

CONTENTS

	PAGE
PREFACE TO THE SECOND EDITION	vii
PREFACE TO THE FIRST EDITION	ix
INTRODUCTION	xix
Definitions and scope of quantitative pharmaceutical chemistry-- References.	
PART I	
General Methods Used in Official Pharmaceutical Analyses	
CHAPTER I	
REMARKS AND GENERAL DIRECTIONS	3
Sampling--Calculation of results and errors--General operations-- The analytical balance--Weights.	
CHAPTER II	
GRAVIMETRIC ANALYSIS	29
Theory of ionization--Reversible reactions--Solubility product principle--Common ion effect.	
CHAPTER III	
GRAVIMETRIC METHODS	37
Assay of sodium chloride, of sodium sulfate, of mercuric chloride, of calcium glycerophosphate, of alum, and of solution of magnesium citrate.	
CHAPTER IV	
PRINCIPLES OF VOLUMETRIC (TITRIMETRIC) ANALYSIS.	57
Definitions--Volumetric apparatus--The calibration of volumetric apparatus--Neutralization methods: Theory--Indicators-- Standard solutions--Preparation and standardization of normal hydrochloric acid, of normal sodium hydroxide, and of tenth- normal barium hydroxide.	
CHAPTER V	
ALKALIMETRY	86
Direct titration methods: Assay of sodium bicarbonate, of sodium hydroxide, and of sodium salicylate.	

Residual titration methods: Assay of zinc oxide, of potassium and sodium tartrate, of magnesium magna, of methenamine, of solution of ammonium acetate, and estimation of nitrogen by the Kjeldahl method.

CHAPTER VI

ACIDIMETRY 105

Direct titration methods: Assay of diluted sulfuric acid, of boric acid, of tablets of sodium salicylate, and of tartaric acid.

Residual titration methods: Assay of aromatic sulfuric acid and of tablets of acetylsalicylic acid.

CHAPTER VII

PRECIPITATION METHODS 116

Determination of the end point--Indicators. Standard solutions. Preparation and standardization of tenth-normal silver nitrate and of tenth-normal ammonium thiocyanate.

Direct titration methods: Assay of strong silver protein.

Residual titration methods: Assay of sodium chloride, of ammonium bromide, of syrup of hydriodic acid, and of elixir of three bromides.

CHAPTER VIII

OXIDATION-REDUCTION METHODS 129

Theory--Standard solutions--Preparation and standardization of tenth-normal potassium permanganate.

Direct titration methods: Assay of ferrous sulfate, of reduced iron, and of solution of hydrogen peroxide.

Indirect titration methods: Assay of calcium gluconate.

Residual titration methods: Preparation and standardization of tenth-normal oxalic acid--Assay of sodium nitrite, of precipitated calcium carbonate, and of potassium chlorate.

Dichromate methods: Preparation of tenth-normal potassium dichromate--Assay of mass of ferrous carbonate.

CHAPTER IX

OXIDATION AND REDUCTION--IODOMETRIC METHODS 155

Starch indicator solutions. Standard solutions: Preparation and standardization of tenth-normal sodium thiosulfate solution and of tenth-normal iodine.

Direct titration with standard iodine solution: Assay of arsenic trioxide.

Direct titration with sodium thiosulfate: Assay of compound solution of iodine.

Residual titration with standard sodium thiosulfate: Assay of mercurous chloride.

Titration of the iodine liberated from potassium iodide with sodium thiosulfate: Assay of solution of ferric chloride, of chlorinated lime, of cupric sulfate, of sodium arsenate, of thyroid, and of spirit of ethyl nitrite.

Titration with tenth-normal bromine: Preparation and standardization of tenth-normal bromine: Assay of phenol and of ammonium hypophosphite.

Titrations with standard potassium iodate: Preparation of standard potassium iodate solution--Assay of potassium iodide.

