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**Designation: E 1569 – 93 (Reapproved 1998)**



**Designation: E 1569 – 03**

## Standard Test Method for Determination of Oxygen in Tantalum Powder<sup>1</sup>

This standard is issued under the fixed designation E 1569; 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.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E-1 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.

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### 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 *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:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>2</sup>

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>3</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>2</sup>

### 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 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 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.

3.3 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 CO<sub>2</sub>. 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<sub>2</sub>, enters the measuring cell last 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 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.

### 4. Significance and Use

4.1 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.

### 5. Interferences

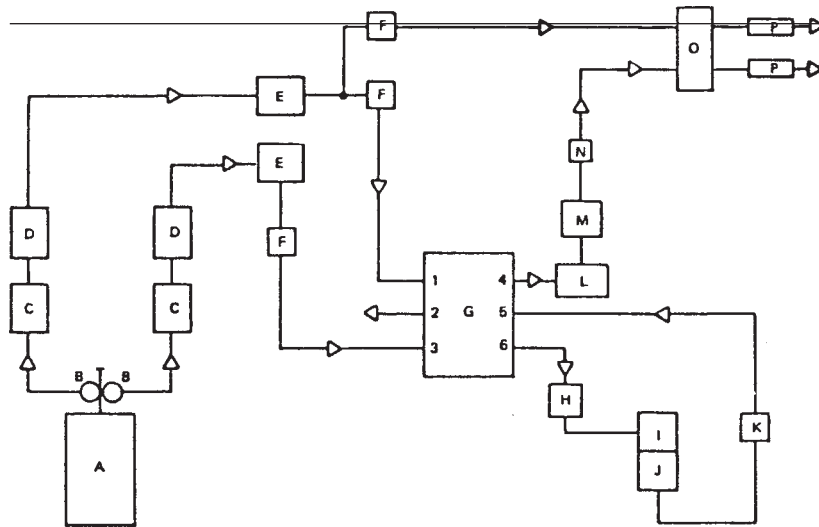
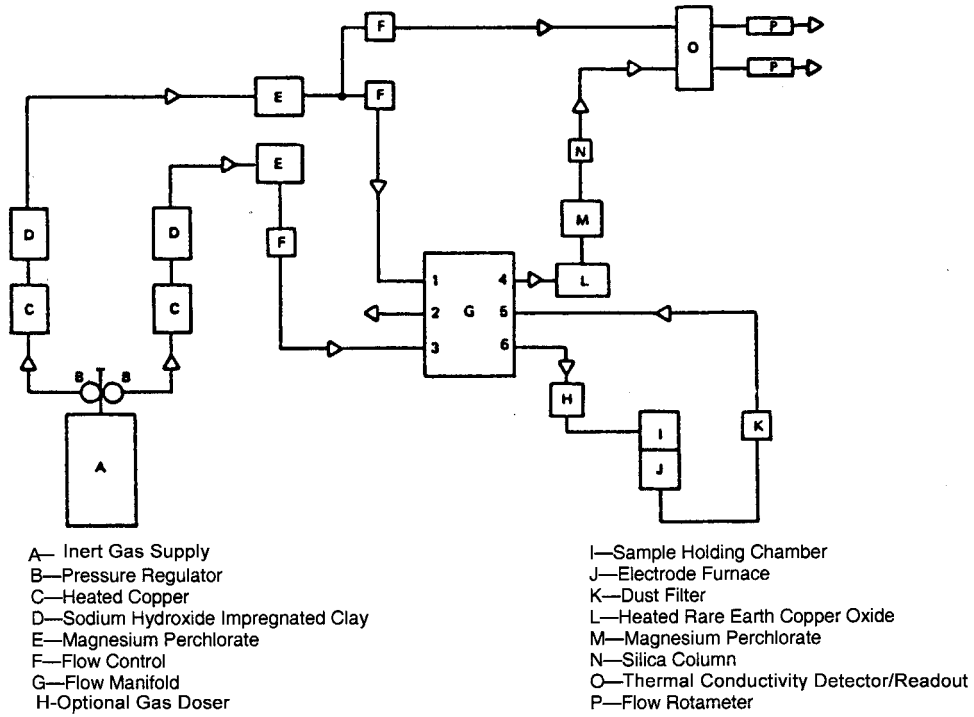
5.1 The elements usually present in this material do not interfere but there is some evidence to suggest that low-purity flux can act as a getter of the released oxygen.

