

POLYDIMETHYLSILOXANE

Prepared at the 37th JECFA (1990), published in FNP 52 (1992) superseding specifications prepared at the 29th JECFA (1985), published in FNP 34 (1986). Metals and arsenic specifications revised at the 61st JECFA (2003). An ADI of 0-1.5 mg/kg bw was established at the 23rd JECFA (1979)

SYNONYMS

Poly(dimethylsiloxane), dimethylpolysiloxane, dimethylsilicone fluid, dimethylsilicone oil; INS No. 900

DEFINITION

Consists of fully methylated linear siloxane polymers containing repeating units of the formula $(\text{CH}_3)_2\text{SiO}$, with trimethylsiloxy end-blocking units of the formula $(\text{CH}_3)_3\text{SiO}-$

The article of commerce used as an antifoaming agent can be further specified as to total silicon

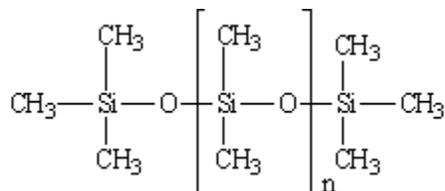
Chemical names

Simethicone (CAS name)

C.A.S. number

8050-81-5

Structural formula



The average value for n is 90 to 410

Formula weight

6,800 to 30,000 (average and approximate)

Assay

Silicon content not less than 37.3% and not more than 38.5% of the total

DESCRIPTION

Clear, colourless, viscous liquid. Polydimethylsiloxane is frequently used in commerce as such, as a liquid containing 4-5% silica gel, and as an aqueous emulsion formulation containing, in addition to silica gel, emulsifiers and preservatives. The pure substance described here can be isolated by centrifuging from the silica gel-containing liquid at about 20,000 rpm.

FUNCTIONAL USES Antifoaming agent, anticaking agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Insoluble in water and in ethanol; soluble in carbon tetrachloride, benzene, chloroform, diethyl ether, toluene and other organic solvents

Specific gravity (Vol. 4)

d (25, 25): 0.964 - 0.977

Refractive index (Vol. 4) $n(25, D): 1.400 - 1.405$

Infrared absorption The infrared spectrum of the sample corresponds with the reference infrared spectrum below. Prepare two solutions containing: (1) 10% of sample in carbon tetrachloride and (2) 2% of sample in carbon disulfide. Obtain the infrared spectrum for the sample using the carbon disulfide solution (2) from 1300 to 650 cm^{-1} . The solvent in the reference cell is also changed at the appropriate wave numbers to correspond to the sample solution.

PURITY

Loss on drying (Vol. 4) Not more than 0.5% (150°, 4 h)

Viscosity 100 - 1500 cSt at 25°
See description under TESTS

Lead (Vol. 4) Not more than 1 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

TESTS

PURITY TESTS

Viscosity

Apparatus:

The Ubbelohde suspended level viscometer, shown in the accompanying diagram, is preferred for the determination of the viscosity.

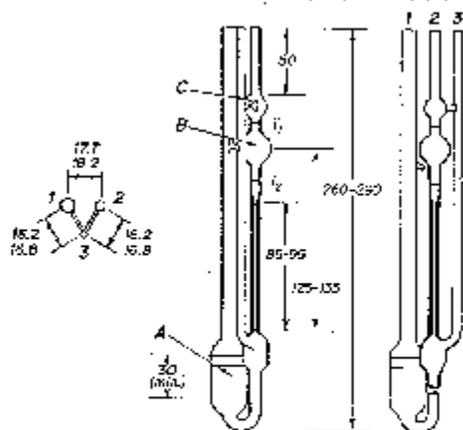


Figure: Ubbelohde Viscometer for Polydimethylsiloxane (all dimensions are in millimeters.)

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For use in the range of 100 to 1.500 centistokes, a No. 3 size viscometer,

having a capillary diameter of 2.00 ± 0.04 mm, is required. The viscometer should be fitted with holders which satisfy the dimensional positions of the separate tubes as shown in the diagram, and which hold the viscometer vertical. Filling lines in bulb A indicate the minimum and maximum volumes of liquid to be used for convenient operation. The volume of bulb B is approximately 5 ml.

