Designation: F1593 – 08 (Reapproved 2016)

Standard Test Method for Trace Metallic Impurities in Electronic Grade Aluminum by High Mass-Resolution Glow-Discharge Mass Spectrometer[1]

This standard is issued under the fixed designation F1593; 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.

1. Scope

1.1 This test method covers measuring the concentrations of trace metallic impurities in high purity aluminum.

1.2 This test method pertains to analysis by magnetic-sector glow discharge mass spectrometer (GDMS).

1.3 The aluminum matrix must be 99.9 weight % (3N-grade) pure, or purer, with respect to metallic impurities. There must be no major alloy constituent, for example, silicon or copper, greater than 1000 weight ppm in concentration.

1.4 This test method does not include all the information needed to complete GDMS analyses. Sophisticated computer-controlled laboratory equipment skillfully used by an experienced operator is required to achieve the required sensitivity. This test method does cover the particular factors (for example, specimen preparation, setting of relative sensitivity factors, determination of sensitivity limits, etc.) known by the responsible technical committee to affect the reliability of high purity aluminum analyses.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1257 Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis

3. Terminology

3.1 Terminology in this test method is consistent with Terminology E135. Required terminology specific to this test method and not covered in Terminology E135 is indicated below.

3.2 *campaign*—a series of analyses of similar specimens performed in the same manner in one working session, using one GDMS setup. As a practical matter, cleaning of the ion source specimen cell is often the boundary event separating one analysis campaign from the next.

3.3 *reference sample*— material accepted as suitable for use as a calibration/sensitivity reference standard by all parties concerned with the analyses.

3.4 *specimen*—a suitably sized piece cut from a reference or test sample, prepared for installation in the GDMS ion source, and analyzed.

3.5 *test sample*— material (aluminum) to be analyzed for trace metallic impurities by this GDMS test method. Generally the test sample is extracted from a larger batch (lot, casting) of product and is intended to be representative of the batch.

4. Summary of the Test Method

4.1 A specimen is mounted as the cathode in a plasma discharge cell. Atoms subsequently sputtered from the specimen surface are ionized, and then focused as an ion beam through a double-focusing magnetic-sector mass separation apparatus. The mass spectrum, that is, the ion current, is collected as magnetic field, or acceleration voltage is scanned, or both.

4.2 The ion current of an isotope at mass M_i is the total measured current, less contributions from all other interfering sources. Portions of the measured current may originate from the ion detector alone (detector noise). Portions may be due to incompletely mass resolved ions of an isotope or molecule with mass close to, but not identical with, M_i. In all such instances the interfering contributions must be estimated and subtracted from the measured signal.

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[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.17 on Sputter Metallization.

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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.

4.2.1 If the source of interfering contributions to the measured ion current at M_i cannot be determined unambiguously, the measured current less the interfering contributions from identified sources constitutes an upper bound of the detection limit for the current due to the isotope.

4.3 The composition of the test specimen is calculated from the mass spectrum by applying a relative sensitivity factor (RSF(X/M)) for each contaminant element, X, compared to the matrix element, M. RSFs are determined in a separate analysis of a reference material performed under the same analytical conditions, source configuration, and operating protocol as for the test specimen.

4.4 The relative concentrations of elements X and Y are calculated from the relative isotopic ion currents $I(X_i)$ and $I(Y_j)$ in the mass spectrum, adjusted for the appropriate isotopic abundance factors ($A(X_i)$, $A(Y_j)$) and *RSFs*. $I(X_i)$ and $I(Y_j)$ refer to the measured ion current from isotopes X_i and Y_j, respectively, of atomic species X and Y.

$$(X)/(Y) = RSF(X/M)/RSF(Y/M) \times A(Y_j)/A(X_i) \times I(X_i)/I(Y_i) \quad (1)$$

where $(X)/(Y)$ is the concentration ratio of atomic species X to species Y. If species Y is taken to be the aluminum matrix (RSF(M/M) = 1.0), (X) is (with only very small error for pure metal matrices) the absolute impurity concentration of X.

