Designation: E 363 – 83 (Reapproved 1997)ε1

Standard Methods for Chemical Analysis of Chromium and Ferrochromium[1]

This standard is issued under the fixed designation E 363; 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—Keywords were added editorially in December 1997.

1. Scope

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

Element	Concentration, %
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 Photometric Method	9-19
Lead by the Dithizone Photometric Method	20-30
Chromium by the Sodium Peroxide Fusion-Titrimetric Method	31-37

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 whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*

A 101 Specification for Ferrochromium[2]

A 481 Specification for Chromium Metal[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition[4]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[4]

E 360 Test Methods for Chemical Analysis of Silicon and Ferrosilicon[5]

E 361 Test Methods for Chemical Analysis of Ferromanganese and Spiegeleisen[5]

3. Significance and Use

3.1 These 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 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 preceding the procedure. 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.

4.2 Photometric practice prescribed in these methods shall conform to Practice E 60.

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

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[2] *Annual Book of ASTM Standards*, Vol 01.02.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] *Annual Book of ASTM Standards*, Vol 03.06.

5. Safety Precautions

5.1 For precautions to be observed in the use of certain reagents in these methods, refer to Practices E 50.

6. Sampling

6.1 For procedures for sampling the material, and for particle size of the sample for chemical analysis, refer to Practices E 32.

7. Rounding Calculated Values

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

8. Interlaboratory Studies

8.1 These methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision and bias section.

ARSENIC BY THE MOLYBDENUM BLUEPHOTOMETRIC METHOD

9. Scope

9.1 This method covers the determination of arsenic in chromium and ferrochromium in concentrations from 0.001 to 0.005 %.

10. Summary of Method

10.1 See Section 10 of Test Methods E 360.

11. Concentration Range

11.1 See Section 11 of Test Methods E 360.

12. Stability of Color

12.1 See Section 12 of Test Methods E 360.

13. Interferences

13.1 See Section 13 of Test Methods E 360.

14. Apparatus

14.1 See Section 14 of Test Methods E 360.

15. Reagents

15.1 Proceed as directed in 15.1 through 15.9 of Test Methods E 360.

16. Preparation of Calibration Curve

16.1 Proceed as directed in 16.1 through 16.5 of Test Methods E 360.

17. Procedure

17.1 Proceed as directed in 17.1 through 17.4 of Test Methods E 360.

18. Calculation

18.1 Proceed as directed in Section 18 of Test Methods E 360.

19. Precision and Bias

19.1 Nine laboratories cooperated in testing this method and obtained the data summarized in Table 1. Samples with arsenic concentrations near the upper limit of the scope were not available for testing. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this method is adequate for the contemplated use.

LEAD BY THE DITHIZONE PHOTOMETRIC METHOD

20. Scope

20.1 This method covers the determination of lead in chromium and ferrochromium in concentrations from 0.001 to 0.05 %.

21. Summary of Method

21.1 See Section 21 of Test Methods E 361.

22. Concentration Range

22.1 See Section 22 of Test Methods E 361.

23. Stability of Color

23.1 See Section 23 of Test Methods E 361.

24. Interferences

24.1 See Section 24 of Test Methods E 361.

25. Apparatus

25.1 See Section 25 of Test Methods E 361.

26. Reagents

26.1 Proceed as directed in Section 26 of Test Methods E 361.

27. Preparation of Calibration Curve

27.1 Proceed as directed in 27.1 through 27.5 of Test Methods E 361.

28. Procedure

28.1 *Test Solution*:

28.1.1 Transfer a sample, selected in accordance with 28.1.1 of Test Methods E 361 and weighed to the nearest 0.1 mg, to a 250-mL beaker. Add 30 mL of HCl (1 + 1) and heat until dissolution is nearly complete. In the case of high-carbon ferrochromium (4.00 to 9.00 % C), add 30 mL of HCl (concentrated) and several drops of HF, and heat until the reaction has subsided.

28.1.2 Add several drops of HF (omit if added in preceding paragraph) plus 10 mL of HNO_3 and 10 mL of $HClO_4$. Evaporate to heavy fumes of $HClO_4$ and fume until the volume is reduced to approximately 5 mL. Add H_2O_2 solution (1 + 9)

TABLE 1 Statistical Information—Arsenic

Ferroalloy Type	Arsenic Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
1. 70Cr-1Si-5C	0.0015	0.0001	0.0005