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**Supporting Information Available :**

**Table S.1** : Results of the simulations performed on the 13C CP and IRCP MAS-NMR spectra of samples prepared at various reaction temperatures. The reaction time was 2 hours, except for the 300°C* sample, 1.5 h.

**Table S.2** : Results of the analysis of the 29Si CP and IRCP MAS-NMR spectra
The percentages for the 220°C, 300°C, 325°C, 380°C and 450°C samples were extracted from the simulation of the CP spectra recorded for a contact time of 10 ms (see text). The reaction time was 2 hours, except for the 300°C* sample, 1.5 h.

**Figure S.1** : Examples of simulations of 13C IRCP MAS-NMR spectra of the 180°C MCPS (contact time : 3 ms; inversion times are indicated for each spectra).

**Figure S.2** : Examples of simulations of 29Si IRCP MAS-NMR spectra of the 180°C MCPS (contact time : 5 ms; inversion times are indicated for each spectra).

| Reaction Temperature | Peak | Chemical Shift (ppm) | % | $T_{CH}$ (ms) | $T_D$ (ms) | $T_{1\rho}H$ (ms) | n |
|---|---|---|---|---|---|---|---|
| 180°C | *a* | -7.6 | 46 | 69 | 2.2 | 19 | 1.1 |
| | *b* | 4.1 | 35 | 67 | 2.1 | 19 | 0.9 |
| | *c* | 11.4 | 19 | 118 | 2.4 | 22 | 0.5 |
| 200°C | *a* | -7.7 | 44 | 47 | 2.2 | 37 | 0.7 |
| | *d* | -6.2 | 10 | 59 | 0.9 | 17 | 1.1 |
| | *b* | 4.4 | 25 | 54 | 2.0 | 32 | 0.8 |
| | *c* | 11.3 | 21 | 75 | 2.5 | 37 | 0.4 |
| 300°C* | *a* | -9.3 | 32 | 52 | 2.0 | 20 | 0.7 |
| | *d* | -6.3 | 32 | 61 | 1.8 | 20 | 1.0 |
| | *b* | 4.7 | 24 | 59 | 2.1 | 19 | 0.8 |
| | *c* | 11.3 | 12 | 81 | 2.5 | 20 | 0.5 |
| 300°C | *a* | -9.4 | 45 | 65 | 1.4 | 16 | 0.6 |
| | *d* | -5.2 | 23 | 56 | 1.3 | 18 | 0.9 |
| | *b* | 4.4 | 13 | 64 | 1.5 | 17 | 0.6 |
| | *e* | 6.5 | 14 | 51 | 1.8 | 17 | 1.2 |
| | *c* | 11.1 | 5 | 115 | 1.8 | 17 | 0.2 |
| 325°C | *a* | -9.4 | 43 | 61 | 1.8 | 16 | 0.8 |
| | *d* | -5.2 | 28 | 57 | 1.2 | 20 | 0.9 |
| | *b* | 3.8 | 11 | 64 | 1.8 | 16 | 0.8 |
| | *e* | 6.5 | 15 | 44 | 1.3 | 21 | 0.7 |
| | *c* | 11.1 | 3 | 108 | 2.6 | 15 | 0.3 |
| 350°C | *a* | -9.2 | 36 | 49 | 1.6 | 24 | 0.7 |
| | *d* | -5.0 | 35 | 56 | 1.2 | 27 | 0.9 |
| | *b* | 3.6 | 2 | 57 | 1.8 | 23 | 0.7 |
| | *e* | 5.5 | 26 | 50 | 1.3 | 26 | 0.8 |
| | *c* | 11.1 | 1 | 71 | 2.1 | 19 | 0.2 |
| 380°C | *a* | -9.2 | 24 | 52 | 1.3 | 46 | 0.7 |
| | *d* | -4.7 | 35 | 47 | 1.1 | 54 | 0.8 |
| | *e* | 4.3 | 34 | 46 | 1.2 | 60 | 0.7 |
| | *f* | 9.3 | 7 | 16 | 0.7 | 32 | 2 |
| 400°C | *a* | -8.6 | 24 | 60 | 1.5 | 107 | 0.9 |
| | *d* | -3.6 | 32 | 55 | 1.1 | 126 | 1.0 |
| | *e* | 4.4 | 36 | 52 | 1.1 | 138 | 0.9 |
| | *f* | 9.3 | 8 | 18 | 0.6 | 75 | 2 |
| 450°C | *a* | -8.6 | 16 | 57 | 1.4 | 38 | 0.9 |
| | *d* | -3.6 | 23 | 47 | 1.2 | 42 | 0.9 |
| | *e* | 4.4 | 36 | 50 | 1.1 | 49 | 0.9 |
| | *f* | 9.3 | 25 | 24 | 1.7 | 33 | 2 |

