Designation: E 50 – 00

Standard Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials[1]

This standard is issued under the fixed designation E 50; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These practices cover laboratory apparatus and reagents that are required for the chemical analysis of metals, ores and related materials by standard methods of ASTM. Detailed descriptions of recommended apparatus and detailed instructions for the preparation of standard solutions and certain nonstandardized reagents are included. An identifying number has been assigned each apparatus (see Section 8) and reagent (see Section 25) for convenience of reference in the analytical methods. Included also are general recommendations on the purity of reagents and protective measures for the use of hazardous reagents.

1.2 The aim of these recommendations is to present descriptions of such apparatus and reagents as are common to several ASTM methods, and thus avoid needless repetition. No attempt has been made to provide a description of every apparatus and reagent prescribed in ASTM methods for the chemical analysis of metals. Other apparatus and reagents that are required will be found listed or specified in the individual methods of analysis.

1.3 These recommendations are intended to apply to the ASTM methods of chemical analysis of metals when definite reference is made to these practices, as covered in Section 3.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards are given in Section 7 and Note 2.

NOTE 1—The use of the verb "shall" (with its obligatory third person meaning) in this standard has been confined to those aspects of laboratory safety where regulatory requirements are known to exist. Such regulations, however, are beyond the scope of these practices.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]
E 1 Specification for ASTM Thermometers[3]
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[4]
E 77 Test Method for Inspection and Verification of Liquid-in-Glass Thermometers[3]
E 100 Specification for ASTM Hydrometers[3]
E 126 Test Method for Inspection and Verification of Hydrometers[3]
E 128 Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use[5]
E 287 Specification for Burets[5]
E 288 Specification for Volumetric Flasks[5]
E 438 Specification for Glasses in Laboratory Apparatus[5]
E 542 Practice for Calibration of Volumetric Ware[5]
E 694 Specification for Volumetric Ware[5]
E 969 Specification for Glass Volumetric (Transfer) Pipets[5]
E 1044 Specification for Glass Serological Pipets (General Purpose and Kahn)[5]
E 1621 Guide for X-Ray Emission Spectrometric Analysis[6]

3. Significance and Use

3.1 The inclusion of the following paragraph, or a suitable equivalent, in any standard (preferably after the section on Scope) is due notification that the apparatus and reagents required in that standard are subject to the recommendations set forth in these practices.

> "*Apparatus and Reagents*—Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in ASTM Practices E 50, for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials."

3.2 It is assumed that the users of these practices will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly-equipped laboratory.

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[1] These practices are under the jurisdiction of ASTM Committee E1 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E 01.20 on Fundamental Practices and Measurement Traceability.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.
[3] *Annual Book of ASTM Standards*, Vol 14.03.
[4] *Annual Book of ASTM Standards*, Vol 15.05.
[5] *Annual Book of ASTM Standards*, Vol 14.04.
[6] *Annual Book of ASTM Standards*, Vol 03.06.

4. Purity of Water and Reagents

4.1 *Water*—Unless otherwise indicated, references to water are understood to mean reagent water of Type II grade, as defined by Specification D 1193.

4.2 *Reagents*—Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society when such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. In addition to this, it is desirable in many cases for the analyst to ensure the accuracy of his results by running blanks or checking against a comparable sample of known composition.

5. Reagents

5.1 *Concentrated Acids, Ammonium Hydroxide, and Hydrogen Peroxide*—When acids, ammonium hydroxide, and hydrogen peroxide are specified by name or chemical formula only, it is understood that concentrated reagents of the specific gravities or concentrations shown in Table 1 are intended. The specific gravities or concentrations of all other concentrated acids are stated wherever they are specified.

5.2 *Diluted Acids and Ammonium Hydroxide*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of the concentrated reagent to be diluted with a given number of volumes of water, as in the following example: HCl (5 + 95) means 5 volumes of concentrated HCl (sp gr 1.19) diluted with 95 volumes of water.

5.3 *Standard Solutions*—Concentrations of standard solutions are stated as molarities or normalities, expressed decimally; or the equivalent of 1 mL of solution in terms of grams, milligrams, or micrograms of a given element expressed as "1 mL = x.xx—g, mg, or µg of..."

5.4 *Nonstandardized Solutions*—Concentrations of nonstandardized solutions prepared by dissolving a given weight of the solid reagent in a solvent are specified in grams of the salt as weighed out per litre of solution, and it is understood that water is the solvent unless otherwise specified. For example, to prepare barium chloride solution (100 g/L) dissolve 100 g of barium chloride ($BaCl_2 \cdot 2H_2O$) in water and dilute to 1 L. In the case of certain reagents, the concentration may be specified as a weight percent. For example, H_2O_2 (3 %) means a solution containing 3 g of H_2O_2 per 100 g of solution. Other nonstandardized solutions may be specified by name only and the designation of the concentration of such solutions will be governed by the instructions for their preparation.

6. Laboratory Ware (1,2)[8,9]

6.1 *Glassware*—Unless otherwise stated all analytical methods are carried out in borosilicate glassware.

6.1.1 *Tolerances*—All glass apparatus and vessels used in analytical work must be carefully selected and calibrated to meet the particular requirements for each operation. Standard volumetric flasks, burets, and pipets must be of Class A or B within the tolerances established by the National Institute of Standards and Technology and ASTM.[9]

6.1.2 *Types*—Glasses are available which include colored glass for the protection of solutions affected by light, alkali-resistant glass, and high-silica glass having exceptional resistance to thermal shock. Standard-taper, interchangeable, ground-glass joints are very useful in analytical work.

6.2 *Plastic Labware*:

6.2.1 *Tolerances*—All plastic apparatus and vessels used in analytical work must be calibrated to meet the particular requirements for each operation. Standard volumetric flasks, burets, and pipets must be of precision grade within the tolerances established by the National Institute of Standards and Technology for the corresponding types of glassware (see 6.2.4).

6.2.2 *Physical Properties*—There are a number of physical properties which influence the usefulness of plastic labware (Table 2).

6.2.3 *Compatibility*—Many reagents can affect the strength, flexibility, surface appearance, color, dimensions, or weight of

TABLE 1 Concentration of Acids, Ammonium Hydroxide, and Hydrogen Peroxide

Name	Formula	Specific Gravity, Approximate	Reagent, Weight %		
			Nominal	Min	Max
Acetic acid	CH_3COOH	1.05	...	99.5	...
Formic acid	HCOOH	1.20	...	88.0	...
Hydrobromic acid	HBr	1.49	48	47.0	49.0
Hydrochloric acid	HCl	1.19	...	35.0	38.0
Hydrofluoric acid	HF	1.15	...	48.0	51.0
Nitric acid	HNO_3	1.42	...	69.0	71.0
Perchloric acid	$HClO_4$	1.67	...	70.0	72.0
Phosphoric acid	H_3PO_4	1.69	...	85.0	...
Sulfuric acid	H_2SO_4	1.84	...	95.0	98.0
Sulfurous acid	H_2SO_3	1.03	...	6.0(SO_2)	...
Ammonium hydroxide	NH_4OH	0.90	...	27.0(NH_3)	30.0 (NH_3)
Hydrogen peroxide	H_2O_2	1.10	30	28.0	...

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[7] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia." United States Pharmacopeial Convention, Rockville, MD 20852.

[8] The boldface numbers in parentheses refer to the list of references at the end of these practices.

[9] For further information the following ASTM Standards may be consulted: Volumetric Labware: Specifications E 287, E 288, E 438, Practice E 542, Specifications E 694, E 969, and E 1044. Thermometers: Specification E 1, and Test Method E 77. Hydrometers: Specification E 100 and Test Method E 126.

TABLE 2 Physical Properties of Plastic Labware[A]

Plastic[B]	Temperature Limit,° C	Specific Gravity	Brittleness Temperature,° C	Water Absorption, %	Flexibility	Transparency
CPE	80	0.92	−100	<0.01	excellent	translucent
LPE	120	0.95	−196	<0.01	rigid	translucent
PA	130	0.90	−40	<0.02	slight	translucent
PP	135	0.90	0	<0.02	rigid	translucent
PMP	175	0.83	−20	<0.01	rigid	clear
FEP	205	2.15	−270	<0.01	excellent	translucent
TFE	315	2.2	−265	<0.01	excellent	translucent
PC	135	1.20	−135	0.35	rigid	clear
SA	95	1.07	−25	0.23	rigid	clear
ETFE	180	1.70	−100	0.1	moderate	translucent

[A] From the publications of the Nalgene Labware Div., Nalge Sybron Corp.

[B] CPE, conventional (low density) polyethylene; LPE, linear (high density) polyethylene; PA, polyallomer (ethylene propylene copolymer); PP, polypropylene; PMP, polymethylpentene; FEP, fluorinated ethylene propylene; TFE, fluorinated ethylene; PC, polycarbonate; SA, styrene-acrylonitrile; ETFE, ethylene-tetrafluoroethylene copolymer.

plastics. The two basic modes of interaction that can cause these changes are described in 6.2.3.1-6.2.3.4.

6.2.3.1 *Chemical*—The analytical reagents can react with the polymer chain by oxidation, by attack on functional groups in or on the polymer molecule, or by depolymerization with a resultant deterioration in physical properties.[10]

6.2.3.2 *Physical*—Absorption of solvents in the plastic can result in softening, swelling, and permeation of the solvent through the plastic. No room temperature solvents are known for the polyolefins, however, it is better not to use them to store reagents. Reagents such as NH_3, Br_2, H_2S, and nitrogen oxides may be absorbed from reagent solutions by the plastic and become a source of error by subsequent release when the vessel is used for a different analysis.[11] Atmospheric contaminants may diffuse through the plastic and spoil contained reagents or samples. Other polymer types may dissolve in some solvents. Plastic labware may crack from interaction of a "stress cracking agent" (present, possibly in the solution to be analyzed) with molded-in stresses. This is, however, a long-time phenomenon and is normally not a factor in analytical work because contact times usually are limited and the labware is washed regularly.

6.2.3.3 Some plastics may contain small concentrations of metals used as catalysts during manufacture. Such metals may dissolve in the analytical reagent system and cause interference, particularly when small amounts of metals are to be determined.

6.2.3.4 A general indication of the effect of individual reagents can often be obtained from manufacturers' publications. It is important, of course, to consider that exposure time, temperature, concentration, and other reagents in the system may alter the effects of a given reagent on a given plastic. Because of these factors, the plastic labware must be thoroughly tested under the conditions of the method.[10] The type of plastic labware (see footnote *B* of Table 2) will be found specified in the method as well as any special precautions for its use.

6.2.4 *Precautions*—Most plastic labware must not be used with strong oxidants at elevated temperatures; or exposed to localized or general temperature above the limits in Table 2.[12] With proper precaution polytetrafluoroethylene labware may be used with strong oxidizing agents at elevated temperatures (see Table 2). For the best performance new volumetric ware should be rinsed with a mild detergent according to the directions of the manufacturer before using. Plastic volumetric ware shrinks slightly as it ages; therefore, it must be recalibrated periodically. Interior surfaces of volumetric ware should not be cleaned by abrasive action.

7. Hazards (3-8)

7.1 *General Requirements*—Nearly all procedures carried out in the chemical laboratory are potentially hazardous. Each of the procedures used in these methods of chemical analysis of metals has been safely performed many times in a number of laboratories. Specific warnings are given in the methods when unusually hazardous steps are required, but the analyst must rely on his own knowledge and skill to avoid the common hazards. The following general concepts have been developed through many years of industrial laboratory operations:

7.1.1 Each person who works in a chemical laboratory should protect himself and others from harm. Each individual should adopt an attitude of anticipating potential hazards and planning means for reducing the associated risk to a tolerable level. This involves the proper implementation of approved procedures, personal protective equipment, and risk management policy.

7.1.2 The employer should provide proper physical facilities, equipment, materials and training to permit employees to work without exposure to undue hazard. The work environment should be engineered to minimize risk and control emergencies. Hoods with recommended face velocities, eyewash and emergency shower stations should be provided. Fire alarm and fire control equipment should be installed. All employer provided risk control equipment, including personal protective equipment, should be on a regular inspection schedule. Management should adopt proper rules to promote safety by establishing low risk operating practices, good housekeeping, and proper personnel behavior. These rules should be

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[10] From the publications of the Nalgene Labware Div., Nalge Sybron Corp.

[11] Theobald, L. S., *Analyst*, Vol 84, 1959, p. 570.

[12] Special care should be used with fluorinated materials, because at temperatures around 250°C traces of possibly hazardous vapors may be emitted. Heat in a hood or well-ventilated area.

enforced consistently and impartially. Employees shall be advised of potential hazards in accordance with applicable federal, state, and local laws and regulations.

7.1.3 Ordinary industrial hazards (such as those which cause thermal burns, slips and falls, electrical shocks, and physical injury from equipment failure or contact with stationary or moving objects) can exist in laboratories along with special chemical hazards. Employee training programs, periodic facilities inspections, and the establishment of low risk practices and procedures may be helpful in minimizing these dangers.

7.2 *Safety Plan*—Every analytical chemistry laboratory shall have a written safety plan. If the laboratory is a part of a larger facility, its plan should be a part of (or coordinated with) the overall safety plan of the larger organization. A safety plan addresses at least the following topics:

7.2.1 Definitions of areas and personnel covered,

7.2.2 General safety rules,

7.2.3 Rules covering specific areas of operations,

7.2.4 Plans and procedures for damage and injury control activities such as, building evacuations and fire fighting,

7.2.5 Lists of safety equipment according to location and type,

7.2.6 Plans for periodic safety and equipment inspections, and personnel safety training.

7.2.7 Descriptions of the duties and identities of personnel who will implement and carry out the provisions of this plan.

7.3 *Personal Protective Equipment*:

7.3.1 *Eye Protection*—Laboratory areas where chemicals are used shall be designated and appropriately posted as eye protection areas. Safety glasses with solid side shields or plastic goggles shall be required for all workers and visitors in these areas.

7.3.2 *Hand Protection*—A variety of glove types afford hand protection for different types of hazard. Rubber gloves are available in a variety of compositions that show differing forms of chemical resistance. For example, nitrile and neoprene rubbers are suitable for work with acids but show poor resistance to some organic solvents. Other materials provide protection from hot objects, cryogenic liquids, or abrasion. The appropriate style and type must be selected for each application. Gloves should be inspected before and decontaminated after each use.

7.3.3 *Miscellaneous Protective Equipment*—The following is a listing of some of the additional personal protective equipment that may be expected to find need in the metals analysis laboratory:

7.3.3.1 Face shields, portable shields, hood sash shields,

7.3.3.2 Ear plugs, sound barrier ear muffs,

7.3.3.3 Lab coats, lab aprons, sleeve protectors,

7.3.3.4 Respirators, gas masks, self-contained breathing apparati, and

7.3.3.5 Safety shoes/boots.

7.4 *Laboratory Equipment*—This section lists common hazards associated with laboratory instruments and equipment. Reference works provide low risk practices and procedures. Suppliers and manufacturers should be consulted for specific information concerning the safe use and maintenance of their products.

7.4.1 Glass is a substance widely used in laboratory equipment. If mishandled, it can shatter into pieces with sharp edges that can inflict serious injury. Its use in pressure and vacuum systems is particularly hazardous.

7.4.2 Electrically operated equipment should always be installed in accordance with applicable local electrical codes, following the manufacturer's instructions. Proper grounding is especially important to prevent electrically conductive cabinets or cases from becoming dangerously charged if an internal short occurs. Electrical interlocks that prevent access to energized internal circuits should be kept in good operating condition and should never be defeated except as a part of carefully performed maintenance procedures. Lock-out/tag-out procedures shall be specified for any repair or maintenance operation that requires defeating electrical safety systems, or any other situation where equipment start-up could result in physical injury. Lock-out means the installation of a physical device (a lock with one key) that prevents re-energization. Tag-out means a prominently displayed clear warning sign that the equipment must not be re-energized. All personnel designated to perform such repair or maintenance shall have been adequately trained in lock-out/tag-out procedures.

7.4.3 Instruments that contain sources of radiation or radioactivity should be operated strictly in accordance with the manufacturer's instructions. Operation of instruments that produce X rays or other ionizing radiation shall conform to applicable local, state, and federal regulations (see Section 10 of Guide E 1621 for protective measures and references). Lasers, high-intensity arcs, sparks, plasmas, and ultraviolet sources can burn exposed skin. Eye protection should always be used. Commonly encountered sources of hazardous high intensity ultraviolet radiation include spectrographic emission sources, electrodeless discharge lamps, and nitrous oxide/acetylene flames.

7.4.4 Compressed gases in cylinders have the potential to cause severe damage and injury. If containers rupture or shatter, the stored energy is released suddenly with devastating force. A damaged cylinder or parts of a system and surrounding structures frequently become destructive projectiles. If the gas is toxic or explosive, its sudden release can quickly flood a vast area in a building with dangerous concentrations of the material. All inert gases present an asphyxiation hazard. The most commonly used inert gases are nitrogen, helium, argon and carbon dioxide. Of these, the latter two are a particular concern. Argon is difficult to clear from lung passages, once inhaled, and carbon dioxide in high concentrations can paralyze the respiratory response. Standard practice is to securely chain or strap a cylinder to a firm support at all times except when it is being moved. Transportation is by means of a specially designed wheeled cart with a security chain and the protective caps should always be screwed on securely when the cylinders are being moved. Storage should be in areas kept at moderate temperatures. Combustible and oxidizing gases should be separated both in storage and in use to reduce the possibility of accidental explosions or fires. Toxic gases should be stored and used in such a manner that normal or abnormally large releases do not endanger life. In use, all gases should be trapped or released in a way that does not endanger property or life. Caution is required to be certain that gases vented outside a building do not inadvertently reenter through ventilating or air-conditioning systems. Fittings, pressure regulators, gages, valves, and tubing should be designed to safely contain the specific gas and pressures to be used in the system. Suppliers of gases and related equipment provide information on the safe use of their products.

7.4.5 Operations that release flammable, corrosive, toxic, or noxious vapors, gases, dusts, or fumes should be conducted in a suitable hood. The hood proper, ducts, and blower parts should be constructed of a material that resists chemical corrosion, solvent action, or heat generated by the process. Exhaust stacks should be positioned to ensure that hood emissions do not reenter the building through ventilating or air-conditioning systems. Periodic inspections should be provided to ensure that efficient air movement is maintained and that no holes develop in the system. Specially constructed hoods should be used for operations producing perchloric acid fumes or mists. In addition to other design features, such hoods are equipped with water valves that allow the stack and hood areas to be flushed down periodically. A regular program of flushing the stack and hood areas must be established for such hoods. Such hoods should never be used for venting easily oxidizable materials (7.5.10).

