Designation: D5774 – 95 (Reapproved 2014)

Standard Test Methods for Rubber—Chemical Analysis of Extractables[1]

This standard is issued under the fixed designation D5774; 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 extractables from synthetic rubbers and are intended for general use on solid uncompounded styrene-butadiene copolymers commonly referred to as SBR. Analysis of other synthetic rubbers is also possible with some of these test methods. The test methods and the sections in which they are covered are as follows:

	Sections
Total Extractables	4 – 11
Organic Acid	12 – 19
Soap	20 – 26
Oil	27 – 42

NOTE 1—The nomenclature used in these test methods is in accordance with Practice D1418.

1.2 *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

D1418 Practice for Rubber and Rubber Latices—Nomenclature

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

3. Reagents

3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without decreasing the accuracy of the determination.

TEST METHOD A—TOTAL EXTRACTABLES

4. Summary of Test Method

4.1 Thin, narrow strips of the dried rubber are extracted three times with 100 cm^3 of hot solvent for 10, 20, and 40-min periods, respectively. The extracted rubber is then extracted for 5 min in 100 cm^3 of hot acetone to thoroughly remove the extraction solvent and aid drying of the rubber. The extracted rubber is then dried to constant mass. The difference in mass between the original sample and the extracted sample is the total extractables.

5. Significance and Use

5.1 This procedure is intended to determine the total extractable materials in rubber. It will give the combined amount of rosin and fatty acids, soaps, extender oils, defoamer tars, antioxidants, and other uncombined organic constituents that are extractable in the solvent used. The rubber hydrocarbon can be estimated by subtracting the sum of the total extract, the total ash, the volatile matter, and carbon black content (for carbon black masterbatch) from 100.

6. Apparatus

6.1 *Wide-Mouth Flask,* 400 to 500 cm^3.

6.2 *Hot Plate and Reflux Condenser.*

NOTE 2—Extraction apparatus in Fig. 1 of Test Methods D297 may be used, but without the extraction siphon cup.

6.3 *Filter Paper and Wire Gauze.*

6.4 *Weighing Dish or Watch Glass.*

---

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

Current edition approved Aug. 1, 2014. Published November 2014. Originally approved in 1995. Last previous edition approved in 2009 as D5774 – 95 (2009). DOI: 10.1520/D5774-95R14.

[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] *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.

6.5 *Vacuum Oven,* capable of maintaining a temperature of 105°C and a pressure of 3.0 kPa (23 mm Hg) or less.

7. Reagents

7.1 *Solvents:*

7.1.1 *ETA (Ethanol-Toluene-Azeotrope)*—Prepare by mixing 70 volumes of ethyl alcohol and 30 volumes of toluene, refluxing the mixture for 4 h over calcium oxide and distilling. Discard the first and last portions, keeping only that distillate coming over within a range of 1°C. Distilling may be omitted if absolute grain alcohol or anhydrous formula 3A alcohol is used.

7.1.2 *H-ITM (Hydrous Isopropanol-Toluene Mixture)*—Prepare by mixing 75 volumes of anhydrous isopropanol with 25 volumes of toluene and then mixing 92 volumes of this solution with 8 volumes of water.

7.1.3 *Acetone.*

8. Preparation of Sample

8.1 Dry the sample in accordance with the procedure described in 8.1.1 or 8.1.2.

8.1.1 Weigh a piece of rubber (at least 450 g) to the nearest 0.1 g. Pass the rubber repeatedly through a laboratory mill, with the rolls of the mill maintained at 100 ± 5°C (212 ± 9°F) and the distance between the rolls at 0.25 ± 0.05 mm (0.010 ± 0.002 in.), as determined by a lead slug. Do not allow the rubber to band, and take care to prevent any loss of sample. At the end of 4 min, weigh the rubber to the nearest 0.1 g. Pass the rubber through the mill for an additional 2 min, and reweigh it. If the weights at the end of the 4 and 6-min periods are within 0.1 g, calculate the volatile matter; if not, continue passing the sample through the mill for 2-min periods until the weight remains constant within 0.1 g.

