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Standard Test Methods for Ash Content of Adhesives[1]

This standard is issued under the fixed designation D 5040; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] NOTE—Section 13, Keywords, was added editorially in September 1997.

1. Scope

1.1 These test methods cover procedures used in determining the ash content of adhesives. This standard is intended as a replacement for Method 4032.1 of Federal Test Method Standard 175B, "Adhesives: Methods of Testing.''

1.2 Two test methods are used:

1.2.1 *Test Method A* is used for a starch adhesive or other type glue, where there is no danger from the non-volatile content forming a rubbery mass when ignited.

1.2.2 *Test Method B* employs nitric acid to avoid the non-volatile residue being transformed into a viscous foam when ignited.

1.3 These methods are not applicable to adhesives containing decomposable salts such as zinc chloride.

1.4 *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.* For a specific hazard statement, see 9.2.3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 301 Test Methods for Soluble Cellulose Nitrate[2]

D 907 Terminology of Adhesives[3]

D 2415 Test Method for Ash in Coal Tars and Pitches[4]

3. Terminology

3.1 *Definitions*—Definitions of terms in this standard may be found in Terminology D 907.

4. Summary of Test Methods

4.1 Ash content of adhesive materials is determined by heating a sample of material to remove all of the volatile components. Complete oxidation and removal of all carbonaceous material may be enhanced by the addition of concentrated HNO_3. The general method of burning the residual ash in a muffle furnace at 600 ± 25°C (1112 ± 45°F) for 8 h or until constant weight, is used.

5. Significance and Use

5.1 This test method measures the amount of inorganic material in the sample.

6. Apparatus

6.1 *Crucible*, porcelain, silica or platinum with a tightly fitting lid, having a capacity of 30 to 45 mL.

6.2 *Evaporating Dish*, porcelain, silica, or platinum, with a capacity of 150 mL.

6.2.1 *Watch Glasses* to cover evaporating dishes.

6.3 *Desiccator*, equipped with drying agent and tray.

6.4 *Analytical Balance*, sensitive to 1 mg.

6.5 *Steam Bath*.

6.6 *Drying Oven*, with temperature control for maintaining temperature at 100 to 105°C (212 to 221°F).

6.7 *Electric Hotplate*.

6.8 *Muffle Furnace*, for igniting crucibles containing test specimens. Capable of maintaining desired temperature regulation (600± 25°C (1112 ± 45°F)).

7. Reagents

7.1 *Nitric Acid* (HNO_3), concentrated, sp gr 1.42.

8. Test Specimens

8.1 For each test, use 5 to 6 g of material.

8.2 The specimen being tested should represent the entire lot of material. Two specimens shall be taken for testing from each sample unit.

9. Procedure

9.1 *Test Method A*— Place a 5.0 ± 0.5-g test specimen in an ignited and tared crucible and evaporate to dryness on a steam bath. Cool in a desiccator and weigh accurately. Heat the crucible and its contents in a muffle furnace, gradually heating the furnace until the temperature reaches 600 ± 25°C (1112 ± 45°F). With the ignition temperature kept at 600°C (1112°F),

[1] These test methods are under the jurisdiction of ASTM Committee D-14 on Adhesives and are the direct responsibility of Subcommittee D14.10 on Working Properties.

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 15.06.

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

allow the specimen to remain in the muffle furnace for 8 h or until constant weight is reached. Check weight by cooling in desiccator and weighing to constant weight.

9.2 *Test Method B*:

9.2.1 Place 5.0 ± 0.5 g test specimen in a tared and ignited dish and evaporate to dryness on a steam bath.

NOTE 1—It is necessary to evaporate all volatile solvent to avoid fire hazard when subjecting material to subsequent drying operation.

9.2.2 Dry the test specimen in the oven at 100 to 105°C (212 to 221°F) for 5 h, cool in the desiccator, then reweigh to the nearest mg. Add 25 mL nitric acid to the dried material in the evaporating dish, cover with a watch glass to prevent loss by spattering, heat on a steam bath until initial vigorous reaction ceases. Repeat addition of 25 mL nitric acid and heating on the steam bath until no further action occurs. Remove the watch glass and continue heating on the steam bath until all excess nitric acid has been removed. Heat the evaporating dish and contents on an electric hotplate, or under an infrared lamp, or in a cold furnace while raising the temperature slowly to prevent spattering and until all volatile matter is driven off and the contents of the dish are charred. Then ignite the evaporating dish and contents in the muffle furnace at 600 ± 25°C (1112 ± 45°F) to constant weight.

NOTE 2—The nitric acid treatment in Test Method B increases oxidation of organic material at relatively low temperatures while burning off only the residual carbon in the muffle furnace. Rubbery residues heated in a muffle furnace can produce a viscous foam which may expand and overflow or spatter due to gaseous decomposition while heating, causing loss of sample.

9.2.3 **Precaution:** All operations involving handling and use of corrosive acids are extremely dangerous. Special care should be taken when using nitric acid.

10. Calculation

10.1 *Test Method A*— Calculate the ash content, C_A, based on the total weight of the specimen as follows:

$$C_A, \% = W_1/W_2 \times 100$$

where:
W_1 = weight of ash, and
W_2 = weight of test specimen.

10.2 *Test Method B*:

10.2.1 Calculate non-volatile content, C_N, in the specimen as follows:

$$C_N, \% = w/W \times 100$$

where:
w = weight of specimen after oven heating, and
W = original weight of sample.

10.2.2 Calculate ash content of the non-volatile content, A_N, based on the weight of the non-volatile content in the sample as follows:

$$A_N, \% = W_1/w \times 100$$

where:
W_1 = weight of ash, and
w = weight of non-volatile content in sample.

NOTE 3—If required, the ash content of the total weight of the specimen shall be calculated as specified in 10.1.

11. Report

11.1 Report the following information:

11.1.1 Pertinent contractual requirements,

11.1.2 Whether the test was determined by Test Method A or B,

11.1.3 The percentage of ash and whether it is based on the original weight of the sample or the weight of non-volatile content,

11.1.4 The temperature and the length of time the specimen was in the muffle furnace, and

11.1.5 The weight percent of ash to the nearest 0.01 %.

12. Precision and Bias

12.1 Use the following criteria to judge the acceptability of results:

12.1.1 Duplicate values in this test method shall not differ by more than 0.01 %.

12.1.2 Duplicate determinations by two sets of laboratories should not differ by more than 0.03 %.

13. Keywords

13.1 adhesive; ash content