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Recommended test procedure for black liquor evaporators

1.0 Scope

Procedures used to measure the performance of black liquor evaporators and concentrators have frequently produced inconsistent and confusing results. There are two underlying causes for this problem. One is a lack of understanding of how to measure work done by an evaporation system. The second is that no standard evaluation procedure is available. This Technical Information Paper (TIP) establishes recommended test requirements for an accurate evaluation of black liquor evaporators. The evaluations discussed are for maximum rate evaluation, performance evaluation, and troubleshooting. It further describes procedures to fulfill test requirements for each. A general understanding of evaporators and the kraft chemical recovery cycle by the user is assumed. *Pulp and Paper Manufacture: Volume 5--Alkaline Pulping (1)* gives extensive background on evaporators for others. Definitions are listed to ensure common understanding of terms. This TIP does not specifically include items unique to evaluation of black liquor concentrator systems; however, it can be used as a guide for evaluation of such systems.

2.0 Definitions

See Appendix A

3.0 Safety precautions

Due to the nature of the kraft pulping process, several substances are generated in the black liquor evaporation system that requires special care. The entire evaporation process is conducted at relatively high temperatures, approaching and in select applications exceeding, 300°F (150°C). Consequently, the fluids used in the process are at elevated temperatures and pressures. Therefore, high temperature and pressure precautions must be taken when opening sample valves, servicing instrumentation, servicing steam traps, etc.

Black liquor used in the process is caustic and can cause severe chemical burns, if not properly handled. Extreme caution must be used when working around black liquor. Wear personal protective equipment (PPE) and use safety procedures related to evaporators, black liquor, and hot surfaces developed for the specific mill and company.

Non-condensable gases (NCG) generated in the evaporation process are potential safety hazards. Some of the vapor components are explosive and easily ignite (for example, methanol and turpentine). Others, such as hydrogen sulfide, are extremely hazardous and can be fatal if inhaled at certain concentrations. When working in these areas, it is essential to know and practice mill and company safety procedures related to evaporators, NCG, and area or personal hydrogen sulfide monitors.

The evaporation system can be a dangerous place if we do not take precautions. Although evaporator bodies have been

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designed with the safety of mill personnel as a chief concern, specific safety procedures must be followed when performing work in and around the system.

One must always use care when opening a valve that releases hot fluid into the atmosphere to avoid being severely burned by splattering fluid and/or flashing steam. Always stand to one side of the opening and use appropriate PPE. Do not open any drain valve on a vessel or pipe that contains hot black liquor above its atmospheric boiling point, i.e., under pressure. When the conditions behind the valve are unknown, always assume the process is under pressure with temperature above the atmospheric boiling point. Cool and dilute the stream before draining. Be extremely careful not to open a sample valve and be driven away by heat and/or vapor so that the valve cannot be closed.

4.0 Procedure

Figure 1 is a flowchart which summarizes the steps detailed in this procedure. The chart indicates early decision points and then the sequence of preparatory work, data acquisition, generation of critical information and data evaluation leading to decision points around output reporting or measures for troubleshooting. Each step references the numbered section(s) of text that describes the corresponding material and procedures.

An evaporation system transfers heat from one fluid to another. The amount of work performed can be quantified in several ways, such as:

- The quantity of heat that is transferred (Btu/h, GJ/h).
- Evaporation rate (lb water/h, kg water/h).
- Solids throughput rate from one solids concentration to another (lb/h or kg/h dry solids from, e.g., 15% to 65%).

Evaporation rate or solids throughput rate are the most suitable measurement methods for specifying this work. Each of these takes into account the three most significant variables affecting the quantity of work: feed liquor concentration, product liquor concentration, and liquor feed rate. If any or all of these change, the evaporation rate will be accordingly influenced.

4.1 Key process performance criteria

Six criteria are typically used to judge overall evaporation system performance. All or selected ones may be test objectives.

- Maximum evaporation rate
- Dry solids throughput rate
- Product dry solids content
- Overall steam economy
- Surface condenser outlet water temperature
- Process condensate quality

These criteria are discussed in subsequent sections.

It is important to have full agreement on test objectives between the client and the test group to ensure that the selected evaporator criteria are achieved and that all necessary data are collected. It is highly desirable, and in many cases mandatory, that all criteria are simultaneously met within a given test. Adjustments are possible for some of the parameters via calculations after the test, if simultaneous achievement of all process conditions cannot be achieved. These cases are covered in Section 5.0.

