STANDARD METHODS

For the Examination of Water and Wastewater

SIXTEENTH EDITION

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PREFACE TO THE SIXTEENTH EDITION

The Fifteenth and Earlier Editions

The first edition of *Standard Methods* was published in 1905. Each subsequent edition presented significant improvements of methodology and enlarged its scope to include techniques 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 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 bacteriological 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 committee report, published in 1905, constituted the first edition of *Standard Methods* (then entitled *Standard Methods of Water Analysis*). Physical, chemical, microscopic, and bacteriological 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),

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* *J. Anal. Chem.* 3:398 (1889).

† *Proc. Amer. Pub. Health Ass.* 23:56 (1897).

and 1923 (Fifth). In 1925, the American Water Works Association 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 examination of industrial wastewaters; this was reflected by a new title: *Standard Methods for the Examination of Water, Sewage and Industrial Wastes.* 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 in the Twelfth Edition (1965), the Thirteenth Edition (1971), the Fourteenth Edition (1976), and the Fifteenth Edition (1981).

In the Fourteenth Edition, the separation of test methods for water from those for wastewater was discontinued. All methods for a given component or characteristic appeared under a single heading. The coordination of methods was reflected in the revised numbering system. The major divisions of the Fourteenth and Fifteenth Editions were 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

The Sixteenth Edition

With minor differences, the organization of the Fourteenth Edition has been retained. Numerous changes, revisions, and improvements in methods have been made and the most noteworthy are mentioned in this preface. Two major policy decisions of the Joint Editorial Board have been implemented for the Sixteenth

‡ *Sewage Works J.* 7:444 (1935).

Edition. First, the International System of Units (SI) has been adopted. Except where prevailing field systems or practices require English units, these have been replaced by SI units. The most obvious changes consistent with this decision are the use of L as the abbreviation of liter (instead of l) and the use of pascal (Pa) or kilopascal (kPa) for pressure. Second, the use of trade names or proprietary materials has been eliminated insofar as possible, in order to avoid potential claims regarding restraint of trade or commercial favoritism. Wherever generic substance names were available, these have been used. Terms such as borosilicate glass, polytetrafluoroethylene (TFE), etc., have been substituted for trademarks or copyrighted names. While this usage still may be unfamiliar or awkward, the Joint Editorial Board hopes that users of this book have adjusted to the changes without difficulty.

The Sixteenth Edition retains the General Introduction (Part 100), containing important information on proper execution of procedures. Every user of this manual must study both the General Introduction and the introductions to all other parts. Each introduction discusses vital matters of general application within the specific subject area to minimize repetition in the succeeding text. Successful analysis rests on close adherence to the introductory recommendations and cautions. Before undertaking an analysis, read and understand the complete discussion of each procedure, including method selection, sampling and sample storage, and interferences.

For the Sixteenth Edition the sections on ion exchange and industrial water in Part 100 have been deleted. The former was dropped because ion-exchange techniques are no longer extraordinary and in need of special emphasis; the appropriate information has been incorporated elsewhere (Section 102). The section on industrial water has been deleted because it was determined that to make it really useful was beyond the scope of a methods manual. A substitute section on reagent water has been added, which specifies reagent-water quality for various tests and the means by which high-quality water can be produced. It amplifies the information on the inside front cover. Section 108 (safety) has been rewritten and expanded considerably. While it deals with most laboratory safety questions, it should not be considered as an alternative to a comprehensive safety manual.

The general subject of laboratory quality assurance continues to occupy a major role in environmental laboratories. In addition to discussion in Section 104 (chemical analysis), quality assurance is dealt with in Sections 701 (radiological analyses), 801 (toxicity testing), and 902 (bacteriological analyses). These sections should be studied carefully to insure that laboratory results correctly and reliably reflect sample composition and that the testing complies with existing legal requirements. As with safety, quality-assurance activities represent a significant and costly, but essential, effort.

