SEMI C1-1000
SPECIFICATIONS FOR REAGENTS

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1 Preface

1.1 In the 1950's, chemicals purchased by the electronics industry were of a quality defined by the Committee on Analytical Reagents of the American Chemical Society, or of lesser quality. With recognition of the importance of impurity content of chemicals in the manufacture of semiconductors, suppliers responded by introducing products with improved analytical characterization, notably for trace elements.

1.2 The SEMI Committee on Standards for Chemical Reagents began its efforts in the fall of 1975. With this publication, the Committee establishes the definitions, general procedures, specifications, and analytical procedures for the chemicals listed in the index.

1.3 Products within all of the requirements can be described as "meeting SEMI specifications."

1.4 Where an analytical procedure different from that provided is substituted by a supplier or user, the burden of proof is on said supplier or user to confirm the equivalency.

1.5 The committee is continuing work on improvement of specifications and procedures and addition of other reagents and systems such as mixed etchants. The committee welcomes comments, suggestions, and recommendations.

1.6 This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Definitions

2.1 *Accuracy and Tolerances in Measurements* — In a test procedure, unless otherwise specified, the accuracy required in the amount of sample taken shall be understood to be within 2.0% of the stated amount, whether a weight or a volume. Where a dilution is performed to a stated volume or a stated volume of a solution is taken, that volume shall be within 2.0% of the stated amount. Unless otherwise stated, a transfer by pipet shall be made by means of a volumetric pipet conforming to the tolerances established by *National Bureau of Standards Circular* 602, April 1, 1959.

2.2 *Abbreviations and Signs* — In this work abbreviations and signs used shall be restricted to those listed in Table 2. Most of the abbreviations are identical to those used in publications of the American Chemical Society as of 1982.

2.3 *Acidity and Alkalinity* — A test for acidity or alkalinity, or both, of organic liquids is common.

2.3.1 In the *acidity test,* usually a stated volume of water (commonly 25 mL) is shaken in a glass-stoppered flask with a stated volume of sample (commonly 10 mL). Then 0.1 mL of phenolphthalein indicator solution is added and 0.01 *N* sodium hydroxide until a slight pink color persists after shaking for 30 seconds. Next, a stated amount of sample is added and mixed, and a titration is performed with 0.01 *N* sodium hydroxide until the pink color is reproduced; the volume of titrant required is recorded.

2.3.2 In the *alkalinity test*, a stated amount of sample is mixed with a stated volume of water (commonly 25 mL). Now 0.05 mL of methyl red indicator solution is added and the mixture is titrated with 0.01 *N* hydrochloric acid until a slight pink color is reached; the volume of titrant required is recorded.

2.3.3 The result of such tests has often been expressed qualitatively as "passes test" or quantitatively as the content of an acid (or base) known or assumed to be present. In this work, the maximum specification limit shall be expressed in microequivalents of acid (or base) per gram of sample (μeq/g). The maximum allowed volume of titrant in a test procedure shall correspond to that specification limit.

2.4 *Apparatus* — Apparatus, unless otherwise qualified, shall be that commonly employed in the conduct of the relevant test.

2.5 *Atomic and Formula Weights* — Atomic weights (that is, atomic masses) used shall be those of the latest report of the International Commission on Atomic Weights. Formula weights shall be calculated from a given empirical (molecular) formula by addition of the stated multiples of the atomic weights. Unless otherwise stated, formula weights shall be rounded to 2 significant figures to the right of the decimal point (see Rounding of Numbers, Section 2.18). All relevant

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factors given in this work are based on the 1975 Table of Atomic Masses.

2.6 *Blank Preparation* — Where a "blank" is specified to be run, unless otherwise stated, it shall be performed with the same quantities of the same reagents and in a manner identical to that followed with the sample portion under test, but with the material itself omitted.

