

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1078b

Tris(1-phenyl-1,3-butanediono)chromium(III)

(Standard for Determination of Chromium in Petroleum Products)

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

Chromium, percent 9.6 ± 0.1

The uncertainty expressed for the above result is twice the standard deviation for a single measurement based on 14 determinations.

Chromium was determined by two methods (after drying the sample at 110 °C for 1 hour) as follows:

Samples of the Standard Reference Material were partially digested using sulfuric and nitric acids. After fuming off the nitric acid, digestion was completed using sulfuric and perchloric acids. The chromium was oxidized using ammonium persulfate in the presence of silver ion and then determined by potentiometric titration with ferrous ion.

Samples were analyzed by non-destructive neutron activation techniques using chromium trioxide as the reference standard.

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 50 elements. Several trace impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use.

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
July 25, 1972

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

STABILITY: Tests show that standard lubricating-oil solutions of this compound with concentrations of chromium 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 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 insure compatibility with the various additives that may be in the oils to be analyzed.)

The tris(1-phenyl-1,3-butanediono)chromium(III) was prepared by Kings Laboratory of Columbia, South Carolina. Chemical analyses were conducted by W. P. Schmidt, activation analysis by P. D. LaFleur, and spectrographic analysis by V. C. Stewart.

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF TRIS(1-PHENYL-1,3-BUTANEDIONO)CHROMIUM(III)

Transfer approximately 0.6 g of this compound from the bottle to a small beaker and dry in an oven at 110 °C for 1 hour. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.521 g of this compound to a weighed 200-ml flask. (This weight of compound is equivalent to 50 mg of chromium.) Add 3 ml of 2-ethylhexanoic acid and 3 ml of 6-methyl-2,4-heptanedione, 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 chromium in this solution is 500 ppm.