Designation: E2371 – 04

StandardTest Method for
Analysis of Titanium and Titanium Alloys by Atomic Emission Plasma Spectrometry[1]

This standard is issued under the fixed designation E2371; 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 method describes the analysis of titanium and titanium alloys by ICP-AES (Inductively Coupled Plasma) and DCP-AES (Direct Current Plasma) for the following elements:

Element	Application Range (wt.%)	Quantitative Range (wt.%)
Aluminum	0–8	0.001 to 8.0
Boron	0–0.04	0.0008 to 0.01
Chromium	0–5	0.005 to 4.0
Copper	0–0.6	0.002 to 0.5
Iron	0–3	0.004 to 3.0
Manganese	0–0.04	0.001 to 0.01
Molybdenum	0–8	0.004 to 6.0
Nickel	0–1	0.001 to 1.0
Silicon	0–0.5	0.02 to 0.4
Tin	0–4	0.02 to 3.0
Vanadium	0–15	0.01 to 15.0
Yttrium	0–0.04	0.001 to 0.004
Zirconium	0–5	0.003 to 4.0

1.2 *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.* Specific safety hazards statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
E1479 Practice for Describing and Specifying Inductively-Coupled Plasma Atomic Emission Spectrometers
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods
E1832 Practice for Describing and Specifying a Direct Current Plasma Atomic Emission Spectrometer
E1914 Practice for Use of Terms Relating to the Development and Evaluation of Methods for Chemical Analysis

3. Terminology

3.1 For definitions of terms used in this method, refer to Terminology E135 and Terminology section in E1914.

4. Summary of Test Method

4.1 A mineral acid solution of the sample is aspirated into an inductively coupled plasma or a direct current plasma spectrometer. The intensities of emission lines from the spectra of the analytes are measured and compared with calibration curves obtained from solutions containing known amounts of pure elements.

5. Significance and Use

5.1 This method for the chemical analysis of titanium and titanium alloys is primarily intended to test material for compliance with specifications of chemical composition.

5.2 It is assumed that all who use this method will be trained analysts capable of performing common laboratory procedures skillfully and safely, and that the work will be performed in a properly equipped laboratory.

5.3 The method is designed to give the maximum flexibility analyzing elements in the titanium matrix. Thus options are given in calibration and analysis to accommodate the variety of ICP and DCP spectrometers and their auxiliary systems.

6. Interferences

6.1 Potential interferences for analytes (see 1.2) are listed in Table 1 for this method's analytical wavelengths. The analytical wavelengths of Table 1 were selected for their freedom from interference by the elements included in this method and the titanium base (see Note 1).

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.06 on Ti, Zr, W, Mo, Ta, Nb, Hf, Re.

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

TABLE 1 Analytical Lines and Potential Interferences

Element	Wavelength (nm)	Potential Interferences	Notes
Al	394.401		
B	249.678		
Cr	267.716		
Cu	327.396		
Fe	259.940		
Mn	257.610		
Mo	202.030		
Ni	231.604		
Si	288.160	Cr	
Sn	175.790		2
	242.949	Mo, Fe	3
V	292.402	Mo, Fe	
Y	360.073	Mo	
Zr	343.823		

Note—Sn 175.790 – Low UV Capable
Sn 242.949 – DCP; non-Low UV capable alternative

NOTE 1—Elements outside the scope of this method may be present in production materials or experimental titanium alloys. These potential interferences must be tested for their effect as an interference on analyte wavelengths and corrections made as necessary as a part of method development according to manufacturer's instructions.

7. Apparatus

7.1 Direct Current Plasma Atomic Emission Spectrometers used in this method may conform to the specifications given in Practice E1832. A differently designed instrument may provide equivalent measurements. Suitability for use is determined by comparing the results obtained with the precision and bias statements contained in this method.

7.2 Inductively Coupled Plasma Atomic Emission Spectrometers used in this method may conform to the specifications given in Practice E1479. A differently designed instrument may provide equivalent measurements. Suitability for use is determined by comparing the results obtained with the precision and bias statements contained in this method.

