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**Supporting Information Available :**

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

**Table S.2** : Results of the analysis of the  $^{29}\text{Si}$  CP and IRCP MAS-NMR spectra

The percentages for the  $220^\circ\text{C}$ ,  $300^\circ\text{C}$ ,  $325^\circ\text{C}$ ,  $380^\circ\text{C}$  and  $450^\circ\text{C}$  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  $300^\circ\text{C}^*$  sample, 1.5 h.

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

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

Reaction Temperature	Peak	Chemical Shift (ppm)	%	T <sub>CH</sub> (ms)	T <sub>D</sub> (ms)	T <sub>1ρH</sub> (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 <sup>13</sup>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 <sub>SiH</sub> (ms) (From CP)	T <sub>1ρ</sub> (ms)	T <sub>SiH</sub> (ms) (From IRCP)
180°C	A <sub>1</sub>	-64.0	46	2.0	30	2.1
	D	-51.2	3	1.9	26	
	B <sub>1</sub>	14.1	18	2.0	31	1.8
	B <sub>2</sub>	24.2	10	1.9	29	1.7
	C	35.3	23	2.7	34	2.7
200°C	A <sub>1</sub>	-62.9	49	1.9	40	1.8
	D	-51.2	4	2.2	39	
	B <sub>1</sub>	14.2	14	1.9	40	1.8
	B <sub>2</sub>	25.5	11	2.1	40	1.9
	C	36.0	22	2.5	44	2.6
220°C	A <sub>1</sub>	-62.8	56			
	D	-46.3	2			
	B <sub>1</sub>	14.2	10			
	B <sub>2</sub>	25.8	16			
	C	36.6	16			
300°C*	A <sub>1</sub>	-64.0	57	2.0	35	2.0
	D	-47.0	6			
	B <sub>1</sub>	14.7	10	2.3	32	2.0
	B <sub>2</sub>	26.0	15	1.9	34	2.1
	C	36.5	12	2.9	36	2.7
300°C	E	-105.1	4			
	A <sub>1</sub>	-67.7	59			
	D	-48.6	8			
	F	-35.1	1			
	B <sub>1</sub>	14.0	11			
	B <sub>2</sub> /J	26.8	10			
	C	34.6	7			
325°C	E	-105.5	5			
	A <sub>1</sub>	-64.9	55			
	D	-48.3	11			
	F	-32.1	6			
	B <sub>1</sub>	19.3	8			
	B <sub>2</sub> /J	28.6	8			
	C	37.2	7			
355°C	E	-105.5	9	4.2	46	7.3
	A <sub>2</sub>	-70.0	26	1.5	37	2.3
	A <sub>1</sub>	-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 <sub>1</sub>	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
	E	-107.4	15			
380°C	A <sub>2</sub>	-72.4	12			
	A <sub>1</sub>	-60.4	44			
	F	-32.2	8			
	G	-13.8	3			
	H	10.3	2			
	B <sub>1</sub>	21.2	2			
	J	28.2	11			
	C	37.2	3			

