



Designation: E 248 – 97

Standard Test Method for Determination of Manganese in Manganese Ores by Pyrophosphate Complexed Permanganate Potentiometric Titration¹

This standard is issued under the fixed designation E 248; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This definitive test method covers the determination of manganese in manganese ore in the range from 15 to 60 %.

1.2 *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:

D 1193 Specification for Reagent Water²

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³

E 882 Guide for Accountability and Quality Control in the Chemical Analysis of Metals⁴

3. Summary of Test Method

3.1 The test sample is decomposed by treatment with hydrochloric, nitric, hydrofluoric, and perchloric acids. After the addition of sodium pyrophosphate and adjustment of the acidity, the manganese is determined by oxidation to trivalent manganese with a standard solution of potassium permanganate. The end point is determined potentiometrically.

4. Significance and Use

4.1 This definitive test method is intended to be used as a referee method for compliance with compositional specifications for manganese 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 E 882.

5. Interferences

5.1 Provision has been made for removal of chromium which, under some conditions, is an interfering element.

6. Apparatus

6.1 *pH Meter* equipped with calomel and glass electrodes.

6.2 *Potentiometric Titration Apparatus* equipped with calomel and platinum electrodes. A suitable pH meter may be used for this purpose.

6.3 *Magnetic Stirrer*—Use of a TFE-fluorocarbon covered stirring bar is recommended.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ 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 Specification D 1193.

7.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

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

7.5 *Hydrofluoric Acid* (48 %)—Concentrated hydrofluoric acid (HF).

7.6 *Hydrogen Peroxide* (3 %)—Mix 1 volume of concentrated hydrogen peroxide (H₂O₂, 30 %) with 9 volumes of water.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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.

Current edition approved April 10, 1997. Published June 1997. Originally published as E 248 – 64 T. Last previous edition E 248 – 95.

² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.05.

⁴ *Annual Book of ASTM Standards*, Vol 03.06.

⁵ “Reagent Chemicals, American Chemical Society Specifications,” Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see “Reagent Chemicals and Standards,” by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the “United States Pharmacopeia.”