In Part 200 (physical examination), the section on oxygen transfer (208) has been deleted because it is inappropriate to a laboratory manual. The method will be available in a publication of the American Society of Civil Engineers. In the section on residue, the formerly used terms of dissolved and suspended solids have

been restored and the confusing terms filtrable and nonfiltrable residue have been discarded.

In Part 300 (metals), the sections dealing with atomic absorption spectrometry have been improved and the section on electrothermal atomic absorption spectrometry, a technique that significantly improves analytical sensitivity, has been expanded. Procedures have been revised to include the determination of palladium and rhenium, and the method for arsenic and selenium has been rewritten. A general discussion of emission spectroscopy using an inductively coupled plasma source has been added. This device makes possible simultaneous or rapid sequential determination of many metals, but without the sensitivity of the electrothermal procedure; it may be suited ideally to sample screening. Despite the emphasis on instrumental methods, classical chemical procedures, colorimetric or other, still are included, although they may be used in relatively few laboratories.

In Part 400 (inorganic nonmetals), the most striking change is the addition of an instrumental method using an ion chromatograph for measuring most anions; it permits rapid, sequential analysis with high precision and accuracy. Methods using specific ion electrodes are included where appropriate. Indicators used for measuring acidity and alkalinity have been changed but the terms methyl orange acidity or alkalinity have been retained despite the deletion of methyl orange.

Part 500 (organics) shows the most changes and additions including deletion of the steam distillation procedure for volatile acids (Section 504), deletion of the functionally replaced carbon chloroform extract procedure (Section 506); addition of the ampule method in measuring chemical oxygen demand (Section 508), and a complete rewrite and a new approach to surfactants analysis (Section 512). The additions are even more significant and include use of a mass spectrometer and an organic halogen analyzer, alternative means of oxidizing and measuring organic material in the total organic carbon test (Section 505), and closed-loop stripping analysis. The combination of gas chromatography (GC) and mass spectrometry (MS) for separating and identifying organic compounds is an extremely powerful tool in organics analysis. A general discussion of GC/MS is included and will be applicable to identification of many organic compounds; more specific is a combined analysis using closed-loop stripping and GC/MS. This CLSA-GC/MS procedure is suitable for analyzing earthy-musty-smelling compounds such as geosmin at concentrations as low as nanograms per liter. The instrumental analysis of total organic halogens (TOX) will permit rapid quantitation of halogenated compounds such as THMs, PCBs, and chlorinated pesticides without specific identification. A relatively small but major change in pesticide analysis (Section 509) is the permitted use of capillary column gas chromatography.

Part 600 (automated methods) has been deleted entirely, completing the change initiated in the Fifteenth Edition. Automated methods, when included, are to be found under the individual constituents. In partial response to a demand by users of this manual that section numbering remain unchanged between editions, Part 600, as a number, has been left. A completely new and permanent numbering system is under consideration for use in the Seventeenth Edition.

Part 700 (radioactivity) includes a new section for uranium. The instruments of choice for measuring gross radioactivity have been changed to thin-window rather than internal proportional counters.

Part 800 (toxicity testing) has been changed relatively little.

In Part 900 (microbiology) a new terminology of heterotrophic plate count (replacing standard plate count) has been accompanied by the addition of spread plate and membrane filter methods. Recognition of waterborne diseases caused by *Campylobacter, Yersinia,* and *Legionella* has led to inclusion of analytical techniques for their isolation. Improved methods for recovering *Giardia* also are included. The most noticeable general change is editorial, in that details for the preparation of microbiological media appear in the section wherein the use of that medium is first described, instead of in a special section on media.

Part 1000 (biological examinations) has been revised and updated, most noticeably in the section on fish and the general taxonomic references.

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 are supported by the largest number of qualified persons, so that they may represent a true consensus of expert opinion.

