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# Proton-conducting Electrolyte Film of Double-decker-shaped Polyhedral Silsesquioxane Containing Covalently Bonded Phosphonic Acid Groups

*Asuman C. Kucuk, Jun Matsui*, and Tokuji Miyashita*

Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Tohoku University, 2-1-1

Katahira, Aoba-ku, Sendai 980-8577, Japan

Author to whom correspondence should be addressed

e-mail: jun_m@tagen.tohoku.ac.jp

**Instrumental Analysis.**

FT-IR spectra were obtained using an FT-IR spectrometer (FT/IR4200; Jasco Corp.). The IR spectra of the amphiphilic DDSQs were recorded between 4000 and 900 $cm^{-1}$ with resolution of 1 $cm^{-1}$ under a continuous nitrogen purge. Then $^{1}H$ and $^{31}P$ NMR measurements were conducted using a spectrometer (JNM-AL 400; JEOL).

For MALDI-TOF MS (Bruker Daltonics) Analysis, the matrix α-cyano-4-hydroxycinnamic acid was dissolved in THF (10 mg ml-1), and mixed with 0.2 μL of sample solution (100 μmol in THF) in 1:1 v/v ratio. The resultant solution was deposited on a stainless steel sample plate and dried. The measurement was done in linear mode with a N2 laser (337 nm), and positive mode with an accelerating voltage 20 kV.

X-ray photoelectron spectrometer (XPS) (PHI 5600; Perkin-Elmer Inc.) was used to determine the element composition of the compounds. All binding energies in XPS measurements were referenced to the C 1s peak for neutral carbon, which was assigned as a value of 285.0 eV.

**Experimental Results**

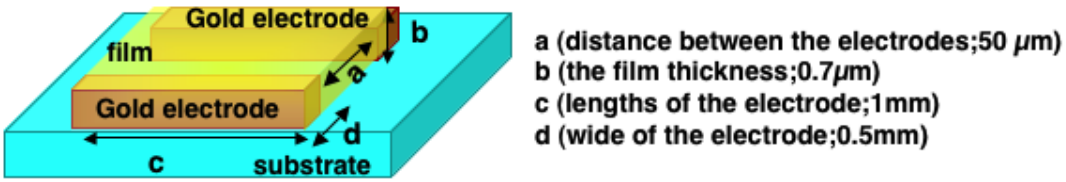


**Figure S1** Glass substrate, where gold electrode is patterned, used for proton conductivity measurement for drop cast film of PHOS-DDSQ.

