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ADAPTING THE BUBBLE-TIME METHOD FOR MEASURING VISCOSITY OF SLASH PINE OLEORESIN

Abstract.--A technique is described whereby the kinematic viscosity of slash pine oleoresin can be easily measured, in stokes, by an adaptation of the bubble-time method.

INTRODUCTION

Naval stores researchers have been studying viscosity of oleoresin in slash pine (*Pinus elliottii* Engelm. var. *elliottii*) since 1950. Schopmeyer et al.[1] established that viscosity of slash pine oleoresin affected the flow rate during the first 24 hours following wounding. Mergen et al.[2] found that viscosity was highest at the beginning of the growing season and then declined rapidly. They also found oleoresin viscosity characteristics in slash pine to be strongly hereditable, with both parents influencing oleoresin viscosity traits of their progeny.

Since 1967 a simple, accurate method of measuring viscosity of slash pine oleoresin has been used extensively at the Naval Stores and Timber Production Laboratory, Olustee, Florida. This method is directly related to Standard D-1545-63, adopted by the American Society for Testing and Materials in 1960 and revised in 1963.[3] It is only useful for transparent liquids because it depends on the visual observation of a rising bubble. Freshly collected, moisture-free slash pine oleoresin is a transparent liquid which lends itself to this technique.

SAMPLE COLLECTION

Equipment

For each sample to be collected and measured, the following items will be needed:

1. One special viscosity tube. A laboratory supplier can make accurate tubes using the dimensions in figure 1. This tube requires only

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[1] Schopmeyer, C. S., Mergen, F., and Evans, T. C. Applicability of Poiseuille's law to exudation of oleoresin from wounds on slash pine. Plant Physiol. 29: 82-87. 1954.

[2] Mergen, F., Hoekstra, P. E., and Echols, R. M. Genetic control of oleoresin yield and viscosity in slash pine. Forest Sci. 1: 19-30. 1955.

[3] American Society for Testing and Materials. ASTM Standard: D-1545-63. Pub. 66-68. 1916 Race St., Philadelphia, Pa. 1963.

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3 ml. of sample; the much larger standard tube is unsuitable for measuring such small, individually collected samples of pine oleoresin.

2. One spout. These can be easily made with aluminum tubing from specifications in figure 2.

3. One connector. These are made from 1/4-inch (ID) black rubber tubing cut into 1-inch lengths.

4. Two size 0 cork stoppers.

In addition to the above, you will need a small hammer, a carpenter's brace with 3/8-inch-diameter bit, and a plastic atomizer bottle, such as those in which nasal sprays are sold. The atomizer should be filled with a 50-percent solution of sulfuric acid. CAUTION: If a nasal spray atomizer is used, be sure to relabel it so it could not possibly be mistaken for its original contents.

