Designation: E50 – 11 (Reapproved 2016)

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 E50; 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 U.S. 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 will be found listed or specified in the individual methods of analysis. Included here are general recommendations on the purity of reagents and protective measures for the use of hazardous reagents.

1.2 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 4.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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 8.

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:*[2]

D1193 Specification for Reagent Water
E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers
E100 Specification for ASTM Hydrometers
E126 Test Method for Inspection, Calibration, and Verification of ASTM Hydrometers
E287 Specification for Laboratory Glass Graduated Burets
E288 Specification for Laboratory Glass Volumetric Flasks
E438 Specification for Glasses in Laboratory Apparatus
E542 Practice for Calibration of Laboratory Volumetric Apparatus
E694 Specification for Laboratory Glass Volumetric Apparatus
E969 Specification for Glass Volumetric (Transfer) Pipets
E1044 Specification for Glass Serological Pipets (General Purpose and Kahn)
E1621 Guide for Elemental Analysis by Wavelength Dispersive X-Ray Fluorescence Spectrometry

3. Terminology

3.1 For definitions of terms used in these practices, refer to Terminology E135.

4. Significance and Use

4.1 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. Apparatus, standard solutions, and certain other reagents shall conform to the requirements prescribed in ASTM Practices E50, for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials."

4.2 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.

5. Purity of Water and Reagents

5.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming

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[1] These practices are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.20 on Fundamental Practices.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 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	$C_6H_4COC_6H\text{-}1,2\text{-}(OH)_2\text{-}3\text{-}SO_3NaCO$
Aluminon (aurintricarboxylic acid-ammonium salt)	$(4\text{-}HOC_6H_3\text{-}3\text{-}COONH_4)_2C:C_6H\text{-}3\text{-}(COONH_4):O$
Aluminum metal (99.9 % min)	Al
* Aluminum metal (sheet or rolled foil)	Al
Aluminum ammonium sulfate	$Al_2(NH_4)_2(SO_4)_4\cdot 24H_2O$
Aluminum nitrate	$Al(NO_3)_3\cdot 9H_2O$
Aluminum sulfate	$Al_2(SO_4)_3\cdot 18H_2O$
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	$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$
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	$BaCl_2\cdot 2H_2O$
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	$BeSO_4\cdot 4H_2O$
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	$CdCl_2\cdot 2\frac{1}{2}\ H_2O$
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

TABLE 1 *Continued*

Name	Formula
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	$CuCl_2\cdot 2H_2O$
* Cupric nitrate	$Cu(NO_3)_2\cdot 3H_2O$
* Cupric oxide (powder)	CuO
Cupric potassium chloride	$CuCl_2\cdot 2KCl\cdot 2H_2O$
* Cupric sulfate	$CuSO_4\cdot 5H_2O$
Curcumin	$(2\text{-}CH_3OC_6H_3\text{-}1\text{-}OH\text{-}4\text{-}CH:CHCO)_2CH_2$
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)	$1\text{-}HOC_{10}H_6\text{-}2\text{-}N:N\text{-}1\text{-}C_{10}H_4\text{-}2\text{-}OH\text{-}4\text{-}SO_3Na\text{-}6\text{-}NO_2$
* 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-methoxyethanol)	$CH_3OCH_2CH_2OH$
* 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	$FeSO_4\cdot 7H_2O$
Fluoroboric acid	HBF_4
Fluorescein, sodium salt	$2NaOCOC_6H_4C:C_6H_3\text{-}3(:O)OC_6H_3\text{-}6\text{-}ONa$
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	$1,4\text{-}(OH)_2C_6H_4$
* Hydroxylamine hydrochloride	$NH_2OH\cdot HCl$
* Hypophosphorous 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	$Mg(ClO_4)_2$

