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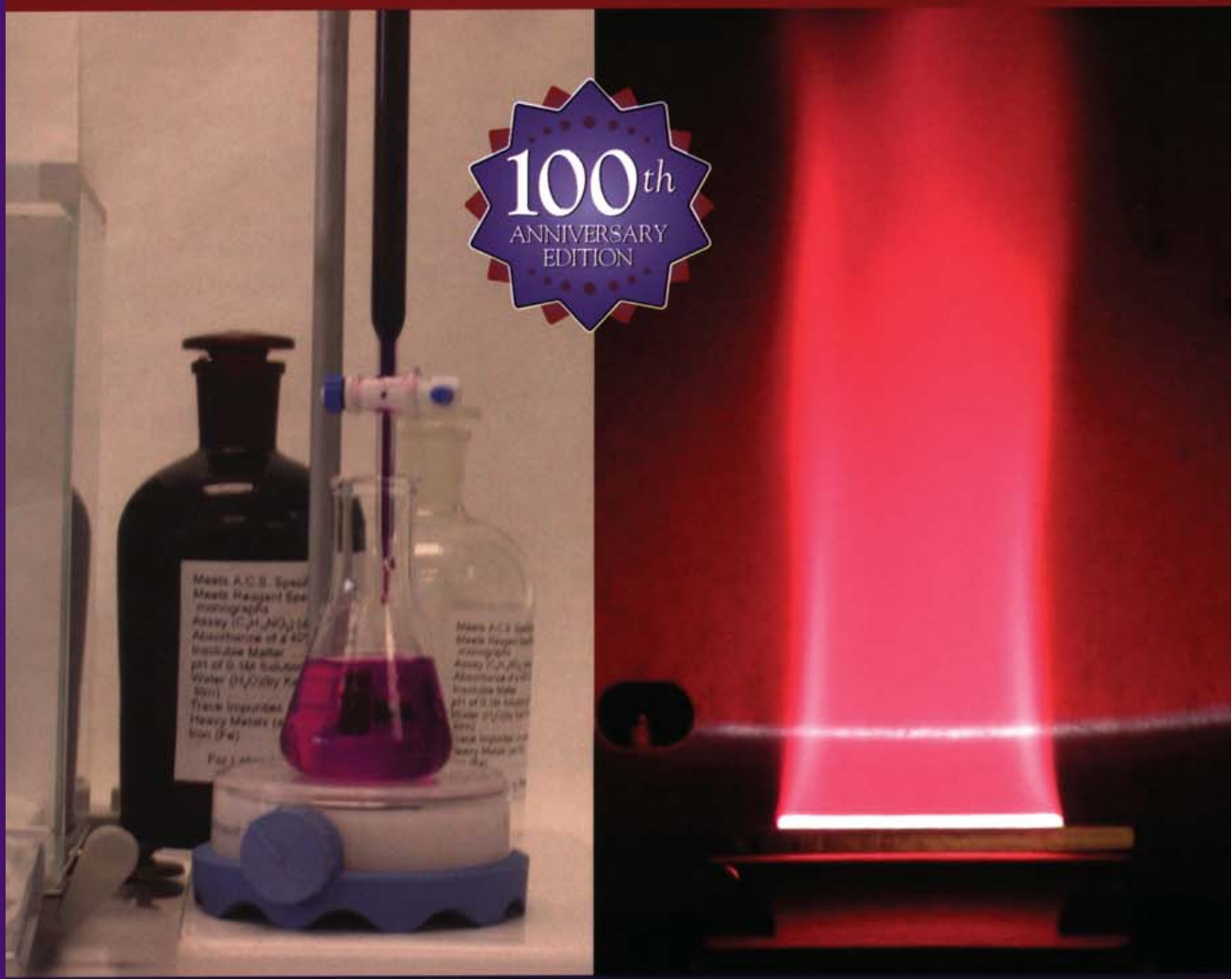

