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Standard Test Methods for Rubber from Natural Sources—Chemical Analysis[1]

This standard is issued under the fixed designation D1278; 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 sampling and chemical analysis of solid natural rubber in the forms supplied to the rubber industry.

1.2 The analytical procedures appear in the following order:

	Sections
Sampling	5
Volatile Matter	6 – 8
Dirt	9 – 13
Ash	14 – 17
Copper (Referee Colorimetric Method)	18 – 22
Copper (Alternative Colorimetric Method)	23 – 27
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Manganese (Colorimetric Method)	29 – 33
Manganese (Alternative Flame Atomic Absorption Method)	34
Iron (Colorimetric Method)	35 – 39
Acetone Extract	40 – 41
Rubber Hydrocarbon	42 – 43
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1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D297 Test Methods for Rubber Products—Chemical Analysis

D1193 Specification for Reagent Water

D1485 Practice for Rubber from Natural Sources—Sampling and Sample Preparation

D3533 Test Method for Rubber—Nitrogen Content (Withdrawn 2006)[3]

D4004 Test Methods for Rubber—Determination of Metal Content by Flame Atomic Absorption (AAS) Analysis

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E131 Terminology Relating to Molecular Spectroscopy

3. Significance and Use

3.1 These test methods are intended for quality control acceptance of natural rubber and may be used for referee purposes.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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 higher purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

5. Sampling

5.1 A lot of natural rubber shall be sampled, the sample prepared for test, and the acceptability of the lot determined from tests on the sample in accordance with Test Methods D1485.

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[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.22 on Natural Rubber.

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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] *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 *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

VOLATILE MATTER

6. Procedure

6.1 Weigh a 10 to 12-g specimen of homogenized rubber to the nearest 1 mg and then dry it in a circulating-air oven at 100 ± 5°C to constant mass. If it is suspected that volatile hydrocarbon oils are present, they should be determined by heating the rubber in a circulating-air oven at 160 ± 5°C to constant weight. The drying is facilitated by cutting the rubber into 25-mm strips having a maximum width and thickness of 2.5 by 1.25 mm or by passing it through a laboratory mill with a clearance between rolls set at 0.5 ± 0.1 mm (0.020 ± 0.004 in.). Keep the dried specimen in a covered weighing vessel or in a desiccator after its removal from the oven until it is ready to be weighed.

7. Calculation

7.1 Calculate the percentage of volatile matter as follows:

$$V = \left[1 - (B \times D)/(A \times C) \right] \times 100 \quad (1)$$

where:

V = percentage of volatile matter,
A = mass of piece taken from bale,
B = mass of piece after homogenizing,
C = mass of specimen before oven drying, and
D = mass of specimen after oven drying.

8. Precision and Bias

8.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

DIRT

9. Apparatus

9.1 *Sieve*—A45-µm (No. 325) sieve conforming to Specification E11.

10. Reagents

10.1 *Petroleum, Light,* boiling between 60 and 80°C.

10.2 *Rubber Peptizing Agent.*

NOTE 1—The peptizing agent selection is not critical but, where one is used that contains inert mineral filler, the peptizer should be added to the rubber solvent and filtered[5] prior to addition of the rubber.

10.3 *Rubber Solvent, Xylene, or a Hydrocarbon Solvent,* with a distillation range within 135 to 220°C.

11. Procedure

11.1 Weigh a 10 to 12-g specimen of homogenized rubber to the nearest 0.1 g and cut into pieces having a maximum dimension of less than 3 mm. Place the pieces in a 250-cm^3 conical flask and cover with 150 cm^3 of rubber solvent containing about 0.5 g of peptizing agent. Heat the mixture and maintain it at a temperature of 125 to 130°C (Note 2) until dissolution is complete (about 3 h).

NOTE 2—Overheating or boiling may cause gelling or charring. Infrared heating lamps and magnetic stirring are aids that will induce rubber solution.

11.2 Pour the hot solution through a 45-µm (No. 325) sieve previously weighed to the nearest 0.1 mg. Rinse the flask three times with about 25 cm^3 of hot rubber solvent and pour the rinsings through the sieve. Transfer any dirt remaining in the flask to the sieve by means of a jet of light petroleum and wash the dirt on the sieve until free of rubber solution. Dry the sieve and contents at 100 ± 5°C and weigh to the nearest 0.1 mg.

