correlation of −0·755 between maltose and bake quality score indicates the possibility that starch damage, as suggested by the natural maltose content, may affect baking quality. I. DICKINSON.

Chemical investigation of wheat. V. Some chromatographic investigations of wheat lipids. M. L. J. Mihailović, G. A. Garton, M. Antić and D. Hadžijev (*Bull. Soc. chim. Beograd*, 1963, **28**, 179—197).—Methods were developed for the extraction of the free and bound lipids of whole wheat flour and gluten. These were fractionated on a silicic acid column, qual. separated by thin-layer chromatography and the methyl esters prepared and separated by gas–liquid chromatography. The relationship between lipid content and baking quality is discussed. (In English.) S. A. BROOKS.

Determination of wheat protein. H. Hecht (*Getreide u. Mehl*, 1964, **14**, 80—84).—The construction and use of the Pro-Meter apparatus for the rapid determination of wheat protein, based on the principle of dye-binding, is critically evaluated. Under appropriate conditions of use, the apparatus gives results agreeing well with those of the Kjeldahl and formol procedures, but errors and variability vary with ripeness and moisture content of the grain. (25 references.) E. C. APLING.

Phospholipids, glycolipids and sterols of wheat endosperm. M. E. McKillican (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 554—557).—The phospholipid–glycolipid mixtures from the endosperm of three varieties of wheat were separated by thin-layer silicic-acid chromatography and column chromatography. A characteristic thin-layer chromatogram showing 20 components is shown. The components were identified and one of them, an esterified sterol glycoside, has not hitherto been reported in wheat lipids. Gas–liquid chromatography was used to determine the fatty acid and sterol compositions of the phospholipid–glycolipid components. Differences were found between varieties and between components. (23 references.) W. E. ALLSEBROOK.

Glutathione in wheat and wheat flour. T. Kuninori and H. Matsumoto (*Cereal Chem.*, 1964, **41**, 252—259).—Methods are described for the determination of glutathione (GSH) and 'total glutathione' (GSH + oxidised glutathione, GSSG) by separation from flour extract by gel filtration on Sephadex. Glutathione was determined in column fractions by reaction with alloxan and measurement of E_{305} mμ (cf. Kay and Murfitt, *Biochem. J.*, 1960, **74**, 303). 'Total glutathione' was determined similarly after individual prior reduction of fractions with 0·1M-$NaBH_4$. Apparent GSH in flour was 0·90 and 0·06 mg. per 100 g. at 1 and 14 days after milling respectively, and GSH + GSSG varied from 8 to 9·6 mg. per 100 g. (18 references.) E. C. APLING.

Mechanism of improver action in cake flours. I. Relation between flour specific surface and chlorine distribution. J. T. Wilson, D. H. Donelson and C. R. Sipes (*Cereal Chem.*, 1964, **41**, 260—274).—The Cl content of fractions from 4- or 5-stage air classification of three control (untreated) and Beta-Chlora or Cl-treated short extraction soft wheat flours was determined by wet ashing and potentiometric titration for total Cl and by direct titration for Cl^-. The specific surface of each flour and flour fraction was computed from particle size data obtained with the Coulter counter. For each treated flour the fines fraction had a Cl uptake three times that of the parent flour and five times that of the coarse, high-starch fraction. Only from 40 to 68% of the added Cl_2 was titratable, indicating the formation of addition or substitution products. The results suggest that uptake of Cl is a random process in which the probability of attack on a flour particle is a function of its surface area. E. C. APLING.

Evaluation of baking quality in several European countries. J. Paquet (*Getreide u. Mehl*, 1964, **14**, 73—78, 89—96).—Baking quality evaluations of ten French wheat types from the crop years 1958—60 by given specialist laboratories in France, Belgium, Holland and Germany, each using their normal test procedures, are reported and systematically compared. While some wheat types were classed similarly in all countries, others were very variously valued. Methods (in order of increasing complexity) considered suitable for use in wheat selection, aimed at eliminating wheat types with baking quality rates only average in all or poor in any one or more of the four countries are: Pelshenke test no.; specific sedimentation value; W value (Alveograph); bread vol. (Detmold test baking method); and bread vol. (Wageningen test baking method). E. C. APLING.

Upgrading amylose content of amylomaize starch by butanol complexing. R. A. Anderson and V. F. Pfeifer (*Stärke*, 1964, **16**, 209—211).—Commercial high-amylose maize starch has an amylose content of up to 70%. Engineering studies of a butanol-complexing method have resulted in a method for fractionating amylomaize into a fraction with >80% amylose content (films prepared from this had improved clarity and fold endurance properties) and an amylopectin fraction. Effects of cooking time, concn. of solids in the cooked starch slurry and pptn. time were investigated and a flowsheet worked out for the process. (11 references.) A. T. CARPENTER.

Acid hydrolysis of glycoside linkages. IX. Hydrolysis of amylose labelled at the reducing and non-reducing end. J. Holló, E. László, J. Szejtli and Gy. Zala (*Stärke*, 1964, **16**, 211—214).—Amylose labelled with ^{14}C at the reducing end was prepared from maltotriose, maltotetraose and a maltopentaose–maltohexaose mixture and labelled at the non-reducing end from inactive dextrins and ^{14}C-glucose-1-phosphate. The radioactivity of the liberated glucose, maltose, malto-tri, tetra-, penta- and hexaoses was measured during the course of hydrolysis with 0·1N-HCl at 100°. The glucose unit at the non-reducing end is split off up to six times more rapidly than that at the reducing end. (17 references.) A. T. CARPENTER.

Glucose reversion as related to the degree of saccharification of the starch hydrolysate. A. Sroczyński and M. Boruch (*Stärke*, 1964, **16**, 215—224).—The effect of time, temp., pH and concn. on di- and trisaccharide formation in pure glucose solutions was measured. Reversion rate is increased (i) in more conc. solutions (max. at 140° and pH 2 after 1·5 h. in 60% and after 2·5 h. in 40% solution); (ii) at low pH levels (min. at pH 5) and (iii) at higher temp. In starch hydrolysates with varying degrees of saccharification reversion is faster than in corresponding pure glucose solutions. Starch hydrolysis experiments using ^{14}C-glucose and chromatographic and radioactive measurements indicate formation of compounds of glucose with maltose and higher oligosaccharides. After reaching max. reversion, continued hydrolysis results in lowering the yield of reversion products and increasing the yield of degradation products. (27 references.) A. T. CARPENTER.

Purification of crude glucose syrup and dextrose juices with separators. H. Huster (*Stärke*, 1964, **16**, 225—229).—Flow schemes are given for batch and continuous flow converters for glucose installations involving separators for removal of protein–fat mixture from the starch milk. The construction and installation of the nozzle-type separator are described. Advantages of its use include elimination of filtration, use of filter aids and cleaning of the filter, economy in personnel, longer life of the activated C used (as it does not become contaminated with fats), recovery of the fat–protein sludge for use in fodder and adaptability of the separator. A. T. CARPENTER.

International standardisation of concepts and definitions in the starch field. I. G. Graefe (*Stärke*, 1964, **16**, 229—235).—Terms (38) and definitions are listed in French, English and German; they were proposed by the Technical Committee ISO/TC 93 on 'Starch including Derivatives and By-products' of the International Organisation for Standardisation as the first stage in setting up a standard vocabulary in this field. A. T. CARPENTER.

Dough rheology. I. Early history up to 1900. H. G. Muller (*Brot. u. Gebäck*, 1964, **18**, 117—121).—A historical review. (34 references.) E. C. APLING.

Dough-improving effect of some aliphatic hydrocarbons. I. Baking and physical dough studies. J. G. Ponte, jun., S. T. Titcomb and R. H. Cotton (*Cereal Chem.*, 1964, **41**, 203—215).—The effects of 47 org. compounds (including alcohols, esters, an ether, fatty acids, ketones and hydrocarbons) on Farinograph properties and bread characteristics when added to doughs at the level of 0·43 per 100 g. of flour are reported. Most of the aliphatic hydrocarbons tested increased Farinograph peak time and stability, and produced finer grain, smoother texture and brighter crumb colour in the bread; max. effect was found with heptane. Esters generally weakened the doughs and lower alcohols showed little effect, but the halogenated and aromatic hydrocarbons, ketones and fatty acids generally increased Farinogram dimensions but showed no corresponding improvement in bread properties. Heptane and hexane both stimulated dough gassing power; heptane improved the baking properties of crude gluten separated from the dough and also produced a finer and more uniform crumb structure in cakes. (12 references.) E. C. APLING.

Action of reducing agents on dough. P. Meredith and I. Hlynka (*Cereal Chem.*, 1964, **41**, 286—299).—The effects of a no. of reducing agents, in concn. up to 2 μmoles per g. of flour, on the properties of doughs mixed under N_2 were examined by Farinograph, Extensograph and gel protein analysis (Meredith and Bushuk, *ibid.*, 1962, **39**, 411). Gel protein analysis showed two types of reaction, possibly depending on the potential acidity of the compound: (i) rapid fall to zero gel followed by recovery and a slow secondary breakdown (L-cysteine hydrochloride, 2-mercaptoethylamine hydrochloride, 2-mercaptoethanol and glycol-dimercaptoacetate), or (ii) rapid fall followed by a very slow recovery (thiomalic acid, thioglycollic acid, glutathione and sulphite). Farinogram results suggest that the effect of O_2 is to re-form linkages in reduced doughs rather than to destroy –SH compounds, and that therefore dough weakening by –SH compounds results from splitting of S–S bonds rather than merely increased –SH mediated mobility of S–S bonds. Extensograms in all cases showed reduction in dough-resistance but little or no variation in total extension with increasing thiol addition. This suggests that the viscous properties of the dough are not decided by the SS–SH system, but that elastic properties are correlated with the integrity and immobility of S–S bonds in the gel protein (glutelin) structure. (31 references.)
E. C. APLING.

Volatile components of white bread prepared by a pre-ferment method. E. L. Wick, M. deFigueiredo and D. H. Wallace (*Cereal Chem.*, 1964, **41**, 300—315).—The results of examinations of concentrates of flavour distillates from bread by GLC, TLC of 2,4-dinitrophenylhydrazine deriv. and flash-exchange GLC of the separated deriv., are reported. Compounds rigorously identified were: ethanol, n-propanol, isobutanol, isopentyl alcohol, acetoin, furfural, AcOH and AcOEt. Tentative identifications were made of: propanal, 2-butenal, 2-ethylhexanal, acetone, biacetyl, 2-methylbutanol-1, formaldehyde, acetaldehyde, n-butanal, isobutanal, n-pentanal, 3-methylbutanal, 2-butanone, 2-pentanone and 2-hexanone. Approximately 17 other unidentified compounds were present in traces. The five alcohols, acetoin, furfural and AcOH were the major components. Addition of 1000 p.p.m. of L-proline to the dough improved bread aroma and increased the amount of odour concentrate obtained, but produced no detectable change in the composition of the volatile material. (25 references.)
E. C. APLING.

Flour lipids and their significance for baking quality. Jean Buré (*Brot. u. Gebäck*, 1964, **18**, 133—143).—An extensive literature review together with the results of comparative analytical and rheological studies with French flour. Topics covered are the content and composition of the lipid of flour, and the effects of flour lipids, glycerides, fatty acids, antioxidants and oxidising improvers on baking quality. (51 references.) E. C. APLING.

Laboratory studies of processing temperatures in continuous bread-making. S. Redfern, B. A. Brachfeld and J. A. Maselli (*Cereal Sci. Today*, 1964, **8**, 190—191).—The effects of variations in temp. of pre-ferments, dough temp. and proof temp. were investigated by use of two formulas, (*a*) no flour is added to the brew, (*b*) has some flour in the brew. In both cases it is shown that the brew temp. and the proof temp. have a significant effect on proof time, whereas dough temp. has no effect. If the brew temp. is changed from 83°F to 90°F in the non-flour brew, the proof time is reduced by 2·5 min., while the proof time is increased by 2·6 min. in the flour brew under the same conditions. The consistencies of the doughs at the higher mixing temp. were lower than those of the cooler doughs. No differences were detected in fineness or uniformity of crumb structure and all baked loaves were scored as being equal. I. DICKINSON.

