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Crude tall oils

Determination of Water-soluble Acids

1. Scope and field of application

This SCAN-test Standard describes the determination of small amounts of water-soluble acids, such as sulphuric acid, that may be present in crude tall oils. It is applicable to all kinds of crude tall oils except those containing considerable amounts of unsplit soap.

2. References

SCAN-T 4, Ash in tall oil
SCAN-T 7, Water in crude tall oil
SCAN-T 8, Sampling of crude tall oil

3. Definition

Water soluble acids (in crude tall oils): Strong mineral acid that can be extracted into an aqueous phase. All acid is calculated as if it were sulphuric acid.

4. Principle

The sample is heated with an added amount of water. The acid content in the aqueous phase is determined by titration with sodium hydroxide solution.

5. Reagents

5.1 *Cresol red indicator solution.* Dissolve 0.1 g of cresol red in 20 ml of hot ethanol. Dilute to 100 ml with distilled water.

5.2 *Sodium hydroxide solution,* standardized, 0.1 mol/l. Dissolve 4.0 g of NaOH in distilled water and dilute to 1 litre.

Standardize against ca. 800 mg of absolutely dry potassium hydrogen phthalate, $\text{KHC}_8\text{H}_4\text{O}_4$, weighed to the nearest 0.1 mg. Transfer the salt to a conical flask and dissolve it in 15 ml of distilled water. Add a few drops of the cresol red indicator solution. Titrate with the sodium hydroxide solution, using a 25 ml burette, graduated to the nearest 0.1 ml. The end-point is indicated by a colour change from yellow to red.

6. Preparation of sample

Obtain a sample as described in SCAN-T 8. Shake the sample bottle before taking a portion for analysis. At the same time take a sample for determination of the water content as described in SCAN-T 7.

7. Procedure

Weigh about 100 g of sample, to the nearest 0.1 g, in a 500 ml conical flask. Add 100.0 ml of distilled water with a pipette and mix carefully. Heat on a boiling water bath and mix thoroughly when hot. Leave the sample flask on the water bath until the water and the tall oil have separated, which normally takes about 5 h. With some samples it may be necessary to add 5 to 10 g of sodium chloride (NaCl) to achieve separation.

NOTE. — If a persistent emulsion is formed this may be due to the presence of unsplit soap in the tall oil. This may be verified by determination of the ash content as described in SCAN-T 4.

After cooling, transfer 50 ml of the water layer to a 200 ml conical flask using a pipette. Blow gently through the pipette and twist it when penetrating the oil layer to ensure that no oil is withdrawn with the aqueous phase. Wipe off any oil adhering to the tip of the pipette. Add a few drops of the cresol red indicator solution and titrate with the sodium hydroxide solution in the same manner as in the standardization (5.2).

8. Calculation

Calculate the content of water soluble acids from the expression

$$X = 98000 \cdot a \cdot c/w$$

where

X = content of water soluble acids, expressed as milligrams of sulphuric acid per kilogram of tall oil (moisture-free basis).

a = volume of sodium hydroxide solution consumed in the titration, millilitres.

c = concentration of the sodium hydroxide solution, in moles per litre.

w = mass of the tall oil sample (moisture-free basis), in grams.

The numerical factor 98000 contains the mass of sulphuric acid equivalent to 1 ml of 1 mol/l sodium hydroxide solution (49 mg), the factor 2 because only half of the aqueous phase is taken to titration and also the factor 1000 so that the result will be obtained in the unit milligram per kilogram.

The results of duplicate determinations should not differ by more than 50 mg/kg.

9. Report

The test report should include reference to this SCAN-test Standard and the following particulars:

- (a) date and place of testing,
- (b) identification mark of the material tested,
- (c) the results,
- (d) any departure from the standard procedure or any other circumstances that may have affected the results.