Designation: E 1868 – 97

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428

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

This standard is issued under the fixed designation E 1868; 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 covers 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 Electronic instrumentation or automated data analysis and data reduction systems or treatments equivalent to this method may be used.

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

1.5 The values stated in SI units are to be regarded as the 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:*

D 6 Test Method for Loss on Heating of Oil and Asphaltic Compounds[2]

D 1509 Test Method for Carbon Black—Heating Loss[3]

D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock[4]

D 2288 Test Method for Weight Loss of Plasticizers on Heating[5]

D 2595 Test Method for Evaporation Loss of Lubricating Greases Over Wide-Temperature Range[6]

D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings[7]

D 3175 Test Method for Moisture in the Analysis Sample of Coal and Coke[8]

D 4893 Test Method for Determination of Pitch Volatility[2]

E 359 Test Method for Chemical Analysis of Soda Ash (Sodium Carbonate)[9]

E 473 Terminology Relating to Thermal Analysis[10]

E 897 Test Method for Volatile Matter in the Analysis Sample of Refuse-Derived Fuel[11]

E 1131 Test Method for Compositional Analysis by Thermogravimetry[10]

E 1142 Terminology Relating to Thermophysical Properties[10]

E 1582 Practice for Calibration of Temperature Scale for Thermogravimetry[10]

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this test method are defined in Terminology E 473 and Terminology E 1142.

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).

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 12 and Test Methods D 6, D 2288, and E 359), heating loss (see Test Method D 1509), evaporative loss (see Test Method D 2595), volatile organic carbon, moisture or water (see Test Methods D 2216 and D 3175), volatility (see Test Method D 4893), highly volatile matter (see Test Method E 897), and

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods.

Current edition approved March 10, 1997. Published May 1998.

[2] *Annual Book of ASTM Standards*, Vol 04.04.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 08.01.

[6] *Annual Book of ASTM Standards*, Vol 05.01.

[7] *Annual Book of ASTM Standards*, Vol 06.01.

[8] *Annual Book of ASTM Standards*, Vol 05.05.

[9] *Annual Book of ASTM Standards*, Vol 15.05.

[10] *Annual Book of ASTM Standards*, Vol 14.02.

[11] *Annual Book of ASTM Standards*, Vol 11.04.

volatile content (see Guide D 2832).

6. Interferences

6.1 Because the specimen size is usually small, care must be taken to ensure that each specimen is homogeneous or 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 capacity of 100 mg capable of continuously recording 10 μg or smaller mass changes.

7.1.2 *Specimen Holders*, that are inert to the specimen and which are 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*, the temperature of which may be controlled from 25 to 1000°C, with 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 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.

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 *Recording Device*, either digital or analog, to record and display the thermogravimetric thermal curve consisting of mass, and temperature as a function of time to the specifications above.

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 an "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.

10. Calibration

10.1 Calibrate the temperature signal from the apparatus according to Practice E 1582 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 and time signals from the apparatus according to the manufacturer's instructions.

NOTE 2—Regular analysis of performance standards (materials of known mass loss characteristics) where available,[13] serves as a check on instrument status, operator efficiency, etc. and provides for a comparison of results from different laboratories.

11. Procedure

11.1 Cool the specimen test area of the apparatus to ambient temperature. For the purpose of this test, ambient temperature is 35°C or lower.

NOTE 3—Cooling of the specimen test area to 25°C following an experiment is time consuming on some apparatus. To improve productivity, it is possible with some test samples to initiate the experiment at a somewhat higher temperature. This must be done with caution since volatility is a function of temperature. For highly volatile materials, appreciable portions of the test specimen mass may be lost in experimental set up, if initiated at too high a temperature.

11.2 With the apparatus closed in the normal operating position, tare the balance so that the empty sample pan indicates zero mass.

11.3 Open the apparatus to expose the specimen holder.

11.4 Carefully place 10 ± 1 mg of the test specimen on the specimen holder. Other specimen sizes may be used but must be indicated in the report.

11.5 Close the apparatus and record the initial mass as m_i. If the apparatus has provisions for direct recording of mass percent, adjust it to read 100 %.

[12] Formulary Vol XVII, United States Pharmacopoeia Convention, 12601 Twinbrook Parkway, Rockville, MD 20852.

[13] Performance Standards for low (2 %), medium (50 %) and high (99 %) LOD values are available from Rose Consulting, 579 Kelly Avenue, Half Moon Bay, CA 94019.