Experiments on the development of a continuous system in heavy batters or thin doughs. I. Hlynka (*Cereal Chem.*, 1964, **41**, 243—251).—The development of a continuous phase was followed by measurements of the amount of dough (determined as wet gluten) wound up on a multi-pin stirrer rotated in the batter for 15 min. at 40 r.p.m. The rate of development of continuous phase was found to increase as the water content of the system was decreased, and to decrease with increasing additions of IO_3^-. Atm. O_2 and added salt showed a similar, but very much smaller, retarding effect. Additions of bisulphite appeared to increase the rate of development, but dough recovery was reduced due to a disruptive effect on the gluten. Additions of both IO_3^- and HSO_3^- at different stages of mixing showed that their effects were distinct and counteracting. The delaying effect of IO_3^- on dough development observed may explain why higher additions of oxidising improver are tolerated in baking methods employing high-speed dough development.
E. C. APLING.

Elastic modulus of bread crumb in linear compression in relation to staling. S. J. Cornford, D. W. E. Axford and G. A. H. Elton (*Cereal Chem.*, 1964, **41**, 216—229).—Changes in crumb firmness during the staling of bread made with and without compound fat, and stored at various temp. in the range from 20 to 90°F, were followed by measurement of crumb modulus, E, under defined conditions of strain and time: namely, compression to half the original thickness within 1 min. Experimental curves of E against time could be fitted by inverse exponential curves of the form: $(E_1 - E_t)/(E_1 - E_0 = \exp(-kt)$, in which the limiting modulus E_1 was characteristic of the particular batch of bread and the rate constant k decreased with increasing temp. Addition of compound fat to the bread formula reduced the value of E without significantly affecting k at any given temp. The results favour a physical explanation of the crumb-firming process which involves the development of a more ordered arrangement, such as in crystallisation, and are in general agreement with the theory of Avrami (*J. chem. Phys.*, 1939, **7**, 1103; 1940, **8**, 212; 1941, **9**, 177) relating to the growth of crystallites. (16 references.) E. C. APLING.

Translocation and equilibration of moisture in canned frozen bread. K. G. Weckel, R. Hawley and E. McCoy (*Food Technol.*, 1964, **18**, No. 9, 234—236).—The pH of bread at four different levels did not change significantly after frozen storage for 20 weeks. Equilibration of moisture (two levels) within the loaf occurred during the first week of storage. Moisture equilibrium is related directly to temp. and not to storage time. (13 references.) E. M. J.

Rôle of wheat pentosans in baking. II. Effect of added flour pentosans and other gums on gluten–starch loaves. R. W. Cawley (*J. Sci. Fd Agric.*, 1964, **15**, 834—838).—Loaves made from gluten and starch were inferior in vol. and texture to loaves made with the addition of flour solubles (**I**). The effect of **I** was destroyed by enzymes which degrade polysaccharides. Other viscous gums were tested for effectiveness in improving the vol. of gluten–starch loaves. Of these eight were effective showing an increase of 8 to 27% increase in vol., 10 materials were without effect. (15 references.)
E. M. J.

Analysis of gums. E. Becker (*Brot u. Gebäck*, 1964, **18**, 150—152).—The identification of locust bean gum, gum arabic, gum tragacanth and alginates by paper chromatography of the products of their hydrolysis with 5% H_2SO_4 is described. E. C. APLING.

Influence of destruction and formation of pigments during processing on the colour of macaroni goods. A. Menger (*Getreide u. Mehl*, 1964, **14**, 85—89).—The pigment classes involved in the development of colour in macaroni goods and methods for their determination are briefly reviewed, and the effects of various additives on their destruction and/or formation during processing are reported. Additions of monoglycerides increased and salt decreased both destruction of carotenoid pigments and development of browning. Lecithin and ascorbic acid both reduced destruction of carotenoids, but their effect was not additive. Lecithin increased the development of browning, particularly in the presence of ascorbic acid. Ascorbic acid also gave rise to the development of orange-red pigments extractable with org. solvents, and of a grey-brown non-extractable colour, and it is suggested that these arise by a melanoidin reaction between ascorbic acid and protein, and by a reaction with flavonoids respectively. (23 references.) E. C. APLING.

Sugars and confectionery

Effect of magnesium on clarification and phosphate content of raw sugars. P. Hidi (*Aust. J. appl. Sci.*, 1964, **15**, 35—40).—From conductiometric measurements made of $Mg^{2+} + HPO_4^{2-} \rightleftharpoons MgHPO_4$ at different temp. and in different amounts of dissolved sugar, Mg ions at concn. ~4·5 mmole in the clarified juice gave approx. four-fold increase in sol. PO_4^{3-} level. Presence of Mg in the pptd. phosphate inhibits the rate of transformation to the more stable form giving an apparent increase in solubility. (20 references.) C. A. P.

Fermentation of cane sugar molasses. A. Sánchez-Marroquin (*Rev. Soc., quím. Mex.*, 1964, **8**, 61—67).—The effect of different media for the production of citric acid from molasses with *A. niger* in submerged culture with shaking has been studied. An optimum medium is molasses (10% sugar) treated with K ferrocyanide, with addition of NH_4NO_3 (0·15), $ZnSO_4$ (0·0044), KH_2PO_4 (0·02) and cornsteep liquor (0·02%), and ethanol (3·5) or methanol (3%). Yields up to 68% were obtained at pH (initial) 6·5 to 7 and fermentation temp. 30 to 32°, with a suitable vegetative inoculum (1·5%). (18 references.) L. A. O'NEILL.

Storage studies of sorgo. O. H. Coleman and I. E. Stokes (*U.S. Dep. Agric. Tech. Bull.*, 1964, No. 1307, 54 pp.).—The effects of various storage procedures of sorgo (*Sorghum vulgare*) on the yield and quality of the syrup were investigated. The results are described and discussed. E.g., storing wet counterbalanced the normal benefits of storage on syrup quality. In general dextrose and levulose increased during the first 10 days of storage, sucrose decreased. No inversion took place in the syrup. Syrup made from unstripped was slightly darker than that made from stripped stalks. Good syrup was produced by processing the entire plant if stalks were stored relatively dry for ~6 days. O. M. WHITTON.

Kinetics of enzymic hydrolysis of maltose and a more precise method of determining maltase activity of mould preparations. D. B. Lifshits, B. Ts. Mikhailovskaya and E. Ya. Kalashnikov (*Mikrobiologiya*, 1964, **33**, 713—718).—A precise method of determining maltase (**I**) activity of enzyme prep. from moulds is described. Purified **I** extracts were prepared by pptg. aq. extracts of cultures with 55% Pr^iOH. Kinetics of action of **I** was followed at 40° for 1 h. using acetate buffered (pH 4·7) 1% solution of maltose (**II**) hydrate. The extent of hydrolysis was measured by determining iodometrically the increase in reducing substances. A single combined curve for extracts from four moulds was plotted showing the relation between **I** activity and quantity of **II** hydrolysed. The activity of **I** is directly proportional to the velocity constant of a monomol. reaction. P. W. B. HARRISON.

Culinary mixes. Procter & Gamble Ltd. (B.P. 937,833, 17.2.60. U.S., 18.2.59).—A dry culinary mix, from which a baking butter may be prepared by addition of liquid, consists of sugar (28—45), farinaceous material (30—48) and shortening (4—25 wt.-%) containing >50 wt.-% of fatty material which is solid at room temp. and 1·5—20 wt.-% of an ester derived from a monomeric polyhydric alcohol (glycerol) and a fatty acid of >20 and I val. <10 (e.g., rapeseed oil or herring oil fatty acid), the ester having <1 free old group (e.g., a monoglyceride). F. R. BASFORD.

Meringues or meringue-like products. Pillsbury Co. (B.P. 937,565, 12.9.61. U.S., 12.9.60).—A food product, especially a proteinaceous, meringue-like product of rigid, aerated structure is claimed and consists of a dispersion of an oil composition (preferably vegetable oil) in a protein dispersion comprising a continuous, external phase of aerated film-forming soya-bean protein composition in which the edible oil is dispersed. More specifically, the protein composition is foamed and heat-set and includes egg albumin, soya-protein and a whipping composition selected from Na hexametaphosphate, triacetin and Et_3 citrate. Such a product has sufficient rigidity to permit its use as a shell for the service of main or dessert dishes which may be hot or cold. F. R. BASFORD.

Air-treating composition. Unilever Ltd. (B.P. 938,039, 1.1.61. U.S., 4.2.60).—There is claimed an aq. gel (for use in air-treatment) comprising aq. medium, volatile air-treatment material (e.g., an alkanol, aldehyde, essential oil, etc.), gelling agent (e.g., alginate, pyroxylin-type nitrocellulose, gum, etc.), and surface-active agent (0·1—10%), preferably a sorbitan monolaurate–ethylene oxide condensate. F. R. BASFORD.

Fermentation and Alcoholic Beverages

Fractionation of must with a continuous press with the object of eliminating certain constituents. J. Carles and C. Laville (*C. R. Acad. Agric. Fr.*, 1964, **50**, 640—645).—Fractionation similar to that previously achieved with a non-continuous press (cf. *ibid.*, 1962, **48**, 773) can also be achieved with the Colin continuous press by collecting the must separately at three additional outlets placed along the length of the compression cylinder. A mixture of the fractions collected from the second and third outlets (representing ~55% of the total) will contain substantially less mineral matter (including Fe, Cu and Mn), N and free acids than a mixture of the fractions from the first and final (fourth) outlets. P. S. ARUP.

Decolorisation of red wines during biological decomposition of acids. U. Vetsch and H. Lüthi (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 93—98).—Experiments on the malolactic fermentation of model wines show the extent of decolorisation to be directly proportional to the content of citric acid and independent of the fermentation of the malic acid. A partial regeneration of the colour is observed immediately after the decolorising action. P. S. ARUP.

Solubility equilibria of calcium and potassium in wine. E. Peynaud, G. Guimberteau and J. Blouin (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 176—186).—The analytical results of 34 samples of Bordeaux wine (<2 years old and decanted from the tartrate deposit) are statistically examined. The pptn. of neutral Ca tartrate and KH tartrate is promoted by high concn. of EtOH, Ca^{2+} and K^+, and inhibited by high concn. of inorg. anions, most particularly by SO_4^{2-}. (15 references.) P. S. ARUP.

Isolation of flavour-producing components of wine by thin-layer chromatography. L. Jakob and O. Bachmann (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 187—192).—The oily matter extracted from the wine with n-pentane is chromatographed on SiO_2-gel with C_6H_6–MeOH (19 : 1) as solvent. The spots are revealed by examination under u.v. light and by spraying with several reagents. An example is given in which the cause of a defect in wine was traced by the taste and smell of an abnormal spot. P. S. ARUP.

Preservation and stability of wines. M. Leglise (*Rev. Ferment.*, 1964, **19**, 52—59).—A lecture giving practical directions as to the selection and application of suitable methods for ensuring the biological and physico-chemical stability in certain wines, and for the removal of excess of Fe or Cu. P. S. ARUP.

Determination of sorbitol in Spanish wine. E. Feduchy Mariño and T. Hidalgo Zaballos (*Bol. Inst. Invest. agron., Madrid*, 1963, **23**, 189—202).—A semi-quant. paper chromatographic method is described and the results obtained for 50 different wines (amounts varying from a trace to 7·5 mg. per 100 ml.) are reported. Wine was defecated with active C and de-ionised by passage through columns of Amberlite IR-120 and IR-400 (carbonate form). The eluate and washings were concentrated and treated with the calculated quantity of 0·1N-I and 0·1N-NaOH to convert glucose to gluconate, and then examined by ascending paper chromatography with n-propanol/ethyl acetate/water (7 : 1 : 2) as mobile phase. Spots were revealed by spraying with 0·01N-$NaIO_4$ (containing 0·5% of AcOH) followed, after 10 min., by 0·01N-benzidine. Four colour plates of typical chromatograms are reproduced. E. C. APLING.

Sensory detection of sorbic acid in wine. C. S. Ough (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 260—265).—The threshold concn. for the detection of the acid is found to be 170 ± 53·5 mg./l. The testing system employed and the method of evaluation of the results are described. Probable reasons for the discrepancies between results obtained by other observers are considered. (11 references.) P. S. ARUP.

Rapid and simple determination of sorbic acid in wine. F. Prillinger (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 193—196).—The method described is based on that of Schmidt (cf. *Anal. Abstr.*, 1962, **9**, 4486). P. S. ARUP.

Large-scale experiments with sorbic acid and diethyl pyrocarbonate [as wine preservatives]. H. Haushofer and A. Rethaller (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 239—250).—The performance of a pump (Orlita) for the dosing of wine with diethyl pyrocarbonate (**I**) was satisfactory. Sorbic acid at 80 or **I** at 50 mg./l. effectively prevented the secondary fermentation of wine kept in bottles under unfavourable conditions without affecting the organoleptic quality. The only case in which a geranium-like flavour was developed in wine dosed with sorbic acid occurred in a sample which had been treated with H_2O_2. (14 references.) P. S. ARUP.