CHAPTER X

GAROMETRIC METHODS 188

Theory--Apparatus--Test of the nitrometer--Assay of carbon dioxide and of spirit of ethyl nitrite.

PART II

Physical Methods Used in Official Pharmaceutical Analyses

CHAPTER XI

SOLUBILITY 203

Definitions.

Determination of the solubility of boric acid in water at 25°C.

CHAPTER XII

SPECIFIC GRAVITY AND DENSITY 207

Methods used to determine the specific gravity of liquids: The use of pycnometers--Determination of the alcohol content of an official preparation. The use of the Westphal balance--Determination of the specific gravity of a volatile oil. The use of hydrometers.

Methods used to determine the specific gravity of solids: By weighing in water--Determination of the specific gravity of camphor: By the flotation method--Determination of the specific gravity of yellow wax.

CHAPTER XIII

MELTING, CONGEALING, AND BOILING POINTS. 226

Melting point: Determination of the melting point of salicylic acid.

Congeeing point: Method of determining--Determination of the solidification temperature of the fatty acids of cottonseed oil.

Boiling and distilling point: Determination of the boiling point of carbon tetrachloride.

CHAPTER XIV

REFRACTOMETRIC MEASUREMENTS	240
Refractive index: Refractometers—The Abbé refractometer.	
Determination of the refractive index of oil of orange.	

CHAPTER XV

ROTATORY POWER	247
Definitions—Polarimeters.	
Determination of the specific rotation of sucrose.	

CHAPTER XVI

VISCOSITY MEASUREMENTS	257
Definitions—Apparatus: The Saybolt viscosimeter.	
Determination of the kinematic viscosity of liquid petrolatum.	

CHAPTER XVII

PHOTOMETRIC METHODS OF ANALYSIS	260
Colorimetry: Determination of the ammonia content of water—	
Determination of the amount of epinephrine hydrochloride in	
solution of epinephrine hydrochloride—Assay of crocus for color.	
Nephelometry: Determination of the amount of arsenic trioxide in	
solution of arsenous acid—Determination of the amount of oil of	
peppermint in spirit of peppermint and limit test for chloride and	
sulfate in calcium gluconate.	

CHAPTER XVIII

DETERMINATION OF HYDROGEN ION CONCENTRATION	272
Acid base equilibrium and pH.	
Potentiometric methods: The hydrogen electrode—Platinization	
of the hydrogen electrode—The calomel electrode—The scheme	
of assembly for hydrogen ion methods.	
Determination of the end point of titration of hydrochloric acid	
with sodium hydroxide potentiometrically—Notes and pre-	
cautions—Determination of the end point of titration of acetic	
acid with sodium hydroxide potentiometrically. Determination	
of the pH of saturated boric acid solution, and of physiological	
salt solution.	
The quinhydrone electrode: Preparation—Use of.	
Determination of the pH of elixir of iron, quinine and strychnine,	
elixir of pepsin, and tincture of aconite by means of the quin-	
hydrone electrode.	
The glass electrode.	
Colorimetric methods: Indicators—Buffer solutions—Color stand-	
ards—Color comparators.	
Determination of the pH of solution of epinephrine hydrochloride,	
and syrup of hydriodic acid.	

CHAPTER XIX

ELECTROLYTIC METHODS. 303

Electrical units and fundamental laws—Theory—Apparatus.
 Assay of copper sulfate and of mercuric chloride. Other electrolytic assays.

PART III

Special Methods Used in Official Pharmaceutical Analyses

CHAPTER XX

ASH AND MOISTURE DETERMINATIONS. 323

Ash content: Determination of the total and acid-insoluble ash content of digitalis leaf.

Moisture content: Determination of the moisture content of scacia—Determination of the moisture content of digitalis leaf by the toluene distillation method.

CHAPTER XXI

EXTRACTIVE AND CRUDE FIBER CONTENT 336

Volatile and non-volatile ether-soluble extractive: Determination of the volatile and non-volatile ether-soluble extractive of clove.

