



# Standard Test Method for Iron in Trace Quantities Using the 1,10-Phenanthroline Method<sup>1</sup>

This standard is issued under the fixed designation E 394; 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 iron in the range from 1 to 100  $\mu\text{g}$ .

1.2 This test method is intended to be general for the final steps in the determination of iron and does not include procedures for sample preparation.

1.3 This test method is applicable to samples whose solutions have a pH less than 2. It is assumed that the pH is adjusted to within this range in the sample preparation.

1.4 Review the current material safety data sheets (MSDS) for detailed information concerning toxicity, first-aid procedures, handling, and safety precautions.

1.5 *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 Water<sup>2</sup>

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.01 on General Standards.

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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.

## 3. Summary of Test Method

3.1 This test method is based upon a photometric determination of the 1,10-phenanthroline complex with the iron(II) ion. The sample is dissolved in a suitable solvent and the iron is reduced to the divalent state by the addition of hydroxylamine hydrochloride. The color is then developed, by the addition of 1,10-phenanthroline. After a short reaction period, the absorbance of the solution is measured at approximately 510 nm using a suitable photometer. The absorbance of the solution, once the color is developed, is stable for at least several months.

## 4. Significance and Use

4.1 This test method is suitable for determining trace concentrations of iron in a wide variety of products, provided that appropriate sample preparation has rendered the iron and sample matrix soluble in water or other suitable solvent (see [10.1](#) and [Note 6](#)).

4.2 This test method assumes that the amount of color developed is proportional to the amount of iron in the test solution. The calibration curve is linear over the specified range. Possible interferences are described in [Section 5](#).

## 5. Interferences

5.1 Fortune and Mellon<sup>3</sup> have made a comprehensive study of the interferences of various inorganic ions in this determination. [Table 1](#) and [Table 2](#), taken from their report, show the effects of various cations and anions on the determination of 2.0 ppm iron. If the maximum level of 500 ppm does not interfere, it is very likely that the ion will not interfere in any quantity. The data were obtained under slightly different conditions than those specified in the present test method, but the interferences should be similar. For a more detailed description of interferences, the original literature should be consulted.

5.2 Aldehydes, ketones, and oxidizing agents interfere by consuming the hydroxylamine hydrochloride added as a reducing agent.

<sup>3</sup> Fortune, W. B., and Mellon, M. G., *Industrial and Engineering Chemistry, Analytical Edition*, IENAA Vol 10, 1938, pp. 60–64.



TABLE 1 Effect of Cations on the Determination of 2 ppm Iron

Ion	Added As	Maximum Added Without Interference, ppm	Applicable pH Range
Aluminum	AlCl <sub>3</sub>	500	2.0–3.0
Ammonium	NH <sub>4</sub> Cl	500	2.0–9.0
Antimony	SbCl <sub>3</sub>	30	3.0–9.0
Arsenic	As <sub>2</sub> O <sub>5</sub>	500	3.0–9.0
Arsenic	As <sub>2</sub> O <sub>3</sub>	500	3.0–9.0
Barium	BaCl <sub>2</sub>	500	3.0–9.0
Beryllium	Be(NO <sub>3</sub> ) <sub>2</sub>	500	3.0–5.5
Bismuth	Bi(NO <sub>3</sub> ) <sub>3</sub>	... <sup>A</sup>	... <sup>A</sup>
Cadmium	Cd(NO <sub>3</sub> ) <sub>2</sub>	50	3.0–9.0
Calcium	Ca(NO <sub>3</sub> ) <sub>2</sub>	500	2.0–9.0
Chromium	Cr <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	20	2.0–9.0
Cobalt	Co(NO <sub>3</sub> ) <sub>2</sub>	10	3.0–5.0
Copper	Cu(NO <sub>3</sub> ) <sub>2</sub>	10	2.5–4.0
Lead	Pb(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>2</sub>	500	2.0–9.0
Lithium	LiCl	500	2.0–9.0
Magnesium	Mg(NO <sub>3</sub> ) <sub>2</sub>	500	2.0–9.0
Manganese	MnSO <sub>4</sub>	500	2.0–9.0
Mercury	HgCl <sub>2</sub>	1	2.0–9.0
Mercury	Hg <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub>	10	3.2–9.0
Molybdenum	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub>	100	5.5–9.0
Nickel	Ni(NO <sub>3</sub> ) <sub>2</sub>	2	2.5–9.0
Potassium	KCl	1000	2.0–9.0
Silver	AgNO <sub>3</sub>	... <sup>A</sup>	... <sup>A</sup>
Sodium	NaCl	1000	2.0–9.0
Strontium	Sr(NO <sub>3</sub> ) <sub>2</sub>	500	2.0–9.0
Thorium	Th(NO <sub>3</sub> ) <sub>4</sub>	250	2.0–9.0
Tin	H <sub>2</sub> SnCl <sub>6</sub>	20	3.0–6.0
Tin	H <sub>2</sub> SnCl <sub>4</sub>	10	2.0–6.0
Tungsten	Na <sub>2</sub> WO <sub>4</sub>	10	2.5–9.0
Uranium	UO <sub>2</sub> (C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>2</sub>	100	2.0–6.0
Zinc	Zn(NO <sub>3</sub> ) <sub>2</sub>	10	2.0–9.0
Zirconium	Zr(NO <sub>3</sub> ) <sub>4</sub>	50	2.0–9.0

<sup>A</sup>Must be completely absent because of precipitation.

TABLE 2 Effect of Anions on the Determination of 2 ppm Iron

Ion	Added As	Maximum Added Without Interference, ppm	Applicable pH Range
Acetate	NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	500	2.0–9.0
Tetaborate	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	500	3.0–9.0
Bromide	NaBr	500	2.0–9.0
Carbonate	Na <sub>2</sub> CO <sub>3</sub>	500	3.0–9.0
Chlorate	KClO <sub>3</sub>	500	2.5–9.0
Chloride	NaCl	1000	2.0–9.0
Citrate	H <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub>	500	2.0–9.0
Cyanide	KCN	10	2.0–9.0
Dichromate	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	20	2.5–9.0
Fluoride	NaF	500	4.0–9.0
Iodide	KI	500	2.0–9.0
Nitrate	KNO <sub>3</sub>	500	2.0–9.0
Nitrite	KNO <sub>2</sub>	500	2.5–9.0
Oxalate	(NH <sub>4</sub> ) <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	500	6.0–9.0
Perchlorate	KClO <sub>4</sub>	100	2.0–9.0
Phosphate	(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	20	2.0–9.0
Pyrophosphate	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	50	6.0–9.0
Silicate	Na <sub>2</sub> SiO <sub>3</sub>	100	2.0–4.5
Sulfate	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	500	2.0–9.0
Sulfite	Na <sub>2</sub> SO <sub>3</sub>	500	2.0–9.0
Tartrate	(NH <sub>4</sub> ) <sub>2</sub> C <sub>4</sub> H <sub>9</sub> O <sub>6</sub>	500	3.0–9.0
Thiocyanate	KCNS	500	2.0–9.0
Thiosulfate	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	500	3.0–9.0

## 6. Apparatus

6.1 *Photometer*, capable of measuring light absorption at 510 nm and holding a 5-cm or 1-cm cell. Check the performance of the photometer at regular intervals according to the guidelines given in Practice E 275 and the manufacturer's manual.

NOTE 1—If a filter photometer is used, a narrow band filter having its

maximum transmission at 480 to 520 nm should be used. A discussion of photometers and photometric practice is given in Practice E 60.

6.2 *Absorption Cells*, 5-cm or 1-cm light path.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of