Rapid method for the detection of diethyl pyrocarbonate in beverages, and natural occurrence of carbonic esters in fermentation products. F. Prillinger and H. Horwatitsch (*Mitt. Wein u. Obstbau, Wien*, 1964, **14A**, 251—257).—The decomposition product diethyl carbonate (**I**) representing ~5% of the originally added diethyl pyrocarbonate (**II**) (cf. *ibid.*, 29) can readily be extracted quant. from fruit beverages or wines into (pure) CS_2; as little as 10 mg./l. of originally added **II** can be detected in the CS_2 extract by a gas chromatographic procedure (described). The sensitivity can be increased tenfold by

concentrating the CS_2 extract by evaporation. No trace of **I** could be found in ~50 wines. P. S. ARUP.

Determination of very weak proteolytic activity in biological media. P. Pantel and C. Barthel (*Brauwissenschaft*, 1964, **17**, 246—250).—The method is based on viscometric measurements at 25° of the rate of ageing of a gelatin solution which has previously been acted on by the enzymic medium at 50°. The rate of increase of η is directly dependent on the enzymic activity of the medium. The time–η graph is compared with a blank graph (prepared from the medium in which the activity has been destroyed) and with graphs prepared from solutions of known activity. The method permits of the detection of 0·02 mg. of trypsin per l. of water. P. S. ARUP.

Rapid determination of moisture in barley with the Super-Beha apparatus. H. Hecht (*Brauwissenschaft*, 1964, **17**, 250—254).—The apparatus was found to be unsuitable for its purpose. P. S. ARUP.

Methodology of determination of air and oxygen in carbonated beverages. H. Kipphan, J. A. Herrmann, C. Karakas and J. Latuszek (*Brauwissenschaft*, 1964, **17**, 336—346).—In order to determine the headspace air, the bottle is opened under water, and the air is collected by immersed inverted funnel connected with a gas burette. The analysis of the air by the Zahm and Nagel apparatus is described. Two methods for the colorimetric determination of dissolved O_2 with the redox indicator indigo disulphonate are described, viz. that of Rothchild and Stone as practised by de Clerck, and the medical syringe method of Jenkinson and Compton. The second method (with minor improvements by the authors) is the more convenient; the accuracy is within $\pm$0·03 mg. of O_2 per l.; a semi-quant. modification of this method is described. The colorimetric methods are not applicable to turbid or dark-coloured beverages. (13 references.) P. S. ARUP.

Oxygen exchange and ITT-increase in beer. J. Mühlbauer and Willi Fischer (*Brauwissenschaft*, 1964, **17**, 329—335).—After beer bottled with known vol. of air in the headspaces had been shaken in the bottles for 100 h. at 1° or at 32°, it was found that 0·05 mg. of O_2 increased the ITT value by 15—20 sec. at 1°, and by 43—61 sec. at 32°. Beer with an initial ITT value of 375 sec. bottled at 20° showed (after storage for 90 days at room temp.) an increase in the ITT value of 165 sec., whilst the same beer bottled at 1° showed an increase of $\geqslant$50 sec., the amounts of O_2 consumed being 0·65 and 0·44 mg., respectively. (23 references.) P. S. ARUP.

Brewing of beer. Weigelwerk A.-G. (Inventor: W. Speilvogel) (B.P. 939,011, 13.1.61).—There is claimed a method for continuously mashing malt and treating the resulting wort, in which part of a cold mash of bruised malt and water produced in one or more cold-mashing appliances is heated to a temp. higher than that required for hot-mashing (e.g., to 100°) and is returned to and mixed with the remaining cold mash at intervals, so that the remixed mash undergoes hot-mashing, then the resulting hot mash is fed to an endless circulating filter belt or centrifuge (to separate the wort from the spent malt), and the wort is fed into a continuous-flow wort-boiling, and, if desired, hopping appliance from which the boiled wort is passed through another endless, circulating filter belt or centrifuge (and is thus purified). Apparatus is figured and claimed. F. R. BASFORD.

Reduction of haze formation in beer. A.-G. Für Brauerei-Industrie Glarus (B.P. 938,153, 20.10.61. Ger., 29.10.60 and 29.4.61).—A process for reducing haze in beer comprises adding finely powdered SiO_2 gel of surface area 200—400 m^2. per g., pore vol. 0·6—1·2 c.c. per g., and pore diameter 60—150 Å, then filtering. Preferably the water-sol. content of the gel is reduced (by washing) to $<1\%$ before use. F. R. BASFORD.

Fruits, Vegetables, etc.

Production of a standard comparator for the skin colour of mature cherries. N. Brearley, J. E. Breeze and R. M. Cuthbert (*Food Technol.*, 1964, **18**, No. 9, 231—233).—Spectrophotometric measurements were made of the skin colour of mature cherries (Bing and Lambert varieties). From the results obtained, standard colour comparators ($\frac{7}{8}$ in dia. plastic spheres) were made for use in the field, to enable growers to pick fruit at optimum maturity, and inspectors to assign a grade to the fruit. Specifically a comparator coated with an acrylic lacquer having a Munsell renotation of 7·5 R 2·02/9·1 was shown to be effective. E. M. J.

Nutritive value of Middle Eastern foodstuffs. I. Composition of fruits and vegetables grown in Lebanon. F. S. Simaan, J. W. Cowan and Z. I. Sabry (*J. Sci. Fd Agric.*, 1964, **15**, 799—805).—Results are presented on the proximate composition, Ca, P, Fe and vitamin A contents of locally available fruits (31) and vegetables (49) grown in Lebanon. Variations were observed among varieties of certain fruits and vegetables in their mineral content and vitamin A potency. The values obtained agreed in general with those obtained by workers in Iraq. (13 references.) E. M. J.

Evaluation of shell fruit, especially walnuts. A. F. Lindner and A. Bauger (*Z. LebensmittUntersuch.*, 1964, **125**, 271—280).—French, Austrian and Swiss official regulations for the testing of nuts for soundness are quoted. The unduly high % of unsound walnuts and chestnuts found in numerous batches purchased in Germany demonstrate the need for regulations similar to those enforced in France. The % of unsound nuts in 1-kg. or 5-kg. samples should not exceed 10—12%. (13 references.) P. S. ARUP.

Detection and estimation of aflatoxin in groundnuts and groundnut materials. III. Classification of aflatoxin B_1 levels. T. J. Coomes, P. C. Crowther, B. J. Francis and G. Shone (*Analyst*, 1964, **89**, 436—437).—Aflatoxin B levels as determined by paper and thin-layer chromatography are revised in terms of aflatoxin B_1 (**I**). Both methods involve determination of the smallest quality of **I** which will exhibit fluorescence under prescribed conditions: 0·1 μg. of **I** with paper chromatography, and 0·003 μg. with thin-layer chromatography. Using kieselgel-G layers, satisfactory results are obtained by diluting the extract until no fluorescence is observed. I. C.

Evaluation of dried foods. Determination of changes in cell and [tissue] structure during drying [of vegetables]. H. Sulser and H. Mohler (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 134—143).—The % of the total moisture that can safely be removed by drying at 60° without injury to the structure is determined by a standardised procedure. Separate portions of the samples in thin slices are dried to remove 10%, 20%, etc. of the total moisture, and the μg. of K dissolved after each portion has, after drying, been shaken with water are determined by flame photometry. The limits at which the curve for dissolved K begins to rise steeply vary from 15—65% for eight vegetables. Beyond these limits the desiccation must be continued by freeze-drying. Samples blanched by steam or hot water show decreases or approx. constant values for dissolved K on progressive desiccation. (15 references.) P. S. ARUP.

Non-alcoholic beverages

Rapid determination of nitrogen in apple-juice and cider. C. Macfarlane and J. F. Mears (*Analyst*, 1964, **89**, 428—431).—The sample is digested in H_2SO_4 with a Kjeldahl catalyst and carborundum. NaOH–$Na_2S_2O_3$ solution is added and the resulting solution is distilled into boric acid, the pH of which is noted on a pH-meter before distillation. N/140 H_2SO_4 is added to the boric acid solution after distillation until the original pH is restored. I. C.

Adulteration of orange juices and concentrates and its detection. J. Royo Iranzo (*Rev. Cienc. apl.*, 1964, **18**, 296—304).—Adulteration may take the form of adding substances normally present in the juice, but from other sources, foreign substances, or other fruit juices. The procedure to detect adulteration would be firstly the determination of the characteristics (*d*, acidity, ash, pulp, etc.) to see whether they were in the normal range, then reducing sugars, ascorbic acid and various indices, then elements (Na, K, P, Ca, Mg), and finally chromatographic examination of amine acids and analysis for carotenes. (28 references.) L. A. O'NEILL.

Composition and quality of carbonated beverages. S. N. Mitra, B. R. Roy, T. V. Mathew, A. K. Roy, A. Basak and P. C. Basu (*J. Instn Chem. India*, 1964, **36**, 205—207).—Samples (24) of different soft drinks were examined for total acidity, sweetening agent, mineral acids, dulcin, arsenic, colouring matter and bacterial count. Sugar, sugar plus saccharin or saccharin only were used for sweetening, ~50% contained non-permitted coal tar dyes and the total acidity varied from 13·6 to 41·6 ml. N/10 NaOH per 100 ml. Dulcin, arsenic and mineral acids were absent and the samples were bacteriologically very pure. E. C. DOLTON.

Analysis and composition of beverages containing quinine; decomposition of quinine during analysis. H. Hadorn and K. Zürcher (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 194—210).—Six samples contained per l., 25—57 mg. of quinine, 87—120 g. of sugar and 3·2—5·6 g. of citric acid. The u.v. spectra of quinine in different solvents were compared. The quinine extracted with Et_2O is pure, but the quinine extracted with $CHCl_3$ or CCl_4 contains decomposition products that can be detected by thin-layer chromatography. P. S. ARUP.

Dehydrated purees. General Foods Corp. (B.P. 938,381, 20.5.60. U.S., 20.5., 4.6., and 2.7.59).—There is claimed an instantly rehydratable dried puree permeated by a plurality of minute

ruptures created by stretching a film in the plastic state and comprising a random and discrete distribution of food solids in a continuous translucent phase of water-sol. solids (sugar). The dried puree may be fruit or tomato. Apparatus for producing the puree is figured and claimed. F. R. BASFORD.

Sterilising and disinfecting fruits. P. Serviere (B.P. 938,908, 24.11.61. Fr., 28.11.60 and 15.11.61).—Grapes and other pruinose fruits are sterilised and disinfected by immersing the fruit in aq. NH_4 lauryl sulphate or triethanolamine lauryl sulphate; rinsing in running potable water; and protecting the fruit against recontamination before pressing. F. R. BASFORD.

Tea, coffee and cocoa

Studies on aroma. I. Cacao aroma. P. Dietrich, E. Lederer, M. Winter and M. Stoll (*Helv. chim. Acta.*, 1964, **47**, 1581—1590).—A scheme is detailed for the separation of aromatic constituents from a light-petroleum extract of roasted Arriba cacao. In addition to substances previously identified, 29 new compounds were isolated including amines, pyrazines and several aliphatic acids and phenols. (In French.) H. S. R.

Milk, Dairy Products, Eggs

Influence of udder inflammation on some characteristics of milk. F. Kiermeier and K. Keis (*Z. LebensmittUntersuch.*, 1964, **125**, 96—101).—Observed decreases in souring capacity due to the disease are not large enough to affect the capacity of mixed milk from substantially sound herds. Coagulation times in the rennet test are found to be directly dependent on the severity of the infection. (26 references.) P. S. ARUP.

Detection of heated milk admixed with raw milk. E. F. McFarren L. A. Black and J. E. Campbell (*J. Milk Tech.*, 1964, **27**, 260—263).—Peroxidase (**I**), lipase, amylase, phosphatase (**II**) and vanillin (**III**) tests are discussed. All depend on the measurement of the enzymes except **III**. In this, heated milk cannot be detected unless the sample was heated >80° but this is equally true of **I**. **II** is used in the present series, samples being heated at 135°F for various periods 15 min. to 6 h. after prewarming; this is necessary for the sample to attain the correct temp. and the time (5 min.) is not included in the holding temp. C. V.

Relationship of fat content to other quality characteristics of milk. F. Kiermeier and E. Renner (*Z. LebensmittUntersuch.*, 1964, **125**, 185—188).—The substitution of silage for hay and roots in cow feeding decreases the well-established coeff. of correlation between the fat and the protein content, and nullifies the coeff. between the fat and carotene. The small negative correlation between the fat content and the I val. is not greatly affected by silage feeding. P. S. ARUP.

