

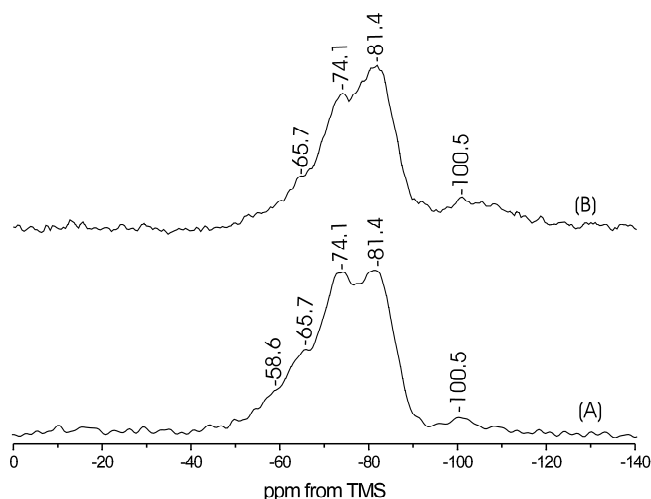
## Spectroscopic evidence of thermally induced metamorphosis in ethenylene-bridged periodic mesoporous organosilicas

Carl Vercaemst<sup>a,\*</sup>, James T.A. Jones<sup>b</sup>, Yaroslav Z. Khimyak<sup>b</sup>, José C. Martins<sup>c</sup>, Francis Verpoort<sup>a</sup> and Pascal Van Der Voort<sup>a,\*</sup>

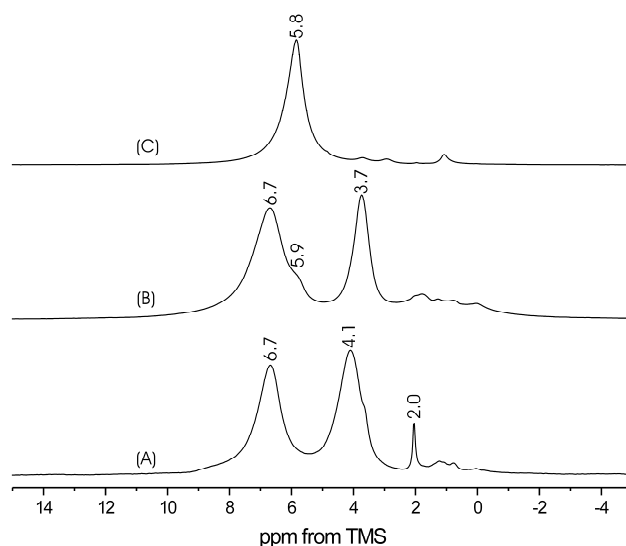
<sup>a</sup> Department of Inorganic and Physical Chemistry, Centre for Ordered Materials, Organometallics and Catalysis, University of Ghent, Krijgslaan 281, building S3, 9000 Ghent, Belgium.

<sup>b</sup> Department of Chemistry, University of Liverpool, Crown street, Liverpool, L69 72D, United Kingdom.

<sup>c</sup> Department of Organic Chemistry, University of Ghent, Krijgslaan 281, building S4, 9000 Ghent, Belgium.



**Figure ESI-1.** (A)  $^1\text{H}-^{29}\text{Si}$  CP/MAS (A) and  $^{29}\text{Si}\{^1\text{H}\}$  MAS NMR (B) spectra of  $-\text{CH}=\text{CH}-\text{PMO}$  after thermal treatment.



**Figure ESI-2.**  $^1\text{H}$  MAS NMR spectra of (A)  $-\text{CH}=\text{CH}-\text{PMO}$  before thermal treatment, (B)  $-\text{CH}=\text{CH}-\text{PMO}$  after thermal treatment and (C) pure vinylsilica.

### **<sup>1</sup>H MAS NMR**

<sup>1</sup>H MAS NMR spectra confirm observations derived from the <sup>13</sup>C and <sup>29</sup>Si NMR experiments. It is clear that after the thermal treatment, the solid displays the presence of vinyl protons and a small proportion of aliphatic resonances.

### **Experimental part**

<sup>1</sup>H Magic Angle Spinning (MAS) NMR spectra were acquired at 400.16 MHz using a bruker 2.5 mm <sup>1</sup>H/X probe with a <sup>1</sup>H  $\pi/2$  pulse length of 2.5  $\mu$ s at a MAS rate of 30.0 kHz and a recycle delay of 10 s. The position of the <sup>1</sup>H resonances is quoted in ppm from external TMS.