



# Standard Test Method for Trace Metallic Impurities in Electronic Grade Titanium by High Mass-Resolution Glow Discharge Mass Spectrometer<sup>1</sup>

This standard is issued under the fixed designation F1710; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of concentrations of trace metallic impurities in high purity titanium.

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

1.3 The titanium 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, aluminum or iron, 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 effect the reliability of high purity titanium 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:<sup>2</sup>

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of

Methods for Chemical Analysis of Metals (Withdrawn 1998)<sup>3</sup>

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)<sup>3</sup>

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, not covered in Terminology E135, is indicated in 3.2.

### 3.2 Definitions:

3.2.1 *campaign*—a series of analyses of similar specimens performed in the same manner in one working session, using one GDMS setup.

3.2.1.1 *Discussion*—As a practical matter, cleaning of the ion source specimen cell is often the boundary event separating one analysis campaign from the next.

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

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

3.2.4 *test sample*—material titanium to be analyzed for trace metallic impurities by this GDMS method.

3.2.4.1 *Discussion*—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

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>1</sup> 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.

Current edition approved May 1, 2016. Published May 2016. Originally approved in 1996. Last previous edition approved in 2008 as F1710 – 08. DOI: 10.1520/F1710-08R16.

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

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, or both, is scanned.

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.

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,  $M$ .  $RSF$ 's 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  $RSF$ 's.  $I(X_i)$  and  $I(Y_j)$  refer to the measured ion current from isotopes  $X_i$  and  $Y_j$ , respectively, of atomic species  $X$  and  $Y$  as follows:

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

where  $(X)/(Y)$  is the concentration ratio of atomic species  $X$  to species  $Y$ . If species  $Y$  is taken to be the titanium 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 between 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 titanium. 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 titanium 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.

## 7. Reagents and Materials

7.1 *Reagent and High Purity Grade Reagents*, as required (MeOH, HNO<sub>3</sub>, HF, H<sub>2</sub>O<sub>2</sub>).

7.2 *Demineralized Water*.

7.3 *Tantalum Reference Sample*.

7.4 *Titanium Reference Sample*.

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

7.4.2 As necessary, non-titanium 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 99.999 % pure (5N-grade) or better titanium 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 99.999 % pure (5N-grade) or better titanium 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.

8.2 The machined surface of the specimen must be cleaned by chemical 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 etching solutions in a clean container insoluble in the contained solution.

8.2.4 Useful etching solutions are HNO<sub>3</sub>:HF::3:1 or HNO<sub>3</sub>:HF:H<sub>2</sub>O<sub>2</sub>: :1:1:1 or H<sub>2</sub>O:HNO<sub>3</sub>:HF:H<sub>2</sub>O<sub>2</sub>::20:5:5:4

**TABLE 1 Suite of Impurity Elements to be Analyzed, with Appropriate Isotope Selection**

NOTE 1—Establish RSFs for the following suite of elements, using the indicated isotopes for establishing RSF values and for performing analyses of test specimens.

NOTE 2—This selection of isotopes minimizes significant interferences (see Annex A1.). Additional species may be determined and reported, as agreed upon by all parties concerned with the analyses. Other isotopes can be selected to assist mass spectrum peak identification or for other purposes.

