

Certificate of Analysis

Standard Reference Material 1065b

Nickel Cyclohexanebutyrate

(Standard for Determination of Nickel 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 nickel in lubricating oils.

CHEMICAL AND SPECTROGRAPHIC ANALYSES

Procedure and Results of Chemical Analysis

Nickel, percent 13.89±0.02

The uncertainty shown represents the 95 percent confidence limit of the mean based on fifteen determinations and allowances for the effects of possible sources of known error.

Nickel was determined by wet-ashing a 1-g sample (dried for 48 hr over phosphorus pentoxide) with sulfuric and nitric acids, precipitating with dimethylglyoxime, and weighing the nickel dimethylglyoxime precipitate after drying at 150 °C. Determinations were also made using a solution prepared by wet-ashing a 0.1 g sample with nitric acid, fuming with perchloric acid, and dissolving the residue in water. The pH of an aliquot of this solution was adjusted to 13, treated with alcoholic dimethylglyoxime, and the nickel determined spectrophotometrically.

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. No significant impurities were found.

STABILITY.—Tests show that standard lubricating-oil solutions of this compound with concentrations of nickel 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 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 nickel cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, N. Y. Chemical analyses were conducted by B. B. Bendigo, spectrophotometric analyses by E. R. Deardorff, and spectrographic analyses by Virginia C. Stewart.

WASHINGTON, D. C. 20234
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W. Wayne Meinke, Chief
Office of Standard Reference Materials

(Over)

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF NICKEL CYCLOHEXANEBUTYRATE

Transfer approximately 0.40 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 48 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.360 g of this dried salt to a weighed 200-ml flask. (This weight of salt is equivalent to 50 mg of nickel.) 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 nickel in this solution is 500 ppm.