POLYDIMETHYLSILOXANE

Prepared at the 69th JECFA (2008), published in FAO JECFA Monographs 5 (2008), superseding specifications prepared at the 37th JECFA (1990), published in the Combined Compendium of Food Additive Specifications, FAO JECFA Monographs 1 (2005). A temporary ADI of 0-0.8 mg/kg bw was established at the 69th JECFA (2008).

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

DEFINITION
Polydimethylsiloxane consists of fully methylated linear siloxane polymers containing repeating units of the formula [$(CH_3)_2SiO$] with trimethylsiloxy end-blocking units of the formula $(CH_3)_3SiO$-. The additive is produced by hydrolysis of a mixture of dimethyldichlorosilane and a small quantity of trimethylchlorosilane. The average molecular weights of the linear polymers range from approximately 6,800 to 30,000.

(NOTE: In commerce, polydimethylsiloxane is frequently used in preparations usually containing silica gel. The pure substance described in this monograph can be isolated from silica gel-containing liquids by centrifuging at about 20,000 rpm. Before testing the Polydimethylsiloxane for *Identification, Refractive index, Specific gravity,* and *Viscosity,* any silica gel present must be removed by centrifugation.)

(NOTE: This monograph does not apply to aqueous formulations of Polydimethylsiloxane containing emulsifying agents and preservatives, in addition to silica gel.)
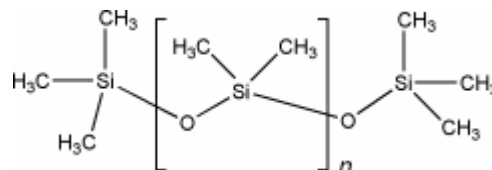
Chemical names
α-(Trimethylsilyl)-ω-methylpoly(oxy(dimethylsilylene))

C.A.S. number
9006-65-9

Structural formula

n ranges from 90 to 410

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

DESCRIPTION
Clear, colourless, viscous liquid.

FUNCTIONAL USES   Antifoaming agent, anticaking agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)   Insoluble in water and in ethanol; soluble in most aliphatic and
                             aromatic hydrocarbon 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 absorption spectrum of a liquid film of the sample
                             between two sodium chloride plates exhibits relative maxima at the
                             same wavelengths as those of a similar preparation of USP
                             Dimethylpolysiloxane Reference Standard (available through
                             http://www.usp.org/referenceStandards/catalog.html or by mail to USP
                             12601 Twinbrook Pkwy, Rockville, MD 20852 USA).

PURITY

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

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

Lead (Vol. 4)   Not more than 1 mg/kg
                             Determine using an AAS/ICP-AES technique appropriate to the
                             specified level. The selection of sample size and method of sample
                             preparation may be based on principles of methods described in
                             Volume 4 (under "General Methods, Metallic Impurities").

TESTS

PURITY TESTS

Viscosity   The Ubbelohde suspended level viscometer, shown in the
                             accompanying diagram, is preferred for the determination of the
                             viscosity.
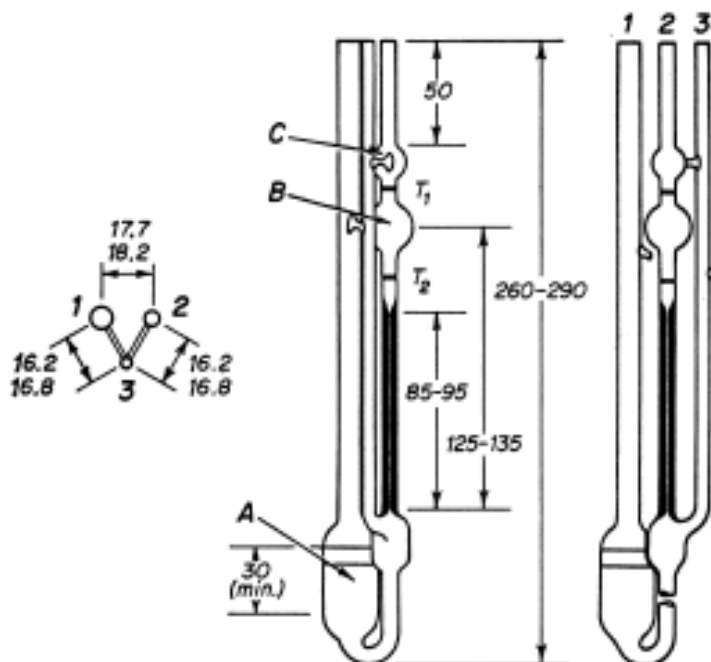
(Dimensions in mm)

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 that satisfy the dimensional positions of the separate tubes as shown in the diagram, and that 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, *k*, for each viscometer by using an oil of known viscosity. [NOTE: Choose an oil with a viscosity as close as possible to that of the sample to be tested.] 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 (*T₁*) to the second (*T₂*). 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 k by the equation

$$k = v/t_1,$$

in which v is the viscosity, in centistokes, and t_1 is the efflux time, in sec, for the standard liquid.

Viscosity determination of Polydimethylsiloxane
Charge the viscometer with the sample in the same manner as described for the calibration procedure; determine the efflux time, t_2; and calculate the viscosity of the sample, v_s, by the equation

$$v_s = kt_2.$$

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 spectrophotometry.

Apparatus
- Fusion apparatus: Parr-type fusion cup; 500-ml nickel beaker; and nickel lid for beaker - or equivalent (avoid use of glassware during fusion and solubilization).
- Instrument: atomic absorption spectrophotometer 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
[CAUTION: 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 of 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 becomes 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 the 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 the nickel lid. When dissolution is complete and the solution has cooled, remove the cup from the

reagent grade glacial acetic acid to the beaker. Cool the solution to room temperature and transfer it 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 that brackets the sample.

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

$$\%\text{Silicon} = 0.05 \times C/W$$

where
C is the silicon concentration of the sample solution (µg/ml)
W is the weight of sample taken (g)