7.3 The sample introduction system shall be constructed of materials resistant to all mineral acids including HF.

7.4 Each instrument shall be set up according to the manufacturer's instructions.

8. Reagents and Materials

8.1 Purity of reagents utilized in this procedure shall conform to the requirements prescribed in Practice E50.

8.2 The purity of water used shall conform to the requirements in Specification D1193 for reagent water, Type II.

8.3 The argon supply for the ICP or DCP plasma shall be of 99.998 % minimum purity; for purging the ICP Optical path, 99.999 % minimum purity.

8.4 The nitrogen supply for purging the ICP Optical path shall be 99.995 % minimum purity.

8.5 Reference solutions are available for purchase as an alternative to the preparation of solutions from pure metals or compounds.

8.5.1 Single element reference solutions in the form of Certified Reference Materials are available from the National Institute of Standards and Technology (NIST) and a number of commercial suppliers. The NIST Standard Reference Materials (SRM) 3100[3] series are available as 10 mg/g (5 mg/g for some elements) solutions. See Notes 2 and 3.

8.5.2 Reference solutions from other sources may be used if they have a documented link of traceability to primary reference materials or CRMs from a national metrology institute and the concentrations of impurities are known or determined (see Note 4).

NOTE 2—To use solution standards of concentration less than the original concentration, prepare the solution by dilution of the concentrated reference solution. Maintain the acid concentration as recommended in the NIST or other certificate. NIST certificates provide instructions for dilution by mass or by volume. Dilution by mass may be preferable to pipetting, especially for solutions containing HF.

NOTE 3—The user should establish internal laboratory procedures that specify a maximum shelf life for a working standard solution. It has been observed that polytetrafluoroethylene (PTFE) bottles preserve the integrity of the solutions stored in them for a long period of time.

NOTE 4—If single element solutions are not provided with values for trace impurities, it is necessary to either determine the concentrations of elements that are in the scope of this method or, if they cannot be detected, ensure that the method detection limit is sufficiently low that the impurities cannot be detected in solutions prepared for calibration of this method.

8.5.3 *Calibrants Prepared from Pure Metals or their Salts* (see Notes 5 and 6):

NOTE 5—Elemental solutions of 10.0 mg/mL or 1.00 mg/mL can be stored in HDPE or polytetrafluoroethylene (PTFE) for one (1) year. The 0.100 mg/mL solutions must be made fresh before each use.

NOTE 6—Elements and compounds for the solutions below are usually supplied on a "metals basis" assay. The concentration of the element sought, as percent in the present usage, is the total metallic content of the compound less the assayed metallic impurities (including B, Si, Y).

8.5.3.1 *Aluminum Standard Solution A* (1 mL = 10.0 mg Al)—Dissolve 10.000 g aluminum wire (purity 99.99 %, min) in 200 mL of HCl (1+1) with gentle heating. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix. (A small crystal of mercuric nitrate may be added to catalyze the dissolution reaction.)

8.5.3.2 *Boron Standard Solution A* (1 mL = 1.00 mg B)—Dissolve 5.720 g of boric acid, H_3BO_3 (purity 99.99 %, min) in 500 mL of water. Transfer to a 1 L volumetric flask, dilute to the mark and mix. Store in a plastic bottle.

8.5.3.3 *Chromium Standard Solution A* (1 mL = 10.0 mg Cr)—Dissolve 10.00 g of chromium (purity 99.99 %, min.) chips in 200 mL of HCl (1+1) with gentle heating. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.4 *Copper Standard Solution A (1 mL = 10.0 mg Cu)*—Dissolve 10.00 g of copper wire (purity 99.99 %, min.) in 100 mL of HNO_3 (1+1) with gentle heating. Continue heating until brown fumes cease to evolve. Cool, transfer to a 1 L volumetric flask, and add 50 mL of HNO_3, dilute to the mark and mix.