**Table S.1 : Results of the simulations performed on the $^{13}C$ CP and IRCP MAS-NMR spectra of samples prepared at various reaction temperatures. The reaction time was 2 hours, except for the 300°C* sample, 1.5 h.**

| Reaction Temperature | Peak | Chemical Shift (ppm) | % | $T_{SiH}$ (ms) (From CP) | $T_{1\rho}$ (ms) | $T_{SiH}$ (ms) (From IRCP) |
|---|---|---|---|---|---|---|
| 180°C | $A_1$ | -64.0 | 46 | 2.0 | 30 | 2.1 |
| | $D$ | -51.2 | 3 | 1.9 | 26 | |
| | $B_1$ | 14.1 | 18 | 2.0 | 31 | 1.8 |
| | $B_2$ | 24.2 | 10 | 1.9 | 29 | 1.7 |
| | $C$ | 35.3 | 23 | 2.7 | 34 | 2.7 |
| 200°C | $A_1$ | -62.9 | 49 | 1.9 | 40 | 1.8 |
| | $D$ | -51.2 | 4 | 2.2 | 39 | |
| | $B_1$ | 14.2 | 14 | 1.9 | 40 | 1.8 |
| | $B_2$ | 25.5 | 11 | 2.1 | 40 | 1.9 |
| | $C$ | 36.0 | 22 | 2.5 | 44 | 2.6 |
| 220°C | $A_1$ | -62.8 | 56 | | | |
| | $D$ | -46.3 | 2 | | | |
| | $B_1$ | 14.2 | 10 | | | |
| | $B_2$ | 25.8 | 16 | | | |
| | $C$ | 36.6 | 16 | | | |
| 300°C* | $A_1$ | -64.0 | 57 | 2.0 | 35 | 2.0 |
| | $D$ | -47.0 | 6 | | | |
| | $B_1$ | 14.7 | 10 | 2.3 | 32 | 2.0 |
| | $B_2$ | 26.0 | 15 | 1.9 | 34 | 2.1 |
| | $C$ | 36.5 | 12 | 2.9 | 36 | 2.7 |
| 300°C | $E$ | -105.1 | 4 | | | |
| | $A_1$ | -67.7 | 59 | | | |
| | $D$ | -48.6 | 8 | | | |
| | $F$ | -35.1 | 1 | | | |
| | $B_1$ | 14.0 | 11 | | | |
| | $B_2$ /$J$ | 26.8 | 10 | | | |
| | $C$ | 34.6 | 7 | | | |
| 325°C | $E$ | -105.5 | 5 | | | |
| | $A_1$ | -64.9 | 55 | | | |
| | $D$ | -48.3 | 11 | | | |
| | $F$ | -32.1 | 6 | | | |
| | $B_1$ | 19.3 | 8 | | | |
| | $B_2$ /$J$ | 28.6 | 8 | | | |
| | $C$ | 37.2 | 7 | | | |
| 355°C | $E$ | -105.5 | 9 | 4.2 | 46 | 7.3 |
| | $A_2$ | -70.0 | 26 | 1.5 | 37 | 2.3 |
| | $A_1$ | -60.0 | 36 | 1.6 | 34 | 1.6 |
| | $D$ | -44.4 | 3 | 1.9 | 54 | 2.8 |
| | $F$ | -31.8 | 4 | 1.9 | 54 | 1.1 |
| | $B_1$ | 19.7 | 6 | 1.7 | 38 | 1.8 |
| | $J$ | 28.9 | 10 | 1.5 | 34 | 1.5 |
| | $C$ | 37.6 | 6 | 2.1 | 35 | 1.8 |
| 380°C | $E$ | -107.4 | 15 | | | |
| | $A_2$ | -72.4 | 12 | | | |
| | $A_1$ | -60.4 | 44 | | | |
| | $F$ | -32.2 | 8 | | | |
| | $G$ | -13.8 | 3 | | | |
| | $H$ | 10.3 | 2 | | | |
| | $B_1$ | 21.2 | 2 | | | |
| | $J$ | 28.2 | 11 | | | |
| | $C$ | 37.2 | 3 | | | |

