

# Certificate of Analysis

## Standard Reference Material 1080a

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

(Standard for Determination of Copper in Petroleum Products)

This compound 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 copper in lubricating oils. The compound is certified to one part per hundred of copper, and every effort should be made to establish a uniform procedure by following the directions in this certificate.

#### CHEMICAL AND SPECTROGRAPHIC ANALYSES

##### Procedure and Results of Chemical Analysis

Copper, percent. . . . . 16.37 ± 0.07

The uncertainty shown represents the 95 percent confidence limit of the mean based on 16 determinations made by two methods and makes allowances for the effects of known sources of possible error.

Copper was determined on samples of bis(1-phenyl-1,3-butanediono)copper(II) (dried for 1 hour at 110 °C) by two methods:

a) Samples of 0.6 g were ignited to destroy the organic material. The residues were dissolved in 20 cm<sup>3</sup> of 6N hydrochloric acid and 20 cm<sup>3</sup> of pyridine, diluted to 1 liter; and the copper determined polarographically by the comparative method.

b) Samples were nondestructively analyzed using the 14 MeV neutron activation technique. The radioactivity induced in these samples and in a standard of known copper content was compared. The determinations made use of the annihilation radiation at 0.51 MeV resulting from the decay of <sup>64</sup>Cu (12.8 h, half life) produced by the <sup>65</sup>Cu(n,2n)<sup>64</sup>Cu nuclear reaction.

##### 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. No impurities at levels greater than 0.01 percent were found. At this level no interference with the intended use is expected.

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 ensure compatibility with the various additives that may be in the oils to be analyzed.)

Washington, D. C. 20234  
February 26, 1969

W. Wayne Meinke, Chief  
Office of Standard Reference Materials

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The bis(1-phenyl-1,3-butanediono)copper(II) was prepared by Distillation Products Industries of Rochester, New York, Chemical analyses were conducted by E. J. Maienthal; activation analyses by S. S. Nargolwalla and J. Suddueth; and, spectrochemical analyses by V. C. Stewart.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of P. D. LaFleur.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

#### DIRECTIONS FOR PREPARING LUBRICATION-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 minutes. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.305 g of this dried salt to a weighed 200-cm<sup>3</sup> flask. (This weight of compound is equivalent to 50 mg of copper.) Add 2 cm<sup>3</sup> of xylene and 4 cm<sup>3</sup> 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 cm<sup>3</sup> of 2-ethylhexanoic acid and 80 to 90 cm<sup>3</sup> 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 copper in this solution is 500 ppm.