



National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1059c

Lead Cyclohexanebutyrate

This Standard Reference Material (SRM) is certified for total lead content and is intended primarily for use in preparing solutions of known lead content in lubricating oils. The certified lead content (weight percent) of this SRM based upon chemical analyses is:

Lead, percent 37.5 ± 0.3

The uncertainty shown represents a statistical tolerance interval at the 95 percent confidence level to cover 95 percent of all subsamples of this material. As such, the uncertainty includes allowances for measurement uncertainty and material inhomogeneity.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

A. Isotope Dilution Mass Spectrometry

Five bottles of SRM 1059c and one of 1059b (control) were analyzed. Samples (0.25 g) were dried over $Mg(ClO_4)_2$ for 24 h, and wet-ashed using HNO_3 and $HClO_4$. Appropriate dilutions were made and aliquots containing about 5 μg Pb were spiked with ^{206}Pb for mass spectrometric analyses. Samples were analyzed using thermal ionization mass spectrometry with a single filament rhenium ion source. All analyses were corrected for systematic bias by comparison with multiple analyses of SRM 981, natural lead isotopic standard.

B. Inductively Coupled Plasma (ICP)

Four bottles of SRM 1059c and one of 1059b (control) were analyzed. Two 0.25-g samples from each bottle were dried over P_2O_5 for 24 h, wet-ashed with HNO_3 and $HClO_4$, and analyzed using ICP instrumentation that was calibrated with aqueous standards. Spike recoveries were checked in each dissolved sample.

C. Spectrographic Analysis

This material 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 characteristic lines of 35 elements.

Several impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use. The following values (in percent) are NOT certified, but are given for information only: Si (0.05), Na (0.01), Bi (0.005), Fe (0.002) and Cu and Mg (0.001). All other impurities are below the limits of detection.

This lot of lead cyclohexanebutyrate was purchased from Alfa Products, Morton Thiokol, Inc., of Danvers, MA.

Gaithersburg, MD 20899
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Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Statistical analysis of the data was performed by K.R. Eberhardt, NBS Statistical Engineering Division.

The analytical measurements leading to certification of this material were performed in the NBS Inorganic Analytical Research Division. Mass spectrometric measurements were made by J.W. Gramlich and E. Beary. ICP measurements were made by L. J. Wood, and R.L. Watters, Jr. Spectrographic analysis was made by J.A. Norris.

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 R.W. Seward.

HOMOGENEITY:

This SRM contains small portions of clumped material that yield slightly higher lead concentrations than the powdered material. The certified lead value and uncertainty represent a sampling scheme that includes both size fractions of the material. Care should be taken to ensure that samples used include both clumped and powdered material. The minimum sample size to be used is 0.25 gram.

STABILITY:

Tests have shown that lubricating-oil solutions of this compound with concentration of 500 ppm are stable for several weeks when prepared as directed below.

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF LEAD CYCLOHEXANEBUTYRATE

Transfer approximately 0.3 g of this material from the bottle to a small beaker and dry over magnesium perchlorate in a desiccator for 24 hours. (Tightly close the bottle containing the remainder of the material.) Quickly and accurately transfer 0.266 g of the dried compound to a weighed 250-mL flask. (This weight of compound is equivalent to 100 mg of lead.) Add 6 mL of xylene and 10 mL of 2-ethylhexanoic acid and heat the flask on a hot plate, while swirling and preventing charring, until a clear solution forms. Add 150 to 180 mL of lubricating oil to the hot solution and gently shake to mix. Allow flask to cool to room temperature and add enough lubricating oil to dilute the contents of the flask by weight to 200 ± 0.5 g. Stopper flask and shake gently to insure a homogeneous solution. The concentration of lead in this solution is approximately 500 ppm.