



Standard Test Methods for the Determination of Arsenic and Lead in Ferromanganese¹

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1. Scope

1.1 These test methods cover the chemical analysis of ferromanganese having chemical compositions within the following limits:

Element	Concentration, %
Arsenic	0.30 max
Carbon	7.50 max
Chromium	0.50 max
Lead	0.050 max
Manganese	16.0 to 90.0
Phosphorus	0.35 max
Silicon	7.00 max
Sulfur	0.050 max
Tin	0.020 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 (0.004 to 0.01 %)	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 hazard statements are given in Section 5 and in special warning paragraphs throughout.

2. Referenced Documents

2.1 ASTM Standards:

A 99 Specification for Ferromanganese²

D 1193 Specification for Reagent Water³

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

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

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.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.

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 99.

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.

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

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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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 ferromanganese and spiegeleisen 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.

14.2 *Zirconium Crucibles*, 30-mL capacity.

15. Reagents

15.1 *Ammonium Bromide* (NH_4Br).

15.2 *Ammonium Molybdate Solution* (10 g/L)—Dissolve 2.5 g of ammonium heptamolybdate tetrahydrate

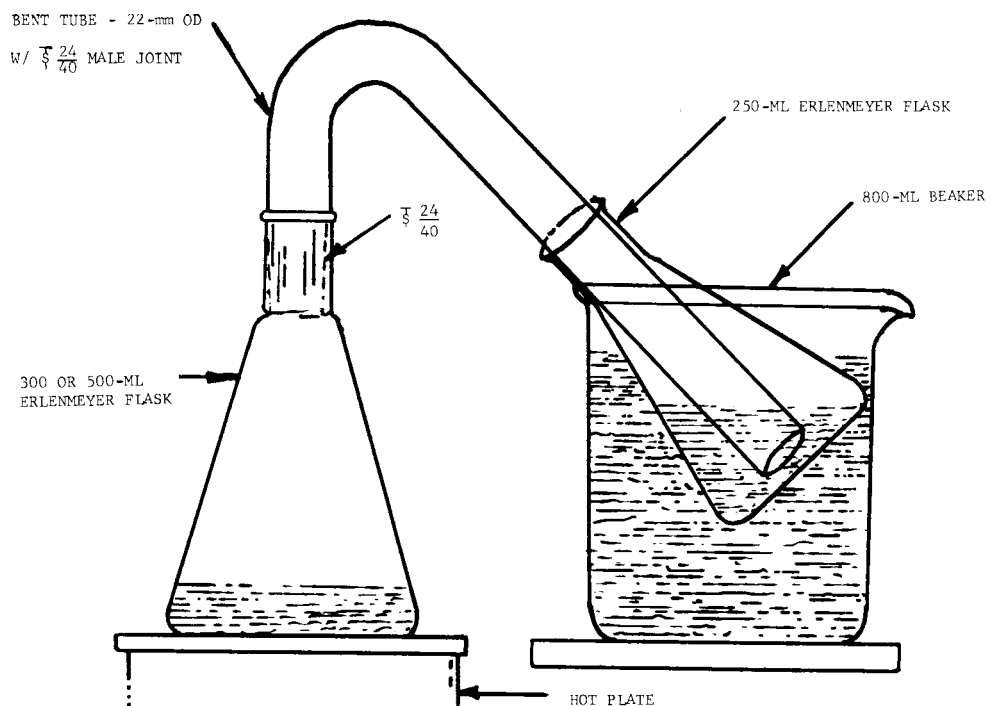


FIG. 1 Arsenic Distillation Apparatus

$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) in 40 mL of warm water. Add 128 mL of H_2SO_4 (1+3), dilute to 250 mL, and mix.

15.3 *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.

15.4 *Arsenic Standard Solution A* (1 mL = 0.10 mg As)—Transfer 0.1320 g of arsenic trioxide (As_2O_3) to a 1-L volumetric flask, dissolve in 100 mL of HCl, cool, dilute to volume, and mix.

15.5 *Arsenic Standard Solution B* (1 mL = 0.01 mg As)—Using a pipet, transfer 100 mL of Arsenic Standard Solution A (1 mL = 0.10 mg As) to a 1-L volumetric flask, dilute to volume, and mix.