8.1.2 Sheet out the rubber (at least 250 g) on a laboratory mill, with the distance between the rolls set at 0.25 ± 0.05 mm (0.010 ± 0.002 in.), as determined by a lead slug, and the temperature of the roll being no greater than 32°C (90°F). Weigh the entire sheet to the nearest 0.1 g. Place in a forced-circulation oven set at 100 ± 5°C (212 ± 9°F) so that both surfaces of the sheet are exposed to the draft. Allow the rubber to remain in the oven until the mass is constant to within 0.1 g. Usually 1 h is sufficient for rubbers containing no more than 1.0 % moisture.

8.2 Sheet the dry polymer to a thickness of 0.5 mm (0.020 in.) or less. Cut approximately 6 g of the dried, sheeted sample into strips not wider than 10 mm or longer than 50 mm.

NOTE 3—To ensure complete extraction of the rubber, the thickness of the sheet must not exceed the prescribed maximum.

For rubber types that during extraction tend to mass or stick together (that is, low viscosity rubber and alum-coagulated rubber) and for rubber types containing materials that are otherwise difficult or slow to extract, the dried sheet may be prepared for extraction by one of the following methods to expedite and provide complete extraction:

(*a*) Using a laboratory press with flat heated platens, press 1.5 to 1.7 g of the rubber into a tared 89 to 94-mm (3.5 to 3.7-in.) diameter circle of either stainless steel screen having openings of approximately 0.177 mm (80 mesh, 5.2 mil wire diameter) or of polyester monofilament screen cloth[4] that has been previously extracted with the solvent being used. Remove any excess rubber that extends beyond the edge of the screen. Accurately weigh the prepared rubber and screen. After the test is completed, the rubber can be burned off within a muffle furnace and the stainless steel screen can be reused.

(*b*) Cut 5 to 6 g of the sheet into squares approximately 38 by 38 mm (1.5 by 1.5 in.) and weigh between two 50-mm (2-in.) squares of polypropylene screen[5] that previously has been extracted in the solvent being used.

9. Procedure

9.1 Accurately weigh 6 g of the dried rubber strips.

9.2 Add 100 cm^3 of the chosen solvent (ETA or H-ITM) to the extraction flask, and add each strip of the weighed polymer separately to the flask, swirling the flask after each addition so that each strip is thoroughly wetted with solvent to avoid sticking. To prevent the sample from sticking to the flask, a filter paper may be placed in the bottom of the flask and a wire gauze may be placed on the hot plate under the flask. Sample preparation, as described in Note 3(*a*) or (*b*) also may be used, if only the total extractables are to be determined.

9.3 Reflux the contents of the flask on the hot plate for at least 10 min (1 h max). Decant the solvent, rinse the rubber with 20 cm^3 of fresh solvent, and add 100 cm^3 of fresh solvent to the flask.

9.4 Reflux the contents of the flask on the hot plate for at least 20 min (1 h max). Again decant the solvent, rinse the rubber with 20 cm^3 of fresh solvent, and add 100 cm^3 of fresh solvent to the flask.

9.5 Reflux the contents of the flask on the hot plate for at least 40 min (1 h max). Again decant the solvent, and then add 100 cm^3 of acetone to the flask.

9.6 Reflux again for 5 min to remove the extraction solvent from the rubber. Decant and discard the acetone solution.

9.7 Transfer the extracted rubber to a tared weighing dish. Place the dish in a vacuum oven and dry for 1 h at 105 ± 3°C and a pressure not greater than 3.0 kPa (23 mm Hg). Reweigh the dried rubber.

10. Calculation

10.1 Calculate the percentage of total extractables as follows:

$$\text{Total extract, } \% = [100(A - B)]/A \tag{1}$$

---

[4] The sole source of supply of the polyester monofilament screen cloth known to the committee at this time is Custom Filter Supply, 8581 Mosley, Houston, TX, 77075, style PES212HC, 74/120 mesh. 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.

