U. S. Department of Commerce
C. R. Smith
Secretary

National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Material 1055b

Cobalt Cyclohexanebutyrate

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

Cobalt, percent..... 14.8 ± 0.1

The uncertainty shown represents the 95 percent confidence limit of the mean based on 28 determinations made by two methods and on allowances for the effects of known sources of possible errors.

Cobalt was determined on samples of cobalt 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 at 800 °C. The resulting oxide was dissolved in hydrochloric acid, electrodeposited as cobalt from an ammoniacal solution and weighed as the metal. The residue cobalt in the electrolyte was determined by the nitroso–R–salt photometric method.

b. A sample was non-destructively analyzed by the 14-MeV activation technique. The 1.81 MeV gamma peak from the decay of 2.58 h ^{56}Mn produced by the nuclear reaction ^{59}Co(n,α)^{56}Mn was counted.

Procedure and Results of Spectrographic Analysis

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. Several impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use. The metallic impurities were estimated to be less than 0.01 percent.

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

COMPATIBILITY. Lubricating-oil solutions of this compound have been found to be compatible with lubricating-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 cobalt 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
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W. Wayne Meinke, Chief
Office of Standard Reference Materials

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