12. Calculation

12.1 Calculate the dirt content as follows:

$$D = \left[(C - B)/A \right] \times 100 \quad (2)$$

where:

D = percentage of dirt,
A = mass of the specimen,
B = mass of the clean, dry sieve, and
C = mass of the sieve plus dirt.

13. Precision and Bias

13.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

ASH

14. Apparatus

14.1 *Crucible*—An unetched porcelain crucible having a capacity of 50 cm^3. If copper is subsequently to be determined, a smooth unetched silica crucible is preferred, but a Vycor crucible or an ignited, acid-washed unetched No. 2 Coors porcelain crucible may be used.

NOTE 3—In cases of dispute where the greatest accuracy is required, use a new, smooth silica crucible each time the test is run.

14.2 *Muffle Furnace,* with temperature indicator and control.

14.3 *Filter Paper, Ashless,* about 150 mm in diameter.

15. Procedure

15.1 Weigh a 5 to 6-g specimen of homogenized rubber to the nearest 1 mg and place it in a crucible previously ignited and weighed to the nearest 0.1 mg. Place the crucible and its contents in a furnace controlled at a temperature of 550 ± 25°C until free from carbon (Note 4). When ashing is complete, cool the crucible in a desiccator and then weigh it to the nearest 0.1 mg.

NOTE 4—The rubber may be charred over a small flame or on a hot plate before it is placed in the furnace. When the rubber is not previously charred before placing it in the furnace, the crucibles shall be placed on a suitable tray to permit placing them in the furnace simultaneously, and the door of the furnace shall then be kept closed for at least 1 h while

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[5] The sole source of supply of the apparatus (Number 40 Watman filter paper) known to the committee at this time is Fischer Scientific, 1600 W. Glenlake Ave., Itasca, IL 60143. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

flammable vapors are evolved. If copper, manganese, or iron is to be determined, the specimen shall be wrapped in a 150-mm ashless filter paper previous to ashing.

16. Calculation

16.1 Calculate the ash content as follows:

$$A = [(C - B)/D] \times 100 \quad (3)$$

where:

A = percentage of ash,
D = mass of the specimen,
B = mass of the empty crucible, and
C = mass of the crucible plus ash.

17. Precision and Bias

17.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

COPPER

(Referee Colorimetric Method)[6]

18. Apparatus

18.1 *Photometer*—A spectrophotometer or a photoelectric photometer with a band pass filter having maximum transmittance at about 435 nm. Matched absorption cells, 1 to 5 cm in path length may be used, however, cells 2 to 5 cm in path length are preferred.

NOTE 5—For definitions of terms used in this procedure, refer to Terminology E131. The cm unit is not a preferred submultiple of the metre but it is used by Committee E13 on Molecular Microscopy. (See Terminology E131).

19. Reagents and Materials

19.1 *Carbon Tetrachloride* (CCl_4).

19.2 *Copper Sulfate, Standard Solution* (1 cm^3 = 0.1 mg Cu)—Dissolve 0.393 g of copper sulfate ($CuSO_4 \cdot 5H_2O$) in water, add 3 cm^3 of concentrated sulfuric acid (H_2SO_4, density = 1.84 Mg/m^3), and dilute to 1 cm^3 with water. This solution should remain stable for at least a month.

19.3 *Copper Sulfate, Standard Solution* (1 cm^3 = 0.01 mg Cu)—Dilute 10 cm^3 of the $CuSO_4$ solution (1 cm^3 = 0.1 mg Cu) to 100 cm^3 with water. Make up this solution fresh each day.

19.4 *Nitric Acid* (1 + 2)—Mix 1 volume of concentrated nitric (HNO_3, density = 1.42 Mg/m^3) with 2 volumes of water.

19.5 *Zinc Dibenzyldithiocarbamate Solution*[7]—Dissolve 0.1 g of zinc dibenzyldithiocarbamate in 1 cm^3 of CCl_4.