[5] The sole source of supply of the screen known to the committee at this time is a screen with 0.84 mm square openings and 0.25 mm diameter wire available from Southern Press and Media Co., P.O. Box 24, Augusta, GA 30903-0024 under the designation of 24 by 24 mesh, 1⁄30 in. square openings, 10 mil diameter green polypropylene screen. 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.

TABLE 1 Type 1 Precision for Total Extractables

Material	Mean	S_r	*r*	*(r)*	S_R	*R*	*(R)*^{A}
No. 6	30.05	0.112	0.317	1.05	0.137	0.39	1.29
No. 5	32.45	0.082	0.232	0.72	0.458	1.30	4.01
No. 3	39.73	0.132	0.374	0.94	0.289	0.82	2.06
Pooled Average	34.08	0.111	0.314	0.92	0.322	0.91	2.29

^{A} S_r = repeatability, standard deviation, in measurement units,
r = repeatability, in measurement units,
(r) = repeatability, (relative) percent,
S_R = reproducibility, standard deviation, in measurement units,
R = reproducibility, in measurement units, and
(R) = reproducibility, (relative) percent.

where:

A = mass of the original dry sample, g, and
B = mass of the extracted dried sample, g.

11. Precision and Bias[6]

11.1 The precision results for these test methods were originally derived from an interlaboratory test program (ITP) conducted prior to the adoption of Practice D4483 as the reference precision standard for Committee D11 test methods and therefore was not conducted according to the guidelines set forth in Practice D4483. However, the results of that ITP have been translated into Practice D4483 precision expression format and are given in this section. See Practice D4483 for terminology and other statistical calculation details.

11.2 The precision results in this precision and bias section give an estimate of the precision of the test method with the materials used in the particular ITP as described in 11.3. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those materials and the specific testing protocols of the test method.

11.3 The Type 1 precision is based on a program that employed three materials (rubbers), with tests on each of two days by eight laboratories. A test result is the average of two determinations. The precision data are given in Table 1.

11.4 Bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method; therefore, bias cannot be evaluated.

TEST METHOD B—ORGANIC ACID

12. Summary of Test Method

12.1 Thin narrow strips of the dried rubber prepared as described in 8.1 and 8.2 are accurately weighed to secure about 6 g. The polymer strips are extracted twice in hot extraction solvent. The solvent extracts and rinsings are combined in a 250-cm^3 volumetric flask and made up to volume with fresh solvent. A 100-cm^3 portion of the extract solution is titrated to the first color change with 0.1 *M* NaOH solution, using the chosen indicator. The titration and sample mass are used to calculate the organic acid.

13. Significance and Use

13.1 This procedure is intended to determine the organic acid remaining in a synthetic rubber. There are two test methods: B for alum-coagulated polymers, and A for all others. While these procedures were developed for emulsion SBR, they may be applicable to other emulsion polymers. Organic acid in the polymer may affect cure rate of compounded stock.

14. Apparatus

14.1 *Typical Laboratory Apparatus.*

15. Reagents

15.1 *Solvents:*

15.1.1 *Ethanol Toluene-Azeotrope (ETA)*—See 7.1.1. Not applicable when testing alum-coagulated polymers.

15.1.2 *Alternative Extraction Solvent—Hydrous Isopropanol-Toluene Mixture (H-ITM)*—See 7.1.2. H-ITM is applicable to all polymers, and especially the alum-coagulated polymers.

15.2 *Indicator Solutions:*

15.2.1 *Meta-Cresol Purple Indicator Solution* (0.1 %), in ethyl alcohol or in water. Neutralize each 0.1 g of indicator in the solution with 26.2 cm^3 of 0.01 *M* NaOH solution before diluting to volume. Store the indicator solution in a brown bottle out of contact with fluorescent lights, which will cause the solution to deteriorate.

15.2.2 *Thymol Blue Indicator Solution* (0.2 %)—Dissolve 0.06 g of indicator in 6.45 cm^3 of 0.02 *M* aqueous NaOH and dilute to 50 cm^3 with distilled water. (Recommended for use with H-ITM extracts.)

15.3 *Sodium Hydroxide Standard Solution* (0.1 *M*)—Prepare and standardize a 0.10 *M* solution of sodium hydroxide (NaOH).

