

Method of estimating total crude tall oil in wood pulp

Scope

This test is used to estimate the total amount of crude tall oil in a given wood pulp sample. Washed pulp will contain (1) tall oil soaps which were not effectively washed from the pulp and (2) unsaponified resin and fatty acids which were not converted to sodium soaps in the digester. In this procedure, the pulp sample is boiled in strong caustic to convert the unsaponified tall oil components to their sodium saponified tall oil components. The pulp is then washed well and the filtrate recovered from the digesting and washing steps is combined and analyzed to estimate the total crude tall oil in the pulp. If this number exceeds 5 kg CTO/ton of pulp, it can indicate problems with either the digesting or washing operations.

To differentiate between the unsaponified tall oil components and the unwashed soap, a second portion of the pulp sample should be washed well using dilute caustic. Analysis of the washing filtrate (using PCTM 24) will give the unrecovered soap content of the washed pulp. The difference between that number and the total crude tall oil in the pulp is an estimate of the unsaponified tall oil content. If this number exceeds 1 kg CTO/ton of pulp, problems with the digester or with calcium soap formation are indicated.

Apparatus

- I. A four liter resin reaction kettle or flask, 2000-mL heating mantle, variable transformer, thermometer, Pyrex stoppers, oil trap and reflux condenser assembled as illustrated in Fig. 1.
2. Three stainless steel screens - 30, 80 and 200 mesh.
3. Separatory funnel, 500-mL.
4. Graduated cylinders, 10-mL, 100-ml, 250-mL, and 1-liter.
5. Beakers, 400-ml and 600-mL.
6. Evaporating dish.
7. Steam bath.
8. Coarse filter paper.

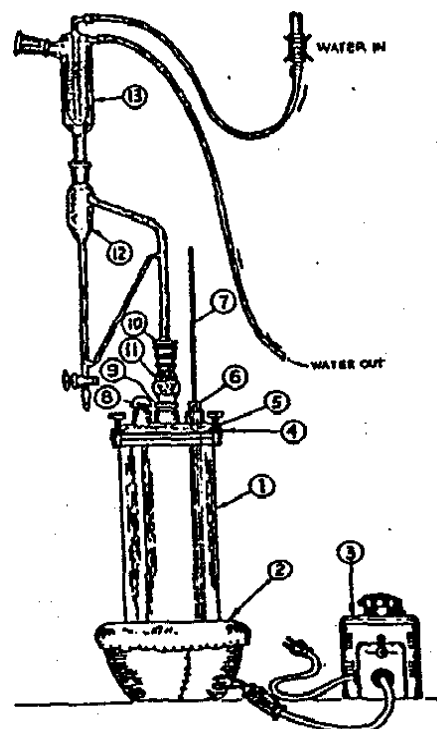


Fig. 1. Apparatus for determining turpentine in pulp or wood chips.

- 1 = 4-liter resin reaction kettle;
- 2 = 2,000-mL electric heating mantle;
- 3 = variable transformer,
- 4 = resin reaction kettle cover;
- 5 = resin reaction kettle damp;
- 6 = thermowell;
- 7 = centigrade thermometer, 0 to 200°C;
- 8 = Pyrex glass stoppers, 24/40;
- 9 = glass adapter, joints 34/45 to 29/42;
- 10 = glass adapter, joints 29/42 to 24/40;
- 11 = packing retainer and packing, joints 29/42 and 24/40; 12 = modified oil trap, ASIMD889 type, altered to have a 10-ml. graduated section;
- 13 = reflux condenser.

Reagents

1. Caustic solution, 0.5N - Mix 20 g of NaOH per liter of water.
 2. HCl (1:1) - Dilute conc. HCl with an equal volume of water.
- NOTE 1:** For safety, add the acid to the water, not the water to the acid.
3. Acetone-methanol - Add 1 volume of methanol to 4 volumes of acetone. Mix well.
 4. Water-acetone-methanol - Mix 2 volumes of acetone with 1 volume of water and 1 volume of methanol.
 5. Petroleum ether, boiling range 40-60°C.
 6. Isopropanol, neutralized to a phenolphthalein end point.
 7. Standardized alcoholic KOH solution, 0.05 N;
maybe prepared by 1:10 dilution of standardized 0.5 N alcoholic KOH (see PCTM 1).
 8. Phenolphthalein indicator solution, 1% in ethanol.