Improvement in the method of Hills and Thiel for determination of degree of oxidation of milk fats. J. Monnin and O. Schetty (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 182—186).—After shaking a solution of the sample in C_6H_6–MeOH (7 : 3) first with aq. $FeCl_3$ plus HCl and then with aq. NH_4SCN, the mixture is heated at 50° for 2 min. The extinction value of the coloration due to the oxidation of $FeCl_2$ to $FeCl_3$ affords a measure of the peroxide value of the fat. The method is accurate within 2—3%. P. S. ARUP.

Chain-length determination of polyphosphates. S. Odagiri and T. A. Nickerson (*J. Dairy Sci.*, 1964, **47**, 920—921).—An acidified polyphosphate solution (pH 2·5) is back titrated with 0·1N-NaOH and a titration curve plotted. The distance between the two points of inflection on the S-shaped curve, is equivalent to the weak acid function (f_w). The strong acid function (f_s) is obtained similarly by back titrating a phosphate solution, hydrolysed to the ortho form. The chain length ($\bar{n}$) is calculated from the equation: $\bar{n} = 2(\text{Equivalents of } f_s)/\text{Equivalents of } f_w$. (10 references.) M. O'LEARY.

Keeping quality of creamed Cottage cheese. T. Kristoffersen and B. K. Chakraborty (*J. Dairy Sci.*, 1964, **47**, 931—936).—The keeping quality of creamed Cottage cheese at 42°F was significantly improved by (*a*) heating the skim milk at 145°F for 30 min. and cooking the curd at 145°F, (*b*) addition of Aureomycin to the wash water, and (*c*) addition of either sorbic acid, K sorbate, lactic acid or H_3PO_4 to the cream dressing. Addition of nisin, citric acid and flavour-producing cultures to the dressing did not improve the keeping quality of the cheese. (15 references.) M. O'LEARY.

Sunlight flavour in milk. II. Complex formation between milk proteins and riboflavin. L. W. Aurand, J. A. Singleton and G. Matrone (*J. Dairy Sci.*, 1964, **47**, 827—830).—Spectral adsorption studies indicated that milk proteins form a loosely bound complex with riboflavin. Formation of the complex is dependent on the availability of O_2 and the presence of tryptophan in the protein. (13 references.) M. O'LEARY.

Changes in composition of Cheddar curd during manufacture as a guide to cheese making by direct acidification. W. M. Breene, W. V. Price and C. A. Ernstrom (*J. Dairy Sci.*, 1964, **47**, 840—848).—A study of conventional Cheddar cheese-making procedure indicated that for the successful production of Cheddar cheese by unconventional methods the curd at hooping should have a pH of 5·3 to 5·4 and contain 41 to 43% moisture and 4 to 6 g. lactose per 100 g. buffering constituents (total solids less fat and lactose). Further studies indicated that Cheddar cheese of acceptable quality could be made by a mechanised process in which acidification during curd-making is achieved by the direct addition of lactic acid to the milk. Normal acidity development during pressing would be accomplished by the addition of an appropriate amount of starter. (28 references.) M. O'LEARY.

***Ad libitum* intake and digestibility of several lucerne hays by cattle and sheep.** D. T. Buchman and R. W. Hemken (*J. Dairy Sci.*, 1964, **47**, 861—864).—Feeding trials indicated that the relative rates of voluntary intake of lucerne hay per 100 lb. body wt. were 3·28, 3·02, 2·91 and 2·23 lb. for dairy calves, dairy cows, wether sheep and yearling dairy heifers respectively. The consumption per 100 kg. of metabolic body wt. was 23·7, 31·4, 17·6 and 23·1 lb. respectively. Hay cut at the bud stage of maturity was more digestible than that cut at the bloom stage. Chopped hay was more digestible than ground and pelleted hay. (14 references.) M. O'LEARY.

Bacteriological changes during the manufacture of non-fat dry milk. M. E. McDivitt, P. P. Huppler and A. M. Swanson (*J. Dairy Sci.*, 1964, **47**, 936—941).—Bacterial examination of samples obtained during different stages of the manufacture of non-fat dry milk at two processing plants indicated that low counts on plate count agar may be misleading as an indication of the safety of the final product. Growth of coagulase-positive staphylococci was demonstrated during storage of conc. milk prior to preheating and drying. The formation of heat-stable enterotoxin at that stage would not be revealed by plate counts of the finished powder. (18 references.) M. O'LEARY.

Sterols in egg-dough products and their determination. II. Determination of sterols after treatment of samples with acid. L. Acker and H. Greve (*Z. LebensmittUntersuch.*, 1964, **125**, 179—184).—The method of treatment with 4N-HCl followed by extraction with Et_2O gives higher results for sterols (by 7—8 mg. per 100 g. of dry matter) than does the method of direct extraction with the EtOH–benzene solvent. Comparative analyses of wheat and of egg-dough products and determinations of the residual sterols detected by applying the method of acid hydrolysis to samples previously extracted with EtOH–C_6H_6 show the difference to be due to the inclusion of wheat sterols by the acid hydrolysis method. Extraction with EtOH–C_6H_6 gives a correct measure of the egg-sterols. P. S. ARUP.

Effect of oil placement in maintaining the albumin condition of the egg. T. L. Goodwin (*Poultry Sci.*, 1964, **43**, 964—966).—Eggs oiled on the small end and stored for 2 weeks or 6 months had higher average Haugh units than had non-oiled eggs or eggs oiled on the large end. There were significant differences due to strain in the effect of oiling position on keeping quality. A. H. CORNFIELD.

Cheese making. Cow & Gate Ltd., G. M. Robertson and G. K. Charles (B.P. 937,441, 13.6.60).—A method of maturing one or more blocks of natural cheese comprises wrapping the block(s) completely in flexible sheet material which is impervious to air and moisture (e.g., metal foil or thin, limp plastic film); and compressing the block(s) between rigid panels which are brought together to effect maturing under pressure. Preferably an intermediate wrapping of relatively stiff but still flexible material (thin cardboard) is interposed between the wrapped block(s) and the container. F. R. BASFORD.

Edible Oils and Fats

Determination of lactic acid in shortening containing lactylated glycerides, by liquid–liquid partition chromatography. R. J. Buswell (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 457—459).—The total or water-insol, combined lactic acid present in prepared shortening mixes as glyceryl lactopalmitate is determined by a liquid–liquid chromatographic method. About 0·4 to 0·44 g. of the shortening is extracted with methylene chloride, which is then washed, evaporated and the residue saponified with alcoholic KOH, and then acidified with H_2SO_4. The resultant material is dispersed on silicic acid, slurried with $CHCl_3$ and transferred to a silicic acid column, where the fatty

acids are removed with $CHCl_3$, and the lactic acid by elution with 15% n-butanol solution which is titrated with methanolic KOH. G. R. WHALLEY.

Oxygen bomb method for determining shortening stabilities. J. E. Bennet and M. J. Byer (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 505—507).—An O_2 bomb method is described for the determination of shortening stability of various vegetable shortening mixtures. A 10-g. sample is employed, which is treated with O_2 at 110 p.s.i. at 135 ± 0·1° for about 1 h. 45 min. The method has twice the accuracy of the active oxygen method. G. R. WHALLEY.

Effects of ionising radiations on fats. II. Accumulation of peroxides and other chemical changes. J. R. Chipault and G. R. Mizuno (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 468—473).—The increase in peroxide, carbonyl and reducing substances content of methyl myristate and linoleate after irradiation with 2, 5 and 8 × 10^{-6} rads was determined. The saturated ester, in vac., gives very small increases, and in the presence of O_2 all are present, and the incorporation of typical fat antioxidants have no effect. Irradiation of methyl linoleate under vac. conditions causes the destruction of preformed hydroperoxides, and in the presence of O_2 about ½ of the peroxides formed arise from a reaction between free radicals and O_2; the remainder are formed via a chain reaction. The increase in peroxide content in the presence of O_2 proceeds at a rate which increases with the increase in radiation dosage. Antioxidants exert a small retarding effect on peroxide formation. (15 references.) G. R. WHALLEY.

Fatty acid components of fried foods and fats used for frying. L. T. Kilgore and W. D. Luker (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 496—500).—When lard and cottonseed oil are used for frying chicken and potatoes for 5- and 10-h. periods at 185 ± 5°, a GLC analysis of the resultant fatty methyl esters shows a loss of 8% of linoleic acid originally present in the cottonseed oil. There was no change in the fatty acid composition of lard after chicken frying, but there is a decrease of 5·5% of linoleic acid when potatoes are fried for 10-h. periods. (22 references.) G. R. WHALLEY.

Factors affecting the rate of deterioration in the frying quality of fats. II. Type of heater and method of heating. S. P. Rock and H. Roth (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 531—533).—The deterioration of partially hydrogenated lard and maize oil in electric, gas and indirectly heated fryers, for periods up to 48 h., was investigated. The fat deterioration in the fryers was studied by the changes in acid value and η (at 212°F) which occurred. Experiments, with fat thermostatically controlled at 375°F, showed that the deterioration varied inversely with the temp. of the heating elements used. This is contrary to what might be expected. It can be explained by rapid convection occurring when very hot elements are used. η changes indicate that well-circulated fat heated indirectly deteriorates more rapidly than fat heated directly. (11 references.) W. E. ALLSEBROOK.

Silicic acid–silver nitrate chromatography as an enrichment technique in fatty acid analysis. M. K. Bhatty and B. M. Craig (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 508—510).—By using column chromatography with silicic acid/$AgNO_3$ and various solvent systems, the enrichment of trace quantities of component fatty acids in various oils is achieved. The technique shows that odd-numbered fatty acids are found in both the saturated and monoene fractions of olive, rapeseed and sunflower seed oils. The method together with oxidation of the unsaturated components and followed by GLC of the fragments is of use in establishing the presence of minor quantities of positional isomers. G. R. WHALLEY.

Spectrophotometric determination of small amounts of 1-monoglycerides in fats. C. Szonyi and K. Sparrow (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 535—537).—Small amounts of 1-monoglycerides in fats were determined using a periodic oxidation method. A glycol aldehyde fatty acid ester was formed and the 2,4-dinitrophenylhydrazone deriv. was analysed spectrophotometrically. (12 references.) W. E. ALLSEBROOK.

Degradation of monocarbonyls from autoxidising lipids. D. A. Lillard and E. A. Day (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 549—552).—The autoxidation of four carbonyl compounds, n-nonanal, n-non-2-enal, n-hepta-2,4-dienal and n-oct-1-en-3-one was studied. The unsaturated aldehydes, non-2-enal and hepta-2,4-dienal, oxidised faster than methyl linoleate or methyl linolenate and produced shorter chain mono- and dicarbonyls as oxidative degradation products. The identification of these compounds helps to explain the monocarbonyl compounds formed from the autoxidation of methyl linoleate and methyl linolenate. (30 references.) W. E. ALLSEBROOK.

Selective use of various chromatographic techniques for the study of the triglyceride structure of natural fats. M. J. McCarthy and A. Kuksis (*J. Amer. Oil Chem. Soc.*, **41**, 527—530).—The separation of a model vegetable oil assumed to be formed with palmitic, stearic, oleic, linoleic and linolenic acids is discussed. Gas–liquid chromatography separates the triglycerides according to their mol. wt. or carbon no. (*C*), $AgNO_3$–thin-layer chromatography separates the triglycerides according to their unsaturation or I no. (*I*) and partition chromatography separates the triglycerides according to their polarity *P* (where *P* is *C* − *I*). An integrated system of triglyceride analysis which uses all three methods of chromatographic analysis for the subsequential fractionation and isolation of triglyceride groups has been developed and is explained. It involves partition chromatography followed by gas chromatography and then separation with thin-layer chromatography. (16 references.) W. E. ALLSEBROOK.

Size reduction of emulsion globules by steam injection. V. A. Jones, C. W. Hall and G. M. Trout (*Food Technol.*, 1964, **18**, No. 9, 217—220).—Homogenisation of maize oil–water emulsions was studied by recirculating the emulsion through a specially designed injector. Steam was introduced and the homogenising effect (**I**) was measured by the optical transmittance of diluted emulsion. **I** increased with steam temp., but was little affected by emulsion pressure and flow rate. A size reduction of emulsion globules was observed in processes where steam and product come into direct contact. (16 references.) E. M. J.

Specific distribution of unsaturated fatty acids in the triglycerides of plants. F. H. Mattison and R. A. Volpenhein (*J. Lipid Res.*, 1963, **4**, 392—396).—The distribution between primary and secondary positions of the triglyceride of 28 species was determined and previous results were confirmed that palmitic (**I**) and stearic (**II**) acids were found to be esterified predominantly in the primary positions and it was also demonstrated that fatty acids with a C-chain >18 were similarly located. Study of the data shows that oleic, linoleic and linolenic acids have a common pattern of distribution, each of these being randomly distributed among the positions of the triglyceride mol. that are not occupied by **I** or **II** acids or by acids with a C-chain >18. Mechanisms are discussed. (15 references.) C. V.

