Designation: E247 – 01 (Reapproved 2015)$^{\varepsilon 1}$

# Standard Test Method for Determination of Silica in Manganese Ores, Iron Ores, and Related Materials by Gravimetry[1]

This standard is issued under the fixed designation E247; 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.

$\varepsilon^1$ NOTE—Editorially corrected 1.1 and Table 1 in November 2015.

## 1. Scope

1.1 This test method covers the determination of silica in iron ores, iron ore concentrates and agglomerates, and manganese ore in the range from 0.5 % to 15 %.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

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

E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition and Physical Properties

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

## 4. Summary of Test Method

4.1 The sample is fused with sodium peroxide in a zirconium crucible. The melt is leached with water and dissolved in HCl. Silica is separated by double dehydration with $HClO_4$. The two precipitates are combined, ignited, and weighed. The silica is volatilized by treatment with HF and $H_2SO_4$ and the residue weighed.

## 5. Significance and Use

5.1 This test method is intended to be used for compliance with compositional specifications for silica content. It is assumed that all who use these procedures will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed such as those described in Guide E882.

## 6. Interferences

6.1 This test method is written for iron and manganese ores containing less than 0.25 % of fluorine. None of the elements normally found in iron ores or in manganese ores interfere with this test method.

## 7. Apparatus

7.1 *Zirconium Crucible* (50-mL capacity).

7.2 *Platinum Filter Cone.*

## 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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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.

such specification are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

8.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated HCl.

8.4 *Hydrochloric Acid* (1 + 49)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 49 volumes of water.

8.5 *Hydrofluoric Acid* (48 %)—Concentrated HF.

8.6 *Perchloric Acid* (70 %)—$HClO_4$.

8.7 *Sodium Peroxide Powder*—$Na_2O_2$.

8.8 *Sulfuric Acid* (1 + 1)—Carefully pour one volume of concentrated $H_2SO_4$, sp gr 1.84, into one volume of water.

## 9. Hazards

9.1 For precautions to be observed in this method, refer to Practices E50.

## 10. Sampling and Sample Preparation

10.1 *Sampling*—The gross sample shall be collected and prepared in accordance with Practice E877.

10.2 *Sample Preparation*—The laboratory sample shall be pulverized to pass a No. 100 (150-μm) sieve.

NOTE 1—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75-μm) sieve.

10.3 *Sample Weight*—Weigh approximately (within ± 25 mg) an amount of test sample specified as following:

| Content of Silica, % | Weight of Test Sample, g |
|---|---|
| Less than 2 | 1.0 |
| 2 and over | 0.5 |

## 11. Procedure

11.1 Transfer the test sample to a small, dry weighing bottle and place in a drying oven. Dry iron ore samples at 110 °C and manganese ore samples at 120 °C for 1 h. Cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize pressure and weigh the capped bottle to the nearest 0.1 mg. Repeat the drying and weighing until there is no further weight loss. Transfer the test sample to a zirconium crucible and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two weights is the weight of the test sample. Add 4 g of sodium peroxide into the crucible. Mix with a stainless steel spatula.

11.2 Fuse the contents over a low flame on a Meker burner, swirling the crucible. When the contents begin to melt, increase the heat to cherry red. Swirl until a clear melt solidifies evenly on the sides of the crucible. Cool for 1 min to 2 min and place into a 250-mL beaker. Cover with a watchglass. Cautiously add 10 mL to 15 mL of water into the crucible. When the reaction ceases, empty the crucible contents and crucible rinsings into the beaker. Add 25 mL HCl by means of the crucible. Finally, rinse and police crucible with water and add rinsings to the beaker. Heat to a boil to obtain a complete and clear solution. Cool for 1 min to 2 min. Add 25 mL of $HClO_4$. Cover with a watchglass and heat to dense white fumes in the $HClO_4$ fume hood. Fume for 15 min to 20 min. Cool for 1 min to 2 min. Add 50 mL of water containing 2 mL HCl (8.3) and heat the contents to dissolve soluble salts. Filter through a fine-textured paper with the aid of paper pulp and a platinum filter cone using suction. Wash filter five times with hot HCl (1 + 49) (8.4) and then ten times with hot water, to remove all perchlorates (see Note 2). Reserve the filtrate. Transfer filter paper and residue to a platinum crucible.

NOTE 2—The residue must be washed free of perchlorates to prevent possible loss of residue by deflagration during the ignition.

11.3 Add 10 mL of $HClO_4$ to the reserved filtrate. Cover with a watchglass and take to dense white fumes and continue fuming for 15 min to 20 min. Cool for 1 min to 2 min. Add 50 mL of water containing 2 mL of HCl (8.3). Heat the contents to dissolve soluble salts. Filter through a fine-textured paper with the aid of paper pulp and a platinum cone using suction. Wash five times with hot HCl (1 + 49) (8.4) and then ten times with hot water (Note 2). Combine the filter and residue with the reserved filter paper and residue in the platinum crucible.

11.4 Ignite crucible and contents at 500 °C for 1 h and then at 1000 °C for 30 min. Cool in a desiccator. After 20 min, weigh.

11.5 Add six drops of $H_2SO_4$ (1 + 1) (8.8) to moisten the contents of the crucible. Add 5 mL of HF. Cautiously and slowly heat to the absence of sulfur trioxide ($SO_3$) fumes. Ignite the residue in a muffle furnace at 1000 °C for 10 min. Cool in a desiccator. After 20 min, weigh. The difference in weight is the weight of the silica.

## 12. Blank Determination

12.1 Perform a blank determination using the same amount of reagents and performing the same operations described in the test procedure.

## 13. Calculation

13.1 Calculate the percentage of silica as follows:

$$\text{Silica, } \% = 100\left[\frac{(A - B)}{C}\right]$$

where:

$A$ = silica in test sample, g,
$B$ = silica in blank, g, and
$C$ = test sample weight, g.

## 14. Precision and Bias[4]

14.1 *Precision*—Table 1 indicates the precision of the test method as determined by Practice E691, but not extended to

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[3] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E16-1005.

obtaining either the repeatability or reproducibility statistic since there were only duplicate determinations made by three cooperating laboratories. (Although one laboratory made quadruplicate analyses on the manganese ore, it was treated as if they were only duplicates.)

14.2 *Bias*—There was no significant bias in the determinations using samples that were all National Institute of Standards and Technology (NIST) certified reference materials.

TABLE 1 Statistical Summary[A]

| Sample[B] | Certified % | Average Determination, % | Standard Deviation | |
|---|---|---|---|---|
| | | | Within-Laboratory | Between-Laboratories |
| Iron Ore | | | | |
| NIST 692 | 10.14 | 10.10 | 0.038 | 0.080 |
| NIST 27F | 4.17 | 4.13 | 0.036 | 0.052 |
| NIST 693 | 3.87 | 3.86 | 0.044 | 0.045 |
| NIST 690 | 3.71 | 3.66 | 0.029 | 0.064 |
| Manganese Ore | | | | |
| NIST 25C | 2.36 | 2.34 | 0.036 | 0.074 |
| Pooled standard deviations[C] | | | 0.037 | 0.064 |

[A] Calculations based on Practice E691. Since there were only three cooperating laboratories, no projection is made to determine repeatability or reproducibility values.

[B] Each concentration represents a different kind of iron ore.

[C] Root mean square of individual standard deviations.

## 15. Keywords

15.1 agglomerates; analytical procedures; concentrates; gravimetric silica; iron ore; manganese ore; silica

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