16. Preparation of Sample

16.1 Prepare a sample in accordance with 8.1 and 8.2.

17. Procedures

17.1 *Procedure A—Non-Alum-Coagulated Polymers:*

17.1.1 Weigh a 6-g specimen of the dried rubber strips to the nearest 1 mg.

17.1.2 Add 100 cm^3 of the chosen solvent (15.1) to the extraction flask and add each strip of the weighed specimen separately to the flask. Swirl the flask after each addition so that each strip is thoroughly wetted with solvent to avoid sticking. To prevent the sample from sticking to the flask, a filter paper may be placed in the bottom of the flask, and a wire gauze may be placed on the hot plate under the flask. Sample preparation, as described in Note 4(*b*), also may be used.

17.1.3 Reflux the contents of the flask on the hot plate for at least 10 min (1 h, max). Decant the solvent into a 250-cm^3 volumetric flask, rinse the sample with 20 cm^3 of fresh solvent, adding the rinsing to the flask. Add 100 cm^3 of fresh solvent to the extraction flask.

17.1.4 Reflux the contents of the flask on the hot plate for at least 20 min (1 h, max). Again decant the solvent into the volumetric flask and rinse the sample with 20 cm^3 of fresh

---

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1002.

solvent, adding the rinsing to the flask. Cool the extract solution to room temperature and add enough fresh solvent to bring the volume to 250 cm^3. Mix the solution thoroughly.

17.1.5 With a pipet, measure two 100-cm^3 portions of the extract solution into 250-cm^3 Erlenmeyer flasks. Save one 100-cm^3 portion for the soap determination (Sections 20 – 26).

17.1.6 To one 100-cm^3 portion of the extract solution, add six drops of the chosen indicator solution. Titrate the solution with 0.1 *M* NaOH solution to the first color change. Run a blank titration using 100 cm^3 of fresh extraction solvent that has been treated in the same manner as the sample solution and using the same type and amount of indicator. Deduct the volume of NaOH solution required for the blank from that used for the sample titration.

17.2 *Procedure B—Alum-Coagulated Polymers:*

17.2.1 Accurately weigh a 6-g sample of the dried rubber strips.

17.2.2 Add 100 cm^3 of the H-ITM solvent (9.2) to the extraction flask. (H-ITM should be used for alum-coagulated polymers.) Add each strip of the weighed sample that has been prepared in accordance with Note 3 to the flask. To prevent the sample from sticking to the flask, a filter paper may be placed in the bottom of the flask and a wire gauze may be placed on the hot plate under the flask.

17.2.3 Reflux the contents of the flask on the hot plate for at least 10 min (1 h, max). Decant the solvent into a 250-cm^3 volumetric flask. Rinse the sample with 20 cm^3 of fresh solvent, adding the rinsing to the flask. Add 100 cm^3 of fresh solvent to the extraction flask.

17.2.4 Reflux the contents of the flask on the hot plate for at least 20 min (1 h, max). Again decant the solvent into the volumetric flask and rinse the sample with 20 cm^3 of fresh solvent, adding the rinsing to the flask. Cool the extract solution to room temperature and add enough fresh solvent to bring the volume to 250 cm^3. Mix the solution thoroughly.

17.2.5 Add 100 cm^3 of fresh solvent to the extraction flask. Reflux the contents of the flask on the hot plate for at least 15 min (1 h, max). Decant the solvent into a 250-cm^3 Erlenmeyer flask and rinse the sample with 20 cm^3 of fresh solvent, adding the rinsing to the flask. Save this extract solution as Supplement Extraction No. 1.

17.2.6 Add 100 cm^3 of fresh solvent to the extraction flask. Reflux the contents of the flask on the hot plate for at least 15 min (1 h, max). Decant the solvent into a 250-cm^3 Erlenmeyer flask and rinse the sample with 20 cm^3 of fresh solvent, adding the rinsing to the flask. Save this extract solution as Supplement Extraction No. 2.

