



Standard Test Methods for Chemical Analysis of Manganese-Copper Alloys¹

This standard is issued under the fixed designation E 581; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analysis of manganese-copper alloys having chemical compositions within the following limits:

Element	Concentration Range, %
Copper	68.0 to 72.0
Manganese	28.0 to 32.0
Carbon	0.03 max
Iron	0.01 max
Phosphorus	0.01 max
Silicon	0.05 max
Sulfur	0.01 max

1.2 The test methods appear in the following order:

	Sections
Iron by the 1,10-Phenanthroline Photometric Method	8-17
Manganese by the (Ethylenedinitrilo) Tetraacetic Acid (EDTA)—Back-Titrimetric Method	18-24
Phosphorus by the Molybdivanadophosphoric Acid Extraction Photometric Method	25-35

1.3 *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.* For precaution to be observed in the use of certain reagents, refer to Practices E 50. A specific precautionary statement is given in Note 2.

2. Referenced Documents

2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications²
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³
- E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition³
- E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals³

¹ These methods are under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, their Alloys and Related Metals.

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² *Annual Book of ASTM Standards*, Vol 14.02.

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

- E 88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition³
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods 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.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections of each test method. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

5. Sampling

5.1 For procedures for sampling the material, refer to Practices E 55 and E 88.

6. Rounding Calculated Values

6.1 Calculated values shall be rounded to the desired number of places as directed in 3.4 to 3.6 of Practice E 29.

7. Interlaboratory Studies

7.1 These test methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision section.

IRON BY THE 1,10-PHENANTHROLINE PHOTOMETRIC METHOD

8. Scope

8.1 This test method covers the determination of iron in concentrations from 0.003 to 0.02 %.



9. Summary of Test Method

9.1 The sample is dissolved in hydrochloric acid and hydrogen peroxide, and the excess oxidant removed by evaporation. The iron is extracted with methyl isobutyl ketone-benzene mixture. The iron is extracted from the organic phase into a hydroxylamine hydrochloride solution and the red-colored 1,10-phenanthroline complex is formed. Photometric measurement is made at approximately 510 nm.

10. Concentration Range

10.1 The recommended concentration range is from 0.005 to 0.125 mg of iron per 50 mL of solution using a 2-cm cell.

NOTE 1—This test method has been written for cells having a 2-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

11. Stability of Color

11.1 The color develops within 5 min and is stable for at least 4 h.

12. Interferences

12.1 Elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

13. Reagents

13.1 *Hydroxylamine Hydrochloride Solution* (10 g/L)—Prepare a solution as directed for Reagent No. 131, but dilute to 500 mL.

13.2 *Iron, Standard Solution A* (1 mL = 0.125 mg Fe)—Prepare a solution as directed for Reagent No. 4, but use 0.1250 g instead of the specified weight.

13.3 *Iron, Standard Solution B* (1 mL = 0.00625 mg Fe)—Using a pipet, transfer 50 mL of iron solution A (1 mL = 0.125 mg Fe) to a 1-L volumetric flask, dilute to volume with HCl (1 + 49), and mix.

13.4 *Methyl Isobutyl Ketone-Benzene Mixture*—Mix 200 mL of methyl isobutyl ketone (MIBK) and 100 mL of benzene.

13.5 *1,10-Phenanthroline-Ammonium Acetate Buffer Solution*—Dissolve 1.0 g of 1,10-phenanthroline monohydrate in 5 mL of HCl in a 600-mL beaker. Add 215 mL of CH₃COOH, and, while cooling, carefully add 265 mL of NH₄OH. Cool to room temperature. Using a pH meter, check the pH; if it is not between 6.0 and 6.5, adjust it to that range by adding acetic acid or NH₄OH as required. Dilute to 500 mL.

14. Preparation of Calibration Curve

14.1 Calibration Solutions:

14.1.1 Using pipets, transfer 1, 2, 5, 10, 15, and 20 mL of iron solution B (1 mL = 0.00625 mg Fe) to 50-mL volumetric flasks. Dilute to 20 mL.

14.1.2 Add 20 mL of NH₂OH·HCl solution, mix, and allow to stand 1 min. Proceed as directed in 14.3.

14.2 *Reference Solution*—Transfer 20 mL of water to a 50-mL volumetric flask and proceed as directed in 14.1.2.

14.3 *Color Development*—Add 5 mL of 1,10-phenanthroline-ammonium acetate buffer solution, dilute to volume, and mix. Allow to stand at least 5 min but not more than 4 h.

14.4 Photometry:

14.4.1 *Multiple-Cell Photometer*—Measure the cell correction using absorption cells with a 2-cm light path and a light band centered at approximately 510 nm. Using the test cell, take the photometric readings of the calibration solutions.

14.4.2 *Single-Cell Photometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 2-cm light path and adjust the photometer to the initial setting, using a light band centered at approximately 510 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

14.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of iron per 50 mL of solution.

15. Procedure

15.1 Test Solution:

15.1.1 Transfer a 2.0-g sample, weighed to the nearest 10 mg, to a 400-mL beaker.

15.1.2 Carry a reagent blank through the entire procedure, using the same amounts of all reagents but with the sample omitted.

15.1.3 Add 25 mL of HCl (7 + 3) and then H₂O₂ as needed to dissolve the alloy completely. When dissolution is complete, add 20 mL of HCl and heat carefully to decompose excess peroxide. Cool to room temperature, transfer to a 125-mL conical separatory funnel. Add HCl (1 + 1), as required, to adjust the volume to 50 mL.

15.1.4 Add 20 mL of MIBK - benzene mixture to the separatory funnel and shake 1 min. Allow the phases to separate, discard the aqueous phase, wash the organic phase 3 times with 3 to 5-mL portions of HCl (1 + 1) to remove copper, and discard the washings. Extract the iron from the organic phase by shaking vigorously 30 s with 10 mL of NH₂OH·HCl solution. Transfer the aqueous phase to a 50-mL volumetric flask. Repeat the extraction with a second 10-mL portion of NH₂OH·HCl solution, and transfer the extract to the 50-mL flask. Dilute to 40 mL and proceed as directed in 15.3.

15.2 *Reference Solution*—Use the reagent blank solution prepared as directed in 15.1.2.

15.3 *Color Development*—Proceed as directed in 14.3.

15.4 *Photometry*—Proceed as directed in 14.4.

16. Calculation

16.1 Convert the net photometric reading of the test solution to milligrams of iron by means of the calibration curve. Calculate the percentage of iron as follows:

$$\text{Iron, \%} = A/(B \times 10) \quad (1)$$

where:

A = milligrams of iron found in 50 mL of the final test solution, and

B = grams of sample represented in 50 mL of the final test solution.