8.5.3.5 *Copper Standard Solution B* (1 mL = 1.00 mg Cu)—Pipet 10 mL of Copper Standard Solution A into a 100 mL volumetric flask, add 10 mL of HNO_3, dilute to the mark and mix.

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[3] Available from National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD 20899, USA.

8.5.3.6 *Iron Standard Solution A* (1 mL = 10.0 mg Fe)—Dissolve 10.00 g of iron rod (purity 99.99 %, min, that has been cleaned to remove oxidation) in 100 mL of HNO_3 by heating to boiling. Continue gentle boiling until brown fumes cease to evolve. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.7 *Manganese Standard Solution A* (1 mL = 1.00 mg Mn)—Dissolve 1.000 g of manganese (purity 99.98 %, min) in 100 mL of HNO_3 (1+1) with gentle heating. Boil gently to expel brown fumes and cool. Transfer to a 1 L volumetric flask, add 50 mL of HNO_3, dilute to the mark and mix.

8.5.3.8 *Molybdenum Standard Solution A* (1 mL = 10.0 mg Mo)—Transfer 5.00 g of molybdenum rod (purity 99.98 %, min) to a 600 mL beaker. Add 200 mL of an acid mixture of HCl, HNO_3 and water (3+2+1) and heat gently to dissolve. Cool, transfer to a 500 mL volumetric flask, dilute to the mark and mix.

8.5.3.9 *Nickel Standard Solution A* (1 mL = 10.0 mg Ni)—Dissolve 10.00 g of nickel wire (purity 99.99 %, min) in 100 mL of HNO_3 with gentle heating. Continue heating until brown fumes are no longer evolved. Cool, transfer to a 1 L volumetric flask, dilute to the mark and mix.

8.5.3.10 *Nickel Standard Solution B* (1 mL = 1.00 mg Ni)—Pipet 10 mL of Nickel Standard Solution A into a 100 mL volumetric flask, add 10 mL of HNO_3, dilute to the mark and mix.

8.5.3.11 *Silicon Standard Solution A* (1 mL = 1.00 mg Si)—Fuse 0.2139 g of silicon dioxide (purity 99.99 %, min) with 2.00 g of sodium carbonate (purity 99.995 %, min) in a platinum crucible. Dissolve the melt in water, transfer to a 100 mL flask, dilute to the mark and mix. Store in a plastic bottle.

8.5.3.12 *Tin Standard Solution A* (1 mL = 10.0 mg Sn)—Dissolve 10.00 g of tin wire (purity 99.99 %, min) in 100 mL of HCl with gentle heating. Cool, transfer to a 1 L volumetric flask, and add 400 mL of HCl, dilute to the mark and mix.

8.5.3.13 *Vanadium Standard Solution A* (1 mL = 10.0 mg V)—Dissolve 17.852 g of vanadium (V) oxide ($V_2 O_5$) (purity 99.99 %, min) in a 1 L volumetric flask by adding 300 mL of HCl and warm (≤ 75°C) to complete dissolution. Cool, dilute to the mark and mix.

8.5.3.14 *Yttrium Standard Solution A* (1 mL = 1.00 mg Y)—Dissolve 1.270 g of yttrium oxide (Y_2O_3) (purity 99.99 %, min) in 30 mL of HCl (1+1). Transfer to a l L volumetric flask, dilute to the mark and mix.

8.5.3.15 *Zirconium Standard Solution A* (1 mL = 10.0 mg Zr)—Transfer 35.326 g of $ZrOCl_2 \cdot 8H_2O$ (purity 99.99 %, min) to a 1 L volumetric flask, add approximately 500 mL of water, and swirl until dissolved. Add 50 mL of HCl, cool, dilute to the mark and mix.

8.6 Titanium base metal for the production of all calibration standards shall be of 99.99 % minimum purity on a metals basis. See Notes 6 and 7.

NOTE 7—Impurities of this titanium base material shall be determined by Glow Discharge Mass Spectrometry (GDMS) or suitable technique that has detection limits for each respective analyte that is less than that of the ICP or DCP in the titanium matrix.