### 6. Apparatus

6.1 *Fusion and Measurement Apparatus*—The general features of the instrument used in developing this test method are shown in Figs. 1 and 2.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

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



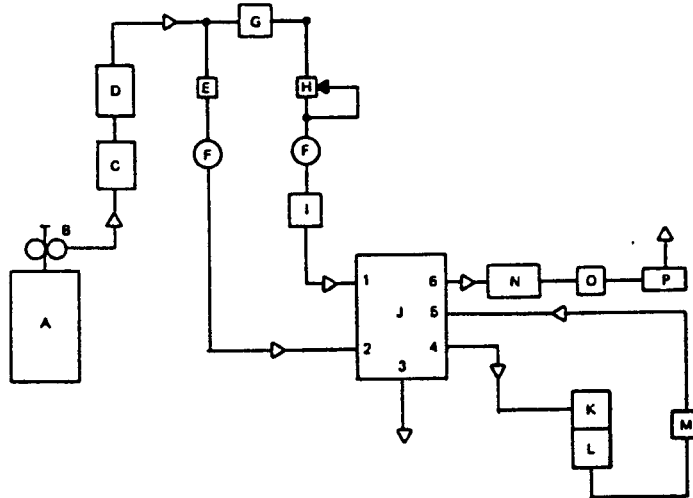
- A—Inert Gas Supply  
 B—Pressure Regulator  
 C—Heated Copper  
 D—Sodium Hydroxide Impregnated Clay  
 E—Magnesium Perchlorate  
 F—Flow Control  
 G—Flow Manifold  
 H—Optional Gas Doser  
 I—Sample Holding Chamber  
 J—Electrode Furnace  
 K—Dust Filter  
 L—Heated Rare Earth Copper Oxide  
 M—Magnesium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

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

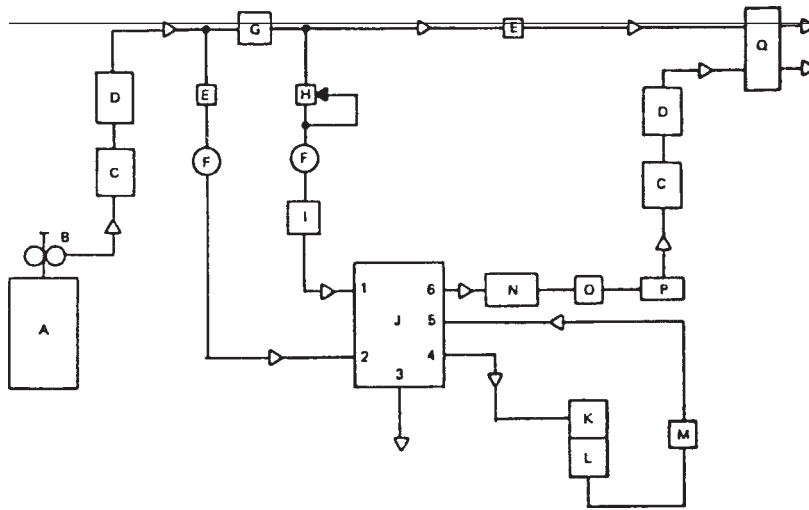
FIG. 1 Apparatus for the Determination of Oxygen by the Inert Gas Fusion—Thermal Conductivity Method

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



- A—Inert Gas Supply
- B—Pressure Regulator 2 Stage
- C—Sodium Hydroxide Impregnated Clay
- D—Magnesium Perchlorate
- E—Flow Restrictor
- F—Flow Meter
- G—Pressure Regulator
- H—Needle Valve
- I—Optional Gas Doser

- J—Flow Manifold
- K—Sample Holding Chamber
- L—Electrode Furnace
- M—Dust Filter
- N—Heated Rare Earth Copper Oxide
- O—Flow Control
- P—IR Detector/Readout



- A—Inert Gas Supply
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- N—Heated Rare Earth Copper Oxide
- O—Flow Control
- P—IR Detector/Readout

Manifold Porting

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

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

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

6.4 *Flux*—The foil or baskets must be made of high-purity nickel and in the case of the baskets, the dimensions must meet the requirements of the automatic sample drop, if present on the instrument.

6.5 *Tweezers*—Solvent and acid-resistant plastic.

## 7. Reagents

7.1 *Acetic Acid*—Reagent grade.

7.2 *Acetone*—Residue after evaporation must be less than 0.0005 %.

7.3 *Inert Gas*—Use the purity and type (helium or argon) specified by the instrument manufacturer.

7.4 *Magnesium Perchlorate, Anhydrous*<sup>4</sup> [ $\text{Mg}(\text{ClO}_4)_2$ ]<sup>2</sup>—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  $\text{HNO}_3$ , and 2 mL of  $\text{HCl}$ . Discard after use.

7.6 *Sodium Hydroxide on Clay*<sup>5</sup>—Reagent used to absorb  $\text{CO}_2$ . Use a purity specified by the instrument manufacturer.

