Standard Methods

For the Examination of Water and Wastewater

FOURTEENTH EDITION

Prepared and published jointly by:
AMERICAN PUBLIC HEALTH ASSOCIATION
AMERICAN WATER WORKS ASSOCIATION
WATER POLLUTION CONTROL FEDERATION

Publication Office:
American Public Health Association
1015 Eighteenth Street NW
Washington, DC 20036

25M3/76
Library of Congress Catalog Number: 55-1979 rev
International Standard Book Number: 0-87553-078-8

Library of Congress Cataloging in Publication Data

Main entry under title:

Standard Methods for the Examination of Water and Wastewater

Includes index.

1. Water – analysis.	2. Sewage – analysis	I. Title
QD142.A5	543.3	55-1979 rev

Printed and bound in the United States of America
Typography: Bru-El Graphic Inc., Springfield, VA
Set in: *Janson*
Text: John D. Lucas Co., Baltimore MD
Binding: Optic Bindery, Inc., Glen Burnie MD

Cover Design: Donya Melanson Assoc., Boston MA

PREFACE TO THE FOURTEENTH EDITION

The first edition of *Standard Methods* was published in 1905. Each subsequent edition has presented significant improvements of methodology and has enlarged its scope to include technics suitable for examination of many types of samples encountered in the assessment and control of water quality and water pollution.

A brief history of *Standard Methods* is of interest because of its contemporary relevance. A movement for "securing the adoption of more uniform and efficient methods of water analysis" led in the 1880's to the organization of a special committee of the Chemical Section of the American Association for the Advancement of Science. A report of this committee, published in 1889, was entitled: A Method, in Part, for the Sanitary Examination of Water, and for the Statement of Results, Offered for General Adoption.* Five topics were covered: (1) "free" and "albuminoid" ammonia; (2) oxygen-consuming capacity; (3) total nitrogen as nitrates and nitrites; (4) nitrogen as nitrites; and (5) statement of results.

In 1895, members of the American Public Health Association, recognizing the need for standard methods in the bacteriologic examination of water, sponsored a convention of bacteriologists to discuss the problem. As a result, an APHA committee was appointed "to draw up procedures for the study of bacteria in a uniform manner and with special references to the differentiation of species." Submitted in 1897† the procedures found wide acceptance.

In 1899, APHA appointed a Committee on Standard Methods of Water Analysis, charged with the extension of standard procedures to all methods involved in the analysis of water. The report of this committee, published in 1905, constituted the first edition of *Standard Methods* (then entitled *Standard Methods of Water Analysis*). Physical, chemical, microscopic, and bacteriologic methods of water examination were included. In its letter of transmittal, the Committee stated:

> The methods of analysis presented in this report as "Standard Methods" are believed to represent the best current practice of American water analysts, and to be generally applicable in connection with the ordinary problems of water purification, sewage disposal and sanitary investigations. Analysts working on widely different problems manifestly cannot use methods which are identical, and special problems obviously require the methods best adapted to them; but, while recognizing these facts, it yet remains true that sound progress in analytical work will advance in proportion to the general adoption of methods which are reliable, uniform and adequate.
>
> It is said by some that standard methods within the field of applied science tend to stifle investigations and that they retard true progress. If such standards are used in the proper spirit, this ought not to be so. The Committee strongly desires that every effort shall be continued to improve the techniques of water analysis and especially to compare current methods with those herein recommended, where different, so that the results obtained may be still more accurate and reliable than they are at present.

Revised and enlarged editions were published by APHA under the title *Standard Methods of Water Analysis* in 1912 (Second Edition), 1917 (Third), 1920 (Fourth), and 1923 (Fifth). In 1925, the American Water Works Association

J. Anal. Chem. 3:398 (1889).
†*Proc. Amer. Pub. Health Ass.* 23:56 (1897).

joined APHA in publishing the Sixth Edition, which had the broader title, *Standard Methods of the Examination of Water and Sewage.* Joint publication was continued in the Seventh Edition, dated 1933.

