

SAPONIFIABLES IN OILS

Application

This procedure is used to determine percent unsaponified saponifiables, soaps, emulsifiers and wetting agents in base soluble oil materials.

Reagents

Isopropyl Alcohol and Distilled Water - (50:50)

Petroleum Ether: Reagent grade

Hydrochloric Acid: Concentrated

Potassium Hydroxide:

1.0 normal alcoholic solution. Weigh 60 g KOH (ACS grade) and heat in 500 mL of methyl alcohol while stirring. Allow to stand overnight to precipitate carbonate. Decant or filter through a fritted glass filter # 0 and dilute to one liter with methyl alcohol.

Ethyl Ether: Reagent grade

Sodium Sulfate: Reagent grade

20 % in water with methyl orange indicator.

Sodium Sulfate: Reagent grade, Anhydrous, granular.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure

1. Weigh 5 - 10 g of sample into a separatory funnel.
2. Add 100 mL 50 % isopropyl alcohol (Reagent #1) and shake well to disperse material.
3. Add 50 mL of petroleum ether and shake well again.
4. Allow to stand and separate into two layers.
5. Drain alcohol layer (bottom) into a second separatory funnel.
6. Extract the alcoholic layer five more times with 30 mL petroleum ether and combine all extracts.

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7. Shake combined ether extracts from above with 25 mL 50 % isopropyl alcohol to remove any soap that may be in this part of the extract, usually three to five extractions.
8. Combine all alcoholic extractions.
9. Drain petroleum ether extract into a tared flask. Evaporate the ether on a steam bath and dry to a constant weight. This portion contains unsaponified saponifiables and non-saponifiables (oil).
10. Drain alcoholic portions from Step 8 into a tared beaker. Evaporate on a steam bath to dryness and place in oven at 100 °C, dry to constant weight. This portion contains soaps, emulsifiers and wetting agents.

$$\begin{array}{l} \text{\% Soaps, emulsifiers} \\ \text{\& wetting agents} \end{array} = \frac{\text{difference in weight}}{\text{weight of sample}} \times 100$$

11. To extract from Step 9 in flask, add 25 mL 1.0 N alcoholic KOH and saponify for 2 1/2 hours.
12. Cool and wash into a separatory funnel, using 50 % isopropyl alcohol water followed by petroleum ether. Rinse flask two or three times to remove all products formed.
13. Shake and separate into layers, repeating Steps 2 - 8.
14. Dry evaporate the petroleum ether extract on a steam bath and dry in oven at 100 +/- 2 °C to constant weight in tared beaker. This is the non-saponifiable matter (oil).

$$\text{Unsaponified total extracted \%} = \frac{\text{difference in weight}}{\text{weight of sample}} \times 100$$

15. Drain alcoholic layer into a beaker and evaporate until alcohol is removed.
16. Add 5 - 10 mL concentrated HCl to break up the soap and heat to coagulate fat.
17. Cool after heating and wash into separatory funnel, using water and 100 mL ethyl ether. Shake well and allow to separate.
18. Extract acid layer three to five times with 25 mL of ethyl ether for each extraction.
19. Combine all ethyl ether extractions and wash with 25 mL, 20 % sodium sulfate. Drain aqueous solution and repeat until washings show neutral to methyl orange (color change from red to orange). Dry ether layer with 10 grams anhydrous, granular sodium sulfate.
20. Drain through cotton into a tared 250 mL beaker. Wash funnel and cotton with ethyl ether until fat free.

Evaporate ethyl ether on steam bath and dry at 100 +/- 2 °C to constant weight.

$$\begin{array}{l} \text{\% Recovered unsaponified} \\ \text{saponifiables} \end{array} = \frac{\text{difference in weight}}{\text{weight of sample}} \times 100$$

Chemicals, materials, parts, and equipment referenced in this document must be used and handled properly. Each party is responsible for determining proper use and handling in its facilities.