17.2.7 With a pipet, measure a 100-cm^3 portion of the extract solution as described in 17.2.4 into a 250-cm^3 Erlenmeyer flask.

17.2.8 To the 100-cm^3 portion of the extract solution and the supplement extractions (total amount), add six drops of the chosen indicator solution. Titrate the three solutions individually with 0.1 *M* NaOH solution to the first color change. Run a blank titration using 100 cm^3 of fresh extraction solvent that has been treated in the same manner as the sample solution and using the same type and amount of indicator. Deduct the volume of NaOH solution required for the blank from that used for each of the sample titrations.

NOTE 4—If the percent acid in the No. 2 supplement extraction is not less than 0.1 % calculated on the original sample mass, run an additional supplement extraction as described in 17.2.6.

18. Calculation

18.1 *Procedure A*—Calculate the percentage of organic acid as follows:

$$\text{Organic acid, \%} = (J \times K \times L \times 0.25)/A \qquad (2)$$

where:

J = volume of NaOH solution used for titration (corrected for blank), cm^3,
K = molarity of standard NaOH solution,
L = equivalent mass of organic acid determined; if unknown, use the following typical values: 284 for stearic acid, 346 for rosin acid, 315 for 50 + 50 mixture of stearic and rosin acids, and
A = mass of original dry sample, g.

18.2 *Procedure B*—Calculate the percentage of organic acid as follows:

$$\text{Organic acid, \%} = \left[J + \frac{X}{2.5} + \frac{Y}{2.5} \times K \times L \times 0.25\right]/A \qquad (3)$$

where:

J = volume of NaOH solution used for titration of 100-cm^3 portion of 250-cm^3 solution (corrected for blank), cm^3,
K = molarity of standard NaOH solution,
L = equivalent mass of organic acid determined; if unknown, use the following typical values: 284 for stearic acid, 346 for rosin acid, 315 for 50 + 50 mixture of stearic and rosin acids,
A = mass of original dry sample, g,
X = volume of NaOH solution used for titration of entire volume of Supplement Extraction No. 1 (corrected for blank), cm^3, and
Y = volume of NaOH solution used for titration of entire volume of Supplement Extraction No. 2 (corrected for blank), cm^3.

19. Precision and Bias[6]

19.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical calculation details.

19.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described in 19.3. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

19.3 A Type 1 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the average value, as specified by this test method, obtained on two determination(s) or measurement(s). Four different materials were used in the interlaboratory program; these were tested in eight laboratories on two different days.

19.4 The results of the precision calculations for repeatability and reproducibility are given in Table 2 in ascending order of material average or level for each of the materials evaluated.

19.5 The precision of this test method may be expressed in the format of the following statements, which use an appropriate value of *r, R,* (*r*), or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 2 closest to the mean level under consideration at any given time for any given material in routine testing operations.

19.6 See 26.6 – 26.9 for repeatability statements as expressed in Table 2.

TEST METHOD C—SOAP

20. Summary of Test Method

20.1 One of the 100-cm^3 portions of solvent extract remaining from the organic acid test described in 17.1.5 is titrated with 0.05 *M* HCl, using the chosen indicator to the first color change. From the weight of the original sample strips and the titrations, the percentage of soap is calculated.

21. Significance and Use

21.1 This procedure is intended to determine the organic soap in an emulsion polymer. That soap is a by-product of the emulsion process; it may affect the cure rate of compounded stock. While this procedure was originally developed for emulsion SBR, it may be applicable to other polymers.

22. Reagents

22.1 *Hydrochloric Acid, Standard (0.05 M).*

22.2 *Indicator Solutions:*

22.2.1 *Meta Cresol Purple Indicator Solution* (0.1 %). See 15.2.1.

22.2.2 *Bromophenol Blue Indicator Solution* (0.1 %), in ethyl alcohol.

22.2.3 *Bromothymol Blue Indicator Solution* (0.1 %), in ethyl alcohol.

22.2.4 *Thymol Blue Indicator Solution.* See 15.2.2 (recommended for use with H-ITM extracts).