Another important criterion is operational time between evaporator cleanings, i.e., boiling of selected bodies, full set boil out, or hydroblasting of selected bodies. An agreed upon long-term monitoring and evaluation plan needs to be developed to evaluate run time performance. This TIP does not address such a plan, although, in general, many of the techniques used to evaluate short-term performance can also be used for long-term monitoring.

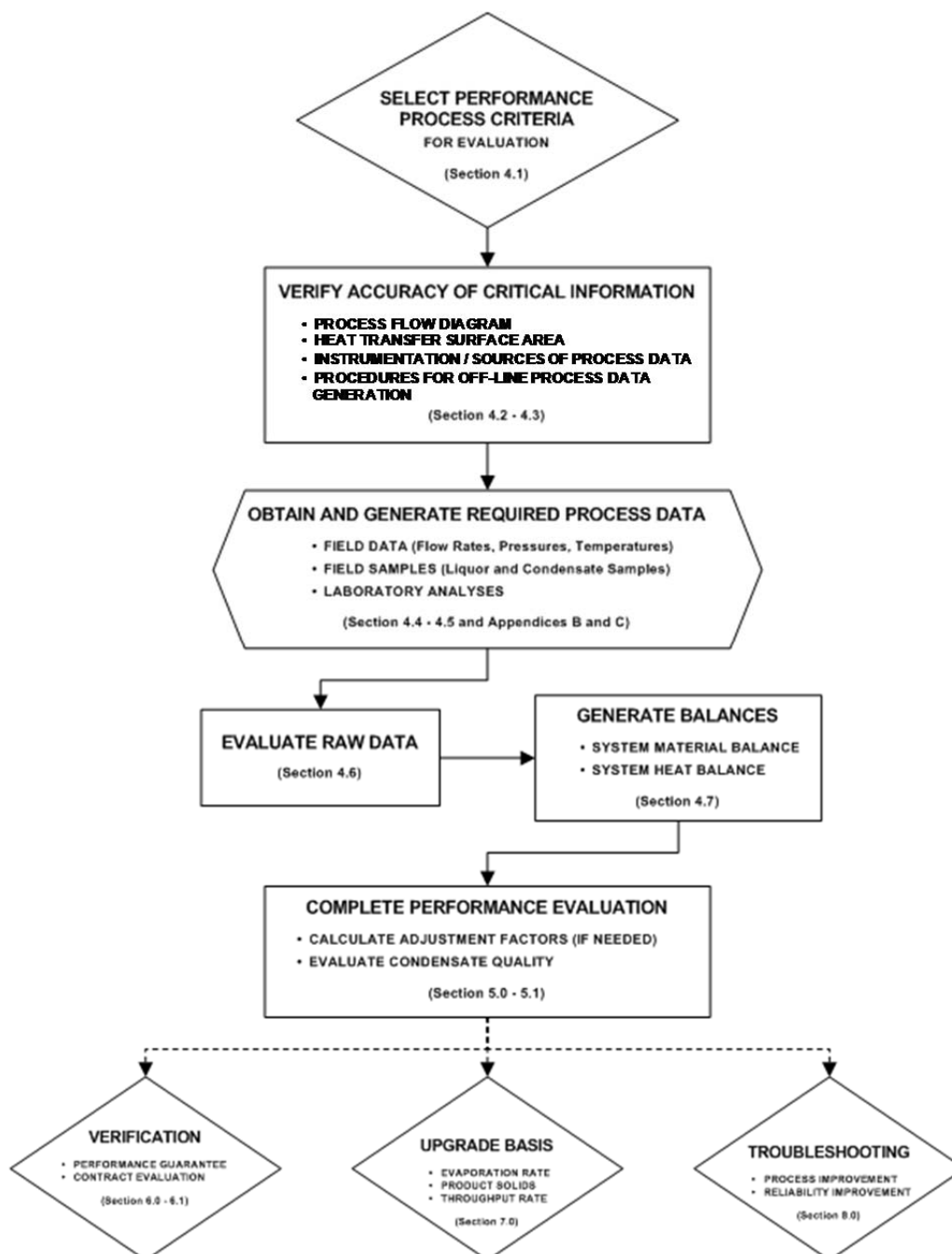


Fig. 1. Flowchart of procedure

4.2 Process flow diagram and heat transfer surface area

An accurate flow diagram showing the overall liquor, steam, vapor, NCGs and condensate flow patterns is required. In many cases, the original design drawing will not reflect the present system. Therefore, the person conducting the evaluation must verify the process equipment, flows, and data on the flow diagram or make a completely new diagram.

A simple block diagram is sufficient in most cases.

Heat transfer surface area is required for each evaporator body, liquor heater and surface condenser; this can be obtained from original design drawings, but may need to be modified for plugged tubes or blanked lamella (plate) elements as noted in the field data collection section.