5. Significance and Use

5.1 This test method is intended for application in the semiconductor industry for evaluating the purity of materials (for example, sputtering targets, evaporation sources) used in thin film metallization processes. This test method may be useful in additional applications, not envisioned by the responsible technical committee, as agreed upon by the parties concerned.

5.2 This test method is intended for use by GDMS analysts in various laboratories for unifying the protocol and parameters for determining trace impurities in pure aluminum. The objective is to improve laboratory to laboratory agreement of analysis data. This test method is also directed to the users of GDMS analyses as an aid to understanding the determination method, and the significance and reliability of reported GDMS data.

5.3 For most metallic species the detection limit for routine analysis is on the order of 0.01 weight ppm. With special precautions detection limits to sub-ppb levels are possible.

5.4 This test method may be used as a referee method for producers and users of electronic-grade aluminum materials.

6. Apparatus

6.1 *Glow Discharge Mass Spectrometer,* with mass resolution greater than 3500, and associated equipment and supplies. The GDMS must be fitted with an ion source specimen cell that is cooled by liquid nitrogen, Peltier cooled, or cooled by an equivalent method.

6.2 *Machining Apparatus,* capable of preparing specimens and reference samples in the required geometry and with smooth surfaces.

6.3 *Electropolishing Apparatus,* capable of removing the contaminants from the surfaces of specimens.

7. Reagents and Materials

7.1 *Reagent and High Purity Grade Reagents,* as required (MeOH, HNO_3, HCl).

7.2 *Demineralized Water.*

7.3 *Tantalum Reference Sample.*

7.4 *Aluminum Reference Sample.*

7.4.1 To the extent available, Aluminum reference materials shall be used to produce the GDMS relative sensitivity factors for the various elements being determined (see Table 1).

7.4.2 As necessary, non-aluminum reference materials may be used to produce the GDMS relative sensitivity factors for the various elements being determined.

7.4.3 Reference materials should be homogeneous and free of cracks or porosity.

7.4.4 At least two reference materials are required to establish the relative sensitivity factors, including one nominally 99.9999 % pure (6N-grade) aluminum metal to establish the background contribution in analyses.

7.4.5 The concentration of each analyte for relative sensitivity factor determination should be a factor of 100 greater than the detection limit determined using a nominally 99.9999 % pure (6N-grade) aluminum specimen, but less than 100 ppmw.

7.4.6 To meet expected analysis precision, it is necessary that specimens of reference and test material present the same size and configuration (shape and exposed length) in the glow discharge ion source, with a tolerance of 0.2 mm in diameter and 0.5 mm in the distance of specimen to cell ion exit slit.

8. Preparation of Reference Standards and Test Specimens

8.1 The surface of the parent material must not be included in the specimen.

TABLE 1 Suite of Impurity Elements to Be Analyzed[A]

NOTE 1—Establish RSFs for the following suite of elements.

silver	arsenic	gold	boron	beryllium	calcium	cerium	chromium	cesium	copper	iron
potassium	lithium	magnesium	manganese	sodium	nickel	phosphorus	antimony	silicon	tin	thorium
titanium	uranium	vanadium	zinc	zirconium						

[A] Additional species may be determined and reported, as agreed upon between all parties concerned with the analyses.

8.2 The machined surface of the specimen must be cleaned by electropolishing or etching immediately prior to mounting the specimen and inserting it into the glow discharge ion source.

8.2.1 In order to obtain a representative bulk composition in a reasonable analysis time, surface cleaning must remove all contaminants without altering the composition of the specimen surface.

8.2.2 To minimize the possibility of contamination, clean each specimen separately immediately prior to mounting in the glow discharge ion source.

