

Electronic Supplementary Information

Silicophosphates containing SiO₆ octahedra - anhydrous synthesis at ambient conditions

Sandra Jähnigen,^a Erica Brendler,^b Uwe Böhme,^a Gerhard Heide^c and Edwin Kroke*^a

^a TU Bergakademie Freiberg, Institut für Anorganische Chemie, Leipziger Straße 29, 09596 Freiberg, Germany. Fax: +49 (0)3731 394058; Tel: +49 (0)3731 393174; E-mail: Edwin.Kroke@chemie.tu-freiberg.de

^b TU Bergakademie Freiberg, Institut für Analytische Chemie, Leipziger Straße 29, 09596 Freiberg, Germany

^c TU Bergakademie Freiberg, Institut für Mineralogie, Brennhausgasse 14, 09596 Freiberg, Germany

Contents:

- Figure S1** ²⁹Si CP/MAS NMR spectra of **SiPO-3** with different contact times.
- Figure S2** ²⁹Si SP/MAS NMR spectrum of **SiPO-3**.
- Figure S3** Comparison of ³¹P CP/MAS and ³¹P MAS NMR spectra of compound **SiPO-3**.
- Figure S4** ¹H NMR spectrum of **SiPO-2**.
- Figure S5** ¹H→³¹P HETCOR NMR spectrum of **SiPO-2**.

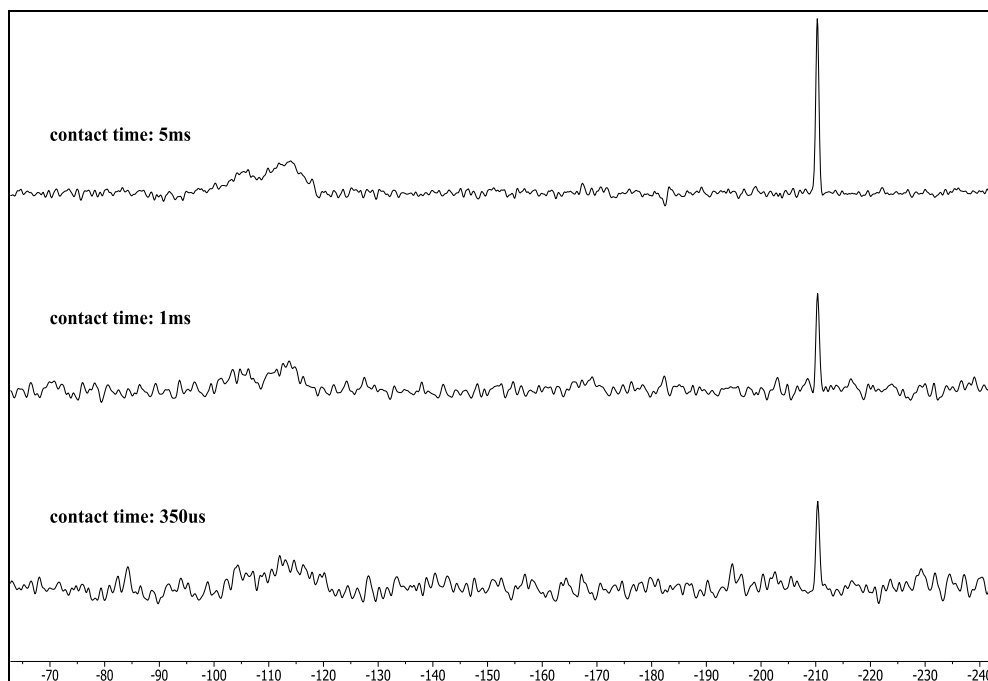


Fig. S1 ^{29}Si CP/MAS NMR spectra of **SiPO-3** [ppm] with contact time 350 μs , 1 ms and 5 ms. With increasing contact times signals at $\delta=-210$ ppm (SiO_6) were amplified compared to the SiO_4 signals.