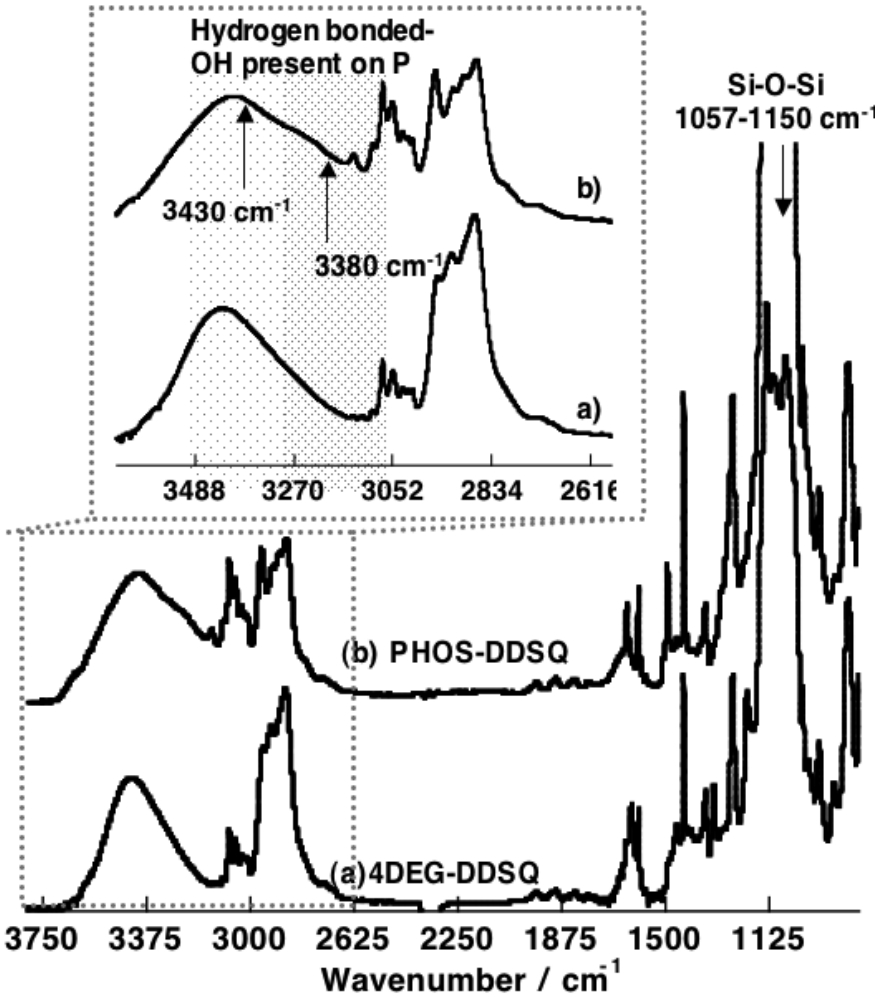


**Figure S2.** FT-IR spectra recorded at room temperature in the 800-3800 $cm^{-1}$ region for (a) 4DEG-DDSQ, (b) PHOS-DDSQ.