## 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.

## 9. Flux Preparation

9.1 Immerse the flux in nickel flux cleaning solution (see 7.5) for 50 to 60 s, then rinse in running water for 2 to 3 min. Pour flux onto paper towels to remove excess water.

9.2 Place flux in sealable glass container, rinse with acetone, and decant. Replace with fresh acetone and store flux under acetone until used (see Notes 1 and 2).

NOTE 1—Nickel is necessary to flux the tantalum fusion reaction but it must be cleaned because significant oxidation can be present on the surface of the baskets or foil to interfere with the analysis.

NOTE 2—The fluxing agent must be of proper size to be introduced through the sample drop mechanism and into the graphite crucible.

## 10. Sample Preparation

10.1 During the weighing and introduction of the sample powder into the tin capsule and nickel basket or foil the entire operation must be accomplished using clean tweezers only. Never touch the flux/sample package with the fingers.

## 11. Calibration

11.1 Selection of reference materials is currently limited to solid steel pins with oxygen content ranging from approximately 0.01 to 0.05 %. The analyst must be aware that the composition of the reference materials is not that of the unknowns.

11.2 *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 with standards because of the fusion characteristics of the furnace/sample combination.

11.3 *Initial Adjustment of Measurement System*—Weigh a steel pin reference material to the nearest milligram and transfer it to an outgassed graphite crucible (some instruments allow for the use of an automatic sample drop port). Proceed as directed in 12.3 and 12.4. Repeat 12.3 and 12.4 until an absence of drift is indicated. Using the last three analyses, adjust the instrument signal to provide a reading within the range of the certified value of the reference material. (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.)

11.4 *Determination of Flux and Crucible Blank*—Proceed as directed in 12.3 and 12.4 for the determination of the entire reagent blank which consists of the type of crucibles, flux, and capsules which will be used in all subsequent analyses. The capsule is folded and the excess air is squeezed out. The capsule is placed within the nickel basket and the package is weighed to the nearest 1 mg. If nickel foil is used, the weight of the foil must be the same as that used for the specimen analysis and the foil is wrapped around the capsule before weighing. Determine the average blank of three individual runs establishing that the blank is low, less than 3  $\mu\text{g}$  of oxygen, and consistent. Enter this value into the appropriate mechanism of the analyzer. Problems with inconsistent or high blank values, generally indicative of reagent depletion, must be corrected before the analysis can be continued. If the unit does not have this provision for automatic blank compensation, then the blank value must be manually subtracted from the total result prior to any other calculation.

<sup>4</sup> Known commercially as Anhydron.

<sup>5</sup> Known commercially as Ascarite II.

11.5 *Calibration*—Follow the calibration procedure recommended by the manufacturer using a steel pin reference material of a concentration different from that of 11.3. Analyze specimens until the maximum range between readings is 0.001 %. Confirm the calibration by analyzing an additional specimen after the calibration procedure is complete. The results should fall within the maximum allowable limit of the reference material. If not, refer to the manufacturers instructions for checking linearity.

## 12. Procedure

12.1 Assemble the apparatus, calibrate it, and test the performance as directed in Sections 8 and 11.

12.2 Transfer 0.08 to 0.15 g of sample powder (the weight of nickel must exceed the weight of the sample by at least a factor of ten) to a tin capsule. Fold the capsule over, squeezing the excess air out, and insert into a nickel basket, or wrap in nickel foil.

12.3 Place the specimen into the automatic sample drop port or into an outgassed crucible.

12.4 Place the crucible on the furnace pedestal, raise the mechanism, and start the analysis cycle. Refer to the specific instructions for your instrument regarding operation, entry of sample weight, and blank value.

## 13. Calculation

13.1 Refer to the manufacturer’s instructions to ensure that all essential variables in the analyses have been accounted for. The output of most modern fusion equipment is given directly in percent oxygen so that post-analysis calculations are normally not required.

## 14. Precision and Bias <sup>6</sup>

14.1 *Precision*—Ten laboratories cooperated in testing four different samples, and the data obtained are presented in Table 1. The testing and statistical analyses were performed in accordance with the provisions of Practice E 691.

14.2 *Bias*—No bias can be evaluated, since the samples used for the round robin had no established values assigned to them.

## 15. Keywords

15.1 oxygen content; tantalum; tantalum powder

<sup>6</sup> Supporting data have been filed at ASTM Headquarters. Request RR:E01-1008.

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**TABLE 1 Statistical Information—Oxygen in Tantalum Powder**

Test Material	Oxygen Found, ppm	Repeatability, <i>r</i>	Reproducibility, <i>R</i>
A	1420	81	83
B	2079	78	136
C	2490	89	151
D	2990	118	144