



Designation: E194 – 20

Standard Test Method for Acid-Insoluble Content of Copper and Iron Powders¹

This standard is issued under the fixed designation E194; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method² covers the determination of the mineral-acid-insoluble matter content of copper and iron powders in amounts under 1.0 %.

1.2 *Units*—With the exception of the values for density and the mass used to determine density, for which the use of the gram per cubic centimetre (g/cm^3) and gram (g) units is the longstanding industry practice, the values stated in SI units are to be regarded as standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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:*³

[B215 Practices for Sampling Metal Powders](#)

[E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

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 Based on the method developed by the Metal Powder Association (now the Metal Powder Producers Association of the Metal Powder Industries Federation) and described in MPIF Standard 06, "Determination of Acid Insoluble Matter in Iron and Copper Powders," which is a standard of the MPIF.

³ 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.

3. Summary of Test Method

3.1 The sample is dissolved in the appropriate acid: nitric acid (HNO_3) for copper, hydrochloric acid (HCl) for iron. The insoluble matter is filtered out and ignited in a furnace at 980 °C for 1 h.

4. Significance and Use

4.1 The purpose of this test method is to determine the amount of gangue, refractory, inert, etc. materials, that may adversely affect compacting tools and sintered properties of components formed from copper and iron powders.

4.2 The insoluble matter consists of those nonmetallic substances that do not dissolve in the mineral acid used to dissolve the metal. In copper powder, which is treated with nitric acid, the acid-insoluble matter includes silica, insoluble silicates, alumina, clays, and other refractory materials that may be introduced either as impurities in the raw material or from the furnace lining, fuel, etc.; lead sulfate may also be present. In iron powder, which is treated with hydrochloric acid, the insoluble matter may include carbides in addition to the substances listed above. The test method excludes insoluble material that is volatile at the ignition temperature specified.

5. Interferences

5.1 Any metallic tin present in the copper powder will be converted into the insoluble tin oxide by the nitric acid treatment; in such cases, provision shall be made for the determination of tin oxide and the appropriate correction applied.

6. Apparatus

6.1 Apparatus and reagents shall conform to the requirements prescribed in Practices E50.

6.2 *Hot Plate.*

6.3 *Muffle Furnace*, capable of operating at 980 °C.

6.4 *Casseroles (non-metallic)*, 250 mL and 750 mL.

6.5 *Glass Funnel.*

6.6 *Quart or Porcelain Crucible.*

6.7 *Desiccator.*

6.8 *Analytical Balance*, readable to 0.0001 g, with a minimum capacity of 100 g.

*A Summary of Changes section appears at the end of this standard

6.9 *Filter Paper*, Whatman No. 541 or one of equivalent pore size and ash content.

6.10 *Vapor Collection System*, suitable to provide adequate operator protection from chemical vapors resulting from the acid digestion steps and muffle furnace ignition steps.

7. Reagents

- 7.1 *Hydrochloric Acid* HCl (1:1).
- 7.2 *Hydrochloric Acid* HCl (1:25).
- 7.3 *Nitric Acid* (HNO₃).
- 7.4 *Nitric Acid* HNO₃ (1:1).
- 7.5 *Ammonium Iodide* (NH₄I).
- 7.6 *Potassium Thiocyanate* (5 %).

8. Sampling

8.1 The metal powder shall be sampled in accordance with Practices **B215**.

8.2 Store the test sample in a tightly stoppered bottle to protect it from moisture which promotes oxidation of copper and iron.

8.3 The test portion shall be approximately 5 g of metal powder.

COPPER POWDER

9. Procedure

9.1 Determine the mass of the test portion to 0.0001 g and transfer to a 250 mL covered casserole.

NOTE 1—Some operators report better reproducibility when increasing the metal powder sample size to 10 g. Nevertheless, the precision statement listed in Section 14 was based on 5 g samples.

9.2 Add 100 mL of HNO₃ (1:1) and let stand at room temperature until the reaction is complete.

9.3 Place the casserole on a hot plate and boil until the volume is reduced to 50 mL.

9.4 Cool, dilute with distilled water to about 100 mL, and bring to a boil. Maintain boiling for about 1 min.

9.5 Filter the hot solution, and wash with hot distilled water until all traces of blue color (copper salts) disappear.

9.6 Prepare a quartz or porcelain crucible by pre-heating for 40 min in air at 980 °C and then cool it in a desiccator.

9.7 Weigh the crucible and determine the mass to 0.0001 g.

9.8 Transfer the filter paper and residue to the crucible.

9.9 Dry in an oven at 105 °C for at least 10 min, and then ignite in a furnace at 980 °C for 1 h.

9.10 Cool in a desiccator and reweigh to determine the mass to 0.0001 g. The difference in mass is the insoluble matter. Reserve the residue.

NOTE 2—If the ignited residue reserved from 9.2 is suspected to contain tin oxide, add 5 g of NH₄I to the crucible. Reheat the crucible and contents in air with a bunsen burner to a dull-red temperature or place in a furnace at 600 °C minimum for 15 min or until all fumes have been dispelled. Remove the crucible from the heat and cool. Add 2 to 3 mL of HNO₃,

evaporate to dryness, ignite, and weigh to determine the mass to 0.0001 g. Repeat the treatment with NH₄I and HNO₃ until constant mass is obtained. The loss in mass represents tin oxide. Subtract this loss in mass from the mass of insoluble matter determined in 9.10 to calculate the insoluble fraction that is free of tin oxide.

10. Calculation

10.1 Calculate the percentage of insoluble matter as follows:

$$\text{Insoluble matter, percent} = [(A - B)/C] \times 100$$

where:

A = insoluble matter, g,

B = correction for grams of tin oxide, if present (Note 2), and

C = sample used, g.

IRON POWDER

11. Procedure

11.1 Determine the mass of the test portion to 0.0001 g and transfer to a 750 mL covered casserole.

NOTE 3—Some operators report better reproducibility when increasing the metal powder sample size to 10 g. Nevertheless, the precision statement listed in Section 14 was based on 5 g samples.

11.2 With caution, add 100 mL of HCl (1:1) (Note 4), and let stand at room temperature until the reaction is complete.

11.3 Heat the solution to boiling on a hot plate. Maintain boiling for about 1 min. Then add 150 mL of water, and reheat to boiling and maintain for about 1 min.

11.4 Filter the hot solution, and wash the residue alternately with hot HCl (1:25) and hot distilled water, six times with each, to ensure the removal of all iron salts. The absence of iron salts in the filtrate may be checked by the addition of a 5 % solution of potassium thiocyanate. If iron salts are present, the filtrate will turn blood-red.

NOTE 4—If it is desired to exclude carbides from the reported insoluble matter, add 20 mL of HNO₃ to the HCl (1:1).

11.5 Prepare a quartz or porcelain crucible by pre-heating for 40 min in air at 980 °C and then cool it in a desiccator.

11.6 Weigh the crucible to determine the mass to 0.0001 g.

11.7 Transfer the filter paper and residue to the crucible.

11.8 Dry in an oven at 105 °C for at least 10 min, and then ignite in a furnace at 980 °C for 1 h.

11.9 Cool in a desiccator and reweigh to determine the mass to 0.0001 g. The difference in mass is the insoluble matter.

12. Calculation

12.1 Calculate the percentage of insoluble matter as follows:

$$\text{Insoluble matter, percent} = [A/B] \times 100$$

where:

A = insoluble matter, g, and

B = sample used, g.