|            |  |
|------------|--|
| Lithium    | <sup>7</sup> Li                        |
| Beryllium  | <sup>9</sup> Be                        |
| Boron      | <sup>11</sup> B                        |
| Carbon     | <sup>12</sup> C                        |
| Nitrogen   | <sup>14</sup> N                        |
| Oxygen     | <sup>16</sup> O                        |
| Fluorine   | <sup>19</sup> F                        |
| Sodium     | <sup>23</sup> Na                       |
| Magnesium  | <sup>26</sup> Mg                       |
| Aluminum   | <sup>27</sup> Al                       |
| Silicon    | <sup>28</sup> Si                       |
| Phosphorus | <sup>31</sup> P                        |
| Sulfur     | <sup>32</sup> S                        |
| Chlorine   | <sup>35</sup> Cl                       |
| Potassium  | <sup>39</sup> K                        |
| Calcium    | <sup>42</sup> Ca                       |
| Scandium   | <sup>45</sup> Sc                       |
| Titanium   | <sup>48</sup> Ti                       |
| Vanadium   | <sup>51</sup> V                        |
| Chromium   | <sup>52</sup> Cr                       |
| Manganese  | <sup>55</sup> Mn                       |
| Iron       | <sup>56</sup> Fe                       |
| Cobalt     | <sup>59</sup> Co                       |
| Nickel     | <sup>60</sup> Ni                       |
| Copper     | <sup>63</sup> Cu                       |
| Zinc       | <sup>66</sup> Zn or <sup>68</sup> Zn   |
| Gallium    | <sup>69</sup> Ga or <sup>71</sup> Ga   |
| Germanium  | <sup>70</sup> Ge or <sup>73</sup> Ge   |
| Arsenic    | <sup>75</sup> As                       |
| Selenium   | <sup>77</sup> Se                       |
| Bromine    | <sup>79</sup> Br                       |
| Rubidium   | <sup>85</sup> Rb                       |
| Yttrium    | <sup>89</sup> Y                        |
| Zirconium  | <sup>91</sup> Zr                       |
| Niobium    | <sup>93</sup> Nb                       |
| Molybdenum | <sup>100</sup> Mo                      |
| Ruthenium  | <sup>101</sup> Ru                      |
| Rhodium    | <sup>103</sup> Rh                      |
| Silver     | <sup>107</sup> Ag                      |
| Palladium  | <sup>106</sup> Pd or <sup>108</sup> Pd |
| Cadmium    | <sup>114</sup> Cd                      |
| Indium     | <sup>115</sup> In                      |
| Tin        | <sup>117</sup> Sn or <sup>119</sup> Sn |
| Antimony   | <sup>121</sup> Sb                      |
| Iodine     | <sup>127</sup> I                       |
| Tellurium  | <sup>125</sup> Te or <sup>130</sup> Te |
| Cesium     | <sup>133</sup> Cs                      |
| Barium     | <sup>138</sup> Ba                      |
| Lanthanum  | <sup>139</sup> La                      |
| Cerium     | <sup>140</sup> Ce                      |
| Neodymium  | <sup>146</sup> Nd                      |
| Hafnium    | <sup>176</sup> Hf or <sup>178</sup> Hf |
| Tantalum   | <sup>181</sup> Ta                      |
| Tungsten   | <sup>184</sup> W                       |
| Rhenium    | <sup>187</sup> Re                      |
| Osmium     | <sup>190</sup> Os or <sup>192</sup> Os |
| Iridium    | <sup>191</sup> Ir                      |
| Platinum   | <sup>194</sup> Pt or <sup>196</sup> Pt |
| Gold       | <sup>197</sup> Au                      |
| Mercury    | <sup>201</sup> Hg or <sup>202</sup> Hg |
| Thallium   | <sup>205</sup> Tl                      |
| Lead       | <sup>208</sup> Pb                      |
| Bismuth    | <sup>209</sup> Bi                      |
| Thorium    | <sup>232</sup> Th                      |
| Uranium    | <sup>238</sup> U                       |

(double etched), etching until smooth, clean metal is exposed over the entire surface.

8.2.5 Immediately after cleaning, wash the specimen with high purity rinses and thoroughly dry the specimen in the laboratory environment.

NOTE 1—Examples of acceptable high purity rinses are very large scale integration (VLSI) grade methanol and distilled water.

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 noncontacting 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 (12.3) to ensure the cleanliness of the surface. Pre-analysis sputtering conditions can be limited by the need to maintain sample integrity. If sputter cleaning and analysis are carried out under different plasma conditions, accuracy should be established for the analytical protocol adopted and elements measured.

## 9. Preparation of the GDMS Apparatus

9.1 The ultimate background pressure in the ion source chamber should be less than  $1 \times 10^{-6}$  torr before operation. The background pressure in the mass analyzer should be less than  $5 \times 10^{-7}$  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 (<sup>181</sup>Ta) using the ion current Faraday cup detector, and measure the ion current from the minor isotope (<sup>180</sup>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 <sup>180</sup>Ta ion current to the <sup>181</sup>Ta ion current by the <sup>181</sup>Ta/<sup>180</sup>Ta isotopic ratio. The result of this calculation is the ion counting detector efficiency (ICE).