TABLE 2 Type 1 Precision for Organic Acid—Procedures A and B

Material	Average Level,%	Within Laboratories			Between Laboratories		
		S_r	r	(r)	S_R	R	$(R)^A$
No. 6	3.21	0.0457	0.129	4.0	0.080	0.226	7.1
No. 5	4.65	0.0621	0.176	3.8	0.145	0.410	8.8
No. 4	6.16	0.0804	0.228	3.7	0.191	0.541	8.8
No. 3	4.99	0.0701	0.198	4.0	0.182	0.515	10.3

[A] S_r = repeatability, standard deviation, in measurement units,
r = repeatability, in measurement units,
(r) = repeatability, (relative) percent,
S_R = reproducibility, standard deviation, in measurement units,
R = reproducibility, in measurement units, and
(R) = reproducibility, (relative) percent.

23. Preparation of Sample

23.1 Prepare the sample in accordance with 8.1 and 8.2 and determine the percentage soap on a portion of the solvent extract remaining from the test for organic acid (17.1.5).

24. Procedure

24.1 Add six drops of the chosen indicator solution to the 100 cm^3 of extract solution saved from the organic acid test (17.1.5). Titrate the solution with 0.05 *M* HCl to the first color change. Run a blank titration, using 100 cm^3 of fresh extraction solvent that has been treated in the same manner as the sample (17.1.2 – 17.1.5), and using the same amount of indicator (Note 5). Deduct the volume of HCl used for the blank from that used for the sample.

NOTE 5—The selection of the indicator solution is left to the discretion of the analyst. Whichever indicator solution gives the clearest, most discernible end-point for the type sample being tested should be used. Thymol Blue has been recommended for use with H-ITM extracts.

25. Calculation

25.1 Calculate the percentage of soap as follows:

$$\text{Soap, \%} = (M \times N \times P \times 0.25)/A \qquad (4)$$

where:

M = volume of standard HCl used for titration (corrected for blank), cm^3 ,
N = molarity of standard HCl,
P = equivalent weight of soap determined; if unknown, use the following typical values: 306 for sodium stearate, 322 for potassium stearate, 368 for sodium rosinate, 384 for potassium rosinate, 337 for 50 + 50 mixture of sodium stearate and sodium rosinate, 353 for 50 + 50 mixture of potassium stearate and potassium rosinate, 345 for 50 + 50 mixture of sodium stearate and potassium rosinate or potassium stearate and sodium rosinate, and
A = mass of original dry sample, g.

26. Precision and Bias[6]

26.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical calculation details.

26.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described in 26.3. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

26.3 A Type 1 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the average value, as specified by this test method, obtained on two determination(s) or measurement(s). Four different materials (rubbers) were used in the interlaboratory program; these were tested in eight laboratories on two different days.

26.4 The results of the precision calculations for repeatability and reproducibility are given in Table 3, in ascending order of material average or level, for each of the materials evaluated.

26.5 The precision of this test method may be expressed in the format of the following statements, which use an appropriate value of *r, R,* (*r*), or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 3 closest to the mean level under consideration at any given time for any given material in routine testing operations.

26.6 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Table 3. Two single test results obtained under normal test method procedures that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

26.7 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Table 3. Two single test results obtained in two different laboratories under normal test method procedures that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

26.8 Repeatability and reproducibility expressed as a percent of the mean level, (*r*) and (*R*), have equivalent application statements as in 26.6 and 26.7 for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results. As is usual with the analysis of materials that are present in low concentrations, the relative repeatability and reproducibility are high.

26.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

TABLE 3 Type 1 Precision for Soap

Material	Average Level,%	Within Laboratories			Between Laboratories		
		S_r	r	(r)	S_R	R	(R)[A]
No. 3	0.044	0.0085	0.0241	54.7	0.0448	0.127	290.0
No. 4	0.013	0.0056	0.0158	122.0	0.0315	0.0891	690.0
No. 5	0.019	0.0093	0.0263	139.0	0.0326	0.0923	490.0
No. 6	0.036	0.0097	0.0275	76.3	0.0625	0.177	490.0

[A] S_r = repeatability, standard deviation, in measurement units,
r = repeatability, in measurement units,
(r) = repeatability, (relative) percent,
S_R = reproducibility, standard deviation, in measurement units,
R = reproducibility, in measurement units, and
(R) = reproducibility, (relative) percent.

