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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron[1]

This standard is issued under the fixed designation E 30; 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 procedures for the chemical analysis of steel, cast iron, open-hearth iron, and wrought iron having chemical compositions as specified in Test Methods E 350, E 351, E 352, E 353, and E 354.

1.2 The analytical procedures appear in the following sections:

	Sections
Beryllium by the Oxide Test Method	78 to 82
Chromium by the Colorimetric Method	[2]
Molybdenum:	
Alpha-Benzoinoxime Test Method	41 to 47
Precipitation as Sulfide and Weighing as Oxide	48 to 55
Phosphorus by the Molybdate-Magnesia Test Method	7 to 17
Titanium by the Photometric Test Method	62 to 70
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Vanadium:	
Electrolytic Separation Test Method	25 to 31
Reduction with Ferrous Sulfate and Titration with Permanganate	32 to 40
Zirconium by the Cupferron-Phosphate Test Method	71 to 77

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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.* For precautions to be observed, refer to Practices E 50. Also refer to specific precautions in the methods.

2. Referenced Documents

2.1 *ASTM Standards:*

E 29 Recommended Practice for Indicating Which Places of Figures Are to Be Considered Significant in Specified Limiting Values[3]
E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]
E 59 Method of Sampling Steel and Iron for Determination of Chemical Composition[3]
E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[3]
E 350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron[3]
E 351 Test Methods for Chemical Analysis of Cast Iron—All Types[3]
E 352 Test Methods for Chemical Analysis of Tool Steels and Other Similar Medium- and High-Alloy Steels[3]
E 353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys[3]
E 354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys[3]

3. Significance and Use

3.1 These test 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 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.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination and 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.

5. Sampling

5.1 For procedures for sampling the material, reference shall be made to Method E 59.

6. Rounding Calculated Values

6.1 Calculated values shall be rounded to the desired number of places as directed in Recommended Practice E 29.

PHOSPHORUS BY THE MOLYBDATE-MAGNESIA TEST METHOD

7. Scope

7.1 This test method covers the determination of phosphorus in concentrations from 0.01 to 0.90 %.

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[1] These test methods are under the jurisdiction of ASTM Committee E-3 on Chemical Analysis of Metals and are the direct responsibility of Subcommittee E03.01 on Ferrous Metals.

These test methods are being revised and issued under new ASTM designations E 350, E 351, E 352, E 353, and E 354.

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[2] Discontinued as of April 28, 1989.

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

8. Summary of Test Method

8.1 The sample is dissolved in acid and carbonaceous residues are oxidized with permanganation. A solution of ammonium molybdate is added to precipitate the phosphorus as ammonium phosphomolybdate. The solution is filtered and the precipitate is dissolved in an ammonium hydroxide-citric acid solution. The phosphorus is then precipitated with a magnesium sulfate solution. The residue is purified, filtered, ignited, and weighed as magnesium pyrophosphate.

9. Reagents

9.1 *Ammonium Molybdate Solution (Ammoniacal)*—Reagent No. 101.

9.2 *Ammonium Nitrate Wash Solution*—Dissolve 50 g of NH_4NO_3 in 1 L of NH_4OH (1+20).

9.3 *Ferrous Sulfate Solution*—Dissolve 100 g of $FeSO_4 \cdot 7H_2O$ in 1 L of H_2SO_4 (5+95).

9.4 *Magnesia Mixture*—Dissolve 130 g of $MgSO_4 \cdot 7H_2O$ and 75 g of $(NH_4)_2SO_4$ in 500 mL of water. Add NH_4OH in slight excess and let stand overnight. Filter if a precipitate appears. Make just acid by adding a very slight excess of H_2SO_4, dilute to 1 L, and keep in a glass-stoppered bottle.

9.5 *Potassium Permanganate Solution* (25 g/L).

10. Procedure for Carbon Steels

10.1 Transfer 3 g of the sample (Note 1) to a 300-mL Erlenmeyer flask, dissolve the sample in 125 mL of HNO_3 (1+3), add 10 mL of $KMnO_4$ solution (25 g/L), and boil for 3 to 5 min. If no precipitate forms, add more $KMnO_4$ solution and boil again. Dissolve the precipitate by the addition of H_2SO_3 drop by drop, and boil for a few minutes to expel oxides of nitrogen. Cool to about 75°C and add 85 mL of ammoniacal ammonium molybdate solution. Stopper the flask, shake for 10 min, and allow to stand for 2 h or more. Filter through a close-texture paper. Wash the flask, precipitate, and paper six to eight times with cold HNO_3 (2+98).

NOTE 1—If the phosphorus content is less than 0.05 %, it is advantageous to treat two or more 3-g samples as described and combine the yellow precipitates either by filtering through the same paper or by combining the precipitates after filtering on separate papers.

