SEMI C1-0310
GUIDE FOR THE ANALYSIS OF LIQUID CHEMICALS

This Standard was technically approved by the Liquid Chemicals Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 16, 2009. Available at www.semi.org in February 2010; originally published in 1978; previously published July 2005.

1 Purpose

1.1 Liquid Chemicals are used extensively during the production of semiconductor devices; their purity has been shown to affect the device yield during the manufacturing processes.

1.2 Since there is a wide range of semiconductor devices being produced, different levels of purity have been established as standards and guidelines for each liquid chemical.

1.3 This Guide describes the various analytical procedures for standards verification procedures required to meet a specific liquid chemical specification.

2 Scope

2.1 All liquid chemical standards consist of guidelines, specifications and validated analytical procedures. Suitable analytical procedures are defined in each standard for each grade of liquid chemical such that their sensitivity meets the requirements of each of the respective grades of liquid chemicals.

2.2 This Guide provides an overview of each of the methods used for liquid chemical certification. Each grade of liquid chemicals (1–5) that have been validated analytically using the appropriate procedures described in this Guide can be described as 'meeting SEMI Specifications.'

2.3 *Analytical Procedures* — The procedures used to establish a liquid chemical specification can be found in § 7. The Analytical Procedures are listed in Table 1.

2.4 *Safety* — Because of the continuing evolution of safety precautions, it is impossible for this publication to provide definite statements related to the safe handling of individual liquid chemicals. The user is referred to product labels, product data sheets, government regulations, and other relevant literature.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

Table 1 Analytical Procedures

7.1	Acidity and Alkalinity
7.2	Assay by Wide Bore Column Gas Chromatography
7.3	Color
7.4	Residue after Evaporation
7.5	Residue after Ignition
7.6	Trace Arsenic (and Antimony) Determination
7.7	Use of a pH Meter
7.8	Ion Chromatography
7.9	Atomic Absorption Spectrometry (AAS)
7.10	Flame Emission Spectrometry
7.11	Graphite Furnace Atomic Absorption Spectrometry (GFAAS)
7.12	Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
7.13	Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
7.14	Calibration and Measurement Method for Particles in Liquids

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3 Referenced Standards and Documents

3.1 *ASTM Standards*[1]

ASTM D5127 — Standard Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

ASTM E203 — Standard Test Method for Water Using Volumetric Karl Fischer Titration

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Requirements

4.1 *Abbreviations and Signs* — In this Guide abbreviations and signs used shall be restricted to those used in publications of the American Chemical Society (ACS).[2]

4.2 *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.

4.3 *Acidity and Alkalinity* — A test for acidity or alkalinity, or both, of liquid chemicals may be specified. (See Acidity and Alkalinity in ¶ 7.1.)

4.4 *Analytical Blank* — 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.

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

4.6 *Atomic and Formula Weights* — Atomic weights (i.e., atomic masses) used shall be those of the latest publication of The International Union of Pure and Applied Chemistry (IUPAC).[3] 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, ¶ 4.17).

4.7 *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, ¶ 4.17). 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 0.149%, 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 ng/mL maximum, a test result as large as 10.5 ng/mL will meet the specification. This use of a decimal point is made to clarify the difference between converting 0.01 mg/mL to 10 ng/mL and converting 0.010 mg/mL to 10 ng/mL.[4]

4.8 *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).

4.9 *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.

4.10 *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

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

[2] American Chemical Society, 1155 Sixteenth Street, NW, Washington DC, 20036, USA; Telephone: +1.202.872.4600, Fax: +1.202.872.4615, http://www.chemistry.org

[3] The International Union of Pure and Applied Chemistry, PO Box 13757, Research Triangle Park, NC 27709-3757, USA; http://www.iupac.org

[4] Flachka, H.A., Barnard Jr., A.J., Sturrock, P.E., "Quantitative Analytical Chemistry." Willard Grant Press, 2nd ed. (1980): p 4.

