



Designation: ~~E342 – 11~~ **E342 – 11 (Reapproved 2016)**

## Standard Test Method for Determination of Chromium Oxide in Chrome Ores by Permanganate Titrimetry<sup>1</sup>

This standard is issued under the fixed designation E342; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of chromium oxide ( $\text{Cr}_2\text{O}_3$ ) in chrome ores in the compositional range from 25 % to 60 %.

NOTE 1—As used in this test method, “%” refers to a mass fraction (wt/wt %) (g/100g).

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:*<sup>2</sup>

[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](#)

[E276 Test Method for Particle Size or Screen Analysis at No. 4 \(4.75-mm\) Sieve and Finer for Metal-Bearing Ores and Related Materials](#)

[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 decomposed by fusion with sodium peroxide. After leaching in water and boiling to decompose peroxides, the solution is acidified with ~~nitric~~ $\text{HNO}_3$  and ~~sulfuric~~ $\text{H}_2\text{SO}_4$  acids. Residual chromium is oxidized to chromate with silver nitrate, potassium permanganate, and peroxydisulfate. The excess of permanganate is destroyed by the addition of ~~sodium chloride~~ $\text{NaCl}$ . After cooling, the chromate is reduced by the addition of a measured excess of a ferrous ammonium sulfate, and the excess is titrated with a permanganate solution.

### 5. Significance and Use

5.1 This test method is intended to be used for compliance with compositional specifications for chromium oxide content in chromium-bearing ores. 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](#).

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 6. Interferences

6.1 None of the elements normally found in chrome ores interfere with this test method.

## 7. Reagents and Materials

7.1 *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 such specifications are available<sup>3</sup>. 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification **D1193**. Type III or IV may be used if they effect no measurable change in the blank or sample.

7.3 *Ammonium Peroxydisulfate Solution (250 g/L)*—Dissolve 25 g of ammonium persulfate (ammonium peroxydisulfate) ( $(\text{NH}_4)_2\text{S}_2\text{O}_8$ ) in water, and dilute to 100 mL. Prepare fresh as needed.

7.4 *Ferrous Ammonium Sulfate Solution (0.2 N)*—Dissolve 78.4 g of ferrous ammonium sulfate ( $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ) in 1 L of cool  $\text{H}_2\text{SO}_4$  (5 + 95). Because the ferrous ammonium sulfate solution gradually weakens in reducing power, it is necessary to standardize the ferrous ammonium sulfate solution daily or at the same time that the chrome ore is analyzed. To standardize, transfer 100 mL of  $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  to a 600-mL beaker, dilute to 300 mL with cold  $\text{H}_2\text{SO}_4$  (5 + 95), add 2 mL of  $\text{H}_3\text{PO}_4$  and 2 drops of ortho-phenanthroline ferrous sulfate (ferroin). Titrate immediately with 0.2 N  $\text{KMnO}_4$  solution (**7.97.8**) to a faint, permanent pink color.

7.5 *Ferrous Sulfate Solution (0.025 M)*—Dissolve 6.95 g of ferrous sulfate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) in 500 mL of water and dilute to 1 L.

7.6 *Ortho-Phenanthroline Ferrous Sulfate Indicator Solution—(0.025 M)*—Dissolve 1.485 g of 1,10-phenanthroline monohydrate in 100 mL of ferrous sulfate solution ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) prepared in **7.5**.

7.7 *Phosphoric Acid (specific gravity 1.69 g/mL)*—Concentrated phosphoric acid ( $\text{H}_3\text{PO}_4$ ).

7.7 *Potassium Permanganate Solution (20 g/L)*—Dissolve 20 g of potassium permanganate ( $\text{KMnO}_4$ ) in water and dilute to 1 L.

7.8 *Potassium Permanganate Solution (0.2 N)*—Dissolve 6.4 g of  $\text{KMnO}_4$  in 1 L of water. Standardize against a National Institute of Standards and Technology Reference Material for sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) or equivalent.

7.9 *Silver Nitrate Solution (8 g/L)*—Dissolve 8 g of silver nitrate ( $\text{AgNO}_3$ ) in water and dilute to 1 L.

7.10 *Sodium Chloride Solution (100 g/L)*—Dissolve 10 g of sodium chloride ( $\text{NaCl}$ ) in water and dilute to 100 mL.

7.11 *Sodium Peroxide ( $\text{Na}_2\text{O}_2$ )*.

## 8. Hazards

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

## 9. Sampling, Test Specimens, and Test Units

9.1 The test material shall be pulverized so that at least 95 % passes a No. 100 (150- $\mu\text{m}$ ) sieve in accordance with Test Method **E276** and shall be dried at 105 °C to 110 °C for a minimum of 1 h.

## 10. Procedure

10.1 Transfer 0.50 g of the previously dried sample, weighed to the nearest 0.1 mg to a 30-mL iron crucible (**Note 2**) and add 8 g of dry  $\text{Na}_2\text{O}_2$  (**Note 3**). Thoroughly mix the contents of the crucible and cover the mixture with an additional 1 g to 2 g of  $\text{Na}_2\text{O}_2$ .

10.2 Cover the crucible with an iron or nickel cover and carefully fuse the contents at 600 °C to 700 °C, preferably in an electric muffle furnace. After the mix has melted, fuse for several minutes at a low red heat while giving a slight rotary motion to the crucible to ensure complete decomposition.

NOTE 2—Ordinary iron crucibles ~~which that~~ may contain significant amounts of chromium should be avoided. Crucibles made of ingot iron are satisfactory.

NOTE 3—Only about 5 g of  $\text{Na}_2\text{O}_2$  is required if about 0.2 g of very fine pulverized sugar charcoal is mixed with the  $\text{Na}_2\text{O}_2$ . After heating for about 30 s, ignition of the charcoal takes place, and the crucible will suddenly become a dull red on the outside. The total time required to complete the fusion is about 2 min. By shortening the time for fusion and contact with the flux, the life of the crucible is extended.

<sup>3</sup> *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 the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.