10.2 Set the filtrate and washings aside after thorough mixing, and note whether further separation of phosphomolybdate occurs. Dissolve the precipitate on the filter and in the flask in a mixture of 20 mL of NH_4OH (1+1) and 2 g of citric acid, catching the solution in a 250-mL beaker. Wash the filter several times with NH_4OH (1+20), then with hot water, and finally several times with HCl (1+20). If the ammoniacal solution of the phosphomolybdate is not clear at this point, heat it to boiling and filter through the same paper. Wash the paper with hot water, ignite in a small platinum crucible, and fuse any residue with as little Na_2CO_3 as possible (not over 0.5 g). Extract the cooled melt with hot water, cool, filter, and add the water solution to the ammoniacal solution.

10.3 Acidify the combined solutions with HCl, add 20 mL of magnesia mixture, cool in ice water, and then add NH_4OH slowly until the solution is just ammoniacal. Stir vigorously for 5 min or until a crystalline precipitate appears (Note 2), and add 5 to 10 mL of NH_4OH. The volume of the solution at this point should not exceed 100 mL. Allow the solution to stand in a cool place for 4 to 6 h or, preferably, overnight. Filter and wash the precipitate moderately with NH_4OH (1+20).

NOTE 2—If the amount of phosphorus is very small, it may take 30 min to 1 h before the precipitate begins to appear.

10.4 Dissolve the precipitate on the filter in 20 mL of HCl (1+1), catching the solution in the original beaker. Wash the filter thoroughly with warm HCl (1+1). Add 0.5 to 1 g of NH_4Br (Note 3) and gently boil the solution to a volume of 5 to 10 mL (but not to dryness) to eliminate arsenic. Dilute to 50 to 75 mL, add 0.1 to 0.2 g of citric acid and 2 to 3 mL of magnesia mixture, and cool in ice water. Make ammoniacal, stir as before, and allow to stand in a cool place for 4 to 24 h.

NOTE 3—With a precipitation temperature of 25°C arsenic, when present in small amounts, is not precipitated and therefore the treatment with NH_4Br can be omitted.

10.5 Filter, and wash with NH_4NO_3 wash solution. Ignite the precipitate in a weighed platinum crucible, carefully and at as low a temperature as possible, until the carbon has been destroyed and the residue is white. Finally, ignite to constant weight at 1000 to 1050°C.

10.6 Dissolve the ignited precipitate in 5 mL of HNO_3 (1+1) and 20 mL of water. If no residue remains, the ignited precipitate may be regarded as $Mg_2P_2O_7$. If a residue remains, filter, wash with hot water, ignite, and weigh. Add a few drops of HF and 1 to 2 drops of H_2SO_4 (1+1). Evaporate to dryness, ignite at 1000°C, and weigh again. The loss in weight represents the correction for impurities.

10.7 *Calculation*—Calculate the percentage of phosphorus as follows:

$$\text{Phosphorus, \%} = [((A - B) \times 0.278)/C] \times 100$$

where:

A = grams of $Mg_2P_2O_7$ (10.5),
B = correction for impurities (10.6), g, and
C = grams of sample used.

11. Procedure for Nickel, Chromium-Nickel, Stainless, and Similar Alloy Steels Containing Neither Tungsten nor Vanadium

11.1 Determine phosphorus in accordance with the procedure described in Section 10.

11.2 High-chromium steels (stainless), high-chromium, high-nickel steels (18 % chromium, 8 % nickel; 20 % chromium, 20 % nickel), and other steels in this group that do not dissolve in HNO_3 (1+3), shall be treated as follows: Transfer 3 g of the sample to a 500-mL Erlenmeyer flask, add 75 mL of a mixture of equal parts of HCl and HNO_3, and heat gently. When decomposition is complete, add 20 mL of $HClO_4$ and evaporate to white fumes. Continue the heating for 5 min to oxidize chromium and to dehydrate SiO_2. Cool somewhat, add 40 mL of water, and filter. Wash the flask, paper, and residue with 55 mL of HNO_3 (3+5). Add 10 mL of $KMnO_4$ solution (25 g/L) to the filtrate, boil 3 to 5 min, add H_2SO_3 to destroy oxides of manganese and to reduce all of the chromium, and complete the determination as described in Section 10.

12. Procedure for Austenitic Manganese Steels (Over 10 % Manganese)

12.1 Transfer 3 g of the sample to a 300-mL Erlenmeyer flask, add 70 mL of HNO_3 (1+3), and digest until action ceases. Add 30 mL of $HClO_4$ and evaporate just to fumes. Add HF drop by drop until all of the SiO_2 is in solution and then an excess of 5 drops. Fume so that the $HClO_4$ refluxes on the sides of the flask for 25 to 30 min. Cool, and add 50 mL of water and 10 mL of HNO_3. Add a few drops of $KMnO_4$ solution (25 g/L) and boil until oxides of manganese are precipitated. Dissolve the precipitated manganese with $H2SO_3$, and boil a few minutes to expel oxides of nitrogen. Cool to about 75 °C, precipitate with ammonium molybdate, and complete the determination as described in Section 10.

