



Designation: E 362 – 99

Standard Test Methods for the Determination of Arsenic and Lead in Silicomanganese and Ferrosilicon Manganese¹

This standard is issued under the fixed designation E 362; 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.

1. Scope

1.1 These test methods cover the chemical analysis of silicomanganese and ferrosilicon manganese having chemical compositions within the following limits:

Element	Concentration, %
Arsenic	0.10 max
Carbon	3.0 max
Chromium	0.50 max
Lead	0.030 max
Manganese	50.0 to 68.0
Molybdenum	0.10 max
Nickel	0.20 max
Phosphorus	0.20 max
Silicon	35.0 max
Sulfur	0.04 max
Tin	0.010 max

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

	Sections
Arsenic by the Molybdenum Blue Photometric Method (0.002 to 0.06 %)	9-19
Lead by the Dithizone Photometric Method (0.02 to 0.05 %)	20-30

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.* Specific hazards statements are given in Section 5 and in special warning paragraphs throughout these test methods.

2. Referenced Documents

2.1 ASTM Standards:

- A 483 Specification for Silicomanganese²
- D 1193 Specification for Reagent Water³
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

¹ These test 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 11.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

E 32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition⁵

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁵

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals⁵

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁵

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory⁶

3. Significance and Use

3.1 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 ASTM Committee A-1 on Steel, Stainless Steel, and Related Alloys, specifically Specification A 483.

3.2 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 E 882.

4. Reagents and Photometric Practice

4.1 Reagents:

4.1.1 *Purity of Reagents*—Unless otherwise indicated, all reagents used in these test methods shall conform to the “Reagent Grade” Specifications of the American Chemical Society.⁷ Other chemicals may be used, provided it is first ascertained that they are of sufficiently high purity to permit their use without adversely affecting the expected performance of the determination, as indicated in the Precision and Bias section.

⁵ Annual Book of ASTM Standards, Vol 03.05.

⁶ Annual Book of ASTM Standards, Vol 03.06.

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

4.1.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

4.2 *Photometric Practice*—Shall conform to Practice E 60.

5. Hazards

5.1 For precautions to be observed in the use of certain reagents and equipment in these test 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 test methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the Precision and Bias section.

ARSENIC BY THE MOLYBDENUM BLUE PHOTOMETRIC METHOD

9. Scope

9.1 This test method covers the determination of arsenic in silicomanganese and ferrosilicon manganese in concentrations from 0.02 to 0.06 %.

9.2 The limits of the scope have been set at 0.02 to 0.06 % because test materials containing other arsenic concentrations were unavailable for testing. However, recognizing that the

procedure should give satisfactory results at lower and higher concentrations, this test method's Calibration and Procedure sections cover the range from 0.001 to 0.1 %.

9.2.1 Users of this test method are cautioned that its use on samples outside of the 0.02 to 0.06 % range is not supported by interlaboratory testing.

10. Summary of Test Method

10.1 The sample is fused with sodium peroxide and sodium carbonate and the melt is dissolved in acid. Arsenic, iron, and other elements are precipitated with ammonium hydroxide. The filtered precipitate is dissolved in acid. Ammonium bromide and hydrazine sulfate are added and the arsenic is distilled as arsenic tribromide. The distillate is evaporated to dryness and reacted with ammonium molybdate and hydrazine sulfate to form the molybdenum blue complex. Photometric measurement is made at 850 nm.

11. Concentration Range

11.1 The recommended concentration range is from 0.005 to 0.15 mg of arsenic per 50 mL of solution using a 1-cm cell.

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.

12. Stability of Color

12.1 The color is stable for at least 2 h.

13. Interferences

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

14. Apparatus

14.1 *Distillation Apparatus*, Fig. 1.

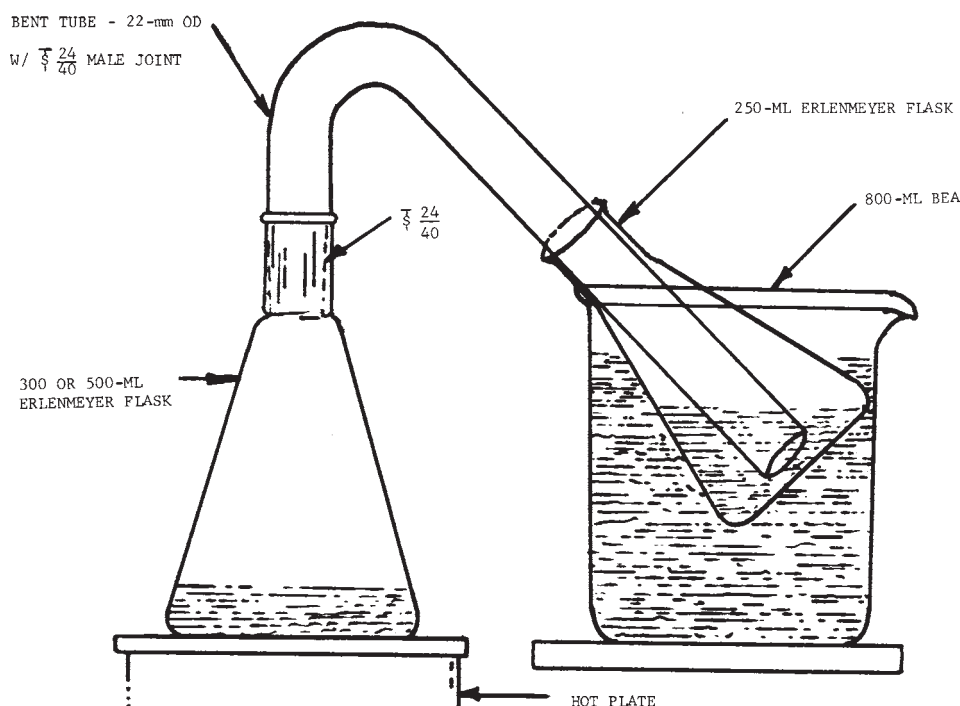


FIG. 1 Arsenic Distillation Apparatus