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## Standard Test Method Practice 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-7 D07 on Wood and is the direct responsibility of Subcommittee D07.06 on Wood Preservatives. Current edition approved July 15, 1995; Sept. 10, 2002. Published September 1995; November 2002. Originally published as D 4278 – 83. Last previous edition D 4278 – 8895.

### 1. Scope

1.1 This test method practice 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 Test 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 1326 Methods for Chemical Analysis of Fluor-Chrome-Arsenate-Phenol Ammoniacal Copper Arsenate<sup>2</sup>
- ~~D 1326~~ Methods for Chemical Analysis of Ammoniacal Copper Arsenate<sup>2</sup>
- ~~D 1627~~ Methods 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 inorganic materials. These materials are often preferably analyzed in dilute aqueous solutions in the absence of organic matter. This test method practice provides a quantitative procedure to prepare means for the preparation of a dilute aqueous solution of the inorganic materials which are in wood by simultaneously decomposing the wood sample and solubilizing the inorganic materials. The aqueous solution may then be analyzed for its inorganic constituents by applicable ASTM test methods.

### 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, 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, 3/8-in.
- 5.2.2 Aspirator, TFE-fluorocarbon.

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

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. For the safe digestion of wood, two essential precautions are vital: (1) the sample should be mixed with nitric acid and further reagents should be withheld until the evolution of brown fumes has subsided; (2) perchloric acid should be diluted with sulfuric acid to form the acid-oxidant before it is added to the digestion mixture. Once the digestion has started, addition of wood, or contact with other organic matter, should be avoided. If such contact should inadvertently occur, flood the digestion flask with large amounts of cold water immediately.

7.2 The following general rules apply to the use of perchloric acid: Virtually all known explosions may be attributed to contact of raw organic matter or other easily oxidized material with concentrated perchloric acid, or to taking perchloric acid to dryness, forming the anhydrous acid. Organic matter should be pretreated with nitric acid, and perchloric acid should be used in conjunction with nitric and sulfuric acids. Do not let a digestion boil dry.

7.3 Special exhausting equipment, such as that illustrated in Fig. 1, must be used, if the digestion is to be carried out in ordinary hoods. Alternatively, special hoods, equipped with washing facilities, and constructed entirely of inorganic materials may be used. In the latter case, references to aspiration in Section 9 may be ignored.

7.4 Use of protective equipment (goggles, shields) should be mandatory. Avoid use of large amounts of acid. Explosions involving one or two grams of acid have caused serious damage and personal injury. Do not store more than one, 1-lb bottle in the laboratory. Keep this on a stone bench or a glass or ceramic tray; not in contact with wood or plastic. Do not increase the amounts in the procedure.

7.5 Clean up all spills with large volumes of water. Do not use sawdust, rags, or other organic material to mop up acid.

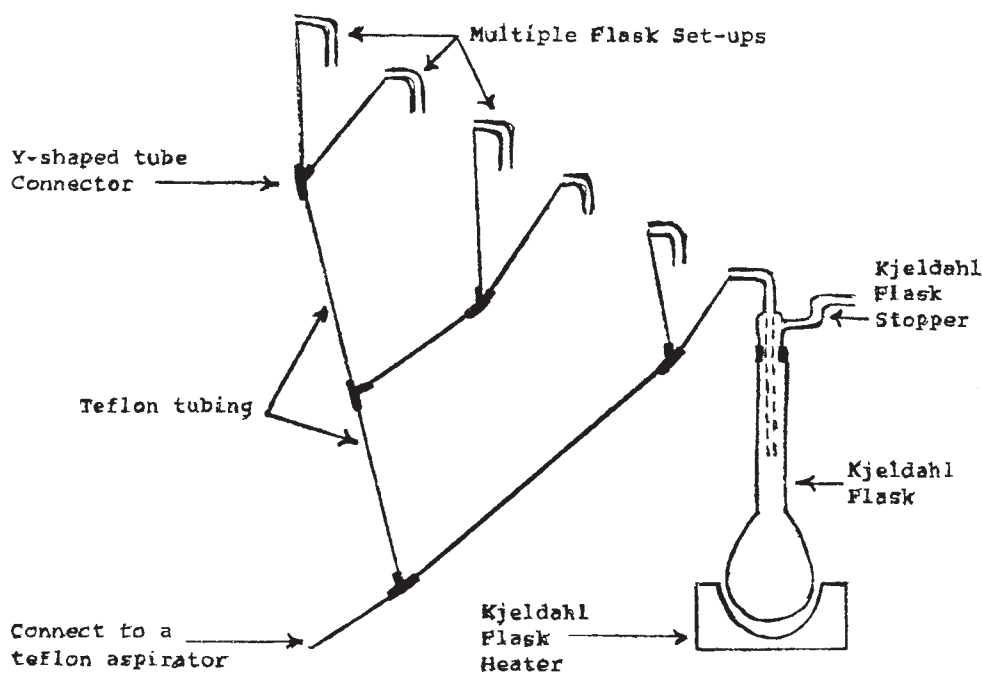


FIG. 1 Schematic Sketch of Setup of Digestion Apparatus

## 8. Preparation of Sample

8.1 Determine the density of the wood sample in pounds per cubic foot. A representative sample is then taken and ground to sawdust in a Wiley mill, or cut into small pieces. Increment borings may be used for determination of retentions, in which case the entire sample is used and the volume is determined for calculations rather than using a weight basis.

## 9. Procedure

9.1 For use with up to 5-g wood samples, as borings or ground wood. Dry the samples in an oven for approximately 2½ h or to constant weight at 125°C. Weigh samples accurately and place them in the 800-mL Kjeldahl flask with 3 to 5 glass beads. Add 30 mL of nitric acid. Digest slowly on low heat (approximately 150°C) with aspiration. The rate of aspiration should be adjusted so that the neck of the Kjeldahl flask is free from brown fumes.

9.2 In about 20 min, the evolution of brown fumes will cease and the wood will be completely dissolved. If this is not the case, add 10 mL additional nitric acid and digest further. Experience may dictate the use of more than 30 mL in the original digestion, but use of excess nitric acid should be avoided. Add 10 mL of acid oxidant, using a syringe, through the intake opening of the glass stopper.

9.3 In about 40 min, dense white fumes will be observed and the solution will be green in color. If the solution turns black, cool, add 10 mL of nitric acid and heat slowly until the solution turns green.

9.4 If chromium is not present, the digestion is now complete. If chromium is present, continue to heat until the solution becomes orange. Remove *immediately* from the heater. The digestion is now complete, and the solution may be analyzed for the various components after cooling to room temperature and following normal dilution procedures.

9.5 *Arsenic, Copper, Zinc, and Phosphate*—Dilute the digestion, using a volumetric flask and suitable aliquots if feasible. Do not use undiluted solutions for arsenic or copper determinations, or permit the solutions to boil to too low a volume during the arsenic procedure or the copper procedure. Explosions have occurred as a result. Follow the applicable procedure on the diluted sample.

9.6 *Chromium*—Care must be taken not to heat too strongly at any time. If this occurs, green insoluble chromic sulfates may form which cannot readily be redissolved. On the final digestion heating should be stopped as soon as the solution becomes a clear orange color. The solution should then be chilled immediately and diluted as above for analysis by standard procedure.

## 10. Precision and Bias

10.1 This section is not applicable to this test method.

## 11. Keywords

11.1 analysis; inorganic; preservative; wet-ashing

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