Alcohol-soluble extractive: Assay of benzoin.

Water-soluble extractive: Assay of aloe.

Purified petroleum benzoin extractive.

Crude fiber: Determination of the crude fiber content of cloves.

CHAPTER XXII

CONSTANTS OF FATS, FATTY OILS, WAXES, BALSAMS, RESINS, ETC. . . . 345

Acid number: Determination of the acid value of rosin.

Saponification value: Determination of the saponification value of cottonseed oil.

Ester number.

Unsaponifiable matter.

Iodine value: Determination of the iodine value of olive oil.

CHAPTER XXIII

ASSAY OF VOLATILE OILS 361

Methods of general application: Specific gravity—Rotatory power—Refractive index—Congealing point—Distilling point—Fractional distillation—Solubility.

Assay for ester content: Preparation of half-normal alcoholic potassium hydroxide—Assay of oil of peppermint for total esters.

Assay for alcohol content: Assay of oil of peppermint for total menthol.

Assay for aldehyde content: Assay of oil of bitter almond for benzaldehyde content.

- Assay for ketone content: Assay of oil of caraway.
 Assay for phenol content: Assay of oil of clove.
 Assay for hydrocyanic acid content: Assay of oil of bitter almond
 for hydrocyanic acid.
 Assay for ascaridol content: Assay of oil of chenopodium.
 Assay for allyl isothiocyanate: Assay of oil of mustard.
 Assay for volatile oil in spirits: Assay of spirit of peppermint.

CHAPTER XXIV

ALKALOIDAL ASSAYING. 380

- General principles: Sources of error—Theory of distribution
 coefficient—Choice of indicators—Test solutions.
 General procedures: Selection of the sample—Extraction with
 immiscible solvents—Evaporation of organic solvents—Gravi-
 metric determination of alkaloids—Volumetric determination of
 alkaloids.

CHAPTER XXV

OFFICIAL TYPE METHODS 403

- Alkaloidal assays by aliquot part method.
 General procedure: Extraction of the drug—Decanting the aliquot
 portion—Shaking out with acid—Shaking out with immiscible
 solvent—Determination of the alkaloidal content.
 Gravimetric assays: Assay of hydrastis for ether-soluble alka-
 loids, of cinchona for total alkaloids, and of compound tincture of
 cinchona.
 Volumetric assays: Assay of ipecac for ether-soluble alkaloids and
 assay of arca.
 Alkaloidal assays by the total extraction method: Assay of hyo-
 scyamus leaves.
 Assay of preparations of hyoscyamus, belladonna, and stramonium:
 Assay of tincture of belladonna and fluidextract of belladonna
 leaf.

CHAPTER XXVI

ALKALOIDAL ASSAYS BY SPECIAL METHODS. 418

- Assay of opium.
 Table of official substances assayed by the same method as opium.
 Assay of colchicum.
 Table of substances assayed by the same method as colchicum.
 Assay of nux vomica.
 Assay of caffeine containing drugs: Assay of guarana.
 Table of official drugs and preparations assayed for caffeine.
 Assay of alkaloidal salts: Assay of citrated caffeine, of eucaine
 hydrochloride, of theobromine with sodium salicylate, and of
 theophylline with sodium acetate.

CONTENTS

xvii

PAGE

CHAPTER XXVII

OTHER OFFICIAL ASSAYS INVOLVING THE USE OF IMMISCIBLE SOLVENTS	437
Assay of cantharides—Assay of aspidium—Assay of jalap—Assay of tablets of phenobarbital.	

CHAPTER XXVIII

ASSAY OF ENZYME-CONTAINING SUBSTANCES	444
Assay of pepsin—Assay of pancreatin for starch digestive power—Assay of pancreatin for casein digestive power—Assay of rennin.	
TABLE OF LOGARITHMS AND ANTILOGARITHMS	453
INDEX	457
TABLE OF ATOMIC WEIGHTS	Inside back cover