

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

WASHINGTON, D.C. 20234

# National Bureau of Standards Certificate

Standard Sample 1060a

## Lithium Cyclohexanebutyrate

(Standard for Determination of Lithium 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 lithium in lubricating oils. The compound is certified to two parts per hundred of lithium, 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

Lithium, percent ..... 4.1

Lithium was determined by igniting a 0.5-g sample (dried for 24 hr over phosphorus pentoxide) wrapped in filter paper and covered with oxalic acid, treating the resulting residue with sulfuric acid, and weighing the lithium as  $\text{Li}_2\text{SO}_4$  after ignition at 600 °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 54 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. Analyst, Elizabeth K. Hubbard.

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

### DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF LITHIUM CYCLOHEXANEBUTYRATE

Transfer approximately 1.5 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 1.22 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of lithium.) 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 lithium in this solution is 500 ppm.

A. V. ASTIN, *Director*.

WASHINGTON, D.C. 20234  
April 24, 1964.