7.4.6 Equipment operating above or below atmospheric pressure should be of special heavy-walled construction. Personnel should be protected from being struck by pieces of the system if it should accidentally explode or implode.

7.4.7 Modern instruments often employ hazardous technology. Such equipment is provided with electrical interlocks, guards, and shields to protect personnel from injury. The equipment should be operated and maintained as specified by the manufacturer. All parts of the equipment, including its safety features, should be in proper working order at all times that it is being operated. Maintenance should be performed by qualified personnel who have been trained to protect themselves and others from the specific hazards present in each system.

7.4.8 Emergency safety equipment should be stored where it is plainly visible and readily available to personnel who need it. The location and manner of storing such equipment requires careful planning. No temporary or permanent storage of equipment or material should be permitted to block access to any safety equipment. Personnel, when first assigned to a new area, should be instructed in the use of this equipment and should be reinstructed at appropriate intervals. The equipment should be inspected periodically to be sure it is in good operating condition. It should not be returned to its proper storage location after use until it is in proper condition for reuse. Examples of such equipment are emergency showers, eye wash stations, various classes of fire extinguishers, gas masks, self-contained breathing equipment, and spill control equipment.

7.5 *Reagents*—Reagent chemicals are normally used in small quantities and by personnel who have been instructed in their hazardous properties. Laboratories shall maintain a file of hazardous property data (Material Safety Data Sheets) for chemicals stored for use. Operating personnel shall have free access to the complete file at all times. However, since nearly all chemicals are hazardous under some circumstances, it is critically essential for all personnel to avoid inhaling or ingesting any chemicals and to permit no substances (with the exception of soap and water) to contact the skin. Some substances or combinations of substances are much more hazardous than others and are normally handled with gloves, protective clothing, barriers, or with other special precautions. Mouth pipetting should never be used. Because of the hazards of inadvertent contamination, it is prudent to establish and maintain a policy that forbids food, drink, tobacco, and cosmetic use in laboratories. A few of the more commonly used hazardous reagents are listed in the sections below. The analyst is cautioned to understand the properties of *any* reagent or combination of reagents before using them for the first time. Every step of a new procedure should be carefully planned, keeping in mind the potentially hazardous properties of the reacting materials and the resulting products. The plan should be designed for low-risk handling, even in the event of such unexpected occurrences as unusually rapid reactions, evolution of large quantities of gases, spillage, or accidental breakage or failure of equipment.

7.5.1 Storage of reagents, chemicals, and solvents should take into account their physical and chemical properties. The general classes of materials that should be stored separately are: bulk acids, strong oxidizers, volatile and flammable solvents, and water-sensitive materials. The latter (for example, calcium carbide and metallic sodium) should be stored where they cannot come in accidental contact with water from such sources as fire protection sprinklers, safety showers, accidental flooding, or leaks. Solvents and other highly flammable materials may require special explosion and fire-resistant storage.

7.5.2 All reagents should be considered hazardous, although some are much more dangerous than others. In many cases, inhalation, ingestion, skin contact, or combination thereof can lead to chronic or acute poisoning, and some chemicals have carcinogenic effects, or mutagenic effects on the unborn. In general, organic solvents have high vapor pressures at room temperatures, are flammable, and form explosive mixtures over a range of concentrations in air, and cause physiological changes in the human body if inhaled, ingested, or absorbed through the skin. Chloroform, carbon tetrachloride, and benzene are examples of solvents with known serious harmful effects. Smoking and open flames or sparking electrical equipment should not be permitted in areas where solvents are stored or used.

7.5.3 Beryllium and its compounds, dry or in solution, present a serious health hazard. Ingestion or inhalation of dusts or sprays containing these materials must be avoided.

7.5.4 Elemental mercury has an appreciable vapor pressure. Hazardous concentrations can build up in the air in enclosed spaces where liquid mercury is exposed. Standard practice is to store mercury in strong, tightly closed containers and to transfer mercury in such a manner that a spill can be contained and thoroughly cleaned up at once.

7.5.5 Mineral dusts that contain any of a number of heavy metals, asbestos, beryllium, chromium compounds, or fluorides are hazardous.

7.5.6 Hydrogen cyanide and alkali cyanides are very toxic substances and should be used in an efficient fume hood. Cyanides must be disposed of with care, avoiding contact with acid that releases highly toxic hydrogen cyanide gas.

7.5.7 Hydrogen sulfide is more toxic than hydrogen cyanide. It is readily detected at low concentrations because of its powerful "rotten egg" smell, but the sense of smell becomes a very unreliable means of detection at higher concentrations. Procedures in which hydrogen sulfide is used or produced (for example, acid dissolution of metal sulfides) should be performed in an efficient fume hood.

7.5.8 Nitric acid fumes and the reaction products of nitric acid with reducing agents (such as metals) are noxious and highly toxic. Reactions with nitric acid should be performed in an efficient fume hood.

7.5.9 The corrosive action of acids and bases on materials, including human tissues, is well known. It is standard practice to use eye protection and protective clothing when handling these materials.

7.5.10 Perchloric acid can be used safely, but only under carefully prescribed conditions. Dilute perchloric acid has the same hazardous properties as other strong acids, but the concentrated acid, especially when it is hot, reacts rapidly and often with violently explosive force with oxidizable materials. Only well-established procedures should be employed for perchloric acid oxidations and the procedures should be followed *exactly* as written. Specially designed hoods are specified for handling perchloric acid fumes and any hood in which perchloric acid may be fumed should not be used for other operations that permit easily oxidizable material to collect in the ducts or blower.

7.5.11 Hydrofluoric acid produces very serious burns which may or may not be painful on first contact. Such burns often damage bone and other tissue within the body. Standard procedure is to use gloves and protective clothing when handling this reagent. After the material is added, the closed container, gloves, and all surfaces that may later be touched are rinsed with large quantities of water. Even one drop of hydrofluoric acid on the skin or fingernail must receive immediate first-aid and medical attention should be promptly sought.

7.5.12 Halogens (fluorine, chlorine, bromine, and iodine) are hazardous materials. Procedures in which halogens are used or produced should be performed in an efficient fume hood. Bromine is the most commonly used halogen in the chemical analysis of metals. Liquid bromine vaporizes at room temperature; its fumes attack organic material and are highly irritating to eyes and lungs. The liquid causes burns and blisters. Inhalation, ingestion, and skin contact with both vapor and liquid must be avoided. Work only in an efficient exhaust hood with proper protective equipment. Familiarity with proper first-aid procedures is essential.

7.5.13 Hydrogen peroxide is commonly used at concentrations of 30 % and lower. At these concentrations the reagent is safer to handle than at higher concentrations but must still be treated as a very serious hazard; it is a very strong oxidizing agent, causes serious burns, and may decompose violently if contaminated.

7.5.14 Sodium peroxide is used both as an aqueous solution reactant and a molten salt flux. It is a very strong oxidant and must be considered a very serious hazard. In particular, sodium peroxide fusions with some sample materials may result in violent or explosive reactions. Such fusions should never be attempted with highly reactive or unknown samples. Sodium peroxide fusions must be performed following well-established procedures with samples of known reactivity and proper safety practices and equipment must be used.

7.5.15 *Spill Control*—Kits are commercially available for dealing with various types of chemical spills. It is also possible to assemble a variety of materials for dealing with such emergencies. Where such equipment is stored and who uses it should be specified in safety planning.

7.5.16 *Disposal of Laboratory Reagents*—As with all work in chemical laboratories, the chemical analysis of metals generates chemical wastes which must be disposed of by means which pose the least harm to health and the environment. All pertinent federal, state, and local laws and regulations shall be strictly followed.[13]

7.6 *Confined Space*—If repair, maintenance, or work of any kind requires personnel to enter a confined space, as defined by the applicable regulations, such personnel shall have been adequately trained in confined space safety procedures.

APPARATUS

8. General

8.1 Apparatus required in the methods of chemical analysis of metals, and referred to by number in the separate sections on apparatus which appear in the chemical methods preceding the procedures, must conform to the requirements prescribed below for the particular apparatus specified.[14]

9. Apparatus No. 1—Apparatus for Determination of Total Carbon by Direct-Combustion

9.1 The apparatus must be suitable for the direct combustion of the metal in purified oxygen and have a suitable purifying train following the furnace. For amounts of carbon above approximately 300 µg, the evolved CO_2 is collected in an absorbent of inert-base impregnated with NaOH. For amounts of carbon above approximately 1600 µg, an ordinary analytical balance may be used to weigh the absorption bulb. For smaller amounts special weighing techniques must be used. Fig. 1

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[13] A valuable reference on the subject of the disposal of laboratory wastes is: *Prudent Practices for Disposal of Chemicals from Laboratories*, National Research Council, National Academy Press, Washington, D.C. 1983.

[14] In addition to the requirements for apparatus given in these practices, reference should be made to the requirements set forth in the *Annual Book of ASTM Standards*, for the following: D 1100, "Specification for Filter Paper for Use in Chemical Analysis;" E 70, "Test for pH of Aqueous Solutions with the Glass Electrode;" E 145, "Specification for Gravity-Convection and Forced-Ventilation Ovens;" E 147, "Specification for Apparatus for Microdetermination of Nitrogen by Kjeldahl Method;" E 287, " Specification for Burets;" E 288, "Specification for Volumetric Flasks;" E 319, "Testing Single-Arm Balances;" E 438, " Specification for Glasses in Laboratory Apparatus;" E 617," Specification for Laboratory Weights and Precision Mass Standards;" E 675, "Specification for Interchangeable Stopcocks and Stoppers;" E 676, "Specification for Interchangeable Taper- Ground Joints."

shows a typical arrangement of the apparatus. Because of the diversity of apparatus by which correct results may be obtained in the determination of carbon, the recommendations given in 9.1.1-9.1.9 are intended to indicate what is acceptable rather than to prescribe definitely what must be used.

NOTE 2—**Caution:** Induction furnaces are capable of inflicting high-frequency burns and high-voltage shocks. All guards must be maintained properly. The furnace must be disconnected from the power line whenever electrical repairs or adjustments are made.

9.1.1 *Combustion Apparatus*—Any apparatus that will heat the sample to the required combustion temperature may be used. Furnaces with wire-wound heating elements generally are limited to a maximum temperature of 1200°C; resistance furnaces equipped with nonmetallic heating elements and induction furnaces (**Caution**, Note 2) and may be operated at temperatures above 1400°C. Some types of induction furnaces contain as integral units the oxygen purifier, catalyzer, particle filter, and carbon dioxide purifier (see 9.1.3, 9.1.7, 9.1.8, and 9.1.9).

9.1.2 *Combustion Tubes*—Tubes may be of porcelain, sillimanite, clay, quartz, or platinum and must be gas tight at operating temperatures. Quartz is susceptible to devitrification when used intermittently at temperatures above 1000°C and may then become porous. Tubes 760 mm (30 in.) long with an inside diameter of 32 mm (1¼ in.), tapered at one end, are generally used with resistance furnaces and are convenient for the small fused silica crucible that is used for the determination of graphitic carbon in cast iron.

9.1.3 *Oxygen Purifiers*—The purity of the oxygen must not be less than 99.5 %. It must be passed through two reduction pressure valves (approximately 207 kPa (30 psig) and 14 to 28 kPa (2 to 4 psig), respectively), or a suitable two-stage reduction valve to provide an even and adequate flow of oxygen. Organic matter is an undesirable impurity; therefore, the oxidation catalyst tube, *E* in Fig. 1, heated to a temperature above 450°C, must be used when the presence of organic contaminants in the oxygen is suspected. Usually organic contaminants are not present, and the catalyst tube *E* (Fig. 1) may be omitted.

9.1.4 *Boats and Covers*—The boats and covers may be of aluminum oxide, clay, zircon, nickel, or platinum. Nickel boats should be made of sheet nickel containing less than 0.02 % carbon. Before use, new boats and covers must be preheated at a minimum of 1100°C in oxygen or air for at least 15 min or until a constant blank is obtained. To prevent damage to the tube by spattering, a platinum, nickel, or ceramic cover or ceramic sleeve, open at both ends to allow free access of oxygen, should be placed over the boat.

9.1.5 *Material for Lining Boats*—Aluminum oxide, 60-mesh or finer, specially prepared for carbon determinations is satisfactory. Ignited low-silica chrome ore, or zirconium oxide, properly sized and freed from materials causing a blank, may be employed.

9.1.6 *Accelerators*—Accelerators are described in the respective methods.

9.1.7 *Catalysts for Resistance Furnace*—Ironized asbestos, copper oxide, platinized quartz or asbestos, or platinum gauze is placed in the exit end of the combustion tube when determining graphitic carbon in cast iron. In other instances when these catalysts are used to ensure complete combustion of carbon to carbon dioxide, it is questionable whether they are of any value except to serve as baffles to hold back finely divided solid metallic oxides, since they soon become fouled. When such baffles are used, the fixed sulfur should be burned out after not more than 200 samples have been run. This may be accomplished by drawing the exit end of the combustion tube into the hot zone while oxygen is passed through. When carbon determinations are made on materials containing high sulfur, baffles are not used in the exit end of the tube, but a special SO_2 absorber or purifier is placed between the tube and the CO_2 absorber.

9.1.8 *Catalyst for Induction Furnace*—A catalyst such as copper oxide heated to about 300°C must be used to ensure complete conversion of CO to CO_2.

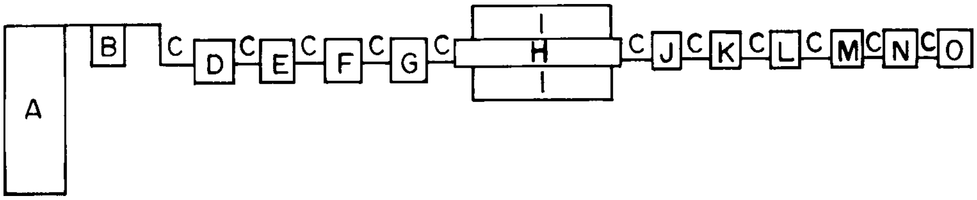
9.1.9 *Carbon Dioxide Purifiers*—The purifiers that follow the combustion tube must remove finely divided solid metallic oxides and oxides of sulfur and selenium, dry the gases before they enter the weighed CO_2 absorber, and protect the absorber from outside effects. Finely divided solid metal oxides are removed from the gases during their passage through the glass wool. The SO_2 given off by materials containing sulfur is removed by MnO_2, or by platinized silica gel heated to about 400°C. Water vapor is absorbed in the tube containing $Mg(ClO_4)_2$. Additional components in the purification train may be required when materials containing very high amounts of sulfur or of halides are being analyzed. A second tube containing MnO_2 may be used when the sulfur content is very high; MnO_2 has also been reported to remove chlorides. Chlorides may also be removed by pumice impregnated with a mixture of sodium thiosulfate and potassium iodide or by a tube containing 20 to 30-mesh zinc metal heated to 300 to 325°C. Other materials may be substituted for those listed; for example, potassium permanganate solution may replace the MnO_2, and H_2SO_4 may be used for removing water vapor, provided satisfactory results are obtained. The materials used in the purification train must be checked frequently to ensure that their absorbing capacity has not been exhausted.

9.1.10 *Gravimetric Determination of Carbon Dioxide by Absorption Bulbs*—No special types of absorption bulbs are recommended, although the Fleming, Miller, and Turner bulbs have proved satisfactory. When charged with absorbent, the bulbs must not weigh over 200 g, and they must always be weighed filled with oxygen. An adequate and uniform time interval between the combustion and the weighing must be maintained.

9.1.11 *Gravimetric Determination of Carbon Dioxide by Carbon Dioxide Absorbents*—The most desirable absorbent for CO_2 is 20 to 30-mesh inert base impregnated with NaOH, followed by anhydrous $Mg(ClO_4)_2$ at the exit end. The latter absorbs the water that is formed during the absorption reaction but not held by the unused CO_2 absorbent.

10. Apparatus No. 2—Filtering Crucible (9)

10.1 Fritted-glass filtering crucibles or porous-bottom porcelain filtering crucibles are recommended for general use. These crucibles are available in different sizes and porosities. When use above 150°C is specified, the porous porcelain



A — Cylinder of oxygen.
B — Reduction valve.
C — Rubber tubing.
D — Flowmeter.
E — Heated oxidation catalyst tube containing CuO or platinum to destroy organic contaminants or CO, if their presence is suspected in the oxygen.
F — Tower containing 20 to 30-mesh CO_2 absorber covered with a 13-mm (½-in.) layer of anhydrous magnesium perchlorate ($Mg(ClO_4)_2$) to remove moisture.
G — Manometer.
H — Combustion tube.
I — Electric furnace, resistance-type with appropriate temperature controls, or induction-type with appropriate power input indicating device.
J — Glass tube lightly packed with glass wool to remove solid particles.
K — Bottle containing asbestos impregnated with manganese dioxide (MnO_2), or heated platinized silica gel, to remove sulfur gases from the stream. For alternative purification trains see 9.1.9.
L — U-tube containing anhydrous $Mg(ClO_4)_2$. (For alternative purification trains see 9.1.9.)
M — Heated oxidation catalyst tube containing CuO or platinized silica gel to ensure complete conversion of CO to CO_2 when using an induction furnace. When using a resistance-type furnace for determining graphitic carbon in cast iron, ironized asbestos or a similar catalyst should be placed in the exit end of the combustion tube.
N — Absorption bulb with exit stopcock containing 20 to 30-mesh CO_2 absorber. A layer of glass wool is placed in the bottom and top of the bulb, and the CO_2 absorbent is covered with a layer of anhydrous $Mg(ClO_4)_2$ approximately 13 mm (½ in.) in thickness.
O — Flowmeter.

FIG. 1 Apparatus No. 1—Typical Arrangement for Determination of Carbon by the Direct-Combustion Method

crucibles should be used. Glass crucibles used at temperatures above 150°C require slower heating and cooling. Gooch crucibles, with fixed or removable perforated bottoms, made of porcelain, fused silica, or platinum and of the following sizes as specified, are also recommended: 10, 20, 25, and 35 mL.