15.6 *Hydrazine Sulfate*— $(\text{NH}_2)_2\text{H}_2\text{SO}_4$.

15.7 *Hydrazine Sulfate Solution* (1.5 g/L)—Dissolve 1.5 g of hydrazine sulfate ($(\text{NH}_2)_2\text{H}_2\text{SO}_4$) in water, dilute to 1 L, and mix. Do not use a solution that has stood more than 1 day.

15.8 *Sodium Carbonate* (Na_2CO_3).

15.9 *Sodium Peroxide* (Na_2O_2)—35 mesh or finer.

16. Preparation of Calibration Curve

16.1 Calibration Solutions:

16.1.1 Using pipets, transfer 1, 2, 5, 10, and 15 mL of Arsenic Standard Solution B (1 mL = 0.01 mg As) to 125-mL Erlenmeyer flasks.

16.1.2 Add 10 mL of HNO_3 and evaporate the solution to dryness on a hot plate. Bake for 30 min at 150 to 180°C. Remove from the hot plate. Add 45 mL of Ammonium Molybdate Hydrazine Sulfate Solution to each flask, warm gently to dissolve the residue, and transfer the solution to a 50-mL volumetric flask. Proceed as directed in 16.3.

16.2 *Reference Solution*—Transfer 10 mL of water to a 125-mL Erlenmeyer flask and proceed as directed in 16.1.2.

16.3 *Color Development*—Heat the flask in a boiling water bath for 15 min. Remove the flask, cool to room temperature, dilute to volume with Ammonium Molybdate-Hydrazine Sulfate Solution and mix.

16.4 Photometry:

16.4.1 *Multiple-Cell Photometer*—Measure the cell correction using the reference solution (16.2) in absorption cells with a 1-cm light path and a light band centered at approximately 850 nm. Using the test cell, take the photometric readings of the calibration solution versus the reference solution.

16.4.2 *Single-Cell Photometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 1-cm light path and adjust the photometer to the initial setting, using a light band centered at approximately 850 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

16.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of arsenic per 50 mL of solution.

17. Procedure

17.1 Test Solution:

17.1.1 Select and weigh a sample to the nearest 0.2 mg in accordance with the following:

Arsenic, %	Sample Weight, g
0.001 to 0.015	0.500
0.01 to 0.04	0.250
0.035 to 0.10	0.125

Transfer the sample to a 30-mL zirconium crucible containing 8 g of Na_2O_2 and 2 g of Na_2CO_3 .

17.1.2 Mix (Note 2) thoroughly with a metal spatula. Fuse carefully over a free flame by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down quietly; raise the temperature gradually to avoid spattering (Note 3). When the contents are molten, give the crucible a rotary motion to stir up any unattached particles of the alloy adhering to the bottom or sides. Finally, increase the temperature until the crucible is bright red for 1 min. Cool the crucible to room temperature. Transfer the crucible to an 800-mL beaker containing 60 mL of H_2SO_4 (1+1) and 200 mL of water. Dissolve the melt; remove and rinse the crucible.

NOTE 2—**Warning:** Use proper safety practices and equipment when performing sodium peroxide fusions.

NOTE 3—If the reaction proceeds violently with spattering because of too rapid heating, the use of insufficient Na_2CO_3 , or the lack of thorough mixing, appreciable loss may occur and the work should be repeated.

17.1.3 Add H_2SO_3 dropwise until the solution clears.

17.1.4 Heat to boiling, and cool. While stirring vigorously, add NH_4OH until the solution is alkaline to litmus, and then add 3 to 5 mL in excess. Heat to boiling, remove from the heat, and allow the precipitate to settle. Filter on a coarse filter paper and wash five times with hot water. Discard the filtrate. Remove the filter paper, carefully open it, and place it on the inside wall of the original 800-mL beaker. Wash the precipitate from the paper using a fine stream of water. Pass 25 mL of HNO_3 (1+1) over the paper, and wash well with water but do not exceed a total volume of 40 mL. Discard the paper. Warm gently until the precipitate dissolves.

17.1.5 Transfer the solution to the distillation flask, add 1 g of NH_4Br and 0.75 g of hydrazine sulfate. Add 20 mL of HNO_3 (1+1) to the receiving flask, and place the flask in an 800-mL beaker containing cold water. Assemble the apparatus (Fig. 1), heat the distillation flask, and distill into the receiving flask.