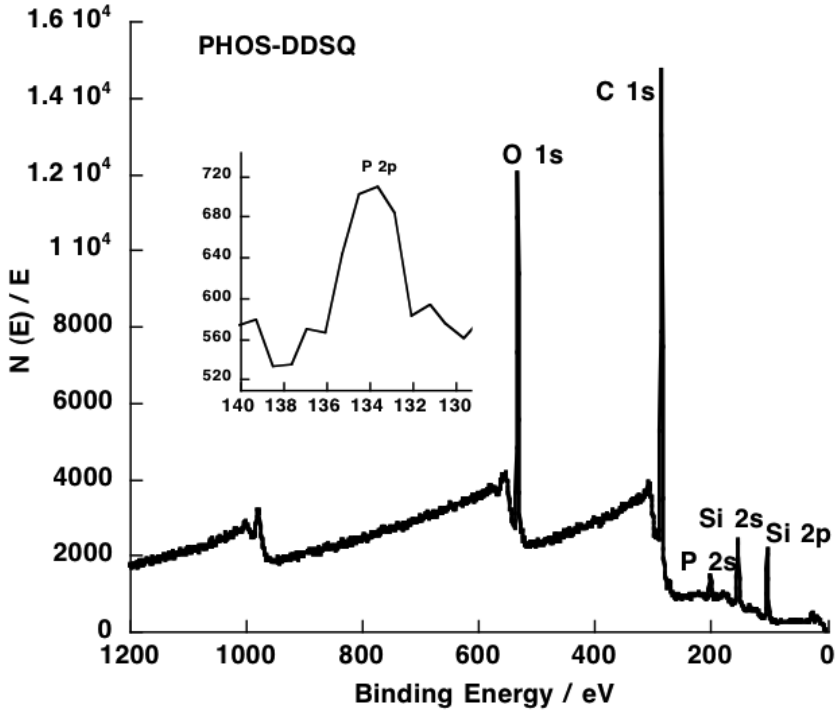


**Figure S3.** XPS spectrum of PHOS-DDSQ.

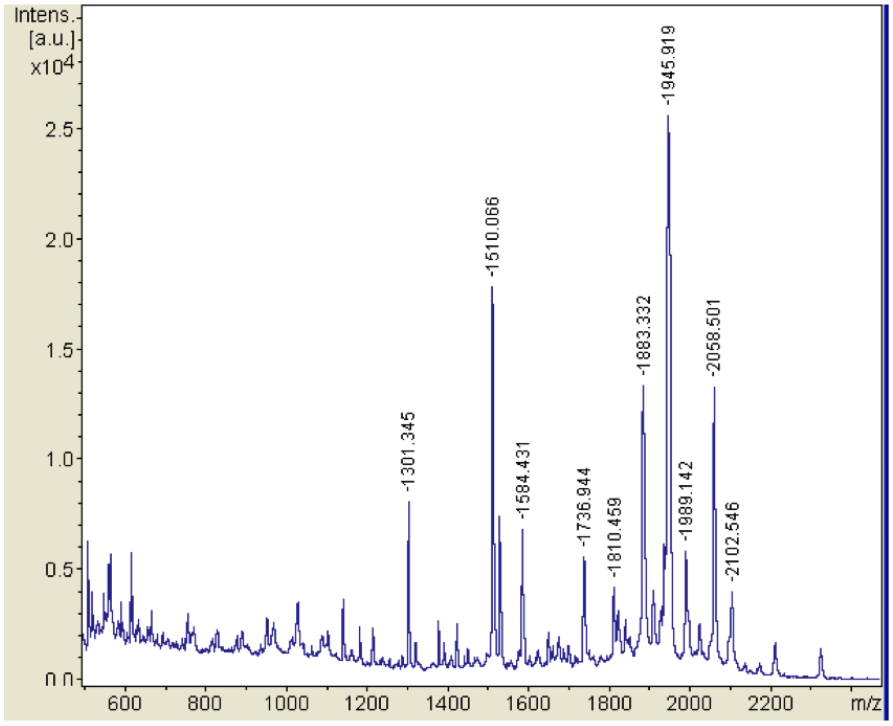


**Figure S4.** Maldi-Tof spectrum of PHOS-DDSQ

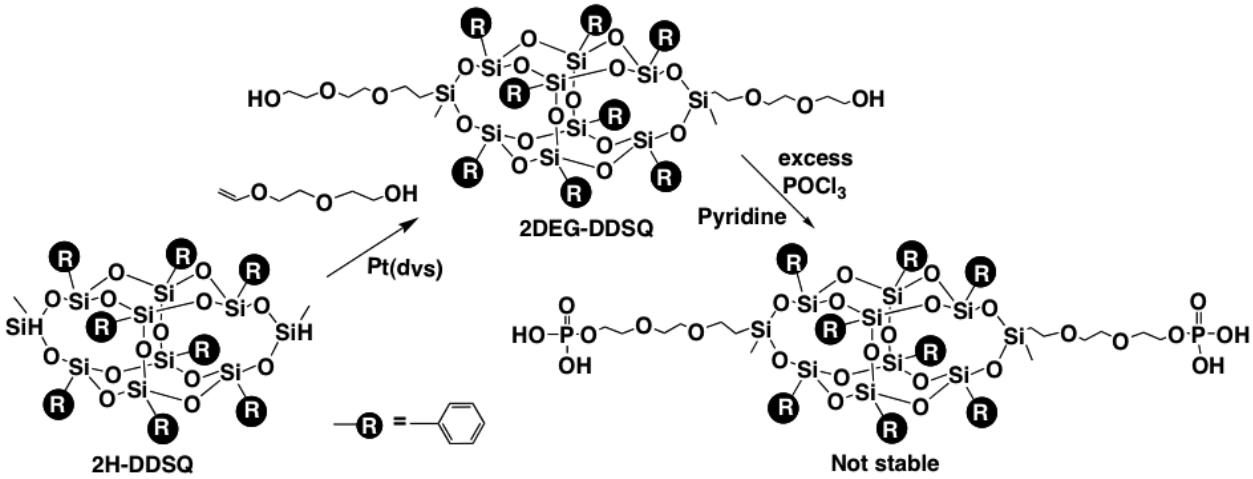


**Figure S5.** Synthesis of phosphonic acid functionalized two armed DDSQ