

U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS  
WASHINGTON, D. C.

PROVISIONAL CERTIFICATE  
STANDARD SAMPLE 1059  
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 being certified provisionally to one part per hundred of lead, pending further studies of analytical procedures, of loss of weight on drying, and of changes in weight when exposed to air. 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 - - - - - 37.5

Lead was determined by wet-ashing a 1-g sample (dried for two hours over phosphorus pentoxide) with sulfuric and nitric acids, filtering the lead sulfate through a porcelain frit, and weighing the  $\text{PbSO}_4$  after ignition at  $600^\circ\text{C}$ . Determinations were also made by direct ignition of a 1-g sample, wrapped in filter paper and covered with oxalic acid. The oxide was dissolved in nitric acid, precipitated with chromate, and weighed as  $\text{PbCrO}_4$  after drying at  $140^\circ\text{C}$ . Analyst, B. B. Bendigo.

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 impurities were tin, estimated to be less than 0.1 percent, and iron, estimated to be less than 0.01 percent. Analyst, Elizabeth K. Hubbard.

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 provisional 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.)

#### 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 two hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.133 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  $100 \pm 0.5$  g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of lead in this solution is 500 ppm.



MERRILL B. WALLENSTEIN, Chief  
Division of Chemistry II

October 31, 1960  
Washington, D. C.

USCOMM-NBS-DC