Designation: E1868 – 09

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

This standard is issued under the fixed designation E1868; 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 describes 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. This test method determines only the mass of material lost, not its identity.

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

1.3 The applicable temperature range for this test method is 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

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[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

D4893 Test Method for Determination of Pitch Volatility

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

E473 Terminology Relating to Thermal Analysis and Rheology

E897 Test Method for Volatile Matter in the Analysis Sample of Refuse-Derived Fuel

E1131 Test Method for Compositional Analysis by Thermogravimetry

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

3. Terminology

3.1 *Definitions*:

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

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).

[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 Calorimetry and Mass Loss.

Current edition approved Sept. 1, 2009. Published October 2009. Originally approved in 1997. Last previous edition approved in 2004 as E1868 – 04. DOI: 10.1520/E1868-09.

[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] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

5. Significance and Use

5.1 This test method is used to estimate the amount of volatile materials present in a material.

5.2 This test method is useful for design purposes, service evaluation, regulatory statues, manufacturing control, quality control, specification acceptance, development and research.

5.3 The results obtained by this test method 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 [4] 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, or both.

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.

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 this test method. Specimen holders, composed of platinum, aluminum or quartz may be used, but other holders may be considered.

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

7.1.4 *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.

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

7.1.6 *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.

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

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

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

7.1.9.1 Specimen mass as a percent of the initial mass.

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

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

7.1.10.1 When an operator selected period of time at an isothermal temperature condition has elapsed, and

7.1.10.2 When an operator selected rate of mass loss is achieved.

7.2 *Gas Exhaust System*, capable of removing from the laboratory the potentially noxious purge gas effluent of the system above.

7.3 *Inert Gas*—Purified grade nitrogen.

7.4 *Micropipets or Syringes*, for liquids, capable of dispensing up to 15 ± 1 µL.

8. Hazards

8.1 Toxic or corrosive effluent, or both, may be released when heating some materials and could be harmful to personnel and to apparatus.

9. Sampling

9.1 Samples are usually analyzed on “as received” basis. Should some thermal or mechanical treatment (such as grinding, or sieving) be applied to the sample prior to analysis, it shall be indicated in the report. Grinding may release volatiles due to the heating generated by grinding process.

9.2 Since small test specimens are used, they must be homogeneous and representative of the sample. The mixing or stirring of samples prior to analysis is recommended whenever possible.

10. Calibration

10.1 Calibrate the temperature signal from the apparatus according to Practice E1582 using a heating rate of 1 °C/min and a transition temperature close to the isothermal test temperature used in this procedure.

10.2 Calibrate the mass signal from the apparatus according to Test Method E2040.

NOTE 1—Regular analysis of performance standards (materials of

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