8.2.3 Prepare and use electropolishing or etching solutions in a clean container insoluble in the contained solution.

8.2.4 *Electropolishing—* perform electropolishing in a solution of methanol and HNO_3 mixed in the ratio 7:5 by volume. Apply 5–15 volts (dc) across the cell, with the specimen as anode. Electropolish for up to 4 min, as sufficient to expose smooth, clean metal over the entire polished surface.

8.2.5 *Etching*—perform etching by immersing the specimen in aqua regia (HNO_3 and HF, mixed in the ratio 3:1 by volume). Etch for several minutes, until smooth, clean metal is exposed over the entire surface.

8.2.6 Immediately after cleaning, wash the specimen with several rinses of high purity methanol or other high purity reagent to remove water from the specimen surface, and dry the specimen in the laboratory environment.

8.3 Immediately mount and insert the specimen into the glow discharge ion source, minimizing exposure of the cleaned, rinsed specimen surface to the laboratory environment.

8.3.1 As necessary, use a non-contacting gage when mounting specimens in the analysis cell specimen holder to ensure the proper sample configuration in the glow discharge cell (see 7.4.6).

8.4 Sputter etch the specimen surface in the glow discharge plasma for a period of time before data acquisition (see 12.3) to ensure the cleanliness of the surface. Pre-analysis sputtering conditions are limited by the need to maintain sample integrity. Pre-analysis sputtering at twice the power used for the analysis should be adequate for sputter etch cleaning.

9. Preparation of the GDMS Apparatus

9.1 The ultimate background pressure in the ion source chamber should be less than 1×10^{-6} Torr before operation. The background pressure in the mass analyzer should be less than 5×10^{-7} Torr during operation.

9.2 The glow discharge ion source must be cooled to near liquid nitrogen temperature.

9.3 The GDMS instrument must be accurately mass calibrated prior to measurements.

9.4 The GDMS instrument must be adjusted to the appropriate mass peak shape and mass resolving power for the required analysis.

9.5 If the instrument uses different ion collectors to measure ion currents during the same analysis, the measurement efficiency of each detector relative to the others should be determined at least weekly.

9.5.1 If both Faraday cup collector for ion current measurement and ion counting detectors are used during the same analysis, the ion counting efficiency (ICE) must be determined prior to each campaign of specimen analyses using the following or equivalent procedures.

9.5.1.1 Using a specimen of tantalum, measure the ion current from the major isotope (^{181}Ta) using the ion current Faraday cup detector, and measure the ion current from the minor isotope (^{180}Ta) using the ion counting detector, with care to avoid ion counting losses due to ion counting system dead times. The counting loss should be 1 % or less.

9.5.1.2 The ion counting efficiency is calculated by multiplying the ratio of the ^{180}Ta ion current to the ^{181}Ta ion current by the ^{181}Ta/ ^{180}Ta isotopic ratio. The result of this calculation is the ion counting detector efficiency (ICE).

9.5.1.3 Apply the ICE as a correction to all ion current measurements from the ion counting detector obtained in analyses by dividing the ion current by the ICE factor.

10. Instrument Quality Control

10.1 A well-characterized specimen must be run on a regular basis to demonstrate the capability of the GDMS system as a whole for the required analyses.

10.2 A recommended procedure is the measurement of the relative ion currents of selected analytes and the matrix element in aluminum or tantalum reference samples.

10.3 Plot validation analysis data from at least five elements with historic values in statistical process control (SPC) chart format to demonstrate that the analysis process is in statistical control. The equipment is suitable for use if the analysis data group is within the 3-sigma control limits and shows no non-random trends.

10.4 Upper and lower control limits for SPC must be within at least 20 % of the mean of previously determined values of the relative ion currents.

11. Standardization

11.1 The GDMS instrument should be standardized using National Institute of Standards Technology (NIST) traceable reference materials, preferably aluminum, to the extent such reference samples are available.

11.2 Relative sensitivity factor (RSF) values should, in the best case, be determined from the ion beam ratio measurements of four randomly selected specimens from each standard required, with four independent measurements of each pin.

