Designation: D6370 – 99 (Reapproved 2014)

Standard Test Method for Rubber—Compositional Analysis by Thermogravimetry (TGA)[1]

This standard is issued under the fixed designation D6370; 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 This test method provides a thermogravimetric (TGA) technique to determine the amounts of organics (oil, polymer), carbon black and ash (filler) in a rubber compound.

1.2 The amount of plasticizer/oil may be determined separately using Test Method D297.

1.3 This test method utilizes previously calibrated, manual or computer assisted TGA instrumentation.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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

D1566 Terminology Relating to Rubber

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

D6085 Practice for Sampling in Rubber Testing—Terminology and Basic Concepts

E473 Terminology Relating to Thermal Analysis and Rheology

E1953 Practice for Description of Thermal Analysis and Rheology Apparatus

3. Terminology

3.1 *Definitions:*

3.1.1 The definitions relating to rubber appearing in Terminology D1566 shall be considered applicable to this test method.

3.1.2 The terminology relating to sampling appearing in Practice D6085 shall be considered applicable to this test method.

3.1.3 The definitions for thermal analysis appearing in Terminology E473 shall be considered applicable to this test method.

3.1.4 The description of thermal analysis equipment appearing in Practice E1953 shall be considered applicable to this test method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *ash, n*—nonvolatile additives (fillers), such as zinc oxide, talc, etc.

3.2.2 *carbon black, n*—carbon black.

3.2.3 *organics, n*—rubber (polymer), noncarbon black organic additives, such as oil, plasticizer, antioxidants, etc.

4. Summary of Test Method

4.1 The mass of the rubber test sample, heated at a controlled, specified rate in a controlled, specified environment is recorded as a function of temperature. The mass loss over the specified temperature range provides a compositional analysis of the sample.

5. Significance and Use

5.1 This test method is intended for use in quality control, material screening, and related problem solving where a compositional analysis, or comparison to a known material, is desired.

5.2 The parameters described are guidelines and may be altered to suit the analysis of other rubber compounds.

5.3 This test method is not suitable for rubber compounds containing filler materials which decompose in the temperature range of 50 to 800°C, for example, $CaCO_3$, $Al(OH)_3(3H_2O)$, etc. Analysis of compounds containing fillers of this type requires knowledge of the filler type and some correction for mass loss.

[1] This test method is 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.

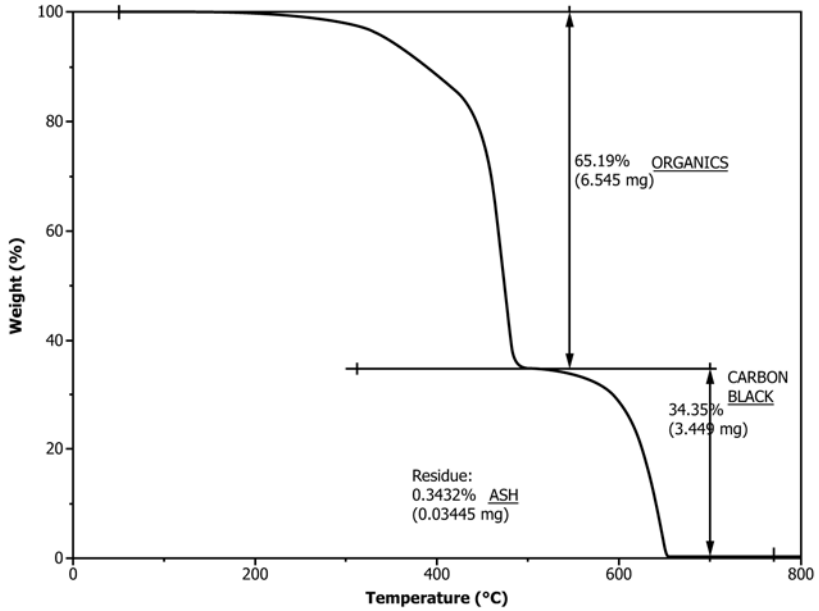

FIG. 1 Typical Thermogram

6. Apparatus

6.1 *Thermogravimetric Analyzer*—A system of related instruments that are capable of continuously weighing a test sample, at a sensitivity of ±2 µg, and recording the change in mass of the test sample under atmospheric control over a specified temperature range.

7. Reagents and Materials

7.1 An inert compressed gas, such as argon or nitrogen, and a reactive gas, such as air or oxygen.

7.2 Compressed gases must be 99.99 % minimum purity.

7.3 The inert purge gas must not contain more than 10 µg/g oxygen.

8. Calibration

8.1 Calibrate the apparatus, according to the prescribed procedures or appropriate operating manual, at the heat (temperature) and purge gas flow rates to be used.