Procedure

1. Obtain a 100 g representative pulp sample for the tall oil availability determination and a 200 g sample for the TAPPI standard moisture determination. In sampling from a drum washer, sample at several locations and include the full pad profile in the composite pulp sample.
2. Add 100 g of pulp (wet' weight) to a four-liter resin reaction kettle along with 2,000 mL of 0.5 N caustic.
3. Set the kettle up for a turpentine determination, as illustrated in Fig. 1.
4. Reflux the turpentine for 5 hours. Control the reflux rate using the variable transformer to prevent foaming problems.
5. After five hours discard the turpentine and separate the pulp from the caustic solution using 30, 80 and 200 mesh screens in series. Save the caustic solution.
6. Transfer the pulp from all three screens back to the kettle and add 2,000 mL of fresh caustic solution. Mix and separate as before. This is a simple washing step - do not recook the sample.
7. Mix the 4000 mL of caustic solution and allow an hour for the solution to become homogeneous. Measure the total solution volume accurately using the graduated cylinders and record the volume as (E).
8. Filter about 1000 mL of the solution through coarse filter paper and discard the remaining solution. Ibis will give enough solution for ten runs.

9. Transfer 100 mL of the solution to a 500 mL separatory funnel.
10. In a fume hood, acidify the solution by adding 15 mL of HCl. Shake and vent continuously for one minute.
11. Add 250 ml, of the acetone-methanol solution, and mix thoroughly for 30 seconds to dissolve any lignin.
12. Add 150 mL of petroleum ether, shaking for about two minutes. Watch gas evolution. Vent frequently, particularly at the start of this step.
13. After shaking, allow about 5 minutes for the phases to separate. Transfer the lower phase to a 600 ml, beaker.
14. Wash the petroleum ether phase in the separatory funnel once with 25 mL of the water-acetone-methanol mixture and add the washings to the aqueous phase in the 600 ml, beaker.
15. Pour, from the top of the separatory funnel, the washed petroleum ether extract into a 400 mL beaker. Start evaporating the petroleum ether by placing the beaker on a steam bath at 80°C.
Caution - A hot plate should not be used to evaporate petroleum ether as ether vapors are heavier than air and a spark could result in a fire or explosion.
16. Pour the aqueous phase from the 600 mL beaker back into the separatory funnel, making sure all solids in the water phase are transferred into the funnel. Extract with 100 mL of petroleum ether. Wash the ether as described above. Combine this extract with the original petroleum ether extract.
17. Evaporate the petroleum ether extract over a water bath at 80°C just until the off appears on the bottom of the beaker. Then add 25 mL of neutralized isopropanol to redissolve the oil.
18. Filter the dissolved oil through coarse filter paper into a 100 ml, beaker. Wash the 400 mL beaker thoroughly with neutralized isopropanol and pour over the filter paper. Then wash the filter paper with neutralized isopropanol until the final filtrate volume is approximately 50 or 60 mL.
19. Add about one mL of 1% phenolphthalein indicator solution and titrate with standardized alcoholic KOH solution, 0.05 N. For a 100 mL sample from pulp off a third washer or decker, the titration is typically only 1-2 mL.

Calculations

1. Assuming an acid number of 160, calculate the weight of tall oil present.

Weight tall oil, g = $A \times N \times 56.1 / 160$

where:

A = mL KOH used

N = normality of KOH solution

2. Calculate the pounds of available tall oil per ton of oven-dried pulp.

Pounds tall oil available/ton OD. pulp =

$$\frac{(C / 100 \text{ mL solution}) \times (E / 100 \text{ g pulp})}{20 D \text{ lbs./ton}} \times$$

where:

C = weight tall oil, g (from the calculation above)

D = pulp consistency, % or g dry pulp/100g pulp mat (measured separately) using Tappi standard method T240 om-93 or T 258 om-94.

E = total volume of caustic used for digesting and washing, mL