4.3 System requirements for evaporator evaluation

Evaluations to establish evaporator production capability or compliance with vendor guarantees require a high degree of accuracy. High accuracy performance data yields a more accurate evaluation. Mill instrumentation, while generally adequate to record daily operating parameters and to provide troubleshooting trend data, seldom meets the requirements for an accurate performance test. For these evaluations, the following minimum instrumentation is recommended:

- Operating pressures (calibrated precision electronic manometer, or calibrated pressure gauge and vacuum gage)
- Operating temperatures (precision thermometers or infrared pyrometer)
- Liquid flows (mill instrumentation noting discussion later in the field data section)
- Liquor solids concentration (oven drying tests, TAPPI T 650 "Solids Content of Black Liquor")

The following is an explanation of each of the evaluation steps. Forms to aid in collecting and recording the required data are enclosed as Appendices B and C. Appendix B, Field Data Summary, summarizes performance data recorded in the field from system performance. Appendix C, Laboratory Analysis, summarizes data obtained from laboratory analysis of test samples.

4.4 Field data

Field data must be taken during the shortest possible time interval while the evaporator is operating at steady conditions and at its maximum sustained rate (MSR) for at least 4 hours before collecting data. Other process areas that affect the liquor cycle should also have been operating at typical and steady conditions, ideally 24 to 72 hours. The key areas are:

- Digester (wood species and quality, alkali charge, and H-factor)
- Washers (dilution ratio, black liquor solids from last washer)
- Soap skimmer (in typical operating mode, if applicable)
- Recausticizing area [causticizing efficiency, white liquor (TTA, AA & sulfidity), make-up chemical flow (s)]
- Recovery boiler reduction efficiency.

Care should be taken to ensure that liquor and condensate samples are representative of each applicable stream. The type of flow meter used to obtain flow measurements should be noted to permit evaluation of flow rate discrepancies. Field data collection is important, but it is only as good as the methods for obtaining it. When taking pressure readings, check the connection to ensure it is free of fiber, scale, or other debris. Drain any condensate or liquor from the sample line that would give inaccurate readings. Before getting liquor samples, thoroughly flush sample ports. This is generally indicated when its discharge temperature has stabilized at the "hot" condition. For samples near or above flashing temperature, run the sample line through a cooler before taking the sample to get the best representation of the liquor solids as well as for safety reasons. Fill the sample bottle completely and cap it tight immediately to prevent evaporation loss and a non-representative sample. The following field data is recommended:

- Obtain the following flow rates from mill instrumentation:
 - Feed liquor (s) at each entry point (if a return "sweetening" liquor is used, measure total feed downstream of entry point and the sweetening liquor flow)
 - Product liquor (s) (review relative accuracy of this flow compared to the feed liquor flow with the mill)
 - Any liquor streams leaving and re-entering the evaporator, e.g., liquor to/from the soap skimmer, ash mixing
 - Chemical streams entering after the weak black liquor flowmeter (s)
 - e.g., salt cake, boiler ash, ClO₂ spent acid, crude tall oil brine, caustic, and boil out reclaim
 - Process condensates and flash vapors (likely via calculations) from other processes, if available
 - Steam, including steam supplied to an integrated condensate stripper
 - Steam condensate (s), if available

Desuperheating water, if available
 Surface condenser water
 Fluids heated by evaporator vapors, e.g. demineralized boiler feedwater
 Foul condensate flow to steam stripper, if one is integrated into the evaporators

- Obtain the following pressures using portable, calibrated gauges and from trusted mill instrumentation where available. Preferably, measurements from the portable calibrated gauge are compared to the mill instrumentation:
 - Surface condenser (s), vapor side inlet
 - Non-condensable gas line to vacuum system (ejector or vacuum pump)
 - NCG pipes around the ejectors or vacuum pumps
 - Each effect, liquor side (vapor head)
 - Each effect, steam/vapor side (only if pressure drop between effects is required)
 - Steam, both upstream and downstream of the control valve
 - Product flash tank (s) vapor pressure (alternately, product liquor temperature at pump suction)
 - Condensate flash tank (s) vapor pressure (alternately, condensate temperature at pump suction)
- Record temperatures listed below. Glass laboratory thermometers provide good results, if open streams are available. Alternately, portable IR sensors can be used on uninsulated non-reflective bare pipes or reflective pipes with a flat-black paint spot. Also record temperature data from mill RTD-type sensors, where available.
 - Feed liquor, downstream of any sweetening liquor, brine addition, or other additions as applicable
 - Product liquor
 - Surface condenser cooling water inlet and outlet
 - Non-condensable gas line to vacuum system (ejector or vacuum pump)
 - NCG pipes around the ejectors or vacuum pumps
 - Liquor out of each body
 - Internal and external liquor heater(s) inlet and outlet liquor temperatures
 - Liquor to/from the soap skimmer
 - NCG vent temperature at each body, both directly upstream and downstream of throttling valve or orifice plate
 - Steam temperature at the steam flowmeter, typically upstream of the desuperheater and control valve
 - Steam temperature downstream of the desuperheater
 - Desuperheater water temperature
 - If a stripper is integrated, the following temperatures are also required:
 - Stripper feed condensate out of preheater or into column
 - Stripped condensate from column / to preheater
- Obtain the following liquor/condensate samples:
 - Weak liquor feed(s), downstream of any sweetening liquor addition
 - Product liquor from each body (especially important for parallel bodies and if checking for leaks within bodies)
 - Liquor to and from salt cake pick-up
 - Product liquor(s), upstream of any sweetening liquor take off
 - Steam condensate(s)
 - Clean process condensate(s) each fraction produced
 - Foul process condensate(s)

