

J. E. PURVIS

AND T. R. HODGSON

The Chemical
Examination of Water,
Sewage and Foods and
Other Substances

THE CHEMICAL EXAMINATION OF WATER, SEWAGE, FOODS AND OTHER SUBSTANCES

BY

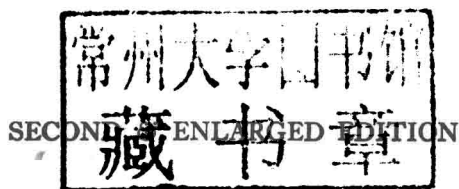
J. E. PURVIS, M.A.

University Lecturer in Chemistry and Physics as applied
to Hygiene and Public Health
St John's and Corpus Christi Colleges, Cambridge

AND

T. R. HODGSON, M.A.

Public Analyst for the County Boroughs of Blackpool and Wallasey
and the Municipal Boroughs of Bacup and Stalybridge
Formerly of Christ's College, Cambridge



CAMBRIDGE
AT THE UNIVERSITY PRESS

1922

CAMBRIDGE UNIVERSITY PRESS

University Printing House, Cambridge CB2 8BS, United Kingdom

Cambridge University Press is part of the University of Cambridge.

It furthers the University's mission by disseminating knowledge in the pursuit of education, learning and research at the highest international levels of excellence.

www.cambridge.org

Information on this title: www.cambridge.org/9781107494732

© Cambridge University Press 1922

This publication is in copyright. Subject to statutory exception and to the provisions of relevant collective licensing agreements, no reproduction of any part may take place without the written permission of Cambridge University Press.

First edition 1914

Second edition 1922

First published 1922

Re-issued 2015

A catalogue record for this publication is available from the British Library

ISBN 978-1-107-49473-2 Paperback

Cambridge University Press has no responsibility for the persistence or accuracy of URLs for external or third-party internet websites referred to in this publication, and does not guarantee that any content on such websites is, or will remain, accurate or appropriate.

CAMBRIDGE PUBLIC HEALTH SERIES

UNDER THE EDITORSHIP OF

G. S. GRAHAM-SMITH, M.D., F.R.S., *University Lecturer in Hygiene and Secretary to the Sub-Syndicate for Tropical Medicine*

AND

J. E. PURVIS, M.A. *University Lecturer in Chemistry and Physics in their application to Hygiene and Preventive Medicine, and Secretary to the State Medicine Syndicate*

THE CHEMICAL EXAMINATION OF WATER, SEWAGE, FOODS AND OTHER SUBSTANCES

EDITORS' PREFACE

IN view of the increasing importance of the study of public hygiene and the recognition by doctors, teachers, administrators and members of Public Health and Hygiene Committees alike that the *salus populi* must rest, in part at least, upon a scientific basis, the Syndics of the Cambridge University Press have decided to publish a series of volumes dealing with the various subjects connected with Public Health.

The books included in the Series present in a useful and handy form the knowledge now available in many branches of the subject. They are written by experts, and the authors are occupied, or have been occupied, either in investigations connected with the various themes or in their application and administration. They include the latest scientific and practical information offered in a manner which is not too technical. The bibliographies contain references to the literature of each subject which will ensure their utility to the specialist.

It has been the desire of the editors to arrange that the books should appeal to various classes of readers: and it is hoped that they will be useful to the medical profession at home and abroad, to bacteriologists and laboratory students, to municipal engineers and architects, to medical officers of health and sanitary inspectors and to teachers and administrators.

Many of the volumes will contain material which will be suggestive and instructive to members of Public Health and Hygiene Committees; and it is intended that they shall seek to influence the large body of educated and intelligent public opinion interested in the problems of public health.

AUTHORS' PREFACE TO FIRST EDITION

THIS book is intended for the use of students who are attending courses of instruction for diplomas and degrees in Public Health, and for those who are studying the chemistry of water, sewage, sewage effluents, foods, disinfectants, etc. during their ordinary laboratory practice. It will probably be also useful to those who are engaged as public analysts or in public health matters generally.

It does not give an exhaustive account of all the available methods of examination, but it describes those which have been used and tested by the authors both in laboratory instruction and in the daily work of a public analyst. Full references are given in the text to the sources of the different methods, and, at the end, there is a short bibliography of some of the more important works which discuss the various subjects. No illustrations have been introduced, as the apparatus, as well as the appearance and characters of the substances which require the use of a microscope, can only be fully understood and appreciated in the actual practical work of the laboratory.

