Designation: ~~E2403 – 06 (Reapproved 2018)~~ E2403 – 23

Standard Test Method for Sulfated Ash of Organic Materials by Thermogravimetry[1]

This standard is issued under the fixed designation E2403; 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~~ Scope*

1.1 This test method describes the determination of sulfated ash content (sometimes called residue-on-ignition) of organic materials by thermogravimetry. This test method converts common metals found in organic materials (such as sodium, potassium, lithium, calcium, magnesium, zinc, and tin) into their sulfate salts permitting estimation of their total content as sulfates or oxides. The range of this test method is from ~~0.1~~0.1 % to 100 % metal content.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D874 Test Method for Sulfated Ash from Lubricating Oils and Additives
D914 Test Methods for Ethylcellulose
D3516 Test Methods for Ashing Cellulose
E473 Terminology Relating to Thermal Analysis and Rheology
E1131 Test Method for Compositional Analysis by Thermogravimetry
E1142 Terminology Relating to Thermophysical Properties
E1582 Test Method for Temperature Calibration of Thermogravimetric Analyzers
E2040 Test Method for Mass Scale Calibration of Thermogravimetric Analyzers
E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

2.2 *Other Standards:*

The United States Pharmacopeia XXII and The National Formulary XVII, United States Pharmacopeial Convention, Rockville, MD, 1990, Section 281, p. 1527

3. Terminology

3.1 *Definitions*—Technical terms used in this standard are defined in Terminologies E473 and E1142.

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[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.

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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.

***A Summary of Changes section appears at the end of this standard**

3.1.1 *residue-on-ignition, ROI, n*—a commonly used alias for sulfated ash.

3.1.2 *sulfated ash, n*—the residue remaining after a specimen has been oxidized, and the residue subsequently treated with sulfuric acid and heated to constant weight.

~~3.1.2 *residue-on-ignition, ROI, n*—a commonly used alias for sulfated ash.~~

3.1.3 *volatiles, n*—for the purpose of this test, those materials evolving as gas at temperatures below ~~160°C~~160 °C in an air atmosphere.

4. Summary of Test Method

4.1 A test specimen is ignited and burned in an air atmosphere at temperatures up to ~~600°C~~600 °C until only ash remains. After cooling, the residue is treated with sulfuric acid and heated to ~~800°C~~800 °C to constant weight. The residue remaining is identified as sulfated ash.

4.2 This test method is similar to Test Method D874 for lubricating oils and additives, Test Methods D914 for ethyl cellulose, Test Methods D3516 cellulose, and that of The United States Pharmacopeia XXII and makes use of thermogravimetric apparatus to perform the determination.

5. Significance and Use

5.1 The sulfated ash ~~may be used to indicate~~ value indicates the level of known metal-containing additives or impurities in an organic material. When phosphorus is absent, barium, calcium, magnesium, sodium and potassium are converted to their sulfates. Tin and zinc are converted to their oxides.

5.2 This test method may be used for research and development, specification acceptance, and quality assurance purposes.

6. Interferences

6.1 If phosphorus is present with metals, it partially or wholly remains in the sulfated ash as metal phosphates.

6.2 Sulfur and chlorides do not interfere.

7. Apparatus

7.1 *Thermogravimetric Analyzer* ~~(TGA)—~~(TGA)—The essential instrumentation required to provide the minimum thermogravimetric analytical capability for this test method includes:

7.1.1 A thermobalance composed of:

7.1.1.1 A furnace to provide uniform controlled heating of a specimen to a constant temperature of ~~850°C~~850 °C and at a constant rate of ~~5~~5 °C/min to ~~60°C/min~~60 °C/min

7.1.1.2 A temperature sensor to provide an indication of the specimen or furnace temperature readable to ~~±1°C.~~±1 °C.

7.1.1.3 A continuously recording balance to measure the specimen mass with a minimum capacity of 50 mg ~~and a sensitivity of~~ readable to ±0.01 mg.

7.1.1.4 A means of maintaining the specimen or container under atmospheric control of air at a purge flow rate of ~~50~~50 mL/min to 100 mL/min ± 5 mL/min.

7.1.2 A temperature controller capable of executing a specific temperature program by operating the furnace between selected temperature limit at a rate of ~~5~~5 °C/min to ~~60°C/min~~60 °C/min and to an isothermal temperature of up to ~~850°C~~850 °C which is maintained constant to ~~±10°C~~±10 °C for a minimum of 70 ~~minutes.~~min.

7.1.3 A ~~*recording*~~ *data collection device*, ~~capable of recording and displaying any fraction of the specimen mass signal (TGA curve) including signal noise.~~ to 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.

7.1.4 Containers (pans, crucibles, etc.) that are inert to the specimen and to concentrated sulfuric acid and that will remain gravimetrically stable up to ~~850°C.~~850 °C. Platinum is a common material of construction for this purpose.

7.2 Graduated micropipette with capacity of ~~40–50~~ 40 µL to 50 µL

8. Reagents and Materials

8.1 Sulfuric acid, concentrated (98 %), with a relative density of 1.84. (**Warning—**Poison. Corrosive. Strong Oxidizer.)

8.2 *Air*—Zero grade or better purity

9. Hazards

9.1 Sulfuric acid may be corrosive to some thermogravimetric apparatus. A regular visual inspection of the apparatus will determine if any corrosion is taking place.

9.2 The exhausted purge gas from the apparatus will contain sulfuric acid fumes. This purge gas shall be treated by exhausting to ~~a~~an acid hood or by bubbling through a solution of sodium bicarbonate to absorb the acidic fumes.

10. Preparation of Apparatus

10.1 After turning the power on, allow the instrument to equilibrate for at least one hour prior to any measurements.

10.2 Perform any cleaning and calibration procedures described by the manufacturer in the apparatus Operator's Manual.

11. Calibration and Standardization

11.1 Perform temperature calibration of the thermogravimetric analyzer according to Test Method E1582 using reference materials suitable for the temperature range of this method, namely ~~25 to 800°C.~~25 °C to 800 °C (see Appendix X1).

11.2 Perform mass calibration of the thermogravimetric analyzer according to Test Method E2040.

NOTE 1—Committee E37 recommends calibration, or calibration verification, of all signals at least annually.

12. Procedure

12.1 Transfer ~~3~~030 mg to 40 mg of the sample into a tared, clean, and dry sample container. Assemble the thermogravimetric analyzer for operation. Record the initial weight of the test specimen as W_o to within ±0.01 mg

NOTE 2—The sample container may be preconditioned by heating in an air atmosphere to ~~800°C.~~800 °C.

NOTE 3—Smaller quantities of test specimen will reduce the quantification capability of this method.

12.2 Heat the specimen from ambient to ~~600°C at 10°C/min~~600 °C at 10 °C/min under an air purge gas with a flow rate of ~~50~~50 mL/min to ~~100~~100 mL ~~± 5~~/min ± 5 mL/min and record the thermal curve.

12.3 Cool the heated sample and thermogravimetric apparatus to ~~20 to 25°C.~~20 °C to 25 °C.

12.4 If desired, record the weight at ~~150°C~~150 °C (W_v) as the mass after apparent loss of volatiles. Record the weight at ~~600°C~~600 °C as the mass of the residue (W_r).

NOTE 4—Mass loss due to apparent loss of volatiles at ~~150°C~~150 °C and residue at ~~600°C~~600 °C are not required for the sulfated ash determination but may be recorded for additional sample characterization (see Test Method E1131).

NOTE 5—The temperature at which the mass loss due to volatiles is determined may range from ~~100 to 160°C~~100 °C to 160 °C depending upon the material. Other values may be used but shall be reported.

12.5 Using a micropipette, add ~~30 –~~30 µL to 40 µL of 98 % grade sulfuric acid to the sample residue (in the container). Reassemble the instrument for operation.

12.6 Heat the specimen from ambient to ~~800°C at 50°C/min~~800 °C at 50 °C/min and hold isothermally at ~~800 ± 25°C for 60 ± 1 minutes~~800 °C ± 25 °C for 60 min ± 1 min under an air purge at ~~50~~50 mL/min to ~~100~~100 mL/min ± ~~5~~ 5 mL ~~mL/min.~~/min.

12.7 Cool the heated specimen and thermogravimetric apparatus to ~~20 to 25°C.~~20 °C to 25 °C.

12.8 Record the residue mass W_s.

12.9 Calculate percent Sulfated Ash (S) using Eq 1.

12.10 In the absence of interferences and if the specific metal in the test specimen is known, then its mass percent (M) may be calculated by multiplying the sulfated ash value by the factors presented in Table 1 using Eq 2.

12.11 If desired, calculate the Percent Volatiles (V) and Percent Residue (R) using equations 3 and 4, respectively.

13. Calculation

13.1 Sulfated Ash (S) may be determined using the following equation:

$$S = W_s \times 100\ \%/W_o \tag{1}$$

where:

S = Sulfated ash, mass %,
W_s = Mass of sulfated ash from Section 12.8, mg,
W_o = Original mass of the test specimen from Section 12.1, mg.

13.2 If the specific metal in the test specimen is known and if this is the only metal ion present, then its mass percent (M) may be calculated by multiplying the sulfated ash value by the factors presented in Table 1 using Eq 2.

$$M\ (metal) = \text{Factor} \times S \tag{2}$$

where:

M = Percent of the identified metal, mass %.

14. Report

14.1 Report the sulfated ash (S) to three significant figures.

TABLE 1 Sulfated Ash Factors

Metal	Sulfated Ash	Factor
Sodium (Na)	Na_2SO_4	0.3237
Potassium (K)	K_2SO_4	0.4487
Lithium (Li)	Li_2SO_4	0.1263
Calcium (Ca)	$CaSO_4$	0.2944
Magnesium (Mg)	$MgSO_4$	0.2019
Zinc (Zn)	ZnO	0.8034
Tin (Sn)	SnO	0.8812