8.7 An internal standard may be used to control drift. If an internal standard is used, it shall not interfere with the analytes of this method.

8.8 A surfactant may be used to control droplet formation in the spray chamber and sample tubes of the DCP spectrometer. A maximum 0.1 % Vol/Vol of a surfactant such as Polyoxyethyene(10)isooctylphenyl ether is recommended.

8.9 *Calibration Solutions:*

8.9.1 *Standard Solution A:*

8.9.1.1 Add 5.00 g (ICP system) or 10.00 g (DCP system) of titanium (purity 99.99 %, min) to a 1 L plastic volumetric flask (see Note 8).

NOTE 8—The ICP procedure and DCP procedure require differing sample sizes. This is maintained consistently throughout the method in terms of sample weights and analyte additions for samples, calibration solutions and quality control solutions.

8.9.1.2 Add 150 mL of HCl, then 20 mL of HF (see warning note below in 8.9.1.4).

8.9.1.3 After all titanium has dissolved, add 10 mL of HNO_3 (ICP) or 15 mL of HNO_3 (DCP) to oxidize.

8.9.1.4 Cool, add internal standard solution and/or surfactant if desired, dilute to the mark and mix. (**Warning**—HF causes burns that require immediate medical attention even though they are not immediately painful; refer to the paragraph about HF in the Safety Precautions of Practice E50.)

8.9.1.5 Store in a polytetrafluoroethylene (PTFE) bottle. Label the bottle and mark with the date of preparation. This solution's shelf life is six (6) months.

8.9.1.6 Adjust the "0" values of Standard A to reflect the actual concentration of the analytes in the titanium base material before calibration.

8.9.2 *Standard Solutions B-G:*

8.9.2.1 Weigh quantities of titanium (purity 99.99 % min) into 1 L plastic volumetric flasks in accordance with Appendix X1 for ICP and Appendix X2 for DCP.

8.9.2.2 To each, add the specified volumes of HCl and HF (see warning in 8.9.1.4) in accordance with Appendix X1 for ICP and Appendix X2 for DCP.

8.9.2.3 After the titanium dissolves completely, add the specified volumes of HNO_3 in accordance with Appendix X1 for ICP and Appendix X2 for DCP.

8.9.2.4 Cool, add volumes of standard solutions in accordance with Appendix X1 for ICP and Appendix X2 for DCP.

8.9.2.5 Add internal standard solution and/or surfactant if desired. Dilute to volume and mix. Store in a polytetrafluoroethylene (PTFE) bottle. Shelf life is three (3) months.

8.9.2.6 Adjust the concentration values of Standard Solutions B-G to reflect the actual concentration of the analytes in the titanium base material before calibration.

8.9.3 *Standard Solution H (Optional):*

8.9.3.1 Some instrument systems require more data points for optimum calibration, not only in the range of the major alloy elements but also the trace elements as their concentration approaches zero. An additional calibration material can be used to satisfy this requirement (see Note 9).

8.9.3.2 This calibration reference material can be a "matrix-matched" solution which contains the major alloying elements (additions of the solutions of section 8.5.3) and the traces of the analytes (made from additions of serial dilutions of the solutions of paragraph 8.5.3) added to the appropriate weight of titanium base. The solution is then acid balanced and the internal standard and/or surfactant are then added so that this optional standard H is consistent with the Standards A-G and the object unknown samples in total solids (Ti plus alloying elements) content, acidity and optional additives (internal standard and surfactant).

(1) Additions of major elements of the alloy array (for example, Al, Cr, Fe, Mo, Si, Sn, V, Zr) are calculated and added to the titanium base material (calculated for weight depending upon the total alloying element content) in the same manner as Standard Solutions B-G.

(2) For elements B, Cu, Mn, Ni, Y at the trace level the target is 25 to 50 % of the values given in Standard Solution B.

(3) For trace levels of elements Al, Cr, Fe, Mo, Si, Sn, V, Zr, the target is set at 0.01 0.05 % in final concentration (metals basis).

