



Designation: E159 – 17

Standard Test Method for Loss of Mass in a Reducing Gas Atmosphere for Cobalt, Copper, Tungsten, and Iron Powders (Hydrogen Loss)¹

This standard is issued under the fixed designation E159; 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 the mass of hydrogen-reducible constituents in the following metal powders: cobalt, copper, iron, and tungsten.

1.2 This test method is useful for cobalt, copper, and iron powders in the range from 0.05 to 3.0 % oxygen, and for tungsten powder in the range from 0.01 to 0.50 % oxygen.

1.3 This test method does not measure the oxygen contained in oxides such as silicon oxide (SiO_2), aluminum oxide (Al_2O_3), magnesium oxide (MgO), calcium oxide (CaO), titanium dioxide (TiO_2), etc. that are not reduced by hydrogen at the test temperatures.

1.4 For total oxygen content, vacuum or inert gas fusion methods are available (see Test Methods E1019).

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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.*

1.7 *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

B215 Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

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

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology B243. Additional descriptive information on powder metallurgy is available in the Related Material section of Vol 02.05 of the *Annual Book of ASTM Standards*.

4. Summary of Test Method

4.1 This test method consists of subjecting a test portion of powder to the action of a hydrogen-containing gas under standard conditions of temperature and time and measuring the resulting loss of mass.

5. Significance and Use

5.1 The oxygen content of a powder affects both its green and sintered properties.

5.2 Hydrogen loss is a term widely used in the powder metallurgy industry even though the measurement represents an approximate oxygen content of the powder.

5.3 Oxygen is the most common hydrogen-reducible constituent of metal powders, and this procedure may be used as a measure of oxygen, reducible under the test conditions, if other interfering elements are absent.

6. Interferences

6.1 If carbon or sulfur is present, or both, are present, they will be largely removed in the test. Their loss in mass is included in the total loss in mass measurement and must be subtracted from the total mass loss.

6.2 If metals or compounds are present that vaporize at the test temperature (such as cadmium, lead, zinc, etc.), their effect is included in the loss of mass measurement and must be subtracted from the total mass loss.

¹ This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.

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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 Summary of Changes section appears at the end of this standard

6.3 If some components are present that are oxidized or hydrided during the test, there is a gain in mass that must be added to the total mass loss.

7. Apparatus

7.1 *Furnace*, capable of operating at the prescribed temperature.

7.2 *Temperature Control*, capable of maintaining temperatures to $\pm 15^\circ\text{C}$.

7.3 *Gastight Ceramic or Metallic Combustion Tube*.

7.4 *Flow Meter*, to measure flow of reducing gas.

7.5 *Combustion Boat*, composed of alundum, quartz, or nickel, depending on the test conditions. The boat shall be of such dimensions, for example 75 mm long and 12 mm wide, that the thickness of powder, when uniformly distributed, does not exceed 3 mm.

7.6 *Balance*, suitable for determining mass to the nearest 0.0001 g.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Dissociated Ammonia*, having a dew point lower than -40°C . May be used interchangeably with the hydrogen specified in section 8.3.

8.3 *Hydrogen*, having an oxygen content less than 20 ppm and a dew point lower than -40°C .

8.4 *Nitrogen*, having an oxygen content less than 20 ppm and a dew point lower than -40°C .

9. Test Portion

9.1 Obtain the test portion in accordance with the procedure described in Practices B215.

9.2 The test portion shall weigh approximately 5 g.

10. Procedure

10.1 Spread the test portion to a uniform depth in a combustion boat that has been preconditioned to a constant mass and weighed to the nearest 0.0001 g. The depth of powder in the boat should be approximately 3 mm. Weigh the boat and specimen to the nearest 0.0001 g.

10.2 Pass the nitrogen through the combustion tube for a period of at least 1 min before inserting the combustion boat at the center of the zone of uniform temperature of the furnace.

