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SAPONIFICATION NUMBER OF TALL OIL

Definition

The saponification number of tall oil is the number of milligrammes of potassium hydroxide required to saponify the esters present in one gramme of the oil.

Scope

This method applies to crude and distilled tall oil, tall oil rosin, tall oil fatty acids, tall light oil and tall oil pitch.

Apparatus

1. A pH-meter, with an alkali-resistant glass electrode and a saturated calomel reference electrode, readable to the nearest 0.05 pH unit. The pH meter should be calibrated and checked as described in SCAN-T 11.
2. A mechanical stirrer with a glass propeller-type paddle, or a magnetic stirrer with a coated magnet.
3. Burette, 50 ml, with 0.1 ml divisions. An automatic burette should be protected by means of soda-lime tubes against absorption of atmospheric carbon dioxide.
4. Erlenmeyer flasks, capacity 250 ml, with ground stoppers — for example, standard taper No. 34.
5. Condenser, water cooled, with a joint to fit the Erlenmeyer flasks.

Reagents

1. Ethanol, C_2H_5OH, 95 %, of highest purity (Note 1).
2. Ethanolic potassium hydroxide solution, 0.5 M. Dissolve 33 g of KOH pellets in *ca.* 30 ml of distilled water and dilute to 1 litre with ethanol. Standardize the ethanolic potassium hydroxide solution as described in SCAN-T 11.
3. Hydrochloric acid, 0.5 M, standardized, molarity known to the nearest 0.001 M.

Procedure

Shake the sample bottle and weigh into an Erlenmeyer flask 2.95 to 3.05 g of the contents, to the nearest milligramme (Notes 2 and 3). Add 50.0 ml of the ethanolic potassium hydroxide solution and connect the flask to the condenser. Heat to boiling in a water-bath and reflux for 30 min. Cool the mixture and transfer it without delay to a 300 ml beaker using a total of 100 ml of ethanol for at least 5 rinsings.

Ensure that the pH meter is in calibration; rinse the electrodes first with distilled water and then with a mixture of 15 ml of distilled water and 100 ml of ethanol.

Fill a burette with the 0.5 M hydrochloric acid. Place the beaker so that the burette tip is close to the surface of the solution. Immerse the electrodes in the solution, switch on the stirrer, and adjust it to mix well without splashing. Record the initial pH and add portions of acid; after each addition allow the pH to stabilize and record it and burette reading. Add 5 ml portions until the pH is about 12, then 1 ml portions until the change in pH exceeds 0.3 unit, and continue with 0.1 ml portions, until the equivalence point has been passed — this is indicated by a significant decrease in the change in pH per unit volume of acid added. Increase the additions in the reverse order, and end the titration at pH 8 or below.

Plot the pH against the added volume of acid, expressed in millilitres, determine the inflection point to the nearest 0.1 ml and record this as the end-point.
Make a blank determination. Instead of the sample solution, use 50.0 ml of the ethanolic potassium hydroxide solution; otherwise follow the procedure described above.

Calculation and report

Carry out two determinations and calculate the saponification number from the expression

$$X = \frac{56.1\ m\ (b - a)}{c}$$

where

a = the volume of hydrochloric acid consumed in titration of the sample, ml

b = the volume of hydrochloric acid consumed in titration of the blank, ml

c = the amount of sample taken, calculated on a water-free basis, g (Note 3)

m = the molarity of the hydrochloric acid

X = the saponification number.

Calculate the mean and report it to the nearest integer (Note 4).

Additional information

This method is based on ASTM D 803 (1) and should give equivalent results.

Note 1

The shelf-life of the ethanolic potassium hydroxide solution depends on the purity of the ethanol used in its preparation.

Note 2

Dissolve samples of tall oil pitch and tall oil rosin in a small volume of a mixture of equal volumes of toluene and either methanol or ethanol.

Note 3

Determine the water content of tall oil samples according to SCAN-T 7.

Note 4

Results of duplicate determinations of the saponification number may differ by 0.6 unit for distilled tall oil, tall oil rosin, tall oil fatty acids and tall light oil and by 1 unit for crude tall oil and tall oil pitch. Results from different laboratories may be expected to agree within 1 and 2 units, respectively.

Literature

1. American Society for Testing and Materials, 1971 Book of ASTM Standards, ASTM Designation: D 803-65, Part 20, pp 369—370.

This method has been published in:

Norsk Skogindustri *26* (1972):4, 103—106. (English, Norwegian).
Paperi ja Puu — Papper och Trä *54* (1972):4, 277—282. (English, Finnish, Swedish)
Svensk Papperstidning *75* (1972):7, 243—244. (English)
Svensk Papperstidning *75* (1972):8, 329—330. (Swedish)

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