NOTE 6—The commercial grade of zinc dibenzyldithiocarbamate[7] is usually suitable for use as a reagent. Lower blanks and more rapid extractions, however, may be obtained with the purified reagent. A method of preparation of pure reagent is available in the literature.[6]

20. Preparation of Calibration Curve

20.1 Pipet 0, 1, 2, 3, and 4-cm^3 separate portions of standard $CuSO_4$ solution (1 cm^3 = 0.01 mg Cu) into separate separatory funnels. Add 5 cm^3 of HNO_3 (19.4) to each funnel and dilute to about 100 cm^3. With a pipet add 25 cm^3 of zinc dibenzyldithiocarbamate solution to each funnel if 2 to 5-cm absorption cells are to be used, or 10 cm^3 of the solution if 1-cm cells are to be used.

20.2 Proceed with the extraction as described in 21.1 and measure the absorbance of the CCl_4 solution as described in 21.1, using the solution with no added copper as the reference solution.

20.3 Prepare a calibration curve by plotting the relationship between copper concentration and absorbance. The calibration curve should be checked whenever necessary, depending on local conditions and on the type of instrument used.

21. Procedure

21.1 Ash a 10-g specimen wrapped in filter paper in accordance with Section 15. Ash a blank consisting of the filter paper in the same manner and carry it through the procedure in the same manner as the specimen. Add 10 cm^3 of HNO_3 (19.4) to the crucible and digest the mixture on a steam bath for 30 min. Transfer the solution to a 100-cm^3 volumetric flask. Cool and dilute it to the 100-cm^3 mark with water. Pipet 50 cm^3 of the solution into a separatory funnel and dilute to about 100 cm^3 with water. With a pipet add 25 cm^3 of zinc dibenzyldithiocarbamate solution if 2 to 5-cm absorption cells are to be used or add 10 cm^3 of the solution if the 1-cm cells are to be used. Shake the mixture vigorously for 1 min, allow the layers to separate, and draw off the CCl_4 layer through a funnel containing a plug of absorbent cotton, directly into the absorption cell. Leave a small amount of CCl_4 in the separatory funnel so as to avoid introducing water into the cell. If the reagents are sufficiently pure, this one extraction is sufficient. Each new reagent solution shall be tested. If a second successive extraction removes additional copper, it will be necessary to make additional extractions and combine the extracts. In this case combine the extracts in a 50-cm^3 volumetric flask and dilute with the reagent to the 50-cm^3 mark.

21.2 Measure the absorbance with a spectrophotometer at 435 nm or, if a photoelectric photometer is used, by using the appropriate filter. Use the blank solution as the reference solution. Determine the concentration of copper in the test solution from the absorbance reading and the calibration curve (20.3). Express the result in milligrams of copper per kilogram of rubber (parts per million).

22. Precision and Bias

22.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

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[6] Martens, R. I., and Githens, Sr., R. E., "Small Amounts of Copper in Dyes and Rubber Chemicals," *Analytical Chemistry*, Vol 24, 1952, pp. 991–3. Reprinted in *Rubber Chemistry and Technology*, Vol 26, 1953, pp. 257–262.

[7] The sole source of supply of this salt (trade name Arazate) known to the committee at this time is Uniroyal Chemical, Elm Street, Naugatuck, CT 06770. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

COPPER
(Alternative Colorimetric Method)

23. Apparatus

23.1 See Section 18 and Note 4.

24. Reagents and Materials

24.1 *Ammonium Hydroxide* (density = 0.90 Mg/m^3)—Concentrated ammonium hydroxide (NH_4OH).

24.2 *Chloroform* ($CHCl_3$).

24.3 *Citric Acid Solution* (33.33 %)—Dissolve 50 g of citric acid in 100 cm^3 of water.

24.4 *Copper Sulfate, Standard Solution* (1 cm^3 = 0.1 mg Cu)—See 19.2.

24.5 *Copper Sulfate, Standard Solution* (1 cm^3 = 0.01 mg Cu)—See 19.3.

24.6 *Hydrochloric Acid-Nitric Acid Mixture*—Mix 2 volumes of concentrated hydrochloric acid (HCl, density = 1.19 Mg/m^3) with 1 volume of concentrated nitric acid (HNO_3, density = 1.42 Mg/m^3) and 3 volumes of water.

24.7 *Sodium Sulfate, Anhydrous* (Na_2SO_4).

24.8 *Zinc Diethyldithiocarbamate Reagent*—Dissolve 1 g of sodium diethyldithiocarbamate in water and add 2 g of zinc sulfate ($ZnSO_4{\cdot}7H_2O$). Extract the resulting zinc diethyldithiocarbamate with 100 cm^3 of chloroform. Separate the chloroform layer and dilute it to 1 cm^3 with chloroform. The reagent is stable for 6 months if stored in an amber-colored bottle.