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the second weighing follows a second drying (for one hour (unless otherwise stated) or ignition for 15 minutes (unless otherwise stated), respectively.

4.11 *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.

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

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

4.14 *Reagent Chemicals* — Unless otherwise stated, reagents to be used in tests shall conform to the minimum standards of quality set forth in the current edition of Reagent Chemicals, published by the American Chemical Society.[5]

4.15 *Residue after Evaporation* — A 'residue after evaporation' test may be used in the assessment of liquid chemicals (see Determination of Residue after Evaporation, ¶ 7.4).

4.16 *Residue after Ignition* — A 'residue after ignition' test serves to assess the amount of nonvolatile inorganic matter present in a sample (see Determination of Residue after Ignition, ¶ 7.5).

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

4.17.1 When the figure next beyond the last place to be retained is less than 5, leave unchanged the figure in the last place retained.

4.17.2 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.

4.17.3 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.

4.17.4 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.

4.17.5 Obtain the rounded value in one step by direct rounding and not in two or more steps of successive roundings.

4.18 *Samples and Sampling* — For liquid chemicals provided in small containers, one (or more) should be freshly opened for testing, thereby reducing possibilities for contamination or change in composition (e.g., by moisture pickup). Where the liquid chemical is provided in larger bulk quantities, one or more drums or other containers shall be sampled to avoid cross contamination, 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.

4.19 *Specifications and Specification Limits* — The specifications provided by this Guide are intended to serve for liquid 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 liquid chemicals of the required quality. The function of the specifications is to establish minimum standards of quality.

4.19.1 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.

[5] Available from American Chemical Society, 1155 Sixteenth Street, NW, Washington DC, 20036, USA; Telephone: +1.202.872.4600, Fax: +1.202.872.4615, http://www.chemistry.org

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

4.19.3 A liquid chemical conforming to the specification will commonly contain either more of the major component than the minimum permissible limit or be within the specified range for the liquid chemicals and contain less of each impurity (or impurities) than the maximum permissible limit. In neither case shall the liquid chemicals be considered as of higher quality than that defined by the specification.

4.19.4 It is manifestly impossible in the specifications and procedures for a liquid 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 liquid 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 liquid chemical.

4.20 *Specification Parameters* — The statement of specification parameters for a particular liquid chemical shall be, as far as practical.

4.20.1 Assay or other test assessing the component(s) of interest.

4.20.2 Appearance or color.

4.20.3 Acidity or alkalinity, or both, or pH range of solution.

4.20.4 Residue after evaporation or ignition.

4.20.5 Water content.

4.20.6 Diverse general tests, arranged alphabetically.

4.20.7 Tests for stated anions and ammonium shall be arranged alphabetically (ammonium, chloride, nitrate, phosphate, sulfate).

4.20.8 Tests for specific trace metals, arranged alphabetically by name. The 21 metals for grade specifications higher than Grade 2 are aluminum, antimony, arsenic, barium, boron, cadmium, calcium, chromium, copper, iron, lead, lithium, magnesium, manganese, nickel, potassium, sodium, tin, titanium, vanadium, and zinc.

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

4.20.10 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.

4.21 *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, 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.

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

4.23 *Water* — References to water in the testing procedures are understood to mean water that is of the appropriate grade for the intended purpose. Depending on the grade of liquid chemical being tested, water meeting the requirements for Types E-1, E-1.1, and E-1.2 water in ASTM D5127 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.

4.24 *Water Content 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. Details of this method are provided in the ASTM E203 and in Reagent Chemicals, published by the American Chemical Society

4.25 *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.

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4.26 *Weights and Measures* — The weights and measures used shall be those of the International System (SI).[6]

4.26.1 Concentration units that are dimensionless and do not indicate the basis for calculation shall be a ratio of masses. For example, 'ppm' may be used to express µg/g or mg/kg.