10.1.1 *Purification of Asbestos for Gooch Crucibles*—Digest a good grade of commercial shredded asbestos in HCl on a steam bath for several hours. Filter on a Büchner funnel and wash with water. Repeat the acid digestion until soluble iron has been removed. Filter and wash the fibers to remove chlorides. If the Gooch crucible is to be used for a particular operation, it may be desirable to introduce at this point further treatments of the asbestos. For example, if the Gooch crucible is intended for use in the bismuthate method for manganese, digest the asbestos for 1 to 2 h with hot HNO_3 (1 + 2) after chlorides have been washed out, to make sure of the elimination of the chlorides and to oxidize traces of ferrous iron. If the Gooch crucible is to be used in combustions for carbon, dry the asbestos, ignite it under oxidizing conditions until all carbonaceous matter has been consumed, and again mix with water.

10.1.2 *Preparation of Filter Pad for Gooch Crucible*—Prepare three sizes of asbestos suspended in water: coarse, medium, and fine. Pour some of the coarse asbestos into the crucible and drain. Then add the medium fibers and drain under gentle suction. Tamp down gently, especially at the sides, with the flattened end of a glass rod. Finally, add a layer of the fine asbestos and drain under stronger suction. Then wash with distilled water and drain under full suction until no fibers pass into the filtrate. The pad should be 2 to 3 mm thick and weigh 50 to 100 mg.

11. Apparatus No. 3—Apparatus for pH Measurements and Potentiometric Titrations

11.1 See Willard, Merritt, Jr., and Dean (**10**) for an excellent discussion of potentiometers for pH and potentiometric measurements.

11.1.1 *Apparatus No. 3A for pH Measurement*—A number of pH meters are commercially available. Many of these instruments can accept a variety of electrodes and therefore can be used also for potential measurements. Although both line- and battery-operated pH meters are manufactured, the former is recommended for laboratory work because this type of pH meter contains an electronic or transistorized potentiometer which makes the emf balancing operation entirely automatic. Electrometer tube input is used on both the electronic and transistorized pH meters.

11.1.1.1 The pH meter must have electrode standardization (or asymmetry potential) and manual or automatic temperature compensation controls. The dial must read in pH directly, and permit readings that are accurate to at least ±0.01 pH unit. For higher accuracies it is recommended that a pH meter with an expanded scale be used.

11.1.1.2 Because there is no accurate method for determining the absolute potential of an individual electrode, two electrodes are used for pH measurements. These are called the reference and indicator electrodes. By international agreement the hydrogen electrode is the standard indicator electrode for pH, but it is inconvenient to use and subject to several limitations. The most widely used reference electrode is the saturated calomel electrode. It is most often used as a pencil-type unit that is immersed directly in the solution, but may also be utilized as an external cell (to prevent possible contamination) contacting the solution by means of a salt bridge. The silver - silver chloride reference electrode is also convenient to use, but it is more difficult to prepare than the saturated calomel electrode. The mercurous sulfate reference electrode may be used in solutions in which the chloride ions that diffuse out of the calomel cell might be harmful.

11.1.1.3 The most commonly employed indicator electrode is the glass electrode. The quinhydrone and antimony - antimonous oxide electrodes are used to a much less extent. Combination electrodes containing both the indicator and reference units are also available. The tips of the electrodes containing solutions must be covered with rubber caps when the electrodes are disconnected from the meter and stored. When pH measurements are not being made the electrodes connected to the pH meter should be kept in a beaker containing water. Prior to measuring the pH of a solution the electrodes must be thoroughly washed with water especially if they have been left standing for a long period of time.

11.1.2 *Apparatus No. 3B for Potentiometric Titrations*—Instruments for detecting the end points in pH (acid-base), oxidation-reduction, precipitation, and complexation titrations consist of a pair of suitable electrodes, a potentiometer, a buret, and a motor-driven stirrer. Titrations are based on the fact that when two dissimilar electrodes are placed in a solution there is a potential difference between them. This potential difference depends on the composition of the solution and changes as the titrant is added. A high-impedance electronic voltmeter follows the changes accurately. The end point of the titration may be determined by adding the titrant until the potential difference attains a predetermined value or by plotting the potential difference versus the titrant volume, the titrant being added until the end point has been passed.

11.1.2.1 An elaborate or highly sensitive and accurate potentiometer is not necessary for potentiometric titrations because the absolute cell voltage needs to be known only approximately, and variations of less than 1 MV are not significant. Such instruments should have a range of about 1.5 V and a readability of about 1 MV. Many of the pH meters satisfying the requirements for Apparatus No. 3A are also suitable for potentiometric titrations.

11.1.2.2 The electrode system must consist of a reference electrode and an indicator electrode. The reference electrode maintains a constant, but not necessarily a known or reproducible potential during the titration. The potential of the indicator electrode does change during the titration; further, the indicator electrode must be one that will quickly come to equilibrium.

11.1.2.3 Saturated calomel and silver-silver chloride half-cells may be used as reference electrodes. In oxidation-reduction titrations tungsten may be used as the reference electrode with platinum as the indicator electrode. Where the hydrogen-ion activity remains constant throughout the titration, the glass electrode may be used as the reference electrode with platinum as the indicator electrode for oxidation-reduction titrations or silver in argentometry for precipitation reactions. Graphite has also been used as a reference electrode.

11.1.2.4 In addition to the indicator electrodes already cited, the glass electrode is used for this purpose in acid-base titrations, but in rare instances a hydrogen, quinhydrone, or antimony electrode may be preferred. In oxidation-reduction reactions the bright platinum or platinum-rhodium alloy may be in the form of wire or foil. Mercury is used as the indicator electrode in certain complexometric titrations. Silver may be used for argentometric halide precipitation reactions.

11.1.2.5 Initially, a titration of the constituent in question is performed manually, and the volumes of titrant added and the corresponding potential differences are noted. By use of established techniques (**10**) the end point potential is determined. For the analytical determinations, titration may be continued to a preset potential, the end point being signaled by a null meter, with or without automatic termination of the titration. This technique is applicable to reasonably rapid reactions involving strong oxidants and reductants, precipitates not more soluble than silver chloride, and ionization constants greater than that of boric acid.

11.1.2.6 Other techniques may be used for both slow and fast reactions. These include automatic recording of the titration curve on a strip chart, and the recording of the titrant end point volume on a tape. In the latter, an adjustable print-out delay prevents undertitrating when the reaction is slow.

11.1.2.7 Further detailed requirements for this apparatus are given in Method E 70.

12. Apparatus No. 4—Apparatus for Determination of Sulfur by Evolution

12.1 *Apparatus No. 4A*—The apparatus shown in Fig. 2 may be used for determining sulfur by the evolution method in testing materials that are soluble in HCl (1 + 1). It should consist of a 300-mL Florence flask fitted with a "sulfur-free" two-hole rubber stopper carrying a thistle tube and a second glass tube dipping into a beaker containing ammoniacal $ZnSO_4$ or $CdCl_2$ solution.

13. Apparatus No. 6—Apparatus for Determination of Arsenic by Distillation

13.1 A simplified solution-distillation apparatus recommended for the determination of arsenic is shown in Fig. 3. The apparatus consists of a 300 or 500-mL Erlenmeyer flask with a standard-taper, ground-glass stopper carrying a distillation tube, a thermometer, and pressure regulator tube that comes within 3 to 6 mm (⅛to ¼ in.) of the bottom of the flask.

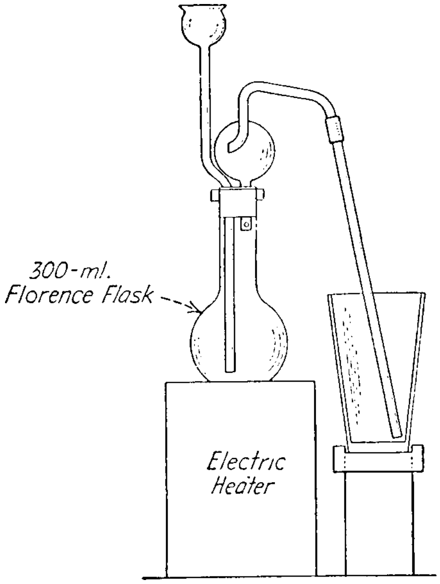


FIG. 2 Apparatus No. 4A for Determination of Sulfur by Evolution Method

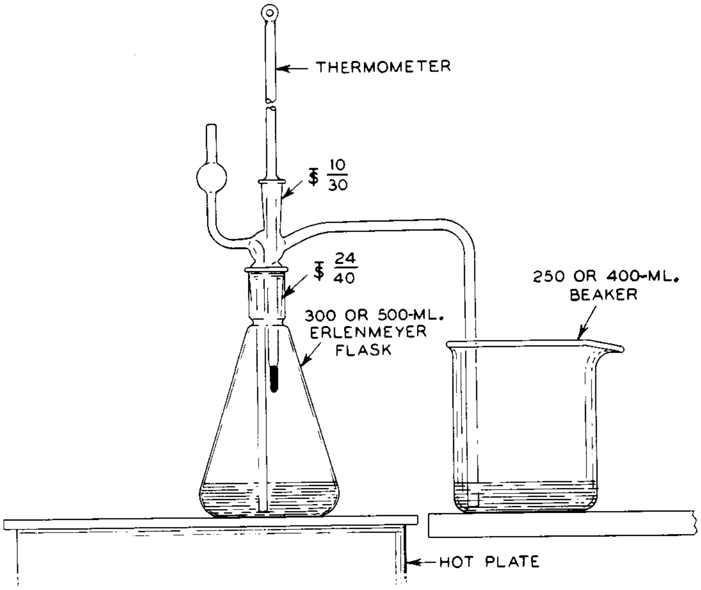


FIG. 3 Apparatus No. 6 for Determinations of Arsenic by Distillation

13.2 *Thermometer*—A 76-mm partial immersion thermometer, having a range from − 20 to + 150°C, conforming to the requirements for ASTM Partial Immersion Thermometer 1C as prescribed in Specifications E 1 is recommended.

14. Apparatus No. 7—Apparatus for Reduction of Tin

14.1 *Apparatus No. 7A*—When tin is to be reduced to the stannous state and determined by titration with standard iodine or iodate solution, air must be excluded during the reduction and titration to prevent oxidation of the stannous tin. This exclusion of air is usually accomplished by keeping the solution under a blanket of gaseous CO_2 and may be accomplished in a variety of ways. One of the simplest methods is by means of the apparatus shown in Fig. 4 in which the reduction of the tin solution is made in a flask capped with a rubber stopper containing an L-shape siphon tube. When reduction is complete, the end of the siphon is dipped into a saturated solution of $NaHCO_3$ and set aside to cool. When cool, the stopper is removed and the solution titrated.

14.2 *Apparatus No. 7B*—For work of high accuracy, it is best to keep the tin solution under gaseous CO_2. Fig. 5 shows one of the many forms of apparatus that may be used when gaseous CO_2 is employed. It consists of a flask closed with a three-hole rubber stopper containing an inlet tube for CO_2, an air condenser, and a hole for the buret (glass plugged). During reduction a very slow stream of CO_2 is passed through the flask. When reduction is complete, the flow is increased to maintain a protecting blanket of CO_2 during the cooling and titration.

15. Apparatus No. 8—Jones Reductor

15.1 The Jones reductor has the dimensional requirements shown in Fig. 6. It consists of a column 19 mm in diameter and 250 mm in length, of 20 to 30-mesh amalgamated zinc. To amalgamate the zinc, shake 800 g of zinc (as free of iron as possible) with 400 mL of $HgCl_2$ solution (25 g/L) in a 1-L flask for 2 min. Wash several times with H_2SO_4 (2 + 98), and then thoroughly with water. The reductor, when idle, should always be kept filled with distilled water to above the top of the zinc.

15.2 When the reductor is to be used for the determination of columbium in steel, the reducing column should be 19 mm in diameter and 760 mm in length. One filling should suffice for about six determinations of columbium.

16. Apparatus No. 9—Electrodes for Electroanalysis

16.1 *Electrodes*—Platinum electrodes of the stationary type are recommended as described in 16.1.1 and 16.1.2, but strict adherence to the exact size and shape of the electrodes is not mandatory. When agitation of the electrolyte is permissible in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed. The surface of the platinum electrodes should be smooth, clean, and bright to promote uniform deposition and good adherence. Sandblasting is not recommended (**11**).

16.1.1 *Cathodes*—Platinum cathodes may be formed either from plain or perforated sheets or from wire gauze, and may be

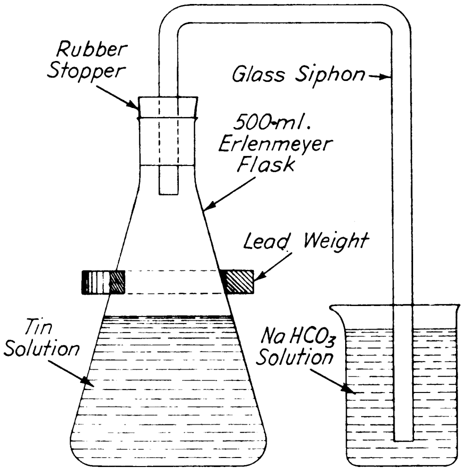


FIG. 4 Apparatus No. 7A for Reduction of Tin

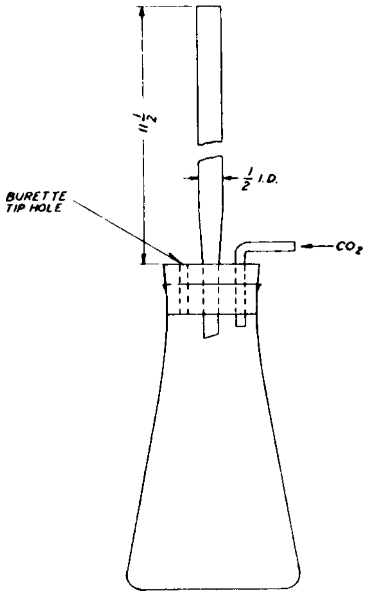


FIG. 5 Apparatus No. 7B for Reduction of Tin

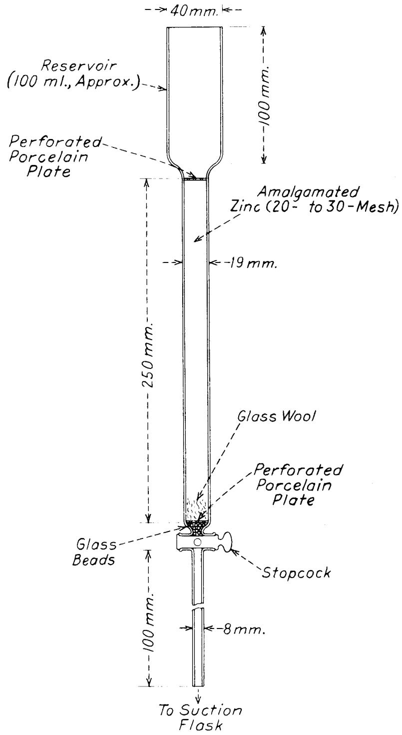


FIG. 6 Apparatus No. 8, Jones Reductor

either open or closed cylinders. Gauze cathodes are recommended, and shall be made preferably from 50-mesh gauze woven from wire approximately 0.21 mm (0.0085 in.) in diameter. The cathode should be stiffened by doubling the gauze for about 3 mm at the top and the bottom of the cylinder or by reinforcing the gauze at the top and bottom with a platinum band or ring. The cylinder should be approximately 30 mm in diameter and 50 mm in height. The stem should be made from a platinum alloy wire such as platinum-iridium, platinum-rhodium, or platinumruthenium, having a diameter of approximately 1.30 mm. It should be flattened and welded the entire length of the gauze. The over-all height of the cathode should be approximately 130 mm. A cathode of these dimensions will have a surface area of 135 cm^2 exclusive of the stem.

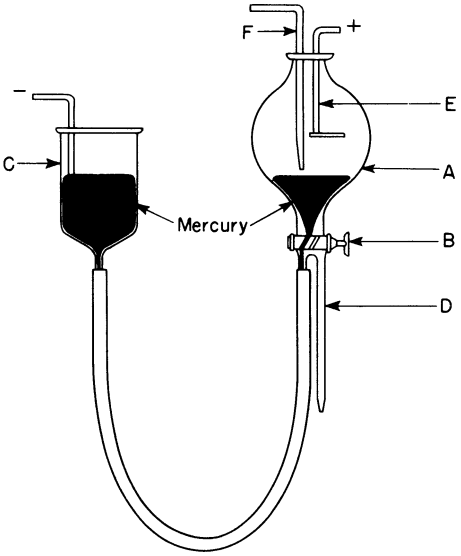
16.1.2 *Anodes*—Platinum anodes may be of the spiral type when anodic deposits are not being determined, or if the deposits are small (as in the electrolytic determination of lead when it is present in amounts not over 0.2 %). When used in analyses where both cathodic and anodic plates are to be determined, the anodes should be of wire gauze. Spiral anodes should be made from 1.00-mm or larger platinum wire formed into a spiral of seven turns having a height of approximately 50 mm and a diameter of 12 mm, the over-all height being approximately 130 mm. A spiral anode of this description will have a surface area of 9 cm^2. Platinum gauze anodes should be made of the same material and of the same general design as platinum gauze cathodes. The anode cylinder should be approximately 12 mm in diameter and 50 mm in height and the over-all height of the anode should be approximately 130 mm. A gauze anode of these dimensions will have a surface area of 54 cm^2. Both areas are exclusive of the stem.

17. Apparatus No. 10—Mercury Cathode Cell

17.1 The mercury cathode separation serves as a means of quantitatively removing iron, chromium, zinc, nickel, cobalt, tin, molybdenum, copper, bismuth, silver, and other ions from solution (**12**). Only partial separations of manganese, antimony, arsenic, and ruthenium are obtained (**13**). The separation is accomplished by reducing the ions electrolytically and causing them to dissolve in the mercury pool that serves as cathode. Electrolysis is generally carried out in a sulfuric acid or perchloric acid solution that is free of chlorides and nitrates. The presence of large quantities of salt is undesirable because it slows down the reduction process. The most rapid electrolysis is obtained if the solution pH is about 1.5 and if solution and mercury are stirred during the process. The d-c current should be 3 to 5 A with a voltage of 6 to 15 across each cell.