In 1935, the Water Pollution Control Federation (then the Federation of Sewage Works Associations) issued a committee report, "Standard Methods of Sewage Analysis."‡ With minor modifications, these methods were incorporated into the Eighth Edition (1936) of *Standard Methods,* which was thus the first to provide methods for the examination of "sewages, effluents, industrial wastes, grossly polluted waters, sludges, and muds." The Ninth Edition, appearing in 1946, likewise contained these methods, and in the following year the Federation became a full-fledged publishing partner. Since 1947, the work of the *Standard Methods* committees of the three associations—APHA, AWWA, and WPCF—has been coordinated by a Joint Editorial Board, on which all three are represented.

The Tenth Edition (1955) included methods specific for the examination of industrial wastewaters; this was reflected by a new title: *Standard Methods for the Examination of Water, Sewage and Industrial Wastes.* In order to describe more accurately and concisely the contents of the Eleventh Edition (1960), the title was shortened to *Standard Methods for the Examination of Water and Wastewater.* It remained unchanged for the Twelfth Edition (1965), the Thirteenth Edition (1971), and the Fourteenth Edition (1976).

The Fourteenth Edition

In the Fourteenth Edition, the separation of test methods for water from those for wastewater is discontinued. Recent developments such as advanced wastewater treatment and reuse of effluents have made the distinction obsolete. All methods for a given component or characteristic now appear under a single heading. This trend was started in the previous edition by combining the methods for industrial wastewaters with those for other polluted samples. The integration of all test methods is now complete.

To accomplish this consolidation, it was necessary to change the organization that had been in effect since 1947. In the past several editions, the major divisions were designed to correspond with the areas of principal interest of the sponsoring societies. Each society accepted primary responsibility for certain major parts of the manual, setting up subcommittees to handle individual methods. The present arrangement required much more interaction among the societies in the early stages of the work. For each test of interest to more than one society, a Joint Task Group was established, to which each interested society could appoint members. The Chair, selected by the Joint Editorial Board, was authorized to appoint additional members or advisors.

The coordination of methods is reflected in the revised numbering system. Although based on that used in the Thirteenth Edition, it has been changed to accommodate the new arrangement of the subject matter and to include two major sec-

‡*Sewage Works J.* 7:444 (1935).

tions that consist almost entirely of new material. The major divisions of the Fourteenth Edition are as follows:

Part 100—General Introduction
Part 200—Physical Examination
Part 300—Determination of Metals
Part 400—Determination of Inorganic Nonmetallic Constituents
Part 500—Determination of Organic Constituents
Part 600—Automated Laboratory Analyses
Part 700—Examination of Water and Wastewater for Radioactivity
Part 800—Bioassay Methods for Aquatic Organisms
Part 900—Microbiological Examination of Water
Part 1000—Biological Examination of Water

Changes, revisions, and improvements in the methods are too numerous for individual listing in this preface. Many changes resulted from consolidation of the former separate sections on water and contaminated samples. Many others represent advances in precision and accuracy, in sensitivity, or in applicability of previously existing technics, while still others are improved technics replacing older methods. For a number of components and characteristics, methods appear in this edition for the first time.

The Fourteenth Edition retains the General Introduction (Part 100) containing important information concerning proper execution of the procedures. The Joint Editorial Board strongly urges that every reader carefully study the General Introduction and the introductions to the individual parts. Each introduction discusses vital matters of wide application within its specific province to minimize repetition in the succeeding text. The success of the analysis may well rest on the manner in which the recommendations are adhered to as set forth in the introductions. The same precaution holds true for individual determinations. The complete discussion of each procedure, embracing selection of method, sampling and storage, general discussion, and interferences, should be read and fully understood before an analysis is undertaken.

Part 200 now includes a procedure for the determination of oxygen transfer. An added method for measurement of salinity has been adapted from the discontinued publication, *Recommended Procedures for Examination of Sea Water and Shellfish* (American Public Health Association). This method is included in Part 200, as are the reversing thermometer and thermograph methods for measuring temperature, from the same source. Section 213, Tests on Activated Sludge, presents methods for determining oxygen consumption rate and zone settling rate, neither of which has appeared in earlier editions.

In Determination of Metals (Part 300), relatively few new components appear because previous editions were quite complete in this respect. Methods applicable to a large number of metals, including atomic absorption spectrophotometry and polarography, have been consolidated in Section 301. The Fourteenth Edition includes the flameless atomic absorption method for mercury, which has not appeared previously. Atomic absorption technics have been extended in accordance with advances

in the capability of these methods. Wherever possible, these procedures have been extended to samples of more or less polluted water, in addition to their former applications to relatively clean samples.

