REAGENT CHEMICALS

SEVENTH EDITION

AMERICAN CHEMICAL SOCIETY SPECIFICATIONS

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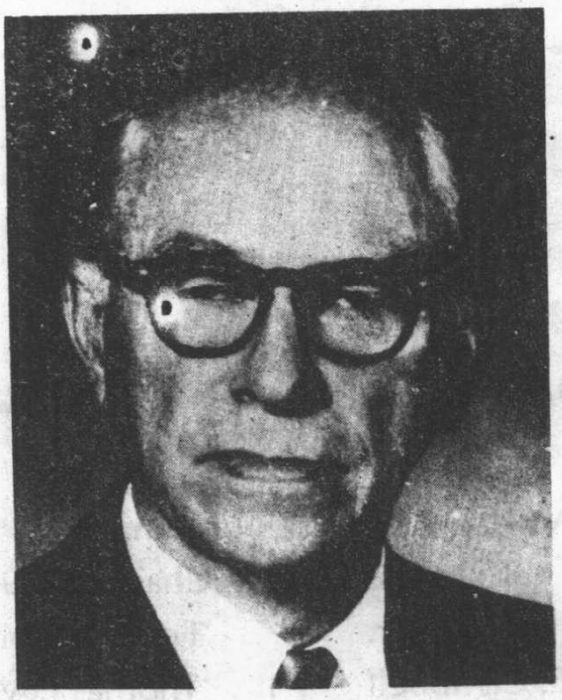

Edward Wichers
1892–1984

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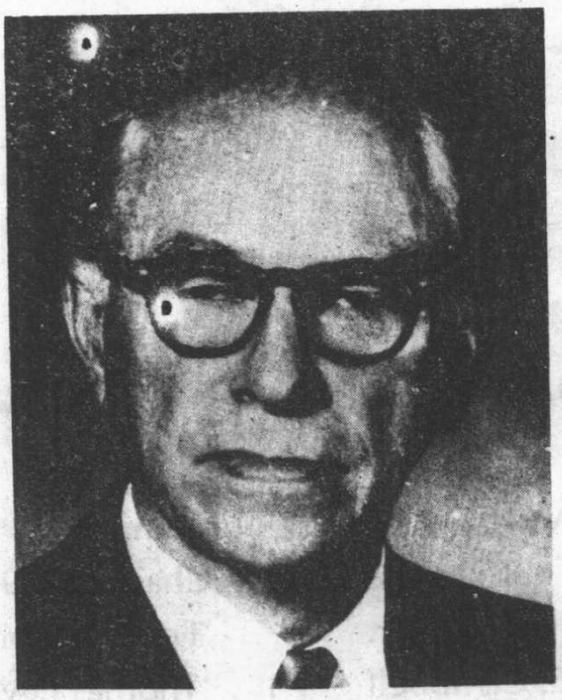
One of the world's outstanding authorities on analytical chemistry, Dr. Wichers retired from the National Bureau of Standards (NBS) in 1962 after a career of 44 years. He joined NBS in 1917, the year he received his Ph.D. degree in inorganic chemistry from the University of Illinois. Three years later, he became chief of the section dealing with platinum metals and reagent chemicals, and in 1948, he was named Chief of the Chemistry Division. He was appointed NBS Associate Director for Chemistry in 1958.

Dr. Wichers was chairman of the Commission on Atomic Weights of the International Union of Pure and Applied Chemistry (IUPAC) from 1949 to 1969. In this role, he was a major influence in the adoption in 1961 of the unified scale of atomic weights, which replaced the different scales used by chemists and physicists. During this period, he also served as President of the Inorganic Division of IUPAC from 1955 to 1959.



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PREFACE

The ACS Committee on Analytical Reagents evolved from the Committee on the Purity of Chemical Reagents established in 1903. Changes in the name and role occurred before the current name was adopted in 1927. Standards in a style close to that now used were proposed in 1917 for ammonium hydroxide, hydrochloric acid, nitric acid, and sulfuric acid and appeared in 1921 in *Industrial and Engineering Chemistry.* Policies for Standards begun in 1924–25 have been pursued continually. Initially, Standards appeared in *Industrial and Engineering Chemistry* and its *Analytical Edition.* In 1941, existing Standards were reprinted in a single pamphlet. Eventually, revisions and new Standards were gathered together into a single book, the 1950 Edition of *Reagent Chemicals.* This edition was followed by the 1955 and 1960 Editions and by the Fourth, Fifth, and Sixth Editions in 1968, 1974, and 1981, respectively.

