Designing Hydrogen Silsesquioxane: Control Over Particle Size, Shelf-Life, and Sensitivity. Part II.

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Hydrogen silsesquioxane(s) (HSQ) are inorganic compounds containing hydrogen, silicon, and oxygen in a ratio of 2:2:3, respectively. HSQ is a popular negative resist. Applied Quantum Materials Inc. (AQM) is a supplier of both solid- and solution-phase HSQ. The solid possesses an indefinite shelf-life when stored appropriately. However, the shelf-life diminishes when dissolved in organic solvents due to the absorption of water vapour. Previously, we defined the HSQ molecular weight variants and presented our initial shelf-life investigation using the organic solvent methyl isobutyl ketone (MIBK). This presentation will provide an update and an analysis of alternative organic solvents that increase the shelf-life of solution-phase HSQ.

Different light scattering methods were used to investigate AQM's standard HSQ (H-SiOx-15) dissolved in various organic solvents. At 5 hours, the mean particle size of the HSQ is between 2.7 and 3.2 nm. The solvation diameter, which includes the HSQ's particle diameter, varies from solvent to solvent. The number-weighted particle diameters and solvation diameters are summarized in Table 1. The solutions are stored in sealed HDPE bottles at room temperature, and on a lab bench. Sampling and analysis of the solutions also occurred at 2, 4, and 6 months. The mean particle diameter for HSQ in MIBK increased to 7.59 (1.55) nm over the 6 months. Incidentally, we have found other solvents that resulted in negligible change to the HSQ particle diameter over the same period. These alternative solvents, their properties, spin curves, and acquired electron beam lithography will be discussed.

Table 1. Particle size distributions for H-SiOx-15 in different solvents at 5 hours.

Solvent	Functional Group	Peak [nm] ^a	Solvation Diameter [nm] ^a	Distribution $[\%]^b$
MIBK	Ketone	3.23 (0.26)	39.7 (6.0)	13.4
Solvent-A	Aromatic	2.71 (0.22)	12.5 (5.3)	12.8
Solvent-B	Ester	2.85 (0.22)	62.5 (8.0)	13.4
Solvent-C	Alkane	3.21 (0.28)	24.2 (3.5)	13.5
Solvent-D	Aromatic	2.88 (0.29)	17.1 (3.3)	13.5

HSQ was dissolved in an appropriate solvent to be a consistent weight percentage concentration. The HDPE bottle was sealed, stored at room temperature, and the solution analyzed at 5 hr. a The particle and solvation diameters reported (standard deviation in brackets) was an average of several runs (\geq 5 runs). b The distribution was that at the peak particle diameter.