METAL POWDER INDUSTRIES FEDERATION

Method for

Determination of Acid Insoluble Matter in Iron and Copper Powders

MPIF Standard 06
Issued 1948
Revised 1954, 1964, 1974, 1983, 1988, 1997, 2004, 2010

STANDARD 06

1. SCOPE

1.1 This standard describes an analytical method for determination of the acid insolubles content of elemental iron and copper metal powders.

1.2 The acid insolubles referred to are compounds that are not completely soluble in ordinary mineral acids. These are generally considered to be silica and silicates, carbides, alumina, clays, other refractory oxides, or difficult-to-dissolve oxides that may be present in the raw material from which the powders are made or were introduced in their manufacturing process. This method excludes insoluble material that is volatile or combustible at the specified ignition temperature.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound units in parentheses were converted in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 *This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. APPARATUS AND REAGENTS

2.1 **Apparatus**

2.1.1 Hot Plate.

2.1.2 Muffle furnace capable of operating at 980°C (1800°F).

2.1.3 250 and 750 mL non-metallic casserole or beaker.

2.1.4 Glass funnel.

2.1.5 Quartz or porcelain crucible.

2.1.6 Desiccator.

2.1.7 Analytical balance having a sensitivity of 0.0001 g.

2.1.8 Fine, ash-less filter paper (Whatman 541 or one of equivalent pore size and ash content.

2.1.9 Vapor collection system suitable to provide adequate operator protection from chemical vapors resulting from the acid digestion steps and muffle furnace ignition steps.

2.2 **Reagents to use in the iron powder procedure.**

2.2.1 Hydrochloric acid solution (1:1) - Mix 1 volume of concentrated HCl with 1 volume of water.

2.2.2 Hydrochloric acid solution (1:25) - Mix 1 volume of concentrated HCl with 25 volumes of water.

2.2.3 Nitric acid (concentrated), HNO_3.

2.2.4 Potassium thiocyanate, KSCN (5%).

2.3 **Reagents to use in the copper powder procedure.**

2.3.1 Nitric acid solution (1:1) - Mix 1 volume of concentrated HNO_3 with 1 volume of water.

2.3.2 Ammonium iodide, NH_4I

3. TEST SPECIMEN

3.1 The test specimen shall be 5 g of metal powder, obtained in accordance with MPIF Standard 01 "Sampling Finished Lots of Metal Powders," weighed to the nearest 0.0001 g.

NOTE 1—Some operators report better reproducibility of results when increasing the metal powder sample size to 10 g. However, the statistical analysis that supports the present method was based on 5 g samples.

4. PROCEDURE

4.1 **Iron Powder**

4.1.1 Place the test specimen, weighed to the nearest 0.0001g in a 750 mL casserole. Add 100 mL of HCl (1:1) with caution and cover with a watch glass. Allow the solution to stand at room temperature until the reaction is complete. Place the casserole on the hot plate. Heat to boiling point and hold until reaction ceases. Add 150 mL of distilled water, reheat to boiling point and maintain for about 1 minute. Filter the hot solution and wash the residue on the filter paper alternately with hot distilled water, and hot HCl (1:25) six times with each to ensure that all iron salts are dissolved. 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 2—If it is desired to exclude carbides as part of the insoluble matter, add 20 mL of concentrated HNO_3 to the HCl (1:1) and proceed as indicated above. This will prevent the inclusion of combined carbon with the insoluble matter being determined.

4.1.2 Prepare a crucible by preheating in a muffle furnace for 40 minutes in air at 980 °C (1800 °F) and then cool it in a desiccator. Weigh the crucible to the nearest 0.0001 g and place the filter paper and residue in it. Dry and place in a muffle furnace at 980 °C (1800 °F). Burn off the paper for 1 hour. Cool in the desiccator. Re-weigh the crucible and ash to the nearest 0.0001 g.

4.2 **Copper Powder**

4.2.1 Place the test specimen weighed to the nearest 0.0001 g in a 250 mL casserole. Add 100 mL of HNO_3 (1:1) and cover with a watch glass. Allow the solution to stand at room temperature until the reaction is complete. Place the casserole on the hot plate, and heat to boiling point. Boil to half volume. Add distilled water to bring the volume to approximately 100 mL. Heat the solution to boiling point and maintain boiling for about 1 minute. Filter the hot solution and wash the filter with hot distilled water until all traces of blue color (copper salts) disappear.

4.2.2 Prepare a crucible by preheating for 40 minutes in air at 980 °C (1800 °F) and then cool it in a desiccator. Weigh the crucible to the nearest 0.0001 g and place the filter paper and residue in it. Dry and place in a muffle furnace at 980 °C (1800 °F) for 1 hour. Cool in the desiccator. Weigh crucible and ash to the nearest 0.0001 g.

NOTE 3—If the ignited residue contains tin oxide, add 5 g of NH_4I to the prepared crucible and heat in a furnace, in air, at 600 °C (1100 °F) for 15 minutes. After the fumes have disappeared, remove the crucible and cool. Add 2 to 3 mL of concentrated nitric acid; evaporate to dryness, ignite and weigh. Repeat this procedure 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 section 4.2.2 to calculate an insoluble fraction that is free of tin oxide.

5. CALCULATIONS

5.1 Calculate the percentage of total insoluble matter as follows:

$$\text{Total insoluble matter, \%} = \left(\frac{A-B}{C}\right) 100$$

where:
A = mass of crucible and ash after ignition, in grams,
B = mass of crucible, in grams, and
C = grams of sample used.

6. REPORT

6.1 Total insoluble matter as a percentage to the nearest 0.01%

7. PRECISION

7.1 The following precision data were developed using the procedures contained in MPIF Standard 06 from an interlaboratory study that performed six sets of tests. The % 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. ASTM Practice E691 was followed for the design and analysis of the data; the details are given in MPPA Research Report No. MPPA-R-06-98.

7.2 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.

INSOLUBLE CONTENT OF IRON AND COPPER POWDERS PRECISION STATISTICS

		-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

7.3 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.

7.4 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.

7.5 Data Source – Data used in developing this statement was contributed by the members of ASTM Committee B09 on Metal Powders and Metal Powder Products and was used for the determination of Precision with the permission of ASTM.

APPENDIX

A1. COMPARABLE STANDARDS
ASTM E194
ISO 4496