11.3 RSF values must be determined for the suite of impurity elements for which specimens are to be analyzed (see Table 1) using the selected isotopes (see Table 2) for measurement and RSF calculation.

12. Procedure

12.1 Establish a suitable data acquisition protocol (DAP) appropriate for the GDMS instrument used for the analysis.

TABLE 2 Isotope Selection[A]

NOTE 1—Use the following isotopes for establishing RSF values and for performing analyses of test specimens.

^{109}Ag
^{75}As
^{197}Au
^{11}B
^{9}Be
^{44}Ca
^{140}Ce
^{52}Cr
^{133}Cs
^{63}Cu/ ^{65}Cu
^{56}Fe
^{39}K
^{7}Li
^{24}Mg
^{55}Mn
^{23}Na
^{60}Ni
^{31}P
^{121}Sb
^{28}Si
^{119}Sn
^{232}Th
^{48}Ti
^{238}U
^{51}V
^{64}Zn/ ^{66}Zn
^{90}Zr

[A] This selection of isotopes minimizes significant interferences (see Annex A1). Additional species may be determined and reported, as agreed upon between all parties concerned with the analyses.

12.1.1 The DAP must include, but is not limited to, the measurement of elements tabulated in Table 1 and the isotopes tabulated in Table 2.

12.1.2 Instrumental parameters selected for isotope measurements must be appropriate for the analysis requirements:

12.1.2.1 Ion current integration times to achieve desired precision and detection limits; and,

12.1.2.2 Mass ranges about the analyte mass peak over which measurements are acquired to clarify mass interferences.

12.2 Insert the prepared specimen into the GDMS ion source, allow the specimen to cool to source temperature, and initiate the glow discharge at pre-analysis sputtering conditions.

12.3 Proceed with specimen analysis using either Procedure A (12.3.1) or Procedure B (12.3.2).

12.3.1 *Analysis Procedure A:*

12.3.1.1 Establish a temporary pre-analysis sputtering data acquisition protocol (TDAP) including the measurement of critical surface contaminants from the specimen preparation steps (refer to Guide E1257).

12.3.1.2 After at least 5 min of pre-analysis sputtering, perform at least three consecutive measurements of the specimen using the TDAP, with appropriate intervals between the measurements to ensure cleanliness of the specimen surface.

(*1*) The concentration values from the last three consecutive measurements must exhibit equilibrated, random behavior, and the relative standard deviation (RSD) of the three measurements of the critical contaminants must meet the criteria tabulated in Table 3 before terminating pre-analysis sputtering and proceeding to the next step.

12.3.1.3 After pre-analysis sputtering, adjust the glow discharge ion source sputtering conditions to the conditions required for analysis, ensuring that the gas pressure required to do so is within normal range.

12.3.1.4 Measure the specimen using the full DAP.

TABLE 3 Required Relative Standard Deviation (RSD) for RSF Determination, Pre-sputtering Period, and Plasma Stability Tests

Analyte Content Range, ppm	Required RSD, %
Major (1000 > X > 100)	5
Minor (100 > X > 1)	10
Trace (1 > X > 100)	20

12.3.1.5 The single full analysis using the DAP is reported as the result of analysis by Procedure A.

12.3.2 *Analysis by Procedure B:*

12.3.2.1 After at least 5 min of pre-analysis sputtering, adjust the glow-discharge ion-source sputtering conditions to the conditions required for analysis, ensuring that the gas pressure required to do so is within normal range.

12.3.2.2 Analyze the specimen using the DAP and accept as final the concentration values determined only as detection limits.

12.3.2.3 Generate a measurement data acquisition protocol (MDAP) including only the elements determined to be present in the sample (from the results of 12.3.2.2).

