



Designation: D 3872 – 86 (Reapproved 1999)

## Standard Test Method for Ferrous Iron in Iron Oxides<sup>1</sup>

This standard is issued under the fixed designation D 3872; 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 quantitative determination of ferrous oxide (FeO) by oxidation of ferrous iron (Fe<sup>++</sup>) in an acid solution to the ferric state (Fe<sup>+++</sup>) and titration with potassium dichromate using diphenylamine as the indicator.

1.2 This test method is applicable to synthetic black iron oxide, natural black iron oxide, magnetite or brown iron oxide where part of the iron content is present in the ferrous state (Note 1). It is applicable to iron oxides where the ferrous iron content ranges from 50 to 0.20 %.

NOTE 1—Natural iron oxides and magnetite may contain traces of metallic iron that will be combined with and analyzed as FeO.

1.3 *This standard does not purport to address the safety concerns if any, problems 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 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese<sup>2</sup>
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments<sup>2</sup>
- D 769 Specification for Black Synthetic Iron Oxide<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>3</sup>
- D 3722 Specification for Natural Red and Brown Iron Oxide Pigments<sup>2</sup>
- D 3724 Specification for Synthetic Brown Iron Oxide Pigment<sup>2</sup>

### 3. Significance and Use

3.1 This test method may be used for production quality control or specification acceptance.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

### 4. Apparatus

4.1 The digestion apparatus consists of a standard 500-mL Erlenmeyer flask fitted with a two-hole rubber stopper and glass tubing, as shown in Fig. 1, to provide for the introduction of the inert gas and vent for gas and digestion fumes.

### 5. Reagents

5.1 *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.<sup>4</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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

5.3 *Diphenylamine Indicator*—Dissolve 1 g of diphenylamine in 100 mL of concentrated (H<sub>2</sub>SO<sub>4</sub>) (sp gr 1.84).

5.4 *Hydrochloric Acid (1+1)*—Dilute concentrated hydrochloric acid (HCl, sp gr 1.19) with an equal volume of reagent water.

5.5 *Iron Ore, Standard, Sibley No. 27-f*.

5.6 *Potassium Dichromate, Standard Solution (0.1 N)*—Dissolve 4.904 g of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in water and dilute to 1 L. Standardize against National Institute of Standards and Technology standard sample No. 27f of Sibley iron ore. Calculate the Fe factor in grams per millilitre for the solution as:

$$Fe = W/V_1 \quad (1)$$

<sup>4</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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>5</sup> May be secured from National Institute of Standards and Technology, Department of Commerce, Washington, DC 20234. Other recognized primary iron standard may be substituted.