9. Procedure

9.1 Place a small piece, 10 to 12 mg, of the rubber test sample into the platinum pan of the calibrated Thermogravimetric Analyzer (TGA).

9.2 Apply a 75 cm^3/min, or the manufacturer's recommended flow, argon or nitrogen purge.

9.3 Heat to 50°C and allow the instrument to equilibrate for a minimum of 2 min.

9.4 Heat from 50 to 560°C at 10°C/min.

9.5 Cool to 300°C and allow the temperature to equilibrate for a minimum of 2 min.

9.6 Change the purge gas to air or oxygen and purge at 75 cm^3/min or the manufacturer's recommended flow.

9.7 Heat from 300 to 800°C at 10°C/min.

10. Calculation

10.1 Record the percent mass loss for organics, carbon black, and ash as follows (see Fig. 1):

10.2 *For EPDM, NR, PE, PP and SBR:*

Component	% Mass Loss
Organics	50 to 550°C (nitrogen)
Carbon black	310 to 790°C (air)
Ash	Residue at 790°C

10.3 *For CPE, CR, NBR and PVC:*

Component	% Mass Loss
Organics	50 to 550°C (nitrogen) + 310 to 560°C (air)
Carbon black	560 to 790°C (air)
Ash	Residue at 790°C

11. Report

11.1 Report the following information:

11.1.1 Identification of the test sample.

11.1.2 *Percents*—organics, carbon black, and ash found, each to the nearest 0.1 %.

12. Precision and Bias[3]

12.1 This precision and bias section has been prepared in accordance with Practice D4483. Please refer to this practice for terminology and other statistical calculation details.

12.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, etc.) used in the particular interlaboratory test program (ITP) as described below. The precision parameters should not be used for acceptance or rejection testing of

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1089.

TABLE 1 Precision for Thermogravimetric Analysis (Type 1 Precision)

NOTE 1—No relative precision given for percent ash, mean values close to zero.

Part 1—Percent Organics

		Within Laboratories			Between Laboratories		
Material	Mean	Sr^A	r^B	$(r)^C$	SR^D	R^E	$(R)^F$
C	63.7	0.171	0.478	0.75	0.248	0.694	1.09
B	64.9	0.252	0.705	1.09	1.933	5.41	8.34
A	65.4	0.109	0.306	0.47	0.181	0.508	0.78

Part 2—Percent Carbon Black

		Within Laboratories			Between Laboratories		
Material	Mean	*Sr*	*r*	(*r*)	*SR*	R	(*R*)
A	34.3	0.113	0.316	0.92	0.157	0.439	1.28
B	34.6	0.224	0.628	1.82	1.73	4.850	14.02
C	34.6	0.106	0.296	0.86	0.186	0.520	1.50

Part 3—Percent Ash

		Within Laboratories		Between Laboratories	
Material	Mean	*Sr*	*r*	*SR*	*R*
A	0.26	0.110	0.307	0.134	0.375
B	0.31	0.094	0.264	0.158	0.442
C	1.64	0.144	0.403	0.244	0.682

[A] *Sr* = repeatability standard deviation, in measured %.
[B] *r* = repeatability, in measured %.
[C] (*r*) = repeatability, relative basis, % of %.
[D] *SR* = reproducibility standard deviation, in measured %
[E] *R* = reproducibility, in measured %
[F] (*R*) = reproducibility, relative basis, % of %

any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocols of the test method.

12.3 A Type 1 interlaboratory test program was conducted in 1998 on three materials or compounds (A, B, C) containing 35 % carbon black; A = EPDM, B = NBR and C = SBR. Thirteen laboratories participated in the ITP conducting duplicate tests on each of two successive test days. A test result is the average of two measurements for each of the tests conducted; % organics, % carbon black and % ash. The database generated by the ITP was subjected to *h*-outlier and *k*-outlier analysis as given by Practice D4483. Several outlying laboratories were found for the tests; the outlier values were deleted and replaced by the average values for all laboratories for that test and material. The revised database (outliers removed) was then analyzed for test method precision. The results are given in Table 1. The results in the table indicate that the precision for the NBR compound is substantially poorer than for EPDM and SBR.

12.4 *Repeatability*—The repeatability *r*, for each test (organics, carbon black, ash) of this test method has been established as the value tabulated in Table 1 for each material. 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.

12.5 *Reproducibility*—The reproducibility *R*, for each test (organics, carbon black, ash) of this test method has been established as the value tabulated in Table 1 for each material. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* must be considered to have come from different or nonidentical sample populations.

12.6 The relative repeatability and reproducibility, (*r*) and (*R*), also are given in Table 1. These precision parameters have the same applicability statements as given in 12.4 and 12.5.

12.7 *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; therefore, bias cannot be determined.

13. Keywords

13.1 ash; carbon black; filler; oil; organics; plasticizer; polymer; rubber; thermogravimetry