



National Institute of Standards & Technology

Certificate of Analysis Standard Reference Material 1069b

Sodium Cyclohexanebutyrate

This Standard Reference Material (SRM) is primarily intended for use in preparing oil solutions of known concentrations of sodium. SRM 1069b is essentially free from other metals and has suitable solubility, compatibility, and uniformity for use with most lubricating oils or petroleum products. The SRM consists of 5 grams of sodium cyclohexanebutyrate that is certified to one part per hundred of sodium. The certified sodium content is given below.

Sodium ----- 12.0 \pm 0.1 weight percent

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) Samples were 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) Samples were analyzed using non-destructive thermal neutron activation analysis. The radioactivity induced in the samples by the ^{23}Na (n, γ) ^{24}Na and in a standard of known sodium concentration was compared by gamma spectrometry, using the 1.37 MeV gamma peak of ^{24}Na .

The SRM was also 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: 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 on the back page.

COMPATIBILITY: Lubricating-oil solutions of this compound have been found to be compatible with lubricating-oil solutions of the other metallo-organic SRMs in this series. However, tests have not been carried out to ensure compatibility with the various additives that are commonly used in many petroleum products.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certified value or any technical data presented in this certificate.

Gaithersburg, MD 20899
June 10, 1991
(Revision of Certificate
dated 2-13-69)

William P. Reed, Chief
Standard Reference Materials Program

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The sodium cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, NY. 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 Standard Reference Materials Program by T.W. Mears.

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 ensure a homogeneous solution. The concentration of sodium in this solution is 500 ppm.