U. S. Department of Commerce
Alexander B. Trowbridge,
Secretary

National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Material 1061b
Magnesium Cyclohexanebutyrate

(Standard for Determination of Magnesium 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 magnesium in lubricating oils.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Procedure and Results of Chemical Analysis

Magnesium, percent.....6.53 ± 0.03

The uncertainty shown represents the 95 percent confidence limit of the mean based on eleven 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 as magnesium ammonium phosphate, and weighing the $Mg_2P_2O_7$ after ignition at 1000 °C. Magnesium was also determined by wet-ashing a 0.5-g sample with sulfuric and nitric acids, followed by titration of the solution of the ash with a standard EDTA solution.

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.

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 magnesium cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, N. Y. Chemical analyses were conducted by B. B. Bendigo and spectrographic analyses by Virginia C. Stewart.

WASHINGTON, D.C. 20234
January 3, 1968

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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 0.765 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 ± 0.5 g. Stopper the flask and shake gently to insure a homogeneous solution. The concentration of magnesium in this solution is 500 ppm.