

**TOTAL SAPONIFIABLE MATTER AND ADDED SULFUR
IN GEAR LUBRICANTS****Application**

This procedure is used to determine the total saponifiable matter and added sulfur in petroleum oil base lubricants which are used for lubrication of gears.

Description of Methods

This procedure describes two methods - one for total saponifiable matter and the other for added sulfur. The two methods are combined in this procedure since it is necessary to separate the saponifiable material when determining added sulfur.

The procedure involves a dilution of the heavy oil, acidification to free the added sulfur compounds, saponification of the neutral fats and fatty acids, and extraction of the soap thus formed from the mineral oil with alcohol and ether. Sulfur is determined by the bomb method on the original sample and the extracted mineral oil and the difference calculated as added sulfur.

Apparatus Required**Saponification Flask**

The 300 mL saponification flask and the condenser tube are both Corning No. 7280. They are made of boron-free, alkali resistant glass with Ts 24/60 joints.

Additional Apparatus

Separatory funnel, 500 mL, Pyrex with ground glass stopper; water or steam bath; laboratory balance; electric hot plate.

Reagents Required

Benzol, ACS-Sulfur free; isopropyl alcohol, ACS; hydrochloric acid (1:1); petroleum ether, reagent grade (BP 30 - 60 C); ethyl ether, ACS.

Alcoholic Potassium Hydroxide

Weigh 60 g of KOH (ACS) and heat in 500 mL of 3A alcohol with stirring. When dissolved, allow to stand overnight to precipitate carbonates. Decant or siphon through asbestos and dilute to one liter with 3A alcohol.

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.

Date	Action	Revisions
2000 08 07		Editorial – no technical change A. Cockman
1992 04 28		

Printed copies are uncontrolled

Page 1 of 3

Copyright © 2000, Ford Global Technologies, Inc.

Procedure

1. Weigh 5 to 6 grams (+/- 0.001 g) of sample into a 250 mL beaker.
2. Add 25 mL of benzol to dilute the sample and then 25 mL of isopropyl alcohol and 20 mL of dilute HCl (1:1).
3. Heat to boiling and boil gently for 20 minutes on the hot plate.
4. Transfer the hot sample to a 200 mL separatory funnel, washing with a little benzol and hot water.
5. Allow the layers to separate as much as possible and draw off bottom layer.

Note: It is difficult to obtain a clean separation between the benzene and water layers because of the formation of an emulsion at the interface. Therefore it is suggested that, in the washing procedure, the emulsion layer be left with the benzene layer and the final separation be made as complete as possible on the last washing.

6. Wash the oil layer five times with 50 mL portions of hot water, allowing each wash to separate as much as possible before drawing off. The washings should be done carefully, as follows:

Add hot water to the benzol in the separatory funnel and swirl with the stopper off. Stopper the flask, invert the separatory funnel and open the stopcock cautiously while in the inverted position to relieve pressure. Close stopcock, swirl gently and again open to relieve pressure. Do this for each of the water additions.

7. Collect the total water washes and add 40 - 50 mL of the benzol. Shake well, let settle and drain off the water. Wash with one or two washings (25 mL each) of warm water and draw off water layer.
8. Combine the benzol extracts from Steps 6 and 7 and transfer to a saponification flask and evaporate the solvent. (Avoid open flame!)
9. Add 50 mL of alcoholic potassium hydroxide, attach condenser to flask, place on electric hot plate and saponify under reflux for one hour.
10. Cool, transfer to a separatory funnel, rinsing flask with small portions of distilled water (45 mL total). Rinse flask with small portions of petroleum ether (50 mL total) and finally rinse with 50 % alcohol. Shake, allow to settle and drain off the alcoholic solution into a separatory funnel.
11. Shake the alcoholic soap solution with portions of petroleum ether to separate mineral oil, and add to the mineral oil extract in Step 10. The first extraction should be made with 50 mL of petroleum ether and then four additional extractions made with 30 mL portions of petroleum ether to completely separate the mineral oil from the soap. Save both the alcohol soap extract and the ether-oil extract.
12. Shake the combined petroleum ether extracts three times with 15 mL portions of 50 % alcohol to which has been added a trace of alkali. Add these extracts of alcohol to the soap solution obtained in Step 11.