17.1.1 *Apparatus No. 10A*—A convenient form of mercury cathode cell is that recommended by Melaven (**14**), shown in Fig. 7. With this apparatus, the electrolyte is removed from the cell through the stopcock.

17.1.2 *Apparatus No. 10B*—An efficient apparatus for mercury cathode separations is that employing a rotating mercury pool cathode. With this instrument the movement of the cathode causes a fresh surface of mercury to be exposed during electrolysis, thus accelerating the separation. This instrument permits use of a current of 15 A in a 400-mL beaker. The electrolyte may be removed from the cell through a stopcock located just above the level of the mercury or siphoned from it. When 1 % or more of aluminum or titanium is present and these are to be determined, it should be initially ascertained if any of the aluminum or titanium is lost to the cathode.



A — Glass vessel.
B — Two-way stopcock, wide-bore.
C — Leveling bulb.
D — Glass tubing constricted in same manner as a buret tip.
E — Platinum gauze or coiled platinum wire.
F — Narrow glass tube for introduction of air to stir the electrolyte and mercury (a motor-driven stirrer can be used instead).

FIG. 7 Apparatus No. 10A, Melaven Type Mercury Cathode Cell

18. Apparatus No. 13—Apparatus for Determination of Sulfur by Direct Combustion

18.1 The apparatus must be suitable for the combustion of the sample in oxygen to form sulfur dioxide (SO_2) and must provide an absorption vessel in which the SO_2 is titrated. A typical arrangement is shown in Fig. 8.

18.1.1 *Oxygen Purifiers*—The regular commercial tank oxygen is satisfactory. It must be passed through two pressure reduction valves (approximately 207 kPa (30 psig) and 14 to 28 kPa (2 to 4 psig), respectively) or a suitable two-stage reduction valve to provide an even and adequate flow of oxygen through a tower containing H_2SO_4 and through an absorption tower containing 20 to 30-mesh inert base impregnated with NaOH and anhydrous magnesium perchlorate ($Mg(ClO_4)_2$). A flowmeter and quick-acting shut-off valve for use during preheating periods must precede the resistance furnace assembly. A flowmeter must also precede the induction furnace assembly.

18.1.2 *Combustion Furnace*—An electric tube furnace capable of continuous operation at 1425 to 1450°C is recommended, since this temperature is required for some alloys. The combustion may be accomplished either by resistance or induction heating. With the former, the temperature must be controlled as specified for each type of alloy. With the latter a rheostat to control the power input to the induction coil is required to avoid heating some types of samples too rapidly during the early stages of combustion. The combustion zone of the resistance furnace must be 200 to 250 mm (8 to 10 in.) in length and that of the induction furnace must amply provide for adequate heating of the sample.

18.1.3 *Combustion Tube*—The combustion tube of the resistance furnace must be of a low-sulfur refractory type that will withstand the maximum operating temperature without becoming porous. The tube must be of a suitable size to fit the particular furnace used and have an inside diameter large enough to accommodate the thimble, boat, and cover. A tapered-end tube is recommended.

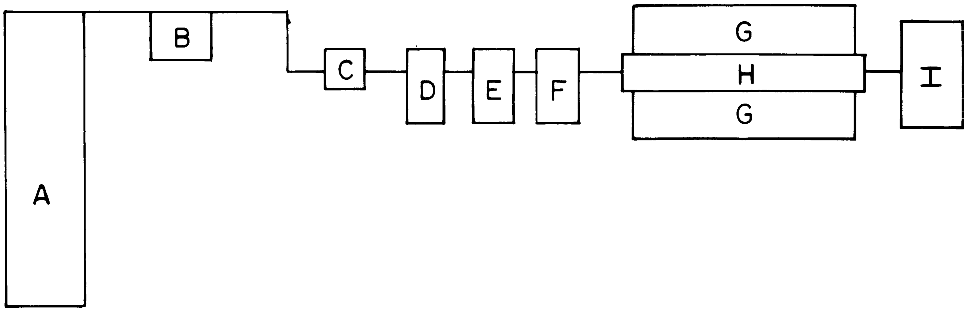
18.1.4 *Combustion Boats, Crucibles, and Covers*—The boats and crucibles for use with the respective types of furnaces must be of adequate thickness to retain the molten slag and have a blank as low and consistent as possible. The boats for use with resistance furnaces should be 90 to 100 mm (3.5 to 4 in.) in length and may be provided with suitable covers. The crucibles for use with induction furnaces must have adequate capacity and may be provided with suitable covers. The blank requirements that apply to the boats and crucibles also apply to their covers. Prior to use, the boats and covers must be prefired at least 15 min at 1100°C and then stored in a desiccator.

18.1.5 *Ceramic Thimble*—A porous ceramic thimble or liner with a small orifice drilled in the closed end is placed (closed end first) in the hot zone of the tube of the resistance furnace to prolong the life of the combustion tube by absorbing spattered slag, and to act as a filter to remove metal oxide fumes from the gas stream.

18.1.6 *Ceramic Filter*—If a ceramic thimble is not available, a porous ceramic filter is placed in the hot zone of the furnace to remove metallic oxide fumes from the gas stream; it can be constructed from porous insulating fire brick capable of withstanding the operating temperatures. In induction furnaces suitable precautions must be taken to prevent metallic oxides from entering the titration vessel.

18.1.7 *Connections*—A metal breech connector at the entrance of the combustion tube is recommended. If a rubber stopper is used it must be protected by heat-reflecting baffles, preferably of the double-disk type. Connection between the outlet end of the combustion tube and the absorption and titration assembly must be as short and free of bends as possible, with glass connections butted to minimize areas of rubber tubing exposed to gases. All rubber stoppers and tubing must be essentially free of sulfur.

18.1.8 *Absorption and Titration Apparatus*—The apparatus should consist of an absorption and titration vessel of appropriate volume and containing an inlet bubbler tube for the sulfur gases with a float valve to prevent back flow of liquid when the sample is starting to consume oxygen. The vessel must be shaped to effect complete absorption of SO_2 in a small volume of solution. The buret should be approximately 10 mL in capacity. Automatic titrations which utilize a photoelectric cell to activate a solution inlet valve are commercially available and may be used.



A — Oxygen tank.
B — Reduction valve.
C — Quick-acting shut-off valve.
D — Tower containing H_2SO_4.
E — Tower containing CO_2 absorber and anhydrous $Mg(ClO_4)_2$.
F — Flowmeter.
G — Furnace, induction or resistance-type.
H — Combustion tube.
I — Absorption and titration assembly.

FIG. 8 Apparatus No. 13, Typical Arrangement for Determination of Sulfur by the Direct-Combustion Method

19. Apparatus No. 14—Apparatus for Determination of Nitrogen by Distillation

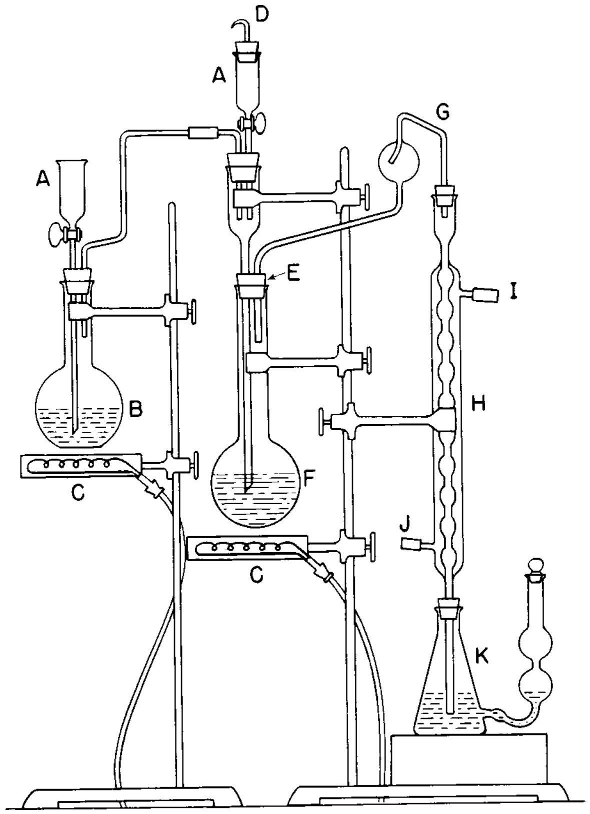
19.1 *Apparatus No. 14B*—The apparatus recommended for the determination of nitrogen by distillation with steam is shown in Fig. 9.

19.2 *Apparatus No. 14C*—The Micro-Kjeldahl apparatus recommended for the determination of nitrogen by distillation is shown in Fig. 10. The apparatus is essentially the same as Apparatus No. 14B in Fig. 9 except the Micro-Kjeldahl flask is 100 mL.

20. Apparatus No. 16—Vacuum Fusion Apparatus

20.1 A suitable apparatus for the vacuum fusion determination of hydrogen, oxygen, and nitrogen is shown diagrammatically in Fig. 11. Such equipment is available commercially[15] or may be constructed in accordance with the following description. If constructed, the following components will be required:

20.1.1 *Vacuum Pump*, used to evacuate the system prior to test, consisting of a suitable mechanical pump[16] in series with an oil-diffusion pump or mercury-diffusion pump,[17] DP_3, Fig. 11.



A —125-mL separatory funnel.
B —1-L Florence flask.
C —Electric heater.
D —Glass hook for hanging up stopper
E —Special Kjeldahl rubber stopper.
F —800-mL longneck Kjeldahl flask.
G —Nitrogen distilling head.
H —Condenser.
I —Water outlet.
J —Water inlet.
K —Modified Volhard nitrogen receiving flask.

FIG. 9 Apparatus No. 14B for Determination of Nitrogen by Distillation

20.1.2 *Manifold Exhaust Pump*—A suitable mechanical pump,[18] to evacuate the manifold to the mercury cutoff bulbs C_1, C_2, etc.

20.1.3 *Transfer Diffusion Pump, DP_1*—A suitable diffusion pump,[19] mercury-in-glass; two stage, having a pumping speed of 25 to 30 L/s.

20.1.4 *Vacuum Furnace, F*, air-cooled and constructed of borosilicate glass, and conforming to the details illustrated in Fig. 12.

20.1.5 *Blower B* (Fig. 11 and Fig. 12) to cool the furnace, capable of delivering 4 m^3 (140 ft^3) of air/min.

20.1.6 *Graphite Crucibles* (Fig. 13) machined from high-grade graphite.[20]

20.1.7 *Graphite Funnels* (Fig. 13) from the same material[20] as the crucibles (20.1.6). Some users prefer to machine the funnels as an integral part of the crucibles.

20.1.8 *Graphite Powder*, which may be prepared from the grade of carbon described in 20.1.6. The powder should pass a No. 200 (75-μm) sieve and should be passed over a magnet to remove any particles of iron or other magnetic material.

20.1.9 *Quartz Tube* (Fig. 14) clear, 51-mm outside diameter by 165 mm long, (2-in. by 6.5 in.), having an approximately 3-mm (⅛-in.) wall, and provided with two holes at the top for insertion of 1.3-mm (0.050-in.) platinum wire. The holes shall be located diametrically opposite.

20.1.10 *Special Tools* (Fig. 15) for insertion of the quartz tube into the furnace and for manipulation of the platinum wire loop over the glass hooks.

20.1.11 *Low-pressure Gas Analysis Apparatus* as shown in Fig. 11 in the area enclosed by the dotted line. Special components are listed as follows:

20.1.11.1 *Circulating Diffusion Pump, DP_2*, mercury-in-glass, three-stage, high forepressure tolerance.

20.1.11.2 *Toepler Pump, T*, manually operated.

20.1.11.3 *Combination McLeod Gage and Gas Pipet, M*, for measuring pressure in the system and for measuring the quantity of gas released by sample.

20.1.11.4 *Expansion Bulbs, H_1 and H_2*, for extending the range of the gas-measuring pipet, *M*.

20.1.11.5 *Trap, G*, copper-rare earth oxide.

20.1.11.6 *Bulb, I*, filled with $Mg(ClO_4)_2$.

20.1.11.7 *Trap, J*, cooled by liquid nitrogen.

20.1.12 *Induction Heater and Coil* adequate to heat the crucible to at least 2400°C, having the general characteristics of approximately 500 kHz and 2 to 5-kW output.

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[15] The apparatus described and referred to in this discussion is a commercially available unit manufactured and sold by the National Research Corp., Cambridge, MA.

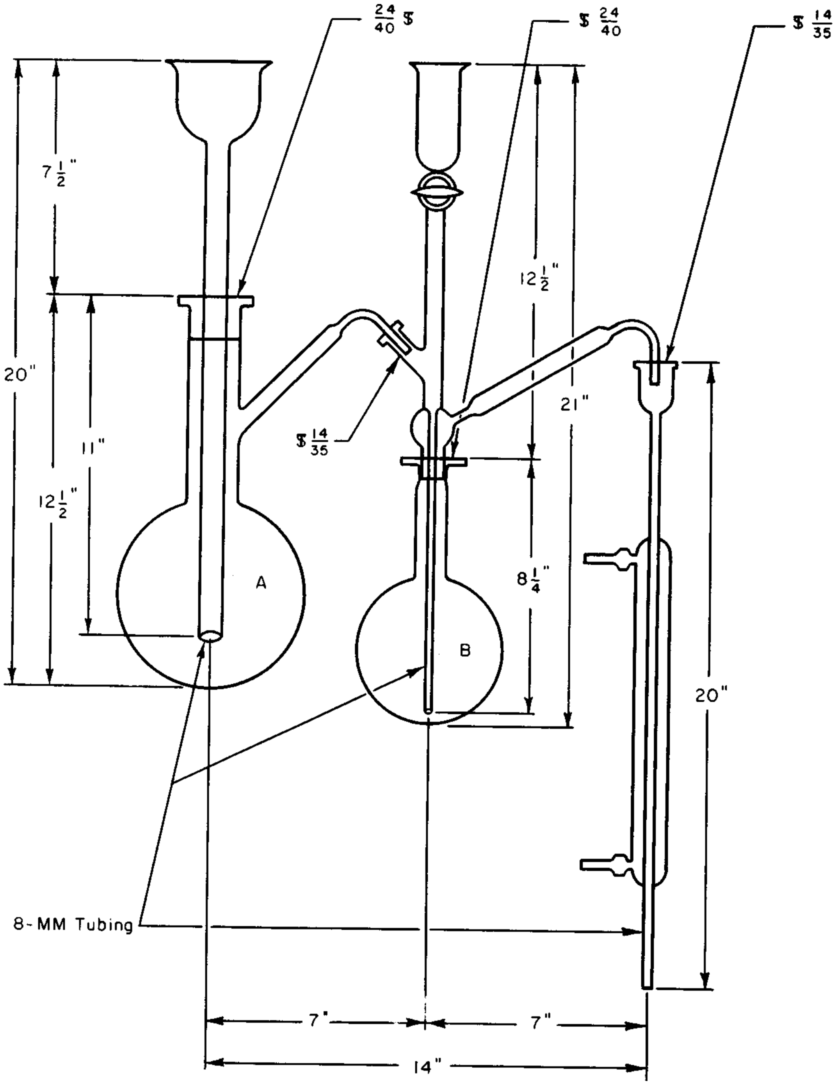
[16] A National Research Corp. No. 20 or Welch No. 1405 mechanical pump, or other suitable mechanical pump of equivalent capacity, has been found satisfactory for this purpose.

[17] A National Research Corp. Model H-2-P oil diffusion pump or CVC mercury diffusion pump have been found satisfactory for this purpose.

[18] A National Research Corp. No. 25 or Welch No. 1400 mechanical pump, or other equivalent, has been found satisfactory for this purpose.

[19] A National Research Corp. Model No. G-2-M diffusion pump or equivalent, has been found satisfactory for this purpose.

[20] National Carbon Co. spectrographic pure grade and United Carbon Co. halogen-treated spectrographic grade graphite have been found satisfactory for this purpose.



A — 1000-mL round-bottom flask.
B — 100 mL round-bottom flask.

FIG. 10 Apparatus No. 14C for Determination of Nitrogen by Distillation

20.1.13 *Optical Pyrometer* to measure the range from 1000 to 2500°C.

20.1.14 *Heaters*, glass mantle type, for copper oxide and $Mg(ClO_4)_2$ bulbs.

20.1.15 *Mercury Cutoff Valves, C_1 to C_{12}.*

20.1.16 *Dewar Flask* for containing liquid nitrogen.

NOTE 3—If liquid nitrogen is not conveniently available the procedure described by W. G. Guldner **(15)**, in which carbon dioxide is removed by Ascarite, may be used.

20.1.17 *Mercury*, high-purity, for pumps, gages, and cutoffs,

20.1.18 *Copper-Rare Earth Oxide Mixture*. **(16)**

20.1.19 *Magnesium Perchlorate*—($Mg(ClO_4)_2$) reagent grade, screened to remove fines, material passing a No. 20 (850-µm) sieve and retained on a No. 200 (75-µm sieve) being acceptable for use.

20.1.20 *Liquid Nitrogen* for cooling trap *J*.

20.1.21 *Stopcock Grease*. [21]

20.1.22 *High-Vacuum Wax*. [22]

20.1.23 *Electrical Control Circuit*—Since a number of the components require electrical heating at various times during the analysis, a rather elaborate electrical control circuit is desirable. Most analysts use an electrically operated Toepler pump and various types of interlocks with a panel board containing lamps so that the analyst can determine, at a glance, if the proper circuits are energized.

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[21] Apiezon L or N, or its equivalent, has been found satisfactory for this purpose.

[22] Apiezon W, or its equivalent, has been found satisfactory for this purpose.

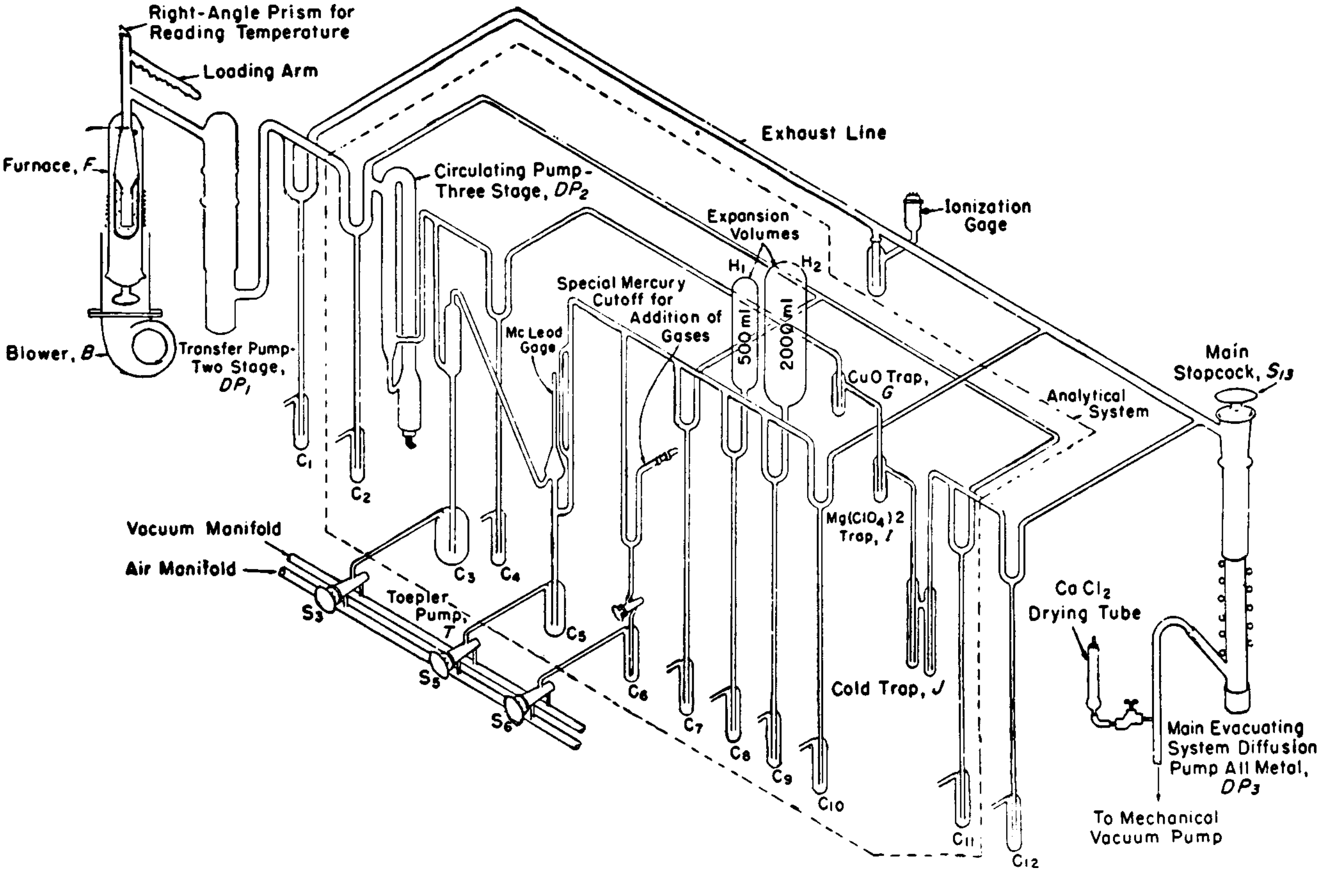

FIG. 11 Apparatus No. 16, Diagram of Complete Vacuum Fusion Apparatus for Determining Hydrogen, Nitrogen, and Oxygen

REAGENTS

STANDARD SOLUTIONS

21. Reagent No. 1—Arsenious Acid,[23] Standard Solution (0.1 N)

NOTE 4—The concentration of this reagent described here has been chosen for convenience in the description of its preparation and standardization. Where the concentration of the reagent specified in a given procedure differs from that given here, it will be necessary to make suitable adjustments in the amounts of materials used in order to produce the desired concentration. Unless otherwise indicated, the same procedure as described here can be followed in preparing other desired concentrations.

21.1 *Standardization*—This is a primary standard.

21.2 *Preparation*—Dissolve 4.9460 g of arsenic trioxide (As_2O_3)[22] in 40 mL of warm potassium hydroxide solution (100 g of KOH/L). When dissolution is complete, make just acid with H_2SO_4 (1 + 1), and cool to room temperature. Transfer to a 1-L volumetric flask, dilute to volume, and mix.

22. Reagent No. 2—Bismuth, Standard Solution (1 mL = 0.10 mg Bi) (Note 4)

22.1 *Preparation*—Dissolve 0.1000 g of bismuth (purity: 99.9 % min) in 10 mL of HNO_3 (1 + 3). Heat gently to dissolve the bismuth and expel brown fumes. Cool, transfer to a 1-L volumetric flask with HNO_3 (1 + 9), dilute to volume with HNO_3 (1 + 9), and mix.

22.2 *Standardization*—This is a primary standard.

23. Reagent No. 3—Sulfatoceric Acid (Ceric Sulfate), Standard Solution (0.1 N) (Note 4)

23.1 *Preparation*—Dissolve 54 g of ceric sulfate ($H_4Ce(SO_4)_4$) in 500 mL of water containing 16 mL of H_2SO_4. When dissolution is complete, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. The solution is approximately 1 *N* with respect to H_2SO_4. When other normalities of this solution are prepared, the acid strength should be maintained at approximately 1 *N*. (Thirty-seven % of the weight of $H_4Ce(SO_4)_4$ used should be counted as H_2SO_4.)

23.2 *Standardization*—Standardize as directed in Method A or B.

23.2.1 *Method A*—Transfer 0.2000 g of iron (purity: 99.8 % min)[24] to a 250-mL beaker. Add 20 mL of HCl (1 + 1) and 1 to 2 mL of bromine water. When dissolution is complete, boil to

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[23] National Institute of Standards and Technology, Standard Sample No. 83 of arsenic trioxide is satisfactory for this purpose.

[24] National Institute of Standards and Technology, Standard Sample No. 365 of electrolytic iron is satisfactory for this purpose.

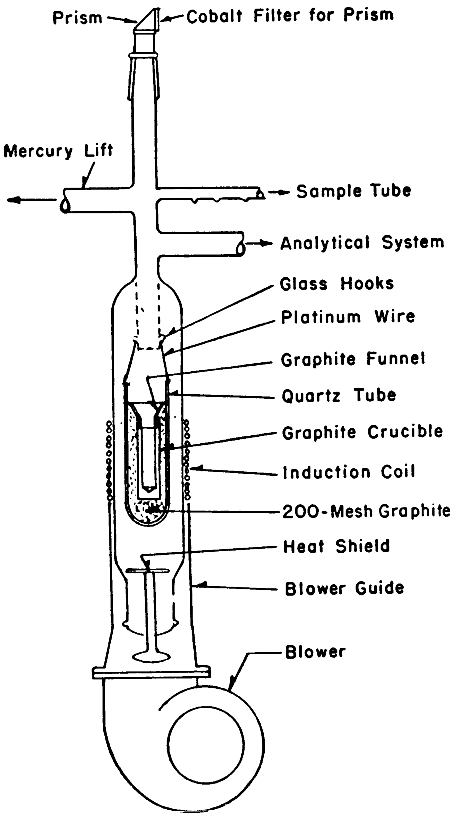

FIG. 12 Apparatus No. 16 ***(continued)*** **for Furnace Assembly**

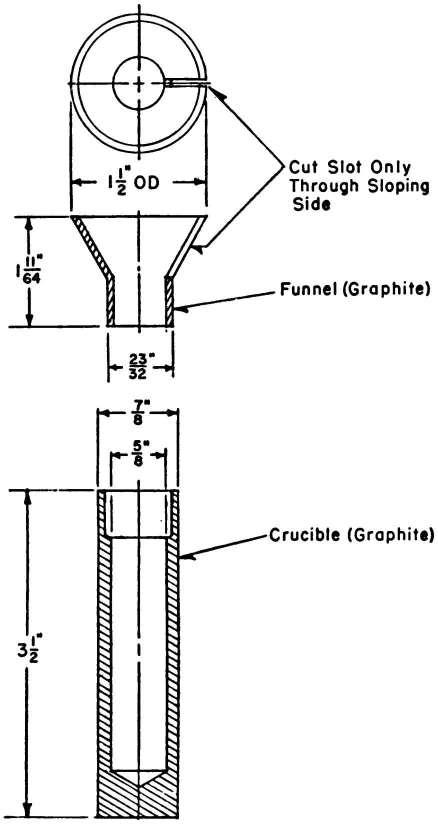

FIG. 13 Apparatus No. 16 ***(continued)*** **for Graphite Crucible and Funnel**

expel the excess bromine. Add stannous chloride solution (150 g of $SnCl_2 \cdot 2H_2O$/L of HCl (1 + 2)) dropwise to the hot solution, while stirring constantly, until the yellow color just disappears; then add 1 or 2 drops in excess. Cool the solution to 25°C or below, and add all at once 10 mL or mercuric chloride solution (50 g of $HgCl_2$/L). The precipitate must be white and milky. If it is gray, indicating the presence of finely divided elemental mercury, discard the solution and start over again. Add 4 to 5 drops of diphenylamine sulfonate indicator solution (Reagent No. 121) and titrate with the sulfatoceric acid solution (Reagent No. 3) until the end point is almost reached. Add 10 mL of H_3PO_4 (1 + 3) and titrate slowly to a persistent purple color.

23.2.2 *Method B*—Transfer 25.0 mL of arsenious acid solution (Reagent No. 1) to a 400-mL beaker and dilute to 100 mL with water (Note 5). Add 10 mL of H_2SO_4 (1 + 1), 3 drops of osmium tetroxide solution (2.5 g of OsO_4/L of 0.1 *N* H_2SO_4) as a catalyst, and 1 to 2 drops of 1,10-phenanthroline ferrous complex indicator solution (Reagent No. 122). Titrate to a pale blue or colorless end point with the sulfatoceric acid solution.

NOTE 5—If Reagent No. 1 is not available, transfer 0.2000 g of arsenic trioxide (As_2O_3)[23] to a 400-mL beaker and add 15 mL of NaOH solutioin (135 g of NaOH/L). When dissolution is complete, dilute to 100 mL with water and proceed as directed in 23.2.2, starting with the addition of H_2SO_4 (1 + 1).

24. Reagent No. 4—Iron, Standard Solution (1 mL = 0.10 mg Fe) (Note 4)

24.1 *Preparation*—Transfer 0.1000 g of iron (purity: 99.9 % min)[24] to a 100-mL beaker. Add 10 mL of HCl (1 + 1) and 1 mL of bromine water. Boil gently until the excess bromine is removed. Add 20 mL of HCl, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

24.2 *Standardization*—This is a primary standard.

25. Reagent No. 5—Ferrous Ammonium Sulfate, Standard Solution (0.1 N) (Note 4)

25.1 *Preparation*—Dissolve 39.2 g of ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$) in 500 mL of cold H_2SO_4 (5 + 95) and dilute to 1 L with H_2SO_4 (5 + 95).

NOTE 6—Saturating the solution with CO_2 will decrease the rate of oxidation of the ferrous ion.

25.2 *Standardization*—Standardize as directed in the methods concerned.

26. Reagent No. 6—Silicon, Standard Solution (1 mL = 0.05 mg Si) (Note 4)

26.1 *Preparation*—Fuse 0.1070 g of anhydrous silicon dioxide (SiO_2) with 1.0 g of sodium carbonate (Na_2CO_3) in a covered platinum crucible. Cool, dissolve the melt in water in a plastic beaker, cover, and heat on a steam bath for ½ to 1 h. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store the solution in a plastic bottle. If anhydrous SiO_2 is not available, the weight should be adjusted according to the actual silicon content of the SiO_2 used, as determined by gravimetric analysis.

26.2 *Standardization*—This is a primary standard.

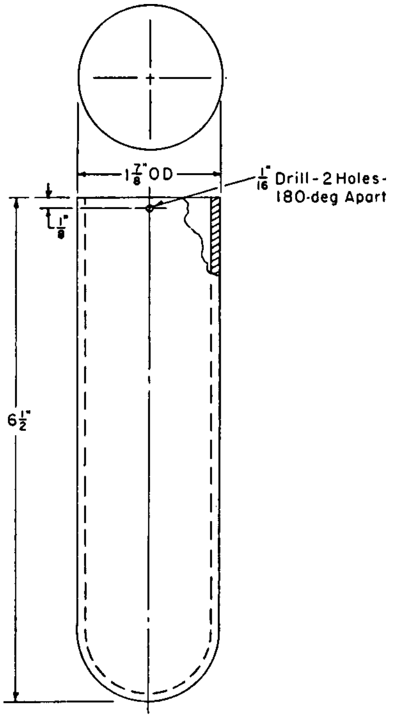

FIG. 14 Apparatus No. 16 *(continued)* for Quartz Tube)

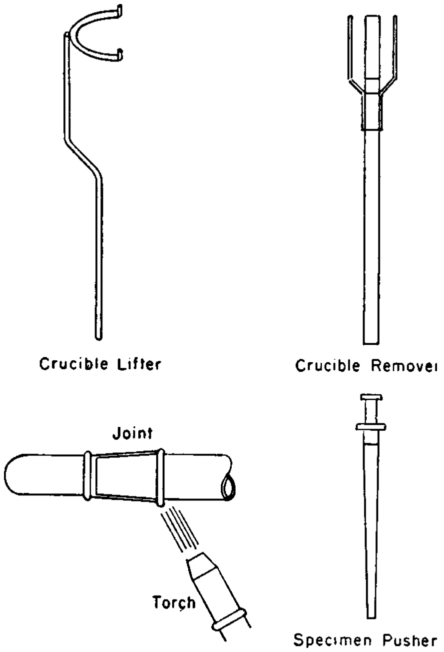

FIG. 15 Apparatus No. 16 *(continued)* for Special Tools)

27. Reagent No. 7—Iodine, Standard Solution (0.1 N) (Note 4)

27.1 *Preparation*—Dissolve 12.7 g of iodine (I_2) and 40 g of potassium iodide (KI) in 25 mL of water. When dissolution is complete, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a dark-colored, glass-stoppered bottle in a cool place.

27.2 *Standardization*—Standardization against arsenious acid solution (Reagent No. 1) is preferable, except when tin is to be determined. Directions for standardization against tin or an alloy of known tin content are given in the individual methods. To standardize against arsenious acid proceed as follows:

27.2.1 Transfer 25.0 mL of arsenious acid solution (Reagent No. 1) to a 250-mL beaker containing 60 mL of water, 0.5 g of KI, and 1 g of sodium bicarbonate ($NaHCO_3$). Add 2 mL of starch solution (Reagent No. 110) and titrate rapidly with the iodine solution to a persistent blue color.

28. Reagent No. 8A—Molybdenum, Standard Solution (1 mL = 0.20 mg Mo) (Note 4)

28.1 *Preparation*—Dissolve 0.500 g of sodium molybdate dihydrate ($Na_2MoO_4·2H_2O$) in water containing 5 mL of H_2SO_4, transfer to a 1-L volumetric flask, dilute to volume, and mix.

28.2 *Standardization*—Standardize as directed in Method A or B.

28.2.1 *Method A (Titrimetric)*—Prepare a ferric iron solution as follows: Dissolve 100 g of ferric sulfate ($Fe_2(SO_4)_3$) in 850 mL of an aqueous solution containing 10 mL of H_2SO_4 and 150 mL of H_3PO_4; add sufficient potassium permanganate solution (25 g of $KMnO_4$/L) to give a faint pink color. Prepare a Jones reductor (Apparatus No. 8). Just before use pass 100 mL of warm (40 to 50°C) H_2SO_4 (5 + 95) through it and then a little cold water, and discard. Transfer 35 mL of the ferric iron solution to the suction flask, and then enough water so that the tip of the reductor dips well beneath the surface of the solution. Transfer 100 mL of the molybdenum solution to a 250-mL beaker and add 10 mL of H_2SO_4 (1 + 1). With gentle suction, draw the cool (about 20°C) molybdenum solution through the reductor, while gently swirling the solution in the suction flask. Just before the surface of the liquid reaches the zinc, add 50 mL of cold H_2SO_4 (5 + 95). Rinse the column two more times by adding 50 mL of water each time. Add the water just before the surface of the solution reaches the zinc. Close the stopcock while a portion of the last rinsing remains in the reductor funnel. Disconnect and raise the reductor as a little water is allowed to run through the stem, and rinse the outside of the stem. Titrate the collected solution with $KMnO_4$ solution (Reagent No. 13). Correct for a reagent blank that has been carried through the entire procedure, using the same amounts of all reagents.

$$2H_2MoO_4 + 3Zn + 6H_2SO_4 = Mo_2(SO_4)_3 + 3ZnSO_4 + 8H_2O$$

$$\begin{aligned} &Mo_2(SO_4)_3 + 2Fe_2(SO_4)_3 + 6H_2O \\ &= 2HMoO_3 \text{ (Note 7)} + 4FeSO_4 + 5H_2SO_4 \end{aligned}$$

NOTE 7—Exact chemical composition not known, $HMoO_3$ used for illustration only; based on the work of G. E. F. Lundell et al. **(17).**

$$\begin{aligned} &10HMoO_3 + 20FeSO_4 + 19H_2SO_4 + 6KMnO_4 \\ &= 10H_2MoO_4 + 10Fe_2(SO_4)_3 + 3K_2SO_4 + 6MnSO_4 + 14H_2O \end{aligned}$$

28.2.2 *Method B (Gravimetric)*—Transfer 25.0 mL of the molybdenum solution to a 250-mL beaker and dilute to 150 mL. Add 1 drop of methyl orange indicator solution (Reagent No. 118) and neutralize with sodium hydroxide solution (200 g of NaOH/L) until the indicator changes to a yellow tint. Add H_2SO_4 (1 + 1) dropwise (Note 8) until the pink color just returns. Add 1 g of sodium acetate and stir (Note 9). Heat the solution just to boiling and add silver nitrate solution (20 g of

$AgNO_3$/L) dropwise until the silver molybdate is precipitated; then add several drops in excess. Stir occasionally, while cooling to room temperature. A curdy precipitate settles out leaving a clear supernatant solution. Filter with gentle suction, using a weighed fritted-glass crucible, and wash with silver nitrate solution (5 g of $AgNO_3$/L). Finally wash three times with 5-mL portions of ethanol (95 %). This washing will remove the last traces of $AgNO_3$. Dry at 250°C and weigh. (Ag_2MoO_4 found × 0.2554 = weight of molybdenum in 25 mL of solution.)

