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WI \_\_\_\_\_ 160802.03 \_\_\_\_\_

T \_\_\_\_\_ 656 \_\_\_\_\_

DRAFT NO. \_\_\_\_\_ 05 - SARG \_\_\_\_\_

DATE \_\_\_\_\_ October 7, 2020 \_\_\_\_\_

WORKING GROUP  
CHAIRMAN \_\_\_\_\_ 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 Material 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.

**Measuring, sampling, and analyzing white waters  
(Reconfirmation of T 656 cm-07)  
(Fourth ballot required due to non-voters dropped on Ballot 3)  
(no changes from Draft 4)**

**1. Scope and significance**

1.1 This method presents procedures of white water evaluation so that different mills may use substantially the same procedures and thus establish a common basis of comparison. The complete method is intended to evaluate as accurately as possible paper mill white waters and to separate fibrous and nonfibrous constituents. For routine testing only those parts dealing with total solids, suspended and dissolved, correlated with rate of flow, for determination of inorganic and organic materials as mineral fillers, fibers, etc., may be adequate. The complete procedure, however, should serve as an excellent periodic check of routine methods. When checks are made, it is recommended that they cover periods of 1 to 2 weeks.

1.2 This method is not intended for the analysis of environmental samples. Please refer to the proper published procedures designed for the analysis of environmental samples collected for regulatory purposes. If in doubt as to which method to choose, refer to the appropriate (Code of Federal Register) CFR reference for your

samples.

## 2. Measurement of white water volume

2.1 The effluent from each manufacturing unit, such as the paper machines, wet machines, blow pits, etc., should be kept as separate groups and each discharged through a metering device of either the recording or integrating type. The average rate of flow can be obtained either by a recorder or by taking readings at 15- or 30-min intervals for a 24-h period.

2.2 Calculate the flow of white water and report in terms as follows:

$$\frac{\text{liters per minute} \times 1440}{\text{net kilograms of product per 24 h}} = \text{liters of white water discharged per kilogram of product}$$

or

$$\frac{\text{gallons per minute} \times 2,880,000}{\text{net pounds of product per 24 h}} = \text{gallons of white water discharged per ton of product}$$

## 3. Sampling

3.1 To secure a representative sample at a white-water outlet, take portions where there is good agitation, such as from a small box receiving the discharge of the metering weir. Obtain a composite sample for each 24-h period either by an automatic sampler or by combining portions collected at 15- or 30-min intervals. Take increments for a composite sample, proportional to the flow and representative of typical conditions. If there are wide fluctuations in rate of flow, give particular attention to the measuring device to avoid accumulation of solids during periods of low flow.

3.2 Take a large enough composite sample, preferably 5000 mL (or about 1.5 gal) to furnish an adequate quantity for analysis. Should the sample be likely to decompose before analysis, add a small amount (1 or 2 mL/L) of chloroform or other preservative to protect it. However, with chipboard and similar grades, decomposition is often very rapid, and the sample should be analyzed within 2 h after collection instead of depending on a preservative. In hot weather it may be advantageous to pack such samples in ice immediately after collection and before analysis.

#### 4. Procedure

##### 4.1 Total suspended solids

##### 4.1.1 For paper machine and groundwood mill white water

4.1.1.1 Using a rather thick but fast-filtering qualitative filter paper which has been dried to constant weight at  $105 \pm 3^\circ\text{C}$ , filter a well-mixed composite sample of not less than 2000 mL through a 15-cm Büchner funnel, using suction. (Where large quantities of filler are used, the amount of sample may be reduced.) If this filtrate is cloudy, filter a second time. Take care that none of the suspended solids (fiber and filler) pass over the edge of the filter paper. Wash the residue on the paper with several small portions of distilled water. Remove the paper, wiping off with it any deposit on the filter walls. Dry the filter paper and contents to constant weight at  $150 \pm 3^\circ\text{C}$ . Do not prolong the drying unnecessarily. Save the filtrate.

4.1.1.2 For the suggested volume, a balance having practical sensitivity of 1 mg is sufficiently accurate. A balance adapted for mounting on a drying oven greatly facilitates this work.