4.26.2 Concentration units that are dimensionless and are a ratio of some quantities other than mass shall indicate of what measurements are the bases for calculation. For example, 'ppmv' may be used to express µL/L, a ratio of volumes.

5 Reagent and Standard Solutions

5.1 The purity of reagents used in each analytical procedure should be concomitant with the grade of liquid chemical being analyzed.

5.2 *Ammonium Molybdate Reagent Solution* — Dissolve 50 g of ammonium molybdate, $(NH_4)_6Mo_7O_{24}\bullet 4H_2O$, in 1 N sulfuric acid and dilute with 1 N sulfuric acid to 1000 mL.

5.3 *Ammonium Thiocyanate Reagent Solution* — Dissolve 150 g of ammonium thiocyanate, NH_4SCN, in water and dilute with water to 500 mL.

5.4 *Antimony Standard Solution* — Dissolve and dilute 0.275 g of potassium antimony $KSbO(C_4H_4O_6)\bullet 1/2H_2O$ with water to 1000 mL (1.0 mL = 0.1 mg of antimony [Sb]).

5.5 *Arsenic Stock and Working Solution* — See ¶ 7.6.3.1.

5.6 *Barium Chloride Solution* — Dissolve 60 g of barium chloride dihydrate, $BaCl_2\bullet 2H_2O$, in water and dilute with water to 500 mL.

5.7 *Boron Standard Solution* — Dissolve 0.572 g of boric acid, H_3BO_3, in water and dilute to 1000 mL with water. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.001 mg of boron [B]).

5.8 *Bromine Water Reagent Solution* — Add sufficient liquid bromine to water in a bottle so that undissolved bromine remains as a separate phase when the mixture is shaken.

5.9 *Brucine Sulfate Solution* — Dissolve 0.6 g of brucine sulfate, $(C_{23}H_{26}N_2O_4)_2\bullet H_2SO_4\bullet 7H_2O$, in dilute sulfuric acid (2 + 1), previously cooled to room temperature, and dilute to 1000 mL with the dilute acid. The sulfuric acid should be nitrate-free acid prepared as follows: Dilute the concentrated sulfuric acid (about 96% H_2SO_4) to about 80% H_2SO_4 by adding it to water, heat to dense fumes of sulfur trioxide, and cool. Repeat the dilution and fuming three or four times.

5.10 *Carminic Acid Reagent Solution* — Dissolve 0.05 g of carminic acid in 100 mL of sulfuric acid and shake until dissolution is complete.

5.11 *Chloride Standard Solution* — Dissolve 0.165 g of sodium chloride, NaCl, in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of chloride [Cl] ion).

5.12 *Dimethylglyoxime Reagent Solution* — Dissolve 1.0 g of dimethylglyoxime in 100 mL of 95% EtOH (ethanol) reagent alcohol.

5.13 *Hydrogen Sulfide Water* — Immediately before use, saturate water with hydrogen sulfide gas.

5.14 *Hydroxylamine Hydrochloride Reagent Solution* — Dissolve 10 g of hydroxylamine hydrochloride, $NH_2OH\bullet HCl$, in water and dilute with water to 100 mL.

5.15 *Iron Standard Solution* — Dissolve 0.702 g of ferric ammonium sulfate hexahydrate, $Fe(NH_4)_2(SO_4)_2\bullet 6H_2O$, in 10 mL of 10% sulfuric acid and dilute with water to 100 mL. Then to 10 mL of this solution, add 10 mL of 10% sulfuric acid and dilute with water to 1000 mL (1.0 mL = 0.010 mg of iron [Fe] ion).

5.16 *Lead Standard Solution* — Dissolve 0.160 g of lead nitrate, $Pb(NO_3)_2$, in 100 mL of dilute nitric acid (1 + 99) and dilute to 1000 mL with water. Dilute 10 mL of this solution with water to 100 mL immediately before use (1.0 mL = 0.010 mg of lead [Pb] ion).

5.17 *Methyl Orange Indicator Solution* — Dissolve 0.1 g of methyl orange in 100 mL of water.