The number of reagents for which ACS Standards have been adopted continues to increase. This Seventh Edition includes 32 new Standards, 21 of which appeared in Supplement No. 1 to the Sixth Edition issued in 1984, and 10, thereafter, in *Analytical Chemistry, 58,* No. 6, May 1986, pp. 1276–80 (*see Chemical and Engineering News,* June 2, 1986, p. 26, for an additional Standard).

Two directions adopted with the Sixth Edition are continued: (1) establishing assay limits and procedures for hydrated salts and some other compounds and (2) unifying the text for general tests.

New practices include providing the Chemical Abstracts number for each Standard as an aid for on-line searching of literature and listing additional secondary names, as well as presenting the various Standards for acetonitrile, chloroform, dichloromethane, hexanes, and methanol in a combined format (suitable for general use, ultraviolet spectrophotometry, high-performance liquid chromatography, or pesticide residue analysis). As an aid to the user, the index is enlarged.

The choice of nomenclature, abbreviations, units, and editing policies has been guided generally by current ACS practices. Usually the degree of hydration of salts is explicitly stated rather than implied by the context. In Standards, secondary names for relevant metal compounds now include the use of Stock numbers, and those for organic com-

pounds include recent practices of *Chemical Abstracts*. Trademarked items appear with an initial capital letter and are listed with their owners on page 71.

The following lists the membership for each person serving on the Committee after issue of the Sixth Edition in 1981:

Alfred J. Barnard, Jr.	1981–	Thomas J. Murphy	1967–85
Donald E. Campbell	1974–84	Robert Parmerter	1985
Anthony D. Debolli	1979–81	Anthony D. Pietrzykowski	1986–
Kishor A. Desai	1981–	Sterling Pomeroy	1976–81
Clifford A. Flanders	1954–81	Charles J. Pouchert	1978–
Norman C. Jamieson	1982–	Wallace G. Rohrbough	1965–
Richard S. Juvet	1986–	*Chairman*	1981–85
Lynn L. Lewis	1976–	William E. Schmidt	1967–
Clarence Lowery	1974–	*Secretary (nonvoting)*	
Chairman	1985–	Vernon A. Stenger	1962–
Murugan Malaiyandi	1982–	*Chairman*	1967–73
Irving May	1969–81	Cyrus M. Strauss	1982–
Loren C. McBride	1983–	Samuel M. Tuthill	1958–
Thomas W. Mears	1966	*Chairman*	1974–80
	1973–	*Consultant*	1982–
John R. Moody	1986–	Paul S. Von Bacho, Jr.	1981–84
Fred A. Morecombe	1966–84	(deceased)	
(deceased)		Frank G. Walthall	1982–
		Donald H. Wilkins	1983–

Wallace G. Rohrbough served as chairman of the Committee during most of the revision; he was succeeded by Clarence Lowery in the fall of 1985.

The Committee normally meets in the spring and fall of each year. After each meeting, changes of an immediate nature are reported in *Chemical and Engineering News*. As such changes accumulate and as changes of a nonurgent nature, including new Standards, are approved, it is expected that they will appear in *Analytical Chemistry*. Such publications will be announced in *Chemical and Engineering News*.

The Committee plans at least one formal supplement, sized to fit this book, before publishing the next full edition. Interim changes published in *Chemical and Engineering News* or *Analytical Chemistry* will be included. The availability of such supplements will be announced in *Chemical and Engineering News*. Cards for requesting them are in the back of this book and should be filled in and returned—best when the book is first received, but also after announcement of the supplements.

Since the publication of the Sixth Edition of *Reagent Chemicals*, the

Committee has adopted written operating procedures that describe and govern its operations. Interested parties may obtain a copy of these procedures by addressing their requests to:

Secretary, ACS Committee on Analytical Reagents
c/o Books Department
American Chemical Society
1155 Sixteenth Street, NW
Washington, DC 20036

The Committee urges that any errors observed be reported, invites constructive criticism, and welcomes suggestions concerning added Standards and tests. Communications on these subjects should be sent to the secretary at the above address.

CONTENTS

DEFINITIONS, PROCEDURES, AND STANDARDS

SPECIFICATIONS AND TESTS

The specifications prepared by the Committee on Analytical Reagents of the American Chemical Society are intended to serve for reagents to be used in precise analytical work of a general nature. It is recognized that there may be special uses for which reagents conforming to other, or more rigorous, specifications may be needed. Therefore, where known and where feasible, some of the specifications herein include requirements and procedures for certain specialized uses. However, it is impossible to include specifications for all such uses, and thus there may be occasions when it will be necessary for the analyst to further purify reagents known to have special purity requirements for certain uses.

