T 532 cm-01

OFFICIAL METHOD – 1981
CLASSICAL METHOD – 1986
REVISED – 2001

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Starch consumption in corrugated board (enzymatic/colorimetric method)

1. Scope

1.1 The colorimetric adaptation of a simple laboratory test to measure the weight of starch present in the combining adhesive per unit area of corrugated board is described. It involves dissolving the starch in the corrugating adhesive with an enzymatic solution and colorimetrically determining the reducing dextrines and sugars produced by the enzymatic conversion. The method permits separate measurements of the adhesive at the double-face and single-face locations.

1.2 The basic enzymatic/gravimetric method, which yields statistically lower values, is described in TAPPI T 531 “Starch Consumption in Corrugated Board (Enzymatic/Gravimetric Method.”

2. Summary

The method is based on the hydrolysis of the starch by an alpha amylase enzyme to maltose and dextrines. The saccharides so produced are optically measured via the Nelson-Somogyi method for quantification of reducing sugars and then correlated to dry starch weight (weight per unit area) by a standard curve.

3. Significance

The test is a useful tool for: establishing the proper weight of adhesive per unit area for various board constructions; securing the optimum adhesive balance at each location; spot checking consumption to maintain continued economy with performance; establishing critical limits of machine operation; investigating the uniformity of adhesive application; facilitating the investigation of board complaints; and conducting development studies concerning the effect of operating conditions such as moisture in paper, waxing, machine settings, speeds, preheaters, showers, adhesive viscosity, and solids.

4. Apparatus

4.1 *Spectrophotometer,* visible region, 12.7 mm (0.5 in.) matched colorimeter tubes.
4.2 *Blood sugar tubes,* 25 mL.

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4.3 *Pipets*, automatic, micropipetter, piston type, 1-10 mL ± 1% accuracy.
4.4 *Volumetric flasks*, 100 and 1000 mL.
4.5 Mason Jars approximately 1 L, with rings and lids.
4.6 *Knife,* for separating single-face and double-face samples.
4.7 *Oven*, adjustable to ± 0.5°C (± 0.9° F), up to at least 150°C (302° F).
4.8 *Miscellaneous:* funnels; filter paper (No. 4 Whatman or equivalent); beakers, 50-mL and 600-mL; amber bottle; volumetric flask, 1000 mL.
4.9 *Analytical balance,* about 200 g capacity reading to 0.1 mg with precision of 0.2 mg.

5. Reagents and materials

5.1 *Amylase enzyme.* Pipet 60 mL enzyme solution having 631.8 DV units/mL into 1000-mL volumetric flask and fill to mark with distilled water to make 6% v/v solution.

NOTE 1: The test requires an enzyme to react with starch. The reaction is influenced by the dextrinizing value (DV) of the enzyme. DV is defined as the number of DV units per dry gram of enzyme solids. The amount of enzyme used will vary according to its DV. For example, 1 mL of Super Excise TX-24[1] weighs 1.139 g, and the enzyme is 21.3% solids; therefore, 1 mL contains 0.243 g dry enzyme. The DV of TX-24 is 2600; hence, 1 mL has 631.8 DV units (0.243 × 2600). If another enzyme is to be used, the concentration can be calculated by knowing its DV value and adding an amount equal to 631.8 DV units.

NOTE 2: Weigh all ingredients to the nearest 10 mg.

5.2 *Somogyi sugar reagent.*

NOTE 3: It is preferable to prepare a 10-L stock of the Somogyi reagent several weeks in advance of its use. This allows impurities and a slight amount of cuprous oxide to settle.

5.2.1 Dissolve 24 g of anhydrous sodium carbonate (Na_2CO_3) and 12 g of Rochelle salt ($KNaC_2H_4O_64H_2O$) in about 250 mL of cooled, boiled, distilled water (to deaerate the water).
5.2.2 To this solution add 4 g of hydrated copper sulfate ($CuSO_45H_2O$) dissolved in 40 mL of boiled, distilled water.
5.2.3 After mixing, add 16 g of sodium bicarbonate ($NaHCO_3$) and, when dissolved, pour the solution into a 1-L graduated cylinder.
5.2.4 Dissolve 180 g of anhydrous sodium sulfate (Na_2SO_4) in about 500 mL of hot water, and boil the solution to expel air.
5.2.5 After cooling, add this solution to the cylinder and dilute to the 1000-mL mark with cooled, boiled, distilled water.
5.3 *Arsenomolybdate reagent*
5.3.1 Dissolve 25 g ammonium molybdate [$(NH_4)_2MoO_4$] in 450 mL of water, add 21 mL concentrated H_2SO_4, and mix.
5.3.2 Dissolve 3 g of sodium acid arsenate ($Na_2HAsO_47H_2O$) in 25 mL of water. Weigh all ingredients to the nearest 10 mg.

CAUTION: Arsenate compounds are *poisonous* and suspected carcinogens. Avoid skin contact and/or ingestion. Before use, consult the warning label for precautions and antidotes.

5.3.3 Mix solutions from 5.3.1 and 5.3.2 together and incubate at 37°C (99° F) for 24 to 48 h. Store in an amber bottle.

[1] Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the bound set of TAPPI Test Methods, or may be available from the TAPPI Standards Department.