13. Procedure for Chromium-Vanadium Steels or Other Steels Containing Vanadium but No Tungsten

13.1 Proceed as described in 12.1 until the solution is ready for the addition of the molybdate reagent. At this point, cool to 10°C and add 5 mL of $FeSO_4$ solution and 2 to 3 drops of H_2SO_3. Mix, add 85 mL of ammonium molybdate solution, shake for 10 min, and allow to stand for 4 h or, preferably, overnight.

13.2 Filter and complete the determination as described in Section 10.

14. Procedure for High-Speed Steels or Other Steels Containing Tungsten and Vanadium

14.1 Decompose 3 g of the sample in 125 mL of HNO_3 (1+3), and 30 mL of HCl, and evaporate to dryness. Dissolve the residue with 20 mL of HCl (1+1), dilute to 100 mL with hot water, and filter off the tungstic acid.

NOTE 4—This precipitate may contain a small amount of phosphorus, and in standardization work shall be treated as follows: Transfer the bulk of the precipitate to an Erlenmeyer flask with a jet of water, and then treat the paper with 25 mL of hot NH_4OH (1+4) containing 0.5 g of citric acid. Catch the filtrate in the flask containing the remainder of the precipitate. Wash the paper with hot water, then a few times with hot HCl (1+20), holding the volume of the filtrate and washings to about 75 mL. Slightly acidify the solution with HCl, add 25 mL of magnesia mixture, and add about 10 mL of NH_4OH in excess. Add a few glass beads, cool the solution in ice water, stopper the flask, and shake thoroughly for 1 h. Allow to stand overnight at about 0°C. Filter, and wash a few times with NH_4OH (1+20). Dissolve the precipitate in a little hot HNO_3 (1+3) and add it to the original solution in which phosphorus is to be determined, or determine the phosphorus separately by precipitating it with ammonium molybdate solution and titrating with standard NaOH and HNO_3. This recovery usually amounts to less than 0.001 % and need not be made in routine analyses.

14.2 Evaporate the filtrate twice with 20-mL portions of HNO_3 to expel the HCl, taking the second evaporation just to a sirup. Add 65 mL of HNO_3 (1+3), and filter the solution if it is not entirely clear. Cool to 10°C. Add 5 mL of $FeSO_4$ solution and 2 to 3 drops of H_2SO_3. Mix, add 85 mL of ammonium molybdate solution, shake for 10 min, and allow to stand for 4 h or, preferably, overnight.

14.3 Filter and complete the determination as described in Section 10.

15. Procedure for Cast Iron and High-Silicon Steels

15.1 Dissolve 2 to 3 g of the sample in a covered casserole in 30 mL of HNO_3 (1+1). When solution is complete, add 10 mL of HCl (1+1), evaporate to dryness, and bake for 15 to 20 min on the hot plate.

15.2 Cool, drench the residue with HCl, dilute to 50 mL with hot water, and warm until the salts are in solution. Filter without delay, wash with HCl (1+20), and evaporate the filtrate to sirupy consistency.

15.3 In the meantime, place the paper containing the graphite and silica in a platinum crucible, burn the carbon in a good oxidizing atmosphere, cool, and add 5 to 10 drops of HCl and 1 to 2 mL of HF. Evaporate just to dryness, take up any residue (Note 5) in 5 mL of HCl, and add the solution to the main solution which is being evaporated.

NOTE 5—If the percentage of titanium or zirconium is high, this residue should be fused with Na_2CO_3, extracted with hot water, cooled, filtered, and the water extract added to the main solution.

15.4 When the solution is of syrupy consistency, transfer it to a 300-mL Erlenmeyer flask by alternate washing with HNO_3 (1+1) and hot water using about 40 mL of HNO_3 (1+1) and 70 mL of hot water. Adjust the temperature of the solution to about 70°C, and add 100 mL of ammonium molybdate solution. Shake for 10 min and allow to stand for 4 to 6 h or, preferably, overnight.

15.5 Filter and complete the determination as described in Section 10.

16. Procedure for Open-Hearth Iron and Wrought Iron

16.1 Determine phosphorus in accordance with the procedure described in Section 10.

17. Precision and Bias

17.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify by the use of certified reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

CHROMIUM BY THE COLORIMETRIC METHOD

(This method which consisted of Sections 18 through 24 of this standard, was discontinued in 1989.)

VANADIUM BY THE ELECTROLYTIC SEPARATION TEST METHOD

25. Scope

25.1 This test method covers the determination of vanadium in concentrations from 0.02 to 2.00 %.

26. Summary of Test Method

26.1 The sample is dissolved in sufuric acid and the vanadium is precipitated with sodium bicarbonate. The precipitate is filtered, dissolved with acid, and electrolyzed by mercury cathode. The resultant solution is treated with sulfur dioxide to reduce the vanadium after which the sample is titrated with standard potassium permanganate. The amount of vanadium present is calculated from the volume of standard permanganate consumed.

27. Apparatus

27.1 *Mercury Cathode Cell*—Apparatus No. 10A or 10B.

28. Reagents

28.1 *Potassium Ferricyanide Solution*—Prepare a very dilute solution as needed by dissolving a crystal the size of the head of a pin in 25 mL of water.