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T 692

BALLOT NO. 05 SARG

DRAFT NO. 04

DATE October 26, 2023

WORKING GROUP CHAIR Charles Courchene

SUBJECT CATEGORY Alkaline Pulping

RELATED METHODS See "Additional Information"

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 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.

Determination of suspended solids in kraft green and white liquors

(Five-year review of Official Method T 692 om-13)

(Underscores, notes, and strikethroughs show changes from Draft 3)

1. Scope

This method provides a means of determining the level of suspended solids in kraft green liquor and kraft white liquor.

2. Significance

A procedure for determining suspended solids is needed to establish the efficiency of filtering equipment or of chemical additives used to reduce the level of suspended solids in these liquors.

3. Summary

The method given is based upon a filtration through glass fiber filter discs, with hot water wash to remove the dissolved solids. It is similar to the method used by the U.S. Public Health Service, called the "Gooch Crucible-Asbestos Fiber Method." The use of the glass fiber filter disc is recommended since the caliper of the glass fiber media is uniform, and insures reproducible results.

4. Apparatus

4.1 *Filter discs*[1], glass fiber, pore diameter less than 2.5 micron, 47 mm overall outer diameter. (If other diameter filter disc is used, report diameter used).

4.2 *Gooch crucibles*[1] (or alternative - see note 2).

4.3 *Gooch crucible filter adapter*[1] (or alternative – see note 2).

4.4 *Filter flask*, 1-L.

4.5 *Vacuum* (aspirator satisfactory).

4.6 *Distilled water*.

4.7 *Drying oven* (105°C ± 3°C).

4.8 *Analytical balance*.

4.9 *Desiccator*.

NOTE 1: The pore size in the glass fiber discs specified in this method is less than 2.5 micron. Other inert filtering media with a comparable pore size may be employed if glass fiber discs are not available. Filter paper should yield comparable results. However, no data are available comparing other media with the glass fiber disc. Whatever medium is employed, it still remains important to filter the sample at the temperature specified, and to rinse with hot distilled water to remove the dissolved solids prior to weighing.

NOTE 2: Membrane or Buchner filter apparatus are acceptable alternative equipment selections, providing that the pore size of the filter material is less than 2.5 microns. Report type of equipment used (if other than Gooch) and the diameter of filter disc used.

5. Procedure

5.1 Collect a representative sample of the liquor to be tested. A minimum of 2 L is required. Record temperature. If possible, maintain at that temperature for testing by using an insulated type container. If not possible, reheat the liquor to its original temperature prior to testing (see Note 3).

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

NOTE 3: Temperature of sample is critical. If the temperature of the sample, or sample aliquot, is allowed to drop before testing, or during the filtration step, the sample should be reheated to the original sampling temperature and stirred for a period of time sufficient to re-dissolve any salts (crystals), which may have precipitated as the sample cooled. The sample should be kept hot, using a water or steam bath if necessary, during the filtration step.

5.2 Insert a glass fiber filter pad, felt side up, into a Gooch crucible. The felt side is easily recognized by its smoother surface as compared with the wire side (which is characterized by a wire mark). Place filter unit in adapter in the filtering flask and turn on medium vacuum. Use about 50 mL of distilled water and seat the filter.

5.3 Place the prepared filter unit in an oven at 105°C and dry to constant weight (about 60 min). Cool filter unit in a desiccator and obtain tare weight. Weigh to nearest 0.1 mg.

5.4 Place the filter unit in the filter adapter in the filtering flask. Wet filter with 10-15 mL distilled water; pull down slightly with vacuum to seat filter in bottom of crucible. Hold small amount of vacuum to keep filter in place.

5.5 Stir sample vigorously, and measure out desired volume into a graduated cylinder. Usual sample size is 200 mL, and duplicate determinations should be made. When liquor suspended solids are low, i.e. after a pressure filter, the sample size should be increased.

5.6 Pour sample into filter unit and adjust vacuum to optimum conditions for each sample. The vacuum should be held to a minimum. Excessive vacuum will pull the suspended solids into the pores of the filter media and prolong the filtering time. If the suspended solids are high and the filtration rate is slow, repeat the test transferring only 30-50 mL aliquots of the sample into the filter unit at any one time. Keep the rest of the sample hot using a hot plate, water bath or steam bath (see Note 3).

CAUTION: Excessive vacuum may also cause flashing of the hot filtrate & unseat the filter disc, thus voiding the test result.

5.7 Rinse the graduated cylinder used in 5.5 onto the filter media with 100-200 mL of hot distilled water. After sample is entirely filtered, rinse the filter with 100-200 mL of hot distilled water to remove dissolved or precipitated solids. Repeat for a total of three or more rinses. Phenolphthalein can be used to check the alkalinity of the filtrate after the third wash to determine if more rinses are needed. The phenolphthalein should not be put on the filtered solids or filter paper. To check the filtrate, you must empty the filtration flask after the filtration step and first two washes, prior to the last wash, or use a second filter flask to capture the filtrate from the third wash. More washes are needed if the phenolphthalein turns pink or mauve when added to the filtrate from the third wash.

