U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
WASHINGTON, D.C. 20234

National Bureau of Standards Certificate

Standard Sample 1052a

Bis(1-phenyl-1,3-butanediono)oxovanadium(IV)

(Standard for Determination of Vanadium in Petroleum Products)

This compound was prepared to insure material that is essentially free from other metals and has suitable solubility, compatibility, and uniformity for use in the preparation of a standard of vanadium in lubricating oils. The compound is certified to one part per hundred of vanadium, and every effort should be made to maintain a uniform procedure by following the directions in this certificate.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Procedure and Results of Chemical Analysis

Vanadium, percent..... 13.1

Vanadium was determined by wet-ashing a 1-g sample (dried for 2 hr over phosphorus pentoxide) with sulfuric and nitric acids, oxidizing the vanadium with nitric acid, and titrating the vanadium with ferrous ammonium sulfate solution. Determinations were also made by direct ignition of a dried 1-g sample, wrapped in filter paper and covered with oxalic acid. The oxide was ignited at 800 °C and weighed as V_2O_5. Analyst, B. B. Bendigo.

Procedure and Results of Spectrographic Analysis

The compound was examined spectrographically for metallic impurities. A 5-mg sample of the compound was excited in a direct-current arc and the photographed spectrum was examined for the characteristic lines of 51 elements. Several impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use. The impurities were each estimated to be less than 0.01 percent. Analyst, Elizabeth K. Hubbard.

STABILITY.—Tests show that standard lubricating-oil solutions of this compound with concentrations of vanadium up to 500 ppm are stable for several weeks when prepared by the directions given below.

COMPATIBILITY.—Lubricating-oil solutions of this compound have been found to be compatible with lubricating-oil solutions of the other compounds in this series. Blends of several different compounds have been prepared by the procedures given in the certificates for the other compounds. (Tests have not been carried out to insure compatibility with the various additives that may be in the oils to be analyzed.)

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF BIS(1-PHENYL-1,3-BUTANEDIONO)OXOVANADIUM(IV)

Transfer approximately 0.4 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 2 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.382 g of this dried compound to a weighed 200-ml flask. (This weight of compound is equivalent to 50 mg of vanadium.) Add 3 ml each of xylene, 6-methyl-2,4-heptanedione, and bis(2-ethylhexyl)amine, and heat the flask on a hot plate, with swirling and without charring, until the compound dissolves. Add to the hot solution 1 ml of 2-ethylhexanoic acid and 80 to 90 ml of lubricating oil, and gently shake the flask to mix the contents. Allow the flask to cool to room temperature and add enough lubricating oil to bring the total weight of the contents of the flask to 100 ± 0.5 g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of vanadium in this solution is 500 ppm.

A. V. ASTIN, *Director.*

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