[6] http://www.bipm.fr/enus/3_SI/, Bureau International des Poids et Mesures, Pavillon de Breteuil, 92312 Sèvres Cedex, France; Telephone: +33.1.45.07.70.70, Fax: +33.1.45.34.20.21, http://www.bipm.fr/

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5.18 *Methyl Red Indicator Solution* — Dissolve 0.1 g of the hydrochloride salt form of methyl red in 100 mL of 95% EtOH (ethanol) reagent alcohol.

5.19 *p-(Methylamino)phenol Sulfate Reagent Solution* — Dissolve 0.2 g of *p*-(methylamino)phenol sulfate in 100 mL of water and add 20 g of sodium bisulfite. The performance of the resulting reagent solution in the determination of trace phosphate can be verified by the following test:

5.20 Place 25 mL of 0.5 N sulfuric acid and 1 mL of ammonium molybdate reagent solution in each of four test tubes. Add 1 mL of the reagent solution to each of the tubes. To the tubes add, respectively, zero, 0.005, 0.010, and 0.020 mg of phosphate ion (PO_4). After two hours at room temperature, an increasing intensity of blue color should be visible in the series.

5.21 *Nickel Standard Solution* — Dissolve 0.448 g of nickel sulfate hexahydrate, $NiSO_4 \cdot 6H_2O$, in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of nickel [Ni] ion).

5.22 *Nitrate Standard Solution* — Dissolve 0.163 g of potassium nitrate, KNO_3, in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of nitrate [NO_3] ion).

5.23 *Nitrogen Standard Solution* — Dissolve 0.382 g of ammonium chloride, NH_4Cl, in water and dilute with water to 100 mL. Dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of nitrogen [N]).

5.24 *Phenolphthalein Indicator Solution* — Dissolve 1 g of phenolphthalein in 100 mL of 95% EtOH (ethanol) reagent alcohol.

5.25 *Phosphate Standard Solution* — Dissolve 0.143 g of monobasic potassium phosphate, KH_2PO_4, in water and dilute to 100 mL with water. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of phosphate [PO_4] ion).

5.26 *Platinum-Cobalt Stock Solution (APHA No. 500)* — See ¶ 7.3.2.

5.27 *Potassium Iodide Reagent Solution* — Dissolve 10 g of potassium iodide, KI, in 100 mL of water. Prepare immediately before use.

5.28 *Potassium Standard Solution* — Dissolve 0.191 g of potassium chloride, KCl, in water and dilute with water to 1000 mL. Dilute 100 mL of this solution with water to 1000 mL (1 mL = 0.010 mg of potassium [K] ion).

5.29 *Silver Nitrate Reagent Solution* — Dissolve 1.7 g of silver nitrate, $AgNO_3$, in water and dilute with water to 100 mL.

5.30 *Sodium Carbonate Reagent Solution* — Dissolve 1.0 g of anhydrous sodium carbonate, Na_2CO_3, in water and dilute with water to 100 mL.

5.31 *Sodium Arsenite Reagent Solution (5%)* — Dissolve 1.0 g of anhydrous sodium arsenite, $NaAsO_2$, in water and dilute with water to 100 mL.

5.32 *Sodium Hydroxide Reagent Solution* — Dissolve 50 g of sodium hydroxide, NaOH, pellets in water and dilute with carbon dioxide-free water to 500 mL.

5.33 *Sodium Standard Solution* — Dissolve 0.254 g of sodium chloride, NaCl, in water and dilute with water to 1000 mL. Then dilute 100 mL of this solution with water to 1000 mL (1 mL = 0.010 mg of sodium [Na] ion).

5.34 *Stannous Chloride Reagent Solution* — Dissolve 20 g of stannous chloride dihydrate, $SnCl_2 \cdot 2H_2O$, in hydrochloric acid and dilute with hydrochloric acid to 100 mL.