Calibration of the Viscometer

Determine the viscosity constant C for each viscometer by using an oil of known viscosity. Charge the viscometer by tilting the instrument about 30 degrees from the vertical, with bulb A below the capillary, and then introduce enough of the sample into tube 1 to bring the level up to the lower filling line.

The level should not be above the upper filling line when the viscometer is returned to the vertical position and the sample has drained from tube 1. Charge the viscometer in such a manner that the U-tube at the bottom fills completely without trapping air.

After the viscometer has been in a constant-temperature bath long enough for the sample to reach temperature equilibrium, place a finger over tube 3 and apply suction to tube 2 until the liquid reaches the center of bulb C. Remove suction from tube 2, then remove the finger from tube 3 and place it over tube 2 until the sample drops away from the lower end of the capillary. Remove the finger from tube 2, and measure the time, to the nearest 0.1 sec required for the meniscus to pass from the first time mark (T_1) to the second (T_2). In order to obtain accurate results within a reasonable time, the apparatus should be adjusted to give an elapsed time of from 80 to 100 sec.

Calculate the viscometer constant C by the equation, $C = cs/t_1$, in which cs is the viscosity, in centistokes, and t_1 is the efflux time, in sec for the standard liquid.

METHOD OF ASSAY

Principle

Silicon in the sample is converted to a soluble form by fusion with sodium peroxide. Soluble silicon is measured in the percent range as total silicon by atomic absorption spectrometry.

Apparatus

- Apparatus for fusion: a Parr-type fusion cup and nickel beaker, or equivalent (avoid use of glassware during fusion and solubilization).
- Instrument: atomic absorption spectrometer with silicon hollow cathode lamp; nitrous oxide - acetylene burner, or equivalent.

Reagents

- Sodium peroxide, glacial acetic acid, silica (of known purity for use as standard).

Procedure

NOTE: Normal safe laboratory practices for Parr-type bomb fusion should be followed.

Equivalent fusions must be performed on sample(s), reagent blank(s) and silica standards for each series of samples. For each sample weigh a portion (*W*) not to exceed 0.3 g into a Parr-type fusion cup (use gelatin capsules for liquid samples). Add 15.0±0.5 g sodium peroxide. Assemble the fusion apparatus and place it in a protective ignition rack. Fill the cavity above the cap with water and keep it full during ignition to prevent the gasket from melting. Heat the bottom of the cup with a blast lamp until the cup is cherry red about 100 mm up from the bottom within 90 sec. Quench the apparatus in ice water and disassemble the apparatus. Place the cup in a 500 ml nickel beaker containing 150 to 200 ml of distilled water. Rinse any material adhering to the inside of the assembly cap into the beaker with distilled water. Cover the beaker with a nickel lid. When the solution is complete and cooled, remove the cup from the beaker and rinse with distilled water into the beaker. Add 55.0 ml of reagent grade glacial acetic acid to the beaker. Cool the solution to room temperature and transfer to a 500 ml volumetric flask. Dilute to volume with distilled water. The solution should contain about 100 µg silicon/ml for a sample weight of about 0.13 g. This method performs best if the silicon concentration of the final analysis solution is 1 to 200 µg/ml. Prepare a series of standards, using the same fusion technique, which bracket the sample.

Adjust the spectrophotometer according to operating instructions to obtain optimum analysis conditions for: maximum lamp output when monochromator is at 251.6 nm, fuel and oxidant flow rate to burner (or equivalent procedures for other vaporizing techniques). Adjust zero absorbance while aspirating the solvent blank used to dilute the samples. Measure the absorbance of sample(s), reagent blank and standards. Estimate the concentration (*C*) of silicon in the sample solution from the standards, correcting for the reagent blank. Calculate percent total silicon in the sample by the formula

$$\% \text{ silicon} = 0.05 \times \frac{C}{W}$$

where

C = the silicon concentration of the sample solution (µg/ml)

W = the weight of sample taken (g)

Infrared spectrum

Polydimethylsiloxane