12.3.2.4 Measure the sample at least two additional times using the MDAP until the criteria of 12.3.2.5 are met.

12.3.2.5 If the concentration differences between the last two measurements are less than 5 %, 10 % or 20 %, depending on concentration (see Table 3), the measurements are confirmed and the last two measurements are averaged.

12.3.2.6 The confirmed values from 12.3.2.4, 12.3.2.5 and the detection limits determined from 12.3.2.2 are reported together as the result of the analysis by Procedure B.

13. Detection Limit Determination

13.1 The following procedures to determine detection limits enable rapid operator assessment of detection limits in the case (*1*) that the analyte signal must be determined in the presence of a substantial signal from an interfering ion and in the case (*2*) that the analyte signal must be determined in the presence of a statistically varying background signal. In the former case, the mass difference between the analyte and an interfering ion is typically less than 1.5 full mass peak width at half-maximum peak intensity (FWHM) of the mass peak and the shape and magnitude of the interfering mass peak determine the analyte detection limit, not the statistical variability of the interfering signal. A Type I (13.2) or Type II (13.3) detection limit should be calculated and reported. If the analyte peak is obscured by statistical variation, a Type III detection limit (13.4 should be calculated and reported.

13.1.1 The procedures outlined below are designed to enable rapid detection limit evaluation as free of operator bias as possible in a circumstance where substantial operator intervention is required for reliable data evaluation.

13.2 *Type I Detection Limit:*

13.2.1 If the analyte signal at the appropriate mass cannot be mass resolved from possible interfering ion signals, and the identification of the analyte signal cannot be confirmed by correlation with a similar signal from a related isotope, the analyte concentration calculated assuming that the entire signal or mass peak is due to the element in question constitutes an upper limit on the actual amount present.

13.2.2 If the ion signal at the analyte mass can be isotopically confirmed as due mainly (greater than 80 %) to an unresolvable interfering ion, then the detection limit is calculated to be 20 % of the interfering ion signal.

13.2.3 If the origin of the analyte ions is ambiguous, the entire signal must be accepted as an upper limit on the concentration of the isotope in the sample unless strong arguments can be made that interfering contributions are less than 20 %. For example, Tantalum ions may originate from the sample but most likely originate from ion source components. Likewise, oxygen ions may derive from the sample or may be a plasma gas contaminant arising from source or instrument outgassing.

13.3 *Type II Detection Limit (see Fig. 1):*

13.3.1 If an analyte and an interfering ion are marginally mass resolvable, but there is no local minimum in the signal to confirm the presence of at least two separate contributions to the mass peak (analyte plus interfering ion), the upper limit on the concentration of the analyte is estimated by integrating the full ion signal over the half-mass peak width at half-maximum peak intensity (HWHM) mass range beginning at the mass position of the analyte and extending away from the mass of the interfering ion and then doubling the result.

13.4 *Type III Detection Limit (see Fig. 2):*

13.4.1 If the mass difference between an analyte and any possible interference ion is greater than 1.5 FWHM of the mass peak, and the analyte signal is superimposed on a signal dominated by detector noise or unstructured signals from ions of nearby masses, the detection limit is calculated using the following procedures.

13.4.1.1 If N is the sum of the ion counts within the FWHM range about M, then the detection limit is as follows:

$$d.l. = 3 + 5\sqrt{N} \tag{2}$$

with appropriate quantitation for the element in question.[3]

13.4.2 An equivalent calculation of detection limit in the case where the analyte signal is superimposed on a smoothly varying, non-zero background signal is obtained as follows.

