REAGENT CHEMICALS

SIXTH EDITION

AMERICAN
CHEMICAL
SOCIETY
SPECIFICATIONS

REAGENT CHEMICALS

SIXTH EDITION

AMERICAN CHEMICAL SOCIETY SPECIFICATIONS

Official from January 1, 1982

American Chemical Society
Washington, D.C.
1981

Library of Congress Cataloging in Publication Data
American Chemical Society. Committee on Analytical Reagents.
Reagent chemicals.
Includes Index.
1. Chemical tests and reagents. I. Title.
QD77.A54 1981 543'.01 81-8111
ISBN 0-8412-0560-4 AACR2

PRINTED IN THE UNITED STATES OF AMERICA

Prepared by the COMMITTEE ON ANALYTICAL REAGENTS (1975–1980)

Samuel M. Tuthill, *Chairman*

Francis J. Amore
Donald E. Campbell
Maurice D. Cooper
Anthony D. Debolli
John A. Fiorino
Clifford A. Flanders
Floyd J. Green
Lucius G. Jordan
Lynn L. Lewis
Clarence Lowery
Jeanne M. Luly
Irving May
Thomas W. Mears
Fred A. Morecombe
Thomas J. Murphy
Sterling Pomeroy
Charles J. Pouchert
Wallace G. Rohrbough
Michael A. Semon
Michael J. Spitulnik
Vernon A. Stenger
G. Edward Wolf

William E. Schmidt, *Secretary*

PREFACE

The present ACS Committee on Analytical Reagents evolved from the Committee on the Purity of Chemical Reagents established in 1903. Several changes of name and role occurred before the current name was designated in 1927. Specifications in approximately the style used today were proposed in 1917 and published in 1921 in *Industrial and Engineering Chemistry* for four chemicals—ammonium hydroxide and hydrochloric, nitric, and sulfuric acids. The present pattern and policy for ACS specifications were begun in 1924–25 and systematically pursued thereafter. Initially, specifications and tests were published in various issues of *Industrial and Engineering Chemistry* and its *Analytical Edition*, and in 1941 previously issued specifications were reprinted in a single pamphlet. Eventually the revisions and new items were collected in a single book, the 1950 Edition of *Reagent Chemicals*. This was followed by the 1955 and 1960 Editions, the 4th Edition (1968), and the 5th Edition (1974).

The number of reagents for which ACS specifications and tests have been adopted continues to increase. In addition to the four new items added to the 5th Edition by Supplement No. 1 (1977), eight new items appear for the first time in the 6th Edition. Among the items added are three solvents especially controlled for use in the analysis of pesticides. Other solvents now may be added in the format that thus has been established.

In the past, many chemicals, and especially those that are marketed as hydrates, have been defined only by their description or formula. In this edition of the book, assay limits and tests have been established to define most of the hydrates for which monographs currently exist.

One of the main objectives of the Committee in developing this 6th Edition of *Reagent Chemicals* has been to standardize as much as possible the general tests. Hence, the section on Definitions, Procedures, and Standards has been extensively expanded to include many of the general procedures used in monograph tests. This has greatly reduced duplication of test descriptions in the monographs, as reference may now be made in the monograph to the general test methods in the front of the book. The most important result, however, is that this change makes the task of adding new reagents to the book much easier. Thus, the stage has been set, we hope, for adding many new reagents—both to supplement the 6th Edition and for the 7th Edition.

The list cited below shows the period of membership of each person who has served on the Committee following the publication of the 5th Edition in 1974. The size of the Committee has been maintained at 18 or 19 members.

Amore, Francis J.	1975–1980
Campbell, Donald E.	1974–1980
Cooper, Maurice D.	1963–1975*
Debolli, Anthony D.	1979–1980
Fiorino, John A.	1972–1977
Flanders, Clifford A.	1954–1980
Green, Floyd J.	1963–1977
Jordan, Lucius G.	1972–1975
Lewis, Lynn L.	1976–1980
Lowery, Clarence	1974–1980
Luly, Jeanne M.	1977–1980
May, Irving	1969–1980
Mears, Thomas W.	1966 1973–1980
Morecombe, Fred A.	1966–1980
Murphy, Thomas J.	1967–1980
Pomeroy, Sterling	1976–1980
Pouchert, Charles J.	1978–1980
Rohrbough, Wallace G.	1965–1980
Schmidt, William E. *Secretary (nonvoting)*	1967–1980
Semon, Michael A.	1969–1980
Spitulnik, Michael J.	1972–1977
Stenger, Vernon A. *Chairman*	1962–1980 1967–1973
Tuthill, Samuel M. *Chairman*	1958–1980 1974–1980
Wolf, G. Edward	1966–1980

*Deceased

Samuel M. Tuthill has served as Chairman of the Committee during the entire revision period.

