

METHODS OF ORGANIC ANALYSIS

Published @ 2017 Trieste Publishing Pty Ltd

ISBN 9780649648245

Methods of Organic Analysis by Henry C. Sherman

Except for use in any review, the reproduction or utilisation of this work in whole or in part in any form by any electronic, mechanical or other means, now known or hereafter invented, including xerography, photocopying and recording, or in any information storage or retrieval system, is forbidden without the permission of the publisher, Trieste Publishing Pty Ltd, PO Box 1576 Collingwood, Victoria 3066 Australia.

All rights reserved.

Edited by Trieste Publishing Pty Ltd.
Cover @ 2017

This book is sold subject to the condition that it shall not, by way of trade or otherwise, be lent, re-sold, hired out, or otherwise circulated without the publisher's prior consent in any form or binding or cover other than that in which it is published and without a similar condition including this condition being imposed on the subsequent purchaser.

www.triestepublishing.com

HENRY C. SHERMAN

**METHODS OF
ORGANIC
ANALYSIS**

METHODS OF ORGANIC ANALYSIS

BY

HENRY C. SHERMAN, PH.D.

ADJUNCT PROFESSOR OF ANALYTICAL CHEMISTRY
IN
COLUMBIA UNIVERSITY



New York

THE MACMILLAN COMPANY
LONDON: MACMILLAN & CO., LTD.

1905

All rights reserved

GENERAL

Copyright 1905

By HENRY C. SHERMAN

PRINTED BY
THE NEW ENGLAND PRINTING COMPANY
LANCASTER, PA.

PREFACE.

The purpose of this work is to give a connected introductory training in organic analysis, especially as applied to plant and animal substances and their manufactured products. No attempt is made to touch upon all important branches of this subject but representative topics are treated in considerable detail with reference both to analytical methods and to the interpretation of results.

The greater part of the book is devoted to quantitative methods for food materials and related substances. Standard works of reference and the publications of the Association of Official Agricultural Chemists have been freely used. The nomenclature adopted in these publications has been followed as closely as possible. As a rule, footnotes show the original sources of statements or methods included in the text, while general or additional references are given at the end of each chapter. The references have been carefully selected and are believed to be sufficient to put the reader in touch with the most important literature.

The descriptions of methods were written primarily for the use of third-year students in the School of Chemistry, Columbia University, and therefore presuppose a knowledge of inorganic quantitative analysis, elementary organic chemistry, and general physics.

The writer takes pleasure in acknowledging his indebtedness to Professor Edmund H. Miller for helpful advice and suggestions throughout the work, and to Mr. Roland H. Williams for assistance in testing methods and in the revision of parts of the manuscript.

H. C. S.

NEW YORK, July 1, 1905.



TABLE OF CONTENTS.

CHAPTER I.

| | |
|---|---|
| INTRODUCTION. | |
| Ultimate and proximate analysis..... | 1 |
| Preliminary treatment of samples..... | 1 |
| Outline of ultimate organic analysis..... | 2 |
| Preparation and analysis of ash..... | 4 |

CHAPTER II.

| | |
|--|----|
| NITROGEN, SULPHUR, AND PHOSPHORUS. | |
| Determination of nitrogen..... | 8 |
| Kjeldahl method..... | 8 |
| Gunning-Arnold-Dyer modification..... | 10 |
| Method for nitrates and nitro-compounds..... | 13 |
| Determination of sulphur..... | 14 |
| Comparative outline of methods..... | 14 |
| Leibig's alkali method..... | 16 |
| Osborne's peroxide method..... | 18 |
| Berthelot's oxygen method..... | 19 |
| Determination of phosphorus..... | 20 |
| Alkali methods..... | 20 |
| Neumann's acid method..... | 21 |

CHAPTER III.

| | | |
|---|--|----|
| ALCOHOLS..... | | 23 |
| Ethyl alcohol..... | | 24 |
| Detection and identification..... | | 25 |
| Determination by the specific gravity method..... | | 26 |
| Determination by the boiling point method..... | | 32 |
| Determination by oxidation..... | | 33 |
| Detection and determination of homologous alcohols..... | | 33 |
| Methods of stating strength of alcohol solutions..... | | 35 |
| Glycerol..... | | 36 |
| Determination by oxidation..... | | 37 |
| Determination by acetylation..... | | 39 |
| Determination by separation and weighing..... | | 40 |
| Examination of commercial glycerol..... | | 42 |

CHAPTER IV.

| | | |
|-----------------------------------|--|----|
| ALDEHYDES..... | | 46 |
| Formaldehyde..... | | 48 |
| Detection and identification..... | | 50 |

| | |
|---|----|
| Determination by oxidation..... | 52 |
| Determination by condensation reactions | 54 |
| Determination by addition reactions | 55 |
| Additional references..... | 57 |

CHAPTER V.