NOTE 8—Due to the limited solubility of silver acetate the amount of sodium acetate must not exceed the specified amount to bring back the yellow tint; therefore, the H_2SO_4 must be added carefully.

NOTE 9—At this point the solution must be yellow. If an excess of acid was added, discard the solution and start over again.

29. Reagent No. 8B—Molybdenum, Standard Solution (1 mL = 0.20 mg Mo) (Note 4)

29.1 *Preparation*—Transfer 0.2000 g of molybdenum metal (purity: 99.8 % min) to a 150-mL beaker and dissolve in 10 mL of HCl and HNO_3 added dropwise. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

29.2 *Standardization*—This is a primary standard.

30. Reagent No. 9—Potassium Bromate, Standard Solution (0.1 N) (Note 4)

30.1 *Preparation*—Dry the crystals of potassium bromate ($KBrO_3$) at 180°C to constant weight. Dissolve 2.7834 g of the $KBrO_3$ in water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

30.2 *Standardization*—This is a primary standard.

31. Reagent No. 10—Potassium Dichromate, Standard Solution (0.1 N) (Note 4)

31.1 *Preparation*—Dissolve 4.9035 g of National Institute of Standards and Technology standard potassium dichromate ($K_2Cr_2O_7$) in water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

31.2 *Standardization*—This is a primary standard.

32. Reagent No. 11—Potassium Ferrocyanide, Standard Solution (1 mL ±i 0.01 g Zn) (Note 4)

32.1 *Preparation*—Dissolve 42.0 g of potassium ferrocyanide ($K_4Fe(CN)_6 \cdot 3H_2O$) in water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

32.2 *Standardization*—Standardize as directed in the methods concerned.

33. Reagent No. 12—Potassium Iodate, Standard Solution (0.1 N) (Note 4)

33.1 *Preparation*—Dry the crystals of potassium iodate (KIO_3) at 180°C to constant weight. Dissolve 3.5667 g of the KIO_3 in 200 mL of water containing 1 g of sodium hydroxide (NaOH) and 10 g of potassium iodide (KI). When dissolution is complete, transfer to a 1-L volumetric flask, dilute to volume, and mix.

33.2 *Standardization*—This is a primary standard. Where empirical standardization is required standardize as directed in the methods concerned.

34. Reagent No. 13—Potassium Permanganate, Standard Solution (0.1 N) (Note 4)

34.1 *Preparation*—Dissolve 3.2 g of potassium permanganate ($KMnO_4$) in 1 L of water. Let stand in the dark for 2 weeks. Filter, without washing, through a Gooch crucible (Apparatus No. 2) or a fine porosity fritted-glass crucible. Avoid contact with rubber or other organic material. Store in a dark-colored glass-stoppered bottle.

34.2 *Standardization*—Dry a portion of the National Institute of Standards and Technology standard sample of sodium oxalate at 105°C. Transfer 0.3000 g of the sodium oxalate to a 600-mL beaker. Add 250 mL of H_2SO_4 (5 + 95), previously boiled for 10 to 15 min and then cooled to 27 ± 3°C, and stir until the oxalate has dissolved. Add 39 to 40 mL (Note 10) of the $KMnO_4$ solution, at a rate of 25 to 35 mL/min, while stirring slowly. Let stand until the pink color disappears (about 45 s) (Note 11). Heat to 55 to 60°C and complete the titration by adding $KMnO_4$ solution until a faint pink color persists for 30 s. Add the last 0.5 to 1 mL dropwise, allowing each drop to become decolorized before adding the next drop. To determine the blank: Titrate 250 mL of H_2SO_4 (5 + 95), treated as above, with $KMnO_4$ solution to a faint pink color. The blank correction is usually equivalent to 0.03 × 0.05 mL.

NOTE 10—A 0.3000-g portion of sodium oxalate requires 44.77 mL of $KMnO_4$ solution (0.1 *N*).

NOTE 11—If the $KMnO_4$ solution is too strong, the pink color will not fade at this point; begin again, adding a few millilitres less of the $KMnO_4$ solution.

35. Reagent No. 14—Silver Nitrate, Standard Solution (1 mL ±i 1.0 mg Ni) (Note 4)

35.1 *Preparation*—Use only colorless crystals of silver nitrate ($AgNO_3$). Dry the $AgNO_3$ at 110°C and finally heat to constant weight at 220 to 250°C, but never over 250°C, for 15 min to drive out the last small amount of water. Dissolve 5.7890 g of the twice recrystallized $AgNO_3$ in water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

35.2 *Standardization*—This is a primary standard. If a check on the standard is desired, proceed as follows:

35.2.1 Dissolve 1.000 g of NaCl in 150 mL of water containing 1 mL of HNO_3(1 + 1). The following operations must be carried out in subdued light. While stirring, add 20 mL of the $AgNO_3$ solution dropwise to the NaCl solution. Heat the suspension nearly to boiling, and stir for 2 min to assist in the coagulation of the AgCl precipitate. Set the beaker aside in the dark for at least 2 h. Collect the precipitate on a Gooch crucible (Apparatus No. 2), wash with 0.01 *N* HNO_3(0.5 mL of HNO_3/L of water) and, finally 1 or 2 times with water. Dry the AgCl at 150°C to constant weight.

36. Reagent No. 15—Sodium Arsenite, Standard Solution

(1 mL ±i 0.50 mg Mn)

36.1 *Preparation*—Dissolve 20 g of sodium arsenite ($NaAsO_2$) in water and dilute to 1 L. Transfer 100 mL of this solution to a 1-L volumetric flask, dilute to volume, and mix. Filter if necessary.

36.2 *Standardization*—Standardize as directed in the methods concerned.

37. Reagent No. 16—Sodium Hydroxide, Standard Solution (0.1 N) (Note 4)

37.1 *Preparation*—Dissolve 50 g of sodium hydroxide (NaOH) in 50 mL of water and transfer to a large test tube; avoid wetting the top. Stopper tightly and let stand in a vertical position until the supernatant liquid is clear. Carefully withdraw and transfer 5.0 mL of the clear solution to a 1-L volumetric flask containing about 750 mL of freshly boiled and cooled water, dilute to volume with more of the freshly prepared water, and mix. Protect the NaOH solution from CO_2 by means of a soda-lime or soda-mica mineral tube (Ascarite II).

37.2 *Standardization*—Transfer 0.4000 g of the National Institute of Standards and Technology standard sample of potassium acid phthalate to a 250-mL Erlenmeyer flask. Dissolve in 100 mL of freshly boiled and cooled water. Add 3 drops of phenolphthalein indicator solution (Reagent No. 117) and titrrate to a faint pink color with the NaOH solution. Where empirical standardization is required standardize as directed in the methods concerned.

38. Reagent No. 17—Sodium Thiosulfate, Standard Solution (0.1 N)

38.1 *Preparation*—Dissolve 24.8 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in water and dilute to 1 L. If sulfur precipitates during preparation or upon subsequent use, discard the solution and prepare a fresh solution.

38.2 *Standardization*—Transfer 25.0 mL of KIO_3 solution (Reagent No. 12) to a 125-mL flask. Add 30 mL of water, 1 g of potassium iodide (KI), and 10 mL of H_2SO_4 (1 + 4), and titrate with $Na_2S_2O_3$ solution to a light straw color. Add 2 mL of starch solution (Reagent No. 110) and titrate slowly to the disappearance of the blue color. Where empirical standardization is required, standardize as directed in the methods concerned.

39. Reagent No. 18—Titanium, Standard Solution (1 mL = 0.50 mg Ti)

39.1 *Preparation*—Prepare as directed in Method A or B.

39.1.1 *Method A*—Fuse 0.85 g of titanium dioxide (TiO_2)[25] in a covered 50-mL platinum crucible with 15 g of potassium bisulfate ($KHSO_4$); cool the melt and dissolve in 200 mL of hot H_2SO_4 (1 + 1), stirring occasionally. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

39.1.2 *Method B*—Transfer 0.85 g of titanium dioxide (TiO_2)[25] to a 250-mL Erlenmeyer flask, add 10 g of ammonium sulfate ($(NH_4)_2SO_4$), and 25 mL of H_2SO_4. Insert a short-stemmed glass funnel in the neck of the flask and heat cautiously to incipient boiling while rotating the flask over a free flame. Continue the heating until complete dissolution has been effected and no unattacked material remains on the wall of the flask. Cool and rapidly pour the solution into 450 mL of cool water which is vigorously stirred. Rinse the flask with H_2SO_4 (5 + 95). Transfer the solution to a 1-L volumetric flask, dilute to volume with H_2SO_4(5 + 95), and mix.

39.2 *Standardization*—Standardize as directed in Method A or B.

39.2.1 *Method A (Gravimetric)*—Transfer 50 mL of the titanium solution to a 400-mL beaker and dilute to 200 mL with boiling water. Heat to boiling, make slightly ammoniacal, and boil for 1 to 3 min. Filter, using an 11-cm fine paper and wash with hot water until free of alkali salts. Place the paper and precipitate in a weighed platinum crucible and char the paper at 500°C. Finally, ignite to constant weight at 1100°C, and weigh as titanium dioxide (TiO_2). TiO_2 found × 0.5995 = weight of titanium in 50 mL of solution.

39.2.2 *Method B (Titrimetric)*—Prepare a ferric iron solution as follows: Dissolve 100 g of ferric sulfate ($Fe_2(SO_4)_3$) in 850 mL of an aqueous solution containing 10 mL of H_2SO_4 and 150 mL of H_3PO_4; add sufficient potassium permanganate solution (25 g of $KMnO_4$/L) to give a faint pink color. Prepare a Jones reductor (Apparatus No. 8). Just before use pass 100 mL of H_2SO_4 (2 + 98) through the reductor and discard. Transfer 30 mL of the ferric iron solution to the suction flask and position the flask so that the tip of the reductor is just below the surface of the solution. Transfer 100 mL of the titanium solution to a 250-mL beaker and heat to 60 to 70°C. Pass the solution through the reductor at the rate of about 20 mL/min. Rinse the reductor, first with 100 mL of hot H_2SO_4 (1 + 9) and then with 50 mL of water. Add 1 drop of 1,10-phenanthroline ferrous complex indicator solution (Reagent No. 122) and titrate with $KMnO_4$ solution (0.05 *N*) (Reagent No. 13; Use 1.6 g of $KMnO_4$/L instead of the specified weight) to a clear green color that persists for 1 min. Correct for a reagent blank that has been carried through the entire procedure, using the same amounts of all reagents.

$$2\,Ti(SO_4)_2 + Zn + H_2SO_4 = Ti_2(SO_4)_3 + ZnSO_4 + H_2SO_4$$

$$Ti_2(SO_4)_3 + Fe_2(SO_4)_3 = 2Ti(SO_4)_2 + 2FeSO_4 10FeSO_4 + 2\,KMnO_4$$
$$+ 8H_2SO_4 = 5Fe_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O$$

40. Reagent No. 19—Tin, Standard Solution (1 mL = 1.0 mgSn) (Note 4)

40.1 *Preparation*—Transfer 1.0000 g of tin (purity, 99.9% min)[26] to a 400-mL beaker, and cover. Add 300 mL of HCl (1 + 1) and warm gently until the metal is dissolved. If dissolution is difficult, add 0.5 to 1.0 g of potassium chlorate ($KClO_3$). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

40.2 *Standardization*—This is a primary standard.

41. Reagent No. 20—Zinc, Standard Solution (1 mL = 4.0 mg Zn) (Note 4)

41.1 *Preparation*—Transfer 4.0000 g of zinc (purity, 99.9 % min)[27] to a 400-mL beaker, and cover. Add 200 mL of HCl (1 + 4) and warm gently until the zinc is dissolved. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

41.2 *Standardization*—This is a primary standard.

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[25] National Institute of Standards and Technology, Standard Sample No. 154 of titanium dioxide is satisfactory for this purpose.

[26] National Institute of Standards and Technology, Standard Sample No. 42 of standard melting point tin is satisfactory for this purpose.

[27] National Institute of Standards and Technology, Standard Sample No. 43 of standard melting point zinc is satisfactory for this purpose.

42. Reagent No. 21—Copper, Standard Solution (1 mL = 1.0 mg Cu) (Note 4)

42.1 *Preparation*—Transfer 1.0000 g of copper (purity, 99.9 % min) to a 250-mL beaker, and cover. Add 15 mL of HNO_3 (1 + 3) and warm gently until the copper is dissolved. Add 5 mL of H_2SO_4 and evaporate to dense white fumes. Cool, wash down the cover and walls of the beaker with 20 mL of water, and again evaporate to white fumes. Cool, and add 200 mL of H_2SO_4 (1 + 1). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

42.2 *Standardization*—This is a primary standard.

43. Reagent No. 22—Disodium (Ethylenedinitrilo) Tetraacetate Dihydrate (EDTA Disodium Salt), Standard Solution (0.01 *M*) (Note 4)

43.1 *Preparation*—Dissolve 3.7225 g of disodium (ethylenedinitrilo) tetraacetate dihydrate in water, transfer to a 1-L volumetric flask, dilute to volume, and mix. The solution is stable for several months when stored in plastic or borosilicate glass bottles.

43.2 *Standardization*—Standardize as directed in the methods concerned.

44. Reagent No. 23—Nitrogen, Standard Solution (1 mL = 0.10 mg N) (Note 4)

44.1 *Preparation*—Dissolve 0.3820 g of dry ammonium chloride (NH_4Cl) in 500 mL of water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

44.2 *Standardization*—This is a primary standard.

45. Reagent No. 24—Manganese, Standard Solution (1 mL = 0.10 mg Mn) (Note 4)

45.1 *Preparation*—Prepare as described in Method A or B.

45.1.1 *Method A (Preparation from Manganese Metal)*[28]—Pretreat manganese metal (purity, 99.8% min) (Note 12) as follows:

Wash in H_2SO_3, rinse with water, and dry. Store in a covered glass beaker in a desiccator. Transfer 0.10 g, weighed to the nearest 0.1 mg (do not use small particles of metal) to a 150-mL beaker, and cover. Add 10 mL of HNO_3 (1 + 1). Heat gently until dissolution is complete and brown fumes are expelled. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

NOTE 12—For the analysis of high-manganese materials, the manganese metal must be assayed. This would include the determination of oxygen as well as all metallics.

45.1.2 *Method B (Preparation from Potassium Permanganate)*—Transfer 90.9 mL of 0.1000 *N* potassium permanganate ($KMnO_4$) solution (Reagent No. 13) to a 400-mL beaker and add 10 mL of H_2SO_4 (1 + 1). (If necessary, the $KMnO_4$ solution should be freshly refiltered and restandarized, as described under Reagent No. 13.) Reduce the $KMnO_4$ by additions of H_2SO_3 and boil the solution until free of SO_2. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

46. Reagent No. 25A—Cobalt, Standard Solution (1 mL = 0.06 mg Co) (Note 4)

46.1 *Preparation*—Prepare as directed in Method A or B.

46.1.1 *Method A*—Transfer 0.3000 g of cobalt metal (purity, less than 0.3 % nickel) to a 400-mL tall-form beaker. Add 15 mL of HCl (1 + 1), cover the beaker, and heat until the cobalt is dissolved. Add 10 mL of HCl and dilute to about 150 mL. Add NH_4OH dropwise until the solution is neutral to pH paper. Add 1 g of hydroxylamine hydrochloride ($NH_2{\cdot}OH{\cdot}HCl$), and stir well until the salt is dissolved. Add 5 mL of NH_4OH and dilute to approximately 250 mL. Electrolyze on a clean platinum electrode, starting with a current density of 3 A/dm^2 until the brown solution begins to turn pink and the cobalt starts to deposit. Change the current density to 1 A/dm^2 and continue the electrolysis until the pink color disappears. Alternatively, the entire electrolysis may be conducted by controlling the cathode potential at − 0.9 V with respect to a saturated calomel reference electrode, preferably with an automatic potentiostat. Rinse, dry, and weigh the electrode. Dissolve the cobalt from the electrode by placing it in a beaker containing enough hot HCl (1 + 9) to cover the electrode. Rinse the electrode and collect the rinsings in the beaker. Dry and reweigh the electrode. Transfer the solution to a 500-mL volumetric flask, dilute to volume, and mix. Transfer a 50-mL aliquot of this solution to a 500-mL volumetric flask, dilute to volume, and mix.

46.1.1.1 *Standardization*—Calculate the exact concentration of cobalt in the solution on the basis of the amount of cobalt dissolved from the electrode and the dilution ratio used.

46.1.2 *Method B*—Dry a weighing bottle in an oven at 130°C for 1 h, cool in a desiccator, and weigh. Transfer 0.7890 g of cobalt sulfate ($CoSO_4$)[29] that has been heated at 550°C for 1 h to the weighing bottle. Dry the bottle and contents at 130°C for 1 h, cool in a desiccator, stopper the bottle, and weigh. The difference in weight is the amount of $CoSO_4$ taken. Transfer the weighed $CoSO_4$ to a 400-mL beaker, rinse the weighing bottle with water, and transfer the rinsings to the beaker. Add 150 mL of water and 10 mL of HCl, and heat to dissolve the salts. Cool, transfer to a 500-mL volumetric flask, dilute to volume, and mix. Transfer a 50-mL aliquot of this solution to a 500-mL volumetric flask, dilute to volume, and mix.

46.1.2.1 *Standardization*—Calculate the cobalt concentration as follows:

$$\text{Cobalt, mg/mL} = \text{weight of } CoSO_4\text{, g, } \times 0.07605$$

47. (Reagent No. 25B—Cobalt, Standard Solution (1 mL = 1.5 mg Co) (Note 4)

47.1 *Preparation*—Dry a weighing bottle in an oven at 130°C for 1 h, cool in a desiccator, and weigh. Transfer 3.945 g of cobalt sulfate ($CoSO_4$)[29] that has been heated at 550°C for 1 h to the weighing bottle. Dry the bottle and contents at 130°C for 1 h, cool in desiccator, stopper the bottle, and weigh. The difference in weight is the amount of $CoSO_4$ taken. Transfer the weighed $CoSO_4$ to a 400-mL beaker, rinse the weighing bottle with water, and transfer the rinsings to the beaker. Add 150 mL

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[28] Elmang, as supplied by the Union Carbide Corp., Metals Div., is satisfactory for this purpose.