8.9.3.3 Alternatively, the optional standard solution H can be a material which is a CRM or other well characterized internal reference material that contains the analytes of interest.

NOTE 9—Some modern instruments are limited in their dynamic range for calibration of several elements and may require additional optional calibration materials of the type described in this section .

9. Hazards

9.1 This method involves the use of concentrated HF. Read and follow label precautions, MSDS information, and refer to Practice E50.

9.2 For precautions to be observed in the use of certain reagents in this test method, refer to Practice E50.

10. Sampling

10.1 Select a sample portion for analysis representing equally all parts of the laboratory sample.

10.2 Laboratory samples shall consist of drillings, chips, millings, turnings, powder, or platelets free from oxidation and other extraneous contaminants. Bulk sampling of materials is not within the scope of this method.

11. Calibration

11.1 Set up the instrument for operation in accordance with the manufacturer's recommendations. Prepare the instrument for calibration as recommended by the manufacturer.

11.2 Optimize the analyte wavelengths with analyte concentrations consistent with the concentrations of the calibration standards and titanium matrix according to the manufacturer's instructions.

11.3 Verify that the instrument is stable and free from drift. Use the calibration solutions to calibrate the instrument in accordance with manufacturer's instructions.

11.4 Calibrate the instrument according to manufacturer's instructions. Select the standard solutions A-H to bracket the analyte concentration. The whole system may be calibrated utilizing the whole array of calibration reference materials. If desired, calibrations may be separated so that certain elements can be analyzed together or determined individually. Alternatively, the major alloying elements and the trace elements may be grouped and analyzed separately.

11.5 Analyze a control sample or samples and apply the values to control charts to demonstrate statistical control of the calibration and measurement system. If the results do not fall within predetermined control limits based on the laboratory's prior experience with the method, identify and correct the problem. If necessary, calibrate the spectrometer again. The user should consult a guide such as Guide E882 for appropriate control chart systems.

12. Procedure

12.1 Weigh 1.000 g (ICP) or 2.000 g (DCP) of a titanium sample into either a polytetrafluoroethylene (PTFE) 250 mL beaker or directly into a 200 mL plastic volumetric flask. Add 30 mL of HCl and 4 mL of HF (see warning in 8.9.1.4).

12.2 When dissolution is complete (approximately 5 to 10 min), add 2 or 3 mL of HNO_3 for the ICP dissolution or the DCP dissolution respectively (see Note 10). If a beaker is used, transfer to a 200 mL plastic flask with 3 generous rinsings as a minimum.

NOTE 10—Alloys containing palladium may not completely dissolve. Some palladium may remain as a finely divided black suspension in beaker or flask. Heat gently to dissolve it before proceeding.

12.3 Cool, add internal standard solution and/or surfactant if used, dilute to volume and mix.

12.4 Analyze sample solutions in accordance with manufacturer's instructions. If more than 10 samples are to be analyzed, analyze control samples after each group of 10. If controls exceed established limits for one or more analytes, recalibrate the instrument for the affected analytes. Reanalyze the previous group of samples before proceeding with other samples. Use a quality control system such as Guide E882.

13. Calculation and Report

13.1 Program the instrument to report results in weight percent.

14. Precision and Bias

14.1 *Precision*—Seven laboratories cooperated in testing this method. Tables 2-14 present summaries of the precision information. The interlaboratory data were evaluated in accordance with Practice E1601 and Guide E1763.

14.2 An approximate value for the expected reproducibility index, R_C, at any concentration, C, within the quantitative range of this method (see 1.1) can be calculated by Eq 1. The constants, K_R and K_{rel}, are presented in Table 15 (see Note 11).

$$R_C = \left(K_R{}^2 + (C \cdot K_{rel})^2\right)^{1/2} \quad (1)$$

NOTE 11—Manganese within this quantitative range follows the constant error model (see Table 7).

14.3 *Bias*—The accuracy of the method has been deemed satisfactory on the basis of the bias information in Tables 2-14. Users are encouraged to use these or similar reference materials to assure proper analytical performance.

