**Designation: ~~E354 – 93 (Reapproved 2006)~~ E354 – 14**

# Standard Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys[1]

This standard is issued under the fixed designation E354; 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.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 1. Scope

1.1 These test methods cover the chemical analysis of high-temperature, electrical, magnetic, and other similar iron, nickel, and cobalt alloys having chemical compositions within the following limits:

| ~~Element~~ | ~~Concentration Range, %~~ | | |
|---|---|---|---|
| ~~Aluminum~~ | ~~0.005~~ | ~~to~~ | ~~18.00~~ |
| ~~Beryllium~~ | ~~0.001~~ | ~~to~~ | ~~0.05~~ |
| ~~Boron~~ | ~~0.001~~ | ~~to~~ | ~~1.00~~ |
| ~~Calcium~~ | ~~0.002~~ | ~~to~~ | ~~0.05~~ |
| ~~Carbon~~ | ~~0.001~~ | ~~to~~ | ~~1.10~~ |
| ~~Chromium~~ | ~~0.10~~ | ~~to~~ | ~~33.00~~ |
| ~~Cobalt~~ | ~~0.10~~ | ~~to~~ | ~~75.00~~ |
| ~~Columbium (Niobium)~~ | ~~0.01~~ | ~~to~~ | ~~6.0~~ |
| ~~Copper~~ | ~~0.01~~ | ~~to~~ | ~~10.00~~ |
| ~~Iron~~ | ~~0.01~~ | ~~to~~ | ~~85.00~~ |
| ~~Magnesium~~ | ~~0.001~~ | ~~to~~ | ~~0.05~~ |
| ~~Manganese~~ | ~~0.01~~ | ~~to~~ | ~~3.0~~ |
| ~~Molybdenum~~ | ~~0.01~~ | ~~to~~ | ~~30.0~~ |
| ~~Nickel~~ | ~~0.10~~ | ~~to~~ | ~~84.0~~ |
| ~~Nitrogen~~ | ~~0.001~~ | ~~to~~ | ~~0.20~~ |
| ~~Phosphorus~~ | ~~0.002~~ | ~~to~~ | ~~0.08~~ |
| ~~Silicon~~ | ~~0.01~~ | ~~to~~ | ~~5.00~~ |
| ~~Sulfur~~ | ~~0.002~~ | ~~to~~ | ~~0.10~~ |
| ~~Tantalum~~ | ~~0.005~~ | ~~to~~ | ~~10.0~~ |
| ~~Titanium~~ | ~~0.01~~ | ~~to~~ | ~~5.00~~ |
| ~~Tungsten~~ | ~~0.01~~ | ~~to~~ | ~~18.00~~ |
| ~~Vanadium~~ | ~~0.01~~ | ~~to~~ | ~~3.25~~ |
| ~~Zirconium~~ | ~~0.01~~ | ~~to~~ | ~~2.50~~ |
| Element | Composition Range, % | | |
| Aluminum | 0.005 | to | 18.00 |
| Beryllium | 0.001 | to | 0.05 |
| Boron | 0.001 | to | 1.00 |
| Calcium | 0.002 | to | 0.05 |
| Carbon | 0.001 | to | 1.10 |
| Chromium | 0.10 | to | 33.00 |
| Cobalt | 0.10 | to | 75.00 |
| Columbium (Niobium) | 0.01 | to | 6.0 |
| Copper | 0.01 | to | 10.00 |
| Iron | 0.01 | to | 85.00 |
| Magnesium | 0.001 | to | 0.05 |
| Manganese | 0.01 | to | 3.0 |
| Molybdenum | 0.01 | to | 30.0 |
| Nickel | 0.10 | to | 84.0 |
| Nitrogen | 0.001 | to | 0.20 |
| Phosphorus | 0.002 | to | 0.08 |
| Silicon | 0.01 | to | 5.00 |
| Sulfur | 0.002 | to | 0.10 |



[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 E01.01 on Iron, Steel, and Ferroalloys.

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| | | | |
|---|---|---|---|
| Tantalum | 0.005 | to | 10.0 |
| Titanium | 0.01 | to | 5.00 |
| Tungsten | 0.01 | to | 18.00 |
| Vanadium | 0.01 | to | 3.25 |
| Zirconium | 0.01 | to | 2.50 |

