

Certificate of Analysis

Standard Reference Material 1069b

Sodium Cyclohexanebutyrate

(Standard for Determination of Sodium 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 sodium in lubricating oils. The compound is certified to one part per hundred of sodium, 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

Sodium, percent 12.0 ± 0.1

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

Sodium was determined on samples of sodium cyclohexanebutyrate (dried for 48 hours over phosphorus pentoxide) by two methods:

- a) A sample was wrapped in filter paper, covered with oxalic acid and ignited. The residue was either converted to sodium chloride and weighed after ignition at 600 °C or converted to sodium sulfate and weighed after ignition at 750 °C.
- b) A sample was analyzed non-destructively using a thermal neutron activation technique. The radioactivity induced in a sample and a standard of known sodium concentration was compared. Determination was by gamma spectrometry, using the 1.37 MeV gamma peak produced by the $^{23}\text{Na}(n,\gamma)^{24}\text{Na}$ nuclear reaction.

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 55 elements. No impurities were found present in sufficient concentration to interfere with the intended use.

STABILITY—Tests show that standard lubricating-oil solutions of this compound with concentrations of sodium up to 500 ppm are stable for several weeks when prepared by the directions given below.

COMPATIBILITY—Lubrication-oil solutions of this compound have been found to be compatible with lubrication-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.)

The sodium cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, N.Y. Chemical analyses were conducted by B. B. Bendigo, activation analyses by S. S. Nargolwalla, J. Suddueth, E. P. Przybylowicz, and G. W. Smith, and spectrochemical 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
February 13, 1969

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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**DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF SODIUM
CYCLOHEXANEBUTYRATE**

Transfer approximately 0.5 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 48 hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.417 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of sodium.) 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 sodium in this solution is 500 ppm.