A considerable number of typical analyses have been introduced to illustrate the variations which may occur in the composition of the articles themselves. We desire to convey our grateful thanks to those who have allowed us to insert the results of their own work and which we have acknowledged by name in the appropriate places.

The methods employed in the bacteriological examination of water, sewage, foods, air, etc. is the subject of a separate volume in this series by Dr W. G. Savage.

J. E. P.
T. R. H.

CAMBRIDGE,
July, 1914.

PREFACE TO THE SECOND EDITION

THE success of the first edition has given an opportunity to the authors to make the book still more useful to students. They have included in this edition a considerable amount of fresh matter and newer methods of analysis. The chapters on water and milk have been expanded, and a more detailed account is described of the methods of analysis of foods and beverages. A chapter has been added containing an outline of elementary toxicological analysis.

The helpful criticisms by reviewers of the first edition have been carefully considered and their suggestions have usually been incorporated and developed in this enlarged edition.

J. E. P.

T. R. H.

CAMBRIDGE,

January, 1922.

CONTENTS

CHAP.	PAGE
I. WATER—SEWAGE—SEWAGE EFFLUENTS—RIVER-WATER AND SEWAGE—SEA-WATER AND SEWAGE—WORKS EFFLUENTS	1
II. MILK—CREAM—DRIED MILK—CONDENSED MILK . . .	100
III. BUTTER—MARGARINE—LARD, DRIPPING, SUET—CHEESE, CREAM CHEESE—EDIBLE OILS	141
IV. TEA—COFFEE—CHICORY—COCOA	176
V. WHEAT FLOUR, SELF-RAISING FLOUR, BAKING POWDER —BREAD—RICE—PEARL BARLEY—STARCHES—IN- FANTS' FOODS	194
VI. PEPPER, CAYENNE, MUSTARD—NUTMEG, CINNAMON, GINGER	213
VII. CANE SUGAR—GOLDEN SYRUP AND TREACLE—HONEY— JAM	220
VIII. ALCOHOLIC BEVERAGES—WHISKEY, RUM, GIN, BRANDY— BEER—CIDER	228
IX. VINEGAR—LIME AND LEMON JUICE	238
X. MEAT AND MEAT PRODUCTS	245
XI. POISONOUS METALS IN FOODS	252
XII. PRESERVATIVES—DISINFECTANTS	260
XIII. AIR—COAL GAS—OTHER GASES	273
XIV. RAG FLOCK—URINE	291
XV. TOXICOLOGY	302
APPENDIX	314
BIBLIOGRAPHY	315
ADDENDA. TABLE OF ATOMIC WEIGHTS. ALCOHOL TABLE. VOLUMES OF OXYGEN AND NITROGEN ABSORBED FROM THE ATMOSPHERE BY DISTILLED WATER AND SEA- WATER. MILK TABLES. CONVERSION OF ALCOHOLIC STRENGTH. SPECIFIC GRAVITY OF ALCOHOL. FACTORS FOR USE IN VOLUMETRIC ANALYSIS. TABLE OF SOME USEFUL CONSTANTS	
INDEX	327

CHAPTER I

WATER—SEWAGE—SEWAGE EFFLUENTS—RIVER- WATER AND SEWAGE—SEA-WATER AND SEWAGE —WORKS EFFLUENTS

WATER

CHEMICALLY pure water does not occur in nature. All water supplies are obtained ultimately from rain water, which contains various substances, both organic and inorganic, dissolved by the water during its passage through the air, soil, subsoil, etc.; and, it may be, by contamination with sewage material and trade wastes.

An analysis of water is undertaken with the object of estimating these impurities and of deciding whether the water is of sufficient purity to be used for domestic purposes. The value of this analysis depends on the accuracy of the results, and, in a large measure, on careful and proper sampling.

Method of sampling. The sample should be collected in a "Winchester Quart," having a ground glass stopper. Stone jars should not be used, and care should be taken to see that the bottle has not been previously used for storing ammonia and ammonium salts. Before collecting the sample, the bottle should be well rinsed at least three times with the water.

If the sample is to be taken from a stream, the bottle should be completely immersed in the stream, as near the centre as possible, and care should be taken not to disturb any mud.