| | | | | | | |
|---|---|---|---|---|---|---|
| 400°C | *E* | -107.4 | 15 | 3.3 | 202 | 2.3 |
| | $A_2$ | -74.4 | 11 | 2.2 | 91 | 2.8 |
| | $A_1$ | -60.6 | 34 | 1.8 | 170 | 1.6 |
| | *F* | -31.8 | 11 | 1.1 | 149 | 1.1 |
| | *G* | -12.8 | 11 | 1.1 | 167 | 1.1 |
| | *H* | 8.2 | 3 | 1.2 | 191 | 1.1 |
| | $B_1$ | 21.7 | 2 | 1.9 | 106 | 1.4 |
| | *J* | 28.1 | 11 | 1.6 | 172 | 1.5 |
| | *C* | 38.3 | 2 | 2.2 | 189 | 2.4 |
| 450°C | *D* | -108.5 | 18 | | | |
| | $A_2$ | -74.5 | 4 | | | |
| | $A_1$ | -60.6 | 31 | | | |
| | *F* | -29.4 | 15 | | | |
| | *G* | -13.4 | 3 | | | |
| | *H* | 3.5 | 17 | | | |
| | $B_1$ | 23.9 | 4 | | | |
| | *J* | 28.7 | 5 | | | |
| | *C* | 37.6 | 3 | | | |

**Table S.2 - Results of the analysis of the $^{29}$Si CP and IRCP MAS-NMR spectra**
**The percentages for the 220°C, 300°C, 325°C, 380°C and 450°C samples were extracted from the simulation of the CP spectra recorded for a contact time of 10 ms (see text). The reaction time was 2 hours, except for the 300°C* sample, 1.5 h.**

