

sulfate tends to low results, while the nitrate has the opposite effect. Only by chance will these errors cancel one another.¹ A few determinations made with double the usual amounts of reagents seem to show that the net error due to occlusion is small.

The present paper is a preliminary one, and it is intended to publish a more detailed discussion of the whole subject later on.

SUMMARY

The desirability of investigating the possible effect of sulfur compounds on the properties of lubricating oils is pointed out.

The disadvantages of the methods now in general use for the determination of sulfur are indicated.

A new procedure, which gives results at least as accurate as those by the bomb calorimeter, is described.

DETERMINATION OF MINUTE QUANTITIES OF OIL IN SULFUR

By L. S. Bushnell and H. Smith Clark

FREESPORT SULPHUR COMPANY, FREESPORT, TEXAS

Received November 25, 1919

The presence of very small amounts of oil in sulfur is objected to by manufacturers of certain sulfur products. The sulfur may contain carbonaceous residues from burned oil, and the method here described is not intended to include these.

The estimation of oil by simple extraction as ordinarily made with a volatile solvent—such as sulfuric ether or petroleum ether—will not suffice in the case of sulfur, because, while the sulfur is only slightly soluble, there is usually such a small amount of oil present (from 0.001 per cent to 0.32 per cent, or more) that the extract contains considerably more sulfur than oil. In this method extraction is made as usual, and the solvent purified from sulfur by boiling it in a Wiley continuous extractor in which has been placed a roll of copper foil.

APPARATUS

A sheet of copper foil 4 in. \times 15 in. is rolled up in such fashion that no two points of its surface come in contact. It is desirable that the copper sheet be rolled uniformly so that there will be a space of about three-sixteenths inch between any one turn of the roll and the next turn. The sheet may be cut so that at one corner a tab three-eighths inch square projects. A hole is punched in this tab so that the coil may be lifted from the Wiley tube with a wire hook. The end with this tab should be rolled first, and the tab project from one end of the coil.

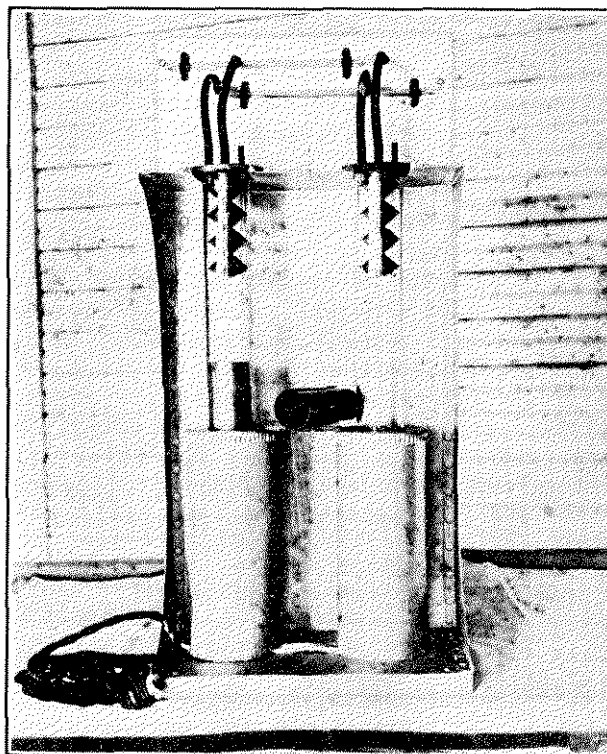
A 100 cc. volumetric flask; a thin glass beaker, tall form, without lip, 300 cc. capacity; and a Wiley continuous extractor, without crucible or thimble, complete the required apparatus.

PROCEDURE

Fifty grams of the pulverized sulfur (or a smaller quantity, if the oil content is known to be high) are weighed and transferred to the 100 cc. flask. On this are poured about 50 cc. of redistilled petroleum ether;

¹ Allen and Johnston, *J. Am. Chem. Soc.*, **32** (1910), 588; Johnston and Adams, *Ibid.*, **33** (1911), 829.

the flask is thoroughly shaken every half hour for several hours, and allowed to settle. The petroleum ether is decanted through a filter into a Wiley tube, and a second smaller quantity of the solvent is added to the flask, shaken, settled, and filtered, as before. The washing is continued in this way until the sulfur and the filter paper are free of oil, and the Wiley tube contains about 175 cc. of solvent, or enough to fill the tube above the top of the copper roll or coil.



The copper coil is cleaned with dilute nitric acid, washed with water, then with alcohol, dried with ether, and placed in the Wiley tube. The petroleum ether is then boiled until all of the sulfur has been deposited on the copper as copper sulfide.

The solvent is transferred to the tall beaker, filtering if necessary to eliminate the copper sulfide, which will doubtless partially flake off. The coil, the Wiley tube, and the filter are washed with small quantities of petroleum ether, the combined filtrate and washings gently evaporated to dryness at very low heat, and the residue weighed.

NOTE ON THE SOLUBILITY OF BENZIDINE SULFATE IN WATER

By C. S. Bisson and A. W. Christie

UNIVERSITY OF CALIFORNIA, BERKELEY, CALIFORNIA

Received September 9, 1919

One of the methods for the determination of sulfates which has found considerable application consists in precipitation as benzidine sulfate, which may be quantitatively titrated with standard alkali or potassium permanganate. In most of the articles describing such methods, the benzidine sulfate is said to be insoluble in water. A review of the literature