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

**Table S.2 - Results of the analysis of the <sup>29</sup>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.

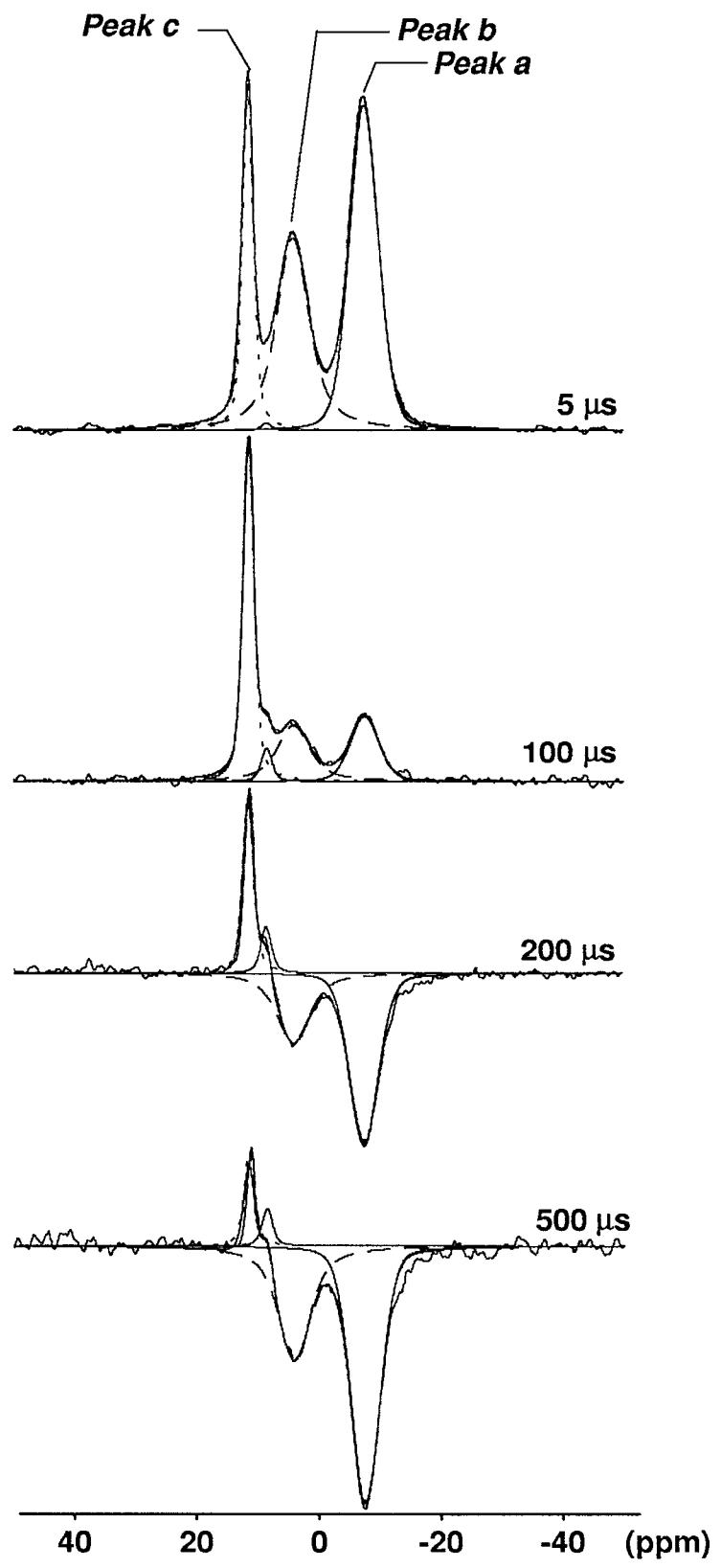


Figure S.1

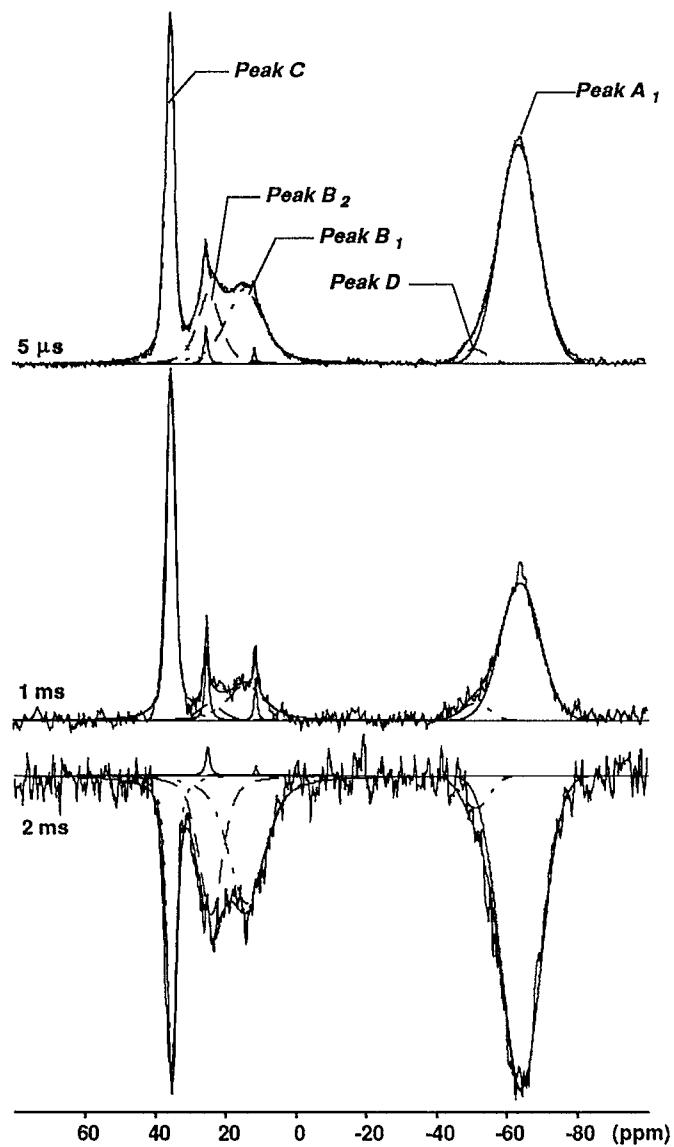


Figure S.2