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REAGENT CHEMICALS

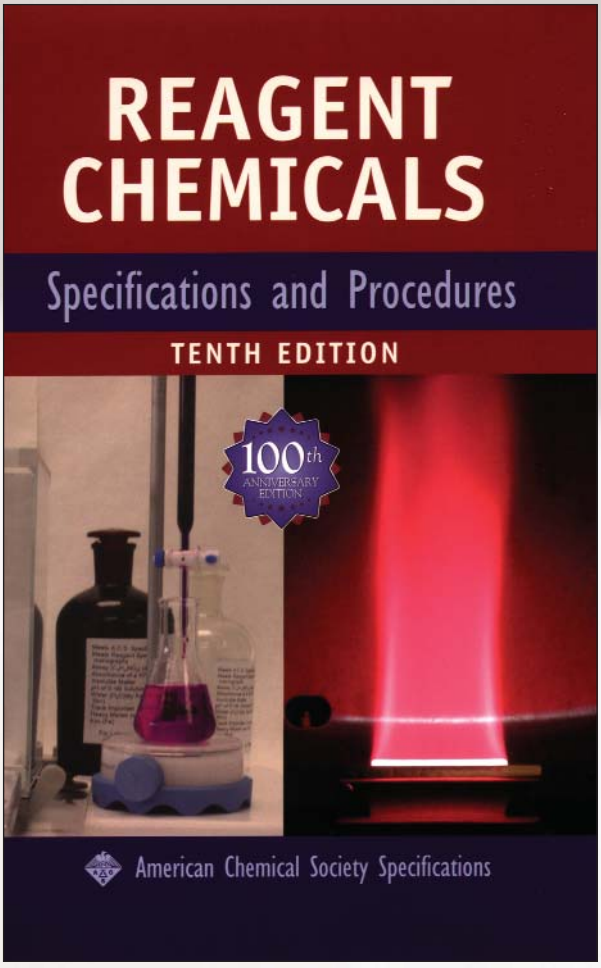
Specifications and Procedures • *10th Edition*

ACS Committee on Analytical Regeants

This rigorous volume, with its many specifications, up-to-date procedures, and constant improvement through years of research, is an essential tool in assuring the required quality of your reagent chemicals. The American Chemical Society Committee on Analytical Reagents is the only organization in the world that sets requirements and develops validated methods for determining the purity of analytical reagents. For the first time, *Reagent Chemicals, 10th Edition* includes general physical properties and analytical uses for all reagent chemicals—nearly 500 chemicals in all. This edition introduces 32 new reagents and three new classes of standard grade reference materials. In addition, the use of Inductively Coupled Plasma Mass Spectrometry (ICP-MS), which is recognized as the most powerful and flexible trace element technique, is now accepted as an analytical method in the 10th edition. Other improvements include a CAS number index, a separate index for standard grade reference materials, updated atomic weights, frequently used mathematical equations, complete assay calculations with titer values, a tutorial on how to read a monograph, and detailed tables of contents introducing each section.

Highlights from the 10th Edition:

- Sets purity specifications for almost 500 reagent chemicals and over 500 standard-grade reference materials
- Provides general physical properties and analytical uses for all reagent chemicals
- Features 32 new reagents and 3 new classes of standard-grade reference material
- Introduces Inductively Coupled Plasma Mass Spectrometry (ICP-MS) as a new method for performing trace metal determinations
- Includes a CAS number index, a separate index for standard grade reference materials, updated atomic weights, frequently used mathematical equations, and a tutorial on how to read a monograph



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**Please note: Standards will be official as of January 1, 2006.*

REAGENT CHEMICALS

Specifications and Procedures • *10th Edition*

ACS Committee on Analytical Regeants

Contents

About the Authors:

The American Chemical Society Committee on Analytical Reagents sets the specifications for most chemicals used in analytical testing. Currently, ACS is the only organization in the world that sets requirements and develops validated methods for determining the purity of reagent chemicals. These specifications have also become the de facto standards for chemicals used in many high-purity applications. Publications and organizations that set specifications or promulgate analytical testing methods—such as the United States Pharmacopeia and the U.S. Environmental Protection Agency—specify that ACS reagent-grade purity be used in their test procedures.

