



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1052b

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

(Standard for Determination of Vanadium in Petroleum Products)

This Standard Reference Material (SRM) was prepared to ensure 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 oil. SRM 1052b consists of 5 g of bis(1-phenyl-1,3-butanediono)oxovanadium(IV) in powder form. The certified vanadium content is given below.

Vanadium 13.01 ± 0.02 wt %*

* wt % = mg/kg $\times 10^{-4}$

The uncertainty shown represents the 95 percent confidence limit of the mean based on fifteen determinations and allows for the effects of possible sources of known error.

Procedure and Results of Chemical Analysis: Vanadium was determined by wet-ashing a 0.5 g sample (dried for 2 h over phosphorus pentoxide) with sulfuric and nitric acids, oxidizing the vanadium with ammonium persulfate in the presence of silver nitrate, and titrating the vanadium with ferrous ammonium sulfate solution. Determinations were also made by direct ignition of a dried sample, wrapped in filter paper and covered with oxalic acid. The oxide was ignited at 800 °C and weighed as V₂O₅.

Procedure and Results of Spectrographic Analysis: The SRM was examined spectrographically for metallic impurities. A 5 mg sample of the SRM was excited in a direct-current arc and the photographed spectrum was examined for the characteristic lines of 51 elements. No significant impurities were found.

STABILITY: Tests show that standard lubricating oil solutions of this compound with concentrations of vanadium up to 500 mg/kg are stable when prepared by the directions given on the reverse of this certificate.

COMPATABILITY: 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 on the certificates for the other compounds. (Tests have not been carried out to ensure compatibility with the various additives that may be in the oils to be analyzed.)

The bis(1-phenyl-1,3-butanediono)oxovanadium(IV) was prepared by Distillation Products Industries of Rochester, NY. Chemical analyses were conducted by B.B. Bendigo, and spectrochemical analyses by V.C. Stewart.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.

Gaithersburg, MD 20899
April 12, 1993
(Revision of certificate dated 3-1-68)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.W. Mears. Revision of this certificate was coordinated through the Standard Reference Materials Program by T.E. Gills.

**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 h. (Tightly close the bottle containing the remainder of the compound). Quickly and accurately transfer 0.384 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 ensure a homogeneous solution. The concentration of vanadium in this solution is 500 $\mu\text{mol/mol}$.