

T 504 cm-07

SUGGESTED METHOD – 1967
OFFICIAL STANDARD – 1974
OFFICIAL TEST METHOD – 1984
REVISED – 1989
CLASSICAL METHOD – 1997
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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.

Glue in paper (qualitative and quantitative determination)

1. Scope and significance

1.1 This method deals with the qualitative and quantitative determination of animal glue in paper in quantities that are used for sizing high grade papers (1). It is applicable to glue hardened with formaldehyde, alum, or amino-formaldehyde resins, and to photographic papers. This method can also be used in the determination of gelatin (a more highly refined form of animal protein).

1.2 The method is based on the color reaction of Ehrlich's reagent (*p*-dimethyl-aminobenzaldehyde) with hydroxyproline, one of the amino acids derived from collagen. It is specific for glue and gelatin; other nitrogenous materials do not interfere.

1.3 Two qualitative procedures are given, which are essentially the same: a macromethod where at least 100 mm² of paper is available and a micromethod where only 10 mm² can be used.

1.4 The scope of the quantitative procedure is given in 3.1.

NOTE 1: *Safety precautions* - This method requires the use of hazardous chemicals. Please refer to specific Material Safety Data Sheets for personal protective equipment, safe handling precautions, and disposal requirements.

2. Qualitative determination

2.1 *Apparatus*

2.1.1 *Boiling water bath*, a 600- or 100-mL beaker heated on an electric hot plate is satisfactory.

2.1.2 *Clamps*, to hold test tubes in the bath. Thermometer clamps attached to the rim of the beaker may be used for the micromethod.

2.1.3 *Glassware*, volumetric flasks, 100-, 500-, and 1000-mL. For macromethod: test tubes, 16 x 150 mm; pipets, two each, 5 and 1 mL with 0.1-mL graduations; one 0.2-mL with 0.01-mL graduations. For micromethod: test tubes, 10 x 75 mm; pipets, two 1-mL with 0.1-mL graduations; three 0.1-mL with 0.01-mL graduations.

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2.2 Reagents

2.2.1 *Sodium hydroxide.* For the macromethod, approximately 12.5N NaOH: dissolve 50 g NaOH in water and dilute to 100 mL. For the micromethod, 6N NaOH: dissolve 24 g NaOH in water and dilute to 100 mL.

2.2.2 *Copper sulfate,* approximately 0.01M CuSO₄: dissolve 1.25 g CuSO₄ • 5H₂O in water and dilute to 500 mL.

2.2.3 *Hydrogen peroxide,* approximately 4% H₂O₂: dilute 13 mL of 30% H₂O₂ with water to 100 mL.

2.2.4 *Sulfuric acid,* approximately 3N H₂SO₄. Add 83 mL concentrated H₂SO₄, slowly and with stirring, to about 800 mL water, cool, and dilute to 1000 mL.

2.2.5 *1-Propanol* (reagent, b. p. 96-98°C).

2.2.6 *p-Dimethylaminobenzaldehyde* reagent, purified grade. Obtain in 10- or 25-g bottles and refrigerate after opening. The crystals should be white and the solution practically colorless.

2.2.7 *Ehrlich's reagent:* dissolve 1 g of *p*-dimethylaminobenzaldehyde in 20 mL of 1-propanol. The solution may be used as long as it is colorless or yellowish but should be discarded if it develops a green, brown, or red color.

2.3 Test specimens

2.3.1 *Macromethod:* From each test unit obtained in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product," take a specimen having a total area of about 100 mm² of the paper and cut into small pieces.

2.3.2 *Micromethod:* Take a specimen approximately 3 mm square. If the paper is thicker than 0.125 mm (5 mils), peel off and use only the surface layer or layers.

2.4 Procedure

2.4.1 Macromethod

2.4.1.1 Carefully pipet 0.20 mL of 12.5N NaOH into the bottom of a 16 x 150-mm dry test tube, touching the bottom of the tube with the tip of the pipet to remove the last drop. Hold the tube in a vertical position and add the test specimen. Place the test tube in the boiling water bath for at least 10 min. Remove from the bath, cool for a few seconds with tap water, add 1 mL of 0.01M CuSO₄, mix and add 0.5 mL of 4% H₂O₂, without allowing it to contact the upper part of the tube. Shake occasionally until most of the foaming stops and return the tube to the boiling water for 5 min. The peroxide must be completely decomposed before the H₂SO₄ is added.

2.4.1.2 Again cool with tap water for a few seconds, add 2.5 mL of 3N H₂SO₄ and 1.5-2.0 mL of Ehrlich's reagent and mix well. Cool the water bath to 80-90°C by adding cool water and return the test tube to the bath. If a rose-red or pink color develops within about 10 min, the presence of glue is indicated.

2.4.2 *Micromethod:* Carefully pipet 0.015 mL of 6N NaOH onto the bottom of a 10 x 75-mm dry test tube, touching the bottom of the tube as before to remove the last drop. Hold the tube in a vertical position, add the test specimen, and proceed as above, except use 0.05 mL of CuSO₄, 0.025 mL of H₂O₂, 0.125 mL of H₂SO₄ and 0.1 mL of Ehrlich's reagent.

2.5 *Report.* Report the presence or absence of glue as indicated above.

3. Quantitative determination

3.1 Scope and summary

3.1.1 This procedure (2) determines glue in the presence of melamine resin. It may be used in the presence of other additives such as glycerin, alum, and rosin. The recovery of glue from papers made from rag pulps is almost complete. Recovery from wood pulp papers and from papers containing groundwood is not complete, but the data are possibly sufficiently accurate for most purposes.

3.1.2 The glue is extracted from the paper by refluxing with 0.1N HCl. An aliquot of the extract is hydrolyzed with 6N HCl at 110°C. The hydroxyproline in the hydrolyzate is reacted with Ehrlich's reagent to give a colored compound with an absorbance maximum of 560 nm. The absorbance is a measure of the amount of glue on the paper.

3.1.3 The procedure is a specific test for glue. It is not intended to replace TAPPI T 418 "Organic Nitrogen in Paper" when interfering materials are absent.

3.2 Apparatus

3.2.1 *Spectrophotometer,* for making absorbance measurements of 560 nm, and equipped with quartz or glass absorption cells. A filter colorimeter may be used, but it is not as satisfactory as a spectrophotometer.

3.2.2 *Reflux assembly,* all glass, consisting of a 250-mL Erlenmeyer flask and water-cooled condenser with standard taper connections.