

# Certificate of Analysis

## Standard Reference Material 1080

### Bis(1-phenyl-1,3-butanediono)copper(II)

(Standard for Determination of Copper 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 copper in lubricating oils. The compound is certified to one part per hundred of copper, 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

Copper, percent.....16.5

The standard deviation of this value is 0.08 percent; the 95 percent confidence limit ( $t\sigma$ ) is 0.18 percent. Spectrochemical analysis indicates that interfering elements are not present in sufficient quantities to affect the accuracy of these determinations.

Copper was determined by wet-ashing a 1-g sample (dried for 30 min at 110 °C) with nitric and sulfuric acids, depositing the copper in the resulting solution by electrolysis, and then weighing as metallic copper. Determinations were also made by EDTA titration of the copper solution resulting from wet-ashing procedure [cf. P. B. Sweetser and C. E. Bricker, *Anal Chem* 25, 253 (1953)].

##### 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.

**STABILITY.**—Tests show that standard lubricating-oil solutions of this compound with concentrations of copper 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.)

The bis(1-phenyl-1,3-butanediono)copper(II) was prepared by Distillation Products Industries of Rochester, N. Y. The material was sized and dried by Connie L. Stanley. Chemical analyses were conducted by B. B. Bendigo, and spectrographic analyses by Elizabeth K. Hubbard.

Washington, D. C. 20234  
June 15, 1967

W. Wayne Meinke, Chief  
Office of Standard Reference Materials

(Over)

## DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF BIS(1-PHENYL-1,3-BUTANEDIONO)COPPER(II)

Transfer approximately 0.5 g of this compound from the bottle to a small beaker and dry in an oven at 110 °C for 30 min. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.303 g of this dried salt to a weighed 200-ml flask. (This weight of compound is equivalent to 50 mg of copper.) Add 2 ml of xylene and 4 ml of 2-ethylhexylamine and heat the flask on a hot plate, with swirling and without charring, until a clear solution forms. Add to the hot solution 2 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  $100 \pm 0.5$  g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of copper in this solution is 500 ppm.