5.35 *Starch Indicator Solution* — Mix 1 g of soluble starch for iodometry with a starch indicator and enough cold water to form a thin paste. Add 200 mL of boiling water and boil for 1 minute with stirring. Cool and transfer to a storage bottle that has been well rinsed with hot water.

5.36 *Sulfate Standard Solution* — Dissolve 0.148 g of anhydrous sodium sulfate, Na_2SO_4, in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of sulfate [SO_4] ion).

5.37 *Thymolphthalein Indicator Solution* — Dissolve 0.10 g of thymolphthalein in 100 mL of 95% EtOH (ethanol) reagent alcohol.

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6 Method Validation

6.1 *Method Validation* — Analytical methods for trace metals, trace anions, moisture and assays require that performance parameters be assessed and compared to the standard and guideline requirements. The accuracy, precision of the measurement and interferences should be reviewed and reported with each method.

6.1.1 *Accuracy for Trace Metals, Trace Anions, and Moisture*

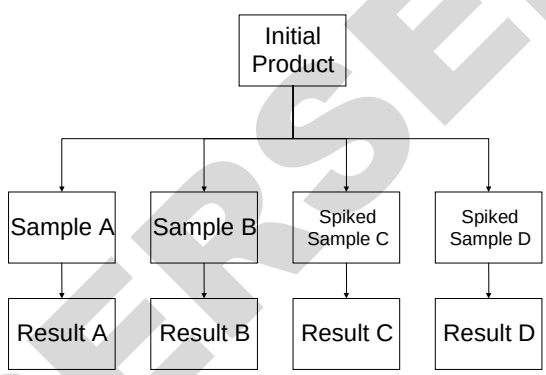
6.1.1.1 Method accuracy is a figure of merit that compares the data generated from the determined concentration of a sample to its 'known' concentration. In some cases, a traceable standard may not be available and a 'best available standard' may be substituted.

6.1.1.2 Determine the accuracy of a test method by utilizing a standard reference material as a sample and analyze it using the proposed test method. Report the test method accuracy as percent (%) bias:

$$\%\,\text{bias} = \frac{X_{\text{"found"}} - X_{\text{"true value"}}}{X_{\text{"true value"}}} \times 100 \qquad (1)$$

6.1.1.3 If no suitable traceable standard exists for the 'true value,' describe the procedures used to qualify the 'best available standard.'

6.1.1.4 Spiked recovery determinations are used to make a limited assessment of accuracy. Method validation is based on the analysis of a minimum of two samples and a minimum of two spiked samples (Figure 1).



#1 Samples A, B, C, and D are originally from the same initial product.

Figure 1
Data Collection Structure

6.1.2 *Measurement of Precision*

6.1.2.1 Typically a repeatability and reproducibility study is done, where samples are analyzed over a period of multiple days.

6.1.2.2 Measures of method precision incorporate all applicable method variability sources (such as sample preparation, calibration, and instrumental performance) into their definitions. An assessment of method precision for trace metals, trace anions, and moisture is detailed in Tables 2 and 4.

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Table 2 Data Reporting Requirements for Trace Metals, Trace Anions, and Moisture

Sample A	Result A
Sample B	Result B
Average	Average of Result A and B
Spike	The amount of spike equivalent to 50% of the specified value for trace impurity, and it should be added to the sample prior to any sample preparation (e.g., sample dilution, evaporation, etc.)
Spike Sample C	Result C
Spike Sample D	Result D
Recovery 1	Result C – Average
% Recovery 1	(Recovery 1 / Spike) × (100)
Recovery 2	Result D – Average
% Recovery 2	(Recovery 2 / Spike) × (100)
Average % Recovery	(% Recovery 1 + % Recovery 2)/2
% Recovery Range	Max. % Recovery − Min. % Recovery
Result Standard Deviation	When applicable, report the Std. Dev. of multiple measurements for each Result
Relative Standard Deviation	When applicable, report the Relative Std. Dev. of multiple measurements for each Result

6.1.2.3 Measurement Repeatability targets how well measurements can be repeated and should be reported as percent (%) relative standard deviation (Tables 2 and 4).