The requirements and the details of tests are based on published work, on the experience of members of the Committee in the examination of reagent chemicals on the market, and on studies of the tests made by members of the Committee. The limits and procedures are designed for application to reagents in freshly opened containers. Reagents in containers of extended age, in containers subject to constant changes in humidity or headspace gas content (as by repetitive opening and closing of the container), or subjected to potential inadvertent contamination by repeated opening of the container may not conform to the designated requirements. Where the possibility of change due to age, humidity, light, or headspace contamination is recognized, the Standard usually contains a warning; nonetheless, the analyst is cautioned to take appropriate steps to ensure the continued purity of his or her reagents, especially after opening the container.

In determining quality levels to be defined by new or revised specifications the committee is guided by the following general principles. When a specification is first prepared, it will usually be based on

the highest level of purity (of the reagent to which it applies) that is competitively available in the United States. Generally, the term "competitively available" is understood to mean that the material is available from two or more producers. If a significantly higher level of purity subsequently becomes available on the same competitive basis, the specification will generally be revised accordingly.

Because the requirements of a specification relating to the content of designated impurities must necessarily be expressed in terms of maximum permissible limits, products conforming to the specification will normally contain less than the maximum permissible proportion of some or all of these impurities. A given preparation of a reagent chemical that has less than the maximum content of one or more impurities permitted by the specification is, therefore, not considered as of higher quality than that defined by the specification.

A lower permissible limit for a given impurity will be adopted only if it is significantly different from the one it is intended to supersede. In general a new requirement for an impurity whose content is not greater than 0.01% will not be considered significantly different unless it decreases the maximum permissible content of the impurity by at least 50%. This principle will also be approximated in the revision of those requirements defined by the term, "To pass test."

Tests as written are considered to be applicable only to the accompanying requirements. Modification of a requirement, especially if the change is toward a higher level of purity, will necessitate reconsideration, and often revision, of the test to ensure its validity.

The assays and tests described herein constitute the methods upon which the ACS Reagent Standards are based. The analyst is not prevented, however, from applying alternative methods of analysis that produce results of at least equal reliability. In the event of doubt or disagreement concerning a substance purported to comply with the ACS Standards, only the methods described herein are applicable.

SOLVENTS FOR SPECIAL PURPOSES

For some solvents, the 6th Edition of *Reagent Chemicals* had separate Standards defining them as either "suitable for use in ultraviolet spectrophotometry" or "suitable for use in determining pesticide residues." In recent years, interest has developed in characterizing certain solvents as "suitable for use in high-performance liquid chromatography." In this edition, these special-use Standards have been treated in a single, integrated presentation. The seller shall designate in product labeling the suitability for one or more of these special uses on the basis of the relevant specifications and tests.

HAZARDOUS SUBSTANCES

Some of the reagent chemicals for which ACS Standards have been adopted are hazardous substances. The use of such reagents and the application of the various test methods specified by the ACS Committee on Analytical Reagents may involve hazardous substances, operations, and equipment. The ACS Committee on Analytical Reagents does not purport in its publications to address all of the safety problems associated with ACS Reagents, their use, or the methods prescribed for testing them. It is the responsibility of whoever uses ACS Reagents and the ACS testing methods to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to such use. Material Safety Data Sheets, which contain precautionary information related to safety and health concerns, are available for many chemicals from manufacturers and/or distributors of reagent chemicals and may be of assistance in carrying out the aforementioned responsibility.

CONTAINERS

The container is the device that holds the reagent and that is, or may be, in direct contact with the reagent. The closure is part of the container.

The container in which a reagent is sold and/or stored must be suitable for its intended purpose and should not interact physically or chemically with the contained reagent so as to alter its quality (within a reasonable period of time) beyond the requirements of the Standard.

Containers for solids normally have wide mouths to facilitate both the filling of the container and the removal of the contents. Containers for liquids normally have narrow mouths so that the contents may be easily poured into other, frequently smaller, containers.

Prior to its being filled, the container should be free from extraneous particulate matter, otherwise clean, and dry if necessary.

INTERPRETATION OF REQUIREMENTS

The requirements of reagent chemicals can be divided into two main classes: an assay or quantitative determination of the principal or active constituent and the determination of the impurities or minor constituents. By far, the majority of the requirements belong to the latter class. In some cases physical properties are specified.