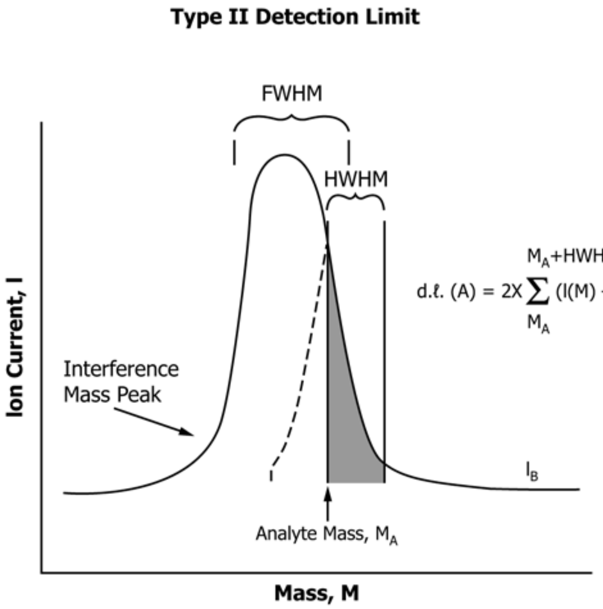

FIG. 1 Type II Detection Limit

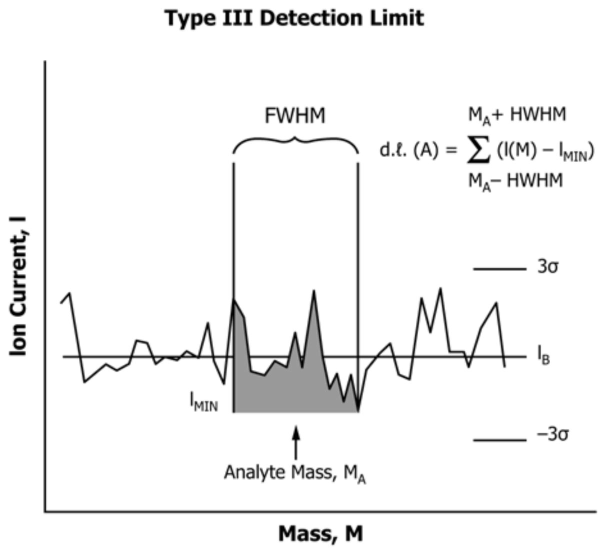

FIG. 2 Type III Detection Limit

13.4.2.1 In a mass interval centered at M and equal in width to FWHM, the lower limit to the measured signal in the interval is noted, excluding up to 5 % of the measurements if it is judged necessary to do so to exclude very extreme measurements. This limiting value is subtracted from each of the other signal measurements in the FWHM mass interval. These difference values are then summed over the mass interval. The sum, properly quantitated for the element in question, constitutes the detection limit for the isotope at mass M.

13.4.3 The Type III procedures above provide a continuity of technique with the assessment procedures for Type I and II detection limits whereby the ion signal over a FWHM mass range is integrated to provide the detection limit estimate.

14. GDMS Analysis for Thorium, Uranium, and Similar Elements

14.1 Use extra caution in determining thorium, uranium and other Group 3 and Group 4 elements because these analytes are especially sensitive to instrument changes and analytical conditions.

14.2 Thorium, Uranium and other elements with significantly lower specification limits should be determined separately according to instrument performance, for example, use increased ion counting times to lower the detection limits.

15. Report

15.1 Provide concentration data for the suite of elements listed in Table 1. Additional elements may be listed as agreed upon between all parties concerned with the analysis.

15.2 Report elemental concentrations in a tabulation arranged in order of increasing atomic number or atomic weight, whichever is more convenient.

15.3 Element concentration shall be reported, typically, in units of parts per million by weight.

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[3] Currie, L. A., "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, Vol 40, 1968, pp 586–593.

15.4 Numerical results shall be presented using all certain digits plus the first uncertain digit, consistent with the precision of the determination.

15.5 Non-detected elements shall be reported at the detection limit.

15.6 Unmeasured elements shall be designated with an asterisk (*) or other notation.

16. Precision and Bias[4]

16.1 *Precision*—Precision calculations have been done in accordance with the practices outlined in Practice E691. The reader is referred to both Practices E691 and E177 for both detailed definitions and statistical derivations of the critical measures developed in this study. The precision calculations were based upon the analysis of three different aluminum samples by eight independent laboratories. The results are summarized in Table 4.