**TABLE 2 Required Relative Standard Deviation (RSD) for RSF Determination, Pre-sputtering Period, and Plasma Stability Tests**

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

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 titanium 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 nonrandom 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 NIST traceable reference materials, preferably titanium, to the extent such reference samples are available.

11.2 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 (Table 1) using the selected isotopes (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.

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

12.1.2 Instrumental parameters selected for isotope measurements must be appropriate for the analysis requirements: (a) ion current integration times to achieve desired precision and detection limits; and (b) 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 analysis sputtering conditions, ensuring that the gas pressure required to do so is within normal range.

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 2 before terminating pre-analysis sputtering and proceeding to the next step.

12.3.1.3 Measure the specimen using the full DAP.

12.3.1.4 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 Subject the sample to at least 10 min of pre-analysis sputtering.

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 for all of the elements included in the MDAP.

12.3.2.5 If the concentration differences between the last two measurements are less than 5 %, 10 % or 20 %, depending on concentration (Table 2), 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 (a) that the analyte signal must be determined in the presence of a substantial signal from an interfering ion and in the case (b), 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 (13.1.1) or Type II (13.2) detection limit should be calculated and reported. If the analyte peak is



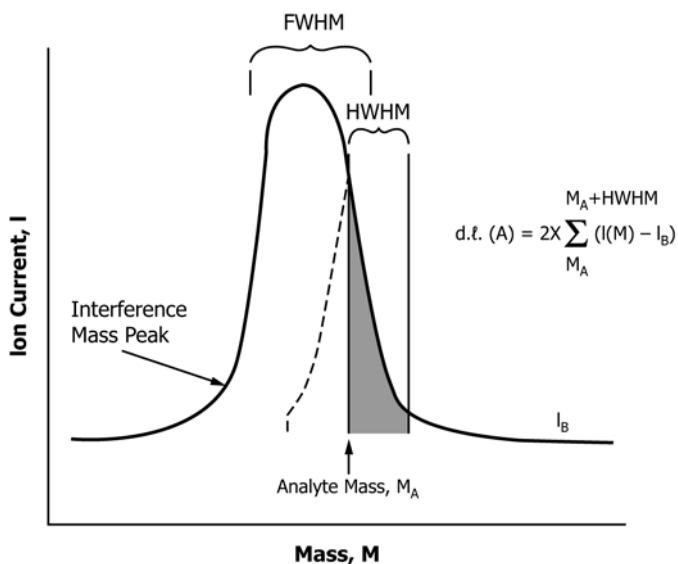


FIG. 1 Type II Detection Limit

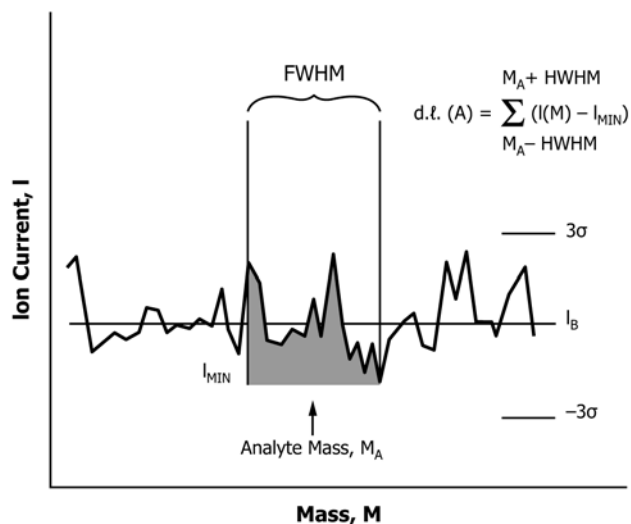


FIG. 2 Type III Detection Limit

obscured by statistical variation, a Type III detection limit (13.3) should be calculated and reported.

13.1.1 The following procedures 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.

*Type I Detection Limit (d.l.):*

13.1.1.1 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.

13.1.1.2 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.

13.1.1.3 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.

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

13.2 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.

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

13.3 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:

13.3.1 If  $N$  is the sum of the ion counts within the FWHM range about  $M$ , then the detection limit is as follows:<sup>4</sup>

$$\text{detection limit} = 3 + 5 \times \sqrt{N} \quad (2)$$

with appropriate quantitation for the element in question.

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

13.3.2.1 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  $M$ .

