CHAPTER 6 OFF-LINE BLACK LIQUOR SOLIDS MEASUREMENT (LABORATORY AND FIELD TECHNIQUES)

6.1 Purpose

The procedures and interlocks described in this recommended good practice take action to remove black liquor from the furnace or prevent its introduction if the liquor solids fall below a predetermined, absolute value (58%). The reference method for determining absolute values for black liquor solids is the TAPPI Standard Method, T650-om-05.

Actions taken to prevent low solids black liquor from entering the recovery furnace are based on readings from continuous recording black liquor solids monitors (normally referred to as refractometers). These continuous monitors measure a property of the black liquor (e.g. refractive index) that is proportional to the dissolved black liquor solids content. In order to convert the continuous solids monitor reading to an absolute basis, it is necessary to calibrate the continuous solids monitor with respect to T650- om-99. T650-om-05 is a laboratory method. It is time consuming and requires skilled laboratory techniques. The recommended practice is to verify the continuous solids monitor reading through periodic, off-line field tests. The field test measurement technique, in turn, should be checked against T650-om-05 at least once per week.

This chapter discusses off-line black liquor solids measurement and makes recommendations as to how this should be carried out. Correct procedures for both collection of samples and sampling techniques are essential for good results. Both of these are discussed in this chapter.

6.2 Sampling

It is essential that the black liquor sample is representative of the black liquor that is being fired into the furnace and that is being measured by the continuous solids monitor. The sampling location should be on the line to the furnace header or on the header itself. There should be no sources of dilution or chemical addition downstream of the sampling point. Use of liquor samples from other locations (such as the cascade flow box), chosen for convenience, can give erroneous results.

The black liquor sample will be under some pressure and could be at a temperature above the boiling point. Precautions need to be taken to deal with the possibility that the liquor may flash. Flashing could result in spattering of hot black liquor and will cause some evaporation of water (and an erroneously high solids content). If flashing is excessive, it may be necessary to cool the sample or use a pre-dilution technique that will be described later. In all cases, the sample line must be adequately flushed before taking a sample for analysis.

Field solids measurements should be made as soon as possible after the sample is taken. Firing strength black liquor normally contains suspended solids that are insoluble at that concentration. These particles can settle rather quickly in hot black liquor and are difficult to re-suspend uniformly once they have settled out. The sample of black liquor should be stirred just prior to removal of a small representative portion for testing. The black liquor sample should not be allowed to cool below 140°F (60°C) before the test portion is taken since suspended saltcake particles can absorb moisture from the liquor on cooling and form large hydrate crystals that are difficult to disperse. In low-odor units without direct contact evaporators, the black liquor sample should be kept closed. This will minimize contact with air which could oxidize some of the sulfide in the liquor and give an erroneously high solids value. One convenient sampling container is a pint-size, screw-capped, wide-mouth, vacuum-jacketed bottle.

Special precautions need to be taken for the laboratory T650-om-05 sample. The relatively long time involved before the measurement is made will lead to extensive settling of un-dissolved solids. It is recommended that the pre-dilution technique be used in taking this sample. After flushing the sample line, about 200 ml of heavy black liquor is drawn into a tared (weighed) sample bottle of at least 400 ml volume. A second weighing establishes the liquor sample weight. The sample can then be diluted with a known amount of water to a working concentration in the 30-40% solids range where all liquor constituents are soluble. The solids concentration in the original undiluted liquor can be calculated from that measured in the diluted sample using the formula given in T650-om-05.

6.3 Off-Line Field Measurement Techniques

There are a number of solids measurement techniques that are reasonably straightforward and suitable for field use. A partial listing includes:

moisture balances,	-Acceptable
microwave oven drying,	-Acceptable
density (Baumé) readings	**not recommended**
hand held refractometers	**not recommended**

All of these methods are procedure dependent. The accuracy and repeatability of results depends strongly on small (but important) details of the equipment and procedures used. Frequently, the absolute value of the solids obtained will vary with the operator running the test regardless of the analytical equipment being used. It is important to have written black liquor solids test procedures that are accurate and are consistently followed to produce repeatable operator solids test results. For best results, post the test procedure at the operator test station and perform periodic audits of the test procedure as compared to the laboratory T650-om-05 test.

Moisture balances are probably the most common method for field solids testing. Moisture balance results can give variable results based on the age or brand of infrared bulb used, the height of the heating element above the sample, the amount of sample used, and the distribution of the sample on the sample pan. In recent years, automated moisture balances have become available and help to take some of the variation out of field solids measurements.

Experience has shown that microwave oven drying can give variable results with black liquor. Overheating of samples can result in pyrolysis of liquor organics and erroneously low values for the measured solids. Microwave fields in most ovens are not uniform and the heat absorbed by the sample may depend on its exact position and orientation in the oven.

Specific gravity (Baumé) readings are not recommended for making calibration adjustments to continuous solids monitors. This technique measures the specific gravity on a pre-diluted sample of firing strength black liquor and uses a conversion chart to determine the percent solids. This is not a direct measurement of solids content. The Baumé vs. solids curve is dependent on liquor composition. Experience has shown that this method lacks the accuracy of other methods when applied to a wide variety of black liquors and liquors subject to changes in makeup rate, recycled dust load, and chemical addition rates.

Hand-held refractometers are also not recommended for making calibration adjustments to continuous solids monitors, especially if the continuous device is also a refractometer. The field test should provide a cross-check on the continuous device and should be a direct measurement of the solids content, not a measure of a property that depends on solids content. Refractive index depends on liquor composition as well as solids content and does not respond to the suspended solids that would be present in firing strength black liquor. For these reasons, confirmation by direct solids measurement is needed.

Moisture balance techniques and microwave testing are both reliable methods for field solids measurement but it is necessary to recognize that the values obtained can be dependent on procedural details. These details need to be standardized as much as possible. It is recommended that a standard, detailed solids testing method be written up for each recovery unit and a copy posted at the testing station. The procedure should specify the liquor sampling point, treatment of the sample before weighing, sample size, and all equipment parameters found to influence the result.

6.3.1 Testing Black Liquor Solids

During start up, at least one operator off-line solids test shall be performed immediately prior to firing black liquor to confirm solids are above 58% and the refractometers are reading correctly. These test results must be recorded. The sampling location should be on the line to the furnace header or on the header itself. There should be no sources of dilution or chemical addition downstream of the sampling point. Use of liquor samples from other locations (such as the cascade flow box), chosen for convenience, can give erroneous results.

6.4 General Comments

Field solids measurements should be made at regular, frequent intervals. During start-up and periods of upset, more frequent field testing is recommended.

- Boilers with an as-fired solids less than or equal to 70% BLS:
  - Every two hours is a recommended minimum frequency during normal operation.
- Boilers firing Black Liquor solids greater than 70% BLS:
  - Manually test the black liquor solids a minimum of every eight (8) hours if the following conditions are met:
    - The refractometers must be within 2%.
    - The boiler must be in an established stable firing condition.
      - The weekly TAPPI test procedures must be in agreement with the refractometers.
    - The low solids divert should be increased to 62% BLS.
      - An audible alarm should sound if the solids drop to 70% solids or below.
      - If the Black Liquor solids drop to 70% or below the two hour testing frequency shall be resumed.

All solids measurement methods should be checked against the TAPPI Standard, T650-om-05, on a regular basis. Once per week is a recommended minimum frequency.

Refractometers do not see suspended solids while direct solids measurements do include them. The liquor composition should be as stable and representative as possible when taking samples for off-line solids measurement. There should be no deliberate changes in salt cake makeup, or chemical addition when the refractometer is being checked against an off-line measuremen