A WEALTH OF USEFUL INFORMATION AT YOUR FINGERTIPS

*Each **monograph** begins with the common name for the reagent.*

The reagent's formula, formula weight, and CAS number are included.

If there is a common alternate name, it is listed below.

*The **General Description** section contains useful (but background) information for each reagent. For more about this section, see page 7.*

*The **Specifications** section lists the purity requirements for a reagent. These must be met for a reagent to be "ACS Reagent Grade." For more about this section, see pages 9–12.*

*The **Tests** section outlines the procedure for verifying that a reagent meets the specifications.*

Solutions in the tests identified as "reagent," "indicator," "buffer," or "volumetric" are described in Part 3 of this book.

Assays by titration include both the classical statement of milliequivalents and the calculation formula.

Special solutions not included in Part 3 are listed within or following each test.

Some tests are described completely in the monograph.

Other test methods are described in Part 2 of this book. Page cross references lead to a detailed discussion of the procedure.

Ceric Ammonium Nitrate

Ammonium Hexanitratocerate(IV)

$(NH_4)_2Ce(NO_3)_6$ **Formula Wt 548.22** **CAS No. 16774-21-3**

GENERAL DESCRIPTION

Typical appearance: orange-red or orange-yellow solid
Analytical use: oxidimetric standard
Aqueous solubility: 141 g in 100 mL

SPECIFICATIONS

	Maximum Allowable
Assay	≥98.5% $(NH_4)_2Ce(NO_3)_6$
Insoluble in dilute sulfuric acid	0.05%
Chloride (Cl)	0.01%
Phosphate (PO_4)	0.02%
Iron (Fe)	0.005%

TESTS

Assay. (By titration of oxidative capacity of Ce^{IV}). Weigh accurately 2.4–2.5 g, and dissolve in 50 mL of water. From a pipet, add 50 mL of 0.1 N ferrous ammonium sulfate volumetric solution, and swirl until the precipitate that forms is redissolved. Add 10 mL of phosphoric acid and 0.10 mL of diphenylaminesulfonic acid, sodium salt, indicator solution (described below). Titrate at once with 0.1 N potassium dichromate volumetric solution to a change from faint green to violet. Record this titration volume as *A* mL. Pipet 25 mL of 0.1 N ferrous ammonium sulfate volumetric solution into 50 mL of water, and treat it in the same way (with phosphoric acid and indicator but without sample). Titrate to a change from green to gray-blue, and record this titration volume as *B* mL. One milliliter of 0.1 N ferrous ammonium sulfate corresponds to 0.05482 g of $(NH_4)_2Ce(NO_3)_6$.

$$\%\ (NH_4)_2Ce(NO_3)_6 = \frac{[(2B - A\ \text{mL}) \times N\ K_2Cr_2O_7] \times 54.82}{\text{Sample wt (g)}}$$

Diphenylaminesulfonic Acid, Sodium Salt, Indicator Solution. Dissolve 0.10 g of the salt in 100 mL of water.

Insoluble in Dilute Sulfuric Acid. To 5.0 g, add 10 mL of sulfuric acid, stir, and then cautiously add 90 mL of water to dissolve. Heat to boiling, and digest in a preconditioned covered beaker on a hot plate (≈100 °C) for 1 h. Filter through a tared filtering crucible, wash thoroughly, and dry at 105 °C.

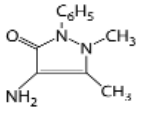
Chloride. (Page 35). Use 0.10 g dissolved in 10 mL of water. The comparison is best made by the general method for chloride in colored solutions.