2.7 *Comparison of Analytical Results with Specification Limits* — In the comparison of an analytical result for a test with the numerical limit associated with the specification, the result shall be rounded to the number of significant figures indicated for that limit. (See Rounding of Numbers, Section 2.18.). Consequently, a specification stated at 96% *minimum* will be met by a result as small as 95.5%, and that stated as 96.0% *minimum* will be met by a result as small as 95.95%. A specification of 0.1% *maximum* will be met by a result as large as 0.14%, and that of 0.10% *maximum* by a result as large as 0.105%. In those specifications given with no digit following the decimal point, such as 10. ppb maximum, a test result as large as 10.5 ppb will meet the specification. This use of a decimal point is made to clarify the difference between converting 0.01 ppm to 10 ppb and converting 0.010 ppm to 10. ppb.[1]

2.8 *Density* — The density of a liquid shall be determined for the compound in air at 25°C and be expressed in grams/milliliter (g/mL). Where the specific gravity, with respect to water, is determined, it shall be expressed as density by the use of tabular values for the density of water at the relevant temperature(s).

2.9 *Desiccator* — A desiccator shall imply a tightly closed container charged with a suitable desiccant (commonly calcium chloride or sulfate) that allows an atmosphere of low humidity to be maintained.

2.10 *Drying or Igniting to Constant Weight* — A statement "dried to a constant weight" or "ignited to a constant weight" shall imply that two separate weighings differ by no more than ± 0.4 mg (unless otherwise specified), where the second weighing follows a second drying (for one hour (unless otherwise stated) or ignition for 15 minutes (unless otherwise stated), respectively.

2.11 *Expression of Content and Concentration* — Unless otherwise stated, a specification limit and experimental results related to it shall be expressed in units of weight by weight. The concentration of a solution of a test reagent shall be expressed either in molarity or normality or as percent weight by volume. A redox normality value shall always be followed, parenthetically, by the relevant molarity. In the approximate dilution of reagents, a parenthetical expression of two numbers with an intervening "plus" sign shall imply that the relative volume of the stated reagent given by the first number shall be admixed with the relative volume of water given by the second number. Thus, "dilute" sulfuric acid (1 + 3) directs that one volume of reagent grade sulfuric acid be added to 3 volumes of water and the mixture stirred to form a uniform solution.

2.12 *Filtration* — A statement to "filter," unless qualified, shall imply filtration through suitable filter paper until the filtrate is clear.

2.13 *Heavy Metals (as Pb)* — The test described as "Heavy Metals (as Pb)" responds to many of the metals precipitated by hydrogen sulfide. Unless otherwise stated, the test shall involve a visual comparison of the color developed by the sample preparation and a lead standard ("control"), upon treatment with hydrogen sulfide at pH 3 to 4. The color comparison shall be made with matched 50 mL Nessler tubes with vertical viewing over a white background. The treatment of the sample shall be specified in detail in the test procedure. Unless otherwise stated, the heavy metals standard shall be prepared by diluting a solution containing 0.02 mg of lead with water to 25 mL, adjusting the acidity of this solution to pH 3 to 4 (using a pH meter), and then diluting with water to 40 mL, and mixing. The test shall then be conducted by adding to that standard and to 40 mL of the stated sample solution 10 mL of freshly prepared hydrogen sulfide water with mixing. The test shall have been passed if the sample solution is no darker than the standard. This test is proximate in nature and does not imply that lead is present either alone or with other heavy metals.

2.13.1 For sodium hydroxide and potassium hydroxide, this test becomes "Heavy Metals (as Ag)," and silver replaces lead in the standard. This change is appropriate as silver and silver-clad equipment are often used in the manufacture of these caustics.

2.14 *Physical Properties* — Physical properties shall not usually be employed for specification purposes; for information, however, representative values for a particular chemical, as supplied, may be included as item 2 in the monograph for that chemical. Where relevant, physical properties shall be specified for 25°C.

2.15 *Reagent Chemicals* — Unless otherwise stated, reagents to be used in tests shall conform to the minimum standards of quality set forth in the 8th Edition of *Reagent Chemicals*, published by the American Chemical Society, and any revisions thereto.

2.16 *Residue after Evaporation* — A "residue after evaporation" test is often used in the assessment of

1 H.A. Flachka, A.J. Barnard Jr., P.E. Sturrock. Quantitative Analytical Chemistry, Willard Grant Press, 2nd ed., 1980, p 4.

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organic liquids. (See Determination of Residue after Evaporation, Section 3.3.)

