

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

WASHINGTON, D.C. 20234

National Bureau of Standards Certificate

Standard Sample 1066

Octaphenylcyclotetrasiloxane

(Standard for Determination of Silicon in Petroleum Products)

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

Silicon, percent 14.1

Silicon was determined by wet-ashing a 1-g sample (dried for 2 hr over phosphorus pentoxide) with sulfuric and nitric acids, dehydrating the silica twice, igniting the silica at 1100 °C, and volatilizing the silicon with hydrofluoric acid. 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 50 elements. Several impurities were found, but none is considered to be present in sufficient concentration to interfere with the intended use. The impurities were each estimated to be less than 0.01 percent. Analyst, Elizabeth K. Hubbard.

STABILITY.—Tests show that standard lubricating-oil solutions of this compound with concentrations of silicon up to 200 ppm are stable at 75 °C 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 OCTAPHENYLCYCLOTETRASILOXANE

Transfer approximately 0.2 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 2 hr. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.142 g of this compound to a weighed 200-ml flask. (This weight of compound is equivalent to 20 mg of silicon.) Add 4 g of xylene and heat the flask on a hot plate, with swirling and without charring, until the solid dissolves. In a separate flask, heat 95 g of lubricating oil to 75 °C, and carefully pour this hot oil into the silicon solution. Allow the solution to cool to room temperature and add enough cool lubricating oil to bring the total weight of the contents of the flask to 100 ± 0.5 g. Reheat the flask immediately to 75 °C under reflux and keep the solution under reflux at this temperature during use. The concentration of silicon in this solution is 200 ppm.

A. V. ASTIN, *Director*.

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April 24, 1964.