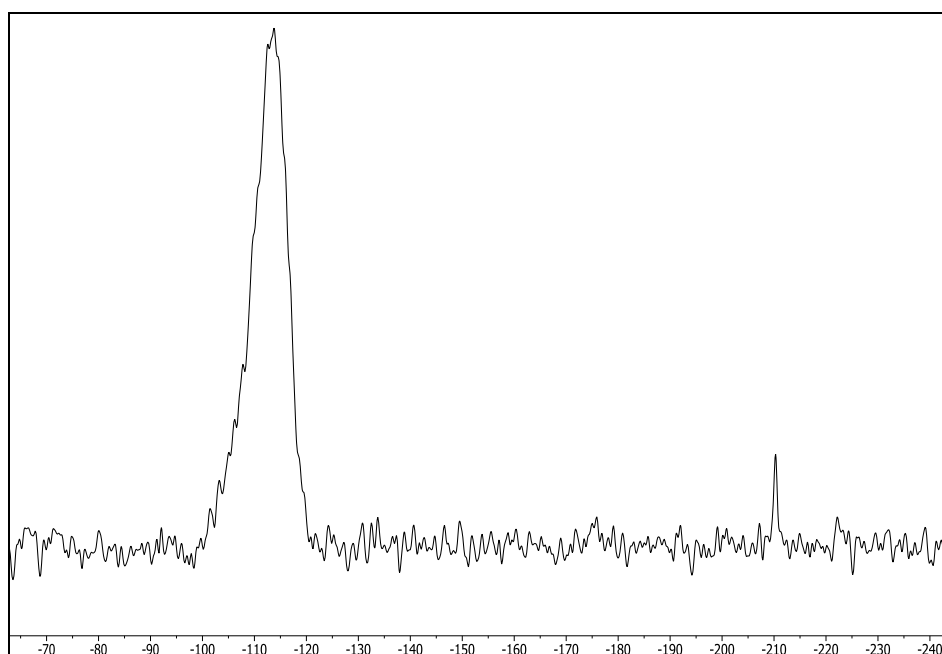


Fig. S2 ^{29}Si SP/MAS NMR [ppm] spectrum from a different batch of **SiPO 3**.

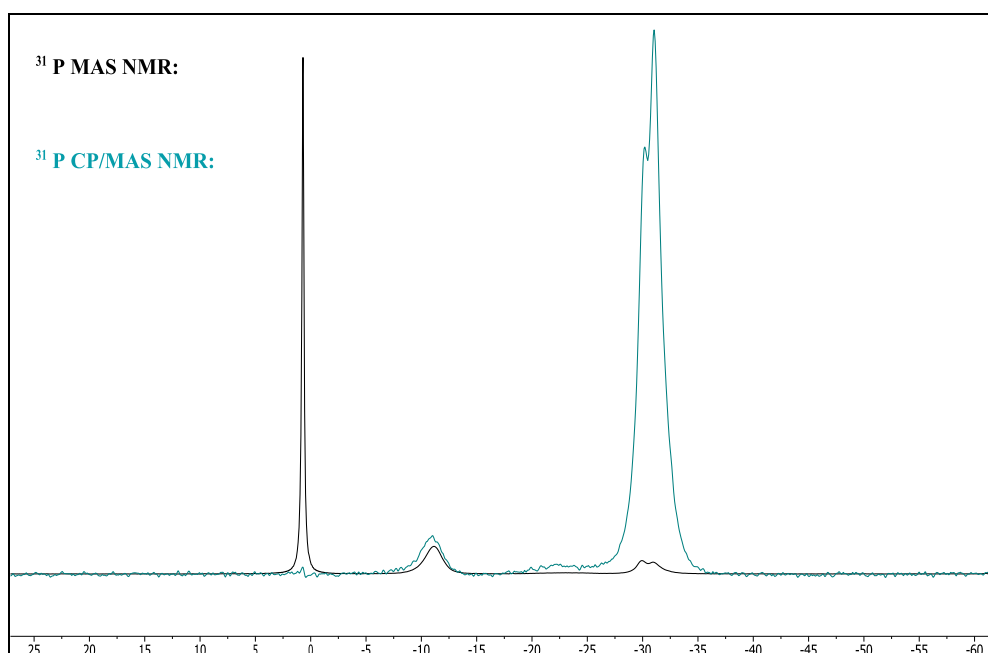


Fig. S3 Comparison of ³¹P CP/MAS and ³¹P MAS NMR spectra [ppm] of compound SiPO-3.