2.17 *Residue after Ignition* — A "residue after ignition" test serves to assess the amount of nonvolatile inorganic matter present in a sample. Unless otherwise stated, the ignition shall be performed at 800° ± 25°C for 15 minutes. Where the addition of sulfuric acid is specified, the result corresponds to what is often termed "sulfated ash." The calculation of the result, expressed in parts per million (µg/g), takes the form:

$$\text{ppm Residue after Ignition} = \frac{\text{Net weight of residue (g)} \times 10^6}{\text{Weight of sample (g)}}$$

2.18 *Rounding of Numbers* — The following rules for "rounding" of measured or calculated values shall be employed:

1. When the figure next beyond the last place to be retained *is less than 5*, leave unchanged the figure in the last place retained.
2. When the figure next beyond the last place to be retained *is greater than 5*, increase by 1 the figure in the last place retained.
3. When the figure next beyond the last place to be retained is *5 and there are no figures beyond this 5 or only zeroes*, (a) increase by 1 the figure in the last place retained if it is odd, or leave the figure unchanged if it is even.
4. When the figure next beyond the last place to be retained is *5 and there are figures other than zeroes beyond this 5*, increase by 1 the figure in the last place retained.
5. Obtain the rounded value in one step by direct rounding and not in two or more steps of successive roundings.

2.19 *Samples and Sampling* — For chemicals provided in small containers, one (or more) shall be freshly opened for testing, thereby reducing possibilities for contamination or change in composition (for example, by moisture pickup). Where the chemical is provided in larger bulk quantities, one or more drums or other containers shall be sampled appropriately, and the combined sample shall be placed in a labeled, well-cleaned container that shall be tightly closed and transferred expeditiously to the testing laboratory.

2.20 *Specifications and Specification Limits* — The specifications provided by this work are intended to serve for chemicals to be used in the manufacture and processing of semiconductors and advanced electronic devices and circuits. The specifications and the associated test procedures are based on the experience of suppliers and users and also on published studies relating to chemicals of the required quality. The function of the specifications is to establish *minimum* standards of quality.

2.20.1 Where feasible, a specification of content shall be expressed as a numerical limit in units of weight by weight. For a specification that cannot be assigned such a limit, the expression "To pass test" shall be used.

2.20.2 For a major component, the value (assay, purity, etc.) shall be expressed as a *minimum* permissible limit. For an impurity, the value shall be expressed as a *maximum* permissible limit.

2.20.3 A chemical conforming to the specification will commonly contain more of the major component than the minimum permissible limit or contain less of an impurity (or several impurities) than the maximum permissible limit. In neither case shall the chemical be considered as of higher quality than that defined by the specification.

2.20.4 It is manifestly impossible in the specifications and procedures for a chemical to consider every impurity or contaminant that might be present. For certain applications, it is recognized that more stringent or additional specifications and procedures might be required. The intent of these specifications and the associated procedures is, on one hand, to assure that a chemical is suitable for the common uses to which it may be put in the manufacture and processing of semiconductor devices and, on the other hand, to be consistent with contemporary manufacturing processes for that chemical.

2.21 *Specification Parameters* — In this work, the order of statement of specification parameters in Item 1 of the monograph for a particular chemical shall be, as far as practical: 1) assay or other test assessing the content of the major component(s); 2) appearance or color; 3) acidity or alkalinity, or both, or pH range of solution; 4) residue after evaporation or ignition; 5) water content; 6) diverse general tests, arranged alphabetically; 7) tests for stated anions, arranged alphabetically (bromide, chloride, phosphate, sulfate, sulfite, etc.); 8) ammonium; 9) heavy metals; 10) arsenic and antimony; 11) tests for specific trace elements, especially metals, arranged alphabetically by name.

2.21.1 All major words in a specification parameter shall have their initial letters capitalized.

2.21.2 Following the statement of a specification parameter, where relevant, the species on which the calculation of the result is based shall be expressed parenthetically as the atomic symbol or empirical formula.

2.22 *Tared Containers* — Where the use of a tared container is specified, it shall be carried through operations identical to those used in the test procedures,

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including drying or ignition, or both, cooling in a desiccator, and weighing. When a new container is placed in service, special measures may be required to assure that it is brought to constant weight by the operations.

