



Designation: D 4278 – 95

## Standard Test Method for Wet Ashing Procedure for Preparing Wood Samples for Inorganic Chemical Analysis<sup>1</sup>

This standard is issued under the fixed designation D 4278; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method consists of a procedure for decomposition of wood as an initial step for analysis for the constituents arsenic, chromium, copper, phosphate, and zinc, all of which may then be analyzed in accordance with Methods D 1035, D 1326, and D 1627, and Test Methods D 1628 and D 5584.

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.* Specific precautionary statements are given in Section 7.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 1035 Test Methods for Chemical Analysis of Fluor-Chrome-Arsenate-Phenol<sup>2</sup>
- D 1326 Methods for Chemical Analysis of Ammoniacal Copper Arsenate<sup>2</sup>
- D 1627 Methods for Chemical Analysis of Acid Copper Chromate<sup>2</sup>
- D 1628 Test Methods for Chemical Analysis of Chromated Copper Arsenate<sup>2</sup>
- D 5584 Test Methods for Chemical Analysis of Ammoniacal Copper Quat, Type B (ACQ-B)<sup>2</sup>

### 3. Summary of Test Method

3.1 A small (up to 5 g) sample of chopped or ground wood (usually treated) is oxidized and solubilized by an initial digestion in hot concentrated nitric acid. This is followed by further digestion after the addition of a solution of aqueous perchloric acid in concentrated sulfuric acid. The cooled solution of metal salts is then diluted and is suitable for analysis by the appropriate method.

### 4. Significance and Use

4.1 Many wood preservatives consist of organic materials. These materials are often preferably analyzed in dilute aqueous

solutions in the absence of organic matter. This test method provides a quantitative procedure to prepare a dilute aqueous solution of the inorganic materials which are in wood by simultaneously decomposing the wood sample and solubilizing the inorganic materials.

### 5. Apparatus

5.1 For each digestion to be run simultaneously, the following apparatus is required:

- 5.1.1 *Kjeldahl Flask*, 800 mL, with 24/40 ground glass top.
- 5.1.2 *Ground Glass Stopper*, 24/40.
- 5.1.3 *Kjeldahl Flask Heater*, rheostat-controlled.
- 5.1.4 *Tubing*,  $\frac{3}{8}$ -in. TFE-fluorocarbon.
- 5.1.5 *Glass Beads*.

5.2 For each group of digestions, the following apparatus is required:

- 5.2.1 *Tube Connectors*, Y-form, glass,  $\frac{3}{8}$ -in.
- 5.2.2 *Aspirator*, TFE-fluorocarbon.

5.3 The apparatus is assembled as shown schematically in Fig. 1. Up to six samples can be handled with a single aspirator. During digestion, clamps may be used on the tubing to balance the rate of aspiration between flasks. It may occasionally be necessary to enlarge the aspirator hole to about  $\frac{1}{16}$  in. to optimize aspiration, either because of extraneous plastic in a new aspirator, or because of build-up during use.

5.4 A syringe is also required for the introduction of acid during digestion. Care should be taken to keep the bulb free of acid oxidant and the syringe should be rinsed after use.

- 5.5 *Grinding Mill*.

### 6. Reagents

- 6.1 *Nitric Acid*, concentrated (sp gr 1.42).
- 6.2 *Sulfuric Acid*, concentrated (sp gr 1.84).
- 6.3 *Perchloric Acid*, 70 %.
- 6.4 *Acid Oxidant*—Add 185 volumes of perchloric acid (70 %) to 100 volumes of distilled water and then add *slowly with mixing* 270 volumes of concentrated sulfuric acid.

### 7. Safety Precautions

7.1 Although several thousand wood analyses have been carried out by this method without accident, the improper or careless use of perchloric acid has caused violent and dangerous explosions. Careful adherence to all directions is essential.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-7 on Wood and is the direct responsibility of Subcommittee D07.06 on Wood Preservatives.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.10.