Electron spin resonance spectra of free radicals in u.v.-irradiated fats and fatty acids. C. U. Deffner, H. Lück and R. Kohn (*Z. LebensmittUntersuch.*, 1964, **125**, 281—288).—The spectra obtained by the previously described technique (cf. *ibid.*, 1963, **123**, 200) from these products after irradiation at −196° differ in conformation from those similarly obtained after γ-irradiation, and are, by comparison, much less stable at higher temp. (−78·5 to 0°). Peroxide radicals can be detected in the spectra after warming the samples up to −110° in the presence of air. Whilst the spectrum of u.v.-irradiated elaidic acid resembles those of fats, the spectra of sorbic and β-elaeostearic acids are of a different pattern. (22 references.) P. S. ARUP.

Composition of cacao butter. H. Woidich, H. Gnauer, O. Riedl and E. Galinovsky (*Z. LebensmittUntersuch.*, 1964, **125**, 91—96).—In continuation of previous work (cf. *ibid.*, 1960, **112**, 184), the fatty acid composition of 77 samples was determined by gas chromatography of the Me esters. Accepted values for the major constituents were confirmed. Special points of interest were: the presence of up to 4·2% of linolic acid, up to 1·3% of arachidic acid, and up to 0·2% of myristic and linolenic acid in genuine samples; traces of several hitherto unsuspected acids were also found. P. S. ARUP.

2-Thiobarbituric acid method for the measurement of oxidative rancidity. T. C. Yu and R. O. Sinnhuber (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 540—543).—The validity of the 2-thiobarbituric acid (**I**) procedure for the measurement of oxidative rancidity has been questioned. The effect of acids, oxidising agents and hydroperoxides on **I** and the reaction of **I** with malonaldehyde was studied. Absorption spectra/wavelength graphs are shown. The absorption tests and column chromatography experiments indicate that the oxidative rancidity test is affected by traces of impurities in the reagents (AcOH and isopentyl alcohol) used. W. E. ALLSEBROOK.

Thin-layer chromatography of antioxidants. I. Gallic acid and its alkyl esters. T. Salo and K. Salminen (*Z. LebensmittUntersuch.*, 1964, **125**, 167—170).—The separation is carried out within 20 min. on 10%-acetylated cellulose with Shell Sol A–PrOH, AcOH–formic acid as developing solvent, the spots being revealed by spraying with 1% $FeCl_3$ in EtOH. R_F values are given for the acid and six of its esters. P. S. ARUP.

Powdered fat compositions. Pillsbury Co. (B.P. 937,564, 12.9.61. U.S., 12.9.60).—Claim is made for a powdered fat composition comprising particles of edible fat, and a hydro-stable, hydrophilic, encapsulating film entirely encasing the fat particles and consisting of a dried film-forming composition comprising a hydrophilic

film-former (egg white, soya protein) and an amount of water not in excess of that required for the hydration of the film-former. E. Enos Jones.

Treatment and preservation of crude animal fats and compositions containing them. A. Mayer (B.P. 937,942, 17.5.62. Ger., 14.7.61). —Animal waste fat, especially beef tallow (intestinal fat) from slaughter houses, is rendered fit for incorporation into animal feeding stuffs by pulverising (after hanging and freezing), admixing with iodised NaCl (20—80 wt.-%), then incorporating ~4000 p.p.m. of lactic acid (as preservative). A typical feeding stuff (claimed) consists of a mixture of treated beef tallow 10, dried and ground meat offal 10, linseed oil meal 10, dried and ground lucerne 10, wheat malt germ 10, skim milk powder 5, sweetened whey powder 5, minerals including trace elements 5, dried yeast 2, and $CaCO_3$ 3%. F. R. Basford.

Meat and Poultry

Beef quality. IV. Comparison of the eating quality of meat from bulls and steers. K. Bryce Jones, J. M. Harries, J. Robertson and J. M. Akers (*J. Sci. Fd Agric.*, 1964, **15**, 790—799).—Twin male calves raised on an intensive system of management were used. Of rib joints and silversides from 10 pairs, the eating quality of steer meat was the more tender. It had significantly more flavour, was significantly more juicy and was significantly more cooked at the same internal temp. than meat from bull twins treated as nearly as possible identically and tasted concurrently. The difference was not equally apparent in all types of joint, or whether tasted hot or cold. The bull meat contained less fat than that of the steers. E. M. J.

Faecal streptococci as food poisoners. H. Lüönd and H. Gasser (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 144—149).—An account is given of a temporary outbreak of gastro-intestinal disturbances following the consumption of meat pies contaminated with enterococci, probably *S. faecium* or *S. durans*. (11 references.) P. S. Arup.

Nitrifying bacteria in pickled meats. R. Buttiaux (*Rev. Ferment*, 1963, **18**, 191—195).—The bacteria which satisfy the requirements for reducing NO_3^- to NO_2^- in a 'pickling' medium belong to the group *Vibrio*. Conditions for culturing this organism and its use in ham pickling are described. (18 references.) J. V. Russo.

Meat emulsions. Griffith Laboratories Ltd. (B.P. 938,711, 1.3.61. U.S., 4.3.60).—A meat emulsion which finally contains free edible acid is produced by adding to a meat emulsion, immediately prior to or during the emulsification process, glucono-δ-lactone (≯1 pt. per 3200 pt. of meat), which slowly hydrolyses with water present to give the required acidity. F. R. Basford.

Water-insoluble defibrillated meat proteins. Armour & Co. (B.P. 937,446, 5.4.61. U.S., 5.4.60).—Comminuted meat is extracted with water and then defibrillated with alkali at pH 8—14 (preferably with aq. NH_3, at ~2—150°), to provide a water-insol. meat protein, substantially colourless and flavourless, suitable for use in the fortification of non-meat products. F. R. Basford.

Meat-like products. Pillsbury Co. (B.P. 937,566, 12.9.61. U.S., 12.9.60).—A highly nutritious meat product, which resembles meat-base products such as luncheon loaves and spreads, consists of an edible, lipophilic discontinuous internal phase (e.g., edible oil) dispersed within and entrapped by cells of an edible, continuous, cellular external phase (of hydrated protein, e.g., egg white which is heat-set). F. R. Basford.

Fish

[A] **Use of γ-radiation for the preservation of scallop meat.** [B] **γ-Irradiation as a means of extending the storage life of haddock fillets.** H. E. Power, D. I. Fraser, W. Neal, W. J. Dyer, C. H. Castell and [A] H. C. Freeman and D. R. Idler (*J. Fish. Res. Bd Can.*, 1964, **21**, 813—826, 827—835).—[A] The storage life at 0° of iced irradiated scallops (*Placopecten magellanicus* Gmelin) is increased by a dose of 75,000 rads of γ-radiation; raw scallops remained acceptable for 28 days, scallops assessed after cooking remained unchanged for 35 days and were acceptable until 43 days of storage. Doses of γ-radiation >75,000 rads caused undesirable changes in texture and odour and loss of quality. Neither irradiated nor fresh iced scallops rated as high organoleptically as frozen control scallops which had been rapidly frozen in a pre-rigor condition and immediately stored at −26°. (21 references.)

[B] The storage life of iced haddock (*Melanogrammus aeglefinus*) fillets after treatment with doses of γ-radiation of 75,000, 150,000 and 250,000 rads was studied. The unirradiated control became unacceptable after 12 days; the fillets irradiated with 75,000 rads and assessed after cooking became unacceptable after ~25 days at 0°; those irradiated at the higher levels showed no significant decrease in acceptability up to 27 days of storage in ice. (17 references.) E. M. J.

Extractives of fish muscle. IV. Seasonal variations of fat, water-solubles, protein and water in cod (*Gadus morhua* L.) fillets. N. Dambergs (*J. Fish. Res. Bd. Can.*, 1964, **21**, 703—709).—The concn. of all major components (protein water-solubles, fat and water) are subject to seasonal variations. During the period of spawning, fat decreases −20% and protein −5%; water solubles increase +10% and water +5%, and these values are restored to average annual values at various rates. The seasonal emaciation of cod is linked to the physiological processes taking place during reproductive activities. (12 references.) E. M. J.

Protein denaturation in frozen fish. VIII. The temperature of maximum denaturation in cod. R. M. Love and M. K. Elerian (*J. Sci. Fd Agric.*, 1964, **15**, 805—809; cf. J.S.F.A. Abstr., 1963, **i**, 103).—Cell fragility of cod muscle frozen at −29° and then stored at −0·5° to −3·5° at half-degree intervals was examined. Denaturation was most rapid at ~−1·5° and depended on the presence of ice in the tissue. When a similar no. of unfrozen pieces of fillet were kept at −1·5° in the supercooled state, after 12 days the optical densities remained almost constant. No denaturation had taken place in absence of ice in the tissue. (33 references.) E. M. J.

Volatile reducing substances in assessment of fish spoilage. N. K. Velankar (*Indian J. Technol.*, 1964, **2**, 247—248).—During storage the concn. of volatile reducing substances (**I**) in uniced fish (2° and 7·5°) increases uniformly, whereas in iced fish the concn. is irregular with time and may even decrease. Values of **I** are therefore not a valid index of spoilage in iced fish. W. J. Baker.

Pilchard oil: an analysis for component fatty acids with particular reference to the C_{24} chain length. R. G. Ackman and J. C. Sipos (*J. Fish. Res. Bd Can.*, 1964, **21**, 841—843).—Comment was made on the report that in the analysis of the oil of the Pacific sardine or pilchard (*Sardinops sagax*) 15·2% of highly unsaturated C_{24} acids were found. The normal range of C_{24} acids in marine teleost fish does not exceed 1—2%. By gas–liquid chromatography the presence of 24 : 1 acid in a no. of fish oils was found but other workers found C_{24} acids only in herring oil; this latter report being in agreement with the present analysis and with the suggestion that the amount of 24 : 1 acid normally exceeds that of C_{24}-polyunsaturated acids in marine oils. The I val. of the oil examined is higher than that found by other workers. This and the composition of the plankton species on which the pilchard feeds are discussed. (14 references.) E. M. J.

Effects of ionising radiation on lipids of fish. V. F. Stout and D. D. DesMarteau (*U.S. atomic Energy Commission*, 1964, Rep. TID 19586, 18 pp.).—Autoxidation of methyl docosahexaenoate (**I**) was studied; two methods of prep. were used and tests were made to ascertain whether pre-exposure to O_2 sensitised the lipid to oxidation, but in both cases similar results were obtained. **I** was insensitive to radiation-induced oxidation but the Fe^{2+} ion catalysed the action slightly and the Co^{2+} on Mn^{2+} ions showed a more pronounced effect. In general irradiation showed a more marked reaction as compared with the un-irradiated controls. C. V.

Spices, Flavours, etc.

Sensory evaluation of foods. R. M. Pangborn. (75 references.) **Interactions of food technology with nutrition during the last 25 years.** A. F. Morgan. (21 references.) **Quality control in processing foods.** C. H. Brokaw and A. Kramer. (11 references.) (*Food Technol.*, 1964, **18**, No. 9, 63—67; 68—72; 73—78.) E. M. J.

Consumer preference evaluation of the storage stability of foods. D. R. Peryam (*Food Technol.*, 1964, **18**, No. 9, 214—217).—A successful system worked out over the last 13 years to test food stability is described. It depends on the preference evaluation using the hedonic-scale method with groups of people selected from a large pool of ordinary consumers. The design is factorial and the data are subjected to analysis of variance. Precise deterioration (if it has occurred) and the relative importance of product variables are shown. Prediction of the end point of useful storage life under a given set of conditions is determined with reference to background information on the particular type of product. E. M. J.

Flavour-modifying properties of disodium inosinate. C. H. Kurtzman and L. B. Sjöström (*Food Technol.*, 1964, **18**, No. 9, 921—223). —Food products (41) comprising soups, canned meats, dairy products, canned and frozen vegetables, cereals and grains, etc. were used. Disodium inosinate (**I**) in concn. ranging from 0·0075 to 0·05% generally improved the flavour blend and fullness of food

products and showed specific trends of flavour modification. A sensation of increased η in liquid or semiliquid food products was created. **I** should be considered favourably as a seasoning compound in products in which it acts as a flavour enhancer, especially for use in soups, canned meats and some canned vegetables. E. M. J.

