U. S. Department of Commerce
C. R. Smith
Secretary

National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Material 1057b

Dibutyltin Bis(2—ethylhexanoate)

(Standard for Determination of Tin in Petroleum Products)

This compound was purified to ensure material that is essentially free from other metals and has suitable solubility, compatibility, and uniformity for use in preparation of a standard of tin in lubricating oils. The compound is certified to one part per hundred of tin, 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

Tin, percent ..... 22.95 ± 0.07

The uncertainty shown represents the 95 percent confidence limit of the mean based on 15 determinations and allowances for the effects of known sources of possible errors.

Tin was determined by wet-ashing a 1-g sample (dried for 2 hours over phosphorus pentoxide) with sulfuric and nitric acids, precipitating the tin with cupferron, and converting the precipitate to SnO_2 by ignition at 1000 °C. A second method followed the same wet-ashing procedure, after which the tin in the resulting solution was reduced with nickel and titrated with a standard solution of potassium iodate.

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 50 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 tin up to 500 ppm are stable for several weeks when prepared by the directions given on the reverse of this certificate.

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 dibutyltin bis(2—ethylhexanoate) was prepared by Distillation Products Industries, Rochester, N.Y. Chemical analyses were conducted by J. R. Baldwin and B. B. Bendigo, 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.

Washington, D. C. 20234
August 5, 1968

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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DIRECTIONS FOR PREPARING LUBRICATING–OIL SOLUTIONS OF DIBUTYLTIN BIS(2–ETHYLHEXANOATE)

Transfer approximately 0.3 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 2 hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.218 g of this dried compound to a weighed 200-ml flask. (This weight of compound is equivalent to 50 mg of tin.) Add 5 ml of xylene and heat the flask on a hot plate, with swirling and without charring, until a clear solution forms. Add to the hot solution 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 tin in this solution is 500 ppm.