



Designation: E 1409 – 97

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

This standard is issued under the fixed designation E 1409; 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 concentrations from 0.04 and 0.3 %.

1.2 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

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 Note 2.

2. Referenced Documents

2.1 ASTM Standards:

- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals²

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 under a flowing inert gas stream (Ar, He, or N₂) at a temperature sufficient to release oxygen. The released oxygen combines with carbon to form CO. Depending on instrument design, the CO is oxidized to CO₂ or left as CO and swept by the inert gas stream into either an infrared or thermal conductivity detector. The detector output is compared to that of calibration standards and the result is displayed as percent oxygen.

3.3 In a typical instrument whose detection is based upon thermal conductivity (see Fig. 1) the sample gases are swept

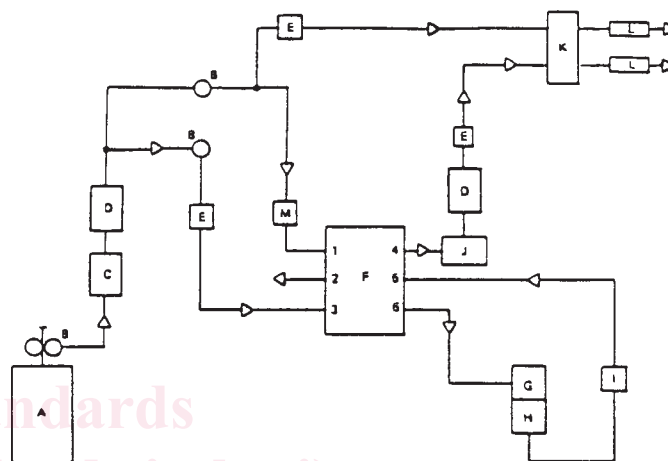


FIG. 1 Apparatus for Determination of Oxygen by the Inert Gas Fusion-Thermal Conductivity Method

with helium through heated rare earth/copper oxide that converts CO to CO₂. The water produced during fusion is absorbed by magnesium perchlorate and the remaining nitrogen and carbon dioxide are separated chromatographically. The oxygen, as CO₂, enters the measuring cell and the thermistor bridge output is integrated and processed to display percent oxygen.

3.4 In a typical instrument based on infrared detection (see Fig. 2) the test sample is fused in a stream of nitrogen or argon and the evolved gases are passed directly into an infrared cell through which infrared energy is transmitted. The CO in the sample gases absorbs some of the transmitted infrared energy and the decrease in energy reaching the detector is processed and displayed directly as percent oxygen. Some instruments utilize helium carrier gas and oxidize the CO to CO₂ before it is passed into the infrared cell.

4. Significance and Use

4.1 This test method is primarily intended as a referee 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.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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.

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² Annual Book of ASTM Standards, Vol 03.05.

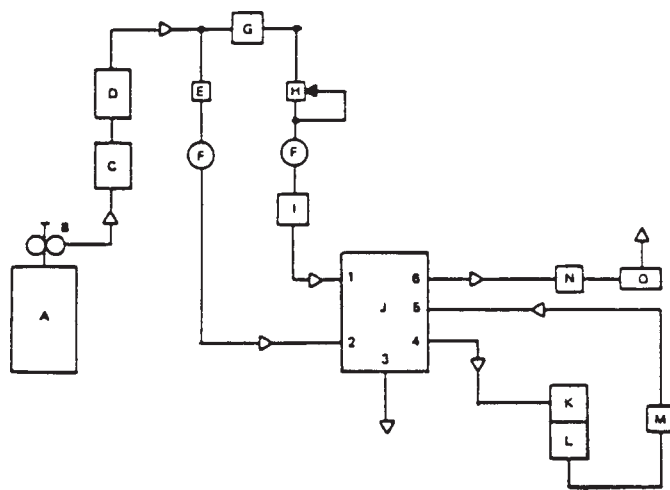


FIG. 2 Apparatus for Determination of Oxygen by the Inert Gas Fusion-Infrared Absorption Method

5. Interferences

5.1 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.

6. Apparatus

6.1 *Instrument*—The general features of the instruments are shown in Figs. 1 and 2.

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

6.3 *Flux*—Wire baskets must be made of high-purity nickel and the dimensions must meet the requirements of the automatic sample drop, if present, on the instrument. (See 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 ratio of flux to sample.

6.4 *Tweezers*—Six inches (152 millimetres), solvent and acid-resistant plastic (used during the sample preparation process).

7. Reagents

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

7.2 *Graphite Powder*—High-purity graphite powder specified by the instrument manufacturer.

7.3 *Inert Gas*—Use the purity and type (Ar, N₂, or He) specified by the instrument manufacturer.

7.4 *Magnesium Perchlorate, Anhydrous*³—Used in the instrument to absorb water. Use the purity specified by the instrument manufacturer.

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 some instruments to oxidize CO to CO₂ for thermal conductivity detection. Use the purity specified by the instrument manufacturer.

7.7 *Sodium Hydroxide on Clay*⁴—Reagent used in some instruments to absorb CO₂. Use a purity specified by the instrument manufacturer.

7.8 *Titanium Sample Pickle Solution*—Prepare a fresh solution of 3 parts 30 % H₂O₂ and 1 part 48 % HF. HNO₃ may be substituted for 30 % H₂O₂ (see Notes 2 and 3).

NOTE 2—**Warning:** HF causes serious burns that may not be immediately painful; refer to the paragraph about HF in the Safety Precautions Section of Practices E 50.

NOTE 3—In 1996, alternative sample preparation procedures were investigated by two laboratories, both active members of subcommittee E01.06, which has jurisdiction over E1409. The first alternative allows direct substitution of nitric acid for hydrogen peroxide when preparing titanium pickle solution and subsequent etching of the test specimen as specified in 10.2. The second entails removing surfaces of the sample specimen by filing with a file, thus eliminating the need to use any leaching procedure. The results of the interlaboratory study comparing these two preparation techniques is summarized in Table 1.

8. Preparation of Apparatus

8.1 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.

8.2 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.

³ Known commercially as Anhydrone.

⁴ Known commercially as Ascarite II.

TABLE 1 Results of Interlaboratory Study

Lab	Sample ID	Sample Desc.	Filed, % Oxygen	HF-HNO ₃ Etched, % Oxygen
Comparison of Filed versus HF-HNO ₃ Etched Sample Prep Procedures				
Lab 1	030	Ti-5A1-2.5Sn	0.160	0.164
Lab 2			0.161 ± 0.002, n = 3	0.165 ± 0.003, n = 3
Lab 1	035	CP-Ti	0.163	0.163
Lab 2			0.154 ± 0.001, n = 3	0.156 ± 0.001, n = 3
Lab 1	040	Ti-6A1-4V	0.164	0.160
Lab 2			0.156 ± 0.003, n = 3	0.155 ± 0.002, n = 3
Comparison of HF-H ₂ O ₂ Etched versus HF-HNO ₃ Etched Sample Prep Procedures				
Lab 2		CP-Ti	HF-H ₂ O ₂ Etched, % Oxygen	HF-HNO ₃ Etched, % Oxygen
			0.1412 ± 0.0025, n = 10	0.1422 ± 0.0016, n = 10
		Ti-6A1-4V	0.1425 ± 0.0026, n = 10	0.1418 ± 0.0021, n = 10