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## Supplementary Information for 'Periodic mesoporous phenylenesilicas with ether or sulfide hinge groups – a new class of PMOs with ligand channels'

Experimental parameters:

Powder X-ray diffraction patterns were collected on a D5000 Siemens diffractometer equipped with high-power long focus CuK $\alpha$  source operating at 50 kV/35 mA. The secondary beam is monochromatized by a Kevex Si/Li Solid State Detector. The experimental patterns are collected on a step scan mode. The obtained diffraction data were processed with DiffracPlus<sup>TM</sup> Eva 8.0 software. TEM images were obtained using an Hitachi HD-2000 microscope (200 kV/20  $\mu$ A) and SEM images collected on an Hitachi S-5200 (30 kV/10  $\mu$ A), with powder samples deposited onto carbon coated copper grids. Nitrogen adsorption measurements were recorded on a Quantachrome Autosorb-1C. Prior to measurement, PMO samples were outgassed at 120 °C for 24 hours. Solid-state NMR spectra were recorded on a Bruker DSX 400 NMR spectrometer using a 4mm rotor: <sup>29</sup>Si (79.5 MHz) CP-MAS NMR experiments {5 kHz spin rate, 3s pulse delay, 5 ms contact time,  $\pi/2$  pulse width of 5.0  $\mu$ s, 12000-18000 scans, external reference: Si[Si(CH<sub>3</sub>)<sub>3</sub>]<sub>4</sub>, <sup>13</sup>C (100.6 MHz) CP-MAS NMR experiments {5 kHz spin rate, 3s recycle delay, 5 ms contact time,  $\pi/2$  pulse width of 6.5  $\mu$ s, 5000-8000 scans, external reference: adamantane}. TGA spectra were recorded on a Perkin-Elmer TGA7 instrument with a heating rate of 5 °C min<sup>-</sup> 1. # Supplementary Material (ESI) for Chemical Communications

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**Fig. S1.** <sup>29</sup>Si solution NMR of the precursors: (a) Bis-4-(triethoxysilyl)phenyl ether (b) Bis-4-(triethoxysilyl)phenyl sulfide.



Fig. S2. <sup>13</sup>C CP/MAS-NMR spectra: (a) 4-phenyl ether PMO (b) 4-phenyl sulfide PMO.

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Fig. S3. Thermogravimetric analysis plots of the template extracted PMOs under a nitrogen atmosphere (1 = 4-phenyl ether, 2 = 4-phenyl sulfide).