



Designation: E1409 – 13 (Reapproved 2021)

Standard Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion¹

This standard is issued under the fixed designation E1409; 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 the determination of oxygen in titanium and titanium alloys in mass fractions from 0.01 % to 0.5 % and the determination of nitrogen in titanium and titanium alloys in mass fractions from 0.003 % to 0.11 %.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in 8.8.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

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

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)³

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

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.

³ The last approved version of this historical standard is referenced on www.astm.org.

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method, refer to Terminology E135.

4. Summary of Test Method

4.1 This test method is intended for use with automated, commercially available, inert gas fusion analyzers. These analyzers typically measure both oxygen and nitrogen simultaneously or sequentially utilizing parallel measurement systems.

4.2 The test sample, plus flux, is fused in a graphite crucible under a flowing inert gas stream at a temperature sufficient to release oxygen and nitrogen. Oxygen combines with carbon to form carbon monoxide (CO) and nitrogen is released as N₂. Depending on instrument design, the CO may be oxidized to carbon dioxide (CO₂). The CO or CO₂, or both, are swept by the inert gas stream into either an infrared or thermal conductivity detector. The detector response generated by analysis of the test sample is compared to the response generated by analysis of reference materials and the result is displayed as percent oxygen. The nitrogen is swept by the inert gas stream into a thermal conductivity detector. The detector response generated by analysis of the test sample is compared to the response generated by analysis of reference materials and the result is displayed as percent nitrogen.

4.3 In a typical instrument for the determination of nitrogen, the sample gases are swept with inert gas through heated rare earth/copper oxide that converts CO to CO₂ and hydrogen (H₂) to water (H₂O). The CO₂ is absorbed on sodium hydroxide impregnated on clay, and the H₂O is removed with magnesium perchlorate. The nitrogen, as N₂, enters the measuring cell and the thermistor bridge output is integrated and processed to display percent nitrogen.

5. Significance and Use

5.1 This test method is primarily intended as a test for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that the work will be performed in a properly equipped laboratory.

6. Interferences

6.1 The elements usually present in titanium and its alloys do not interfere but there is some evidence to suggest that low purity flux can cause some adsorption of the released oxygen.

7. Apparatus

7.1 *Instrument*—Fusion and measurement apparatus, automatic oxygen and nitrogen determinator consisting of an electrode furnace, provision for scrubbing impurities from analytical gas stream; infrared or thermal conductivity measurement system(s), or both, and auxiliary gas purification systems (Note 1).

NOTE 1—Several models of commercial oxygen and nitrogen determinators are available and presently in use by industry. Each has its own unique design characteristics and operational requirements. Consult the instrument manufacturer's instruction manual for operational details.

7.2 *Graphite Crucibles*—The crucibles must be made of high-purity graphite and be of the dimensions recommended by the instrument manufacturer.

7.3 *Flux*—Flux must be made of high-purity nickel. If nickel baskets are used, the dimensions must meet the requirements of the automatic sample drop, if present, on the instrument. (See Note 2.) Ultra high-purity nickel flux is commercially available and may eliminate the need to clean the flux before using it.

NOTE 2—In some instruments, nitrogen and oxygen are run sequentially and platinum is the required flux for nitrogen. High-purity platinum can be substituted for nickel in the same ratio of flux to sample.

7.4 *Tweezers or Crucible Tongs*, made of solvent and acid resistant material.

8. Reagents

8.1 *Acetone*—Low residue reagent grade or higher purity.

8.2 *Graphite Powder (optional)*—High-purity as specified by the instrument manufacturer.

8.3 *Inert Gas*—Use the purity and type specified by the instrument manufacturer.

8.4 *Magnesium Perchlorate, Anhydrous*⁴—Used in the instrument to absorb water. Use the purity specified by the instrument manufacturer.

8.5 *Nickel Flux Cleaning Solution*—An acid solution capable of removing surface contamination from the nickel flux. A solution made by combining 75 mL of acetic acid, 25 mL of HNO₃, and 2 mL of HCl has been found suitable for this purpose.

8.6 *Copper Oxide or Rare Earth/Copper Oxide*—Reagent used in some instruments to oxidize CO to CO₂ for detection. Use the purity specified by the instrument manufacturer.

8.7 *Sodium Hydroxide on Clay*⁵—Reagent used in some instruments to absorb CO₂. Use a purity specified by the instrument manufacturer.

8.8 *Titanium Sample Pickle Solution*—Three parts 30 % hydrogen peroxide (H₂O₂) and 1 part 48 % HF. Other pickle solutions may be substituted if there are data supporting the effectiveness of the solution on removing contaminants. For example, substituting concentrated HNO₃ for 30 % H₂O₂ has been found effective (see Note 3). (Warning—HF causes serious burns that may not be immediately painful; refer to the paragraph about HF in the Hazards Section of Practices E50.)

NOTE 3—In 2004, alternative sample preparation procedures (Section 12) were tested by seven laboratories. Three laboratories processed the sample materials by pickling their samples in HF-H₂O₂ (8.8). Two laboratories utilized the HF-HNO₃ alternative pickle solution (8.8). Two laboratories utilized abrasion (in this case diamond saw and shear) in accordance with 12.2. The prepared samples were distributed among the laboratories for analysis. Six laboratories analyzed these samples in random order under a single operator, single-day, single calibration sample run. The results of this testing are given in Tables X1.1 and X2.1 for oxygen and nitrogen, respectively. In both cases, the analysis of variance (ANOVA) indicates that there is no significant difference at the 95 % level of confidence for either oxygen or nitrogen due to the preparation technique.

9. Hazards

9.1 Use care when handling hot crucibles and operating furnaces to avoid personal injury by either burn or electrical shock.

9.2 For precautions to be observed in the use of HF and other reagents in this test method, refer to Practices E50.

10. Preparation of Apparatus

10.1 Assemble the apparatus as recommended by the manufacturer. Make the required power, gas, and water connections. Turn on the instrument and allow sufficient time to stabilize the equipment.

10.2 Change the chemical reagents and filters as required. Test the furnace and analyzer to ensure the absence of leaks (Note 4). A minimum of two test runs using a sample as directed in 14.3 and 14.4 is recommended to condition the newly changed filters. This should be done before attempting to calibrate the system or to determine the value of the blank.

NOTE 4—Typical leak checks should be 0.0 mm Hg to 1.5 mm Hg. The maximum allowable leak check should follow the manufacturer's recommendation.

11. Nickel Flux Preparation

11.1 Ultra high-purity nickel is commercially available that does not require the nickel cleaning procedure below. Its sufficiency must be verified by satisfactory blank determinations. If ultra high-purity nickel is not used, the nickel must be cleaned to remove contamination (11.2).

11.2 Immerse the flux in freshly prepared nickel flux cleaning solution for 50 s to 60 s, then rinse in running water for 2 min to 3 min. Pour flux onto paper towels to remove excess water. Place flux in sealable glass container, rinse with acetone and decant. To prevent new oxidation from forming, the flux may be stored under fresh acetone until used. (See Note 5.)

⁴ Known commercially as Anhydron.

⁵ Known commercially as Ascarite II.