

U. S. Department of Commerce

C. R. Smith

Secretary

National Bureau of Standards

A. V. Astin, Director



Certificate of Analysis

Standard Reference Material 1051b

Barium Cyclohexanebutyrate

(Standard for Determination of Barium 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 barium in lubricating oils. The compound is certified to one part per hundred of barium, and every effort should be made to maintain a uniform procedure by following the directions in this certificate.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Barium, percent. 28.7±0.1

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

Barium was determined by wet-ashing a 0.5 g sample (dried for 72 hours over phosphorus pentoxide) and the resulting insoluble material removed by filtration, ignited at 900 °C and weighed as barium sulfate.

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, other than strontium, were found.

The strontium content of this material was the only impurity element present that would interfere with the barium determination. Samples, 0.5 g, were wet-ashed, diluted and analyzed by both flame-emission and atomic-absorption spectrometry. A strontium content of 0.05 percent was found. The barium determination given above has been corrected to account for this interference.

STABILITY.—Tests show that standard lubricating-oil solutions of this compound with concentrations of barium up to 500 ppm are stable for several weeks when prepared by the directions given on the reverse 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 barium cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, N.Y. Chemical analysis was conducted by B. B. Bendigo, spectrochemical analysis by V. C. Stewart, and flame-emission and atomic-absorption analysis by T. C. Rains.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. K. Taylor.

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
July 15, 1968

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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DIRECTIONS FOR PREPARING LUBRICATING—OIL SOLUTIONS OF BARIUM
CYCLOHEXANEBUTYRATE

Transfer approximately 0.2 g of this compound to a small beaker and dry at 110 °C for 2 hours (alternatively, the sample may be dried over phosphorus pentoxide for 72 hours.) (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.174 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of barium.) 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 barium in this solution is 500 ppm.