Determination of Inorganic Nonmetallic Constituents (Part 400) has undergone extensive editorial revision in many sections. The syringaldazine method for residual chlorine has been introduced and the methyl orange method and all acid orthotolidine methods have been deleted. In Section 419, Nitrogen (Nitrate), the electrode method appears for the first time as a screening method, and the phenoldisulfonic acid method, and the zinc reduction method of the Thirteenth Edition have been deleted. Procedures for cyanides amenable to chlorination also are new.

In Determination of Organic Constituents (Part 500), the distillation method for volatile acids has been reinstated. A new "miniature" method for determination of total organic contaminants by activated carbon adsorption has been added. The section on organic pesticides has been amplified by inclusion of methods for chlorinated phenoxy acids, organophosphates, and carbamates. The Thirteenth Edition method for tannin and lignin has been replaced by a new, improved technic. For determination of grease and oil, previously presented technics have undergone substantial modification. Infrared determination is recommended for low concentrations of highly volatile oils, while gravimetric readout is retained where applicable. The Soxhlet extraction procedure and liquid-liquid extraction are retained, but the semiwet procedure has been deleted. Silica gel has replaced activated alumina for the separation of polar lipids from nonpolar compounds.

Completely new divisions deal with automated laboratory analyses (Part 600) and with bioassay methods (Part 800). Each of these areas represents a substantial extension of the scope of these standards. Each embraces a type of laboratory testing that is already important and that is expected to become even more so in the future. With the exception of the fish toxicity bioassay, all the material in these sections is new.

Part 700, dealing with the determination of radioactivity, has been changed relatively little from the Thirteenth Edition. Part 900 has been revised, updated, and expanded to include procedures for *Klebsiella*, pathogenic leptospires, fungi, actinomycetes, and nematodes. The material on iron and sulfur bacteria now appears in Part 900. Part 1000 likewise has been revised and updated.

Selection and Approval of Methods

For each new edition, both the technical criteria for selection of methods and the formal procedures for their approval and inclusion are reviewed critically. In regard to the approval procedures, it is considered particularly important to assure that the methods presented have been reviewed and supported by the largest possible number of qualified persons, in order that they may represent a true consensus of expert opinion.

It has been mentioned that for the Fourteenth Edition a Joint Task Group was established for each test. Appointment of an individual member to a Joint Task Group generally was based on the expressed interest of the individual or his recog-

nized expertise. The effort in every case was to assemble a group having maximum available expertise in the test methods of concern.

Each Joint Task Group was charged with reviewing the pertinent methods in the Thirteenth Edition along with other methods from the literature, recommending the methods to be included in the Fourteenth Edition, and presenting those methods in the form of a proposed manuscript section. Subsequently, each section of the manuscript was ratified by vote of the membership of the pertinent committees of the societies. Every negative vote and every comment submitted in the balloting was reviewed by the Joint Editorial Board. All relevant suggestions were referred to the appropriate Joint Task Groups for resolution.

The methods presented here, as in previous editions, are believed to be the best available and generally accepted procedures for the analysis of water, wastewaters, and related materials. They represent the recommendations of specialists, ratified by a large number of analysts and others of more general expertise, and as such are truly consensus standards, offering a valid and recognized basis for control and evaluation.

The technical criteria for selection of methods were applied by the Joint Task Groups and by the individuals reviewing their recommendations, with the Joint Editorial Board providing only general guidelines. In addition to the classical concepts of precision, accuracy, and minimum detectable concentration, selection of a method must also recognize such considerations as the time required to obtain a result, needs for specialized equipment and for special training of the analyst, and other factors related to the cost of the analysis and the feasibility of its widespread use.

Status of Methods

All methods in the Fourteenth Edition are "standard" unless designated "tentative". No other categories are used. Methods with "standard" status have been studied extensively and accepted as applicable within the limits of sensitivity, precision, and accuracy given. "Tentative" methods are those still under investigation that have not yet been evaluated fully or are not considered sufficiently tested at present to be designated "standard".

Technical progress makes advisable the establishment of a program to keep *Standard Methods* abreast of advances in research and general practice. The Joint Editorial Board has developed the following procedure for effecting interim changes in methods between editions:

1. Any method given "tentative" status in the current edition may be elevated to "standard" by action of the Joint Editorial Board, on the basis of adequate published data supporting such a change as submitted to the Board by the appropriate Joint Task Group. Notification of such a change in status shall be accomplished by publication in the official journals of the three associations sponsoring *Standard Methods*.