15. Keywords

15.1 atomic emission spectrometry; DCP; direct current plasma; ICP; inductively coupled plasma; titanium; titanium alloys

TABLE 2 Aluminum in Titanium and Titanium Alloys by Atomic Emission Spectrometry

Test Material	Number of Laboratories	Aluminum Found, %	Minimum SD (s_M, E1601)	Reproducibility SD (s_R, E1601)	Reproducibility Index (R, E1601)	R_{rel}%
6	6	0.000383	0.000804	0.00161	0.00451	1177.
5	6	0.0339	0.000969	0.00299	0.00839	24.7
4	6	0.099	0.00168	0.00545	0.0152	15.4
1	6	3.136	0.0371	0.0632	0.177	5.6
3	6	6.435	0.0478	0.133	0.373	5.8
2	6	7.723	0.0589	0.128	0.359	4.6

Test Material	Certified Aluminum, %	Bias, %	Material Identification (Source)	Description (Uncertainty or Standard Deviation)
1	3.08	0.083	NIST 649	Ti-15V-3Al-3Cr-3Sn (0.02)
2	7.63	0.102	NIST 2433	Ti-8Al-1Mo-1V (0.05)
3	6.36	0.075	NIST 173b	Ti-6Al-4V (0.04)
4	0.096	0.003	HTL 572	TIMET ILT, Ti-CP (0.009)
5	0.031	0.0029	HTL AA16#4	TIMET Internal, Ti-CP (0.001)
6	0.0001	0.000283	HTL 559	TIMET Electro-refined (no data)

Note—The General Analytical Error model estimates relative errors decrease above 0.1 % Al to a minimum of 5.2 %.

TABLE 3 Boron in Titanium and Titanium Alloys by Atomic Emission Spectrometry

Test Material	Number of Laboratories	Boron Found, %	Minimum SD (s_M, E1601)	Reproducibility SD (s_R, E1601)	Reproducibility Index (R, E1601)	R_{rel}%
3	6	0.000095	0.000094	0.000137	0.000384	404.
4	5	0.00487	0.000458	0.000462	0.00129	26.6
1	7	0.00488	0.000076	0.000340	0.00095	19.5
2	5	0.00822	0.000393	0.000480	0.00134	16.3

Test Material	Certified Boron, %	Bias, %	Material Identification (Source)	Description (Uncertainty or Standard Deviation)
1	0.0048	0.00007	HTL 572	TIMET ILT, Ti-CP (0.009)
2	0.0093	-0.00108	HTL AA16#4	TIMET Internal, Ti-CP (0.001)
3	<0.0001	0.0000	HTL 559	TIMET Electro-refined (no data)
4	0.0052	-0.00033	HTL 573	TIMET ILT, Ti-6Al-4V (0.0003)

Note—The General Analytical Error model estimates relative errors decrease above 0.002 % to a minimum of 18.5 %.

TABLE 4 Chromium in Titanium and Titanium Alloys by Atomic Emission Spectrometry

Test Material	Number of Laboratories	Chromium Found, %	Minimum SD (s_M, E1601)	Reproducibility SD (s_R, E1601)	Reproducibility Index (R, E1601)	R_{rel}%
4	5	0.00035	0.000335	0.000862	0.00241	692.
3	5	0.0149	0.000191	0.000600	0.00168	11.3
2	5	0.0245	0.000203	0.001633	0.00457	18.7
1	5	3.809	0.01878	0.03363	0.0942	2.47

Test Material	Certified Chromium, %	Bias, %	Material Identification (Source)	Description (Uncertainty or Standard Deviation)
1	3.84	-0.031	NIST 648	Ti-5Al-2Sn-2Zr-4Cr-4Mo (0.03)
2	0.025	-0.0005	NIST 173b	Ti-6Al-4V (NIST 654B, 0.002)
3	0.0145	0.0004	HTL 572	TIMET ILT, Ti-CP (0.0005)
4	0.0003	0.00005	HTL 559	TIMET Electro-refined (no data)

Note—The General Analytical Error model estimates relative errors decrease above 0.1 % Cr to a minimum of 2.5 %.