17.1.6 Distill until the volume is reduced to 10 mL or until oxides of nitrogen are noted in the distillation flask. Remove the distillation flask from the heat source. Place the receiving flask on a hot plate and evaporate the solution to dryness. Bake for 30 min at 150 to 180°C. Add 45 mL of Ammonium Molybdate-Hydrazine Sulfate Solution to the flask, warm gently to dissolve the residue, and transfer the solution to a 50-mL volumetric flask. Proceed as directed in 17.3.

17.2 *Reference Solution*—Carry a reagent blank through the entire procedure using the same amounts of all reagents with the sample omitted, for use as a reference solution.

17.3 *Color Development*—Proceed as directed in 16.3.

17.4 *Photometry*—Take the photometric reading of the test solution as directed in 16.4.

18. Calculation

18.1 Convert the net photometric reading of the test solution to milligrams of arsenic by means of the calibration curve.

Calculate the percentage of arsenic as follows:

$$\text{Arsenic, \%} = A/(B \times 10) \quad (1)$$

where:

A = arsenic found in 50 mL of final test solution, mg, and
 B = sample represented in 50 mL of final test solution, g.

19. Precision and Bias

19.1 *Precision*—Although samples covered by this test method were not available for testing, the precision data obtained for other types of ferroalloys, using the methods indicated in Table 1, should apply. 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.

19.2 *Bias*—No information on the bias of this test method is available.

LEAD BY THE DITHIZONE PHOTOMETRIC METHOD

20. Scope

20.1 This test method covers the determination of lead in ferromanganese in concentrations from 0.004 to 0.01 %.

20.2 The limits of the scope have been set at 0.004 to 0.01 % because test materials containing other lead 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 0.05 %.

20.2.1 Users of this test method are cautioned that its use on samples outside of the 0.004 to 0.01 % range is not supported by interlaboratory testing.

21. Summary of Test Method

21.1 After dissolution of the sample, lead is precipitated with ammonium hydroxide. Interfering metals are complexed with sodium citrate and sodium cyanide, and the lead dithizone complex is extracted with chloroform. Photometric measurement is made at 520 nm.

22. Concentration Range

22.1 The recommended concentration range is from 0.001 to 0.025 mg of lead per 10 mL of solution, using a 1-cm cell.

NOTE 4—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 amounts of sample and reagents used.

23. Stability of Color

23.1 The color is quite stable if the solution is protected against evaporation and decomposition of chloroform. Because

of the volatility of the solvent, it is advisable to make all readings promptly. The color develops almost immediately.

24. Interferences

24.1 The elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1. If more than 0.005 % bismuth is present, it must be removed as directed in Note 8 in order to avoid high results for lead.

25. Apparatus

25.1 *Glassware*—Use only borosilicate beakers, covers, and funnels. Wash all glassware with hot HNO₃ (1+1) and reserve for this determination only. Before using separatory funnels, rinse them with dithizone solution and then with water. Store all reagents in glass-stoppered borosilicate bottles which have been previously washed with hot HNO₃ (1+1) and rinsed with distilled water.

25.2 *pH Meter*—A pH meter for measurements to within ± 0.10 pH units is required.

26. Reagents

26.1 *Chloroform* (CHCl₃).

NOTE 5—**Warning:** Chloroform is highly toxic and is to be used in a well-ventilated hood. Consult the Material Safety Data Sheet or other source of data prior to use.

26.2 *Dithizone Solution* (0.04 g/L in chloroform)—Dissolve 0.02 g of dithizone (diphenylthiocarbazon) in 80 mL of CHCl₃ in a 500-mL conical separatory funnel, add 100 mL of cold water and 10 mL of NH₄OH, stopper, and shake vigorously for 1 to 2 min. Draw off the CHCl₃ layer and discard. Wash the aqueous layer with 5 mL of CHCl₃ and discard the latter. Add HCl (1+9) to the aqueous layer until it is just acidic to litmus paper, cool, and extract with three 50-mL portions of CHCl₃. Combine the CHCl₃ extracts, wash several times with water until the aqueous phase does not give an acid test with pH paper, and discard the aqueous layer. Dilute the CHCl₃ layer to 500 mL with CHCl₃ and store in an amber glass bottle preferably in a refrigerator.

