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D2288 Test Method for Weight Loss of Plasticizers on Heating (Withdrawn 2010)[3]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

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[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.

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:

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

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

7.1.11.2 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 *Micropipettes 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.

9.3 If experiments are to be initiated at room temperature, equilibrate samples in their sealed containers to room temperature prior to testing.

NOTE 6—For SCAQMD Rule 1144 purposes, samples containing solid particles should be filtered prior to analysis.

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 7—Regular analysis of performance standards (materials of known mass loss characteristics) where available,[6] serves as a check on instrument status, operator efficiency, etc., and provides for a comparison of results from different laboratories. Verify mass measurement conformity to better than 1 %. Verify time measurement conformity to better than 1 %.

10.3 Calibrate the time signal from the apparatus according to Test Method E1860.

NOTE 8—For SCAQMD Rule 1144 purposes, calibrate the mass signal every week, the temperature signal every three months, and the time signal every six months.

11. Procedure

NOTE 9—For SCAQMD Rule 1144 purposes, additional proceedures may be required by the agency.

11.1 Obtain a suitable baseline correction.

11.2 Set the furnace and flow parameters.

NOTE 10—For SCAQMD Rule 1144 purposes, the total flow must be 50 mL/min, divided (balance protection flow:sample purge flow) either 10:40 mL/min or 20:30 mL/min.

11.3 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 11—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 of a temperature.

NOTE 12—For SCAQMD Rule 1144 purposes, initial temperature is 25 ± 2°C.

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

11.5 Open the apparatus to expose the specimen holder.

11.6 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.

NOTE 13—Care should be taken to ensure that the sample is free of air bubbles.

NOTE 14—For SCAQMD Rule 1144 purposes, specimen size is 10 ± 1 µL, autosamplers are not to be used and the specimen must be distributed evenly over the bottom of the specimen holder.

11.7 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 %.

NOTE 15—Should the test specimen have appreciable volatility at ambient temperature, it may lose a significant amount of mass as it is placed into the test position. In such cases, care should be taken to perform 11.6 and 11.7 as quickly as is practical.

11.8 Heat the test specimen at 5°C/min to the isothermal test temperature, while recording specimen mass (or mass percent), temperature and elapsed time. Other heating rates may be used, but must be indicated in the report.

NOTE 16—Selection of the optimum operating parameters of isothermal test temperature and test time (t_t) is dependent on the class of materials being tested. The technique of response surface methodology (RSM) may be useful in predicting optimum operating parameters.[7,8] Some common experimental conditions are presented in Tables 1 and 2.

NOTE 17—For SCAQMD Rule 1144 purposes, heating rate is 25°C/min and isothermal test temperature is 81°C.

11.9 Once the isothermal test temperature is reached, it is held there for the remainder of the experiment. The time from start to the isothermal portion of the experiment is taken as t_i.

11.10 After 5 min at the isothermal test temperature, the specimen temperature may be recorded.

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[6] Performance Standards for low (2 %), medium (50 %), and high (99 %) LOD values are available from several suppliers. Contact ASTM International for list of suppliers.

[7] Box, G.E.P., and Hunter, J.S., "Multi Factor Experimental Designs for Exploring Response Surfaces," *Annals of Mathematical Statistics*, Vol 28, 1997, pp. 195–241.

[8] Pino, J.A., Barry, T.A., and Rose, J.E., "Estimation of Volatile Emission Potential of Pesticides by Thermogravimetric Analysis," *Proceedings 110th American Official Analytical Chemists Meeting*, 1996.

TABLE 1 Commonly Used Isothermal Test Temperatures and Times

Material	Temperature, °C	Time, h	Test Method
Grease	93	20	D2595
Barium Hydroxide	105	2	[A]
Barium Sulfate	105	4	[A]
Calcium Carbonate	200	4	[A]
Lubricants	81	1.83 (110 m. from start)	E1868
Pesticides	65	11	E1868
Refuse Derived Fuel	950	0.1	E897
Pitch	360	0.5	D4893
Soil and Rock	110	12	D2216
Polymers	155	24	D2288
Carbon Black	125	1	D1509
Soda Ash	250	4	E359
Asphalt	163	5	D6

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

TABLE 2 Commonly Used Rates of Loss and Delay Times

Material	Rate of Loss, %/min	Delay Time, min	Reference no
Pesticides	0.1	15	2.2[A]

[A] Pino, J., Barry, T., and Rose, J., *Estimation of Volatile Emission of Pesticides by Thermogravimetric Analysis*, California Environmental Protection Agency, 1020 N Street, Room 161, Sacramento, CA 95814-5624.