2. No method having "standard" status may be abandoned or reduced to "tentative" status during the interval between editions.

3. A new method may be adopted as "tentative" or "standard" by the Joint Editorial Board between editions, such action being based on adequate published data as submitted by the Joint Task Group concerned. Upon adoption, the details of the method, together with a resume of the supporting data, must be published in the official journal of any one of the three sponsoring associations, and reprints shall be made available at a nominal charge. Notice of such publication and of the availability of reprints shall appear in the official journals of the other two sponsors.

Even more important to maintaining the current status of these standards is the intention of the sponsors and the Joint Editorial Board that subsequent editions will appear regularly at intervals of approximately two years.

Acknowledgments

For the major portion of the work in preparing and revising the methods in the Fourteenth Edition, the Joint Editorial Board gives full credit to the Standard Methods Committees of the American Water Works Association and of the Water Pollution Control Federation, and to the Subcommittee on Standard Methods for the Examination of Water and Wastewater, and the Committee on Laboratory Standards and Practices of the American Public Health Association. Members of these committees chair and serve as members of the Joint Task Groups. They were assisted in many cases by advisors, not formally members of the committees, and in many cases not members of the sponsoring societies. To the advisors, special gratitude is extended in recognition of their efforts. A list of the committee members and advisors follows these pages.

The Joint Editorial Board expresses its appreciation to James R. Kimmey, M.D., and William H. McBeath, M.D., past and present Executive Director, respectively, American Public Health Association, to Eric F. Johnson, Executive Director, American Water Works Association, and to Robert A. Canham, Executive Secretary, Water Pollution Control Federation, for their continuous cooperation and helpful advice. James B. Ramsey, Director of Standards, American Water Works Association, has acted as secretary to the Joint Editorial Board for this edition and has provided an endless variety of helpful services as well as useful advice. In the final stages of compiling the manuscript, obtaining approval of every section, and readying it for printing, Mr. Ramsey and the Joint Editorial Board were assisted most ably by Paul A. Schulte, Deputy Director of Standards, American Water Works Association. Special recognition for her valuable services is due to Mary Ann H. Franson, Managing Editor of the Fourteenth Edition, who has discharged most efficiently the extensive and detailed responsibilities on which a complete volume depends.

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TABLE C. INTERNATIONAL RELATIVE ATOMIC WEIGHTS, 1975

Scaled to the relative atomic mass. $A_r(^{12}C)=12$

The atomic weights of many elements are not invariant but depend on the origin and treatment of the material. The footnotes to this table elaborate the types of variation to be expected for individual elements. The values of $A_r(E)$ given here apply to elements as they exist naturally on earth and to certain artificial elements. When used with due regard to the footnotes they are considered reliable to ±1 in the last digit or ±3 when followed by an asterisk*. Values in parentheses are used for certain radioactive elements whose atomic weights cannot be quoted precisely without knowledge of origin; the value given is the atomic mass number of the isotope of that element of longest known half life.