16.2 *Bias*—The bias of this test method could not be determined because adequate certified standard reference materials were unavailable at the time of the testing. The user is cautioned to verify, by the use of certified reference materials if available, that the accuracy of this test method is adequate for the contemplated use.

17. Keywords

17.1 aluminum; electronics; glow discharge mass spectrometer (GDMS); purity analysis; sputtering target; trace metallic impurities

TABLE 4 Statistical Summary

Precision Statistics

Silicon

Material	Average (ppm)	Sx^A	Sr^B	SR^C	r^D	R^E
SAX 300	10.413	1.333	0.313	1.364	0.876	3.819
SAX 300-1	1.459	0.142	0.064	0.154	0.178	0.432
SAX 300-2	1.469	0.261	0.501	0.537	1.404	1.503

Iron

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	44.31	6.496	2.103	6.788	5.889	19.006
SAX 300-1	2.797	0.412	0.136	0.431	0.382	1.208
SAX 300-2	0.891	0.131	0.057	0.142	0.16	0.397

Copper

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	17.126	3.448	1.5687	3.747	4.392	10.492
SAX 300-1	1.600	0.537	0.126	0.459	0.352	1.538
SAX 300-2	0.946	0.448	0.190	0.482	0.533	1.349

Maganese

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	19.85	2.744	1.379	3.032	3.860	8.489
SAX 300-1	1.169	0.141	0.074	0.157	0.207	0.439
SAX 300-2	0.319	0.046	0.024	0.051	0.066	0.143

Magnesium

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	7.716	0.717	0.250	0.754	0.699	2.111
SAX 300-1	0.971	0.072	0.054	0.088	0.151	0.247
SAX 300-2	0.641	0.045	0.039	0.059	0.111	0.164

Titanium

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	6.870	1.333	0.896	1.575	2.509	4.409
SAX 300-1	0.720	0.113	0.042	0.119	0.116	0.333
SAX 300-2	0.227	0.037	0.013	0.039	0.038	0.110

Nickel

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	16.698	2.695	0.812	2.800	2.272	7.841
SAX 300-1	0.958	0.135	0.058	0.145	0.161	0.406
SAX 300-2	0.231	0.035	0.017	0.039	0.049	0.109

Zinc

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	25.135	3.592	1.035	3.719	2.898	10.415
SAX 300-1	1.566	0.196	0.105	0.219	0.294	0.613
SAX 300-2	0.407	0.068	0.024	0.071	0.067	0.199

Chromium

Material	Average (ppm)	Sx^A	Sr^B	SR^C	r^D	R^E
SAX 300	16.188	2.871	0.783	2.963	2.194	8.296
SAX 300-1	1.045	0.161	0.037	0.165	0.103	0.461
SAX 300-2	0.342	0.059	0.027	0.064	0.077	0.180

Zirconium

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	15.250	2.999	2.205	3.639	6.174	10.192
SAX 300-1	1.014	0.196	0.076	0.209	0.214	0.585
SAX 300-2	0.26	0.045	0.018	0.048	0.050	0.135

Boron

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	16.271	5.938	1.588	6.121	4.446	17.137
SAX 300-1	4.125	1.485	0.726	1.633	2.034	4.573
SAX 300-2	2.078	0.789	0.132	0.798	0.368	2.237

Lead

Material	Average (ppm)	Sx	Sr	SR	r	R
SAX 300	20.868	6.020	2.575	6.484	7.210	18.155
SAX 300-1	1.173	0.368	0.095	0.378	0.267	1.059
SAX 300-2	0.295	0.079	0.043	0.088	0.119	0.247

Thorium

Material	Average (ppb)	Sx	Sr	SR	r	R
SAX 300	1.033	0.353	0.072	0.360	0.203	1.007
SAX 300-1	1.019	1.310	1.423	1.867	3.984	5.228
SAX 300-2	0.622	0.362	0.190	0.403	0.531	1.129

Uranium

Material	Average (ppb)	Sx	Sr	SR	r	R
SAX 300	2.514	1.091	0.235	1.113	0.657	3.115
SAX 300-1	0.646	0.358	0.099	0.369	0.276	1.034
SAX 300-2	0.537	0.392	0.094	0.402	0.263	1.124

[A] Sx = the standard deviation of the averages across all participating laboratories.