10.2.1 For cobalt, the temperature is 875°C .

10.2.2 For copper, the temperature is 875°C .

10.2.3 For iron, the temperature is 1120°C .

10.2.4 For tungsten, the temperature is 1120°C .

10.3 Ensure that the pilot flames at both ends of the tube furnace are lit and then start the flow of reducing gas and stop the flow of nitrogen. Record the time when the reducing gas is introduced. The flow rate of the reducing gas should be 10 to 30 mL/min.

10.4 Maintain a positive flow of reducing gas through the system for the time of reduction during which the temperature of the furnace shall be held within $\pm 15^\circ\text{C}$ of the target reduction temperature.

10.4.1 For cobalt, the time shall be 30 min.

10.4.2 For copper, the time shall be 30 min.

10.4.3 For iron, the time shall be 60 min.

10.4.4 For tungsten, the time shall be 60 min.

10.5 At the end of the prescribed time, discontinue the flow of reducing gas and restart the flow of nitrogen into the tube.

10.6 After nitrogen has been flowing through the combustion tube for at least 1 min, open the tube and withdraw the boat under nitrogen atmosphere to the cooler part of the combustion tube.

10.7 Allow the specimen to cool in the nitrogen atmosphere for 15 to 30 min, then remove the specimen from the tube and permit it to cool to room temperature in a desiccator.

10.8 When the boat has cooled to room temperature, reweigh it to the nearest 0.0001 g.

11. Calculation

11.1 Calculate the mass loss as follows:

$$\text{Loss in mass, \%} = \frac{A - B}{A - C} \times 100 \quad (1)$$

where:

A = original mass of boat and powder specimen, g;

B = mass of boat and powder specimen after reduction, g; and

C = original mass of the boat, g.

11.2 If the loss of mass value calculated in 11.1 is to be used as the oxygen content of the powder, correct the value obtained in accordance with 6.1 – 6.3.

12. Report

12.1 Results shall be reported as percent loss in mass to the nearest 0.01 %.

13. Precision and Bias

13.1 A statement of precision provides guidelines as to the kind of variability that can be expected between test results when the method is used in one or more reasonably competent laboratories.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

13.2 The precision of this test method has not been determined by a statistically valid interlaboratory test because of the limited number of participating laboratories (less than six).

13.3 The following precision data were developed using the procedures contained in Test Method E159 from an interlaboratory test conducted by four laboratories in 1994. The percent loss in mass in hydrogen was determined for four samples, a cobalt powder, a copper powder, an iron powder, and a tungsten powder. Except for the use of only four laboratories, Practice E691 was followed for the design and analysis of the data. The details are given in an ASTM research report.

13.4 The precision information given below is for the results. The results were obtained from the running of three replicates by each lab on each sample.

	Cobalt	Copper	Iron	Tungsten
Average, %	0.45	0.15	0.22	0.47
<i>r</i> , %	0.03	0.03	0.03	0.09
<i>R</i> , %	0.12	0.03	0.07	0.11

13.5 Repeatability, *r*, is a measure of the variability between test results obtained within a single laboratory in the shortest practical period of time by a single operator with a specific set of test apparatus using test specimens taken at random from a single quantity of homogeneous material.

13.6 Reproducibility, *R*, is a measure of the variability between test results obtained in different laboratories using test specimens taken at random from a single quantity of homogeneous material.

13.7 Bias cannot be stated since there is no certified reference material.

14. Keywords

14.1 chemical analysis; cobalt powder; copper powder; hydrogen loss; iron powder; metal powders; oxygen content; tungsten powder

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (E159 - 10) that may impact the use of this standard. (April 1, 2017)

- (1) Replaced “hydrogen” with “reducing gas” throughout the standard.
- (2) Replaced “specimen” with “portion” throughout the standard.

- (3) Replaced “sample” with “test portion” in 4.1.
- (4) Subsection 10.3—Added reducing gas flow rate.
- (5) Subsection 10.4—Clarified the furnace temperature.

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