If the sample is to be taken from a well, at least ten gallons should be allowed to run to waste before filling the bottle, and the water should then be allowed to flow directly into the bottle; in no case should a funnel be used.

When the sample is to be collected from a tap, at least ten gallons should be allowed to run to waste; and the sample should then be collected directly from the tap.

When a sample of water is submitted for analysis, the largest possible amount of information regarding it should be forwarded at the same time. In order to assist the analyst in

arriving at his opinion, information on the following points is essential and should always be supplied: (a) risk of pollution; (b) nature of the surface and subsoil, *e.g.* clay, chalk, gravel, sand, etc.; (c) if the water comes from a well the approximate depth of the well.

Physical characters. A careful preliminary examination of a water is of great importance.

(a) Test the water with blue and red litmus papers, and notice if the reaction is acid or alkaline. Most drinking waters are slightly alkaline or neutral in reaction. Peaty waters and waters from upland surfaces are often acid, as well as waters from chemical works and breweries. Waters which are contaminated with sewage are usually alkaline. Waters which contain carbon dioxide give a slightly acid reaction.

(b) Warm a portion in a test-tube and notice if any smell is produced. Polluted water may give off sulphuretted hydrogen.

(c) Place a portion in a two-foot glass tube and examine it downwards in a good light. Notice if it is clear and bright or turbid. Pure water has a bluish tinge; polluted waters may be yellow or brown; peaty waters and upland surface waters are usually coloured yellow or brown.

(d) Allow a portion of the water to stand for an hour or two in a conical shaped tube, and collect the sediment. Mount a little of the sediment on a glass slide, and examine it carefully with a microscope to see if it is mineral matter, such as sand or chalk or oxide of iron; or vegetable matter, such as vegetable cells, starch granules, cotton or linen or silk fibre, yeasts, moulds, bacteria, diatoms and other water organisms; or animal refuse such as worms, insects, hairs, fish scales, animal fibre, etc.

(e) Also notice if it is well aerated, by shaking a portion of it in a glass bottle and noticing the liberation of gas bubbles.

Free and saline ammonia

Solutions required:

Nessler's solution. Weigh out

35 grams of potassium iodide,
13 grams of mercuric chloride,
120 grams of caustic potash,

dissolve each separately in water, and gradually add the potassium iodide solution to the solution of mercuric chloride, a precipitate of mercuric iodide appears at first which is afterwards redissolved in the excess of potassium iodide; the caustic potash solution is then added gradually; and, afterwards, a saturated solution of mercuric chloride drop by drop, until a very slight precipitate, which will not redissolve, is formed. The whole is then finally made up to 1000 c.c. and allowed to stand until the precipitate settles; the solution should always be kept in contact with the precipitate.

Ammonium chloride solution (strong): weigh out 3.147 grams of pure dry ammonium chloride, dissolve it in water and make up to 1000 c.c.

Ammonium chloride solution (dilute): measure out 10 c.c. of the strong ammonium chloride solution and add ammonia-free distilled water to 1000 c.c. 1 c.c. of this solution = 0.00001 gram of ammonia.

Estimation of free and saline ammonia. The most convenient apparatus for this determination is a 1500 c.c. round-bottomed distilling flask, with a ground glass stopper, having a fused-in delivery tube, bent at right angles, this delivery tube is fitted into a double surface condenser.

It is necessary first to free the apparatus from ammonia; and, in order to do this, 200 c.c. of distilled water and about one gram of solid sodium carbonate, which has been fused in a platinum dish to drive off any ammonium salts, are placed in the distilling flask and boiled; the distillate is collected in a Nessler cylinder, and the boiling continued until the distillate gives no colour with 2 c.c. of the Nessler solution. As soon as the apparatus has been freed from ammonia, 500 c.c. of the sample are added to the contents of the distilling flask and the boiling continued; the distillate being collected in Nessler cylinders, 50 c.c. at a time; to the contents of each cylinder, 2 c.c. of the Nessler solution are added and the colour obtained is compared with the standard colours.

Standard colours are obtained by placing known quantities of the dilute ammonium chloride solution (e.g. 0.5 c.c., 1.0 c.c., 1.5 c.c., etc.) in a Nessler cylinder, filling up to the mark with distilled water free from ammonia, and adding 2 c.c. of the

Nessler solution. In some cases it may be found that the colour obtained with the first glass of the distillate is too deep to be compared with a standard solution, the contents of the glass are therefore diluted to 250 c.c. and then compared with the standard; or an aliquot portion may be taken and diluted with ammonia-free water to 50 c.c. and then compared with the standard. The amount of ammonia obtained in the distillation is calculated from the known strength of the dilute ammonia solution.

