



Designation: E 1937 – 04

Standard Test Method for Determination of Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique¹

This standard is issued under the fixed designation E 1937; 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 provides a procedure for the determination of nitrogen in titanium and titanium alloys in concentrations from 0.007 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in 7.8 and Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*²

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals

E 173 Practice For Conducting Interlaboratory Studies of Methods For Chemical Analysis of Metals

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Summary of Test Method

3.1 This test method is intended for use with automated, commercially available inert gas fusion analyzers.

3.2 The test sample, plus flux, is fused in a graphite crucible in a flowing helium gas stream at a temperature sufficient to release nitrogen. The nitrogen is swept by the helium gas stream into a thermal conductivity detector. The detector response is compared to that of calibration standards and the result is displayed as percent nitrogen.

3.3 In a typical instrument (Fig. 1) the sample gases are swept with helium through heated rare earth/copper oxide which converts CO to CO₂ and H₂ to H₂O. The CO₂ is absorbed on sodium hydroxide impregnated on clay, and the

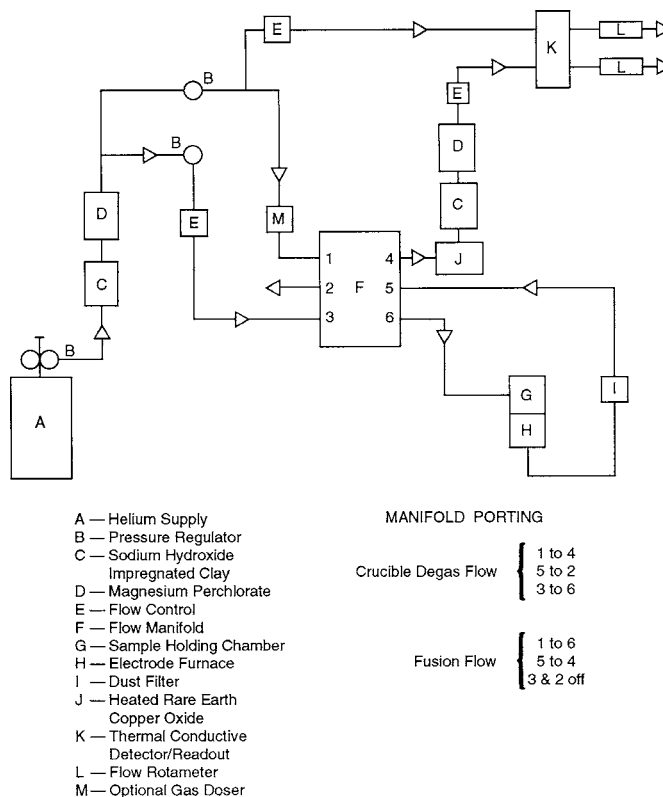


FIG. 1 Apparatus for Determination of Nitrogen by the Inert Gas Fusion-Thermal Conductivity Method

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.

4. Significance and Use

4.1 This test method is primarily intended as a referee method 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.

5. Interferences

5.1 The elements usually present in titanium and its alloys do not interfere.

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 Titanium, Zirconium, Tungsten, Molybdenum, Tantalum, Niobium, Hafnium, and Rhenium.

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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.

6. Apparatus

6.1 *Instrument*—The general features of the typical instrument are shown in Fig. 1.

6.2 *Graphite Crucibles*, made of high-purity graphite of the dimensions recommended by the instrument manufacturer.

6.3 *Flux*—Wire baskets consisting of platinum or high-purity nickel of dimensions that meet the requirements of the automatic sample drop, if present, on the instrument (Note 1).

NOTE 1—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 weight ratio of flux to sample. When using platinum as a flux, graphite powder should not be added to the crucible.

6.4 *Tweezers*—Six inch solvent and acid-resistant plastic.

7. Reagents

7.1 *Acetone*—Residue after evaporation must be < 0.0005 %.

7.2 *Graphite Powder*, of purity specified by the instrument manufacturer.