2.23 *Temperature* — Temperature values shall be expressed in degrees Celsius (°C).

2.24 *Water* — References to water in the testing procedures are understood to mean distilled or deionized water which is suitable for the intended purpose. Water meeting the requirements for Water, Reagent in *Reagent Chemicals*, ACS specifications, current edition, or Type II Reagent Water in ASTM specification D1193 may be used. In tests for nitrogen compounds, water should be "ammonia-free" or "nitrogen-free." For some tests, freshly boiled water must be used in order to ensure freedom from material absorbed from the air such as ammonia, carbon dioxide, or oxygen.

2.25 *Water by Karl Fischer Titration* — The Karl Fischer method for the determination of water involves a titration with the so-called Karl Fischer reagent, consisting of iodine, sulfur dioxide, pyridine, and methanol. For most relevant chemicals intended for use in semiconductor manufacture and other electronics applications, the water content is small and electrometric detection of the end point is preferred. Details of this method are provided by ASTM E 203 and in *Reagent Chemicals*, published by the American Chemical Society.

2.26 *Water and Steam Baths* — A water bath, unless otherwise stated, shall imply a bath of vigorously boiling water. A steam bath (at 1 atmosphere pressure), unless otherwise qualified, shall imply either exposure to flowing steam or to another source of heat at the temperature of flowing steam.

2.27 *Weights and Measures* — The weights and measures used shall be, to the extent possible, those of the International System (SI), adopted by the General Conference of Weights and Measures of 1960. Some relevant quantities are listed below:

Physical Quantity	*Name*	*Symbol*	*Expression in SI Base Units*
length	meter	m	(base)
mass	kilogram	kg	(base)
time	second	s	(base)
electric current	ampere	A*	(base)
amount of substance	mole	mol	(base)
electric conductance	siemens	S	$m^{-2}•kg^{-1}•s^{3}•A^{2}$
electric potential difference	volt	V	$m^{-2}•kg•s^{-3}•A^{-1}$
electric resistance	ohm	Ω	$m^{2}•kg•s^{-3}•A^{-2}$
energy, work quantity of heat	joule	j	$m^{2}•kg•s^{-2}$
force	newton	N	$m•kg•s^{-2}$
frequency	hertz	Hz	s^{-1}
power, radiant flux	watt	W	$m^{2}•kg•s^{-3}$
pressure	pascal	Pa	$m^{-1}•kg•s^{-2}$
quantity of electricity, electric charge	coulomb	C	s•A

*In this work, the ampere is abbreviated amp to avoid confusion with absorbance.

3 General Procedures and Guidelines to Certain Methods

3.1 *Guidelines for Assay by Wide Bore Column Gas Chromatography* — The theory of gas chromatography is given in many monographs, reviews, etc. The purpose here is to delineate some practical aspects for the assessment of volatile organic reagents by gas chromatography. A complete detailed procedure for the gas chromatographic assay is not given since available instruments vary from laboratory to laboratory.

However, the use of wide bore capillary columns is recommended to replace conventional packed columns. Capillary columns, constructed of fused silica onto which is bonded a liquid phase, have greater resolving power and chemical inertness.

3.1.1 *Equipment* — A standard gas chromatograph equipped with capillary wide bore column adaptors of isothermal and multi-step linear and temperature programmed operation is recommended.

3.1.2 *Sample* — Direct flash vaporization or on-column injection of the sample with standard gauge needles can be used with wide bore columns. On column injection can minimize sample degradation while increasing the reproducibility of results. On column injection is ideal to use with very volatile materials such as the reagents assayed in this monograph.

3.1.3 *Columns* — Many types of columns exist and can be used. Until recently, packed columns in which a liquid phase is coated on a porous solid support were extensively used for assay of reagents. The introduction of wide bore capillary columns having an i.d. of typically 530 microns and a thick film of liquid phase from 1 to 5 microns allows a laboratory to use a standard packed column instrument and conditions, while gaining the advantages of capillary technology. Columns can be used either in the high resolution capillary mode (low carrier gas flow rates) to achieve optimum resolution of sample components, or at higher flow rates (20–30 mL/min) where they will perform

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packed column-like separations in a shorter time. Packed columns require a multitude of stationary phases to accomplish typical separations performed in an analytical laboratory. The increased length of capillary columns allows for better separation of components so that three columns of low (Type 1, METHYL SILICONE), moderate (Type 2, MIXED CYANO, PHENYL, METHYL SILICONE), and high (Type 3, CARBOWAX) polarity can handle the majority of analytical requirements. Columns which have been found acceptable for assay of the reagents in this manual are listed in Table 1.

