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Standard Test Method for Compositional Analysis by Thermogravimetry¹

This standard is issued under the fixed designation E 1131; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method provides a general technique incorporating thermogravimetry to determine the amount of highly volatile matter, medium volatile matter, combustible material, and ash content of compounds. This test method will be useful in performing a compositional analysis in cases where agreed upon by interested parties.

1.2 This test method is applicable to solids and liquids.

1.3 The temperature range of test is typically room temperature to 1000 °C. Composition between 1 and 100 weight % of individual components may be determined.

1.4 This test method utilizes an inert and reactive gas environment.

~~1.5 Computer or electronic-based instruments, techniques, or data treatment equivalent to this test method may also be used.~~

~~Note 1—Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to determine the necessary equivalency prior to use.~~

~~1.6 SI units are the standard.~~

~~1.7 This standard is related ISO 11358 but is more detailed and specific.~~

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

1.6 This standard is related ISO 11358 but is more detailed and specific.

1.7 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 3172 Practice for Proximate Analysis of Coal and Coke

E 473 Terminology Relating to Thermal Analysis and Rheology

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E 1142 Terminology Relating to Thermophysical Properties

E 1582 Practice for Calibration of Temperature Scale for Thermogravimetry

E 2040 Test Method for Mass Scale Calibration of Thermogravimetric Analyzers

2.2 *ISO Standard:*

11358 Plastics-Thermogravimetry (TG) of Polymers — General Principles³

3. Terminology

3.1 *Definitions:*

3.1.1 Many of the technical terms used in this test method are defined in Terminologies E 473 and E 1142.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *highly volatile matter*—moisture, plasticizer, residual solvent or other low boiling (200 °C or less) components.

3.2.2 *medium volatile matter*—medium volatility materials such as oil and polymer degradation products. In general, these materials degrade or volatilize in the temperature range 200 to 750 °C.

¹ This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Thermal Analysis Methods—Test Methods and Practices.

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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*, Vol 05.05, volume information, refer to the standard's Document Summary page on the ASTM website.

³ *Annual Book of ASTM Standards*, Vol 14.02.

³ Supporting data available from ASTM. Request RR: E37-1009.

3.2.3 *combustible material*—oxidizable material not volatile (in the unoxidized form) at 750 °C, or some stipulated temperature dependent on material. Carbon is an example of such a material.

3.2.4 *ash*—nonvolatile residues in an oxidizing atmosphere which may include metal components, filler content or inert reinforcing materials.

3.2.5 *mass loss plateau*—a region of a thermogravimetric curve with a relatively constant mass.

4. Summary of Test Method

4.1 This test method is an empirical technique using thermogravimetry in which the mass of a substance, heated at a controlled rate in an appropriate environment, is recorded as a function of time or temperature. Mass loss over specific temperature ranges and in a specific atmosphere provide a compositional analysis of that substance.

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 is desired or a comparison can be made with a known material of the same type.

5.2 The parameters described should be considered as guidelines. They may be altered to suit a particular analysis, provided the changes are noted in the report.

5.3 The proportion of the determined components in a given mixture or blend may indicate specific quality or end use performance characteristics. Particular examples include the following:

5.3.1 Increasing soot (carbon) content of used diesel lubricating oils indicates decreasing effectiveness.

5.3.2 Specific carbon-to-polymer ratio ranges are required in some elastomeric and plastic parts in order to achieve desired mechanical strength and stability.

5.3.3 Some filled elastomeric and plastic products require specific inert content (for example, ash, filler, reinforcing agent, etc.) to meet performance specifications.

5.3.4 The volatile matter, fixed carbon, and ash content of coal and coke are important parameters. The “ranking” of coal increases with increasing carbon content and decreasing volatile and hydrocarbon, (medium volatility) content.

6. Interferences

6.1 This test method depends upon distinctive thermostability ranges of the determined components as a principle of the test. For this reason, materials which have no well-defined thermostable range, or whose thermostabilities are the same as other components, may create interferences. Particular examples include the following:

6.1.1 Oil-filled elastomers have such high molecular weight oils and such low molecular weight polymer content that the oil and polymer may not be separated based upon temperature stability.

6.1.2 Ash content materials (metals) are slowly oxidized at high temperatures and in an air atmosphere, so that their mass increases (or decreases) with time. Under such conditions, a specific temperature or time region must be identified for the measurement of that component.

6.1.3 Polymers, especially neoprene and acrylonitrile butadiene rubber (NBR), carbonize to a considerable extent, giving low values for the polymer and high values for the carbon. Approximate corrections can be made for this if the type of polymer is known.

6.1.4 Certain pigments used in rubber lose weight on heating. For example, some pigments exhibit water loss in the range 500 to 600 °C, resulting in high polymer values. Others, such as calcium carbonate, release CO₂ upon decomposition at 825 °C, that may result in high carbon values. The extent of interference is dependent upon the type and quantity of pigment present.

