



Designation: E 539 – 02

# Standard Test Method for X-Ray Emission Spectrometric Analysis of 6Al-4V Titanium Alloy<sup>1</sup>

This standard is issued under the fixed designation E 539; 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 X-ray emission analysis of 6Al-4V titanium alloy for the following elements in the ranges indicated (Note 1):

Element	Concentration Range, %
Aluminum	4.6 to 7.2
Vanadium	2.6 to 5.4
Iron	0.1 to 0.3

NOTE 1—The concentration ranges can be extended by use of suitable reference materials. The ranges for aluminum and vanadium represent the actual concentration ranges of the NIST<sup>2</sup> Standard Reference Materials<sup>®</sup> used when this method was tested. The maximum concentrations of the unknowns used in the cooperative test program were all lower than the reference materials. The iron concentration range was determined by in-house reference materials used by the cooperating laboratories.

1.2 This test method is designed for control analysis in the production of titanium alloys and for inspection testing of the product shipped to the consumer. It is applicable for analyzing cast or wrought samples having a diameter of approximately 32 mm and a thickness of approximately 25 mm.

NOTE 2—Samples of greater or lesser size than the size designed may be used with specially designed sample holders.

1.3 *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 precautionary statements are given in Section 8.

<sup>1</sup> 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 E1.06 on Ti, Zr, W, Mo, Ta, Nb, Hf.

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<sup>2</sup> NIST Standard Reference Materials<sup>®</sup> used when this study include NIST SRM<sup>®</sup> 653, 654, and 655 from NIST SRM<sup>®</sup> Program, 100 Bureau Drive, Mail Stop 2322, Building 202, Room 204, Gaithersburg, MP 20899–2322.

## 2. Referenced Documents

2.1 *ASTM Standards:*

E 120 Test Methods for Chemical Analysis of Titanium and Titanium Alloys<sup>3</sup>

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>3</sup>

## 3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology E 135.

## 4. Summary of Test Method

4.1 The sample is finished to a clean, uniform surface and then irradiated by an X-ray beam of high energy (short wavelength). The secondary X rays produced are dispersed by means of crystals and the intensities measured by detectors at selected wavelengths. The outputs of the detectors in voltage pulses are integrated or counted. Data are collected using time required to reach a fixed number of counts or using total counts for a fixed time. Concentrations of the elements are determined by relating the measured radiation of unknown samples to analytical curves or charts prepared from certified reference materials of known compositions. A fixed channel or polychromator system or a sequential system may be used to provide simultaneous or sequential determinations of elements.

## 5. Significance and Use

5.1 The chemical composition of 6Al-4V titanium alloy must be accurately determined to ensure the desired metallurgical properties. This test method is suitable for manufacturing control and inspection testing.

## 6. Apparatus

NOTE 3—It is not within the scope of this test method to prescribe all details of equipment to be used. Equipment varies between laboratories.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.05.

### 6.1 Sample Preparation Equipment:

6.1.1 *Surface Grinder*, with 60 to 600-grit silicon carbide belts or disks capable of providing test specimens with a uniform flat finish. A wet belt or wet disk grinder is preferred to prevent work hardening of the sample.

### 6.2 Excitation Source:

6.2.1 *X-Ray Generator*, providing constant potential or rectified power of sufficient energy to produce secondary radiation of the sample for the elements specified. The generator may be equipped with a line voltage regulator and current stabilizer, with voltage and current regulations up to 75 kV and 40 mA.

6.2.2 *X-Ray Tube*, with a high-purity tungsten or rhodium target capable of continuous operation up to potentials and currents shown in **Table 1**.

**TABLE 1 Operating Voltages and Currents**

Element	Voltage, kV with Current, mA
Iron	60-32, 51-48, 48-48, 48-22
Aluminum	60-32, 51-48, 41-26, 48-22
Vanadium	48-24, 40-24, 51-48, 48-46, 48-22
Chromium	48-24, 40-24, 51-48, 48-48, 48-22

6.3 *Spectrometer*, designed for X-ray emission analysis using air vacuum or helium, and equipped with specimen holders and specimen chamber. The chamber should contain a sample specimen spinner.

