Method of estimating crude tall oil in wood chips

Scope

This test is used to determine the amount of available tall oil in a given wood supply. A wood chip sample is ground and then digested twice in 0.5 N caustic. During the digestion the tall oil components are saponified to form their soluble sodium soaps. The resulting pulp is washed to recover the sodium soaps. The digesting and washing filtrates are combined and analyzed to estimate the total crude tall oil in the wood chip sample.

Apparatus

1. Waring blender.
2. A four-liter resin reaction kettle or flask, 2,000 mL heating mantle, variable transformer, thermometer, Pyrex stoppers, oil trap and reflux condenser assembled as illustrated in Figure 1.
3. Three stainless steel mesh screens- 30, 80 and 200 mesh.
4. Separatory funnel, 500-mL.
5. Graduated cylinders, 10-mL, 100-mL, 250- mL, and 1-liter.
6. Beakers, 400-mL and 600-mL.
7. Evaporating dish.
8. Steam bath.
9. Filter paper, coarse

Reagents

1. Caustic solution 0.5 N – Mix 20 g of NaOH per liter of water.
2. HCl (1:1) - Dilute cone. HG with an equal volume of water.
   a. **NOTE 1: For safety, add the acid to the water, not water to the acid.**
3. Acetone-methanol - Add 1 volume of methanol to 4 volumes 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,
6. Isopropanol, neutralized to a phenolphthalein end point.
7. Standardized alcoholic KOH solution 0.05 N; may be prepared by 1:10 dilution of standardized 0.5 N alcoholic KOH (see PCTM 1)
8. Phenolphthalien indicator solution 1% in ethanol.

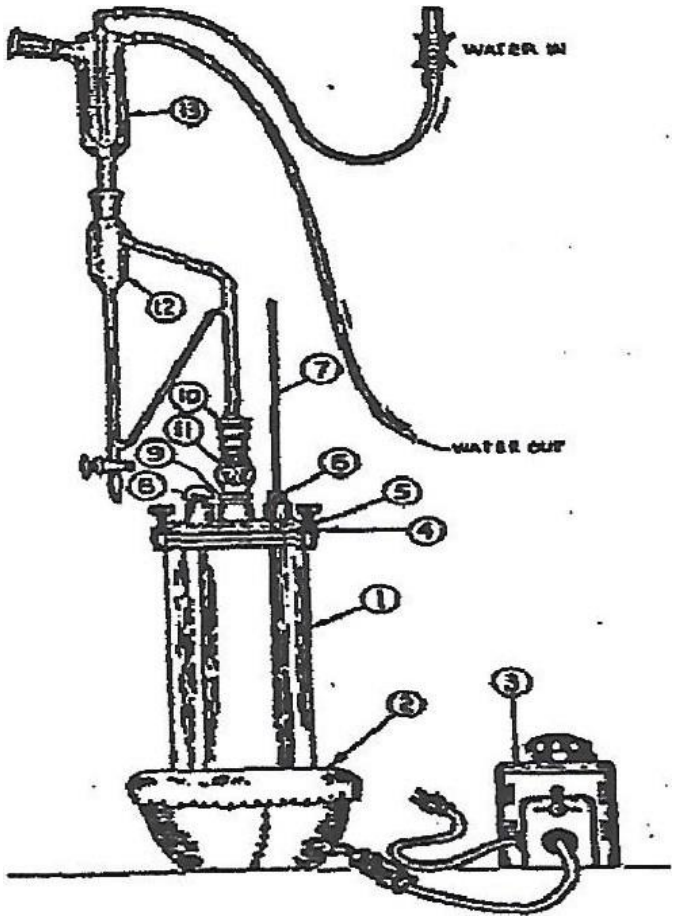

Fig.1. Apparatus for determining crude tall oil in pulp wood chips.

1. 4-liter rosin reaction kettle;
2. 2,000-mL electric heating mantle;
3. Variable transformer;
4. Rosin reaction kettle cover;
5. Rosin reaction kettle clamp;
6. Thermowell;
7. Non mercury centigrade thermometer, 0° to 200° C or other temperature measuring device;
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. Mmodified oil trap, ASTM D889 type, altered to have a 10-mL graduated section;
13. Reflux condenser.

Procedure

Wood Chip Digestion

1. Use the TAPPI standard for sampling a chip population to get a 100 g sample for tall oil availability and a 200 g sample for the TAPPI standard moisture determination.
2. Reduce the 100 g sample of chips (wet weight) to fines in a laboratory Waring blender. Add a small amount of the 0.5 N caustic to the fines to help the blender.
3. Transfer the fines and caustic to a four-liter resin reaction kettle using caustic solution to wash the blender. Add the wash to the fines and bring the total volume of 0.5 normal caustic to 2000 ml.
4. Set the kettle up as shown in Figure 1.
5. Reflux for five hours. Use the variable transformer to control refluxing. Watch that fines do not plug the necks of the condensers. Use a straightened coat hanger or heavy gauge wire to periodically clean the throat of the condenser if necessary.
6. After five hours, discard the volatile oil and separate the fines from the caustic solution using the 30, 80, and 200 mesh screens in series. Save the caustic solution.
7. After draining, transfer all of the fines to the Waring blender, add a small amount of fresh 0.5 N caustic solution and blend until the fines are reduced to pulp.
8. Transfer the pulp and caustic solution from the blender back to the kettle, once again washing with caustic and bringing the caustic volume to2000 ml.
9. Reflux, as before, for five (5) hours.
10. After five hours, separate the pulp from the caustic solution using the screens. Combine the caustic solution with the caustic solution used in the first digestion.
11. Transfer the pulp back to the kettle and add 2000 ml of fresh caustic solution. Mix and separate as before. Repeat with 2000 ml of fresh caustic solution and separate again. These are simple washing steps. Do not reflux.
12. Mix the 8000 mL of caustic solution and allow one hour for the solution to become homogenous. Measure the volume of the combined digestion and washing liquors accurately using the 1-liter graduated cylinder and record this volume as **(E).**
13. Filter about 1000 mL of the solution through coarse filter paper and discard the remaining solution. This will give you enough solution for ten (10) runs.

Tall Oil Extraction

14. Transfer 100 mL of the filtered solution to a 500 mL separatory funnel..
15. In a fume hood, acidify the solution by adding 15 mL of HCI. Shake and vent continuously for one minute.
16. Add 250 ml, of the acetone-methanol solution, and mix thoroughly for 30 seconds to dissolve any lignin.
17. Add 150 mL of petroleum ether, shaking for about two minutes. Watch gas evolution. Vent frequently, particularly at the start of this step.
18. After shaking, allow about 5 minutes for the phases to separate. Transfer the lower phase to a 600 ml, beaker.
19. 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.
20. 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.

21. 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.
22. 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.
23. 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.
24. 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.
25. Add about one mL of 1% phenolphthalein indicator solution and titrate with standardized alcoholic KOH solution, 0.05 N.

Calculations

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

$$\textit{Weight Tall Oil, g} = \frac{A \times N \times 56.1}{160} = C$$

where:
A = mL KOH used
N = normality of KOH solution (about 0.05N)

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

Pounds of tall oil available/ton O.D. Chips =

$$\frac{C}{\textbf{100 ml. Solution}} \times \frac{E}{\textbf{100 g Chips}} \times \frac{\textbf{100 g chips}}{D} \times \frac{\textbf{2000 lb.}}{\textbf{ton}}$$

where:
C - Weight tall oil, g

D - Chips solids content, % or g dry wood/100 g of wood chips. measured separately.

E - Total Volume of caustic digestion solution.