CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name: AWPA A7: Standard Wet Ashing Procedure for Preparing Wood for Chemical Analysis

CFR Section(s): 7 CFR 1728.202(g)(1)(v)(D)

Standards Body: American Wood Preservers Association

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

AMERICAN WOOD-PRESERVERS' ASSOCIATION
STANDARD

A7–75

STANDARD WET ASHING PROCEDURE FOR PREPARING WOOD FOR CHEMICAL ANALYSIS

Scope

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 according to the procedures given in AWPA Standards A2, A9, and A11.

Preparation of Sample

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.

Important Warning

Although several thousand wood analyses have been carried out by this procedure 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.

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 dry-

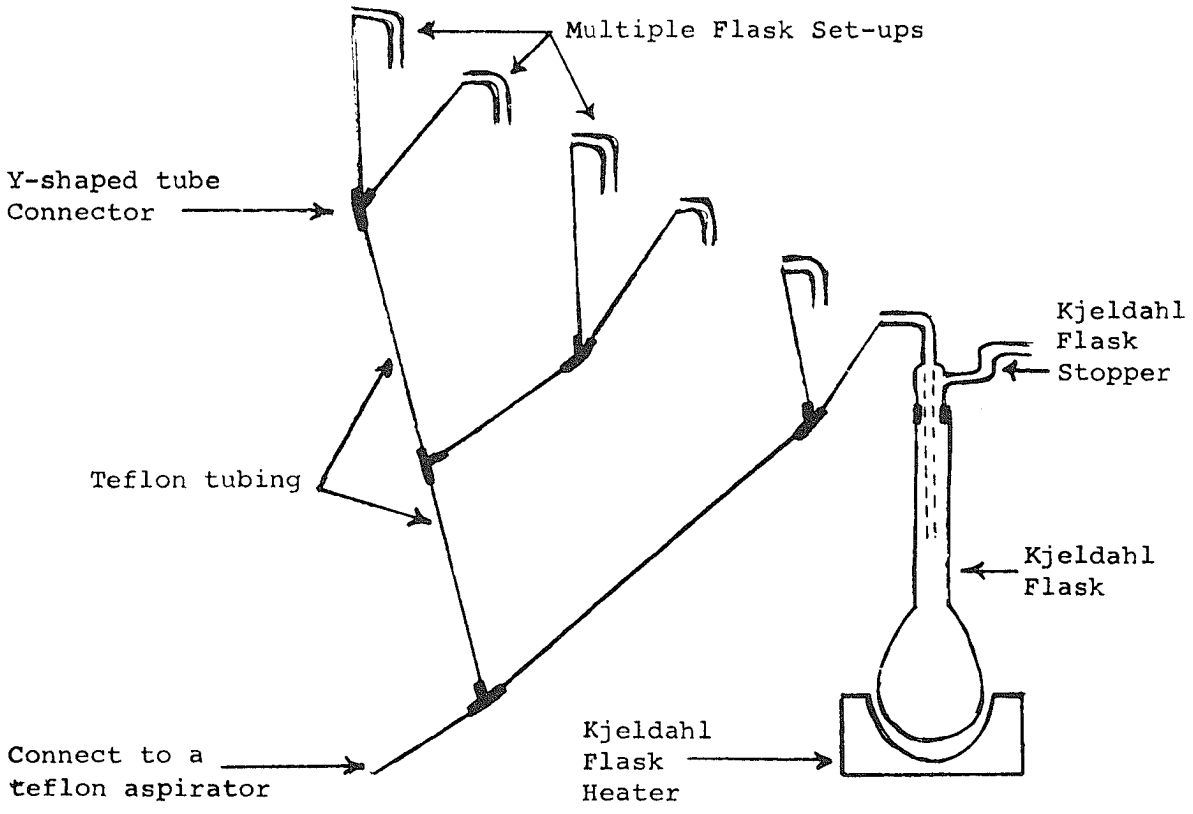

Figure 1.—Schematic Sketch of Set-up of Digestion Apparatus

ness, 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.

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 the *procedure* may be ignored.

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, one-pound 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.

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

Method One—Acid Digestion

Equipment

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

1. Kjeldahl flash, 800 ml, with 24/40 ground glass top. (SGA Scientific JF-6030 or equivalent)
2. Ground glass stopper, 24/40 (SGA Scientific JB-1290 or equivalent)
3. Kjeldahl flask heater, rheostat-controlled (Fischer Scientific 11–425 or equivalent)
4. Teflon tubing, 3/8″
5. Glass beads

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

6. Tube connectors, Y-form, glass, 3/8″
7. Aspirator, Teflon, (SGA Scientific Mallinckrodt plastic P-590-0038 or equivalent)

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 1/16″ to optimize aspiration, either because of extraneous plastic in a new aspirator, or because of build-up during use.

8. 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.

Reagents

Nitric acid, concentrated

Sulfuric acid, concentrated

Perchloric acid, 70 percent

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.

Procedure

For use with up to 5 gram wood samples, as borings or ground wood. Dry the samples in an oven for approximately 2½ hours 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.

In about 20 minutes, 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.

In about 40 minutes, 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. 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.

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 Step 4 of the arsenic procedure or Step 1 of the copper procedure in Standard A2. Explosions have occurred as a result. Follow the A2, A9 or A11 procedures on the diluted sample.

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.

Method Two—Peroxide Digestion[1]

Reagents

Add slowly two volumes hydrogen peroxide (30%) to one volume concentrated sulfur acid.

Analytical Procedure

Accurately measure or weigh wood sample into 250 or 500 ml. Erlenmeyer flask. For each gram of wood (5 maximum) add 15 ml. of reagent. Warm slowly on hot plate. Increase heat after initial reaction until solution clears. If solution starts to char, add 5 ml. of 30% hydrogen peroxide.

When solution no longer chars, and if wood was resinous or preservative contains chromium, cool, add 2 ml. concentrated nitric acid and 2 ml. of perchloric acid (70%). Continue heating to white fumes. Dilute solution in volumetric flask. Follow the A2, A9, or A11 procedures on the diluted sample. Note that the mixture now contains perchloric acid.

[1] Chromium results by this procedure may be low.