7.3 *Helium*, of purity and type specified by the instrument manufacturer.

7.4 *Magnesium Perchlorate, Anhydrous*—Used in the instrument to absorb water. Use the purity specified by the instrument manufacturer. (Known commercially as Anhydron.)

7.5 *Nickel Flux Cleaning Solution*—Prepare a fresh solution of nickel cleaning solution by combining 75 mL of acetic acid, 25 mL of HNO₃ and 2 mL of HCl.

7.6 *Rare Earth/Copper Oxide*—Reagent used in the instrument to oxidize CO to CO₂. Use the purity specified by the instrument manufacturer.

7.7 *Sodium Hydroxide on Clay*—Reagent used in some instruments to absorb CO₂. Use a purity specified by the instrument manufacturer. (Known commercially as Ascarite II.)

7.8 *Titanium Sample Pickle Solution*—Prepare a fresh solution of 3 parts 30 % H₂O₂ and 1 part 48 % HF. (WARNING: HF causes serious burns which may not be immediately painful; refer to the paragraph about HF in the Safety Precautions section of Practices E 50.)

8. Hazards

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

8.2 For precautions to be observed in the use of HF and other reagents in this test method, refer to Practices E 50.

9. Preparation of Apparatus

9.1 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.

9.2 Change the chemical traps and filters as required. Test the furnace and analyzer to ensure the absence of leaks. Make a minimum of two test runs using a sample as directed in 12.3

and 12.4 to condition the newly changed filters before attempting to calibrate the system or to determine the value of the blank.

10. Nickel Flux Preparation

10.1 Nickel is necessary to flux the titanium fusion reaction but contamination can be present on the surface of the nickel wire baskets that must be removed before use.

10.2 Immerse the flux in Nickel Flux Cleaning Solution for 50 to 60 s, then rise in running water for 2 to 3 min. Pour flux onto paper towels to remove excess water. Place flux in sealable glass container, rinse with acetone and decant. Replace with fresh acetone and store flux under acetone until use.

11. Sample Preparation

11.1 The optimum test sample is a pin approximately 1/8 in. in diameter and nominally weighing 0.12 to 0.15 g. Cut the sample to this approximate weight range.

11.2 Leach the test sample in the Titanium Sample Pickle Solution until the surface is clean. This will normally require approximately 5 s from the time of the initial vigorous reaction.

11.3 Immediately remove the reacting test sample with tweezers and rinse it twice with water and once with acetone and then air dry. This test sample should now weigh between 0.100 and 0.140 g.

11.4 All subsequent operations on the test sample and flux must be done without introducing contamination to either. Use only clean tweezers and never let the test sample or flux contact the analyst's skin. In the event this does happen, rinse the sample plus nickel basket with acetone and air dry before analysis.

12. Calibration

12.1 *Calibration Standards*—Select only titanium or titanium alloy standards. Select one containing approximately 0.02 % nitrogen. The accuracy of the test method is dependent upon the accuracy of the methods used to certify the nitrogen concentration of the certified reference materials, as well as upon the their homogeneity. Thus, wherever possible, standards used to confirm instrument calibration should be NIST Standard Reference Materials or other certified reference materials.

12.2 *Gas Dosing*—Automatic and manual gas dosing, recommended by some manufacturers, can be used to set up the instrument, but instrument response must be verified by calibration with titanium standards because of the fusion characteristics of the furnace/sample combination.

12.3 *Initial Adjustment of Measurement System*—Weigh a titanium standard to the nearest milligram, place it in a nickel basket and transfer it to an outgassed graphite crucible containing graphite powder (Note 2). Proceed as directed in 13.3 and 13.4. Repeat until an absence of drift is indicated. Using the average of the last three analyses, adjust the instrument signal to provide a reading within the range of the certified value of the standard. (Outgassing is accomplished automatically either as part of the continuous analysis cycle used with the automatic sample drop, or as the first step in a two-stage cycle associated with the manual addition of the sample to the crucible.)