13.3.3 The Type III procedures 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.

<sup>4</sup> Currie, L. A., "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, Vol 40, 1968, pp. 586–593.

#### 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 Report the following information:

15.1.1 For a survey analysis, provide concentration data for the suite of elements (isotopes) listed in **Table 1**. Fewer elements may be listed as agreed upon between all parties concerned with the analysis, but care must be taken to include elements that may cause mass spectral interferences for the requested elements.

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

15.1.3 Element concentration shall be reported, typically, in units of parts per million by weight (ppmw).

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

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

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

#### 16. Precision and Bias

16.1 An inter-laboratory comparison test (“round robin”) in accordance with Practices E173, E180, and E691 is being organized and executed by the responsible technical subcommittee.

#### 17. Keywords

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

## ANNEX

### (Mandatory Information)

#### A1. MASS SPECTRUM INTERFERENCES FOR TITANIUM ANALYSIS BY GDMS

A1.1 Ions of the following atoms and molecular combinations of titanium, argon plasma gas isotopes, plasma impurities (C, H, O, Cl) and tantalum source components can significantly interfere with the determination of the ion current of the selected isotopes at low element concentrations. They are listed as follows:

$^{38}\text{Ar}^{++}$  interferes with  $^{19}\text{F}^+$   
 $^{46}\text{Ti}^{++}$  interferes with  $^{23}\text{Na}^+$   
 $^{12}\text{C}^{16}\text{O}^+$  interferes with  $^{28}\text{Si}^+$

$^{40}\text{Ar}^{38}\text{Ar}^1\text{H}^+$  interferes with  $^{79}\text{Br}^+$   
 $(^{40}\text{Ar}_2)^+$  scattered ions interfere with  $^{79}\text{Br}^+$   
 $^{36}\text{Ar}^{49}\text{Ti}^+$  interferes with  $^{85}\text{Rb}^+$

$(^{16}\text{O}_2)^+$  interferes with  $^{32}\text{S}^+$   
 $^{38}\text{Ar}^1\text{H}^+$  interferes with  $^{39}\text{K}^+$   
 $^{40}\text{Ar}^+$  scattered ions interfere with  $^{39}\text{K}^+$   
 $^{41}\text{K}^+$   
 $^{36}\text{Ar}^{48}\text{Ti}^{++}$  interferes with  $^{42}\text{Ca}^+$   
 $^{40}\text{Ar}^{48}\text{Ti}^{++}$  interferes with  $^{44}\text{Ca}^+$   
 $^{12}\text{C}^{16}\text{O}_2^+$  interferes with  $^{44}\text{Ca}^+$   
 $^{40}\text{Ar}^{12}\text{C}^+$  interferes with  $^{52}\text{Cr}^+$   
 $^{40}\text{Ar}^{16}\text{O}^+$  interferes with  $^{56}\text{Fe}^+$   
 $^{47}\text{Ti}^{16}\text{O}^+$  interferes with  $^{63}\text{Cu}^+$   
 $^{40}\text{Ar}^{35}\text{Cl}^+$  interferes with  $^{75}\text{As}^+$   
 $^{40}\text{Ar}^{36}\text{Ar}^1\text{H}^+$  interferes with  $^{77}\text{Se}^+$

$^{40}\text{Ar}^{48}\text{Ti}^+$  interferes with  $^{88}\text{Sr}^+$   
 $^{40}\text{Ar}^{49}\text{Ti}^+$  interferes with  $^{89}\text{Y}^+$   
 $^{46}\text{Ti}^{47}\text{Ti}^+$  interferes with  $^{93}\text{Nb}^+$   
 $^{50}\text{Ti}^{50}\text{Ti}^+$  interferes with  $^{100}\text{Mo}^+$   
 $^{40}\text{Ar}^{16}\text{O}^{47}\text{Ti}^+$  interferes with  $^{103}\text{Rh}^+$   
 $^{40}\text{Ar}^{36}\text{Ar}^{38}\text{Ar}^+$  interferes with  $^{114}\text{Cd}^+$   
 $^{48}\text{Ti}^{48}\text{Ti}^{50}\text{Ti}^+$  interferes with  $^{146}\text{Nd}^+$   
 $^{181}\text{Ta}^{16}\text{O}^+$  interferes with  $^{197}\text{Au}^+$

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