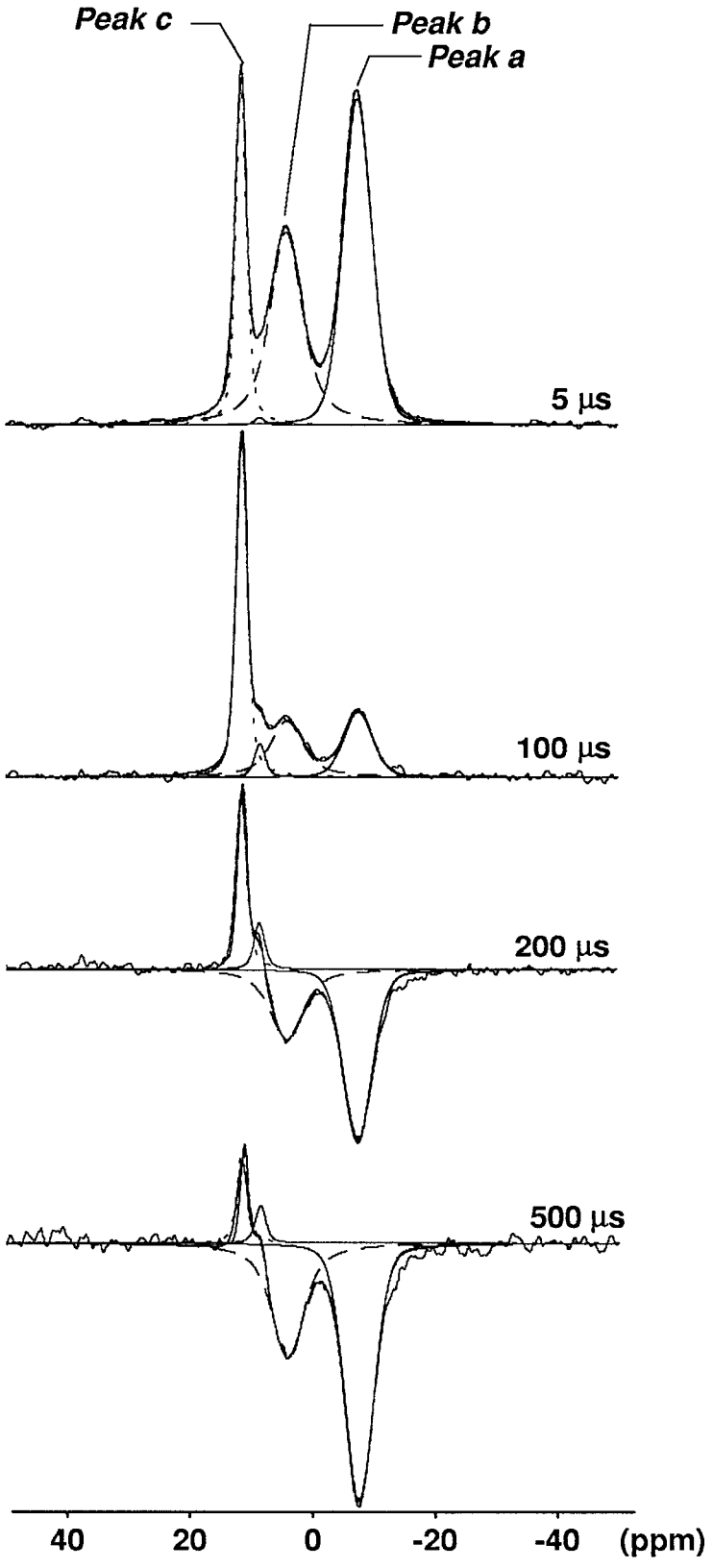


Figure 8.1

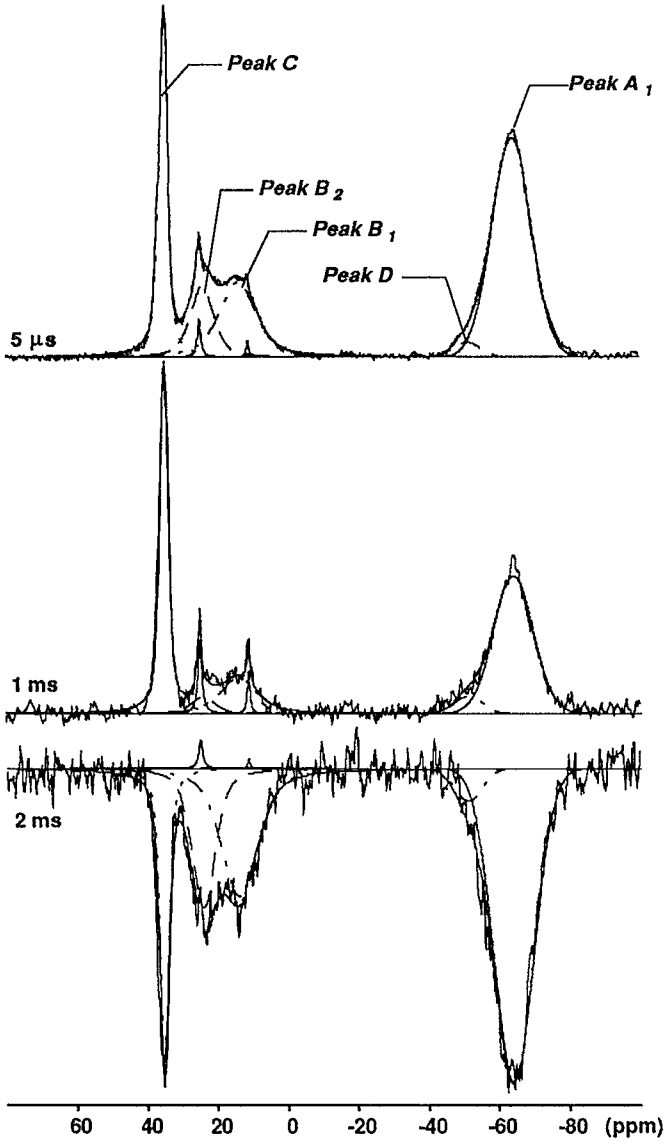
Peak C
Peak A 1
Peak B 2
Peak B 1
Peak D
5 µs
1 ms
2 ms
60
40
20
0
-20
-40
-60
-80
(ppm)

Figure 5.2