



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 1066a

#### Octaphenylcyclotetrasiloxane

This Standard Reference Material (SRM) is primarily intended for use in preparing oil solutions of known concentrations of silicon. SRM 1066a is essentially free from other metals and has suitable solubility, compatibility, and uniformity for use with most lubricating oils or petroleum products. The SRM consists of 5 grams of octaphenylcyclotetrasiloxane that is certified to at least one part per hundred of silicon. The certified silicon content is given below.

Silicon ----- 14.14  $\pm$  0.07 weight percent

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) Samples weighing 1-gm each were wet-ashed with nitric and sulfuric acids. The silica formed was double dehydrated, ignited at 1200 °C, and weighed. This was followed by volatilization of the silica with hydrofluoric acid.

(b) Samples were analyzed using 14 MeV neutron activation analysis. The radioactivity induced in the samples by the  $^{28}\text{Si}$  (n,p)  $^{28}\text{Al}$  reaction and in a standard of known silicon concentration were compared by gamma spectrometry, using the 1.78 MeV gamma peak of  $^{28}\text{Al}$ .

SRM 1066a was also examined spectrographically for metallic impurities. A 5-mg sample of the SRM 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 total impurities were estimated to be less than 0.01 percent by weight.

**STABILITY:** Lubricating-oil solutions of this SRM with concentrations of silicon up to 200 ppm are stable at 75 °C when prepared by the directions given on the back page.

**COMPATABILITY:** Lubricating-oil solutions of this SRM have been found to be compatible with lubricating-oil solutions of the other metallo-organic SRMs in this series. However, tests have not been carried out to ensure compatibility with the various additives that are commonly used in many petroleum products.

*This Certificate of Analysis has undergone editorial revisions to reflect program and organization changes at NIST and the Department of Commerce. No attempt was made to reevaluate the certified value or any technical data presented in this certificate.*

Gaithersburg, MD 20899  
June 10, 1991  
(Revision of Certificate  
dated 4-23-69)

William P. Reed, Chief  
Standard Reference Materials Program

(over)

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 Standard Reference Materials Program 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 70 °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.