



Designation: E363 – 23

Standard Test Methods for Chemical Analysis of Chromium and Ferrochromium¹

This standard is issued under the fixed designation E363; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 These test methods cover the chemical analysis of chromium and ferrochromium having chemical compositions within the following limits:

Element	Composition, %
Aluminum	0.25 max
Antimony	0.005 max
Arsenic	0.005 max
Bismuth	0.005 max
Boron	0.005 max
Carbon	9.00 max
Chromium	51.0 to 99.5
Cobalt	0.10 max
Columbium	0.05 max
Copper	0.05 max
Lead	0.005 max
Manganese	0.75 max
Molybdenum	0.05 max
Nickel	0.50 max
Nitrogen	6.00 max
Phosphorus	0.03 max
Silicon	12.00 max
Silver	0.005 max
Sulfur	0.07 max
Tantalum	0.05 max
Tin	0.005 max
Titanium	0.50 max
Vanadium	0.50 max
Zinc	0.005 max
Zirconium	0.05 max

1.2 The analytical procedures appear in the following order:

	Sections
Arsenic by the Molybdenum Blue Spectrophotometric Test Method [0.001 % to 0.005 %]	10 – 20
Lead by the Dithizone Spectrophotometric Test Method [0.001 % to 0.05 %]	21 – 31
Chromium by the Sodium Peroxide Fusion-Titrimetric Test Method [50.0 % to 99.5 %]	32 – 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. Specific hazard statements are given in Section 6 and in special “Warning” paragraphs throughout these test methods.

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

- A101 Specification for Ferrochromium
- A481 Specification for Chromium Metal
- D1193 Specification for Reagent Water
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition
- 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 1997)³
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 For definition of terms used in this test method, refer to Terminology E135.

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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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. Significance and Use

4.1 These test methods for the chemical analysis of chromium metal and ferrochromium alloy are primarily intended to test such materials for compliance with compositional specifications such as Specifications A101 and A481. 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.

5. Apparatus, Reagents, and Spectrophotometric Practice

5.1 Apparatus, standard solutions, and other reagents required for each determination are listed in separate sections preceding the procedure. Spectrophotometers shall conform to the requirements prescribed in Practice E60. (See 5.1.1.)

5.1.1 In these methods, cells utilized to contain the reference material and sample solutions in spectrophotometers are referred to as “absorption cells.” The radiant energy passed through the cells can be measured as absorbance or transmittance. These methods refer to absorbance measurements. Refer to Practice E60 for details.

5.2 Spectrophotometric practices prescribed in these test methods shall conform to Practice E60.

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

5.4 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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. Hazards

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

6.2 Specific hazard statements are given in 27.1, 27.7, and 36.2.

7. Sampling

7.1 For procedures to sample the material, and particle size requirements of the sample, refer to Practices E32.

8. Rounding Calculated Values

8.1 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.

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E173, unless otherwise noted in the Precision and Bias section. Practice E173 has been replaced by Practice E1601. The Reproducibility R_2 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.

ARSENIC BY THE MOLYBDENUM BLUE SPECTROPHOTOMETRIC TEST METHOD

10. Scope

10.1 This test method covers the determination of arsenic in chromium and ferrochromium in compositions from 0.001 % to 0.005 %.

11. Summary of Method

11.1 Arsenic is first separated by distillation as the trivalent chloride. Ammonium molybdate is added to form arsenomolybdate, which is then reduced by hydrazine sulfate to form the molybdenum blue complex. Spectrophotometric absorbance measurement is made at 850 nm.

12. Concentration Range

12.1 The recommended concentration range is 0.01 mg to 0.15 mg of arsenic per 50 mL of solution using a 1 cm cell. (See Note 1.)

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

13. Stability of Color

13.1 The color is stable for at least 2 h.

14. Interferences

14.1 The elements ordinarily present do not interfere if their compositions are under the maximum limits shown in 1.1.

15. Apparatus

15.1 *Distillation Apparatus*, Fig. 1.

15.2 *Zirconium Crucibles*, 30 mL capacity.

16. Reagents

16.1 *Ammonium Bromide* (NH_4Br).

16.2 *Ammonium Molybdate Solution* (10 g/L)—Dissolve 2.5 g of ammonium heptamolybdate tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$) in 40 mL of warm water. Add 128 mL of H_2SO_4 (1 + 3), dilute to 250 mL, and mix.

16.3 *Ammonium Molybdate-Hydrazine Sulfate Solution*—Dilute 100 mL of ammonium molybdate solution to 900 mL, add 10 mL of hydrazine sulfate solution, dilute to 1 L, and mix. Do not use a solution that has stood more than 1 h.

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, www.usp.org.