For comparison of analytical results with requirements for assays and impurities, the observed or calculated values are rounded to the number of decimal places carried in the requirement. The method and the rounding procedure are in accord with the American Society for Testing and Materials Practice E 29, for "Indicating Which Places of Figures Are to Be Considered Significant in Specified Limiting Values."

The procedure is as follows: If the digit following the last place to be retained is not equal to 5, round to the nearest number; if the digit to be dropped is 5 or 5 followed by zeros, round to an even number. This rounding procedure is illustrated in the following table:

Rounding Procedure

Requirement	*Observed Value*	*Rounded Value*	*Pass/Fail*
Not less than 98	97.6	98	pass
	97.5	98	pass
	97.4	97	fail
Not less than 98.0	97.95	98.0	pass
	97.94	97.9	fail
Not more than 0.01	0.014	0.01	pass
	0.015	0.02	fail
	0.016	0.02	fail
Not more than 0.02	0.015	0.02	pass
	0.025	0.02	pass
	0.026	0.03	fail

(Observe that the foregoing procedure does not conform to the common electronic calculator and computer procedure of rounding up when the digit to be dropped is 5 or 5 followed by zeros.) The rounded value should be obtained in a single step by direct rounding of the most precise value available and not in two or more steps of successive rounding. For example, 97.5487 rounds to 97.5 against a requirement of 97.6 and not in two possible steps of 97.55 and then 97.6.

The formula weights and factors for computing results are based on the 1983 International Atomic Weights shown on the endpapers. The formula weights are rounded to two decimal places.

Assay Requirements

Assay requirements are included in many of the Standards in this book. An assay value, in the sense used herein, is the content or concentration of a stated major component in the reagent. Unless otherwise specified, assay requirements are on an as-is basis, that is, without drying, ignition, or other pretreatment of the sample.

Unless described in great detail and carried out with exceptional skill, available assay methods seldom are accurate enough to permit using

a weighed quantity of a reagent so assayed in an exacting stoichiometric operation. This use of reagent chemicals should be limited to those designated as standards (for example, acidimetric or reductometric standards) because especially exacting assay methods are provided for such reagents.

Except in the case of standards, assays, through their minimum and maximum limits, mainly serve to assure acceptable consistency of the strength of reagents offered in the marketplace. They are particularly useful, for example, in the requirements for acid–water systems to control strength; for alkalies to limit the content of water and carbonate; for oxidizing and reducing substances that may change strength during storage; and for hydrates to control, within reasonable limits, deviations in the amount of water from that indicated in the formula. If, however, it should be necessary to use such reagents in stoichiometric operations, the user should satisfy himself or herself as to the exact values to employ.

Chromatographic assays, where applicable, are advantageous in that they are selective in many cases for the substance being assayed and also may provide an indication of the impurities present.

Impurity Requirements

Requirements for impurities are expressed as one of the following: (1) as numerical limits; (2) in terms of the expression "To pass test," with an accompanying approximate numerical limit; (3) in terms of the expression "To pass test" without an approximate numerical limit. The distinction between these forms of expression is based on the committee's opinion as to the relative quantitative significance of the prescribed methods of test. The methods given for determining conformity to requirements of the first type are considered to yield, in competent hands, what are usually thought of as "quantitative" results whereas those of the second type can be expected to yield only approximate values. Those in class 3 give definitions that cannot be expressed in numbers. It is obvious, however, that these distinctions as to quantitative significance cannot be sharp and that even the numerically expressed requirements are not all defined with equal accuracy. The final and essential definition of any requirement must, therefore, reside in the prescribed method of test rather than in its numerical expression.

If a method of test yields results that are adequately reproducible on repeated trials in different laboratories, it offers a satisfactory definition of the content of an impurity whether or not the result can be expressed by a number. Although the committee has endeavored to base requirements, so far as possible, on methods of testing that meet this criterion, there are a considerable number that are based on essentially undefined statements such as "no turbidity," "no color," or "the color shall not be completely discharged in ..... minutes." While some of the requirements of this kind could be replaced by others based on quantitative compari-

sons or measurements, to do so would require more costly or time-consuming procedures than appear at this time to be justified. The approach to an ultimate goal of replacing in every instance the word "none," or its equivalent, by the expression "not more than ....." therefore is limited both by deficiencies of knowledge and by practical considerations of expediency.