26.3 *Lead Standard Solution* (1 mL = 0.001 mg Pb)—Dissolve 0.2000 g of lead (purity 99.9 % minimum) in 20 mL of HNO₃ (1+1), and heat moderately to expel oxides of nitrogen. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Using a pipet, transfer 5 mL of this solution to a 1-L volumetric flask, dilute to volume, and mix.

26.4 *Sodium Citrate Solution*—Dissolve 30 g of sodium citrate dihydrate in 100 mL of distilled water. Add NH₄OH until the pH is between 9.5 and 10.0. Add 10 mL of CHCl₃ and 1 mL of dithizone solution, and shake. If the CHCl₃ solution is red or gray, add a few drops more of the dithizone solution and shake again. Repeat until the color becomes green. Discard the organic layer and re-extract with a 10 mL portion of fresh CHCl₃. If the color becomes green, draw off the organic phase and then extract several times more with CHCl₃ until the aqueous phase is colorless and the CHCl₃ phase is almost colorless or very light green.

26.5 *Sodium Cyanide Solution* (300 g/L)—Dissolve 60 g of sodium cyanide (NaCN) in 200 mL of water. Store in a polyethylene bottle.

TABLE 1 Statistical Information—Arsenic Molybdenum Blue Photometric Method

Test Material	Arsenic Found, %	Repeatability (R_1 , Practice E 173)	Reproducibility (R_2 , Practice E 173)
1. 70Mn-16Si	0.025	0.001	0.002
2. 66Mn-16Si	0.059	0.001	0.002

NOTE 6—**Warning:** The preparation, storage, use and disposal of NaCN solutions requires special care and attention. Avoid any possibility of inhalation, ingestion, or skin contact with the compound, its solutions, or its vapors. Work only in a well-ventilated hood. Refer to the Safety Precautions section of Practices E 50.

NOTE 7—Because of the strongly alkaline properties of NaCN solutions, contact with borosilicate glass may result in contamination of the reagent.

26.6 *Sodium Sulfite Solution (Saturated)*—Prepare a saturated solution of sodium sulfite (Na₂SO₃).

26.7 *Wash Solution*—Add 10 mL of NH₄OH, 40 mL of Na₂SO₃ solution, and 20 mL of NaCN solution (**Warning**, Note 6) to 100 mL of water, and dilute to 1 L with water.

26.8 *Water*—Distilled water should be free of any lead salts. Low-quality water may be passed through a laboratory-type mixed-bed demineralizer prior to use.

27. Preparation of Calibration Curve

27.1 *Calibration Solutions*—Using pipets, transfer 1, 5, 10, 15, 20, and 25 mL of Standard Lead Solution (1 mL = 0.001 mg Pb) to 250-mL beakers and add enough water to make a total volume of approximately 25 mL. Proceed as directed in 27.3.

27.2 *Reference Solution*—Add 25 mL of water to a 250-mL beaker. Proceed as directed in 27.3.

27.3 Color Development:

27.3.1 In a well-ventilated hood, add 10 mL of sodium citrate solution, 10 mL of Na₂SO₃ solution, and 10 mL of NaCN solution (**Warning**, Note 6), heat at 80°C for 3 min, and cool. Using a pH meter, adjust the pH to 10.5 ± 0.2 with NH₄OH (1 + 1) or HCl (1 + 1) as required. Cool to 10°C and transfer to a 125-mL conical separatory funnel with a minimum of washing.

27.3.2 Using a pipet, transfer 10 mL of dithizone solution to the funnel, shake vigorously for 1 min, and allow the layers to separate. Draw off the lower CHCl₃ layer into a second 125-mL separatory funnel containing 50 mL of wash solution. Shake for 30 s, allow the layers to separate, and drain off the lower CHCl₃ layer into a third 125-mL separatory funnel containing 50 mL of wash solution. Shake for 30 s and allow the layers to separate thoroughly (Note 3). Eliminate water droplets in the CHCl₃ solution by transferring this solution to a clean, dry test tube before transferring to the absorption cell.