The Committee plans to issue one or two supplements prior to publication of the next full edition. These will be published in *Analytical Chemistry*, and reprints to fit this book will be made available. Cards for requesting the supplement reprints are in the back of this book. In order to be sure of receiving a copy of the supplement, it is suggested that you fill out and send in both cards at the time you receive your book. Alternatively, the respective cards may be sent in when supplements are announced in *Analytical Chemistry*.

Following its Spring and Fall meetings, the Committee will announce in *Analytical Chemistry* any changes in specifications and/or tests that merit early notification.

As in the past, the Committee urges readers to report any errors noted in the book.

CONTENTS

DEFINITIONS, PROCEDURES, AND STANDARDS

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. 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. It is recognized that there may be special uses for which reagents conforming to these specifications must be further purified with respect to one or more particular impurities.

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.

INTERPRETATION OF REQUIREMENTS

The requirements of reagent chemicals can be divided into two main classes—namely, 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, the results are rounded off to the number of places carried in the requirement. *A requirement of a minimum of 46% will be met by a product whose analysis shows 45.5%, but not by one whose analysis shows 45.4%. A minimum of 46.0% would demand at least 45.95%;* 45.9% would fail. Rounding off is accomplished according to the commonly used practice of reducing or increasing numbers less than or greater than 5. When the number to be considered is exactly 5, it is reduced or increased by 5, whichever gives an even number for the last digit retained. Thus, 36.465 and 36.455 are both rounded off to 36.46. The formula weights and factors for computing results are based on the 1977 International Atomic Weights shown on the endpapers. The formula weights are rounded off to two decimal places.

Assay Requirements

Assay requirements are included in these specifications only when they are essential to the definition of quality. Such requirements are included, for example, in specifications for standards, for acid-water systems to control strength, for alkalies to limit the content of water and carbonate, for oxidizing or reducing substances which may change strength on storage, and for most hydrates to control, within reasonable limits, the deviations in the amount of water from that indicated by the formula.

In most other instances assays would serve only as a form of identity test of uncertain reliability. Unless described in great detail and carried out with exceptional skill and care, available assay methods seldom are accurate enough to permit using a weighed quantity of a reagent, so assayed, in a precise stoichiometric operation. This use of reagents should be limited to those designated as standards. In this connection see the statement concerning moisture in crystals under "Unlisted Impurities," page 4.

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 comparisons 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 monograph 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 monographs 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 monograph.

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

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 E 11.

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.

3. *Stage micrometer graduated in 10 to 100 micrometers.*
4. Measuring scale for particles at the nominal lower and upper limit of the suggested range. Prepare a photomicrograph of the calibrated stage micrometer at the 50× magnification. Place a sheet of frosted acetate or other suitable material over the photomicrograph and draw in ink with a fine-line drawing pen two pairs of parallel lines, separated by distances corresponding to the minimum and the maximum diameters of interest. For convenience, the parallel lines should be long enough to span the full width of the photomicrograph.

Procedure. Soak a representative portion of the resin in water (2 to 4 g per 100 mL) for at least 30 minutes. While stirring vigorously to maintain a uniform suspension, immediately place a drop of the slurry on a 1× 3-inch microscope slide and cover with a cover glass. If necessary, add a drop of water at the edge of the cover glass to ensure that the entire area under the cover glass is completely covered with water. Apply pressure to the cover glass to form a monolayer of beads.

Place the slide on the stage of the microscope and select an area in which the beads are fairly well separated from one another. Prepare a photomicrograph with a double exposure in the following manner. Focus on the large particles (at the nominal upper limit of the suggested range) and expose for about half the normal time; then, without moving the slide or the film, focus the microscope on the small particles (at the nominal lower limit of the suggested range) and expose for the same time. A single exposure may be used if a reasonably sharp focus can be attained on both the largest and the smallest beads to be measured.

Prepare a sufficient number of photomicrographs from no fewer than four slides to provide at least 1000 beads for examination. In each photomicrograph count the beads with diameters less than that of the lower limit as determined with the measuring scales, taking care to measure the larger, sharp image of each bead; then count all of the beads in the photomicrograph. Calculate and record the percent by number having (1) less than the specified minimum diameter and (2) greater than the specified minimum diameter.

Irregular Bead Fragments. Dry about 50 g of well mixed sample at 105° for 4 hours. Prepare a nest of sieves to classify the resin in the range of particle size sought. Add 0.5 g of minus 325 mesh magnesium trisilicate to 25 g of the dried sample, mix, and transfer to the top sieve. Place the nest of sieves, with pan and cover, on a vibrating mechanical shaker which features a combined vertical and lateral gyration. (A Cenco-Meinzer shaker has been found satisfactory for this purpose.) Shake, without tapping, for 25 minutes. Weigh the fractions and calculate the particle size range. (Allow for the weight of magnesium trisilicate.)

PRECAUTIONS FOR TESTS

The descriptions of the individual tests are intended to give all essential details without repetition of considerations that should be obvious to an experienced analyst. A few suggestions are given for precautions and procedures that are particularly applicable to the routine testing of reagent chemicals.