Unlisted Impurities

The primary objective of the Committee in preparing reagent specifications is to assure the user of the strength, quality, and purity of the reagents. It is, however, manifestly impossible to include in each Standard a test for every impurity and contaminant that may be present. The Committee recognizes that for certain uses more stringent or additional specifications may be required, and for such uses additional testing beyond that specified in the Standards should be employed by the user. The Committee's intent in establishing the specifications is to recognize the common uses for which the reagent is employed and to establish specifications that are consistent both with these uses and with the manufacturing processes and quality of the available reagents. The presence of moisture, either as water of crystallization or as an adventitious impurity, falls within the purview of contamination unless permitted by the specifications in the applicable Standard.

While tests for foreign particulate matter are not usually included in the specifications for solid reagents, such matter constitutes undesirable contamination. Similarly, while tests for clarity are not usually included in the specifications for liquid reagents or for solutions of solid reagents, the presence of haze, turbidity, or foreign particulate matter not common to the production of a quality reagent also constitutes undesirable contamination.

In some instances residual amounts of substances that have been added as aids in the process of purification may be present. An example is the use of complexing agents to keep certain metal ions in solution during recrystallization. These substances not only are impurities but may interfere with the tests.

Certain reagents, such as desiccants and indicators, have specifications that assure suitability for their intended use. Such reagents may contain impurities that do not interfere with their intended use, but that may make these reagents unsuitable for other uses.

When the Committee becomes aware of an unlisted impurity that affects adversely the known or specified uses of a reagent, a new requirement is added to the specifications, provided a suitable method of test is available. Users of reagents can protect themselves against the effects of unlisted impurities on a specific analytical procedure by applying, *ad hoc*, an appropriate "suitability" test.

Added Substances

Unless otherwise specified for an individual reagent chemical, the reagents described in this volume may contain suitable preservatives or stabilizers, intentionally added to retard or inhibit natural processes of deterioration. Such preservatives or stabilizers may be regarded as suitable only if the following conditions are met:

1. That they do not exceed the minimum quantity required to achieve the desired effect.
2. That they do not interfere with the tests and assays prescribed for the individual reagents.
3. That the presence of any added substance be declared on the label of the individual package. Unless of a proprietary nature the name and concentration of any added substance should be stated on the label.

Identity Requirements

Identity requirements and tests are not included in the specifications. If there is any question as to the identity of a chemical, identity can be ascertained by appropriate analytical methods.

Particle Size for Granular Materials

When a mesh or a mesh range is stated on a reagent label, the label shall include reference to a coarse sieve and to a finer sieve. The sieve number (mesh) is related to the sieve opening as indicated in the accompanying table.

Sieve No. (mesh)	*Sieve Opening* (mm)
2	9.52
4	4.76
8	2.38
10	2.00
20	0.84
30	0.59
40	0.42
50	0.297
60	0.250
80	0.177

8 Interpretation of Requirements

When tested according to the procedure given below, at least 95% of the material shall *pass* through the coarse sieve and at least 70% shall be *retained* on the finer sieve. This requirement applies only to materials that are 60 mesh or coarser.

Procedure

The sieves used in this procedure shall be those known as the U.S. Standard Sieve Series. For the details of the standardization of such sieves, reference may be made to ASTM E11, Specification for Wire-Cloth Sieves for Testing Purposes.

Place 25 to 100 g of the materials to be tested upon the appropriate coarser standard sieve, which is mounted above the finer sieve to which a close-fitting pan is attached. Place a cover on the coarser sieve and shake the stack in a rotary horizontal direction and vertically by tapping on a hard surface for not less than 20 minutes or until sifting is practically complete. Weigh accurately the amount of material remaining on each sieve.

An alternate procedure may be used in which the screening through the standard sieves is carried out in a mechanical *sieve shaker*. This shaker reproduces the circular and tapping motion given to the testing sieves in hand sifting, but with a mechanical action. Follow the directions provided by the manufacturer of the shaker.

Evaluation of Particle Size Distribution for Anion Exchange Resins

Spherical Beads

Apparatus. This method requires the following special equipment or its functional equivalent:

1. A microscope capable of resolving the smallest particle to be counted and equipped with a mechanical stage and camera. The magnification recommended is 50×—provided by a combination of 4× objective, 12.5× eyepiece, and a condenser that fills the back lens of the objective with light. This system uses a 1× camera. If the camera differs from 1×, the magnification at the eyepiece should be modified to maintain the overall magnification at 50×—for example, a 4× objective, a 25× eyepiece, and a ½× camera will give the same results.
2. Provisions for variable intensity illumination by transmitted light.