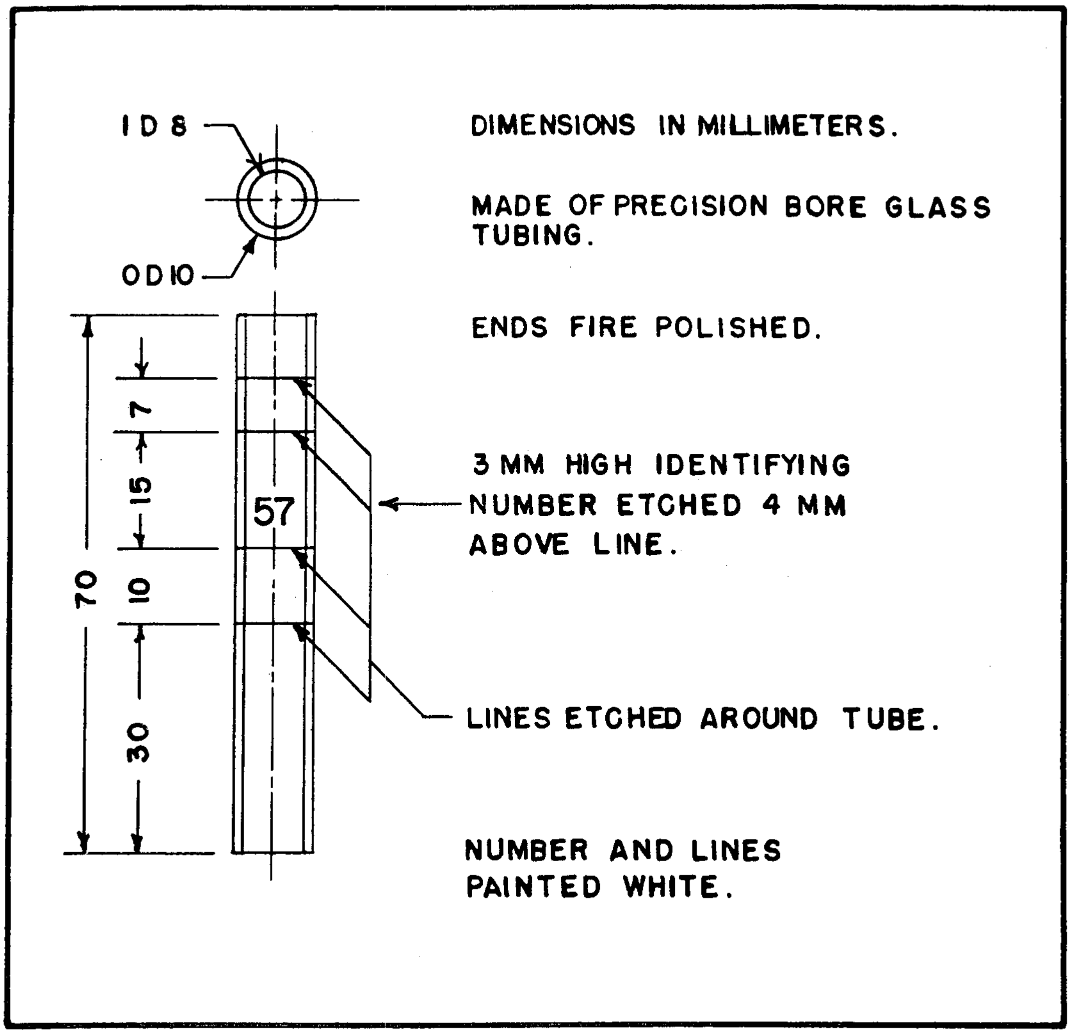

Figure 1.--Details of special viscosity tube.