4.1.1.3 Calculate the moisture-free total suspended solids present, and report as follows:

$$\frac{\text{grams of dry suspended solids} \times 1000}{\text{mL of sample taken}} = \frac{\text{kilograms of dry total suspended solids}}{\text{per 1000 L of white water}}$$

or

$$\frac{\text{grams of dry suspended solids} \times 8345}{\text{mL of sample taken}} = \frac{\text{pounds of dry total suspended solids}}{\text{per 1000 gal of white water}}$$

4.1.2 For sulfite blow pits, sulfate mill diffusers, and similar wastes. Since the filter paper in the foregoing would retain, in addition to pulp fiber, chemicals and incrustants which cannot properly be regarded as pulpmaking or papermaking material, use a disk of 200-mesh wire cloth mounted above the wire of the usual handsheet machine. With this exception follow the weighing and drying procedure as outlined above for the use of filter paper. In this case calculate only the fiber content and discard the filtrate.

##### 4.2 Fixed suspended solids

4.2.1 This determination is particularly important in the case of paper mill white water when fillers are

used in the furnish.

4.2.2 Place the filter paper and contents in a previously ignited and weighed crucible, carefully burn off the organic matter, and ignite the residue to constant weight at  $925 \pm 25^\circ\text{C}$ . Cool the crucible and contents in a desiccator and weigh on an analytical balance. The weight of the residue minus the weight of the ash in the filter paper is a measure of the fixed suspended solids (largely filler residue).

4.2.3 Calculate and report as follows:

$$\frac{\text{grams of fixed suspended solids} \times 1000}{\text{mL of sample taken}} = \frac{\text{kilograms of fixed suspended solids}}{1000 \text{ L of white water}}$$

or

$$\frac{\text{grams of fixed suspended solids} \times 8345}{\text{mL of sample taken}} = \frac{\text{pounds of fixed suspended solids}}{1000 \text{ gal of white water}}$$

4.2.4 In mills where fillers are used which decompose on heating, such as carbonates, sulfides, and sulfates, also give the corrected result, when possible, for the loss of volatile matter caused by heating.

4.2.5 Fillers such as clay contain a certain amount of absorbed moisture which is driven off by drying at  $105^\circ\text{C}$ . Usually they also contain other volatile constituents, such as water of constitution, which are driven off during ignition. By drying specimens of the filler constituents present at  $105 \pm 3^\circ\text{C}$  and subsequently igniting them, factors may be obtained for converting the fixed suspended solids to the moisture-free basis and to the basis as furnished into the beaters.

4.3 *Volatile suspended solids.* Obtain and report the moisture-free volatile suspended solids (largely fiber) per 1000 L (1000 gal) of white water by subtracting the fixed suspended solids after correction for volatile matter from the total suspended solids. Convert, if desired, to the air-dried basis, to loss per 24 h or to per kilogram (ton) of production.

4.4 *Total dissolved solids (from process)*

4.4.1 Place an aliquot portion (equivalent to at least one-tenth) of the filtrate and washings from 4.1.1 in a beaker and evaporate to about 25 mL. Transfer quantitatively to a previously ignited and weighed platinum or glazed porcelain crucible (platinum is preferable). Evaporate to dryness on a steam bath and dry to constant weight

at  $105 \pm 3^\circ\text{C}$  (avoid prolonged heating).

4.4.2 Similarly, evaporate the same quantity of the raw water supply and subtract the resulting weight from the weight of the residue from the total dissolved solids. Calculate the moisture-free total dissolved solids as follows:

$$\frac{\text{grams of dry total dissolved solids from process} \times 1000 \times \text{aliquot factor}}{\text{mL of original sample}} = \frac{\text{kilograms of dry total dissolved solids from process per 1000 L of white water}}$$

or

$$\frac{\text{grams of dry total dissolved solids from process} \times 8345 \times \text{aliquot factor}}{\text{mL of original sample}} = \frac{\text{pounds of dry total dissolved solids from process per 1000 gal of white water}}$$