6.1.3 *Interference Checks*

6.1.3.1 List all interferences that were examined during the course of method validation. Also note any known potential interferences.

6.1.4 *Data Reporting Requirements*

6.1.4.1 Table 2 along with Figure 1 illustrates the minimal data structure and analysis required to support method validation for trace metals, trace anions, and moisture. Table 3 is used for assays.

Table 3 Data Reporting Requirements for Assays

Specification Width	W = upper spec. limit − lower spec. limit If no upper spec. limit is stated for an assay of an ultrapure product, use 100%.
Pooled Standard Deviation	$$s_p = \sqrt{s_p^2} = \sqrt{\frac{\sum_{i=1}^{N}(n_i - 1)s_i^2}{\sum_{i=1}^{N}(n_i - 1)}}$$ where N ≥ 1 groups of current or historical $n_i \geq 2$ assays taken on the ith sample have their standard deviations pooled and s_i^2 is the standard deviation of the data in the ith group squared.
Degrees of Freedom	$$df = \sum_{i=1}^{N}(n_i - 1)$$ where degrees of freedom ≥4

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90% Upper Confidence Limit	$$90\%\ \text{UCL} = t_{df,0.10}\frac{s_p}{\sqrt{n}}$$ where n is the total number of assay measurements taken on the sample in question and t is a t-statistic with the associated degrees of freedom and 10% of the distribution to its right.

6.1.4.2 In cases for trace metals, trace anions, and moisture where more samples are measured, statistical quantities analogous to those in Table 2 should be constructed. For methods where automatic consecutive measurements can be obtained from the instrument, Results A, B, C, and D should be the mean of five or more instrumental measurements.

6.1.5 *Summary of Criteria for Success* — The success of the criteria for each figure of merit depends on the specification, the matrix of the analyte, and levels of detection required. For trace metals, trace anions and moisture, method success criteria are found in Table 4. For assays the 90% upper confidence limit cannot exceed 30% of the specification's width.

Table 4 Method Success Criteria for Trace Metals, Trace Anions and Moisture

Figure of Merit	*Criteria for Successful Method*
Accuracy Spiked Recovery Study	75%–125% Spiked Recovery for each spiked sample.
Method Precision Spiked Re°overy Study	% Recovery Range less than or equal to 35%.
Measurement Precision	Maximum RSD = 20% of the concentration of each spiked sample where: $$\text{RSD} = \frac{\text{Std.Dev. Result}}{\text{Conc. of Spiked Sample}} \times 100$$
Method Detection Limit	As per SEMI C10, Guide for Determination of Method Detection Limits; Method Detection Limits cannot exceed the specification.
Interference Checks	If interferences were examined or potential interferences exist, list them.

6.1.6 *Applicability to Existing Specifications* — The current implementation of Method Validation only applies to new or revised specifications. Any prior method validated by SEMI to support a given specification still remains applicable to that specification.

6.1.7 *Example: Trace Metals, Trace Anions and Moisture* — Suppose that repeat measurements from four samples; A, B, C and D were available as follows:

- Sample A: 0.19, 0.21, 0.25, 0.15, 0.23
- Sample B: 0.31, 0.21, 0.17, 0.22, 0.26
- Spiked Sample C: 0.83, 0.62, 0.76, 0.79, 0.80
- Spiked Sample D: 0.77, 0.87, 0.69, 0.83, 0.84

NOTE 1: Table 5 illustrates the calculations required as per Table 2.

Table 5 Data Reporting – Example 6.1.7

Sample A	(0.19 + 0.21 + 0.25 + 0.15 + 0.23) / 5 = 0.206
Sample B	(0.31 + 0.21 + 0.17 + 0.22 + 0.26) / 5 = 0.234
Average	(0.206 + 0.234) / 2 = 0.220

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