7. Apparatus

7.1 The essential equipment required to provide the minimum thermogravimetric analyzer capability for this method includes:

7.1.1 *A thermobalance*, composed of (a) a furnace to provide uniform controlled heating or a specimen to a constant temperature or at a constant rate within the 25 to 1000 °C temperature range of this test method; (b) a temperature sensor to provide an indication of the specimen/furnace temperature to $\pm 1^\circ\text{C}$; (c) an electrobalance to continuously measure the specimen mass with a minimum capacity of 30 mg and a sensitivity of $\pm 1\ \mu\text{g}$; and (d) a means of sustaining the specimen/container under atmosphere control with a purge rate of 10 to 100 $\pm 5\ \text{mL/min}$.

7.1.2 *A temperature controller*, capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change between 10 and 100 °C/min constant to within $\pm 1\ \%$ for a minimum of 100 minutes: min.

7.1.3 *A recording device*, capable of recording and displaying the change in mass (TGA curve) on the Y-axis with a resolution of 5 μg and temperature or time on the X-axis with a resolution of 1 K or 0.1 min. A data collection device, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for Thermogravimetric analyzers are mass, temperature, and time.

NOTE2—The 1—The capability to display the first derivative of the signal may be useful in the measurement of obscure thermostability ranges.

7.1.4 *Containers (pans, crucibles, and so forth)*, which are inert to the specimen and which will remain dimensionally stable within the temperature limits of this method.

7.2 Gas Flow Control Device, with the capability of switching between inert and reactive gases.

8. Reagents and Materials

8.1 An inert compressed gas such as argon or nitrogen and a reactive compressed gas such as air or oxygen are required for this method.

8.2 Purity of Purge Gases:

8.2.1 0.01 % maximum total impurity.

8.2.2 1.0 µg/g water impurity maximum.

8.2.3 1.0 µg/g hydrocarbon impurity maximum.

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

9. Test Specimen

9.1 Specimens are ordinarily measured as received. If some heat or mechanical treatment is applied to the specimen prior to test, this treatment shall be noted in the report.

9.2 Since the applicable samples may be mixtures or blends, take care to ensure that the analyzed specimen is representative of the sample from which it is taken. If the sample is a liquid, mixing prior to taking the specimen is sufficient to ensure this consideration. If the sample is a solid, take several specimens from different areas of the sample and either combine for a single determination, or each run separately with the final analysis representing an average of the determinations. Note the number of determinations in the report.

10. Calibration

10.1 Calibrate the mass signal from the apparatus according to Test Method E 2040.

10.2 Calibrate the temperature signal from the apparatus according to Practice E 1582.

11. Procedure

11.1 Establish the inert (nitrogen) and reactive (air or oxygen) gases at the desired flow rates. For most analyses, this rate will be in the range of 10 to 100 mL/min. Higher flow rates may be used for some analyses, particularly when utilizing high heating rates.

11.2 Switch the purge gas to the inert (nitrogen) gas.

~~11.3 Zero the recorder and tare the balance. It is recommended that this be done in a range at least one recorder setting more sensitive than that to be used in the final ash weighing.~~

~~11.3 Zero the mass signal r and tare the balance.~~

11.4 Open the apparatus to expose the specimen holder.

11.5 Prepare the specimen as outlined in 9.2 and carefully place it in the specimen holder. Typically, a sample mass of 10 to 30 mg shall be used (see Table 1).

NOTE 32—Specimens smaller than 10 mg may be used if larger specimens cause instrument fouling or poor reproducibility.

11.6 Position the specimen temperature sensor to the same location used in calibration. (See Section 10.)

11.7 Enclose the specimen holder.

11.8 Record the initial mass. If the apparatus in use has provisions for direct percentage measurements, adjust to read 100 %.

11.9 Initiate the heating program within the desired temperature range. See Table 1 for suggested heating rates and temperature ranges. Record the specimen mass change continuously over the temperature interval.

11.9.1 The mass loss profile may be expressed in either milligrams or mass percent of original specimen mass. Expanded scale operation may be useful over selected temperature ranges.

11.9.2 If only one or two components of the compositional analysis are desired, specific, more limited temperature ranges may be used. Similarly, several heating rates may be used during analysis in those regions of greater or lesser interest. Isothermal periods may be necessary for some materials. See Table 1 for suggested parameters.

11.10 Once a mass loss plateau is established in the range 600 to 950 °C, depending on the material, switch from inert to reactive (air or oxygen) environment.

TABLE 1 Suggested Compositional Analysis Parameters

Material	Sample Size mg	Flow Rate mL/min ^A	Purge Time Min	Temperature				Heating Rate °C/min	Gas Switchover °C
				Initial	X	Y	Z ^B		
coal	20	50	5	ambient	110	900	900	10 to 150	900
elastomers	20	50	2	ambient	325	550	750	10	600
thermoplastics	20	50	2	ambient	200	600	750	10	600
lubricants	20	40 to 500	1	50	150	600	750	10 to 100	600
thermosets	20	50	2	ambient	200	550	750	10	600

^AMay differ depending upon instrument design.

^BZ is not necessarily the final temperature.