NOTE 8—If more than 0.005 % bismuth is present in the sample, the CHCl₃ layer should be back-washed with a solution of hydroxylamine hydrochloride (10 g/L) adjusted to a pH of 3.0.

27.4 Photometry:

27.4.1 *Multiple-Cell Photometer*—Measure the cell correction using the reference solution (27.2) in absorption cells with a 1-cm light path and using a light band centered at approximately 520 nm. Using the test cell, take photometric readings of the calibration solutions versus the reference solution (27.2).

27.4.2 *Single-Cell Photometer*—Transfer a suitable portion of the reference solution (27.2) to an absorption cell with a 1-cm light path and adjust the photometer to the initial setting, using a light band centered at approximately 520 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

27.5 *Calibration Curve*—Plot the net photometric readings

of the calibration solutions against milligrams of lead per 10 mL of solution.

28. Procedure

28.1 Test Solution:

28.1.1 Select a sample in accordance with the following:

Lead, %	Sample Weight, g	Dilution, mL	Aliquot Volume, mL
0.001 to 0.01	1.000	100	25
0.01 to 0.025	1.000	250	25
0.025 to 0.05	0.500	250	10

Weigh the sample to the nearest 0.1 mg and transfer it to a 250-mL beaker.

28.1.2 Add 15 mL of HCl and 15 mL of HNO₃, and heat until dissolution is nearly complete.

28.1.3 Add 20 drops of HF and 10 mL of HClO₄. Evaporate to heavy white fumes, fume until the volume is approximately 5 mL, and cool. Add H₂O₂ solution (1+9) dropwise with stirring until any precipitated manganese dioxide is dissolved. Boil to remove excess H₂O₂, and cool.

28.1.4 Dilute to approximately 100 mL, add NH₄OH (1+1) until the solution is neutral to litmus paper (Note 9), and add 10 mL in excess. Boil for approximately 1 min, and cool.

NOTE 9—If the sample does not contain sufficient iron, add a volume of iron solution equivalent to about 100 mg of iron to act as a carrier, and then adjust the pH again. Prepare the iron solution as follows: Dissolve 1 g of iron (lead content 0.001 % maximum) in 10 mL of HCl (1+1) and 10 mL of HNO₃. Add 10 mL of HClO₄, heat to strong fumes, cool, and dilute to 100 mL.

28.1.5 Filter using a medium paper and wash 3 or 4 times with NH₄OH (1+9). Discard the filtrate. Dissolve the precipitate with 30 mL of HCl (1+9) into the original 250-mL beaker, and wash the paper 6 to 8 times with hot HCl (2+98). Add 10 mL of HNO₃ and 10 mL of HClO₄ to the beaker and evaporate to approximately 5 mL, and cool.

28.1.6 Transfer the solution to the appropriate volumetric flask, selected in accordance with 28.1.1, dilute to volume, and mix. In accordance with 28.1.1, and using a pipet, transfer a suitable aliquot to a 250-mL beaker. Proceed as directed in 28.3.

28.2 *Reference Solution*—Carry a reagent blank through the entire procedure using the same amounts of all reagents but with the sample omitted. Proceed as directed in 28.3.

28.3 *Color Development*—Proceed as directed in 27.3.

28.4 *Photometry*—Proceed as directed in 27.4.

29. Calculation

29.1 Convert the net photometric reading of the test solution to milligrams of lead by means of the calibration curve. Calculate the percentage of lead as follows:

$$\text{Lead, \%} = A/(B \times 10) \quad (2)$$

where:

A = lead found in 10 mL of the final test solution, mg, and
 B = sample represented in 10 mL of the final test solution, g.

30. Precision and Bias

30.1 *Precision*—Five laboratories cooperated in testing this

test method and obtained the data summarized in Table 2. The user is cautioned to verify by the use of reference materials, if

available, that the precision and bias of this test method is adequate for the contemplated use.

30.2 *Bias*—No information on the bias of this test method is available.

TABLE 2 Statistical Information—Lead Dithizone Photometric Method

Test Method	Lead Found	Repeatability (R_1 , Practice E 173)	Reproducibility (R_2 , Practice E 173)
1.78 Mn-7C	0.0052	0.0010	0.0021

31. Keywords

31.1 chemical analysis; ferromanganese

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