

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
WASHINGTON, D.C. 20234

National Bureau of Standards Certificate

Standard Sample 1065a

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. The compound is certified to one part per hundred of nickel, 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

Nickel, percent ----- 16.8

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. Analyst, B. B. Bendigo.

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 principal impurities were copper and sodium, each less than 0.01 percent. Analyst, Elizabeth K. Hubbard.

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 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 insure compatibility with the various additives that may be in the oils to be analyzed.)

DIRECTIONS FOR PREPARING LUBRICATING-OIL SOLUTIONS OF NICKEL CYCLOHEXANEBUTYRATE

Transfer approximately 0.35 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 0.298 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.

A. V. ASTIN, *Director.*

WASHINGTON, D.C. 20234
April 24, 1964.