Phosphate. (Page 40, Method 2). Dissolve 0.25 g in 30 mL of dilute sulfuric acid (1 + 9), add hydrogen peroxide until the solution just turns colorless, then boil to destroy excess

Figure 1-1. Features of a typical reagent monograph.

4-Aminoantipyrine

Ampyrone



$C_{11}H_{13}N_3O$ **Formula Wt 203.25** **CAS No. 83-07-8**

GENERAL DESCRIPTION

Typical appearance: yellow solid
Analytical use: indicator for trace phenol determinations in water
Change in state (approximate): melting point, 107 109 °C
Aqueous solubility: soluble

SPECIFICATIONS

Assay	≥98.0% $C_{11}H_{13}N_3O$
Sensitivity to phenol	Passes test
	Maximum allowable
Residue after ignition	0.1%
Loss on drying	0.5%

TESTS

Assay. (Total alkalinity by indirect nonaqueous titration). Weigh accurately about 0.8 g of sample, and dissolve in 100 mL of glacial acetic acid. Add 50.0 mL of 0.1 N perchloric acid in glacial acetic acid volumetric solution and 0.05 mL of crystal violet indicator solution. Titrate the excess perchloric acid with 0.1 N sodium acetate in glacial acetic acid volumetric solution to a violet end point. One milliliter of 0.1 N $HClO_4$ corresponds to 0.02033 g of 4-aminoantipyrine.

$$\% C_{11}H_{13}N_3O = \frac{[(mL \times N\ HClO_4) - (mL \times N\ CH_3COONa)] \times 20.33}{\text{Sample wt (g)}}$$

Sensitivity to Phenol. Prepare a 500-mL water blank and 500-mL phenol standards containing 0.005 mg (described below), 0.010 mg, 0.020 mg, 0.030 mg, 0.040 mg, and 0.050 mg of phenol. To the blank and standards, add 12.0 mL of 0.5 N ammonium hydroxide (described below), and adjust the pH of each to 7.9 ± 0.1 with phosphate buffer solution (described below) using a suitable pH meter. About 10 mL of the phosphate buffer is required. Transfer to a 1-L separatory funnel, add 3.0 mL of 4-aminoantipyrine sample solution (described below), mix well, add 3.0 mL of potassium ferricyanide solution (described below), mix well, and let the color develop for 3 min. The solutions should be clear and light yellow. Extract each solution immediately with 25.0 mL of chloroform; shake the separatory funnel at least 10 times. Allow the layers to separate, and shake again at least 10 times. Allow to fully separate. Draw off and filter the chloroform extracts through filter paper, and collect the dry extracts. Determine the absorbance of the extracts using a suitable spectrophotometer at 460 nm. Run the standards in 1-cm cells, using the blank as the reference. Plot the absorbance against mg phenol concentration. The graph should be linear with a correlation greater than 0.99.

Phenol Standard (0.005 mg of C_6H_5OH in 1 mL). Dissolve 0.50 g of solid phenol in water, and dilute to 1L. Dilute 10.0 mL of this solution to 1L.

Ammonium Hydroxide Solution, 0.5 N. Dilute 3.5 mL of fresh concentrated ammonium hydroxide to 100 mL with water.

4-Aminoantipyrine Sample Solution. Dissolve 2.0 g of sample in water, and dilute to 100 mL. Prepare at time of use.

Potassium Ferricyanide Solution. Dissolve 8.0 g of $K_3Fe(CN)_6$ in water, and dilute to 100 mL. Filter and store in an amber glass bottle. Prepare fresh weekly.

Phosphate Buffer Solution. Dissolve 10.45 g of potassium phosphate, dibasic, and 7.23 g of potassium phosphate monobasic in water, and dilute to 100 mL.

Residue after Ignition. (Page 26). Ignite 1.0 g.

Loss on Drying. (Page 26). Use 1 g. Dry at 90 °C.

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