Quick and easy method of analysis on organoleptic data. Kan-ichi Hayakawa and E. F. Stier (*Food Technol.*, 1964, **18,** No. 9, 224—227).—A quick and easy test of the significance of two-part % by applying tables for partial sums of binomial distribution is presented. Examples of analysis are shown. (10 references.) E. M. J.

Flavour studies of solidified water-in-milk-fat emulsions. J. B. Mickle, C. V. Patel, M. V. Malkus, M. E. Leidigh, H. J. Baker and R. D. Morrison (*Food Technol.*, 1964, **18,** No. 9, 237—239).—Solidified emulsions, water in milk fat, containing 40 or 50% of milk fat, emulsifier and water had the physical characteristics of butter. Emulsifiers with hydrophile–lipophile balance (HLB) no. of 2—5 were the most effective in producing water-in-oil stable emulsions. Untrained panel judges preferred 80% milk fat product to margarine and all of the low fat products. (12 references.) E. M. J.

Ultraviolet absorbancy of volatiles as a measure of oxidative flavour deterioration in egg powders. O. S. Privett, O. Romanus and L. Kline (*Food Technol.*, 1964, **18,** No. 9, 239—242).—Oxidative flavour deterioration in egg-yolk powders was examined by peroxide determination on extracted fat, carbonyl and thiobarbituric acid values on steam distillates of whole powders, and u.v. absorbancy (**I**) of volatiles at 280 mμ; the results were compared with those by tasting tests. The **I** increased in all deteriorated samples and the max. at 280 mμ correlated well with the degree of off-flavour in the powders and over a wider range than did the other chemical techniques. (13 references.) E. M. J.

Chemistry of food odours. R. V. Golovnya, G. A. Mironov and S. D. Sokolov (*Usp Khim.*, 1964, **33,** 816—854).—Methods of separation and identification are discussed. Materials found in the odour of milk, milk products (cream, butter, cheese), also bread, cocoa, coffee, fish and meat products, are identified and related to aldehydes, ketones, alcohols, complex esters, fatty acids, amines, heterocyclic compounds (furan and its deriv.) and S compounds. Variations which occur in odour of food are considered in relation to accumulation or breakdown of different food compositions, e.g., aromatic compositions which can form volatile components, distribution of fats and protein and autoxidation reactions. (299 references.) A. L. B.

Micro-analysis of essential oils. XIV. Detection and quantitative determination of phenols in essential oils by paper-chromatographic separation as the 4-phenylazobenzoate esters. R. Pohloudek-Fabini and P. Münchow (*Pharmazie*, 1964, **19,** 591—596).—Thymol in oil of thyme is determined by conversion into its 4-phenylazobenzoate, chromatographic separation on paper impregnated with paraffin, elution with hexane and measurement of the extinction of the eluate at 325 mμ. The coeff. of variation is $\simeq \pm 7\%$. A. R. ROGERS.

Preservatives

Effects of mixed preservatives. IX. Effects of binary and trinary combinations of preservatives with nisin and tylosin. H.-J. Rehm, F. Senser and E.-M. Lukas (*Z. LebensmittUntersuch.*, 1964, **125,** 258—271).—In continuation of previous work (cf. *ibid.*, 1964, **124,** 437), useful synergisms against Lactobacilli are observed for binary mixtures of nisin or tylosin with sorbic acid, BzOH or esters of *p*-hydroxybenzoic acid, and against *Escherichia coli* for an admixture with formic acid (only). Synergisms are observed for many trinary combinations, thus sorbic acid and benzoic acid (which are ineffective alone) can increase the effectiveness against *L. buchneri* as additions to binary mixtures containing nisin or tylosin. *L. buchneri* readily develops resistance to nisin, but not to tylosin; this effect is greatly retarded in the presence of other preservatives. P. S. ARUP.

Physical and chemical characterisation of cellulose ethers used as food additives. I. Production, properties and uses. S. W. Souci, F. Crössmann and E. Mergenthaler (*Z. LebensmittUntersuch.*, 1964, **125,** 247—258).—A review. British and American standards for purity are discussed with some alternative proposals. (22 references.) P. S. ARUP.

Food Processing, Refrigeration

Food preservation. I. Progress in dehydration 1939—1964. E. Seltzer. **II. Refrigerated foods.** W. H. Cook. **III. Heat processing.** C. R. Stumbo. **IV. Chemicals.** L. A. Hall. **V. Radiation.** S. A. Goldblith (*Food Technol.*, 1964, **18,** No. 9, 117—120, 122—124, 127—129, 131—135, 138, 143—145).—I. The advances and applications of dehydration to, e.g., instant beverages (milk, coffee, tea and fruit drinks), pastry mixes, dessert- and soup-mixes, etc., control of browning, various types of dryers, freeze drying and special dryers are discussed. (21 references.)

II. The general principles and practices required to produce frozen foods of high quality (Tressler and Evers, 1957) and the commercial application (Williams, 1963) are discussed. By reducing the size of the unit to be frozen to 2 in. or less in one dimension, the freezing time was reduced to $\sim\frac{1}{10}$ of that previously required. The products had to be prepared for cooking, packaged before freezing and kept frozen until they reached the consumer. (12 references.)

III. Progress in the successful preservation of perishable food products by heat-processing in glass jars, bottles as demonstrated by Nicholas Appert in 1810 and the development of the canning industry are reviewed.

IV. New chemicals developed for food use over the past 25 years are classified according to use as bacteriostatic, fungistatic or germicidal agents, antioxidants, stabilisers, emulsifiers, thickening agents, plasticisers, firming agents, coating agents, humectants and neutralisers. The Commissioner [of Foods and Drugs] (U.S.) regards food ingredients as NaCl, pepper, sugar, vinegar, baking powder and monosodium glutamate as safe for their intended use. Lists are given of additional substances that when used for the purposes indicated, are regarded by the Commissioner as generally recognised as safe for such uses.

V. The attempts of food scientists to utilise ionising energy for food preservation from 1943 when the effects of *X*-rays on a hamburger were studied, are briefly summarised. The greatest single problem that prevents the large-scale use of ionising energy for food preservation is still the undesirable side reactions induced when ionising energy bombards foodstuffs. E. M. J.

[A] **Applications of refrigeration to the preservation of acid fruits.** [B] **Deodorisation of cold stores.** Alejandro Reig Feliú, Carlos Pérez Nievas and Antonio Albert Bernal (*An. Inst. nac. Invest. agron., Madr.*, 1963, **12,** 69—118, 119—134).—[A] Studies of the effects of N fertilisers on the refrigeration properties of Washington Navel oranges are reported in detail. Trees were treated with from 0 to 9 kg. of $(NH_4)_2SO_4$ together with 2 kg. of superphosphate and 1 kg. of K_2SO_4 per tree in spring, with or without treatment with $Ca(NO_3)_2$ during the summer. Oranges were treated with Na *o*-phenylphenate, stored for 90 days at 1° and 5° at 85% R.H. with forced air circulation, and at 3°, 5° and 10° without aeration or humidity control, and examined at intervals for loss in wt., desiccation, % of pulp, juice and cortex, thickness of cortex, sol. solids, acidity, sucrose, reducing sugars and vitamin C. At 10° loss in wt. and attack by micro-organisms was excessive; reducing sugar content and loss in wt. increased with temp. and duration of storage, and vitamin C was preserved best at 1°, but otherwise there was little difference in the results under different conditions of storage. Increased N application caused increase in wt. loss, % of pulp and % and thickness or cortex, and decrease in % of juice and vitamin C content. N treatment reduced liability of the fruit to mechanical damage, but high treatments gave excessive thickness of cortex. (11 references.)

[B] Cold stores (capacity 15·6 cu. m.) were pre-treated by washing the walls with a 16% solution of chlorinated lime (using 36 g. of $CaCl_2$ per sq. m.) and introducing 6 sq. m. of paper impregnated on one side with melted lard. The stores were then loaded with apples (100 kg.), oranges (40 kg.), onions (100 kg.) and potatoes (100 kg.) together with 120 eggs and some packets of butter as odour absorbers. Odour levels were assessed organoleptically and quant. by aspiration of the air through 0·004N-$KMnO_4$. Uptake of odours from one food to another was found only to occur when reducing conditions were detectable. The effects of both deodorants, and of the periodic removal of deteriorated food were marked. Chlorinated lime had immediate effect; the action of lard was slower, but was effective for longer periods. Deodorisation by lard is most effective between 1 and 5° and between 28 and 32°, but is unaffected by the odour level originally present. It is recommended that periodic analysis of the air should be performed and that lard should be exposed in closed cold stores where incompatible foodstuffs are stored together. E. C. APLING.

Drying particulate foodstuffs. Unilever Ltd. (Inventors: D. V. S. K. Rao, J. H. Pennington and G. J. Pickard) (B.P. 937,461, 24.6.60).—The foodstuff is fluidised in a bed (of depth 1—8 in.) above a horizontal rigid air-permeable base by passing warm air upwardly therethrough (at a temp. preferably 110—120°F at a rate of 200—600 ft. per min.), whilst the base is vibrated (at a frequency of 1000—1500 cycles per min.) in a direction inclined to the horizontal). The process is especially useful in the drying of peas. Apparatus is figured. F. R. BASFORD.

Pesticides in Food

Residual toxicity of pesticides in foods. G. Baluja Marcos (*Rev. Cienc. apl.*, 1964, **18**, 205—216).—The mechanism of action of pesticides, chemical and biochemical transformations, permanence, the toxicity of residues on foodstuffs, and methods of determining the permissible levels are discussed. (12 references.)
L. A. O'Neill.

Degradation of malathion on stored maize and wheat grains. D. G. Rowlands (*J. Sci. Fd Agric.*, 1964, **15**, 824—829).—The samples of grain were treated with malathion, stored in sealed jars for 6 months and tested monthly to examine metabolites produced by enzymic or chemical action in the grains themselves. Dimethyl phosphorothiolothionic acid (**I**), malathion mono- and di-acids (**II**) were identified by thin-layer chromatography. The malathion breaks down slowly by chemical and by enzymic hydrolysis to give **I** and **II**, respectively. (16 references.) E. M. J.

Polarographic determination of residual 2,6-dichloro-4-nitroaniline (dichloran) in fruits. J. Vogel and J. Deshusses (*Mitt. Lebensm. Hyg., Bern.*, 1964, **55**, 151—154).—Dichloran is extracted from a mixture of the fruit pulp by stirring with MeCN. After evaporation of the MeCN, the residue is dissolved in water, and the aq. solution is continuously extracted with PhMe. A solution in aq. NaOH of the residue obtained after evaporation of the PhMe is then used for the polarographic determination of the Dichloran by the reduction of the NO_2 group. The accuracy is within $\pm 3\%$ for 2—7 p.p.m. P. S. Arup.

Detection and determination of biphenyl in citrus fruits by gas chromatography. J. Vogel and J. Deshusses (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 84—92).—A mixture of the peel and dil. aq. H_2SO_4 is refluxed in an apparatus designed to trap the biphenyl (**I**) in cyclohexane. After acids and phenols have been removed by extraction with aq. NaOH + Na_2SO_4, an aliquot of the cyclohexane solution is cautiously evaporated to ~0·2 ml. for gas-chromatographic examination as described in detail. A known quantity of **I** is added to a second aliquot to function as an internal standard. Tangerine peel contains methyl *N*-methylanthranilate which interferes with the determination of **I**. A procedure is described by which this substance is removed by conversion into the insol. K salt of the acid. The reproducibility of the results of 10 analyses was $\pm 2\%$ at P = 0·95. The peel of citrus fruits (excepting tangerines) from various sources contained 0·4—45 p.p.m. of **I**. P. S. Arup.

Detection of residual insecticides (chlorinated hydrocarbons and phosphoric esters) on fruits and vegetables by means of paper and thin-layer chromatography. F. Eder, H. Schoch and R. Müller (*Mitt. Lebensm. Hyg., Bern*, 1964, **55**, 98—144).—In continuation of the survey published by Müller, Ernst and Schoch (cf. *Anal. Abstr.*, 1958, **5**, 1014), comments on available methods are followed by full directions for the determination of 21 pesticides. (36 references.)
P. S. Arup.

Thin-layer chromatography of substances used for treatment of peel of citrus fruits. Biphenyl, o-phenylphenol and 2,4-dichlorophenoxyacetic acid. T. Salo and R. Mäkinen (*Z. LebensmittUntersuch.*, 1964, **125**, 170—171).—These three substances can be separated on SiO_2-gel with Shell Sol. A–AcOH (24 : 1) as developing solvent and a solution in MeOH of the laundry whitening agent Blankophor DCB as spraying reagent. The chromatograms are examined under u.v. light. P. S. Arup.