11.11 The experiment is terminated in one of two ways. These termination criteria are identified as Test Method A and Test Method B.

11.12 *Test Method A:*

11.12.1 After the test specimen is held at the isothermal test temperature for a fixed period of test time (t_t).

NOTE 18—Commonly used isothermal test temperatures and times are tabulated in Table 1.

NOTE 19—For SCAQMD Rule 1144 purposes, test time (t_t) is 110 minutes. Test time is measured from the start of the heating cycle to the end of the isothermal test time.

11.12.1.1 Experiments terminated using Test Method A shall indicate the termination conditions in parentheses following the LOD values. For example: $LOD = XX\,\%$ (2 h at 105°C).

11.13 *Test Method B:*

11.13.1 After a delay time (t_d) initiated when the mass loss rate becomes less than a specified value (R).

NOTE 20—Commonly used values for mass loss rate and delay time are tabulated in Table 2.

NOTE 21—For apparatus so equipped, the mass loss rate may be conveniently observed using the derivative of mass as a function of time.

11.13.1.1 Experiments terminated using Test Method B shall indicate the termination conditions in parentheses following the LOD values. For example: $LOD = XX\,\%$ (15 min at <1 %/min).

11.14 Record the mass at the time of the termination of the experiment. This value is the final mass, m_f.

11.15 Calculate the percent mass loss value according to Eq 1 (see 12.1).

11.16 Perform duplicate determinations and report the loss-on-drying value as the mean value of duplicate determinations of percent mass loss.

NOTE 22—For SCAQMD Rule 1144 purposes, perform duplicate determinations. Unless SCAQMD quality control procedures fail, calculate and report VOC content according to Eq 3 (see 12.3) as the mean value of duplicate determinations.

12. Calculation

12.1 *Loss-on-Drying:*

12.1.1 Calculate percent mass loss according to the following equation:

$$m = \frac{(m_i - m_f) \times 100\,\%}{m_i} \tag{1}$$

$$LOD = \text{mean value of } m \text{ for duplicate determinations}$$

where:

LOD = loss-on-drying (%),
m = percent mass loss (%),
m_i = initial mass (mg), and
m_f = final mass (mg).

12.2 *Volatile Material Content:*

12.2.1 Calculate the amount of volatile material, V, in grams per litre from a liquid sample, according to the following equation:

$$v = \left[\frac{(m_i - m_f)}{m_i}\right] \times \rho \times 1000\,\text{mL/L} \tag{2}$$

where V, the volatile content in g/L, is the mean value of v for replicate determinations, where the number of determinations may be one, and where:

v = volatile content of a single measurement (g/L),
ρ = density of liquid sample (g/mL),
m_i = initial mass (mg), and
m_f = final mass (mg).

12.3 *Volatile Organic Compounds (VOC) Content:*

12.3.1 Calculate the volatile organic compounds content, *VOC* content, in grams per litre from a liquid sample according to the following equation:

$$voc = \left\{\left[\frac{(m_i - m_f)}{m_i}\right] - \frac{w}{100\,\%}\right\} \times \rho \times 1000\,\text{mL/L} \tag{3}$$

where *VOC*, the volatile organic compounds content in g/L, is the mean value of *voc* for replicate determinations, where the number of determinations may be one, and where:

voc = volatile organic compounds content of a single measurement (g/L),
ρ = density of liquid sample (g/mL),
w = water content (%),
m_i = initial mass (mg), and
m_f = final mass (mg).

NOTE 23—For SCAQMD Rule 1144 purposes, use Test Method D1475 for the determination of density and Test Method D4017 for the determination of the water content of the sample.

NOTE 24—Measured values should be entered into the equation retaining all decimal places. Only the final report value should be rounded in accordance with the established precision of the test.

13. Report

13.1 Report the following information:

13.1.1 A complete identification and description of the material being tested,

13.1.2 A description of the instrument used to perform the test including manufacturer and model number,

13.1.3 A description of the temperature calibration material and procedure used,

13.1.4 The thermal curves for the determination via the method, and

13.1.5 The LOD value as well as the experiment termination conditions (see 11.12.1.1 and 11.13.1.1). For example: 5.0 % (2 h at 105°C). V or VOC values may also be reported as necessary.

NOTE 25—For SCAQMD Rule 1144 purposes, report VOC.

13.2 The specific dated version of this method used.

NOTE 26—For SCAQMD Rule 1144 purposes, consult with the agency for any other specific reporting requirements.