[29] Cobalt sulfate (99.9 % min), prepared from the hexamine salt, by G. Frederick Smith Chemical Co., Columbus, OH, is satisfactory for this purpose.

of water and 20 mL of HNO_3, and heat to dissolve the salts. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

47.2 *Standardization*—Calculate the cobalt concentration as follows:

$$\text{Cobalt, mg/mL} = \text{weight of } CoSO_4\text{, g, } \times 0.38026$$

48. Reagent No. 26—Tungsten, Standard Solution (1 mL = 0.10 mg W) (Note 4)

48.1 *Preparation*—Dissolve 0.1794 g of sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$) (Note 13) in 60 mL of water, transfer to a 100-mL volumetric flask, dilute to volume, and mix. Transfer a 10-mL aliquot of this solution to a 100-mL volumetric flask, dilute to volume, and mix. Prepare the final dilute solution as needed.

NOTE 13—Conforms to Dr. Folin's specifications. **(18)**

48.2 *Standardization*—This is a primary standard.

49. Reagent No. 27—Aluminum, Standard Solution (1 mL = 1.0 mg Al) (Note 4)

49.1 *Preparation*—Transfer 1.0000 g of aluminum metal (purity, 99.7 % min) to a 250-mL beaker, and cover. Add 100 mL of HCl (1 + 1), a small crystal of mercuric nitrate and heat gently until dissolution is complete. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

49.2 *Standardization*—This is a primary standard.

NONSTANDARD REAGENTS

50. Reagent No. 101—Ammonium Molybdate Solution (Ammoniacal)

50.1 Transfer 65 g of ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) and 225 g of ammonium nitrate (NH_4NO_3) to an 800-mL beaker. Add 15 mL of NH_4OH and 600 mL of water. Stir and heat gently until the crystals have dissolved. Filter through a medium paper (without washing) and dilute to 1 L.

51. Reagent No. 102—Ammonium Molybdate Solution (Acidic)

51.1 *Solution No. 1*—Transfer 100 g of molybdic acid (85 % MoO_3) to a 600-mL beaker containing 240 mL of water and mix thoroughly. Add 140 mL of NH_4OH while stirring vigorously. When dissolution is complete, filter through a medium paper, add 60 mL of HNO_3, and cool.

51.2 *Solution No. 2*—Add 400 mL of HNO_3 to 960 mL of water in a 2-L beaker and cool.

51.3 Add Solution No. 1 to Solution No. 2 while stirring constantly. Add 0.1 g of ammonium phosphate, dibasic ($(NH_4)_2HPO_4$), and let stand at least 24 h before using. Use only the clear supernatant liquid.

52. Reagent No. 103—Nitric Acid (3 + 97) for Manganese Determination

52.1 Boil 40 mL of HNO_3 until decolorized, cool, and pass in a current of clean air for 5 min. (If necessary, clean the air in a suitable manner to remove particulate matter and organic vapors.) Add 30 mL of this solution to 970 mL of water. Add 1 g of sodium bismuthate ($NaBiO_3$), shake, and let settle. Use only the clear supernatant liquid.

53. Reagent No. 104—Dimethylglyoxime Solution in Alcohol (10 g/L)

53.1 Dissolve 10 g of dimethylglyoxime in ethanol, methanol, or No. 30 specially denatured alcohol and dilute to 1 L with alcohol. Filter before using. This solution keeps almost indefinitely.

54. Reagent No. 105—Dimethylglyoxime Solution in Alcohol (40 g/L)

54.1 Dissolve 4 g of dimethylglyoxime in 100 mL of hot ethanol, methanol, or No. 30 specially denatured alcohol. Keep the solution hot to prevent the reagent from crystallizing out. Prepare fresh as needed.

55. Reagent No. 106—Ammonium Persulfate (Ammonium Peroxydisulfate) Solution (250 g/L)

55.1 Dissolve 25 g of ammonium persulfate (ammonium peroxydisulfate) ($(NH_4)_2S_2O_8$) in water, and dilute to 100 mL. Prepare fresh as needed.

56. Reagent No. 107—Antimony Trichloride Solution (10 g/L)

56.1 Dissolve 1 g of antimony trichloride ($SbCl_3$) in 20 mL of HCl and dilute to 100 mL.

57. Reagent No. 108—Formic Acid Mixture

57.1 Dilute 200 mL of formic acid (sp gr 1.20) to approximately 900 mL with water. Add 30 mL of NH_4OH and dilute to 1 L.

58. Reagent No. 109—Formic Acid Mixture-H_2S Wash Solution

58.1 Dilute 25 mL of formic acid mixture (Reagent No. 108) to 1 L with water and saturate with H_2S.

59. Reagent No. 110—Starch Solution (10 g/L)

59.1 Add about 5 mL of water gradually to 1 g of soluble (or arrowroot) starch, with stirring, until a paste is formed, and add this to 100 mL of boiling water. Cool, add 5 g of potassium iodide (KI), and stir until the KI is dissolved. Prepare fresh as needed.

60. Reagent No. 111—Zinc Oxide Suspension (166 g/L)

60.1 Add 10 g of finely divided zinc oxide (ZnO) to 60 mL of water, and shake thoroughly. Prepare fresh daily as needed.

61. Reagent No. 112—1-Nitroso-2-Naphthol (α-Nitroso-β-Naphthol) Solution (70 g/L)

61.1 Dissolve 7 g of dry 1-nitroso-2-naphthol in 100 mL of acetic acid, and filter. Prepare fresh as needed.

62. Reagent No. 113—Cinchonine Solution (125 g/L)

62.1 Dissolve 125 g of cinchonine in HCl (1 + 1), dilute to 1 L with HCl (1 + 1), and filter.

63. Reagent No. 114—Cinchonine Wash Solution

63.1 Dilute 30 mL of cinchonine solution (Reagent No. 113) to 1 L with water.

64. Reagent No. 115—Cupferron Solution (60 g/L)

64.1 Dissolve 6 g of cupferron in 80 mL of cold water, dilute to 100 mL, and filter. Prepare fresh as needed.

65. Reagent No. 116—Tartaric Acid Solution (250 g/L)

65.1 Dissolve 250 g of tartaric acid in 700 mL of water, bring to a boil, cool, filter, and dilute to 1 L.

66. Reagent No. 117—Phenolphthalein Indicator Solution (10 g/L)

66.1 Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %).

67. Reagent No. 118—Methyl Orange Indicator Solution (1 g/L)

67.1 Dissolve 0.1 g of methyl orange in 100 mL of water. Filter if necessary.

68. Reagent No. 119—Methyl Red Indicator Solution (0.4 g/L)

68.1 Dissolve 0.1 g of methyl red in 3.72 mL of 0.1 *N* sodium hydroxide solution (Reagent No. 16) and dilute to 250 mL with water. Filter if necessary.

69. Reagent No. 120—Bromcresol Purple Indicator Solution (0.4 g/L)

69.1 Dissolve 0.1 g of bromcresol purple in 1.85 mL of 0.1 *N* sodium hydroxide solution (Reagent No. 16) and dilute to 250 mL with water.

70. Reagent No. 121—Sodium Diphenylamine Sulfonate Indicator Solution (2 g/L)

70.1 *Method A (Preparation from Barium Diphenylamine Sulfonate)*—Dissolve 0.32 g of barium diphenylamine sulfonate in 100 mL of hot water. Add 0.5 g of sodium sulfate (Na_2SO_4), stir, and filter through a fine paper to remove the $BaSO_4$. Store in a dark-colored bottle.

70.2 *Method B (Preparation from Sodium Diphenylamine Sulfonate)*—Dissolve 0.20 g of sodium diphenylamine sulfonate in 100 mL of water. Store in a dark-colored bottle.

71. Reagent No. 122—1,10-Phenanthroline (*o*-Phenanthroline) Ferrous Complex Indicator Solution (0.025 *M*)

71.1 Dissolve 1.485 g of 1,10-phenanthroline monohydrate in 100 mL of ferrous sulfate solution ($FeSO_4 \cdot 7H_2O$), Reagent No. 151.

72. Reagent No. 123—α-Benzoin Oxime Solution in Alcohol (20 g/L)

72.1 Dissolve 10 g of α-benzoin oxime in 500 mL of ethanol (95 %) or methanol. Filter if necessary.

73. Reagent No. 124—α-Benzoin Oxime Solution in Acetone (50 g/L)

73.1 Dissolve 5 g of α-benzoin oxime in 95 mL of acetone and 5 mL of cold water. Store in a dark-colored bottle in a cool place. Prepare fresh every 5 days.

74. Reagent No. 125—Potassium *m*-Periodate Solution (7.5 g/L)

74.1 Dissolve 7.5 g of potassium *m*-periodate (KIO_4) in 200 mL of hot HNO_3 (1 + 1), add 400 mL of H_3PO_4, cool, and dilute to 1 L.

75. Reagent No. 126—Sodium Nitrite Solution (20 g/L)

75.1 Dissolve 2 g of sodium nitrite ($NaNO_2$) in water and dilute to 100 mL. This solution is sufficiently stable for use over a 24-h period.

76. Reagent No. 127—Sodium Acetate Solution (500 g/L)

76.1 Dissolve 500 g of sodium acetate trihydrate ($CH_3COONa \cdot 3H_2O$) in about 600 mL of water, filter, and dilute to 1 L.

77. Reagent No. 128—1-Nitroso-2-Naphthol-3,6-Disulfonic Acid Disodium Salt (Nitroso-R Salt) Solution (7.5 g/L)

77.1 Dissolve 1.50 g of nitroso-R salt in about 150 mL of water, filter, and dilute to 200 mL. This solution is stable for only 1 week.

78. Reagent No. 129—Citric Acid Solution (100 g/L)

78.1 Dissolve 100 g of citric acid in water and dilute to 1 L. The addition of 1 g of benzoic acid per litre will prevent bacterial growth.

79. Reagent No. 130—8-Quinolinol (8-Hydroxyquinoline) Solution (25 g/L)

79.1 Dissolve 12.5 g of 8-quinolinol in 25 mL of acetic acid, and dilute to 500 mL with water. Filter if necessary.

80. Reagent No. 131—Hydroxylamine Hydrochloride Solution (100 g/L)

80.1 Dissolve 5.0 g of hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in 50 mL of water. Prepare fresh as needed.

81. Reagent No. 132—2,9-Dimethyl-1,10-Phenanthroline (Neocuproine) Solution (1 g/L)

81.1 Dissolve 0.1 g of neocuproine in 100 mL of absolute ethanol.

82. Reagent No. 133—Silver Nitrate Solution (8 g/L)

82.1 Dissolve 8 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

83. Reagent No. 134—Potassium Permanganate Solution (20 g/L)

83.1 Dissolve 20 g of potassium permanganate ($KMnO_4$) in water and dilute to 1 L.

84. Reagent No. 135—Barium Chloride Solution (100 g/L)

84.1 Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in water, and dilute to 1 L.

85. Reagent No. 136—Boric Acid Solution (50 g/L)

85.1 Dissolve 50 g of boric acid (H_3BO_3) in hot water, cool, and dilute to 1 L.

86. Reagent No. 137—Sodium Thiocyanate Solution (100 g/L)

86.1 Dissolve 100 g of sodium thiocyanate (NaSCN) in about 500 mL of water, filter, and dilute to 1 L. Store in a dark bottle.

87. Reagent No. 138—Stannous Chloride Solution (50 g/L)

87.1 Dissolve 5 g of stannous chloride dihydrate ($SnCl_2 \cdot 2H_2O$) in 10 mL of HCl, and dilute to 100 mL with water. Prepare just before use.

88. Reagent No. 139—Ammonium Phosphate Solution (100 g/L)

88.1 Dissolve 100 g of ammonium phosphate, dibasic ($(NH_4)_2HPO_4$), in water, and dilute to 1 L. Store in a plastic bottle.

89. Reagent No. 140—Ammonium Thiocyanate Solution (100 g/L)

89.1 Dissolve 100 g of ammonium thiocyanate (NH_4SCN) in water and dilute to 1 L. Filter, if necessary, and store in a plastic bottle.

90. Reagent No. 141—Hydrogen Sulfide Wash Solution

90.1 Saturate H_2SO_4 (1 + 99) with hydrogen sulfide (H_2S). Prepare fresh as needed.

91. Reagent No. 142—Potassium Iodide Solution (100 g/L)

91.1 Dissolve 10 g of potassium iodide (KI) in water, and dilute to 100 mL. Prepare fresh as needed.

92. Reagent No. 143—Potassium Cyanide Solution (50 g/L)

92.1 Dissolve 50 g of potassium cyanide (KCN) in water, and dilute to 1 L. Store in a plastic bottle.

NOTE 14—**Caution:** The preparation, storage, and use of KCN solutions require care and attention. Avoid inhalation of fumes and exposure of the skin to the chemical or its solutions. Work in a well-ventilated hood.

93. Reagent No. 144—Eriochrome Black-T Indicator Solution (8 g/L)

93.1 Dissolve 0.4 g of the sodium salt of eriochrome black-T in a mixture of 20 mL of ethanol and 30 mL of triethanolamine. This solution is stable for at least 3 months when kept in a tightly closed plastic dropping bottle.

94. Reagent No. 145—1,10-Phenanthroline (*o*-Phenanthroline) Solution (2 g/L)

94.1 Dissolve 0.4 g of 1,10-phenanthroline monohydrate in 200 mL of water. Filter if necessary.

95. Reagent No. 146—Sodium Hydroxide Solution (100 g/L)

95.1 Dissolve 100 g of sodium hydroxide (NaOH) in about 100 mL of water. When dissolution is complete, cool, and dilute to 1 L. Store in a plastic bottle.

96. Reagent No. 147—Mercuric Chloride Solution (Saturated)

96.1 Add 60 to 100 g of mercuric chloride ($HgCl_2$) to 400 mL of hot water, shake, and cool to room temperature. More water may be added from time to time as long as crystals remain undissolved.

97. Reagent No. 148—Ferric Nitrate Solution (200 g/L)

97.1 Dissolve 20 g of ferric nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$) in water, and dilute to 100 mL.

98. Reagent No. 149—Ammonium Acetate Solution (200 g/L)

98.1 Dissolve 200 g of ammonium acetate (CH_3COONH_4) in water, and dilute to 1 L.

99. Reagent No. 150—Nessler Reagent

99.1 *Solution No. 1*—Dissolve 143 g of sodium hydroxide (NaOH) in 150 mL of water and then add 550 mL of water.

99.2 *Solution No. 2*—Dissolve 50 g of red mercuric iodide (HgI_2) and 40 g of potassium iodide (KI) in 200 mL of water.

99.3 Pour Solution No. 2 into Solution No. 1 and dilute to 1 L. Allow to settle and use the clear supernatant liquid.

100. Reagent No. 151—Ferrous Sulfate Solution (0.025 *M*)

100.1 Dissolve 6.95 g of ferrous sulfate ($FeSO_4 \cdot 7H_2O$) in 500 mL of water and dilute to 1 L.

101. Keywords

101.1 apparatus; chemical analysis; reagents; safety

TABLE 3 Stoichiometrical Equivalents for Standard Solutions[A,B]

Reagent Number	Standard Solution	Equivalent of 1.0000 mL of 1.0000 *N* Solution	
		Reagent Contained in Solution, *g*	Equivalent in Terms of Element or Compound Named, *g*
1	As_2O_3	0.04946	0.03746 As
			0.52840 $H_4Ce(SO_4)_4$
			0.03161 $KMnO_4$
3	$H_4Ce(SO_4)_4$	0.52840	0.04946 As_2O_3
			0.05585 Fe
			0.06700 $Na_2C_2O_4$
5	$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$	0.39214	0.52840 $H_4Ce(SO_4)_4$
			0.01733 Cr
			0.03161 $KMnO_4$
7	I_2	0.12690	0.03746 As
			0.06088 Sb
			0.05935 Sn
9	$KBrO_3$	0.02783	0.03746 As
			0.06088 Sb
	KCN	0.13024	0.02936 Ni
10	$K_2Cr_2O_7$	0.04903	0.01733 Cr
			0.05585 Fe
11	$K_4Fe(CN)_6 \cdot 3H_2O$	0.14080	0.03269 Zn
12	KIO_3	0.03567	0.03746 As
			0.24818 $Na_2S_2O_3 \cdot 5H_2O$
			0.01603 S
			0.05935 Sn
13	$KMnO_4$	0.03161	0.04946 As_2O_3
			0.02004 Ca
			0.04645 Cb
			0.01733 Cr
			0.05585 Fe
			0.01099 Mn
			0.03198 Mo
			0.06700 $Na_2C_2O_4$
			0.05094 V
14	$AgNO_3$	0.16987	0.03646 HCl
			0.13024 KCN
			0.02936 Ni
15	$NaAsO_2$	0.06496	0.01099 Mn
16	NaOH	0.04000	(0.0107)[C] Al
			0.03646 HCl
			0.20423 $KHC_8H_4O_4$
			0.001347 P
17	$Na_2S_2O_3 \cdot 5H_2O$	0.24818	0.06354 Cu
			0.12690 I
			0.03567 KIO_3
			0.00304 Mg
			0.01974 Se

[A] These equivalents are based on the 1965 Table of Relative Atomic Weights of the International Commission on Atomic Weights based on Atomic Mass of C^{12} = 12.

[B] These equivalents are calculated for the reactions involved in the procedures in which they are used in this book. They may not pertain to other procedures.

[C] This equivalent is empirical; the theoretical equivalent is 0.01079.