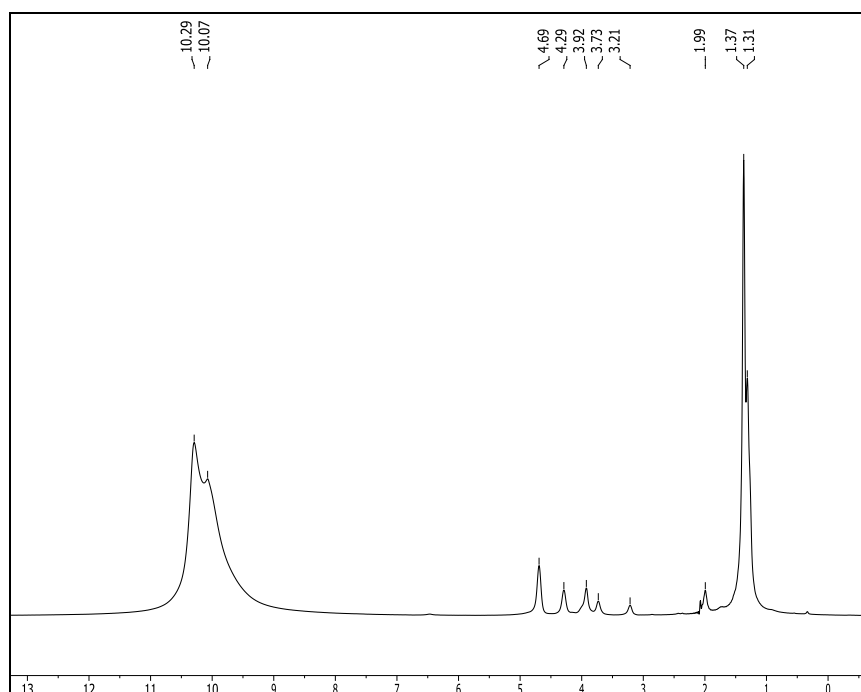


Fig. S4 ¹H MAS NMR spectrum [ppm] of compound SiPO-2 at 14 kHz spinning speed. Signals at 10 ppm can be assigned to remaining OH groups of phosphoric acid, values at 1.3 ppm and around 3.9 ppm represent CH₃ and CH of *i*-propoxy groups and remaining solvent.

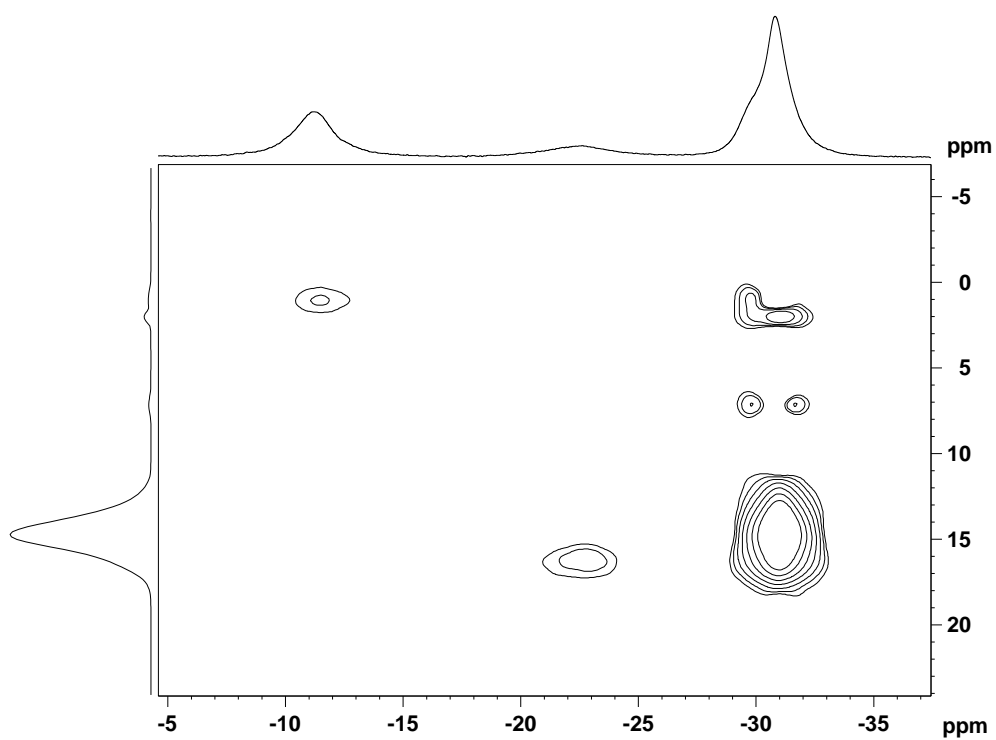


Fig. S5 $^1\text{H} \rightarrow ^{31}\text{P}$ HETCOR NMR spectrum of **SiPO-2** at 14 kHz. At the ^{31}P axes (horizontal) the ^{31}P single pulse MAS spectrum is shown.