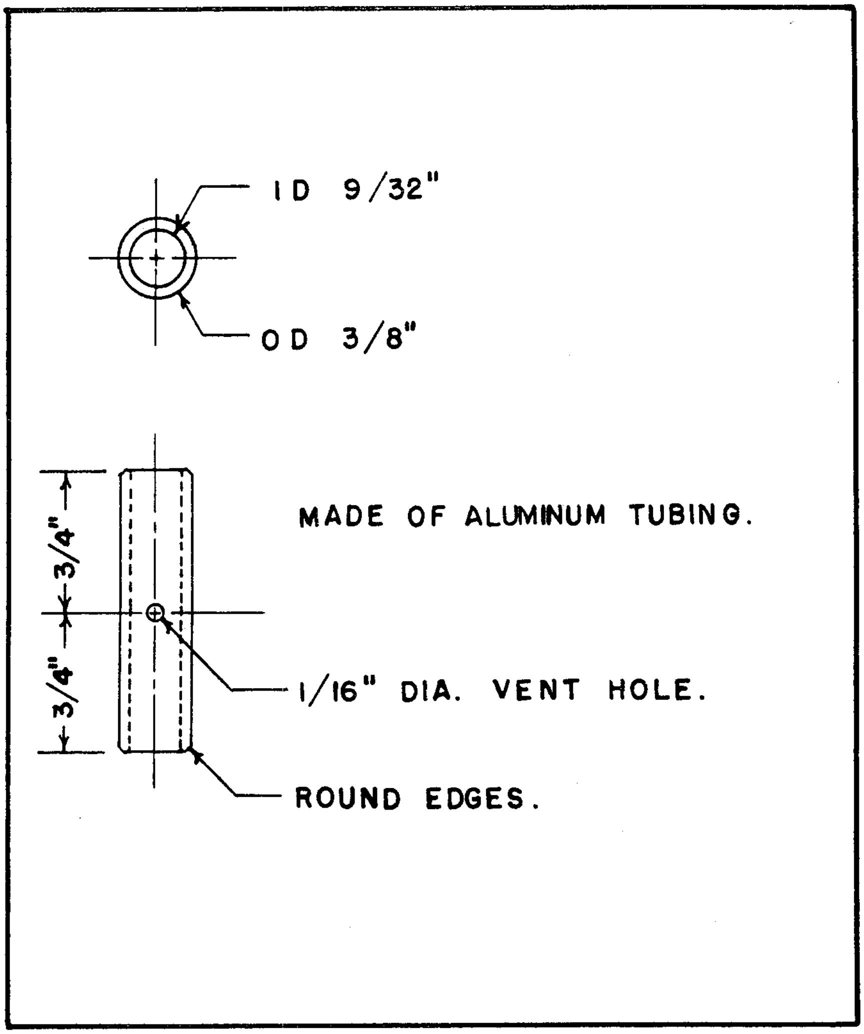

Figure 2.--Details of oleoresin collecting spout.

Procedure

Individual samples of oleoresin are obtained as follows: At a point you wish to sample, bore a 3/8-inch hole at an upward angle of about 45 degrees through the bark and 1/2 inch into the wood. Clear the hole of borings and spray sulfuric acid solution into it; one good squeeze is all that is needed. Oleoresin will collect only very slowly unless the acid stimulant is used.

After the acid has been applied, use a light hammer to tap an aluminum spout into the hole until snug, about 3/8 inch. Be sure the vent hole in the spout is upward to prevent loss of oleoresin. Stopper the bottom end of a viscosity tube. Fasten the top end to the spout in the tree with a rubber connector, as shown in figure 3. (On slash pine in northeast Florida, this special viscosity tube will fill in 2 to 25

Figure 3.--Viscosity tube attached to a slash pine tree.

hours, depending on season and temperature. There are times in winter when insufficient oleoresin can be collected for measurement of viscosity.) After the tube fills, remove it from the tree and stopper the open end.

VISCOSITY DETERMINATION

Equipment

The measurement of viscosity is made in a temperature-controlled waterbath. The following items are needed to set up such a bath:

1. A large glass water container. A 10-gallon aquarium serves well.

2. An immersion water heater.

3. A temperature control thermostat.

4. An electric stirrer.

Items 2, 3, and 4 are available from laboratory suppliers. The thermostat turns the heater on and off to maintain an exact temperature, and the stirrer circulates the water so that bath temperature is the same at all points. The thermostat has to be capable of maintaining a temperature of 25° C. ± 0.1° C. to meet the ASTM standard.

5. A thermometer. An accurate laboratory thermometer is needed to adjust the thermostat properly.

6. An inverting rack. Racks for holding viscosity tubes within 1° of vertical and inverting them 180° while immersed in a waterbath are available from companies supplying test equipment to the paint and varnish industry.

7. A stopwatch or clock calibrated to 0.1 second.

All this equipment assembled for use is shown in figure 4.