[B] Sr = the repeatability standard deviation. Describes the pooled standard deviations across all laboratories.

[C] SR = the reproducibility standard deviation. Deals with the variability between laboratories.

[D] r = the 95 % repeatability limits and is calculated by 2.8 × Sr.

[E] R = the 95 % reproducibility limits and is calculated by 2.8 × SR.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F01-1013.

ANNEX

(Mandatory Information)

A1. MASS SPECTRUM INTERFERENCES

A1.1 Ions of the following atoms and molecular combinations of aluminum, argon plasma gas isotopes, plasma impurities (carbon, hydrogen, oxygen, chlorine) and tantalum source components can significantly interfere with the determination of the ion current of the selected isotopes at low element concentrations.

$^{27}\mathrm{A1}\ ^{1}\mathrm{H}^{+}$ interferes with $^{28}\mathrm{Si}^{+}$
$^{38}\mathrm{Ar}^{++}$ interferes with $^{19}\mathrm{F}^{+}$
$^{12}\mathrm{C}\ ^{16}\mathrm{O}^{+}$ interferes with $^{28}\mathrm{Si}^{+}$
$(^{16}\mathrm{O}_2)^{+}$ interferes with $^{32}\mathrm{S}^{+}$
$^{38}\mathrm{Ar}\ ^{1}\mathrm{H}^{+}$ interferes with $^{39}\mathrm{K}^{+}$
$^{40}\mathrm{Ar}^{+}$ scattered ions interfere with $^{39}\mathrm{K}^{+}$
$^{12}\mathrm{C}\ ^{16}\mathrm{O}_2^{+}$ interferes with $^{44}\mathrm{Ca}^{+}$
$^{40}\mathrm{Ar}\ ^{12}\mathrm{C}^{+}$ interferes with $^{52}\mathrm{Cr}^{+}$
$^{40}\mathrm{Ar}\ ^{16}\mathrm{O}^{+}$ interferes with $^{56}\mathrm{Fe}^{+}$
$^{36}\mathrm{Ar}\ ^{27}\mathrm{Al}^{+}$ interferes with $^{63}\mathrm{Cu}^{+}$
$^{40}\mathrm{Ar}\ ^{35}\mathrm{Cl}^{+}$ interferes with $^{75}\mathrm{As}^{+}$
$^{40}\mathrm{Ar}\ ^{36}\mathrm{Ar}\ ^{1}\mathrm{H}^{+}$ interferes with $^{77}\mathrm{Se}^{+}$
$^{40}\mathrm{Ar}\ ^{38}\mathrm{Ar}\ ^{1}\mathrm{H}^{+}$ interferes with $^{79}\mathrm{Br}^{+}$
$(^{40}\mathrm{Ar}_2)^{+}$ scattered ions interfere with $^{79}\mathrm{Br}^{+}$
$^{40}\mathrm{Ar}\ ^{36}\mathrm{Ar}\ ^{27}\mathrm{Al}^{+}$ interferes with $^{103}\mathrm{Rh}^{+}$
$^{40}\mathrm{Ar}\ ^{36}\mathrm{Ar}\ ^{38}\mathrm{Ar}^{+}$ interferes with $^{114}\mathrm{Cd}^{+}$
$^{181}\mathrm{Ta}\ ^{16}\mathrm{O}^{+}$ interferes with $^{197}\mathrm{Au}^{+}$

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