

# Certificate of Analysis

## Standard Reference Material 1061c

### Magnesium Cyclohexanebutyrate

(Standard for Determination of Magnesium 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 magnesium in lubricating oils.

#### CHEMICAL AND SPECTROGRAPHIC ANALYSES

##### Procedure and Results of Chemical Analysis

Magnesium, percent . . . . . 6.45 ± 0.02

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

Magnesium was determined by wet-ashing a 1-g sample (dried for 48 hr over phosphorus pentoxide) with sulfuric and nitric acids, precipitating twice with diammonium hydrogen phosphate, and weighing the  $Mg_2P_2O_7$  after ignition at 1050 °C.

##### 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 significant impurities were found.

**STABILITY:** Tests show that standard lubricating-oil solutions of this compound with concentrations of magnesium up to 500 ppm are stable for several weeks when prepared by the directions given on the reverse side of this certificate.

The magnesium cyclohexanebutyrate was prepared by the Eastman Kodak Company of Rochester, N. Y. Chemical analyses were conducted by R. K. Bell and spectrographic 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  
October 1, 1981  
(Revision of Certificate  
dated 9/9/70)

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

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.)

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

Transfer approximately 1.0 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 48 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.775 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of magnesium.) 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 ensure a homogeneous solution. The concentration of magnesium in this solution is 500 ppm.