Standard Oil Company
(Indiana)

INFORMATION DIVISION TRANSLATION T46-66

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API-TOM Reel 67 - Frames 940-941
Ruhrbenzine Oberhausen-Holten August 23, 1940
Testing of Products Like Soap Prepared from Fats, Etc.

The water content is determined indirectly or through determination of the weight loss of a 50 average sample, which has been loosened up with ignited sand or the like, after slowly drying up to 105°. Or 50-20 g. soap is heated and distilled with 40-80 cc. benzol pre-dried with anhydrous sodium sulfate. After the clear layer separation in the receiver or in the graduated distillation apparatus the water amount is recalculated by percent.

Total fat content: 5-10 grams are dissolved in water by heating and the fatty substance is precipitated out with diluted sulfuric acid. Acid is added in excess until methyl orange colors permanently red. Separated fat or acids are cleared through further heating, it is mixed with an amount of paraffin that is about the same as the amount of substance that one had used in case the fatty acid did not solidify upon cooling. Separated fats etc. is taken off as a cake; it is washed by repeated melting with hot water. All the water drops that cling to it are absorbed or dried and it is weighed. For the preparation of pure fatty acids these are taken up with trichloroethylene, petroleum- or ethyl-ether instead of paraffin. They are separated in a separating funnel after thorough shaking and the lower aqueous solution is put in a second separation funnel. Here one washes again with petroleum ether. The ether extracts are combined and are evaporated either at low temperature, or, in the presence of unsaturated fatty acids, in carbon dioxide stream. It may be dried with dehydrated sodium sulfate which has to be washed later with ether to be fat-free. This fatty material is determined with simultaneous determination of alkali content and filler in special apparatus (Huggenberche separating burette, Spiepelscher soap analysis apparatus, Luring burette, apparatus Rohring).

Fatty acids that are separated from soap powders are not absorbed by paraffin because otherwise the materials that cause turbidity accumulate with the fat layer and are weighed in the paraffin-cake along with the fatty acid, and the determined fat content is too large for the small amount of substance that is used in the analysis. Long, vigorous shaking in the separatory funnel directly with the volatile solvent is absolutely necessary for soap powder for sharp separation, possibly with admixture of salt. The mixture ratio of kernel and glue fats in the soap ingredients is determined --either in absence or after previous removal of possible resin content and unsaponifiable admixtures--from the saponification number of the total fatty acids.

$$\frac{(250 - \text{saponification number of total})\ 100}{250-200} = \text{fatty acids of kernel fats}$$

$$\frac{\text{(Saponification number total - 200) x 100}}{250-200} = \text{fatty acids of the glue fats}$$

In these formulas, 250 stands for the median saponification number of glue fat fatty acids, 200 that of kernel fat fatty acids.

Total potassium content: is determined by the use of the excess half normal H_2SO_4 of the above total fat determination and by back-titrating of the amount that has not been used in the neutralisation, in the aqueous portion that has been freed of fatty acids. The last titration is done with hydroxide with the use of methyl orange as indicator.

1 cc N/1 acid = 0.04006 g NaOH = 0.0311 g Na_2O
= 0.05611 g KOH = 0.0473 g K_2O
= 0.03691 g $Ca(OH)_2$ = 0.02793 g CaO

$$\text{Total potassium contact in sodium soaps} = \frac{1.55 \text{ used up n/2 acid g}}{\text{g substance}}$$

Combined alkali $\frac{1.55a}{\text{sample wt.}}$ calculated as Na_2O resp. $\frac{1.905 \times a}{\text{sample wt.}}$

Calculated as K_2O where a = the total amount of half normal NaOH, (resp. KOH), that was used for the saponification of the total fatty acids.

Caustic alkali content: 5 to 10 times the amount of soap is dissolved in alcohol which has been dehydrated by distillation over NaOH and which has been neutralized shortly before use. The insoluble material (alkali carbonate, filler) is filtered off. Phenophthalein (red color shows free alkali, colorless shows neutrality or acid content of the soap) is added and one titrates with n/10 HCl. Free alkali in sodium soaps equals $\frac{0.4 \text{ acid}}{\text{sample}}$ calculated as NaOH, in potassium soaps 0.56 is the factor.

The free alkali content can also be determined by dissolving the soap in 20-50 times the amount of water and precipitating with hot concentrated $BaCl_2$ solution 30:100. The barium soap is filtered off and the filtrate is titrated after addition of phenolphthalein with n/10 HCl. In the case of the soft soaps, 5 grams are dissolved under the reflux cooler in 50 cc. of neutralized alcohol. In a cooled solution 5 grams dehydrated Na_2SO_4 (and phenolphthalein?) is stirred in, then titrated with N/10 alcoholic HCl.

Translated Oct. 22, 1946 - Rochelle H. Bondy
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