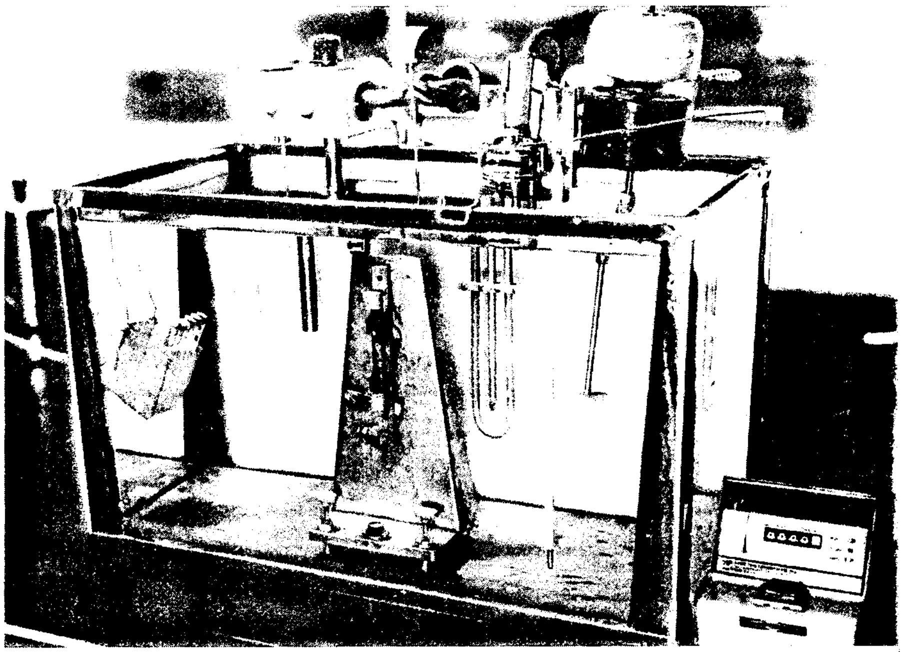

Figure 4.--Items in waterbath include (left to right): basket with tubes to be measured, thermostat, inverting rack, water heater, thermometer, stirrer; outside the tank, a timer.

Procedure

Set up the waterbath and allow time for its temperature to become stable at 25° C. Place all of the oleoresin filled tubes to be measured in the waterbath. After they have had time to reach 25° C., remove one tube at a time and perform the following:

1. Unstopper the top of the tube and remove enough oleoresin so the bottom of the meniscus is level with the second etched line down from the top. A glass stirring rod is useful for removing a little oleoresin, but be sure to clean it between tubes to prevent contamination of one sample with another.

2. Recork the tube so the bottom of the cork is even with the top line on the tube.

3. Return the tube to the waterbath.

Continue until the oleoresin level has been adjusted in all tubes. After the tubes have been in the waterbath another 20 minutes, begin reading viscosities:

4. Place a tube on the inverting rack in the waterbath, and invert the tube.

5. Start timer when top of bubble comes tangent to lower of the two middle lines. A magnifying glass helps.

6. Stop timer when top of bubble comes tangent to upper of the two middle lines.

Continue inverting tube and timing bubble until you obtain three bubble times agreeing within 1 percent of their mean. If you work carefully, the first three readings are usually within this range.

7. The final step is to multiply the average bubble time obtained by a factor which will give the bubble time that would have been obtained had a standard viscosity tube been used. This factor is about 2.58, but it is best to determine the factor for special tubes by comparing them to a standard tube using transparent liquids of viscosities similar to pine oleoresin, such as motor oils and motor oil additives.

Equipment can be cleaned of oleoresin with such solvents as turpentine, alcohol, or mineral spirits.

Accuracy and Units of Measurements

Accurate temperature control of the waterbath is essential. A variation of 0.1° C. in the temperature of the bath will cause a 1-percent variation in bubble time.

The tube must be vertical. A tube deviating from vertical by one-half its diameter (one radius) will give a 10-percent error in bubble time.

The bubble-time method gives viscosity in "bubble-seconds" or "approximate stokes," and for convenience, standard viscosity tubes are designed so bubble seconds are approximately equal to stokes, for most liquids. The absolute unit of viscosity is the poise. Poises are equal to stokes times density, but stokes are adequate for most practical comparative work.

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