1.2 The test methods in this standard are contained in the sections indicated below:

| | Sections |
|---|---|
| ~~Aluminum, Total, by the 8-Quinolinol Gravimetric Method (0.20 to 7.00 %)~~ | ~~100~~ |
| ~~Carbon,Total, by the Combustion-Thermal Conductivity Method~~ | ~~1a~~ |
| ~~Carbon, Total, by the Combustion Gravimetric Method (0.05 to 1.10 %)~~ | ~~79~~ |
| ~~Chromium by the Atomic Absorption Method (0.006 to 1.00 %)~~ | ~~165~~ |
| ~~Chromium by the Peroxydisulfate Oxidation—Titration Method (0.10 to 33.00 %)~~ | ~~175~~ |
| ~~Chromium by the Peroxydisulfate-Oxidation Titrimetric Method~~ | ~~1b~~ |
| ~~Cobalt by the Ion-Exchange-Potentiometric Titration Method (2 to 75 %)~~ | ~~53~~ |
| ~~Cobalt by the Nitroso-R-Salt Photometric Method (0.10 to 5.0 %)~~ | ~~61~~ |
| ~~Copper by Neocuproine Photometric Method (0.01 to 10.00 %)~~ | ~~90~~ |
| ~~Copper by the Sulfide Precipitation-Electrodeposition Gravimetric Method (0.01 to 10.00 %)~~ | ~~71~~ |
| ~~Iron by the Silver ReductionTitrimetric Method (1.0 to 50.0 %)~~ | ~~192~~ |
| ~~Manganese by the Periodate Photometric Method (0.05 to 2.00 %)~~ | ~~8~~ |
| ~~Molybdenum by the Ion Exchange—8-Hydroxyquinoline Gravimetric Method (1.5 to 30 %)~~ | ~~184~~ |
| ~~Molybdenum by the Photometric Method (0.01 to 1.50 %)~~ | ~~153~~ |
| ~~Nickel by the Dimethylglyoxime Gravimetric Method (0.1 to 84.0 %)~~ | ~~135~~ |
| ~~Phosphorus by the Molybdenum Blue Photometric Method (0.002 to 0.08 %)~~ | ~~18~~ |
| ~~Silicon by the Gravimetric Method (0.05 to 5.00 %)~~ | ~~46~~ |
| ~~Sulfur by the Gravimetric Method~~ | ~~1c~~ |
| ~~Sulfur by the Combustion Iodate Titration Method (0.005 to 0.1 %)~~ | ~~37~~ |
| ~~Sulfur by the Chromatographic Gravimetric Method~~ | ~~1b~~ |
| ~~Tin by the Solvent Extraction- Atomic Absorption Method (0.002 to 0.10 %)~~ | ~~143~~ |

| | Sections |
|---|---|
| Aluminum, Total, by the 8-Quinolinol Gravimetric Method (0.20 % to 7.00 %) | 100 |
| Carbon, Total, by the Combustion-Thermal Conductivity Method | Discontinued |
| Carbon, Total, by the Combustion Gravimetric Method (0.05 % to 1.10 %) | Discontinued |
| Chromium by the Atomic Absorption Method (0.006 % to 1.00 %) | 165 |
| Chromium by the Peroxydisulfate Oxidation—Titration Method (0.10 % to 33.00 %) | 175 |
| Chromium by the Peroxydisulfate-Oxidation Titrimetric Method | Discontinued |
| Cobalt by the Ion-Exchange-Potentiometric Titration Method (2 % to 75 %) | 53 |
| Cobalt by the Nitroso-R-Salt Spectrophotometric Method (0.10 % to 5.0 %) | 61 |
| Copper by Neocuproine Spectrophotometric Method (0.01 % to 10.00 %) | 90 |
| Copper by the Sulfide Precipitation-Electrodeposition Gravimetric Method (0.01 % to 10.00 %) | 71 |
| Iron by the Silver ReductionTitrimetric Method (1.0 % to 50.0 %) | 192 |
| Manganese by the Periodate Spectrophotometric Method (0.05 % to 2.00 %) | 9 |
| Molybdenum by the Ion Exchange—8-Hydroxyquinoline Gravimetric Method (1.5 % to 30 %) | 184 |
| Molybdenum by the Spectrophotometric Method (0.01 % to 1.50 %) | 153 |
| Nickel by the Dimethylglyoxime Gravimetric Method (0.1 % to 84.0 %) | 135 |
| Phosphorus by the Molybdenum Blue Spectrophotometric Method (0.002 % to 0.08 %) | 19 |
| Silicon by the Gravimetric Method (0.05 % to 5.00 %) | 46 |
| Sulfur by the Gravimetric Method | Discontinued |
| Sulfur by the Combustion-Iodate Titration Method (0.005 % to 0.1 %) | Discontinued |
| Sulfur by the Chromatographic Gravimetric Method | |
| Tin by the Solvent Extraction–Atomic Absorption Method (0.002 % to 0.10 %) | 143 |

1.3 Methods for the determination of ~~several elements~~ carbon and sulfur not included in this standard can be found in Test Methods ~~E30 and Test Methods~~ E1019.

1.4 Some of the ~~concentration~~composition ranges given in 1.1 are too broad to be covered by a single method and therefore this standard contains multiple methods for some elements. The user must select the proper method by matching the information given in the Scope and Interference sections of each method with the composition of the alloy to be analyzed.

1.5 The values stated in SI units are to be regarded as standard. ~~In some cases, exceptions allowed in Practice E380 are also used.~~

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.* Specific hazards statements are given in Section ~~5~~6 and in special "Warning" paragraphs throughout these test methods.