TABLE 1 *Continued*

Name	Formula
* 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)	$4\text{-}NaOSO_2C_6H_4N{:}NC_6H_4\text{-}4\text{-}N(CH_3)_2$
Methyl purple	formula unknown, patented
* Methyl red (*o* -[[(*p*-dimethylamino)phenyl]azo]benzoic acid)	$4\text{-}(CH_3)_2NC_6H_4N{:}NC_6H_4\text{-}2\text{-}COOH$
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)	$5,7\text{-}(HO)_2C_6H_2\ OC(C_6H_3\text{-}2,4\text{-}(OH)_2){:}C(OH)CO$
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
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

TABLE 1 *Continued*

Name	Formula
Pyrogallic acid (pyrogallol)	$C_6H_3\text{-1,3-}(OH)_3$
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	$CH_3C({:}NONa)C({:}NONa)CH_3\cdot 8H_2O$
Sodium diphenylamine sulfonate	$C_6H_5NHC_6H_4\text{-4-}SO_3Na$
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	$2\text{-}HOC_6H_3\text{-1-}COOH\text{-5-}SO_3H\cdot 2H_2O$
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

TABLE 1 *Continued*

Name	Formula
Zinc (99.9 % min)	Zn
Zinc metal (S-free)	Zn
Zinc oxide	ZnO
Zinc sulfate	$ZnSO_4·7H_2O$
Zirconium oxide	ZrO_2
Zirconium metal	Zr
Zirconyl chloride	$ZrOCl_2·8H_2O$

[A] * Reagent on which ACS specifications exist.
† ACS specification exists but does not cover all requirements.
For concentration of laboratory reagent, see Table 2.

[B] Contains at least 50 % H_3PO_2.

to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

5.2 *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.[3] 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.

6. Reagents

6.1 *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 2 are intended. The specific gravities or concentrations of all other concentrated acids are stated wherever they are specified.

6.2 *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.

6.3 *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..."

6.4 *Nonstandard Solutions*—Composition of nonstandard solutions prepared by dissolving a given mass of the solid reagent in a solvent are specified in grams of the salt as weighed 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 ($BaCl_2·2H_2O$) in water and dilute to 1 L. In the case of certain reagents, the composition may be specified as a mass fraction percent. For example, H_2O_2 (3 %) means a solution containing 3 g of H_2O_2 per 100 g of solution. Other nonstandard solutions may be specified by name only and the designation of the composition of such solutions will be governed by the instructions for their preparation.

7. Laboratory Ware (1,2)[4,5]

7.1 *Glassware*—Unless otherwise stated all analytical methods are conducted in borosilicate glassware.

TABLE 2 Composition of Acids, Ammonium Hydroxide, and Hydrogen Peroxide

Name	Formula	Specific Gravity, Approximate	Reagent, Mass Fraction, %		
			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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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, www.chemistry.org . For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia* and *4.2 National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, www.usp.org.

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

7.1.1 *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.[5]

7.1.2 *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.

7.2 *Plastic Labware:*

7.2.1 *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 plastic labware (see 7.2.4).

7.2.2 *Physical Properties*—There are a number of physical properties which influence the usefulness of plastic labware (Table 3).

7.2.3 *Compatibility*—Many reagents can affect the strength, flexibility, surface appearance, color, dimensions, or weight of plastics. The two basic modes of interaction that can cause these changes are described in 7.2.3.1 – 7.2.3.4.

7.2.3.1 *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.[6]

7.2.3.2 *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, they are best not used 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. 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.

7.2.3.3 Some plastics may contain small amounts 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.

7.2.3.4 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, amount, 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.[6] The type of plastic labware (see footnote *B* of Table 3) will be found specified in the method as well as any special precautions for its use.

7.2.4 *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 3.[7] With proper precaution polytetrafluoroethylene labware may be used with strong oxidizing agents at elevated temperatures (see Table 3). 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.

8. Hazards (see Refs 3-7)

8.1 *General Requirements*—Nearly all procedures conducted in the chemical laboratory are potentially hazardous. Each of the procedures used in these methods of chemical

TABLE 3 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.

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[5] For further information the following ASTM Standards may be consulted: Volumetric Labware: Specifications E287, E288, and E438; Practice E542; and Specifications E694, E969, and E1044. Thermometers: Specification E1 and Test Method E77. Hydrometers: Specification E100 and Test Method E126.