Effects of processing and storage of dairy products on chlorinated insecticide residue. I. DDT and lindane. B. E. Langlois, B. J. Liska and D. L. Hill (*J. Milk Tech.*, 1964, **27**, 264—270).—The only change in structure was found in DDT and lindane during drying of milk into powder but in general the finished products, other than dry whole milk, contained the same amount of insecticide as the raw milk when expressed on a fat basis. C. V.

Analysis of animal food products for chlorinated insecticide residues using electron-capture gas chromatography. I. Column clean-up of samples. II. Various factors involved in this form of analysis. B. E. Langlois, A. R. Stemp and B. J. Liska (*J. Milk Tech.*, 1964, **27**, 202—204, 231—234).—I. A. one-step method applicable to whole-dried whole- and evaporated milk, cream, butter, cheese, egg-yolk, chicken tissue, blood, soil, etc. is outlined. Sufficient accuracy is attained to enable the method to be employed either as a screening procedure or a research tool, and 25–35 samples can be examined in an 8-h. day.

II. Levels of 0·01 p.p.m. of several insecticides may be determined under ideal conditions. Representative samples must be available, the linearity of the instrument must be determined for each compound, all the reagents must be re-distilled, only high-purity N_2 must be used as carrier gas, the chromatographic clean-up column must not be overloaded with fat and suitable elution techniques must be followed. Proper choice of column packing in relation to the insecticide is necessary together with the use of borosilicate glass, correct temp. control, and calibration of instruments. (13 references.) C. V.

Total phosphorus technique for determining organophosphorus pesticide residues using Schöniger flask combustion. R. C. Blinn (*J. agric. Fd Chem.*, 1964, **12**, 337—338).—A procedure is described for the determination of organo-P pesticide residues by means of the Schöniger Flask Combustion technique. P is determined by a Mo blue colorimetric procedure. Sensitivity of 0·1 p.p.m. is attained with 100 g. samples of plant material. (25 references.)
W. Elstow.

Packaging

Past, present and future of packaging processed foods. A. H. Andersen (*Food Technol.*, 1964, **18**, No. 9, 153—157).—In the war years food packers learned the new mediums of pouch packing, waxed paperboards, waterproofing, multiple and individual unit containers, etc. Developments in packaging continued in post-war years. Of the thousands of developments in the packaging of processed foods over the past 25 years a few of the more striking and revolutionary are described. These include the use of the Al can for highly sensitive freeze-dry products, use of resistant, light-wt. glass, application of glass-coating systems which have reduced skin fractures of glass containers, rigid plastic containers, flexible pouches, etc. E. M. J.

Campaign to increase use of petroleum waxes in packaging. Anon. (*Food Technol.*, 1964, **18**, No. 9, 157—158).—The market position of wax for packaging materials, with suggestions for improvement, are discussed, covering: the introduction of new additives, e.g., polyethylenes, copolymers and resins; the use of waxed paper in bread wrapping; new application methods; authorisation for use. The use of Al, e.g., in the beer industry, in one-time baking dishes, is also discussed. E. M. J.

Determination of permeability to aromas of foils and of papers impregnated with plastic products. H. Hadorn and K. Zürcher (*Mitt. Lebensm. Hyg., Bern.*, 1964, **55**, 154—181).—The apparatus includes equipment for the supply of a current of pure air at a constant rate and pressure, R.H. and temp. to the underside of the sample sheet which is fixed horizontally in a diffusion chamber. A little aromatic material (e.g., powdered cloves) is placed on the top surface of the sheet; the permeating aroma is carried off from below, absorbed in acid 0·25N-$K_2Cr_2O_7$, and determined by back-titration. The permeability is expressed as the no. of mg. of aromatic matter permeating per h., per sq. m. of the sample; values quoted for six samples are 7—174 mg. P. S. Arup.

Packaging of foods in laminate and aluminium-film combination pouches. I. Tomato paste. B. S. Luh and G. de la Hoz (*Food Technol.*, 1964, **18**, No. 9, 227—230).—Tomato paste (26% total solids) was processed in three-layer laminate (**I**) and Al-film combination (AFC) pouches and stored at 32, 75 and 86°F. The **I** pouches were permeable to moisture and on storage at 86°F for 10 months lost 10% of the initial wt. Serum colour darkened rapidly at 86°F and very slowly at 32°F and the rate was faster in **I** than in AFC pouches; the latter being impermeable to light protected the paste more effectively. Paste lost ascorbic acid more rapidly in **I** than in AFC pouches. (12 references.) E. M. J.

Polymer packaging for freezing and preservation of fish. W. Pichel (*ASHRAE J.*, 1964, **6**, No. 10, 78—79).—A general discussion. C. V.

Miscellaneous

Nutrition, proteins, amino-acids, vitamins

Infant food based on coconut protein, groundnut protein isolate and skim milk powder. I. Preparation, chemical composition and shelf-life. M. R. Chandrasekhara, G. Ramanatham, G. Rama Rao, D. S. Bhatia, M. Swaminathan, A. Sreenivasan and V. Subrahmanyan. **II. Overall growth-promoting value and supplementary value to poor cereal diets.** G. Rama Rao, K. Indira, G. Ramanatham and M. R. Chandrasekhara (*J. Sci. Fd Agric.*, 1964, **15**, 839—841, 841—846).—I. A process for the prep. of spray-dried infant food containing protein 26% and fat 18% is described. The blend contained coconut skim milk concentrate and skim milk powder, the composition being adjusted so that the proteins contributed by coconut, groundnut and skim milk were in the proportion of 2 : 2 : 1 respectively. Dextrins and hydrogenated groundnut oil were added and the powder was fortified by dry-blending with minerals, thiamine, niacin, riboflavin, ascorbic acid and calciferol; nine other

vitamins were added (details given). Samples were free from *Escherichia coli* and other pathogenic anaerobes. Packed in polyethylene bags and stored in tin containers it kept well for 9 months. Losses of vitamins A and C and thiamine during that time were 25, 32 and 20%, respectively. (15 references.)

II. The growth of rats fed the infant food (**I**) was 15·3 g. as compared with 13·9 in those fed a control milk food (**II**) of a similar composition. A 3 : 1 blend of **I** and cane sugar promoted a weekly growth of 18·1 g. as compared with 16·1 g. with **II** and sugar. Protein and Ca contents of the carcasses of animals given **I** were lower than of those given **II**. In maize–tapioca diets, added at 20% level (5% of extra protein) and in rice diets at 10% level, **I** promoted growth. The supplement of **I** or **II** was effective in correcting protein deficiency in the diets and in preventing liver damage. (19 references.) E. M. J.

Improved recovery of protein from rendering-plant raw materials and products. I. Liquid cyclone separation with carbon tetrachloride. L. G. Criswell, R. W. Schatz, J. H. Litchfield, V. G. Vely, G. H. Sachsel, J. C. Picken, jun. and H. E. Biester. **II. Acid and enzyme hydrolysis.** L. G. Criswell, J. H. Litchfield, V. G. Vely and G. F. Sachsel (*Food Technol.*, 1964, **18**, No. 9, 243—247, 247—251).—I. Renderer's scrap meat treated in liquid cyclone with CCl_4 gave an enriched protein (75%) fraction; >90% of the bone was rejected. Treatment of the raw material by grinding, agitation and attritioning and separation in liquid cyclone with CCl_4 gave an enriched product containing 60% protein and 20% bone. Calf-toxicity assay was made with two Holstein calves and on a 60-day trial no indication of *S*-(dichlorovinyl)-L-cysteine toxicity was observed. (23 references.)

II. By acid (H_2SO_4) hydrolysis of protein fractions of meat scrap a final product containing 90% protein was obtained. Excess acid was neutralised with lime giving $CaSO_4$ waste. This and the fact that tryptophan was lost in the process are disadvantageous. In enzyme hydrolysis of meat scrap, fungal protease, bromelin and papain were used. The raw meat and bone were ground, heated and hydrolysed with fungal protease, centrifuged and dried. A clean separation of fat from protein and protein from bone was obtained. The protein fraction contained 70% protein, 75% of which was sol. in water. E. M. J.

Availability of sulphur amino-acids in protein foods. I. Total sulphur amino-acid content in relation to sulphur and nitrogen balance studies in the rat. E. L. Miller and K. J. Carpenter (*J. Sci. Fd Agric.*, 1964, **15**, 810—820).—Seven commercial concentrates (fish, meat and whale meals) were fed to rats. There was no correlation between the S content (expressed per 16 g. of N) and their net protein utilisation, although the quality of the protein was limited by the S-containing amino-acids. Cystine and methionine accounted for $<\frac{2}{3}$ of S found in five of the samples. Digestibility of the S ranged from 53 to 90% and, except for two meals which had the highest ratio of cystine to methionine, was similar to the corresponding figure for N. The product of S amino-acid content and S digestibility of the meals correlated with their net protein utilisation. Chromatographic and iodometric techniques were used in recovery experiments for the estimation of cystine and methionine. (51 references.) E. M. J.

Effect of amino-acid suplementation on nutritive value of yeast hydrolysate. M. K. Rastogi and C. R. Krishna Murti (*Indian J. exp. Biol.*, 1964, **2**, 143-145).—Rats were fed diets with the sole protein consisting of casein, or of yeast hydrolysate, alone or supplemented by lysine, cystine + methionine, or lysine + cystine + methionine. Rates of growth and protein efficiency ratios were determined. Cystine and methionine supplementation raises the nutritive value of yeast hydrolysate to that of casein; lysine has no added advantage. Feeding yeast hydrolysate alone has an adverse effect on respiratory rate in liver slices, but no effect on liver transaminase activity or on the histological appearance of the liver. (26 references.) A. T. CARPENTER.

Automatic amino analysis: Reagent and instrumental improvements. N. G. Cadavid and A. C. Paladini (*Analyt. Biochem.*, 1964, **9**, 170—174).—The procedure described uses the Auto Analyzer apparatus combined with a modification of Rosen's reagent. A narrow column is used (0·6 cm. i. d) and it is possible to estimate 0·03μ mole of a single amino-acid with a standard deviation of $\pm$5%. Amounts 10 times above the lower limit can be detected. (11 references.) C. V.

Blood amino-acid studies. V. Determination of the limiting amino-acid in diets. J. M. McLaughlan (*Canad. J. Biochem.*, 1964, **42**, 1353—1360).—Diets containing limiting amounts of various amino-acids were fed to male weanling rats and levels of plasma lysine, methionine, threonine and tryptophan determined. The limiting amino-acids in various materials were then assessed by means of the plasma amino-acid score. (17 references.) S. A. BROOKS.

Gas-liquid chromatography of fat-soluble vitamins. K. K. Carroll and D. C. Herting (*J. Amer. Oil Chem. Soc.*, 1964, **41**, 473—474).—On a 6 ft. × $\frac{1}{8}$ in. column containing Gas Chrom P 100—140 mesh coated with 3% SE-30 liquid phase; the *d*-isomers of mono-, di- and tri-methyltocopherols emerged in this order, when the column temp. is 205°, although *d*-γ- and *d*-δ-tocopherols could not be resolved. Vitamin A prep. show evidence of decomposition and ubiquinones gave no peaks on the chromatogram, although two peaks were obtained with vitamin K_1. G. R. WHALLEY.

Suitability of bagasse hydrolysate medium for production of riboflavin by *Candida utilis*. P. N. Agarwal, T. N. Rawal and A. K. Gurtu (*Indian J. Technol.*, 1964, **2**, 246—247).—Variables affecting intra- and extra-cellular biosynthesis of riboflavin (**I**) were studied. Yield of **I** increases with duration of fermentation (to 48 h.) and with concn. of added niacin (**II**), methionine or glycine as N source (**II** almost doubles the yield). Speed of stirring has no effect, but 0·03—0·8 p.p.m. of Na, Mn and Zn, or mixture thereof, decreases the yield. Fermentation for 48 h. at 125 r.p.m. in a medium containing bagasse hydrolysate (23 ml., with 1% xylose), 2·5% KH_2PO_4 solution (1 ml.) and 1·5 μg. **II** plus 0·063 g. of methionine results in an intracellular yield of 79 μg. per g. and an extracellular yield of 81 μg. per 100 ml., approx. the same as when molasses medium is used. W. J. BAKER.