3.1.4 *Conditions* — Appropriate conditions will vary from one gas chromatographic system to another. Conditions which have been found acceptable are listed under each individual standard. When using packed column in the isothermal mode, the temperature of the column is usually maintained at 10° to 20°C below the boiling point of the reagent. The efficient mass transfer associated with wide bore capillary columns may necessitate initial column temperatures to be more than 20°C below the boiling point of the reagent of interest. Temperature programming may be used to improve the shape of eluting peaks and to shorten analysis times. The remaining two heated zones (injector and detector) should be maintained at as low a temperature as possible to lessen thermal decomposition effects. Typically, for the injector this is 50°C above the boiling point of the material of interest; the detector is held 25°C above the highest temperature reached by the column, but never less than 100°C. The split or splitless mode of injection can be used depending on the chromatographic equipment available. The use of a wide bore capillary column simplifies conversion of a packed column to a capillary instrument.

3.1.5 *Detector* — Use of a thermal conductivity detector minimizes the need to correct for detector response to differences in chemical composition and is usually sufficient for determining reagent assay. To qualify trace impurities, the more sensitive flame ionization detector is recommended. The detector output, after amplification, is used to produce the chromatogram, a plot of component quantity versus elution time. Modern systems digitize the analog output, allowing direct printout of peak areas and retention times.

3.1.6 *Calculation of Results* — Results from a chromatogram, used to determine the assay of a reagent, are expressed in area percent normalized to 100%. A response factor correction for each component is required for the most accurate results, especially when the sample components differ markedly in their detector response. An internal standard reduces error due to variations in injection quantities, column, and detector conditions. The use of control charts, to aid the analyst in visualizing chromatographic variability, is suggested. To verify that assay results are valid, use of a system suitability test is recommended.

Table 1

Reagent	*Recommended[1] Column*	*Alternate[2] Column*
Acetone	III	II, I
n-Butyl Acetate	I	II
Dichloromethane	I	II
Methanol	I	
Methyl Ethyl Ketone	I	
2-Propanol	I	III
Tetrachloroethylene	I	
Toluene	I	III
Trichloroethylene	I	
Trichlorotrifluoroethane	I	
Hexamethyldisilizane[3]	I	
1-Methyl-2-Pyrrolidone	I	
1,1,1 Trichloroethane	I	

Type I Column — Methyl Silicon Bonded Phase

Type II Column — Mixed Cyano, Phenyl, Methyl Silicon Bonded Phase

Type III Column — Carbowax Bonded Phase

1. Satisfactory for both assay and SEMI-specified impurity determinations.

2. Column will separate all SEMI-specified impurities from assay component. Elution order will differ from typical given in individual reagent procedures.

3. Presilanized column recommended.

3.2 *Color*

3.2.1 *Color (APHA)* — For the rapid assessment of "colorless" liquids for contamination by colored materials, visual comparison with platinum-cobalt solution standards is appropriate, following the methodology of the American Public Health Association (APHA) established for the evaluation of the color of water.

3.2.2 *Preparation of Platinum-Cobalt Stock Solution (APHA No. 500)* — Dissolve 1.246 g of reagent grade potassium chloroplatinate, K_2PtCl_6, and 1.000 g of cobalt chloride hexahydrate, $CoCl_2 \cdot 6H_20$, in water. Add 100 mL of reagent grade hydrochloric acid (~37%) and dilute with water to exactly 1,000 mL. Store in a tightly closed bottle.

3.2.3 *General Procedure for "Color (APHA)" Test* — For the working standard, dilute one or more suitable aliquots of the Platinum-Cobalt Stock Solution (APHA No. 500) with water to 100 mL. In 100 mL Nessler tubes, compare the working standard with an equal volume of the liquid under assessment. View each tube

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vertically over a white background. The color of the liquid should not exceed the maximum APHA standard value allowed by the specification for that liquid. On the dilution to 100 mL, aliquots of the stock standard of 1, 2, 3 . . . mL correspond to APHA 5, 10, 15, etc.

