

# Certificate of Analysis

## Standard Reference Material 1066a Octaphenylcyclotetrasiloxane

(Standard for Determination of Silicon 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 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. . . . . 14.14 ± 0.07

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

Silicon was determined on samples of octaphenylcyclotetrasiloxane (dried for two hours over phosphorus pentoxide) by two methods:

a) A sample was wet-ashed with nitric and sulfuric acids. The silica formed was doubly dehydrated, ignited at 1200 °C, and weighed. This was followed by volatilization of the silica with hydrofluoric acid.

b) The radioactivity induced in the sample and in a standard of known silicon concentration were compared. Determination was by gamma spectrometry, using the 1.78 MeV gamma peak of  $^{28}\text{Al}$  produced by the reaction  $^{28}\text{Si}(n,p)^{28}\text{Al}$ .

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.

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

The octaphenylcyclotetrasiloxane was prepared by the Silicone Products Department of the General Electric Company and obtained through the Eastman Kodak Company of Rochester, N. Y. Chemical analyses were conducted by B. B. Bendigo, activation analyses by S. S. Nargolwalla and J. Suddueth, 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.

#### DIRECTIONS FOR PREPARING LUBRICATION-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 hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.141 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  $100 \pm 0.5$  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.