Designation: E194 – 10 (Reapproved 2015)

Standard Test Method for Acid-Insoluble Content of Copper and Iron Powders[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method[2] covers the determination of the mineral-acid-insoluble matter content of copper and iron powders in amounts under 1.0 %.

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:*[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

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,* having a sensitivity of 0.1 mg.

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* (HNO_3).

7.4 *Nitric Acid* HNO_3 (1:1).

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[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.

7.5 *Ammonium Iodide* (NH_4I).

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.

COPPER POWDER

9. Procedure

9.1 Transfer 5 g of the sample, weighed to the nearest 0.0001 g, 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 HNO_3 (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 to the nearest 0.0001 g.

9.8 Transfer the filter paper and residue to the crucible.

9.9 Dry, and then ignite in a furnace at 980 °C for 1 h.

9.10 Cool in a desiccator and reweigh to the nearest 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 NH_4I 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 HNO_3, evaporate to dryness, ignite and weigh to the nearest 0.0001 g. Repeat the treatment with NH_4I and HNO_3 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 Transfer 5 g of the sample, weighed to the nearest 0.0001 g, 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 HNO_3 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 dessicator.

11.6 Weigh the crucible to the nearest 0.0001 g.

11.7 Transfer the filter paper and residue to the crucible.

11.8 Dry, and then ignite in a furnace at 980 °C for 1 h.

11.9 Cool in a desiccator and reweigh to the nearest 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.

13. Report

13.1 Report the total insoluble matter as a percentage to the nearest 0.01 %.

14. Precision and Bias

14.1 *Precision*—The following precision data were developed using the procedures contained in Test Method E194 from an interlaboratory study that performed six sets of tests. The percent insoluble was determined for four samples: a –325 mesh iron, a –60 mesh iron, a –325 mesh copper, and a –60 mesh copper. The different particle sizes were used to determine if there were any effects on the precision of testing based on differences in particle size distribution. Practice E691 was followed for the design and analysis of the data; the details are given in an ASTM Research Report.[4]

14.1.1 The precision information given below is for the comparison of two test results. The results were obtained from the running of three replicates in each test on each sample.

	–325 Iron	–60 Iron	–325 Copper	–60 Copper
Average, %	0.29	0.08	0.11	0.09
S_r, %	0.022	0.012	0.013	0.019
S_R, %	0.051	0.025	0.036	0.063
r, %	0.06	0.03	0.04	0.05
R, %	0.14	0.07	0.10	0.18

14.1.2 Duplicate results from the same laboratory should be considered acceptable at the 95 % confidence level unless they differ by more than *r*, the repeatability interval.

14.1.3 Duplicate results from two different laboratories should be considered acceptable at the 95 % confidence level unless they differ by more than *R*, the reproducibility interval.

14.2 *Bias*—No information can be presented on the bias of the procedure in Test Method E194 for measuring the acid insoluble content of copper and iron powders because no material having an accepted reference value is available.

15. Keywords

15.1 acid insolubles; copper powder; iron powder

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1009.

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