Designation: E394 – 22

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

This standard is issued under the fixed designation E394; 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.

1. Scope*

1.1 This test method covers the determination of trace concentrations of iron in the range from 1 to 100 µg/g in a wide variety of products.

1.2 In determining the conformance of the test results using this method to applicable specifications; results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials used in this specification.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

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

E300 Practice for Sampling Industrial Chemicals

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 4).

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

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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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

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.