

KES

**Method for Analysis of Soap
and Base oil for Grease**

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1. Scope of Application

This standard shall be applied to analyze content of mineral oil of grease made of mainly mineral oil and soap.

This method is not proper for grease produced by thickening material insoluble to n-Hexane, thickening material, which is not soap, and similar ingredients except the mineral oil or hydroxyl fatty acid soap, however, if you are familiarized with the analysis, this can be applied to those greases.

2. Specimen

Since required amount of specimen depends on soap content, make 8 ~ 30 gr depending on size of main material.

Measure the weight of the specimen to unit of 0.1 gr.

3. Reagent

(1) Normal Hexane (n-Hexane)

(2) Alcohol (50 %) : Treat 95 % industrial ethyl alcohol or denatured alcohol with potassium hydroxide, take extracted alcohol phenolphthalein as indicator solution, and exactly neutralize with potassium hydroxide or sodium hydroxide.

(3) Phenolphthalein solution : Add 1.0 gr of phenolphthalein to 50 ml of the alcohol distilled in item (2).

(4) Hydrochloric acid

(5) Weak hydrochloric acid : The one that is diluted to 10 ± 5 %

(6) Benzene

(7) T-butanol

(8) Butter yellow indicator: Use by melting 0.02 gr per 1 ml of P-dimethylamino azobenzene

(9) Potassium hydroxide alcohol solution (0.5 N)

4. Method A

4.1 Hydrochloric acid decomposition

- (1) First phase decomposition procedure : Obtain the specimen in 250 mL triangular flask, add 20 mL of n-hexane and 50 mL of 10 % hydrochloric acid, put a boiling chip, and heat until it boils a little for about 3 hours using a reflux refrigerator. During this, shake the flask horizontally from time to time, and decompose until the oil layer is completely transparent. If it is difficult to decompose according to kinds of soaps, add n-hexane or boil hard using the hydrochloric acid. Cool down the specimen in the room temperature.
- (2) Single phase decomposition procedure : Obtain the specimen in 250 mL triangular flask, put 50 mL of t-butanol, 50 mL of n-hexane and 2 mL of strong hydrochloric acid (Specific gravity 1.19), drop 2 drops of butter yellow indicator, put a magnetic bar, and heat and agitate on a heating-agitator. While agitating, be careful of overflowing due to boiling when specimen lump is large, and smash with a glass rod. For greases with large molecular weight, put 50 mL of benzene instead of n-hexane and heat and agitate, and if the temperature drops to 24 ~ 26 °C, be careful for t-butanol to be congealed. If the indicator colors yellow, add about 1 mL of the strong hydrochloric acid until it changes to red, and continuously heat and agitate for 10 ~ 15 minutes to dissolve greases. At this time, the indicator is completely decomposed. Cool down the dissolved specimen in the room temperature.

4.2 Separation of Produced Solution

Move the specimen to separatory funnel (A). (If the benzene is used instead of the n-hexane in method 4.1 (2), extract the benzene and move to the separatory funnel.) Wash out with small amount of the n-hexane and water, and if the specimen is congealed, wash out with hot n-hexane and warm water. After moving to the separatory funnel (A), leave the funnel for about 30 minutes, separate an oil layer and water layer