U. S. Department of Commerce
Maurice H. Stans
Secretary

National Bureau of Standards
M. Branscomb, Director

Certificate of Analysis

STANDARD REFERENCE MATERIAL 1053a

Cadmium Cyclohexanebutyrate

(Standard for Determination of Cadmium in Petroleum Products)

This compound was prepared to ensure material that is essentially free from other metals and has suitable solubility, compatibility, and uniformity, for use in the preparation of a standard of cadmium in lubricating oils. The compound is certified to one part per hundred of cadmium, 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

Cadmium, percent . . . . . 24.8 ± 0.2

The uncertainty shown represents the 95 percent confidence limit of a single measurement based on 11 determinations and allowances for the effects of known sources of possible errors.

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

A. Polarographic Analysis

Samples of approximately 0.4 g were ignited slowly to destroy organic material. The residue was dissolved in hydrochloric acid and cadmium determined polarographically.

B. Activation Analysis

Samples were non-destructively analyzed using the 3-MeV neutron activation technique. The radioactivity induced in these samples and in standards of known cadmium content was compared. The determinations were made using the gamma radiations from the reactions $^{110}Cd(n,\gamma)\,^{111m}Cd$ and $^{111}Cd(n,n'\gamma)\,^{111m}Cd$.

Procedure and Results of Spectrographic Analysis

The material was examined spectrographically for metallic impurities. A 5-mg sample was excited in a direct-current arc and the photographed spectrum was examined for the characteristic lines of 51 elements. Several impurities were found, but none are present in sufficient concentration to interfere with the intended use. The sodium concentration is 0.1 percent.

The cadmium cyclohexanebutyrate was prepared by Wamack Industries, Inc. of West Chester, Pennsylvania. Polarographic analyses were conducted by E. J. Maienthal, activation analyses by S. S. Nargolwalla and J. E. Suddueth, and spectrochemical analysis 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
January 23, 1970

J. Paul Cali, Acting Chief
Office of Standard Reference Materials

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DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF CADMIUM CYCLOHEXANEBUTYRATE

Transfer approximately 0.3 g of sample 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 0.202 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of cadmium.) Add 2 ml of xylene and 4 ml of 2-ethylhexylamine and heat the flask on a hot plate, with swirling and without charring, until a clear solution forms. Add to the hot solution 2 ml of 2-ethylhexanoic acid and 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 cadmium in this solution is 500 ppm.

STABILITY: Tests show that standard lubricating-oil solutions of this compound with concentrations of cadmium 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 are compatible with lubricating-oil solutions of the other compounds in this series. Blends of several different compounds have been prepared by use of the procedures given in the certificates for the other compounds. (Tests have not been made to ensure compatibility with the various additives that may be in the oils to be analyzed.)