TABLE 5 Copper in Titanium and Titanium Alloys by Atomic Emission Spectrometry

Test Material	Number of Laboratories	Copper Found, %	Minimum SD (s_M, E1601)	Reproducibility SD (s_R, E1601)	Reproducibility Index (R, E1601)	R_{rel}%
4	5	0.000353	0.00038	0.00066	0.00185	524.
1	7	0.00410	0.00033	0.00170	0.00476	116.
3	7	0.0206	0.00057	0.00281	0.00786	38.2
5	6	0.0211	0.00058	0.00215	0.00601	28.5
2	6	0.473	0.00938	0.0218	0.0610	12.9

Test Material	Certified Copper, %	Bias, %	Material Identification (Source)	Description (Uncertainty or Standard Deviation)
1	0.008	-0.0039	NIST 173b	Ti-6Al-4V(0.001) NIST 654B(0.004±0.001)
2	0.49	-0.017	IARM-178A	ARMI RM Ti-6Al-6V-2Sn (0.028)
3	0.025	-0.0044	HTL 572	TIMET ILT, Ti-CP (0.003)
4	<0.0001	<0.0003	HTL 559	TIMET Electro-refined (no data)
5	0.025	-0.0039	HTL 573	TIMET ILT, Ti-6Al-4V (0.001)

Note—The General Analytical Error model estimates relative errors decrease above 0.015 % Cu to a minimum of 13 %.

TABLE 6 Iron in Titanium and Titanium Alloys by Atomic Emission Spectrometry

Test Material	Number of Laboratories	Iron Found, %	Minimum SD (s_M, E1601)	Reproducibility SD (s_R, E1601)	Reproducibility Index (R, E1601)	R_{rel}%
4	6	0.00188	0.00015	0.00073	0.00203	108.
3	6	0.0444	0.00092	0.00144	0.00404	9.1
2	6	0.227	0.00196	0.00696	0.0195	8.6
1	6	1.706	0.0108	0.0205	0.0575	3.37

Test Material	Certified Iron, %	Bias, %	Material Identification (Source)	Description (Uncertainty or Standard Deviation)
1	1.77	-0.064	NIST 2432	Ti-10V-2Fe-3Al (0.05)
2	0.23	-0.003	NIST 173b	Ti-6Al-4V (0.01)
3	0.048	-0.0036	HTL 572	TIMET ILT, Ti-CP (0.003)
4	0.0011	0.00078	HTL 559	TIMET Electro-refined (no data)

Note—The General Analytical Error model estimates relative errors decrease above 0.06 % Fe to a minimum of 3.5 %.

TABLE 7 Manganse in Titanium and Titanium Alloys by Atomic Emission Spectrometry

Test Material	Number of Laboratories	Manganse Found, %	Minimum SD (s_M, E1601)	Reproducibility SD (s_R, E1601)	Reproducibility Index (R, E1601)	R_{rel}%
4	5	-0.00071	0.000008	0.00056	0.00157	219.
3	5	0.00388	0.000030	0.00046	0.00128	33.0
1	5	0.00420	0.000043	0.00050	0.00140	33.3
2	5	0.01277	0.000124	0.00041	0.00115	9.0

Test Material	Certified Manganse, %	Bias, %	Material Identification (Source)	Description (Uncertainty or Standard Deviation)
1	0.0048	-0.0006	HTL 572	TIMET ILT, Ti-CP (0.0006)
2	0.013	-0.00023	HTL AA16#4	TIMET Internal, Ti-CP (0.002)
3	0.0044	-0.00052	HTL 573	TIMET ILT, Ti-6Al-4V (0.0008)
4	0.0001	-0.00081	HTL 559	TIMET Electro-refined (no data)

Note—All laboratories reported negative concentrations for Material 4. Three materials cover the range of 0.004 to 0.013 % Mn. Within that range, the Constant Analytical Error [KC] model estimates the Reproducibility Index is 0.0013 % Mn.