

Certificate of Analysis

Standard Reference Material 1059b

Lead Cyclohexanebutyrate

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

Lead, percent..... 36.65 ± 0.06

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

Samples of lead cyclohexanebutyrate weighing about 0.27 g (dried over phosphorus pentoxide for 24 hr) were slowly ashed at 500 °C and the residue dissolved in 10 ml of conc. hydrochloric acid, followed by dilution to one liter. Lead was determined polarographically by the comparative mode of operation using standard solutions prepared from freezing-point standard lead (SRM 49d).

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 principal impurity was silicon, estimated to be 0.05 percent. The metallic impurities other than silicon were estimated to be less than 0.01 percent.

STABILITY.—Tests show that standard lubricating-oil solutions of this compound with concentrations of lead 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 lead cyclohexanebutyrate was prepared by the Sadtler Research Laboratories, Inc., of Philadelphia, Pa. Chemical analyses were conducted by E. June Maienthal, and spectrographic analyses by Elizabeth K. Hubbard.

Washington, D.C. 20234
July 15, 1967

W. Wayne Meinke, Chief
Office of Standard Reference Materials

(Over)

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF LEAD CYCLOHEXANEBUTYRATE

Transfer approximately 0.2 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 24 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.136 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of lead.) Add 3 ml of xylene and 5 ml of 2-ethylhexanoic acid 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 lead in this solution is 500 ppm.