WI 260802.01

T 650

BALLOT NO. 02 - SARG

DRAFT NO. 01

DATE June 9, 2026

WORKING GROUP CHAIR N/A

SUBJECT CATEGORY Chemical Properties

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.

Solids Content of Black Liquor

(Five-year review of Official Method T 650 om-21)

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. Significance

This method will measure correctly the gravimetric "solids" of weak and strong black liquors to be used as fuel in recovery furnaces. The "solids" are what remains after the normal evaporation process. This method is the reference method and can be used to calibrate rapid procedures or sensors.

4. Apparatus and materials

4.1 *Glass weighing bottle,* low-form, ground-glass stoppered, about 70 mm internal diameter × 33 mm high with an approximate capacity of 82 mL.

4.2 *Oven*, forced air, controlled at 105 ± 3°C and adjusted so that the air will be replaced about once or twice per minute.

4.3 *Sand*, high silica (~ 98% +) approximately 20−30 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).

4.4 *Plastic ware*, volumetric flask, 500 mL bottle with cap; pipet or liquid dropper.

4.5 *Balance*, capable of weighing to 0.001 g or better.

5. Sampling and test specimens

5.1 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.

5.2 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.

5.3 Procedure for diluting a hot liquor: Weigh an empty 500-mL or 1-L plastic bottle with cap to the nearest 0.001 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.

5.4 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 5.3 above.

6. Procedure

6.1 Place 45 to 50 g of sand in the weighing bottle and heat the bottle and stopper in the oven (stopper off) at 105°C to constant weight. Cool in a desiccator and weigh to the nearest 0.001 g. By means of a pipet or 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 5.2 - 5.4.) Cap the bottle and weigh to the nearest 0.001 g .

6.2 Place the weighing bottle and stopper in the oven (stopper off) and heat for a minimum of 6 h. Remove, cool in desiccator, and weigh (stopper on). Repeat this heating procedure at 1-h intervals until the weight loss is less than 0.1% per hour (approximately 0.001 to 0.003 g loss per hour). If samples are left overnight in the oven, the time to constant weight will be minimized.

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

7. Calculation

All weights are in grams.

7.1 Undiluted liquors

$$\% \, Solids = \frac{Weight \, of \, \, dried \, solids}{Weight \, of \, \, specimens} \times 100$$

7.2 Diluted liquors

$$Dilution \, ratio = \frac{Weight \, of \, \, diluted \, sample}{Original \, sample \, weight} = \frac{Weight \, of \, \, H_2O + Sample \, weight}{Original \, sample \, weight}$$

$$\% \, Solids \, (diluted \, liquor) = \frac{Weight \, of \, \, dried \, solids}{Weight \, of \, \, specimen} \times 100$$

% Solids (original sample) = % solids of diluted liquor × dilution ratio

8. Report

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

9. Precision

The following estimates of repeatability and reproducibility are based on data from an interlaboratory trial involving 5 laboratories and one sample of heavy black liquor. The trial was conducted in February 1998 using the T 650 om-89 revision of this method. Testing is based on 3 determinations per test result and one result per lab, per material.

% Solids

Material description	*Grand mean*	*Range*	*Repeatability r and r%*		*Reproducibility R and %R*		*Labs included*
Heavy Black Liquor	49.11	48.39 - 49.37	0.53	1.1%	1.22	2.5%	4/5

All data in % solids

Repeatability and reproducibility are estimates of the maximum difference (at 95%) which should be expected when comparing test results for materials similar to those described above under similar test conditions. These estimates may not be valid for different materials or testing conditions.

9.2 The above are in accordance with the definitions of these terms in TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility.”

10. Keywords

Solids content, Black liquors

11. Additional information

11.1 Effective date of issue: To Be Assigned

11.2 This method was originally part of T 625 “Analysis of Soda and Sulfate Black Liquor.”

11.3 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.

11.4 In 1999 a Significance section was added and a new Precision statement was done.

11.5 Changes in the 2015 version include adjusting the mesh size of the sand in 4.3 and adjusting the amounts to be used in 6.1. The original sand was no longer commercially available. The following study was run at one lab doing duplicate measurements on three liquor samples to determine the effect of the sand mesh size.

	20-30 mesh sand	*60-100 mesh sand*
Sample	*% solids*	*% solids*
1	12.51	12.55
2	18.29	18.25
3	21.72	21.59

References

1. Parker, J. L., Hensel, R. P., and Wagoner, C. L., "Measurement of Total Solids in Kraft Black Liquors," *Tappi* **53** (5): 874 (1970).
2. Phillips, J. H., "Determination of Total Nonaqueous Constituents in Pulp Mill Liquors," *Tappi* **34** (10): 101A (1951).
3. Phillips, J. H., and Rubright, M. M., "The Determination of Total Nonaqueous Constituents in Pulp Mill Liquors, Part II, Application to All Types of Residual Pulping Liquors," *Tappi* **36** (9): 392 (1953).
4. McDonald, K. L., *Tappi* **62**(1): 80 (1979). Also private communication: Grace, T. G.; Nelson, W.; and Tostevin, J. E.

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

■