National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1051b

Barium Cyclohexanebutyrate

(Standard for Determination of Barium in Petroleum Products)

This Standard Reference Material (SRM) is primarily intended for use in preparing standard oil solutions containing barium. SRM 1051b 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 barium cyclohexanebutyrate that is certified to one part per hundred of barium. The certified barium content is given below.

Barium ---------- 28.7 ± 0.1 weight percent

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); the resulting insoluble material was 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 side of this certificate.

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 insure 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 July 15, 1968)

William P. Reed, Chief
Standard Reference Materials Program

(over)

The barium cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, NY. 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 Standard Reference Materials Program by T.E. Gills.

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.