Electronic Supplementary Information

Silicophosphates containing SiO₆ octahedra - anhydrous synthesis at ambient conditions

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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 $^{31}$P CP/MAS and $^{31}$P MAS NMR spectra [ppm] of compound SiPO-3.

**Fig. S4** $^1$H 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 CH$_3$ and CH of i-propoxy groups and remaining solvent.
Fig. S5 $^1$H$\rightarrow$$^{31}$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.