TEST METHOD D—OIL

Procedure A

27. Summary of Test Method

27.1 The total extractables of the dried rubber is determined in accordance with Sections 4 – 11. The oil content of the sample is estimated by subtracting from the total extractables a correction value that is the total of the major extractable constituents of the particular type being tested other than the oil. The difference is an estimate of the oil content.

28. Significance and Use

28.1 This test method covers the estimation of oil content of oil-extended polymers and carbon black masterbatches. It is intended for use in checking the uniformity of shipments. While this method was developed for emulsion SBR, it may be applicable to other polymers and carbon black masterbatches.

29. Apparatus

29.1 See Section 6.

30. Reagents

30.1 See Section 7.

31. Preparation of Sample

31.1 See Section 8.

32. Procedure

32.1 See Section 9.

33. Calculation

33.1 Calculate the percentage of oil as follows:

$$\text{Oil content, \%} = [100(A - I)/A] - (B + C + D) \tag{5}$$

where:

A = mass of the original dry sample, g,
I = mass of extracted sample, g,
B = organic acid, %,
C = soap, %, and
D = antioxidant/antiozonant, %. If antioxidant/antiozonant cannot be determined, the following value may be calculated:

$$D = (E/F) \times 100 \tag{6}$$

where:

E = nominal antioxidant added to rubber, phr, and
F = masterbatch recipe, in parts = 100 + phr of oil + phr of carbon black.

34. Precision

34.1 *Repeatability*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 0.28 %.

Note 6—Although the repeatability and reproducibility of this test method are good, the accuracy depends on the accuracy of the analysis of acid, soap, and antioxidant/antiozonant.

34.2 *Reproducibility*—Results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 0.34 % (Note 6).

34.3 The precision of the H-ITM test method is being evaluated.

Procedure B (Quick)

35. Summary of Test Method

35.1 The total extractables of the rubber are determined by extracting a 1-g sample in the chosen solvent and then in acetone and drying the extracted rubber. The oil content is estimated by subtracting from the total extract a correction value that is an average of the major extractable constituents of the particular type being tested other than the oil. This difference is an estimate of the oil content.

36. Significance and Use

36.1 Procedure B is an alternative quick test method for estimating the oil content of oil-extended SBR type polymers (oil masterbatches) and oil-black masterbatches of SBR polymers. This test method is intended for use as a quick test method for determining the total extract of a rubber sample for estimation of oil content where other constituents of the sample may be determined or where their normal average values are used. While this method was developed for emulsion SBR, it may be applicable to other polymers and carbon black masterbatches.

37. Apparatus

37.1 See 6.1 – 6.4.

37.2 *Convection Oven,* 130 ± 3°C (266 ± 5°F).

38. Reagents

38.1 See Section 7.

39. Preparation of Sample

39.1 Prepare the sample in accordance with the procedure described in Section 8.

40. Procedure

40.1 Accurately weigh a 1-g sample of the dried strips.

40.2 Add 150 cm^3 of the chosen solvent to the 400-cm^3 extract flask and add each strip of the weighed sample separately to the flask. Swirl the flask after each addition so that each strip is thoroughly wetted with solvent to avoid sticking.

40.3 Reflux the sample at least 10 min (1 h, max) on the hot plate. Decant and discard the solvent and rinse the sample with 10 cm^3 of fresh solvent. Decant and discard the rinse solvent.

40.4 Add 150 cm^3 of acetone to the flask and reflux for 15 min on the hot plate. Decant and discard the acetone.

40.5 Transfer the extracted sample to a tared weighing dish. Dry the sample for 15 min or to constant mass in a convection drying oven at 130 ± 3°C (266 ± 5°F).

41. Calculation

41.1 See Section 33.

42. Precision

42.1 Precision of this test method has not been determined.

43. Keywords

43.1 extractables; oil; organic acid; soap