

DETERMINATION OF FREE OR ACTIVE SULFUR IN LUBRICATING OIL**Application**

This procedure is used for the determination of free sulfur in petroleum lubricating oils. The sample is dissolved in a solvent and refluxed in contact with copper gauze or crimp and the resulting copper sulfide is ignited in a combustion furnace and sulfur determined by absorption and titration or by a gravimetric method as in the alternate procedure.

Solvents and Apparatus Required**Copper Gauze or Crimp**

Clean 40-mesh copper gauze or crimp by immersing in dilute nitric acid (1:10). Rinse in distilled water and finally in acetone.

Condenser

Any reflux water cooled condenser is satisfactory.

Sulfur Determinator

An instrument such as the Leco or Lindberg, capable of determining sulfur by the combustion method.

Solvents

C.P. acetone and C.P. benzene.

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**Size of Sample**

The sample size is varied depending on the percentage of sulfur present in the oil and the amount of sulfur which can be determined by the sulfur apparatus used.

Example: If the sulfur determinator such as the Leco reads sulfur directly in percentages up to .20 % on a 0.5 g sample, then for an oil containing approximately 2 % active sulfur 50 milligrams are used.

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Weigh the appropriate size sample into a 500 mL flask. Add 50 mL of C.P. benzene, 100 mL of C.P. acetone and agitate to effect complete solution of the sample. Place a piece of copper gauze or crimp (approximately 2 g formed to fit the combustion boat used in the combustion sulfur apparatus) in the flask and reflux the solution under a water cooled condenser for 2 hours. Add an additional small piece of copper gauze and again reflux. When no more sulfur deposits on a fresh piece of gauze the pieces are removed and washed with C.P. benzene and dried. The gauze is then placed in a sulfur combustion boat and sulfur determined as usual. (See Procedure AA 005-01).

Alternate Procedure

Application

When no combustion sulfur apparatus is available, the copper sulfide is converted to sulfate and determined by precipitating as BaSO₄.

Solutions Required

Bromine - Potassium Bromide Solution

Dissolve 160 g of KBr in just enough water to effect solution. Add 100 mL of bromine, mix well and dilute to 1000 mL.

Barium Chloride Solution - 10 %

Dissolve 100 g of BaCl₂•2HCl₂O in distilled water and dilute to 1000 mL.

Barium Chloride - Hydrochloric Wash Solution

To 10 mL of BaCl₂ solution (10 %) add 10 mL of HCl (conc.) and dilute to 1000 mL with distilled water.

Procedure

Treat a sample of the oil as in the preceding method to the point where the copper gauze containing the deposit of sulfide is washed and dried. Transfer the sample to a 400 mL beaker, cover and add 10 mL of KBr-Br mixture. Let stand for 15 minutes in a cold water bath near the hood with occasional stirring. Add 50 mL of HNO₃ (conc.) carefully and allow to stand another 15 minutes at 23 +/- 2 °C. Evaporate on a steam bath to a syrupy consistency, add 30 mL of HCl (conc.) and evaporate to dryness.

Add 30 mL of HCl (conc.) and evaporate to a syrup. Add 5 mL of HCl (conc.), dilute to 100 mL with hot water and filter. Wash with dilute HCl (2:98). Dilute to 250 mL, heat to 150 °F and stir in 10 mL of BaCl₂ (10 %). Allow to settle overnight. Filter on Whatman No. 42 paper and wash with BaCl₂-HCl wash solution, and finally with hot water. Ignite in a tared platinum crucible at 1000 °F until carbon is destroyed and then at 1800 °F to constant weight. Calculate the sulfur as follows:

$$\frac{A \times 0.1373}{B} \times 100 = \% S$$

Where A = Weight in g of BaSO₄
and B = Weight in g of sample used

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.