4.5 *Fixed dissolved solids (from process).* Ignite the crucibles containing the solids from 4.4.1 to constant weight at  $925 \pm 25^\circ\text{C}$ , cool in a desiccator, and weigh. The weight of the residues is the fixed dissolved solids from the process. Calculate as follows:

$$\frac{\text{grams of fixed dissolved solids from process} \times 1000 \times \text{aliquot factor}}{\text{mL of original sample}} = \frac{\text{kilograms of dry total dissolved solids from process per 1000 L of white water}}$$

or

$$\frac{\text{grams of fixed dissolved solids from process} \times 8345 \times \text{aliquot factor}}{\text{mL of original sample}} = \frac{\text{pounds of fixed dissolved solids from process per 1000 gal of white water}}$$

4.6 *Volatile dissolved solids.* Subtract the result of 4.5 from 4.4.2 to obtain the volatile dissolved solids per 1000 L (1000 gal) of white water.

4.7 *Biochemical oxygen demand (BOD)*

4.7.1 All effluents from pulp and paper mills are classified as industrial sewage. As such they usually have a deleterious effect upon the dissolved oxygen content of the receiving stream or body of water. The BOD determination should be run periodically on a sample of water from the trunk sewer or samples from each sewer discharging to the stream. *BOD determinations on composite samples from several sewers mean little and should be discouraged.*

4.7.2 In order to conduct these tests in a manner acceptable to all local, federal, and state authorities having jurisdiction over stream pollution, it is recommended that the latest edition of the "Standard Methods for the Examination of Water and Wastewater" published by the American Public Health Association be followed.

## **5. Report**

It is recommended that a complete report be in the following form:

**7 / Measuring, sampling, and analyzing white waters**

**T 656 cm-07**

Item or department surveyed \_\_\_\_\_ Date \_\_\_\_\_

Survey period \_\_\_\_\_

Net production during period \_\_\_\_\_ kg (lb)

Average liters (gallons) of white water discarded per second \_\_\_\_\_

Average liters (gallons) of white water discarded per kilogram (ton) of product \_\_\_\_\_

	<i>per</i>		<i>per kg</i>
	<i>1000 L</i>	<i>per</i>	<i>(ton) of</i>
Loss and rate of loss	<i>(1000 gal)</i>	<i>24 h</i>	<i>product</i>

Item 1 - Total suspended solids lost, kg (lb)

(a) Moisture-free	_____	_____	_____
(b) 10% moisture basis	_____	_____	_____

Item 2 - Fixed suspended solids, kg (lb)

(a) As determined <sup>1</sup>	_____	_____	_____
(b) As furnished <sup>2</sup>	_____	_____	_____

Item 3 - Volatile suspended solids (volatile on ignition), kg (lb)

(a) As determined <sup>1</sup>	_____	_____	_____
(b) 10% moisture basis, organic portion only	_____	_____	_____

Item 4 - Total dissolved solids (less total solids in water supply), kg (lb)

(a) Moisture-free	_____	_____	_____
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Item 5 - Fixed dissolved solids (less fixed solids in water supply), kg (lb)

(a) As determined <sup>1</sup>	_____	_____	_____
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Item 6 - Volatile dissolved solids, kg (lb)

(a) As determined <sup>1</sup>	_____	_____	_____
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<sup>1</sup>The term "as determined" means as weighed according to the method of analysis without accounting for the decomposition of the inorganic matter during ignition.

<sup>2</sup>The term "as furnished" refers to fillers on the basis of their weight before furnishing.

Item 7 - Biochemical oxygen demand, ppm

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**6. Keywords**

White water, Flow measurement, Suspended solids, Dissolved solids, BOD, Volume, Sampling, Solids content

**7. Additional information**

7.1 Effective date of issue: To be assigned.

7.2 This method, formerly T 656 os-61 and T 656 cm-83, has been reaffirmed as classical in 1997 by committee action.

7.3 Related method: Canadian PAPTAC A-6.

*Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Director of Quality and Standards.* ■