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Standard Test Methods for Loss-On-Drying by Thermogravimetry[1]

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1. Scope

1.1 These test methods describe a procedure for determining the amount of volatile matter of any kind that is driven off from a test specimen under a specific set of temperature and time conditions. These test methods determine only the mass of material lost, not its identity.

1.2 These test methods are applicable to a wide variety of solid or liquid materials, mixtures, or blends where the major component is stable at the test temperature.

NOTE 1—These test methods can be applied to the analysis of volatile organic compounds (VOC) content in metalworking fluids and direct contact lubricants subject to South Coast Air Quality Management District (SCAQMD) Rule 1144.

1.3 The applicable temperature range for these test methods are generally between ambient temperature and 1000°C.

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 There is no ISO method equivalent to this test standard.

1.6 *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]

D6 Test Method for Loss on Heating of Oil and Asphaltic Compounds

D1475 Test Method For Density of Liquid Coatings, Inks, and Related Products

D1509 Test Methods for Carbon Black—Heating Loss

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D2288 Test Method for Weight Loss of Plasticizers on Heating (Withdrawn 2010)[3]

D2595 Test Method for Evaporation Loss of Lubricating Greases Over Wide-Temperature Range

D2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings

D3175 Test Method for Volatile Matter in the Analysis Sample of Coal and Coke

D4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method

D4893 Test Method for Determination of Pitch Volatility

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E359 Test Methods for Analysis of Soda Ash (Sodium Carbonate)

E473 Terminology Relating to Thermal Analysis and Rheology

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E897 Test Method for Volatile Matter in the Analysis Sample of Refuse-Derived Fuel (Withdrawn 2011)[3]

E1142 Terminology Relating to Thermophysical Properties

E1582 Practice for Calibration of Temperature Scale for Thermogravimetry

E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers

E2040 Test Method for Mass Scale Calibration of Thermogravimetric Analyzers

2.2 *SCAQMD Documents:*[4]

Rule 1144 Metalworking Fluids and Direct-Contact Lubricants

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this test method are defined in Terminology E473 and Terminology E1142, including *thermogravimetry, thermogravimetric analyzer, repeatability,* and *reproducibility*.

[1] These test methods are under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Calorimetry and Mass Loss.

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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] Available from South Coast Air Quality Management District (SCAQMD), 21865 Copley Drive, Diamond Bar, CA, 91765, http://aqmd.gov.

4. Summary of Test Method

4.1 A specimen of known mass is heated at a constant temperature while its mass is continuously measured as a function of time. At the end of a pre-determined time interval, or when the loss reaches a pre-determined rate, the mass loss of the specimen is recorded as a percent of the original mass. This value is identified as the loss-on-drying (LOD) value. The LOD value is a function of both temperature and time. Therefore these values must be identified and reported. A typical LOD value is reported as *LOD* = XX % (60 min at 120°C). The volatile content, *V* (g/L), or VOC content, *VOC* (g/L), may be calculated.

NOTE 2—For SCAQMD Rule 1144 purposes, at the end of a pre-determined time interval and specified temperature, the mass loss of the specimen is recorded as a percent of the original mass. Additionally, the density and water content of the sample are determined. These values are then used to calculate the VOC content.

5. Significance and Use

5.1 These test methods are used to estimate the amount of volatile materials present in a material.

5.2 These test methods are useful for design purposes, service evaluation, regulatory statutes, manufacturing control, quality control, specification acceptance, development, and research.

5.3 The results obtained by these test methods may be equivalent to those obtained by other test methods and may be known by other terms in their respective fields. Other tests and terms encountered include loss-on-heating (see Footnote [5] and Test Methods D6, D2288, and E359); heating loss (see Test Method D1509); evaporative loss (see Test Method D2595); volatile organic carbon, moisture, or water (see Test Methods D2216 and D3175); volatility (see Test Method D4893); highly volatile matter (see Test Method E897); and volatile content (see Guide D2832).

6. Interferences

6.1 Because the specimen size is usually small, care must be taken to ensure that each specimen is representative of the sample as a whole.

6.2 This test procedure measures total mass loss under specific experimental conditions. If more than one volatile component is present, the results will reflect the total of all those volatile components present.

6.3 If the test temperature is set too high, the resultant weight loss may include some decomposition of the matrix material.

6.4 When calculating VOC content for SCAQMD Rule 1144 purposes, it may be necessary to measure and correct for water content. Refer to Test Method D4017 to determine the water content of the specimen.

[5] *Formulary Vol XVII* is available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, http://www.usp.org.

7. Apparatus

7.1 *Thermogravimetric Analyzer,* capable of continuously recording specimen mass and temperature as a function of time consisting of:

7.1.1 *Electrobalance,* with a minimum specimen capacity of 100 mg capable of continuously recording 10 µg or smaller mass changes. Performance may be verified in accordance with Test Method E2040.

7.1.2 *Specimen Holders,* that are inert to the specimen and of suitable structural shape and integrity to contain the 10 mg test specimen used in these test methods. Specimen holders, composed of platinum, aluminum, or quartz may be used, but other holders may be considered.

NOTE 3—For SCAQMD Rule 1144 purposes, specimen holders must be shallow and composed of platinum.

7.1.3 *Furnace,* whose temperature can be controlled from 25 to 1000°C, capable of a heating rate, at a minimum, of 5°C/min and of maintaining a set temperature isothermally within that range to ±2°C.

7.1.4 *Temperature Sensor* to provide an indication of the specimen or furnace temperature, or both, to ±0.1°C.

NOTE 4—The temperature sensor shall be placed as close as practical to the test specimen without disturbing weight measurements or as specified by the manufacturer. In addition, it must be located in exactly the same position during analytical determinations as used during calibration.

7.1.5 *Specimen Atmosphere Control System,* capable of supplying inert dry gas (usually purified grade nitrogen) with an operator selectable flow rate of 50 to 100 mL/min to within ±5 mL/min.

NOTE 5—For SCAQMD Rule 1144 purposes, use purified grade nitrogen for both the sample purge flow and balance protection flow.

7.1.6 *Measurement System,* to continuously record specimen temperature to within ±0.1°C over the range from 25 to 1000°C.

7.1.7 *Timer,* capable of continuously recording elapsed time up to 20 h to within ± 0.1 min or ±1 %, whichever is greater. Performance may be verified in accordance with Test Method E1860.

7.1.8 *Controller,* capable of executing a temperature program by operating the furnace from 25 to 1000°C at a minimum rate of 5°C/min to within ±0.1°C/min and of maintaining a set temperature isothermally within the range of ±2°C.

7.1.9 *Data Collection Device,* provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for thermogravimetric analysis (TGA) are mass, temperature, and time.

7.1.10 While not required, it is convenient to have a data analysis device that will continuously perform and display the following calculation:

7.1.10.1 Specimen mass as a percent of the initial mass.

7.1.10.2 Specimen mass rate of change (in mass %/min) capable of detecting 0.01 %/min.

7.1.11 While not required, it is convenient to have an experiment control device capable of terminating the experiment under the following conditions: