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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Ash in Polybasic Acids[1]

This standard is issued under the fixed designation E 347; 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 the determination of the ash content of polybasic acids after a sample has been completely burned and ignited. This test method is particularly designed for low ash content (5 to 200 ppm).

1.2 *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:*

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[2]

3. Summary of Test Method

3.1 The sample is burned in a tared platinum dish or crucible, the residue ignited in an electric muffle furnace, and the dish or crucible reweighed. Results are expressed in ppm ash.

4. Significance and Use

4.1 This test method provides a measurement of the ash content of polybasic acids. Ash is defined as the residual inorganic matter obtained on ignition of a sample in air at a specified temperature. As the ash content is frequently an important factor in the intermediate and end use of these acids, this test method provides a test procedure for specification acceptance and manufacturing control.

5. Apparatus

5.1 *Platinum Dish*, 125-mL capacity.

5.2 *Electric Heater*,[3] 550-W, or bunsen burner.

5.3 *Electric Muffle Furnace*, capable of maintaining a temperature of 800 ± 25°C and 850± 25°C.

6. Procedure

6.1 Carefully clean the platinum dish until it is bright and free from stains, both inside and outside. This can best be accomplished by scouring with sea sand, fusing with potassium bisulfate and removing the cooled melt with water, boiling in 1 % sulfuric acid, rinsing with distilled water, and wiping dry with a cloth. Ignite the cleaned dish in the muffle furnace for 30 min at 800 ± 25°C.

6.2 Remove the dish, allow it to cool in air for 3 min, then place it in a desiccator for 10 min or longer to attain room temperature. Weigh the dish at least two times (separate weighings) and record the tare weight only after two consecutive weighings have agreed to ±0.0001 g. Subsequent weighings should be made on the same balance and in the same manner.

NOTE 1—To obtain the required test precision in absolute values at low ash concentrations, the best possible weighing practices must be employed.

6.3 Weigh 100 ± 1 g of sample into the dish and place it on the electric heater located in a well-ventilated fume hood (Note 2). Turn the heater on full and allow to remain until the sample melts. (If more convenient, a bunsen burner may be used, the dish being supported on a wire triangle whose sides are covered with clay or silica tubes.) When the sample has just melted, turn off the heater and ignite the liquid with an ashless flame, for example, a gas burner.

NOTE 2—The sample may be added in increments, if necessary.

6.4 After the major portion of the sample has burned off, place the dish in the front part of the open muffle furnace to allow the residual vapors to ignite slowly to avoid mechanical loss. When the vapors stop burning, set the dish in the center of the muffle, close the door, and ignite at 800 ± 25°C (Note 3) for 15 to 30 min. Remove the dish, allow it to cool in quiet air for 3 min, place it in a desiccator for at least 10 min, then weigh it at least two times (separate weighings) until two consecutive weighings agree to ±0.0001 g. (See 6.2.)

NOTE 3—Materials that experience has shown to be difficult to ash to constant weight (±0.0001 g) should be ashed at 850 ± 25°C.

7. Calculation

7.1 Calculate the ash content as follows:

$$\text{ash, ppm} = (R/W) \times 10^6 \quad (1)$$

[1] This test method is under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and is the direct responsibility of Subcommittee E15.51 on Acids.

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

[3] Heater No. 61526, with a No. 61825 Top, available from the Precision Scientific Co., 3737 W. Cortland St., Chicago, IL 60647, has been found satisfactory for this purpose.

where:
R = grams of residue, and
W = grams of sample.

8. Report

8.1 Report the ash value to the nearest whole ppm. Duplicate runs that agree within the amount given in Table 1 are acceptable for averaging (95 % probability).

9. Precision and Bias

9.1 The following precision estimates are based on interlaboratory studies[4] of two samples of polybasic acid (adipic acid and sebacic acid having average ash values of 5 ppm and 220 ppm, respectively). Seven laboratories analyzed each sample in duplicate on separate days for a total of 56 determinations. Practice E 180 was used in developing these precision statements.

9.1.1 *Repeatability (Single Analyst)*—The standard deviation of results (each the average of duplicate determinations), obtained by the same analyst on different days, has been estimated to the amount given in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the value given in Table 1.

9.1.2 *Reproducibility (Multilaboratory)*—The standard deviation of results, obtained by analysts in different laboratories, has been estimated to be the amount given in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the value given in Table 1.

9.2 The bias of this test method has not been determined.

10. Keywords

10.1 ash; polybasic acids

[4] Supporting data are available on loan from ASTM Headquarters. Request RR: E15-100.

TABLE 1 Ash Precision Data

	Repeatability			Laboratory Precision			Reproducibility		
Level, ppm	Standard Deviation, ppm	Degrees of Freedom	95 % Limit, ppm	Standard Deviation, ppm	Degrees of Freedom	95 % Limit, ppm	Standard Deviation, ppm	Degrees of Freedom	95 % Limit, ppm
5	1.22	12	3	0.791	6	2	1.93	5	5
200	13	12	36	9.3	6	26	20.2	5	56