Designation: E581 – 17a (Reapproved 2022)ε1

Standard Test Methods for Chemical Analysis of Manganese-Copper Alloys[1]

This standard is issued under the fixed designation E581; 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.

ε[1] NOTE—Editorial changes were made throughout in September 2022.

1. Scope

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

Element	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 Spectrophotometric Method [0.003 % to 0.02 %]	11 – 20
Manganese by the (Ethylenedinitrilo) Tetraacetic Acid (EDTA)—Back-Titrimetric Method [28 % to 32 %]	21 – 27
Phosphorus by the Molybdivanadophosphoric Acid Extraction Spectrophotometric Method [0.002 % to 0.014 %]	28 – 38

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

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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

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

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition

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

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1997)[3]

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

4. Significance and Use

4.1 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.

[1] These test methods are under the jurisdiction of ASTM Committee E01 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, Precious Metals, their Alloys, and Related Metals.

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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.

5. Apparatus

5.1 Spectrophotometers shall conform to the requirements prescribed in Practice E60.

6. Reagents and Materials

6.1 Reagents required for each determination are listed in separate sections of each test method. The standard solutions and certain other reagents used in more than one procedure shall conform to the requirements prescribed in Practices E50.

6.2 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] 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.

6.3 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D1193.

7. Hazards

7.1 For precautions to be observed in this method, refer to Practices E50.

7.2 A warning statement is given in 24.7.

8. Sampling

8.1 For procedures for sampling the material, refer to Practices E55 and E88.

9. Rounding Calculated Values

9.1 Rounding of test results obtained using this test method shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

10. Interlaboratory Studies

10.1 These test methods have been evaluated in accordance with Practice E173, unless otherwise noted in the precision section. The Reproducibility R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

[4] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC, www.acs.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.

TABLE 1 Statistical Information

Test Sample	Labs	Iron Found, %	Repeatability (r, Practice E1601)	Reproducibility (R, Practice E1601)
Manganese Copper	7	0.0137	0.0013	0.0028

IRON BY THE 1,10-PHENANTHROLINE SPECTROPHOTOMETRIC METHOD

11. Scope

11.1 This test method covers the determination of iron from 0.003 % to 0.02 %.

12. Summary of Test Method

12.1 The sample is dissolved in HCl 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. Spectrophotometric absorbance measurement is made at 510 nm.

13. Iron Range

13.1 The recommended range is from 0.005 mg 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.

14. Stability of Color

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

15. Interferences

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

16. Reagents

16.1 *Hydroxylamine Hydrochloride Solution* (10 g/L)—Dissolve 5.0 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) in 500 mL of water. Prepare fresh as needed.

16.2 *Iron, Standard Solution A* (1 mL = 0.125 mg Fe)—Transfer 0.1250 g of iron (purity: 99.9 % min) to a 100 mL beaker. Add 10 mL of HCl (1 + 1) and 1 mL of bromine water. Boil gently until the excess bromine is removed. Add 20 mL of HCl, cool, transfer to a 1 L volumetric flask, dilute to volume, and mix.

16.3 *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.

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

16.5 *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 acetic acid (CH_3COOH), and, while cooling, carefully add 265 mL of NH_4OH. 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 NH_4OH as required. Dilute to 500 mL.

17. Preparation of Calibration Curve

17.1 *Calibration Solutions:*