Table 2 Abbreviations and Signs

A	absorbance
Å	angstrom (s) [NOTE 1]
a.c.	alternating current
amp.	ampere(s) [NOTE 2]
addn.	addition [NOTE 3]
alc.	alcohol(ic) [NOTE 3]
amt.	amount [NOTE 3]
approx.	approximate(ly)
aq.	aqueous [NOTE 3]
atm.	atmosphere(s)
av.	average [NOTE 3]
b.p.	boiling point [NOTE 3]
ca.	circa (i.e., about or approximately)
cal.	calorie(s)
cc	cubic centimeter(s) (mL)
c.d.	current density [NOTE 3]
cm	centimeter(s)
cm^2	square centimeter(s)
compd.	compound [NOTE 3]
compn.	composition [NOTE 3]
concn.	concentration [NOTE 3]
d	density (or difference in statistical analysis)
d.c.	direct current
diam.	diameter [NOTE 3]
dil.	dilute [NOTE 3]
EDTA	ethylenediaminetetraacetate (i.e., [ethylenedinitrilo] tetraacetate)
equiv.	equivalent(s)
f.p.	freezing point [NOTE 3]
FW	formula weight
g	gram(s)
GC	gas chromatography (or chromatographic)
GLC	gas-liquid chromatography (or chromatographic)
hr.	hour [NOTE 3]
I.D.	inside diameter [NOTE 3]
insol.	insoluble [NOTE 3]
ir	infrared [NOTE 3]
kg	kilogram(s)
L	liter(s)
m	meter(s) or milli (10)
M	molar or molarity
ma	milliampere(s)
max.	maximum (or maxima)
meq	millequivalent(s)
mg	milligram(s)
min	minute(s)
min.	minimum
mixt.	mixture [NOTE 3]
mL	milliliter(s)
mm	millimeter(s)
mM	millimolar (or millimolarity)
mmol	millimole
mol	mole [NOTE 3]
m.p.	melting point [NOTE 3]
mv	millivolt(s)
mμ	millimicron(s) (=nm) [NOTE 4]
N	normal or normality
n	nano (10^{-9})
ng	nanogram(s)
nm	nanometer(s)
no.	number [NOTE 3]
O.D.	outside diameter [NOTE 3]
p	pico (10)
pg	picogram(s)
powd.	powdered [NOTE 3]
ppb	part(s) per billion (=ng/g or ng/mL) [NOTE 5]
ppm	part(s) per million (= μg/g or μg/mL)
ppt.	precipitate [NOTE 3]
prepn.	preparation [NOTE 3]
psi	pounds per square inch
PTFE	poly (tetrafluoroethylene) (e.g., Teflon®)
satd.	saturated [NOTE 3]
SCE	saturated calomel electrode [NOTE 3]
s	second(s)
sepn.	separation [NOTE 3]
soln.	solution [NOTE 3]
soly.	solubility [NOTE 3]
std.	standard [NOTE 3]
T	transmittance
temp.	temperature [NOTE 3]
TLC	thin-layer chromatography (or chromatographic)
titrn.	titration [NOTE 3]
UV	ultraviolet [NOTE 3]
v	volts [NOTE 3]
vol.	volume [NOTE 3]
W	watts [NOTE 3]
wt.	weight [NOTE 3]
μ	micron(s) (=μm) or micro (10) [NOTE 4]
μa	microampere(s)
μeq	microequivalent(s)
μg	microgram(s)
μL	microliter(s)
μm	micrometer(s)
μM	micromolar

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µmol	micromole(s)
Ω	ohm(s)
°C	degrees Celsius
~	circa (about or approximately)
%	percent
>	greater than
≥	greater than or equal to
<	less than
≤	less than or equal to

NOTE 1: The angstrom does not conform to the SI program, and its use is restricted to emission spectrographic data (1 Å = 0.1 nm).

NOTE 2: The International System (SI) symbol, A, for ampere(s) is avoided, owing to possible confusion with absorbance.