For the Fourteenth Edition a Joint Task Group was established for each test. This scheme has continued for the Sixteenth Edition. Appointment of an individual to a Joint Task Group generally was based on the expressed interest or recognized expertise of the individual. 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 Fifteenth Edition along with other methods from the literature, recommending the methods to be included in the Sixteenth Edition, and presenting those methods in the form of a proposed section manuscript. Subsequently, each section manuscript was ratified by vote of the membership of the Standard Methods Committee, which has some 400 members. 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. When negative votes on the first ballot could not be resolved by the Joint Task Group, or the Joint Editorial Board, the section was reballoted among all who voted (affirmatively or negatively) on the original ballot. Only a few issues could not be resolved in this manner and the Joint Editorial Board made the final decision.

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 also must 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 Sixteenth Edition are dated to assist users in determining those methods that have been changed significantly between editions. The year the section was approved by the Standard Methods Committee is indicated in a footnote at the beginning of each section. Sections or methods that appeared in the Fifteenth Edition that are unchanged, or changed only editorially in the Sixteenth Edition, show the publication date of the Fifteenth Edition, 1981. Sections or methods that were changed significantly, or that were reaffirmed by general balloting of the Standard Methods Committee, are dated 1985. If only one individual method within a section was revised, then that individual method is dated 1985, and the remaining methods retain the 1981 date.

All methods in the Sixteenth 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.§ A tentative method may be added by decision of the Joint Editorial Board or may be balloted formally as a tentative method.

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§ The Committee on Laboratory Standards and Practices (CLaSP) of APHA adopted a somewhat different methods classification scheme, which is presented here for consideration and comment by the reader.

Class 0—a method or procedure that has been subjected to a thorough evaluation, has been widely used, and through wide use has demonstrated its utility by extensive application, but has not been formally, collaboratively tested. This classification will include methods that are referred to as standard methods in the current APHA publications; essentially it is a grandfather clause.

Class A—a method or procedure that has been subjected to a thorough evaluation, has demonstrated its applicability for a specific purpose on the basis of extensive use, and has been successfully, collaboratively tested.

Class B—a method that has been used successfully in research or other disciplines, has been devised or modified explicitly for routine examination of specimens, has had limited evaluation, and has not been tested collaboratively.

Class C—(1) a new unproved or suggested method not previously used but one that has been proposed

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 the usual consensus procedure.

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 reasonably short intervals. Reader comments and questions concerning this manual should be addressed to: STANDARD METHODS, American Water Works Association, 6666 West Quincy Avenue, Denver, CO 80235.

Acknowledgments

For the major portion of the work in preparing and revising the methods in the Fifteenth 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 often by advisors, not formally members of the committees, and in many

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by recognized laboratory workers as useful or gives promise of being suitable; (2) a method that previously has been placed in Classes O, A, or B but which, through technological advances or significant change in numerical level of acceptable exposure or other circumstances, has been rendered not suitable for its intended purpose and presumably has been superseded by a method of a higher classification. In essence C-1 includes proposed new methods and C-2 includes methods no longer recommended.

Except for Class O, the scheme allows for a progression from Class C to Class A thereby permitting a new unproven method or procedure to be made available pending further evaluation (Class C). As the procedure is tested and evaluated it progresses to Class B and, after thorough evaluation and a successful collaborative test, it becomes a Class A method.

The scheme is most readily applied to manuals of methods that are periodically reissued and the additions, deletions and changes are a challenge to the user. The scheme could foreseeably be applied to a wider range of publilcations other than manuals.

Note, however, that standard methods as defined herein are comparable to CLaSP's Class O or A while tentative methods are comparable to Class B or (possibly) C.

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 William H. McBeath, M.D., Executive Director, American Public Health Association, to David B. Preston, Executive Director, American Water Works Association, and to Robert A. Canham, Executive Director, Water Pollution Control Federation, for their continuous cooperation and helpful advice. Frederick W. Pontius, Water Quality Engineer, American Water Works Association, acted as secretary to the Joint Editorial Board for this edition and provided an endless variety of helpful services as well as useful advice. Adrienne Ash, Director of Publications, American Public Health Association, ably functioned as the publisher. Special recognition for her valuable services is due to Mary Ann H. Franson, Managing Editor of the Sixteenth Edition, who has discharged most efficiently the extensive and detailed responsibilities on which a complete volume depends.

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