Designation: E1569 – 09

Standard Test Method for Determination of Oxygen in Tantalum Powder by Inert Gas Fusion Technique[1]

This standard is issued under the fixed designation E1569; 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 tantalum powder in concentrations from 0.05 % to 0.50 %.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety problems, 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.*

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test 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.

4.2 The sample, plus flux, is fused in a graphite crucible under a flowing inert gas stream at a temperature sufficient to release oxygen. The released oxygen combines with carbon from the crucible to form carbon monoxide (CO) that is swept by the inert gas stream into either an infrared or thermal conductivity detector. The detector output is compared to that of calibration reference materials and the result is displayed as percent oxygen.

4.3 In an instrument whose detection is based upon thermal conductivity (see Fig. 1), the sample gases are passed through heated rare earth copper oxide that converts CO to carbon dioxide (CO_2). The water produced during fusion is absorbed onto magnesium perchlorate and the remaining nitrogen and carbon dioxide are separated chromatographically. The nitrogen elutes first and can be measured (on a dual capability instrument) or disregarded. The oxygen, as CO_2, enters the measuring cell last and the thermistor bridge output is integrated and processed to display percent oxygen.

4.4 In a typical instrument based on infrared detection (see Fig. 2), the sample is fused in a stream of argon and 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.

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 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 this material do not interfere but there is some evidence to suggest that low-purity flux can act as a getter of the released oxygen.

7. Apparatus

7.1 *Capsules*—The capsules must be made of high-purity tin.

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

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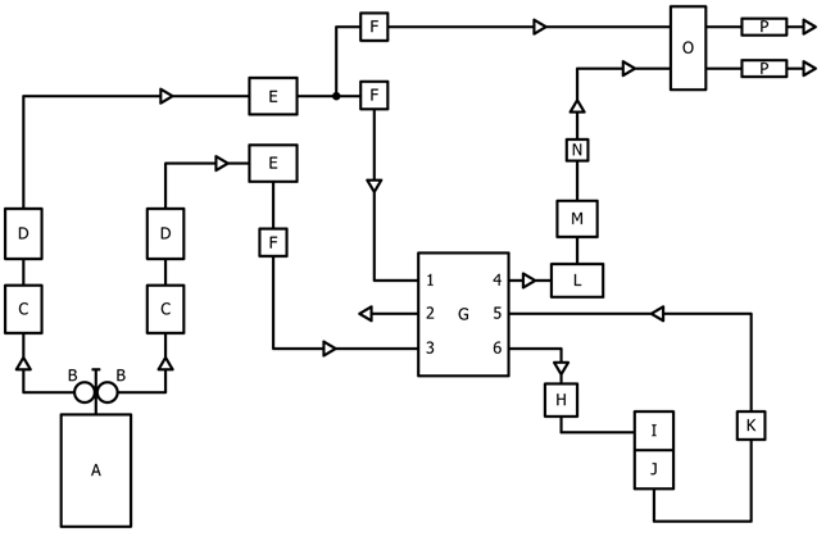
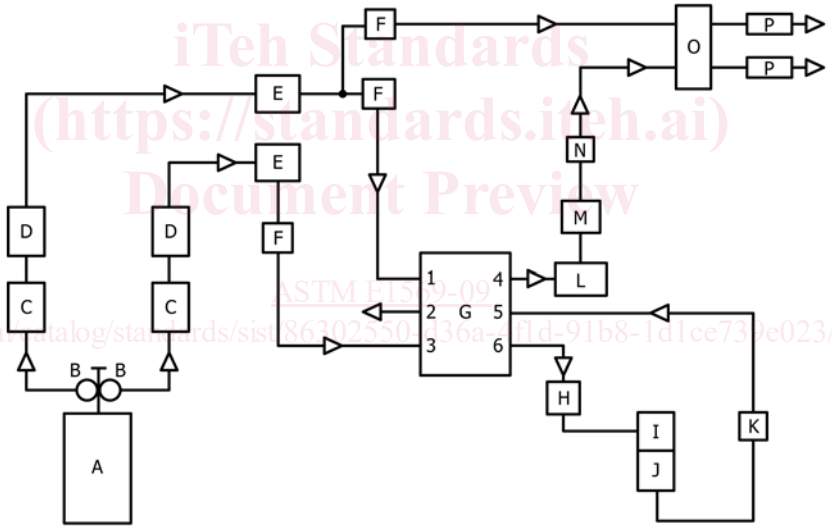
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



A— Inert Gas Supply
B—Pressure Regulator
C—Heated Copper
D—Sodium Hydroxide Impregnated Clay
E—Magnesium Perchlorate
F—Flow Control
G—Flow Manifold
H-Optional Gas Doser
I— Sample Holding Chamber
J— Electrode Furnace
K— Dust Filter
L— Heated Rare Earth Copper Oxide
M— Magnesium Perchlorate
N— Silica Column
O— Thermal Conductivity Detector/Readout
P— Flow Rotameter



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	Manifold Porting
Crucible Degas Flow	1 to 4 5 to 2 3 to 6
Fusion Flow	1 to 6 5 to 4 3 and 2 off

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