Solids content of black liquor

1. Scope

1.1 This method is designed to measure gravimetrically the solids content of weak and strong black liquors as they exist, or will exist, at the point of injection into the recovery furnace (1-3).
1.2 This method can be used to calibrate rapid or routine control procedures.
1.3 The method will measure the “solids” remaining after removal of water and other nonaqueous volatile materials normally lost in commercial evaporation systems.
1.4 The sampling procedure is compatible with additional black liquor analytical procedures such as chemical analyses, heating value, etc.

2. Summary

2.1 Black liquor specimens are dried at 105°C for a minimum of 6 h with inert surface extender and a controlled flow of dried air to increase drying rate and eliminate moisture entrapment. Strong black liquors are diluted to allow volumetric handling and to reduce scum formation.

3. Apparatus and materials

3.1 Glass weighing bottle, ground-glass stoppered, about 50 mm internal diameter x 30 mm high.
3.2 Oven, forced air, controlled at 105 ± 3°C and adjusted so that the air will be replaced about once or twice per minute.
3.3 Sand, high silica (~ 98% +) approximately 60 x 100 mesh, that has been previously heated at 850°C.

NOTE 1: Diatomaceous earth and alundum have been found to react with residual carbonate and caustic in black liquors leading to significantly lower solids results. High silica sand is the only material found to be inert (4).

3.4 Glassware, volumetric flask, 500 mL; pipet or medicine dropper.
4. Sampling and test specimens

4.1 Weak liquor will usually contain fibers. Eliminate these by filtering through coarse filter paper or by inserting the pipet below the fibers, which tend to float to the surface.

4.2 Concentrated liquors should be diluted with distilled water to 20-30% concentration so that they can be handled as a fluid, be made homogenous, and provide no chance of flashing when drying. This should be done by estimating the concentration of the sample and calculating the amount of liquor and water needed to give 20-30% concentration. A 500-mL volume of diluted sample is desirable if subsequent chemical analyses are required.

4.3 Procedure for diluting a liquor that is hot: Weigh an empty 500-mL or 1-L plastic bottle with cap on a top-loading balance to the nearest 0.05 g. Add an appropriate amount of distilled water and reweigh. Add the hot liquid black liquor to the bottle and cap immediately. Gently swirl the bottle to mix. When well-mixed, cool and weigh. Calculate the dilution ratio.

4.4 Procedure for diluting a cold liquor: Liquor to be sampled must be in the liquid state to ensure homogeneity. Place the container with the concentrated sample in a hot water bath to melt it. Stir until the sample is liquid and homogenous. Sample and dilute as in 4.3 above.

5. Procedure

5.1 Place 25 to 30 g of sand in the weighing bottle and heat the bottle and cap in the oven at 105°C to constant weight. Cool in a desiccator and weigh to the nearest 0.5 mg. By means of a pipet or medicine dropper, transfer enough specimen to the weighing bottle to ensure 1 to 3 g of dry solids. (For weak liquors under 30%, a 5- to 10-g specimen is required. For concentrated liquors, dilute as directed in 4.2 - 4.4.) Cap the bottle and weigh to the nearest 0.5 mg.

5.2 Place the weighing bottle and cap in the oven (cap off) and heat for a minimum of 6 h. Remove, cool in desiccator, and weigh (cap on). Repeat this heating procedure at 1-h intervals until the weight loss is less than 0.1% per hour (approximately 1 to 3 mg loss per hour). (If samples are left overnight in the oven, the time to constant weight will be minimized.)

5.3 Make triplicate determinations, weighing and diluting in separate containers (4.3).

6. Calculation

6.1 Undiluted liquors

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\% \text{ Solids} = \frac{\text{Weight of dried solids}}{\text{Weight of specimen}} \times 100
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6.2 Diluted liquors

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\text{Dilution ratio} = \frac{\text{Weight of diluted sample}}{\text{Original sample weight}}
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\frac{\text{Weight of } H_2O + \text{sample weight}}{\text{Original sample weight}}
\]

\[
\% \text{ Solids (diluted liquor)} = \frac{\text{Weight of dried solids}}{\text{Weight of specimen}} \times 100
\]
% Solids (original sample) =
% solids of diluted liquor x dilution ratio

7. Report

Report the average percentage of solids, based on the original weight of the sample, to the nearest 0.1%.

8. Precision

The precision of this method has not been determined fully in accordance with T 1206 “Precision Statement for Test Methods.” However, repeatability has been determined by a laboratory using the above procedure. All samples were run in triplicate. The average percent relative standard deviation is 0.49% on a series of 12 different samples (range of 25-65%) including dilution and which were sampled hot. The percent relative standard deviation is 0.46% for repeated analysis of the same sample when sampled hot. For a 65% liquor this would be a repeatability of 0.83%. The average percent relative standard deviation on a series of samples which were remelted for sampling was 1.75%.

9. Additional information

   9.1   Effective date of issue: December 1, 1989
   9.2   This method was originally part of T 625 “Analysis of Soda and Sulfate Black Liquor.”
   9.3   The 1979 version of this method (T 650 pm-79), which specified use of diatomaceous earth as the extender, is in error and should not be used.

References


Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Technical Divisions Administrator.