[6] From the publications of the Nalgene Labware, www.nalgenelabware.com.

[7] 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.

TABLE 4 Stoichiometrical Equivalents for Standard Solutions[A]

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*
As_2O_3	0.04946	0.03746 As 0.52840 $H_4Ce(SO_4)_4$ 0.03161 $KMnO_4$
$H_4Ce(SO_4)_4$	0.52840	0.04946 As_2O_3 0.05585 Fe 0.06700 $Na_2C_2O_4$
$Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$	0.39214	0.52840 $H_4Ce(SO_4)_4$ 0.01733 Cr 0.03161 $KMnO_4$
I_2	0.12690	0.03746 As 0.06088 Sb 0.05935 Sn
$KBrO_3$	0.02783	0.03746 As 0.06088 Sb
KCN	0.13024	0.02936 Ni
$K_2Cr_2O_7$	0.04903	0.01733 Cr 0.05585 Fe
$K_4Fe(CN)_6{\cdot}3H_2O$	0.14080	0.03269 Zn
KIO_3	0.03567	0.03746 As 0.24818 $Na_2S_2O_3{\cdot}5H_2O$ 0.01603 S 0.05935 Sn
$KMnO_4$	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
$AgNO_3$	0.16987	0.03646 HCl 0.13024 KCN 0.02936 Ni
$NaAsO_2$	0.06496	0.01099 Mn
NaOH	0.04000	(0.0107)[B] Al 0.03646 HCl 0.20423 $KHC_8H_4O_4$ 0.001347 P
$Na_2S_2O_3{\cdot}5H_2O$	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] This equivalent is empirical; the theoretical equivalent is 0.01079.

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:

8.1.1 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.

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

8.1.3 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.

8.2 *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:

8.2.1 Definitions of areas and personnel covered,

8.2.2 General safety rules,

8.2.3 Rules covering specific areas of operations,

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

8.2.5 Lists of safety equipment according to location and type,

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

8.2.7 Descriptions of the duties and identities of personnel who will implement and conduct the provisions of this plan.

8.3 *Personal Protective Equipment:*

8.3.1 *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.

8.3.2 *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.

8.3.3 *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:

8.3.3.1 Face shields, portable shields, hood sash shields,

8.3.3.2 Ear plugs, sound barrier ear muffs,

8.3.3.3 Lab coats, lab aprons, sleeve protectors,

8.3.3.4 Respirators, gas masks, self-contained breathing apparatus, and

8.3.3.5 Safety shoes/boots.

8.4 *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.

8.4.1 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.

8.4.2 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.

8.4.3 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 Hazards section of Guide E1621 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 spectrometric emission sources, electrodeless discharge lamps, and nitrous oxide/acetylene flames.

8.4.4 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 amounts 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 amounts 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 installed 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 ensure 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.

8.4.5 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 (8.5.10).

8.4.6 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.

8.4.7 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.

8.4.8 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.

8.5 *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 (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.

8.5.1 Storage of reagents, chemicals, and solvents should consider 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.

8.5.2 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 amounts 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.

8.5.3 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.

8.5.4 Elemental mercury has an appreciable vapor pressure. Hazardous amounts 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.

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

8.5.6 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.

8.5.7 Hydrogen sulfide is more toxic than hydrogen cyanide. It is readily detected at low amounts 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.

8.5.8 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.

8.5.9 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.

8.5.10 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.

8.5.11 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.

8.5.12 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.

8.5.13 Hydrogen peroxide is commonly used at compositions of 30 % and lower. At these compositions the reagent is safer to handle than at higher compositions 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.

8.5.14 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.

8.5.15 *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.

8.5.16 *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.[8]

8.6 *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.

9. Keywords

9.1 apparatus; chemical analysis; reagents; safety

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[8] A valuable reference on the subject of the disposal of laboratory wastes is: *Prudent Practices in the Laboratory: Handling and Disposal of Chemicals, 1995* National Research Council, National Academy Press, Washington, D.C., www.nap.edu.

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