5.8 Dry filter unit in oven at 105°C to constant weight (2 h is usually sufficient). Cool in a desiccator and weigh.

5.9 The difference in the two weighings is calculated as suspended solids in milligrams per liter:

$$\text{Suspended solids, mg/L} = (W_c - W_T)1000/V$$

where

W_c = weight of crucible + solids, g
W_T = tare weight, g
V = sample volume, L

Examples:

Sample size: 1.0 L of white liquor
Temperature: 82°C

Weight of crucible plus solids:	76.9083 g
Tare weight:	76.8005 g
	0.1078 g

Report: 108 mg of suspended solids/L

6. Report

6.1 Report the difference in the two weighings as suspended solids in milligrams per liter. Test results should be based upon the average of duplicate determinations.

6.2 Report the temperature of the liquor sample at time of testing.

7. Precision

Background information:

The precision statement below is based upon an experiment involving 2 laboratories on a single green liquor and a single white liquor sample in 2000. Each sample was analyzed in duplicate in each laboratory. A complete precision trial may not be practically conducted due to the hazardous nature and potential decomposition of the samples. As this method is primarily applicable to process control efficiency at a single location, it is recommended that users rely primarily on internally developed method performance metrics.

Repeatability (within a laboratory) 2% for the white liquor sample.
Reproducibility (between laboratories) 2% for the white liquor sample.

Repeatability (within a laboratory) 5% for the green liquor sample.
Reproducibility (between laboratories) 9% for the green liquor sample.

8. Caution

Kraft liquors are hazardous. Safety precautions should be followed when obtaining samples and when handling these liquors. As a minimum precaution when obtaining samples of these liquors, face and eye protection should be required and rubber gloves worn.

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Deleted: Both a green and white liquor sample were analyzed in duplicate according to this procedure at two laboratories. ¶
Repeatability (within a laboratory) was 2 to 5%.¶
Reproducibility (between laboratories) was 2% for the white liquor sample and 9.4% for the green liquor sample. ¶
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9. Keywords

Kraft liquors, Green liquors, White liquors, Suspended solids, Solids content

10. Additional information

10.1 Effective date of issue: To be assigned.

10.2 The filter pore size range was determined through research performed at Paprican on lime mud particulate size. See Figure 1. Based on this research, a filter disc with less than a 2.5 micron pore diameter is considered acceptable for particulate retention.

10.3 In response to the comments received in this 2008 revision, in section 4.1, the word “outer” was added before “diameter” to avoid confusion on the size of the filter discs. In section 5.7, a sentence was added to clarify when more washing was necessary.

10.4 The precision statement was revised to include information about the original laboratory testing that was done to establish repeatability and reproducibility.

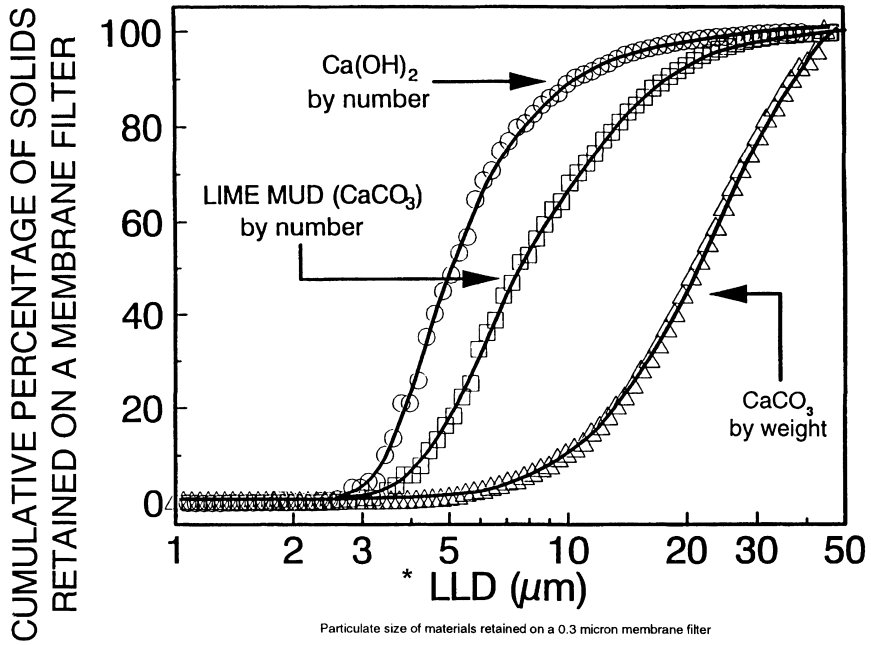

Fig. 1. Cumulative distributions by number for hydrated lime [$Ca(OH)_2$] and by number and weight for $CaCO_3$.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■