25. Preparation of Calibration Curve

25.1 Make up a series of solutions each containing 10 cm^3 of hydrochloric acid-nitric acid mixture and 5 cm^3 of citric acid solution. To these solutions add portions of copper solution (1 cm^3 = 0.01 mg Cu) ranging from 0 to 10 cm^3. Neutralize by dropwise addition of ammonium hydroxide using litmus paper and add about 2 cm^3 in excess. Cool to room temperature, transfer to a separatory funnel, and adjust the volume to about 40 cm^3. Pipet 25 cm^3 of zinc diethyldithiocarbamate reagent into each funnel and proceed with the extraction, separation, and measurement of absorbance exactly as described in 20.2 and 20.3, using the solution with no added copper as the reference solution. Prepare a calibration curve by plotting the relationship between copper concentration and absorbance. The calibration curve should be checked whenever necessary, depending on local conditions and on the type of instrument used.

26. Procedure

26.1 Ash a 5-g specimen as described in Sections 14 and 15. Ash a blank consisting of the filter paper in the same manner and carry it through the procedure in the same manner as the specimen. Moisten the ash with 0.5 to 1 cm^3 of water, add 10 cm^3 of the hydrochloric acid mixture, (24.6) cover the crucible with a watch glass, and digest on a steam bath for 30 to 60 min.

26.2 Wash the contents of the crucible into a small beaker with water. Add 5 cm^3 of citric acid solution and make the solution alkaline by dropwise addition of NH_4OH using litmus paper. Add about 2-cm^3 of NH_4OH in excess. Cool the solution to room temperature, transfer to a separatory funnel, and dilute to about 40 cm^3. Pipet 25 cm^3 of zinc diethyldithiocarbamate reagent into the funnel and shake vigorously for 2 min, taking care not to lose any of the chloroform solution. Separate the chloroform layer into a dry flask containing 0.1 g of anhydrous Na_2SO_4. If the solution is turbid, stopper the flask and allow it to stand. If turbidity persists after 30 min, make further small additions of Na_2SO_4 until the solution is clear.

26.3 Filter the solution through a plug of glass wool or a filter paper directly into the absorption cell of the spectrophotometer or photoelectric photometer. Measure the absorbance at 435 nm with a spectrophotometer or, if a photoelectric photometer is used, by using the appropriate filter. Use the blank solution as the reference solution. Determine the concentration of copper in the test solution from the absorbance reading and the calibration curve (Section 25). Express the results in milligrams of copper per kilogram of rubber (parts per million).

27. Precision and Bias

27.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

COPPER
(Alternative Flame Atomic Absorption Method)

28. Procedure

28.1 See Test Methods D4004, Method D, Copper.

MANGANESE
(Colorimetric Method)

29. Apparatus

29.1 *Photometer*—A spectrophotometer or filter photometer suitable for measurements at approximately 525 nm with absorption cells 1 to 5 cm in path length (Note 5). The 5-cm cells are preferred.

30. Reagents

30.1 *Manganese Sulfate, Standard Solution* (1 cm^3 = 1 mg Mn)—Dissolve 0.77 g of manganese sulfate ($MnSO_4{\cdot}H_2O$) in water, add 2 cm^3 of sulfuric acid (H_2SO_4, density = 1.84 Mg/m^3), and dilute to 250 cm^3 with water. This solution should be stable for at least a month.

30.2 *Managense Sulfate, Standard Solution* (1 cm^3 = 0.02 mg Mn)—Dilute 10 cm^3 of the $MnSO_4$ solution (1 cm^3 = 1 mg Mn) to 500 cm^3 with water. Make up this solution fresh each day.

30.3 *Orthophosphoric Acid* (85 to 90 %)—Concentrated orthophosphoric acid (H_3PO_4).

30.4 *Potassium Hydrogen Sulfate* ($KHSO_4$).

30.5 *Potassium Periodate* (KIO_4).

30.6 *Potassium Permanganate Rinse Solution* (0.03 g/cm^3)—Dissolve 0.03 g of potassium permanganate ($KMnO_4$) in water and dilute to 1 cm^3.