## 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

~~E30 Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (Withdrawn 1995)~~[3]

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

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

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 1998)[3]

E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron

E351 Test Methods for Chemical Analysis of Cast Iron—All Types

E352 Test Methods for Chemical Analysis of Tool Steels and Other Similar Medium- and High-Alloy Steels

E353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys

~~E380 Practice for Use of the International System of Units (SI) (the Modernized Metric System) (Withdrawn 1997)~~[3]

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

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

E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

2.2 *Other Document:*

ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility for Inter-Laboratory Tests[4]

## 3. Terminology

3.1 For definitions of terms used in these test methods, refer to Terminology E135.

## 4. Significance and Use

4.1 These test methods for the chemical analysis of metals and alloys are primarily intended as referee methods to test such materials for compliance with compositional specifications, particularly those under the jurisdiction of the ASTM Committee ~~D1193~~on Steel, Stainless Steel and Related Alloys. 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 under appropriate quality control practices such as those described in Guide E882.

## 5. Apparatus, Reagents, and Instrumental Practice

5.1 *Apparatus*—Specialized apparatus requirements are listed in the "Apparatus" Section in each method. ~~In some cases reference may be made to Practices E50.~~

5.2 *Reagents:*

[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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

5.2.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, ~~all reagents used in these test methods shall~~ it is intended that all reagents conform to the ~~"Reagent Grade" Specifications of the American Chemical Society.~~specifications of the Committee on Analytical Reagents of the American Chemical Society ~~Other chemicals~~where such specifications are available. Other grades may be used, provided it is first ascertained that ~~they are~~ the reagent is of sufficiently high purity to permit ~~their~~its use without ~~adversely affecting the expected performance of the determination, as indicated in the section on "Precision and Bias."~~lessening the accuracy of the determination.

5.2.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as ~~defined by Type~~ conforming to Type I or Type II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

5.3 ~~*Photometric*~~*Spectrophotometric Practice*—~~Photometric~~Spectrophotometric practice prescribed in these test methods shall conform to Practice E60.

## 6. Hazards

6.1 For precautions to be observed in the use of certain reagents and equipment in these methods, refer to Practices E50.

## 7. Sampling

7.1 For procedures for sampling the material, reference shall be made to Practice E1806.

## 8. Interlaboratory Studies and Rounding Calculated Values

8.1 These test methods have been evaluated ~~using~~ in accordance with Practice E173 ~~or ISO 5725.~~(withdrawn 1997) or ISO 5725. The Reproducibility R2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601

8.2 Calculated values shall be rounded to the desired number of places ~~as directed in 3.4 to 3.6~~in accordance with the Rounding Method of Practice E29.



## MANGANESE BY THE METAPERIODATE ~~PHOTOMETRIC~~SPECTROPHOTOMETRIC METHOD

## 9. Scope

9.1 This method covers the determination of manganese in ~~concentrations~~compositions from 0.05 % to 2.00 ~~percent.~~%.

## 10. Summary of Method

10.1 Manganous ions are oxidized to permanganate ions by treatment with periodate. Tungsten when present at ~~concentrations~~compositions greater than ~~0.5 %~~ 0.5 % is kept in solution with ~~phosphoric~~$H_3PO_4$ ~~acid.~~ . Solutions of the samples are fumed with ~~perchloric~~$HClO_4$ ~~acid~~ so that the effect of periodate is limited to the oxidation of manganese. ~~Photometric~~Spectrophotometric measurements are made at approximately 545 nm.

## 11. Concentration Range

11.1 The recommended concentration range is 0.15 mg to 0.8 mg of manganese per 50 mL of solution, using a 1-cm cell (Note 1) and a spectrophotometer with a band width of 10 nm or less.

NOTE 1—This method has been written for cells having a 1-cm light path and a "narrow-band" instrument. The concentration range depends upon band width and spectral region used as well as cell optical path length. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

## 12. Stability of Color

12.1 The color is stable for at least 24 h.

## 13. Interferences

13.1 ~~Perchloric~~$HClO_4$ acid treatment, which is used in the procedure, yields solutions which can be highly colored due to the presence of Cr (VI) ions. Although these ions and other colored ions in the sample solution undergo no further change in color quality upon treatment with metaperiodate ion, the following precautions must be observed when filter ~~photometers~~spectrophotometers are used: Select a filter with maximum transmittance between 545 nm and 565 nm. The filter must transmit not more than 5 % of its maximum at a wavelength shorter than 530 nm. The band width of the filter should be less than 30 nm when measured at 50 % of its maximum transmittance. Similar restrictions apply with respect to the wavelength region employed when ~~other "wide-band"~~other "wide-band" instruments are used.

13.2 The spectral transmittance curve of permanganate ions exhibits two useful minima, one at approximately 526 nm, and the other at 545 nm. The latter is recommended when a "narrow-band" spectrophotometer is used.