In this process the sodium carbonate decomposes any ammonium salts in the water, and the ammonia so obtained, together with any free ammonia in the water, passes over and is collected in the distillate. Ammonia is one of the first products of the decomposition of animal matter and its presence in water in very large quantities must be looked upon with suspicion; but it must be remembered that ammonium salts are not in themselves harmful and their presence in water does not necessarily render the sample unfit for drinking purposes.

Albuminoid ammonia

Solution required:

Alkaline permanganate solution. Weigh out

8 grams of potassium permanganate,
200 grams of caustic potash,

dissolve these together in about 1200 c.c. of distilled water and evaporate the solution to about 900 c.c., add 200 c.c. of distilled water and again evaporate the solution to 1000 c.c.; the solution, which will be found to be free from ammonia, is kept in a glass stoppered bottle.

Estimation of albuminoid ammonia. To the remainder of the water in the distilling flask, after the estimation of free and saline ammonia, add 50 c.c. of the alkaline permanganate solution, which has been previously heated to boiling, and continue the boiling; the ammonia is estimated colorimetrically as before.

The nitrogen in organic combination (as urea, uric acid, indol, skatol, and other proteid substances) is oxidized by the action of the alkaline solution of permanganate to ammonia.

The amount of ammonia obtained is, therefore, a measure of the organically combined nitrogen present in the sample.

Estimation of total solid matter

Measure out 100 c.c. of the sample into a weighed platinum dish, and evaporate it to dryness on a water bath; then place the dish in a water oven at 100°C . for three hours; cool in a desiccator and weigh; the increase in weight represents the total solid matter in 100 c.c. of the water. Each mgm (= 0.001 of a gram) of solid matter represents one part per 100,000.

Chlorine as chlorides

Solutions required:

Silver nitrate solution. Weigh out 2.4 grams of pure dry silver nitrate, and dissolve it in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0.0005 gram of chlorine; or when working on 50 c.c. 1 c.c. = 1 part of chlorine per 100,000.

Potassium chromate solution. Weigh out 10 grams of potassium chromate, free from chloride, and dissolve it in 100 c.c. of distilled water.

Estimation of chlorine as chlorides. Place 50 c.c. of water in a porcelain dish; add three drops of the potassium chromate solution, and run in the silver nitrate solution from a burette, with constant stirring, until a slight but permanent red brown colour is obtained; then read off the number of c.c. of silver nitrate solution used. The silver nitrate precipitates the chlorine present in the water, as silver chloride; and, as soon as all the chloride is precipitated, a reddish insoluble compound of silver chromate is formed.

Oxygen absorbed from potassium permanganate solution

(1) Solutions required:

Potassium permanganate solution. Weigh out 0.395 gram of pure potassium permanganate; dissolve it in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0.0001 gram of available oxygen.

Oxalic acid solution. Weigh out 0.7875 gram of crystallized

oxalic acid; dissolve it in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 1 c.c. of the permanganate solution.

Sulphuric acid solution. Measure out 33 c.c. of pure concentrated sulphuric acid, and add it gradually, with constant stirring, to 66 c.c. of distilled water; allow it to cool; to the cold solution add, drop by drop, potassium permanganate solution until a faint pink colour remains after keeping the solution at 80° F. for four hours.

Estimation of the oxygen absorbed. Into each of two conical flasks, of about 800 c.c. capacity, pour a small quantity of concentrated sulphuric acid and thoroughly rinse, taking care that the sulphuric acid comes in contact with the whole of the inside of the flask; wash out the flasks thoroughly first with tap water and afterwards with distilled water.

Into one flask measure out 250 c.c. of the sample; into the other flask 250 c.c. of distilled water; and to each add 10 c.c. of the potassium permanganate solution from a pipette, and 10 c.c. of the sulphuric acid solution; raise the temperature to 80° F. and maintain it at this temperature for four hours; if, at any time, the pink colour disappears a further quantity of 10 c.c. of the potassium permanganate solution must be added. At the end of four hours titrate the solution in each flask with the oxalic acid solution; the difference in the two titrations multiplied by 0.0001 will give the amount of oxygen absorbed by 250 c.c. of the sample.