Operating conditions at the time of the evaluation must be documented. List any factors affecting performance per the following examples:

- Liquor levels in bodies via sight glasses or level sensors, especially if out of range
- Fiber content of the black liquor feed
- Condensate levels in bodies, especially if high or below minimum indication
- Document evidence of foaming
- Liquor content in condensate (conductivity or Na content)
- Describe any abnormal pump performance (have experienced operator review motor current readings and pump noise levels)
- Stability of the system pressure profile (i.e. any “swinging”?) Document swings using PI or DCS printouts

- Condition of internal / external liquor heaters (in / out of service?)
- Vent control strategy for flash tanks (auto / manual and temperature / pressure) and valve position
- Control strategy for product solids (auto / manual and measurement method)
- Type of vacuum system (ejector or vacuum pump)
- Temperature of non-condensable gases from the surface condenser and pre-cooler ahead of the vacuum system
- Time since last full and partial boil out
- Boil out frequency and media used
- Number of mechanically plugged tubes or blanked plate elements in each body including condensers (maintenance records)
- Soap skimmer performance (residual soap)
- Reausticizing area performance (causticizing efficiency)
- Recovery boiler reduction efficiency
- Type of wood furnish

4.5 Laboratory analysis

The following laboratory analyses are required:

- Oven dry solids on all liquor samples (use TAPPI T 650 “Solids Content of Black Liquor”). Feed and product solids should be performed in duplicate or triplicate.
- Feed and product liquor specific gravity TAPPI T625 “Analysis of Soda and Sulfate Black Liquor” or equivalent method to determine specific gravity at liquor flow meter temperature (correlations given in references 2-4 or tabulations e.g., Appendix D may also be used).
- Liquor boiling point rise (BPR curve). This analysis is best done on the current liquor by a laboratory with this capability. If this cannot be done, use the design BPR data for the set to construct a curve. Alternatively, the vapor pressure in each vapor head can be converted to a saturation temperature and subtracted from the temperature of the liquor exiting each body then compared with the measured solids from each body to determine an approximate BPR curve.

4.6 Raw data evaluation

From the raw data, calculate the mass feed rate, solids rate, product rate and evaporation rate. As a check, compare the solids rates based on both feed and product flows and percent solids. Discrepancies normally occur between the two solids rates. Since the product rate is a smaller flow and typically has more variability, the feed rate generally offers a higher degree of accuracy, unless there is reason to believe the feed flow measurement is more variable. Some processes are configured for product flow rate control. In these cases the product flow and percent solids should be used. These calculations involve liquor flows, TAPPI oven-dry solids tests, and liquor specific gravity. Each of these values should be checked.

Solids determination

TAPPI T 650 should be accurate to $\pm 0.10\%$ solids units. Shortcut methods are rarely better than $\pm 0.50\%$ solids units.

Specific gravity values

Specific gravity at a given liquor concentration and temperature can be estimated from tables such as Appendix D (supplied by Kadant/ Chemical Pulping Division). References 2-4 show specific gravity data and provide equations to estimate black liquor specific gravity as functions of total solids content and temperature. Appendix D and the equations in References 2-4 agree to within approximately 0.2% up to 50% solids. When possible it is best to measure the specific gravity of the actual liquor.

Flows

Flow measurements are the most likely source of discrepancies. Evaluate sensor type from which measurements were obtained and determine if one flow should be more reliable than another. Magnetic flow meters or mass flow meters are generally more accurate than other types such as orifice meters. The recovery boiler flow meter and firing solids