Samples for Testing

To eliminate accidental contamination or possible change in composition, samples for test must be taken from freshly opened containers.

ACCURACY OF MEASUREMENTS

In specifying the weights or volumes of sample to be used in the individual test procedures, it is intended—unless otherwise specified in the individual procedure—that the accuracy of measurement be such that the amount of sample used is within 2.0% of the stated amount. Thus, where a 10 g (mL) sample is specified, the amount actually taken for analysis must be between 9.8 and 10.2 g (mL). Similarly, where a test procedure directs that a solution be diluted to a specific volume or that a specified volume of solution be used, it is intended that the volume actually be within 2.0% of the stated amount.

Where the term "pipet" is used in a verbal sense, it is intended that the specified volume be taken in a volumetric pipet conforming to the tolerances accepted by the National Bureau of Standards (*cf.* "The Calibration of Small Volumetric Laboratory Glassware," NBSIR 74-461, Dec., 1974).

The addition of small volumes of liquid reagents is generally stated to the nearest 0.05 mL (0.05 mL, 0.10 mL, 0.15 mL, etc). Use of the term "drop" to represent 0.05 mL is avoided.

Reagents

Reagents used in the testing should conform to ACS specifications. Reagents not covered by ACS specifications should be of the best grade obtainable and should be examined carefully for interfering impurities.

Blank Tests

Many of the tests are for minute quantities of the impurities sought. Hence complete blank tests must be made covering the water and other reagents used in each step of the tests—including, for example, filtration and ignition. Frequently, however, the directions do stipulate a control, a blank, or other device that corrects for possible impurities in the water and other reagents (see page 57).

GENERAL DIRECTIONS AND PROCEDURES

The general directions and/or procedures for the most common ACS specifications and tests are described in this section. To conserve space in the monographs for particular reagent chemicals, these procedures are not repeated in the individual test descriptions. Page references to this section are cited for most of the following tests: gravimetric methods (insoluble matter and residue after ignition); measurements of physical properties (boiling range, color, density, freezing point, and melting point); colorimetric and turbidimetric determinations of selected impurities (ammonium, arsenic, chloride, heavy metals, iron, nitrate, nitrogen compounds, phosphate, silicate, and sulfate); and certain instrumental methods (flame photometry, atomic absorption spectrophotometry, potentiometry, polarography, electrometric endpoint detection, and gas chromatography).

Gravimetric Method for Determining Small Amounts of Impurities

In many of the reagent specifications it is directed that a precipitate or residue be collected and either dried or ignited so as to provide certain information concerning the purity of the reagent. Detailed instructions for every reagent would be unnecessarily repetitious, so the following procedures are provided. Except where it is directed otherwise in the specific reagent tests, these directions and precautions will be used in collecting, drying, and igniting precipitates and residues.

GENERAL CONSIDERATIONS

It is imperative that the analyst use the best techniques in performing any of the operations included in this book. Exceptional cleanliness and protection against accidental contamination from dirt and fumes are rigid requirements for acceptable results. The choice of equipment is generally left to the discretion of the analyst, but it must provide satisfactory precision and accuracy. All weighings must be determined with an uncertainty of not more than ±0.0002 g.

When the use of a tared container is specified, the container will be carried through a series of operations identical to those used in the procedure, including drying, igniting, cooling in a suitable desiccator, and weighing. The length of the drying or ignition and the temperature employed must be the same as specified in the procedure in which the tared equipment is to be used. Where it is directed to dry or ignite to constant weight, two successive weighings may differ by not more than ±0.0002 g, the second weighing following a second drying or ignition period.

In those operations wherein a filtering crucible is specified, a fritted-glass crucible, a porous porcelain crucible, or a crucible having a sponge platinum mat may be used. Certain solutions may attack the filtering vessel—for example, both fritted glass and porcelain are affected by strongly alkaline solutions. Even platinum sponge mats are attacked by hydrochloric acid unless the crucible is first washed with boiling water to remove oxygen.

COLLECTION OF PRECIPITATES AND RESIDUES

In many of the tests the amount of residue or precipitate may be so small as to escape easy detection. Therefore, the absence of a weighable residue or precipitate must never be assumed. However small, it must be properly collected, washed, and dried or ignited. The size of the filter paper to be used is selected according to the amount of precipitate to be collected, not by the volume of solution to be filtered.

Often when ammonia is added in slight excess to precipitate the R_2O_3 group of elements, the amount of precipitate produced is so small that it is hard to see and collect. If a small amount of a suspension of ashless filter paper pulp is added toward the end of the period of digestion on the steam bath or hot plate, the flocculation and collection of the hydroxides are facilitated.

Some precipitates have a strong tendency to "creep"; others, like magnesium ammonium phosphate, stick fast to the walls of the container. The use of the rubber-tip "policeman" is recommended, sometimes supplemented by a small segment of ashless filter paper. The analyst