(2) *Potassium iodide and sodium thiosulphate method.*

Solutions required:

Standard solution of potassium permanganate.	} prepared as described above.
Dilute sulphuric acid solution.	

Potassium iodide solution; 10 grams of KI are dissolved in 1000 c.c. of water.

Standard sodium thiosulphate solution; 1 gram of pure recrystallized sodium thiosulphate is dissolved in 1000 c.c. of water.

Starch solution; 1 gram of pure potato starch is mixed into a smooth paste with a small quantity of water; and 200 c.c. of boiling water are then carefully added with constant stirring and the solution allowed to cool.

The process. The actual determination is carried out exactly as described above, except that after standing for four hours at 80° F. a few drops of the potassium iodide solution are added to each flask, when a brown solution will be obtained owing to the reaction of the potassium iodide with the excess of potassium permanganate and the liberation of free iodine; the standard solution of sodium thiosulphate is then run in from a burette with constant shaking until a straw yellow colour is obtained; a few drops of starch solution are then added, producing a deep blue colour, and the titration with the thiosulphate solution continued until the blue colour is discharged.

Example. A blank experiment required 34 c.c. of the thiosulphate solution, and 250 c.c. of a water required 31.3 c.c.
 $400 \times 0.001 \times \frac{34 - 31.3}{34} = 0.032$ parts of oxygen absorbed per 100,000 parts of water.

The purer natural waters, as a rule, contain only very small quantities of oxidizable organic substances of vegetable origin; this process, therefore, is a measure of the oxidizable organic substances of animal origin in the water.

With a view of coming to a provisional conclusion more rapidly than can possibly be done in all bacteriological tests, and to a certain extent with chemical tests, Sir A. C. Houston, in his 5th Report (1911) and 7th Report (1913) to the London Metropolitan Water Board, made a considerable number of detailed experiments to correlate the oxygen absorbed from potassium permanganate in *five minutes*, as compared with that absorbed in three hours, both at 80° F. He found that, taking the arbitrary figure 0.1 per 100,000 as the basis for objecting to a water in a three hours' test, the comparable figure for the five minutes' test was somewhat less than 0.038, the ratios varying slightly with different waters. When a water is appreciably worse than 0.1 in the three hours' test, the five minutes' test will be usually above 0.038; and when a water is appreciably better than 0.1 in the three hours' test, the five minutes' test may be nearly always trusted not to exceed 0.038. Dr Houston states that since April, 1910, the figure 0.038 has been adopted for the five minutes' test, and if on

the examination of a sample of water, the results reach or exceed this amount, a telephone communication is sent at once to the district concerned. The five minutes' method is of little use when the waters are very good, but when they are unsatisfactory, the parallelism between the two sets of results is very striking, although, of course, the *actual* results differ in the two cases.

Nitrogen as Nitrates

Estimation of nitrogen as nitrates. Nitrogen occurring as nitrates is determined by one of several methods:

- (a) Gasometric method.
- (b) Colorimetric method.
- (c) Ammonia method.
- (d) Indigo method.
- (e) Titanous Chloride method.

(a) *Gasometric method.* The residue from the determination of the total solid matter is dissolved in the smallest possible quantity of water, the sides of the dish being well scrubbed with a rubber-tipped glass rod; the solution is then poured into the cup of a Crum's nitrometer, filled with mercury and standing in a dish containing mercury, the tap is carefully opened and the solution allowed to run into the nitrometer; an equal volume of concentrated sulphuric acid is then poured into the cup and allowed to flow into the nitrometer in its turn, care being taken that no air is admitted; the bottom of the tube is closed with the thumb and the whole tube removed from the dish and well shaken, at an angle of 45° so that the mercury comes in contact with the mixture at the top of the tube; from time to time during the shaking the tube is replaced in the dish and the thumb removed to release the pressure; when the whole of the nitrate is decomposed the volume of gas obtained (nitric oxide) is noted, together with the temperature and pressure, and the volume is reduced to normal temperature and pressure. From the volume of the gas thus obtained the weight of nitrogen as nitrate in 100 c.c. of the water is calculated. The nitrogen present as nitrate is converted by the action of the sulphuric acid and mercury into nitric oxide (NO).