Qualitative and quantitative method of determining vitamin B_{12} synthesised by Actinomycetes on nutrient agar medium. L. Nachev and R. Gesheva (*Mikrobiologiya*, 1964, **33**, 739—743).—An easy, rapid method is described for qual. and quant. determination of vitamin B_{12} synthesised in nutrient agar medium by Actinomycetes (**I**). The build-up of vitamin B_{12} in over 500 **I** cultures prepared from Bulgarian soils was studied and 49 results are tabulated. *Escherichia coli* 113-3 in agar medium containing detector dye was used as test-culture. Cultures of **I** were grown for 4—15 days on nutrient agar medium containing Co. The vitamin B_{12} content was determined after 4, 7, 10 and 15 days by the usual disk method. A chromatostrip method was used to determine factors present in vitamin B_{12}. Three factors of vitamin B_{12} were detected: (i) occurred in two strains, (ii) (cyanocobalamin) in all and (iii) in a few. P. W. B. HARRISON.

Factors affecting the stability of vitamin C in tropical fruit juices and nectars. A. R. Rahman, J. Anziani and J. R. Cruz-Cay (*J. Agric. Puerto Rico*, 1964, **48**, 1—12).—The rate of destruction of vitamin C was high in fruit juices and nectars during boiling for 1 min. or incubation at 250° for 10—15 min., particularly when vitamin C concn. was <2 g./l. Variation in pH (3—10) during incubation did not affect rate of destruction of vitamin C. Juices stored exposed to air had greater losses of vitamin C than had those in sealed containers, and the % loss was highest with low concn. of vitamin C. A. H. CORNFIELD.

Unclassified

Public health aspects of food microbiology. G. Wilson (*Chem. & Ind.*, 1964, 1854—1859).—Milk-borne diseases, the bacteriology of spray-dried egg, ice-cream and staphylococcal food poisoning, the problems of communal feeding and *Clostridium welchii* destruction, processed egg and synthetic cream and salmonella infection arising from imported foods are reviewed with special reference to their early recognition and the steps that have been taken for their elimination. Fertilisers and animal foodstuffs, meat and salmonella infection are also considered. (31 references.) C. V.

Comparison of media for isolation of *Clostridium perfringens* from food. J. M. L. Southworth and D. H. Strong (*J. Milk Tech.*, 1964, **27**, 205—209).—Five agar culture media were studied; it was found that in every case that described by Angelotti (*Appl. Microbiol.*, 1962, **10**, 193) proved the best for the isolation and identification of clostridia. The presence of sulphadiazine and polymyxin-β-sulphate did not interfere in the recovery. (17 references.) C. V.

Production of fermentation citric acid by surface [culture] methods. III. Production of crystalline citric acid from fermented liquors and of a pectinase preparation from the mycelium resulting from the fermentation. A. Pelc, J. Bartfay, L. Vamos, E. Szep, F. Dolanszky and S. Gavalya (*Nahrung*, 1964, **8**, 229—233).—In continuation of previous work (cf. *ibid*, 147), it is pointed out that the removal of Ca^{2+} by treatment with a cation-exchanger, from the solution of the pptd. Ca citrate in dil. H_2SO_4 obviates the necessity for interrupting the subsequent evaporation of the solution to filter off the pptd. $CaSO_4$. This expedient also permits of the use of slaked lime instead of pptd. $CaCO_3$ for the pptn. of the citric acid as Ca citrate from the fermented liquor, and can be relied on to remove traces of Fe that may not have been removed by the treatment with $K_4Fe(CN)_6$. An efficient pectinase prep. can be obtained by pressing the washed and air-dried mycelium. P. S. ARUP.

Effect of potassium ferrocyanide on the preparation of molasses solutions for citric acid fermentation. I. H. Leopold and Z. Valtr (*Nahrung*, 1964, **8**, 37—48).—Potassium ferrocyanide levels in ppt. and bound and free in solutions of molasses as prepared in various ways were studied. Under industrial conditions most of the ferrocyanide is precipitated. Ferrocyanide precipitates heavy metals in the molasses solutions and more of it appears in the ppt. as the no. of sterilisations increases, the temp. is raised, the pH is lowered and the concn. of the fermentation medium increases. Under lab. conditions it was not possible to precipitate all the ferrocyanide. Industrial prep. gave an increased yield of citric acid on addition of more ferrocyanide but only if the depth of the culture was limited.
J. B. Woof.

Influence of cerium^{III} nitrate and lanthanum^{III} nitrate on growth and acid production of *Aspergillus niger* and the formation of acids. III. Possibilities of chemical influence on vital manifestations of micro-organisms. R. Springer and G. Mühlpoltner (*Z. Lebensmitt-Untersuch.*, 1964, **125**, 81—90).—In continuation of previous work (cf. *Planta med.*, 1960, **8**, 411), it is found that acid formation is initially decreased by the addition to the medium of $Ce(NO_3)_3$ or $La(NO_3)_3$ in concn. 0·1—10 mg./100 ml., but that a marked recovery occurs after the first 8—10 days. These results are explained by an interference with the phosphatic–citric acid cycle leading to an increased production of H_3PO_4 and oxalic acid which inactivate the Ce and La by pptn. A marked increase in the production of gluconic acid is also observed. The mycelium becomes spongy and prone to an excessive fructification. (42 references.) P. S. Arup.

Counting of hydrocarbon-oxidising bacteria. Z. S. Smirnova (*Mikrobiologiya*, 1964, **33**, 737—738).—Pure cultures of *Mycobacterium luteum* (**I**) and *M. rubrum* (**II**), which propagate by oxidation of propane, were grown in liquid mineral medium in propane–air atm. The rate of growth depends on the proportion of propane in atm. The no. of organisms was determined (1) visually on an arbitrary scale, (2) by direct counting of cells under the microscope and (3) by measuring the optical *d* of the bacterial suspension. Tables show the relation between intensity in arbitrary units and optical *d* for red, green and orange filters, and the count of **II** determined microscopically and arbitrary units of intensity.
P. W. B. Harrison.

Activity induced in food by electron, *X*-ray, and cobalt-60 γ-irradiation. F. Rogers (*U.K. Atomic Energy Authority A.E.R.E. Rep.*, 1964, R-4601, 28 pp.).—In a study of possible irradiation hazards arising from the irradiation of food semi-empirical formulae, from published data for induced activity levels in nucleides, are applied to ' max. credible concn.' (M.C.C.) in foods for ^{60}Co 1·33 MeV γ-irradiation and for electron accelerator energies up to 5, 7, 10 and 15 MeV. As activities in terms of curies were not biologically comparable, results were expressed, for each of these irradiation sources, as fractions of the max. permissible concn. in drinking water for public use for irradiation periods of 5 min. to 10 days with an indication of the most significant impurity. The largest factor of uncertainty was the value of (M.C.C.) assumed. It is suggested that ^{60}Co, electrons <5 or 7 MeV and *X*-rays <5MeV appear incapable of inducing any significant activity in any foods; the other forms of radiation appear generally safe subject to certain restrictions on type and quantum energy, food composition, etc. Comparisons with previous similar estimates are made.
J. W. Taylor.

Salad dressing. National Dairy Products Corp. (B.P. 938,717, 25.1.62. U.S., 6.2.61).—An emulsified salad dressing which is stable against freeze–thaw conditions is made by preparing a starch base containing a modified freeze-resistant starch (product of action of phosphate on a waxy starch, e.g., tapioca starch, waxy maize), enough to imbibe all the moisture present in the dressing and retain it under said conditions, then adding edible oil (5—35 wt.-%) and emulsifying agent, e.g., egg yolk (>0·12 pt. per pt. of oil).
F. R. Basford.

3.—SANITATION, WATER, etc.

Progress in food plant sanitation. R. H. Vaughn (*Food Technol.*, 1964, **18**, No.9, 147—150).—Problems being dealt with in the 1930's and after World War II are discussed. Advances in food plant sanitation after the War cover: legal responsibility, break-point chlorination, cleaning in place, washing of raw products, preventive control of insects and rodents, sanitary design of buildings and equipment and field sanitation. Other problems are discussed. (17 references.) E. M. J.

Rôle of microbiological methods in the sanitary control of cereals and cereal products. P. S. Prickett (*Cereal Sci. Today*, 1964, **8**, 192—194, 197—199).—A commonsense approach to microbiological control is emphasised. Careful evaluation and well-understood universally applied methods are needed. The reasons for sanitary control, the methods in manuals and factors common in all methods are outlined. Regular and routine methods and specific bacterial counts are examined. I. Dickinson.

Pesticidal potentiality of Petkolin in comparison with other chlorinated insecticides. S. H. Ashrafi, S. M. Murtaza and Dilshad Asmatullah (*Pakist. J. sci. industr. Res.*, 1964, **7**, 211—213).—Pesticidal potentiality of Petkolin in comparison with other chlorinated insecticides was studied by using topical application method against cockroaches and houseflies. The LD_{50} was found to be in the range 64—96 μg. per cockroach and 8—20 μg. per fly. Against mosquito larvae, LC_{50} was in the range 5—8 p.p.m. and LC_{95} in the range 8·5—15 p.p.m. Petkolin was more toxic than Makrolin against houseflies and mosquito larvae, but less toxic to cockroaches. A. Jabbar Mian.

Cholinesterase variation as a factor in organophosphate selectivity in insects. W. C. Dauterman and R. D. O'Brien (*J. agric. Fd Chem.*, 1964, **12**, 318—319).—The claim that di-isopropyl *p*-nitrophenyl phosphorothionate (**I**) is more toxic to houseflies than bees has been examined and shown to be true. **I** is 100 times more toxic to houseflies than bees. Four other dialkyl paraoxons are examined from this aspect. *In vitro* experiments show that whilst fly and human cholinesterase are similar bee cholinesterase is not. (16 references.)
W. Elstow.

Water wastes and sewage

Biological treatment of protein water from manufacture of potato starch. II. Experimental results and discussion. J. C. Buzzell, jun., A.-L. Caron, S. J. Ryckman and O. J. Sproul (*Wat. Sewage Wks.*, 1964, **111**, 360—365).—This waste can be successfully treated by standard biological treatment procedures. The activated sludge gave BOD removals of ~95% at loading intensities up to 80 lb. of BOD per 1000 lb. mixed liquor suspended solids per h. aeration. Standard rate trickling filters showed 90% or better BOD removals with org. loadings up to 1300 of BOD/acre ft./d. Similar values were attained on high-rate filters with loadings up to 3000 lb. A higher org. loading limit for high rate of filtration might be obtained with a larger-size stone than that used in the present study. C. V.

Mass spread of plankton algae in self-purification of sewage waters in biological ponds. T. N. Sivko and T. A. Sokolova (*Mikrobiologiya*, 1964, **33**, 699—704).—The use of biological ponds for cultivation of algae is described. Observations were made on ponds operated on a fill-and-draw cycle, fed with raw sewage containing industrial and domestic waste. The outlet height was set to retain enough water to inoculate a fresh charge of sewage. Each pond developed its own characteristic algal flora, but the rate of accumulation of algae was roughly same in all, equivalent to 750—1250 kg./ha. Phytoplankton (**I**) was assessed from difference in $K_2Cr_2O_7$ oxidation figures for raw and centrifuged pond water. With **I** having ash content of 5%, 1 mg. of O_2 consumed represents 0·735 mg. of dry algal matter. Rate of growth of algae falls off after the pond has been filled and this is attributed to reduction in light penetration with algal growth. Algae production (g./m.^{2}/24 h.) is highest in May and June, but falls later because Rotiferae, which flourish with high temp. of water, consume large quantities of algae. In Sept. Rotiferae decrease and algae again increase. Assuming that respiration constitutes 15—20% of photosynthesis under optimum conditions, the efficiency and utilisation of light energy appeared greatest under feeble lighting conditions. The total wt. of algae produced from May 1 to Nov. 15 was 11 tons/ha. This method of producing algae uses a culture medium which costs nothing; it also purifies sewage economically. (12 references.)
P. W. B. Harrison.

4.—APPARATUS AND UNCLASSIFIED

Composition studies on tobacco. XX. Bases of cigarette smoke. I. Schmeltz, R. L. Stedman, W. J. Chamberlain and D. Burdick (*J. Sci. Fd Agric.*, 1964, **15**, 774—781).—A method was developed for studying the entire basic fraction by gas chromatography, the major constituents found being: pyridine, isomeric picolines, 3-vinylpyridine, nicotine, nornicotine, myosmine and 2,3′-bipyridyl and evidence for the presence of isomeric lutidines, pyrrole, nicotinonitrile, 3-ethylpyridine, methyl 3-pyridyl ketone, metanicotine and anabasine. Differences in levels of the major bases were found in smoke from blended or unblended flue-cured, burley, Maryland and Turkish cigarettes. Levels of certain bases in cigarette smoke condensates (from flue-cured tobacco) having different organoleptic properties were determined; relationships between smoke flavour and chemical composition were discussed. (34 references.)
E. M. J.

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