NOTE 3: Abbreviation is not recommended for text, but only for formulas, equations, tables, specification statements and illustrations where brevity is required.

NOTE 4: The units micron and millimicron do not conform to the SI program, and the use of the equivalent terms micrometer (mm) and nanometer (nm), respectively, is recommended.

NOTE 5: A "billion" is interpreted differently in various countries and the term "parts per billion" is therefore not recommended by the International Union of Pure and Applied Chemistry (IUPAC). The term should only be used where it is defined parenthetically in the same text passage, e.g., as ng/g or ng/mL.

3.3 *Determination of Residue after Evaporation*

3.3.1 *Introduction* — The residue after evaporation is defined as the relative amount of residue remaining after evaporation of a sample of solvent followed by heating of the residue at 105°C for 30 minutes. Drying to constant weight is not utilized since protracted heating may lead to slow volatilization of high-boiling matter.

3.3.1.1 For the determination of the residue after evaporation at the level of 5 parts per million (µg/g) or below, determinate errors must be kept extremely small and random errors should be minimized. In the procedure given below, the positive error due to exposure to airborne particulate matter is reduced by the use of the closed system afforded by the Thiers assembly. Possible negative errors associated with losses caused by transfer of a concentrated sample or the residue are avoided by effecting the evaporation and weighing in a single dish. The determinate and random errors associated with the weighing operations are reduced by using an aluminum dish for most solvents, weighing on a semi-microbalance, eliminating any static electricity, and conducting all heating and cooling steps in a reproducible manner.

3.3.2 *Equipment* — The Thiers assembly is fashioned from a crystallizing dish. (See Figure 2.) The aluminum dishes of 250 mL capacity are prepared by cutting out a 30 cm circle of aluminum foil, rinsing it with the solvent, and, using gloves, pressing it around the base of an 800 mL beaker. The balance is a semi-micro type, and the static electricity eliminator is a low-level, radioactive type available from supply houses.

3.3.3 *Procedure*

3.3.3.1 Rinse the 250 mL aluminum dish (or platinum dish in the case of halogenated solvents) with a portion of the sample. Heat the dish at 105°C in an oven for 30 min and cool for 30 min (1 hr for platinum) in a desiccator charged with calcium chloride and placed near the balance. Discharge static electricity using the eliminator and weigh the dish on a semi-microbalance to the nearest 0.01 mg.

3.3.3.2 Start the flow of filtered nitrogen gas through the side arm of the Thiers assembly and place the dish inside. Raise the cover briefly to add the specified amount of solvent from a clean, graduated cylinder. Adjust the nitrogen stream so that the surface of the solvent is barely rippled. Also, adjust the heat lamp and hot plate so that the evaporation rate does not exceed 8 mL/min and that no sample condenses on the top inner surface of the assembly.

3.3.3.3 When all of the sample has evaporated, heat the dish for 30 min in an oven at 105°C. Cool as given above, discharge the static electricity, and reweigh. Calculate the residue as parts per million (ppm).

$$\frac{[\text{wt of dish plus residue (g)} - \text{wt of dish (g)}] \times 10^6}{\text{sample wt (g)}}$$

NOTE 1: For best results, room temperature should be kept constant to ± 5°C.

3.4 *General Procedure for Trace Arsenic (and Antimony) Determination*

3.4.1 *Introduction* — This photometric procedure is based upon the color reaction of silver diethyldithiocarbamate with arsine (and stibine). It provides for measurement of the absorbance of the sample and standards with subsequent calculation of the arsenic and antimony content of the sample, expressed as arsenic. The procedure given below differs from conventional ones by the omission of potassium iodide and stannous chloride. By this omission, antimony is evolved as stibine and reacts with the silver diethyldithiocarbamate solution. The color development is consequently due to both arsenic and antimony. The test sensitivity for arsenic is greater than for antimony.

3.4.2 *Apparatus* — The apparatus (see Figure 2) consists of a 125 mL arsine generator (a) fitted with a scrubber unit (c) and an absorber tube (e), with a 24/40 standard-taper joint (b) and a ground glass ball-and-socket joint (d), secured with a No. 12 clamp, connecting the units. In order to insure a more uniform rate of flow of gas into the absorbing solution, various modifications may be employed, such as the use of a

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