30.7 *Sulfuric Acid* (1 + 19)—Mix 1 volume of concentrated sulfuric acid (H_2SO_4, density = 1.84 Mg/m^3) with 19 volumes of water.

31. Preparation of Calibration Curve

31.1 Prepare a series of standard solutions by diluting portions of $MnSO_4$ solution (1 cm^3 = 0.02 mg Mn) ranging from 0 to 20 cm^3 to about 25 cm^3 with water, adding 20 cm^3 H_2SO_4 (30.7) and 0.3 g KIO_4 (Note 7). Heat each solution as described in 32.3.

NOTE 7—If the expected manganese content is below 20 mg/kg (ppm), the calibration curve need not extend beyond 10 cm^3 of standard $MnSO_4$ solution.

31.2 Cool the solutions, transfer to 50-cm^{3}volumetric flasks, and dilute to volume. Measure the absorbance at approximately 525 nm, using the solution to which no manganese was added as the reference solution. Use absorption cells having the same path length and shape as used in 32.4.

31.3 Prepare a calibration curve by plotting the relationship between manganese concentration and absorbance. The calibration curve should be checked whenever necessary, depending on local conditions and on the type of instrument used.

32. Procedure

32.1 Wrap to a 10 to 12-g specimen in a 150-mm ashless filter paper, place 5 g of $KHSO_4$ on top of the specimen in a porcelain crucible, and ash in accordance with Section 15. Ash a blank consisting of the filter paper and the $KHSO_4$ in the same manner and carry it through the procedure in the same manner as the sample.

32.2 Add 20 cm^3 of H_2SO_4 (30.7) to the crucible and heat the crucible on a steam bath for 30 min, crushing the residue occasionally with a glass rod to facilitate dissolution. Filter the solution into a 150-cm^3 beaker, rinse the crucible with water, and pass the rinsings also through the filter.

NOTE 8—A filter crucible is preferred, although filter paper may be used if it in no way affects the final color development. In either case, the filtrate shall be perfectly clear.

32.3 Add 3 cm^3 of H_3PO_4 and 0.3 g of KIO_4 to the filtrate, and evaporate by careful boiling to a volume of less than 50 cm^3 over a period of approximately 15 min. Transfer the solution to a 50-cm^3 volumetric flask, and after cooling to room temperature dilute the solution with water to the 50-cm^3 mark.

NOTE 9—If a turbidity appears at this point or after transfer of the solution to an absorption cell (32.4), it is probably due to crystallization of KIO_4. In this case the solution must be allowed to stand until it is clear or it must be refiltered and reheated in accordance with 32.2 and 32.3 but without further addition of KIO_4.

32.4 Rinse the cell of the photometer with the $KMnO_4$ rinse solution, then with water, and finally with the test solution. Fill the cell with the test solution and measure its absorbance at approximately 525 nm, using the blank solution as a reference solution.

NOTE 10—If 1-cm path length cells are used, report the results only to the nearest 1 mg/kg (ppm). If greater accuracy is desired use a cell of greater path length, preferably a 5-cm cell, in order to obtain absorbance readings between 0.3 and 0.8.

32.5 Determine the concentration of manganese in the test solution from the absorbance reading and the calibration curve (31.3). Express the result in milligrams of manganese per kilogram of rubber (parts per million).

33. Precision and Bias

33.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

MANGANESE
(Alternative Flame Atomic Absorption Method)

34. Procedure

34.1 See Test Methods D4004, Method E, Manganese.

IRON
(Colorimetric Method)

35. Apparatus

35.1 *Photoelectric Photometer*—A spectrophotometer or filter photometer suitable for measurements at approximately 510 nm with absorption cells 1 to 3 cm in path length (Note 5).

36. Reagents

36.1 *Buffer Solution*—Dissolve 164 g of anhydrous sodium acetate in water, add 28.5 cm^3 of acetic acid, and dilute the mixture with water to 500 cm^3.

36.2 *Hydrochloric Acid* (density = 1.19 Mg/m^3)—Concentrated hydrochloric acid (HCl).

NOTE 11—If this buffer solution gives highly colored reference solutions, alternative buffer solutions can be prepared by dissolving 80 g of sodium hydroxide (NaOH) and 107 g of sodium carbonate (Na_2CO_3) in 200 cm^3 of water, adding 142.5 cm^3 of acetic acid, and diluting to 500 cm^3.

