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

This standard is issued under the fixed designation D 5774; 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.

ϵ^1 NOTE—The title was revised editorially in December 2004.

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 D 1418.

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]

D 297 Test Methods for Rubber Products—Chemical Analysis

D 1418 Practice for Rubber and Rubber Latices—Nomenclature

D 4483 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 D 297 may be used, but without the extraction siphon cup.

6.3 *Filter Paper and Wire Gauze or Asbestos Mat*.

6.4 *Weighing Dish or Watch Glass*.

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

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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] *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.

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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 or asbestos mat 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:

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

[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.

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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 D 4483 as the reference precision standard for Committee D11 test methods and therefore was not conducted according to the guidelines set forth in Practice D 4483. However, the results of that ITP have been translated into Practice D 4483 precision expression format and are given in this section. See Practice D 4483 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.

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

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 or asbestos mat 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

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