TABLE 4 Chemical Reagents Specified in ASTM Methods for Chemical Analysis of Metals

Name	Formula
*Acetic acid	CH_3COOH
Acetone	CH_3COCH_3
Acetylacetone (2,4-pentanedione)	$CH_3COCH_2COCH_3$
Alizarin-Red-S	$C_6H_4COC_6H$-1,2-$(OH)_2$-3-SO_3NaCO
Aluminon (aurintricarboxylic acid-ammonium salt)	(4-HOC_6H_3-3-$COONH_4)_2C$:C_6H-3-$(COONH_4)$:O
Aluminum metal (99.9 % min)	Al
*Aluminum metal (sheet or rolled foil)	Al

TABLE 4 *Continued*

Name	Formula
Aluminum ammonium sulfate	$Al_2(NH_4)_2(SO_4)_4\cdot24H_2O$
Aluminum nitrate	$Al(NO_3)_3\cdot9H_2O$
Aluminum sulfate	$Al_2(SO_4)_3\cdot18H_2O$
Aluminum oxide, fused (Alundum)	
1-Amino-2-naphthol-4-sulfonic acid	$NH_2C_{10}H_5(OH)SO_3H$
Ammonium acetate	CH_3COONH_4
Ammonium benzoate	$C_6H_5COONH_4$
Ammonium bifluoride	NH_4FHF
Ammonium bisulfate	NH_4HSO_4
Ammonium bisulfite	NH_4HSO_3
Ammonium carbonate	$(NH_4)_2CO_3$
*Ammonium chloride	NH_4Cl
*Ammonium citrate	$CH_2(COONH_4)C(OH)(COOH)CH_2COONH_4$
Ammonium fluoride	NH_4F
*Ammonium hydroxide[A]	NH_4OH
Ammonium iodide	NH_4I
Ammonium molybdate	$(NH_4)_2MoO_4$
*Ammonium heptamolybdate tetrahydrate	$(NH_4)_6Mo_7O_{24}\cdot4H_2O$
Ammonium nitrate	NH_4NO_3
*Ammonium oxalate	$NH_4OCOCOONH_4\cdot H_2O$
*Ammonium phosphate, dibasic (diammonium acid phosphate)	$(NH_4)_2HPO_4$
*Ammonium persulfate (ammonium peroxydisulfate)	$(NH_4)_2S_2O_8$
*Ammonium sulfate	$(NH_4)_2SO_4$
*Ammonium tartrate	$NH_4OCO(CHOH)_2COONH_4$
Ammonium thiocyanate	NH_4SCN
Ammonium vanadate	NH_4VO_3
Antimony metal (powder)	Sb
Antimony trichloride	$SbCl_3$
*Arsenic trioxide	As_2O_3
Asbestos (for use with Gooch crucible)	
Barium Chloride	$BaCl_2\cdot2H_2O$
Barium diphenylamine sulfonate	$(C_6H_5NHC_6H_4\text{-}4\text{-}SO_3)_2Ba$
*Benzoic acid	C_6H_5COOH
α-Benzoin oxime (benzoin anti-oxime)	$C_6H_5CHOHC{:}NOHC_6H_5$
Beryllium sulfate	$BeSO_4\cdot4H_2O$
Bismuth metal (99.9 % min)	Bi
Boric acid	H_3BO_3
Bromocresol green (3′,3″,5′,5″-tetrabromo-*m*-cresolsulfonephthalein)	$C_6H_4SO_2OC(C_6H\text{-}3,5\text{-}Br_2\text{-}2\text{-}CH_3\text{-}4\text{-}OH)_2$
Bromocresol purple (5′,5″-Dibromo-*o*-cresolsulfonephthalein)	$C_6H_4SO_2OC(C_6H_2\text{-}3\text{-}CH_3\text{-}5\text{-}Br\text{-}4\text{-}OH)_2$
Bromine (liquid)	Br_2
Bromophenol blue (3′,3″,5′,5″-tetrabromophenolsulfonephthalein)	$C_6H_4SO_2OC(C_6H_2\text{-}3,5\text{-}Br_2\text{-}4\text{-}OH)_2$
1-Butanol	$CH_3CH_2CH_2CH_2OH$
Butyl acetate (normal)	$CH_3COOCH_2CH_2CH_2CH_3$
*Cadmium chloride	$CdCl_2\cdot2\frac{1}{2}\,H_2O$
Cadmium chloride, anhydrous	$CdCl_2$
*†Calcium carbonate (low-boron)	$CaCO_3$
Carbon dioxide (gas)	CO_2
Carbon dioxide (solid)	CO_2
Carbon tetrachloride	CCl_4
Carminic acid	$1,3,4\text{-}(HO)_3\text{-}2\text{-}C_6H_{11}O_6C_6COC_6H\text{-}5\text{-}COOH\text{-}6\text{-}OH\text{-}8\text{-}CH_3CO$
*Chloroform	$CHCl_3$
Cinchonine	$C_{19}H_{22}N_2O$
Citric acid	$HOC(COOH)(CH_2COOH)_2$
Cobalt metal	Co
Cobalt sulfate	$CoSO_4$
Coke	
Congo red test paper	
Copper metal (99.9 % min)	Cu
*Copper metal (powder or turnings)	Cu
Copper metal (P-free)	Cu
Copper metal (Mn, Ni, and Co-free, less than 0.001 % of each)	Cu
Copper-rare earth oxide mixture	
m-Cresol purple (*m*-cresolsulfonephthalein)	$C_6H_4SO_2OC(C_6H_3\text{-}2\text{-}CH_3\text{-}4\text{-}OH)_2$
Cupferron	$C_6H_5N(NO)ONH_4$
Cupric chloride	$CuCl_2\cdot2H_2O$
*Cupric nitrate	$Cu(NO_3)_2\cdot3H_2O$
*Cupric oxide (powder)	CuO
Cupric potassium chloride	$CuCl_2\cdot2KCl\cdot2H_2O$
*Cupric sulfate	$CuSO_4\cdot5H_2O$

TABLE 4 *Continued*

Name	Formula
Curcumin	$(2-CH_3OC_6H_3-1-OH-4-CH:CHCO)_2CH_2$
Devarda's alloy	50Cu-45Al-5Zn
Diethylenetriamine pentaacetic acid ([[(carboxymethyl)imino]bis(ethylenenenitrilo)] tetraacetic acid)	$((HOCOCH_2)_2NCH_2CH_2)_2NCH_2COOH$
*Dimethylglyoxime	$CH_3C:NOHC:NOHCH_3$
N,N′ Diphenylbenzidine	$C_6H_5NHC_6H_4C_6H_4NHC_6H_5$
Diphenylcarbazide (1,5-diphenylcarbohydrazide)	$C_6H_5NHNHCONHNHC_6H_5$
*Disodium (ethylenedinitrilo) tetraacetate dihydrate	See (ethylenedinitrilo) tetraacetic acid disodium salt
Dithiol (toluene-3,4-dithiol)	$CH_3C_6H_3(SH)_2$
Dithizone (diphenylthiocarbazone)	$C_6H_5NHNHCSN:NC_6H_5$
Eriochrome black-T (1(1-hydroxy-2-naphthylazo)-6-nitro-2-naphthol-4-sulfonic acid sodium salt)	$1-HOC_{10}H_6-2-N:N-1-C_{10}H_4-2-OH-4-SO_3Na-6-NO_2$
*EDTA (Disodium salt)	See (ethylenedinitrilo) tetraacetic acid disodium salt
*Ethanol	C_2H_5OH
*Ethyl ether (diethyl ether)	$C_2H_5OC_2H_5$
*(Ethylenedinitrilo) tetraacetic acid disodium salt	$HOCOCH_2(NaOCOCH_2)NCH_2N(CH_2COONa)CH_2COOH\cdot 2H_2O$
Ethylene glycol monomethyl ether (2-methoxy-ethanol)	$CH_3OCH_2CH_2OH$
*Ferric chloride	$FeCl_3\cdot 6H_2O$
*Ferric nitrate	$Fe(NO_3)_3\cdot 9H_2O$
Ferric sulfate	$Fe_2(SO_4)_3\cdot nH_2O$
*Ferrous ammonium sulfate	$Fe(NH_4)_2(SO_4)_2\cdot 6H_2O$
*Ferrous sulfate	$FeSO_4\cdot 7H_2O$
Fluoboric acid	HBF_4
Fluorescein, sodium salt	$2NaOCOC_6H_4C:C_6H_3-3(:O)OC_6H_3-6-ONa$
Formaldehyde	HCHO
*Formic acid[A]	HCOOH
Gelatin	
Graphite	C
Glass wool	
Glycerol	$CH_2OHCHOHCH_2OH$
Hydrazine sulfate	$NH_2NH_2\cdot H_2SO_4$
*Hydrobromic acid[A]	HBr
*Hydrochloric acid[A]	HCl
*Hydrofluoric acid[A]	HF
Hydrogen chloride gas	HCl
*Hydrogen peroxide	H_2O_2
Hydrogen sulfide gas	H_2S
Hydroquinone	$1,4-(OH)_2C_6H_4$
*Hydroxylamine hydrochloride	$NH_2OH\cdot HCl$
*Hypophosphorus acid[B]	H_3PO_2
Invert sugar	
*Iodine	I_2
Iron metal or wire (99.8 % min)	Fe
Isopropyl ether	$(CH_3)_2CHOCH(CH_3)_2$
Lead metal	Pb
*Lead acetate	$Pb(CH_3COO)_2$
Lead chloride	$PbCl_2$
*Lead nitrate	$Pb(NO_3)_2$
Litmus	
Lithium fluoride	LiF
Magnesium metal (Sn-free)	Mg
Magnesium perchlorate, anhydrous	$Mg(ClO_4)_2$
*Magnesium sulfate	$MgSO_4\cdot 7H_2O$
Manganese metal (99.8 % min)	Mn
Manganous nitrate	$Mn(NO_3)_2$
Manganous sulfate	$MnSO_4\cdot H_2O$
Mannitol	$CH_2OH(CHOH)_4CH_2OH$
Marble chips	
*Mercuric chloride	$HgCl_2$
*Mercury	Hg
*Methanol	CH_3OH
Methyl isobutyl ketone (4-methyl-2-pentanone)	$CH_3COCH_2CH(CH_3)_2$
*Methyl orange (*p*[[*p*-dimethylamino)phenyl]azo]benzenesulfonic acid sodium salt)	$4-NaOSO_2C_6H_4N:NC_6H_4-4-N(CH_3)_2$
Methyl purple	formula unknown, patented
*Methyl red (*o*-[[(*p*-dimethylamino)phenyl]azo]benzoic acid)	$4-(CH_3)_2NC_6H_4N:NC_6H_4-2-COOH$
Molybdenum metal (99.8 % min)	Mo
Molybdic acid, anhydride (molybdenum trioxide)	MoO_3
Molybdic acid (ammonium paramolybdate)	Assay: as MoO_3—85 %
Morin, anhydrous (2′,3,4′,7-penta hydroxyflavone)	$5,7-(HO)_2C_6H_2OC(C_6H_3-2,4-(OH)_2):C(OH)CO$
β-Naphthoquinoline (5,6-benzoquinoline)	$C_{10}H_6CH:CHCH:N$
Neocuproine (2,9-dimethyl-1,10-phenanthroline)	$(CH_3)_2C_{12}H_6N_2\cdot{}_{12}H_2O$
Nickel metal (99.8 % min)	Ni
Nickel metal (sheet)	Ni

TABLE 4 *Continued*

Name	Formula
Nickelous nitrate	$Ni(NO_3)_2\cdot 6H_2O$
Nickelous sulfate	$NiSO_4\cdot 6H_2O$
*Nitric acid[A]	HNO_3
Nitrogen gas (oxygen-free)	N_2
Nitrogen, liquid	N_2
_m_-Nitrophenol	$NO_2C_6H_4OH$
1-Nitroso-2-naphthol(α-nitroso-β-naphthol)	$NOC_{10}H_6OH$
Nitroso-R-salt (1-nitroso-2-naphthol-3,6-disulfonic acid disodium salt)	$1\text{-}NOC_{10}H_4\text{-}2\text{-}(OH)\text{-}3,6\text{-}(SO_3Na)_2$
Osmium tetraoxide	OsO_4
Oxalic acid	$(COOH)_2$
Oxygen gas	O_2
*Perchloric acid[A]	$HClO_4$
1,10-Phenanthroline (_o_-phenanthroline)	$CH{:}CHCH{:}NC{:}CCH{:}CHC{:}CN{:}CHCH{:}CH\cdot H_2O$
*Phenolphthalein	$C_6H_4COOC(C_6H_4\text{-}4\text{-}OH)_2$
*Phosphoric acid	H_3PO_4
Piperidine	$NH(CH_2)_4CH_2$
Platinized quartz	
Platinized silica gel	
Platinum gauze	Pt
*Potassium biphthalate	$1\text{-}KOCOC_6H_4\text{-}2\text{-}COOH$
Potassium bisulfate	$KHSO_4$
*Potassium bromate	$KBrO_3$
*Potassium bromide	KBr
*Potassium chlorate	$KClO_3$
*Potassium chloride	KCl
*Potassium chromate	K_2CrO_4
Potassium columbate	$4K_2O\cdot 3Cb_2O_5\cdot 16H_2O$
*Potassium cyanide	KCN
*Potassium dichromate	$K_2Cr_2O_7$
*Potassium ferricyanide	$K_3Fe(CN)_6$
*Potassium ferrocyanide	$K_4Fe(CN)_6\cdot 3H_2O$
*Potassium fluoride	$KF\cdot 2H_2O$
*Potassium hydroxide	KOH
*Potassium iodate	KIO_3
*Potassium iodide	KI
Potassium iodide starch paper	
*Potassium nitrate	KNO_3
*Potassium _m_-periodate	KIO_4
*Potassium permanganate	$KMnO_4$
Potassium persulfate	$K_2S_2O_8$
Potassium phosphate, monobasic	KH_2PO_4
*Potassium pyrosulfate	$K_2S_2O_7$
*Potassium sulfate	K_2SO_4
Potassium tantalum fluoride	K_2TaF
Potassium thiocarbonate	K_2CS_3
*Potassium thiocyanate	$KSCN$
Pyrogallic acid (pyrogallol)	$C_6H_3\text{-}1,3\text{-}(OH)_3$
Quinine sulfate	$(C_{20}H_{24}N_2O_2)_2\cdot H_2SO_4\cdot 2H_2O$
8-Quinolinol (8-hydroxyquinoline)	$HOC_6H_3N{:}CHCH{:}CH$
Sebacic acid	$HOCO(CH_2)_8COOH$
Selenium (powder)	Se
Silicon dioxide (silica)	SiO_2
*Silver nitrate	$AgNO_3$
Soda-lime	
Soda-mica mineral (CO_2 absorbent)	
Sodium acetate	CH_3COONa
Sodium arsenite	$NaAsO_2$
Sodium azide	NaN_3
*Sodium bicarbonate	$NaHCO_3$
*Sodium bismuthate	$NaBiO_3$
Sodium bisulfate	see sodium hydrogen sulfate
*Sodium bisulfate, fused	see sodium hydrogen sulfate, fused
Sodium bisulfite	$NaHSO_3$
*Sodium borate	$Na_2B_4O_7\cdot 10H_2O$
*Sodium carbonate, anhydrous	Na_2CO_3
Sodium chlorate	$NaClO_3$
Sodium chloride	$NaCl$
Sodium citrate	$HOC(COONa)(CH_2COONa)_2\cdot 2H_2O$
Sodium cyanide	$NaCN$
Sodium diethyldithiocarbamate	$(C_2H_5)_2NCSSNa\cdot 3H_2O$
Sodium dimethylglyoximate	$CH_3C({:}NONa)C({:}NONa)CH_3\cdot 8H_2O$

TABLE 4 *Continued*

Name	Formula
Sodium diphenylamine sulfonate	$C_6H_5NHC_6H_4$-4-SO_3Na
Sodium dithionite (hydrosulfite)	$Na_2S_2O_4$
*Sodium fluoride	NaF
Sodium hydrogen sulfate	$NaHSO_4$
Sodium hydrogen sulfate, fused	A mixture of $Na_2S_2O_7$ and $NaHSO_4$
*Sodium hydroxide	$NaOH$
Sodium hypophosphite	$NaH_2PO_2 \cdot H_2O$
Sodium molybdate	$Na_2MoO_4 \cdot 2H_2O$
Sodium nitrate	$NaNO_3$
Sodium nitrite	$NaNO_2$
Sodium oxalate	$NaOCOCOONa$
Sodium perchlorate	$NaClO_4$
Sodium peroxide	Na_2O_2
Sodium phosphate, dibasic, anhydrous	Na_2HPO_4
Sodium pyrophosphate	$Na_4P_2O_7 \cdot 10H_2O$
Sodium pyrosulfate	$Na_2S_2O_7$
Sodium sulfate, anhydrous	Na_2SO_4
Sodium sulfide	$Na_2S \cdot 9H_2O$
Sodium sulfite	$Na_2SO_3 \cdot 7H_2O$
Sodium sulfite, anhydrous	Na_2SO_3
Sodium thiocyanate	$NaSCN$
*Sodium thiosulfate	$Na_2S_2O_3 \cdot 5H_2O$
*Sodium tungstate	$Na_2WO_4 \cdot 2H_2O$
*Stannous chloride	$SnCl_2 \cdot 2H_2O$
*Starch	$(C_6H_{10}O_5)_x$
Succinic acid	$HOCOCH_2CH_2COOH$
Sulfamic acid	NH_2SO_3H
Sulfatoceric acid (ceric sulfate)	$H_4Ce(SO_4)_4$
5-Sulfosalicylic acid	2-HOC_6H_3-1-COOH-5-$SO_3H \cdot 2H_2O$
Sulfur dioxide gas	SO_2
*Sulfuric acid[A]	H_2SO_4
*Sulfurous acid[A]	H_2SO_3
Talc	
*Tartaric acid	$HOCO(CHOH)_2COOH$
Test lead	Pb
Tetrapropylammonium hydroxide	$(CH_3CH_2CH_2)_4NOH$
Thioglycollic acid (mercaptoacetic acid)	$CH_2SHCOOH$
Thiourea	NH_2CSNH_2
Tin metal (99.9 %min)	Sn
Titanium dioxide	TiO_2
Titanium metal (low Sn)	Ti
Triethanolamine (2,2′,2″-nitrilotriethanol)	$(CH_2OHCH_2)_3N$
Uranium oxide	U_3O_8
*Uranyl nitrate	$UO_2(NO_3)_2 \cdot 6H_2O$
Urea	NH_2CONH_2
Zinc (99.9 % min)	Zn
Zinc metal (S-free)	Zn
Zinc oxide	ZnO
Zinc sulfate	$ZnSO_4 \cdot 7H_2O$
Zirconium oxide	ZrO_2
Zirconium metal	Zr
Zirconyl chloride	$ZrOCl_2 \cdot 8H_2O$

[A] * Reagent on which ACS specifications exist.

† ACS specification exists but does not cover all requirements.

For concentration of laboratory reagent, see Table 1.

[B] Contains at least 50 % H_3PO_2.

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