6.3.1 *Analyzing Crystals*, flat or curved lithium fluoride (LiF), pentaerythritol (PET), or ethylenediamine *d*-tartrate (EDdT).

6.3.2 *Collimator*, for limiting the characteristic X rays to a parallel bundle when flat crystals are used in the instrument. For curved crystal optics, no collimation is necessary.

6.3.3 *Detectors*—Sealed or gas-flow proportional counters, or equivalent.

6.3.4 *Vacuum System*, providing for the determination of elements whose radiation is absorbed by air. The system should consist of a vacuum pump, gage, and electrical controls to provide automatic pump-down of the optical path and to start the analysis at a pressure of 100  $\mu\text{m}$  or less, controllable to  $\pm 20 \mu\text{m}$ . A helium system can also be used.

6.4 *Measuring System*—An electronic circuit capable of amplifying, counting, and integrating pulses received from the detector tube. The system should be equipped with visual and automatic recording devices and have a pulse height analyzer, which is used for pulse voltage discrimination.

## 7. Reagents

7.1 *Detector Gas* (P-10), consisting of a mixture of 90 % argon and 10 % methane.

## 8. Safety Precautions

8.1 *Occupational Health and Safety Standards*<sup>4</sup> on ionizing radiation shall be observed at all X-ray emission spectrom-

eter installations. It is also recommended that personnel follow the guidelines of safe operating procedures given in the NIST Handbook *X-Ray Protection, HB76*,<sup>5</sup> the booklet *Radiation Safety Recommendations for X-Ray Diffraction and Spectrographic Equipment, #MORP 68-14*, 1968,<sup>6</sup> by T. M. Moore and D. J. McDonald, and the U. S. Government Handbook 93, *Safety Standard for Non-Medical X-Ray and Sealed Gamma-Ray Sources*, Part 1, General,<sup>5</sup> or similar handbooks of latest issue.

8.2 X-ray equipment should be used only under the guidance and supervision of a responsible, qualified person.

8.3 Suitable monitoring devices, either film badges or dosimeters, shall be worn by all personnel using the equipment. To meet local, state, and national radiation standards, periodic radiation surveys of the equipment for leaks and excessive scattered radiation shall be made by a qualified person using an ionization-chamber detector. The personal film badge survey record, the radiation survey record, and a maintenance record shall be available upon request.

8.4 Special precautions for the operator shall be posted.

8.5 X-ray caution signs shall be posted near the X-ray equipment and at all entrances to the radiation area.

8.6 Fail-safe “X-ray On” warning lights shall be used at the X-ray tube.

## 9. Reference Materials

9.1 Certified Reference Materials may include NIST SRM® 653, 654, 655, 655a or their replacements.<sup>7</sup>

9.2 Additional Certified Reference Materials are available from commercial sources. Select a minimum of three samples that contain vanadium, aluminum, and iron at the top and bottom of the concentration range and one intermediate.

## 10. Preparation of Standards and Samples

10.1 Grind the specimens to provide a flat, clean area over the entire surface to be exposed to the X-ray beam. Adhere rigorously to the preparation technique established.

## 11. Preparation of Apparatus

11.1 *Start Up*—Energize the power supply and electronic circuits for at least 1/2 h prior to taking measurements.

11.2 *Power Supply*—Adjust the voltage of the power supply to produce secondary fluorescence according to the expression:

$$V_f = 12 \ 350/\lambda_{K_{\text{abs}}} \quad (1)$$

where:

$V_f$  = minimum voltage required for exciting the element, and

$\lambda_{K_{\text{abs}}}$  = wavelength of the *K* absorption edge of the fluorescent element.

<sup>5</sup> Available from Superintendent of Documents, U. S. Government Printing Office, Washington, DC 20025.

<sup>6</sup> Available from U. S. Department of Health, Education and Welfare, Rockville, MD 20850.

<sup>7</sup> NIST SRM® Program, 100 Bureau Drive, Mail Stop 2322, Room 204, Gaithersburg, MD 20899-2322.

<sup>4</sup> Federal Register, Vol 36, No. 105, May 29, 1971, Sec. 1910.96 or of latest issue of Subpart G; Superintendent of Documents, U. S. Government Printing Office, NBS Handbook III.