CARBOHYDRATES—GENERAL METHODS.

| | |
|--|----|
| Occurrence and relations..... | 58 |
| Solubilities..... | 61 |
| Reactions with phenylhydrazine..... | 63 |
| Preparation and properties of the osazones..... | 64 |
| Reduction of copper solutions..... | 67 |
| Fehling's volumetric method..... | 69 |
| Defren's gravimetric method..... | 72 |
| Kjeldahl's gravimetric method..... | 74 |
| Barfoed's cupric acetate method..... | 75 |
| Reactions with acids..... | 76 |
| Molisch's α -naphthol reaction..... | 76 |
| Furfural reaction of pentoses and pentosans..... | 77 |
| Levulinic acid reaction of hexoses..... | 78 |
| Oxidation by nitric acid..... | 79 |
| Hydrolysis by dilute acids..... | 80 |
| Rotation of polarized light..... | 81 |
| Measure of rotating power—Specific rotation..... | 81 |
| Preparation of solutions for polarization..... | 83 |
| Determination of angular rotation | 84 |
| Reference books..... | 85 |

CHAPTER VI.

CARBOHYDRATES—SPECIAL METHODS.

| | |
|---|----|
| Analysis of raw sugar..... | 86 |
| Polariscopic examination..... | 86 |
| Clerget's method for sucrose..... | 91 |
| Determination of reducing sugars..... | 92 |
| Determination of moisture and ash | 93 |
| Official methods and standards of purity..... | 93 |
| Determination of sucrose in beets and cane..... | 94 |
| Commercial glucose..... | 96 |
| Official definitions and standards of purity..... | 96 |
| Analysis by Wiley's method..... | 97 |
| Analysis by other methods..... | 98 |

CHAPTER VII.

CARBOHYDRATES—SPECIAL METHODS (*Continued*).

| | |
|------------------------------|-----|
| Determination of starch..... | 100 |
|------------------------------|-----|

CONTENTS.

ix

| | |
|--|-----|
| Method of direct acid hydrolysis..... | 100 |
| Method of digestion with diastase or saliva..... | 103 |
| Comparison of results..... | 105 |
| Determination of starch in meat products..... | 105 |
| Additional references..... | 106 |
| Separation of carbohydrates in cereal products..... | 107 |
| Determination of reducing sugars, sucrose, dextrin, starch, pentosans, and cellulose..... | 107 |
| Determination of maltose, dextrin, and starch in malted cereal | 108 |
| References to other special methods..... | 109 |
| Substances rich in sucrose or invert sugar..... | 109 |
| Artificial mixtures containing lactose..... | 110 |
| Animal tissues and fluids other than milk..... | 111 |

CHAPTER VIII.

| | |
|--|-----|
| ACIDS..... | 112 |
| Acetic acid and acetates..... | 112 |
| Determination of acetic acid in calcium acetate..... | 112 |
| Separation of acetic acid from its homologues..... | 114 |
| Vinegar..... | 115 |
| Determination of constituents..... | 116 |
| Determination of source..... | 117 |
| Official standards..... | 118 |
| References..... | 118 |
| Fatty acids..... | 119 |
| Acids of the stearic series..... | 119 |
| Acids of the oleic series..... | 120 |
| Acids of the linoleic series..... | 122 |
| Acids of the linolenic series..... | 122 |
| Hydroxy-acids..... | 122 |
| Separation of fatty acids..... | 123 |

CHAPTER IX.

| | |
|---|-----|
| OILS, FATS, AND WAXES — GENERAL METHODS. | |
| Properties of fats and fatty oils..... | 125 |
| Analytical methods..... | 126 |
| Saponification or Koettstorfer number..... | 127 |
| Hehner number..... | 130 |
| Reichert-Meissl number..... | 130 |
| Iodine or Hübl number..... | 130 |
| Maumené number — Specific temperature reaction..... | 136 |
| Acetyl number..... | 138 |
| Specific gravity..... | 140 |
| Index of refraction..... | 141 |
| Melting and solidifying points — Titer test..... | 142 |