14. Precision and Bias[9]

14.1 *Test Method A:*

14.1.1 An interlaboratory study (ILS) was conducted for Test Method A in 2009–2010 on behalf of the SCAQMD (ILS 500).[4] Eight laboratories participated in this study. Each of the labs was asked to report five replicate test results for four different liquid lubricant sample materials. Samples D, F, and H were analyzed by all eight laboratories. Sample G was analyzed by seven laboratories. Every "test result" reported represented a single determination or measurement; data is expressed as volatile organic compounds in grams per litre. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:E37-1039.[10] See the research report for additional method parameters specific to these lubricant samples.

14.1.2 *Precision:*

14.1.2.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

(1) Repeatability limits are listed in Table 3 below. The repeatability value estimates the 95 % confidence limit.

14.1.2.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

TABLE 3 Volatile Compounds (g/L)

Sample	Average[A] $\bar{X}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
D	633	12	16	32	44
F	59.0	3.6	11	9.9	32
G	970.1	3.6	21	10	59
H	89.2	5.1	11	14	30

[A] The average of the laboratories' calculated averages.

(1) Reproducibility limits are listed in Table 3 below. The reproducibility value estimates the 95 % confidence limit.

14.1.2.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

14.1.2.4 Any judgment in accordance with statements 14.1.2.1 and 14.1.2.2 would have an approximate 95 % probability of being correct.

14.1.3 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

14.1.4 The precision statement was determined through statistical examination of 155 results, from 8 laboratories, on a total of 4 different sample materials. These materials were described as:

D	Rust preventive, formulated from a combination of proprietary rust preventive, 80 % Stoddard Solvent (flash point, 40°C) and 10 % napthenic mineral oil (viscosity, approximately 1.3 cSt.), density, 0.7873 g/mL
F	Oil-based cutting fluid (general purpose), formulated from a proprietary combination of lubricity agents and 70 % napthenic mineral oil (viscosity, approximately 32.3 cSt.), density, 0.9439 g/mL
G	Proprietary water-diluted semisynthetic cutting fluid, 93 % water, density, 1.0084 g/mL
H	Oil-based cutting fluid (aluminum), formulated from a combination of lubricity agents and 70 % napthenic mineral oil (viscosity approximately 6.9 cSt.), density, 0.8852 g/mL

To judge the equivalency of two test results, it is recommended to choose a sample material closest in characteristics to the test material.

14.2 *Test Method B:*

14.2.1 An ILS was conducted for Test Method B in 1995–1996 by the California Environmental Protection Agency. The ILS involved participation by 15 laboratories using 3 performance standards, 3 liquid and 2 solid end-use materials (that is, pesticides). The performance standards were synthetic materials made up to contain low, medium and high levels of volatile corresponding to 2, 50, and 99 % mass loss.

14.2.2 *Precision:*

14.2.2.1 Within laboratory variability may be described using the repeatability value (*r*), obtained by multiplying the standard deviation by 2.8. The repeatability value estimates the 95 % confidence limit.

14.2.2.2 Repeatability values of $r = 0.6$, 0.4, and 0.5 % mass loss were obtained on the low, medium and high loss performance standards, respectively.

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[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1020. Contact ASTM Customer Service at service@astm.org.

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1039. Contact ASTM Customer Service at service@astm.org.

14.2.2.3 Repeatability values of $r = 4.2$, 1.8, and 0.6 % mass loss were obtained on the three liquid end-use materials.

14.2.2.4 Repeatability values of $r = 6.2$ and 1.4 % mass loss were obtained on the two solid end-use materials.

14.2.2.5 Between laboratory variability may be described using the reproducibility value (R) obtained by multiplying the standard deviation by 2.8. The reproducibility value estimates the 95 % confidence limit.

14.2.2.6 Reproducibility values of $R = 4.4$, 1.0, and 0.3 % mass loss were obtained on the low, medium, and high loss performance standards, respectively.

14.2.2.7 Reproducibility values of $R = 4.9$, 2.2, and 0.7 % mass loss were obtained on the three liquid end-use materials.

14.2.2.8 Reproducibility values of $R = 6.6$ and 2.0 % mass loss were obtained on the two solid end-use materials.

14.2.3 *Bias:*

14.2.3.1 An estimation of bias is obtained by comparing the mean mass loss values obtained for each performance standard to their synthesize values. That is:

$$\text{bias} = (\text{mean mass loss}) - (\text{known values}) \tag{4}$$

(1) The bias was found to be less than 0.6 % mass loss for all three levels of the performance standards. This indicates an absence of significant relative bias.

15. Keywords

15.1 loss on drying; mass loss; thermogravimetry; thermogravimetric analysis; volatiles; weight loss

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