Standard Test Method for
Iron in Trace Quantities Using the 1,10-Phenanthroline Method[1]

1. Scope*

1.1 This test method covers the determination of iron in the range from 1 to 100 µg.

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 Safety Data Sheets (SDS) for detailed information concerning toxicity, first-aid procedures, handling, and safety precautions.

1.5 The values given in SI units are the standard. Values in parentheses are for information only.

1.6 *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

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

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 5).

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[4] 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 µg/g (ppm) iron. If the maximum level of 500 µg/g (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.

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[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.15 on Industrial and Specialty General Standards.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Fortune, W. B., and Mellon, M. G., *Industrial and Engineering Chemistry, Analytical Edition*, IENAA Vol 10, 1938, pp. 60–64.

***A Summary of Changes section appears at the end of this standard**

TABLE 1 Effect of Cations on the Determination of 2 µg/g (ppm) Iron

Ion	Added As	Maximum Added Without Interference, µg/g (ppm)	Applicable pH Range
Aluminum	$AlCl_3$	500	2.0–3.0
Ammonium	NH_4Cl	500	2.0–9.0
Antimony	$SbCl_3$	30	3.0–9.0
Arsenic	As_2O_5	500	3.0–9.0
Arsenic	As_2O_3	500	3.0–9.0
Barium	$BaCl_2$	500	3.0–9.0
Beryllium	$Be(NO_3)_2$	500	3.0–5.5
Bismuth	$Bi(NO_3)_3$	...[A]	...[A]
Cadmium	$Cd(NO_3)_2$	50	3.0–9.0
Calcium	$Ca(NO_3)_2$	500	2.0–9.0
Chromium	$Cr_2(SO_4)_3$	20	2.0–9.0
Cobalt	$Co(NO_3)_2$	10	3.0–5.0
Copper	$Cu(NO_3)_2$	10	2.5–4.0
Lead	$Pb(C_2H_3O_2)_2$	500	2.0–9.0
Lithium	$LiCl$	500	2.0–9.0
Magnesium	$Mg(NO_3)_2$	500	2.0–9.0
Manganese	$MnSO_4$	500	2.0–9.0
Mercury	$HgCl_2$	1	2.0–9.0
Mercury	$Hg_2(NO_3)_2$	10	3.2–9.0
Molybdenum	$(NH_4)_6Mo_7O_{24}$	100	5.5–9.0
Nickel	$Ni(NO_3)_2$	2	2.5–9.0
Potassium	KCl	1000	2.0–9.0
Silver	$AgNO_3$	...[A]	...[A]
Sodium	$NaCl$	1000	2.0–9.0
Strontium	$Sr(NO_3)_2$	500	2.0–9.0
Thorium	$Th(NO_3)_4$	250	2.0–9.0
Tin	H_2SnCl_6	20	3.0–6.0
Tin	H_2SnCl_4	10	2.0–6.0
Tungsten	Na_2WO_4	10	2.5–9.0
Uranium	$UO_2(C_2H_3O_2)_2$	100	2.0–6.0
Zinc	$Zn(NO_3)_2$	10	2.0–9.0
Zirconium	$Zr(NO_3)_4$	50	2.0–9.0

[A] Must be completely absent because of precipitation.

TABLE 2 Effect of Anions on the Determination of 2 µg/g (ppm) Iron

Ion	Added As	Maximum Added Without Interference, µg/g (ppm)	Applicable pH Range
Acetate	$NaC_2H_3O_2$	500	2.0–9.0
Tetraborate	$Na_2B_4O_7$	500	3.0–9.0
Bromide	$NaBr$	500	2.0–9.0
Carbonate	Na_2CO_3	500	3.0–9.0
Chlorate	$KClO_3$	500	2.5–9.0
Chloride	$NaCl$	1000	2.0–9.0
Citrate	$H_3C_6H_5O_7$	500	2.0–9.0
Cyanide	KCN	10	2.0–9.0
Dichromate	$K_2Cr_2O_7$	20	2.5–9.0
Fluoride	NaF	500	4.0–9.0
Iodide	KI	500	2.0–9.0
Nitrate	KNO_3	500	2.0–9.0
Nitrite	KNO_2	500	2.5–9.0
Oxalate	$(NH_4)_2C_2O_4$	500	6.0–9.0
Perchlorate	$KClO_4$	100	2.0–9.0
Phosphate	$(NH_4)_2HPO_4$	20	2.0–9.0
Pyrophosphate	$Na_4P_2O_7$	50	6.0–9.0
Silicate	Na_2SiO_3	100	2.0–4.5
Sulfate	$(NH_4)_2SO_4$	500	2.0–9.0
Sulfite	Na_2SO_3	500	2.0–9.0
Tartrate	$(NH_4)_2C_4H_9O_6$	500	3.0–9.0
Thiocyanate	$KCNS$	500	2.0–9.0
Thiosulfate	$Na_2S_2O_3$	500	3.0–9.0

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

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 E275 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 E60.