36.3 *Hydroxylamine Hydrochloride Solution* (100 g/cm^3)—Dissolve 10 g of hydroxylamine hydrochloride in 100 cm^3 of water.

36.4 *Iron, Standard Solution* (1 cm^3 = 0.1 mg Fe)—Dissolve 0.7021 g of ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_26H_2O$) in water containing 3 cm^3 of concentrated hydrochloric acid (HCl, density = 1.19 Mg/m^3) and dilute to 1000 cm^3 with water. This solution should remain stable for at least a month.

36.5 *Iron, Standard Solution* (1 cm^3 = 0.01 mg Fe)—Dilute 10 cm^3 of the iron solution (1 cm^3 = 0.1 mg Fe) to 100 cm^3 with water. The iron solution must be made up fresh each day.

36.6 *1,10 Phenanthroline Solution* (1 g/cm^3)—Dissolve 0.5 g of 1,10 phenanthroline monohydrate in hot water and dilute to 500 cm^3.

37. Preparation of Calibration Curve

37.1 Prepare a series of standard solutions by pipetting portions of 0, 5, 10, 15, and 20 cm^3 of iron solution (1 cm^3 = 0.01 mg Fe) into 50-cm^3 volumetric flasks. To each add 1 cm^3 of HCl (36.2).

37.2 Make an analysis of each of the standard solutions as described in 38.2 and 38.3, starting with the addition of the buffer solution and continuing through the measurement of the absorbance, using the solution containing no added iron as the reference solution.

37.3 Prepare a calibration curve by plotting the relationship between iron concentration and absorbance. The calibration curve should be checked whenever necessary, depending on local conditions and on the type of instrument used.

38. Procedure

38.1 Ash a 10 to 12-g specimen of homogenized rubber according to the ashing procedure described in Section 15, except that the temperature shall be maintained at 525 ± 25°C and the sample shall be wrapped in a 150-mm ashless filter paper. Ash a blank consisting of the filter paper and carry it through the procedure in the same manner as the sample. Add 5 cm^3 of HCl and 5 cm^3 of water to the crucible and digest the mixture on a steam plate for 30 to 60 min. If the solution has a deep yellow color, indicating the presence of much iron, add 5 cm^3 more of HCl and continue the digestion for 30 min more. Filter the solution, collect the filtrate in a 50-cm^3 volumetric flask, and dilute to the 50-cm^3 mark.

38.2 Transfer an aliquot containing not more than 2 cm^3 of HCl to a 50-cm^3 volumetric flask. Add 10 cm^3 of the buffer solution, then 1 cm^3 of hydroxylamine solution, and 10 cm^3 of 1,10-phenanthroline solution. Dilute the solution to the mark with water and allow to stand for 15 min. Treat an equal aliquot of the blank solution by the same procedure.

38.3 Fill the cell of the photoelectric photometer with this solution and measure the absorbance at a wavelength of approximately 510 nm, using the treated blank aliquot as a reference solution. If the absorbance is greater than 0.8, repeat this step using a smaller aliquot. If the absorbance is below 0.3, repeat this step with a larger aliquot if this is possible.

38.4 Determine the concentration of iron in the test solution from the absorbance reading and the calibration curve (37.3). Express the results in milligrams of iron per kilogram of rubber (parts per million).

39. Precision and Bias

39.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

ACETONE EXTRACT

40. Procedure

40.1 Make the quantitative determination for acetone-extractable material in crude natural rubber according to the procedure given for acetone extract in Test Methods D297.

41. Precision and Bias

41.1 Task groups for the purpose of obtaining precision and bias data in accordance with Practice D4483 are being organized and precision and bias statements will be added to this test method when available.

RUBBER HYDROCARBON

42. Procedure

42.1 Make the quantitative determination for rubber hydrocarbon in natural rubber according to the procedure given in Test Methods D297, sections 53.1 to sections 53.2.5.

43. Precision and Bias

43.1 The precision and bias for this test method will apply to the determination from 42.1.

NITROGEN

44. Procedure

44.1 Make the quantitative determination for nitrogen in natural rubber according to the procedure given in Test Method D3533.

45. Precision and Bias

45.1 The precision and bias for this test method will apply to the determination from 44.1.

46. Keywords

46.1 ash; copper; dirt; iron; manganese; nitrogen; rubber hydrocarbon; volatile matter