Name	Symbol	Atomic number	Atomic weight	Footnotes	Name	Symbol	Atomic number	Atomic weight	Footnotes
Actinium	Ac	89	(227)		Mercury	Hg	80	200.59*	
Aluminium	Al	13	26.98154	a	Molybdenum	Mo	42	95.94*	
Americium	Am	95	(243)		Neodymium	Nd	60	144.24*	
Antimony	Sb	51	121.75*		Neon	Ne	10	20.179*	c, e
Argon	Ar	18	39.948*	b, c, d, g	Neptunium	Np	93	237.0482	f
Arsenic	As	33	74.9216	a	Nickel	Ni	28	58.70	
Astatine	At	85	(210)		Niobium	Nb	41	92.9064	a
Barium	Ba	56	137.33*		Nitrogen	N	7	14.0067	b, c
Berkelium	Bk	97	(247)		Nobelium	No	102	(255)	
Beryllium	Be	4	9.01218	a	Osmium	Os	76	190.2	g
Bismuth	Bi	83	208.9804	a	Oxygen	O	8	15.9994*	b, c, d
Boron	B	5	10.81	c, d, e	Palladium	Pd	46	106.4	
Bromine	Br	35	79.904	c	Phosphorus	P	15	30.97376	a
Cadmium	Cd	48	112.41		Platinum	Pt	78	195.09*	
Caesium	Cs	55	132.9054	a	Plutonium	Pu	94	(244)	
Calcium	Ca	20	40.08	g	Polonium	Po	84	(209)	
Californium	Cf	98	(251)		Potassium	K	19	39.0983*	
Carbon	C	6	12.011	b, d	Praseodymium	Pr	59	140.9077	a
Cerium	Ce	58	140.12		Promethium	Pm	61	(145)	
Chlorine	Cl	17	35.453	c	Protactinium	Pa	91	231.0359	f
Chromium	Cr	24	51.996	c	Radium	Ra	88	226.0254	f, g
Cobalt	Co	27	58.9332	a	Radon	Rn	86	(222)	
Copper	Cu	29	63.546*	c, d	Rhenium	Re	75	186.207	c
Curium	Cm	96	(247)		Rhodium	Rh	45	102.9055	a
Dysprosium	Dy	66	162.50*		Rubidium	Rb	37	85.4678*	c
Einsteinium	Es	99	(254)		Ruthenium	Ru	44	101.07*	

Erbium	Er	68	167.26*		Samarium	Sm	62	150.4	
Europium	Eu	63	151.96		Scandium	Sc	21	44.9559	a
Fermium	Fm	100	(257)		Selenium	Se	34	78.96*	
Fluorine	F	9	18.998403	a	Silicon	Si	14	28.0855*	d
Francium	Fr	87	(223)		Silver	Ag	47	107.868	c
Gadolinium	Gd	64	157.25*		Sodium	Na	11	22.98977	a
Gallium	Ga	31	69.72		Strontium	Sr	38	87.62	g
Germanium	Ge	32	72.59*		Sulfur	S	16	32.06	d
Gold	Au	79	196.9665	a	Tantalum	Ta	73	180.9479*	b
Hafnium	Hf	72	178.49*		Technetium	Tc	43	(97)	
Helium	He	2	4.00260	b, c	Tellurium	Te	52	127.60*	
Holmium	Ho	67	164.9304	a	Terbium	Tb	65	158.9254	a
Hydrogen	H	1	1.0079	b, d	Thallium	Tl	81	204.37*	
Indium	In	49	114.82		Thorium	Th	90	232.0381	f, g
Iodine	I	53	126.9045	a	Thulium	Tm	69	168.9342	a
Iridium	Ir	77	192.22		Tin	Sn	50	118.69*	
Iron	Fe	26	55.847*		Titanium	Ti	22	47.90*	
Krypton	Kr	36	83.80	e	Tungsten (Wolfram)	W	74	183.85*	
Lanthanum	La	57	138.9055*	b	Uranium	U	92	238.029	b, c, e, g
Lawrencium	Lr	103	(260)		Vanadium	V	23	50.9414*	b, c
Lead	Pb	82	207.2	d, g	Xenon	Xe	54	131.30	e
Lithium	Li	3	6.941*	c, d, e, g	Ytterbium	Yb	70	173.04*	
Lutetium	Lu	71	174.97		Yttrium	Y	39	88.9059	a
Magnesium	Mg	12	24.305	c, g	Zinc	Zn	30	65.38	
Manganese	Mn	25	54.9380	a	Zirconium	Zr	40	91.22	
Mendelevium	Md	101	(258)						

a Element with only one stable nuclide.

b Element with one predominant isotope (about 99 to 100 percent abundance); variations in the isotopic composition or errors in its determination have a correspondingly small effect on the value of $A_r(E)$.

c Element for which the value of $A_r(E)$ derives its reliability from calibrated measurements (i.e. from comparisons with synthetic mixtures of known isotopic composition).

d Element for which known variations in isotopic composition in terrestrial material prevent a more precise atomic weight being given; $A_r(E)$ values should be applicable to any 'normal' material.

e Element for which substantial variations in A_r from the value given can occur in commercially available material because of inadvertent or undisclosed change of isotopic composition.

f Element for which the value of A_r is that of the most commonly available long-lived isotope.

g Element for which geological specimens are known in which the element has an anomalous isotopic composition.

SOURCE: International Union of Pure and Applied Chemistry.

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