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SUPPORTING INFORMATION

Optimization and intensification of hydrosilylation reaction using microreactor system

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GC vs. *in situ* IR monitoring of the reaction progress

Initially the selectivity and conversion to be reported in this article were calculated from gas chromatography (GC) measurements. However *in situ* IR method has many advantageous. Monitoring of the reaction using in-situ IR is relatively simple, and as one can see on the example in Figure S1, the peak corresponding to vibrations of Si-H band disappears with the progress of the reaction and on the basis of data obtained from IR spectroscopy, it easy to determine the conversion of substrate.

Comparing the results of GC and *in situ* IR analysis for all performed reactions we have got to the conclusions that in situ IR is more accurate and faster analytical technique that can be successfully used in the determination of the hydrosilylation reaction progress. The obtained results, of the conversion of Si-H bond in reactions between HMTS and 1-octene, from GC and *in situ* IR were very similar (Figure S2 and Table S1). However for the reactions of HMTS with 3-allyloxy-1,2-propanediol (Figure S3 and Table S2) and allyl glycidyl ether (Figure S4 and Table S3) data obtained from GC was higher than results calculated from IR spectra. Such observation was anticipated by us, as the difference in the observed results from both analytical techniques may come from higher values of the convention of substrates from samples submitted for the GC analysis as a result of longer time the reaction mixture had before being analyzed.

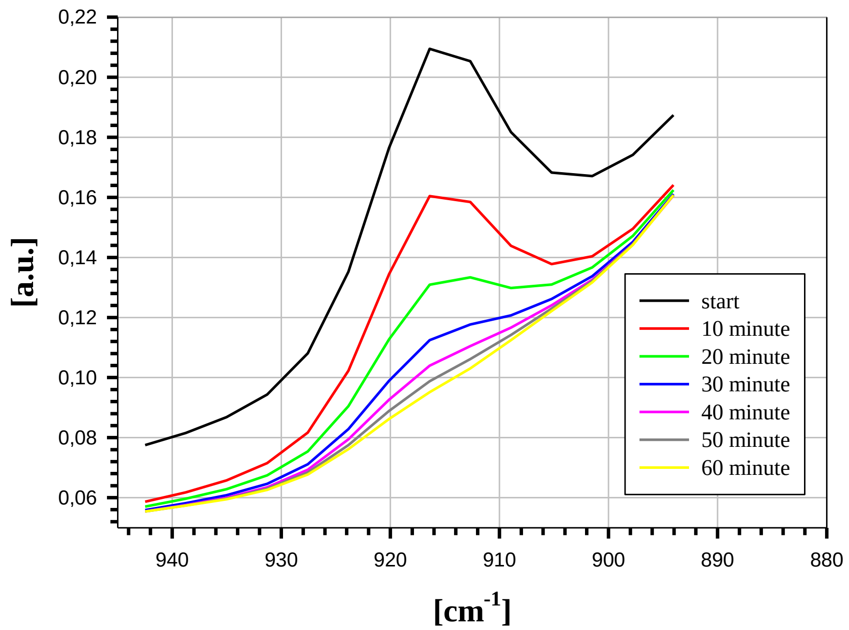

Figure S1. The disappearance of Si-H band with the progress of the reaction (temperature 80°C, concentration of catalyst: 1×10^{-4} mol Pt/mol Si-H monitored by *in situ* IR.

Thus, in our opinion, *in-situ* IR is a more precise method of controlling the progress of the reaction due to: (i) no additional analyzes being required to determine the Si-H bond conversion and because (ii) one can obtain the results in real time. In result, we have focused on presenting the results collected from *in situ* IR as we believe this is better reaction monitoring method.

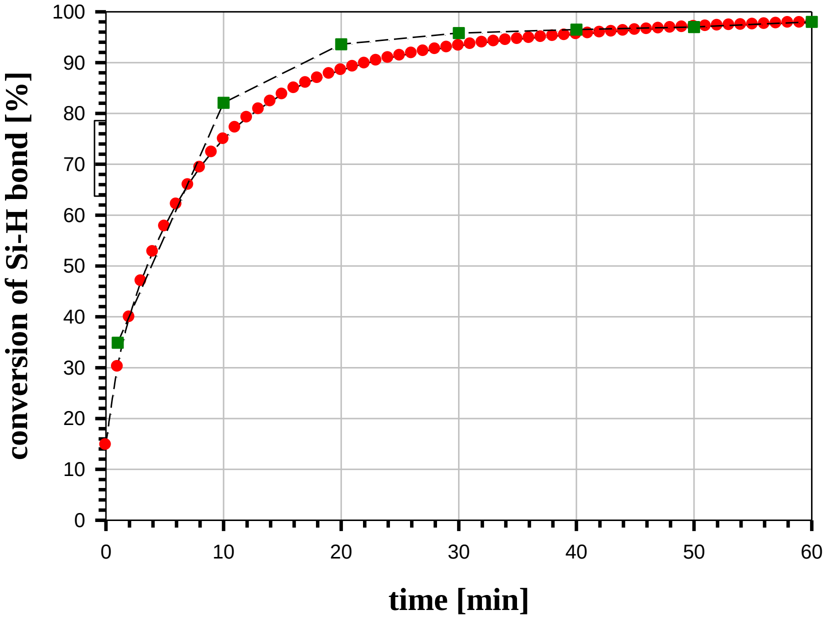

Figure S2. Conversion of Si-H bond (at 913 cm^{-1}) – comparison data obtained from GC and IR for reaction between HMTS and 1-octene performed at 65°C and with concentration of catalyst $1x10^{-6}$ mol Pt/mol Si-H (■ – data from GC; ● - data from *in situ* IR).

Table S1. Comparison of data obtained from GC and *in situ* IR (conversion of Si-H bond, in percent) for the reaction between HMTS and 1-octene in batch.

T, conc. of the catalyst [°C; mol Pt/mol Si-H]	conversion of Si-H bond [%]													
	1 min		10 min		20 min		30 min		40 min		50 min		60 min	
	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR
80°C, 1x10^{-4}	98.1	77.7	98.7	92.6	99.5	96.0	100	97.9	100	99.0	100	99.6	100	100
80°C, 1x10^{-5}	92.8	66.3	96.6	94.3	96.8	96.3	97.1	97.0	97.1	97.3	98.1	97.8	98.2	98.2
80°C, 1x10^{-6}	41.4	12.5	85.6	73.1	93.5	87.3	96.3	93.3	97.8	95.9	97.8	97.0	97.7	97.7
65°C, 1x10^{-4}	93.7	69.3	95.9	93.6	97.4	95.7	98.1	97.6	99.0	99.0	99.1	99.4	99.2	100
65°C, 1x10^{-5}	96.6	31.0	96.3	93.4	96.9	95.0	97.5	96.3	97.7	97.1	98.0	97.7	98.1	98.1
65°C, 1x10^{-6}	34.9	14.8	82.1	74.9	93.6	89.8	95.8	93.0	96.5	95.3	97.0	96.8	98.0	97.8

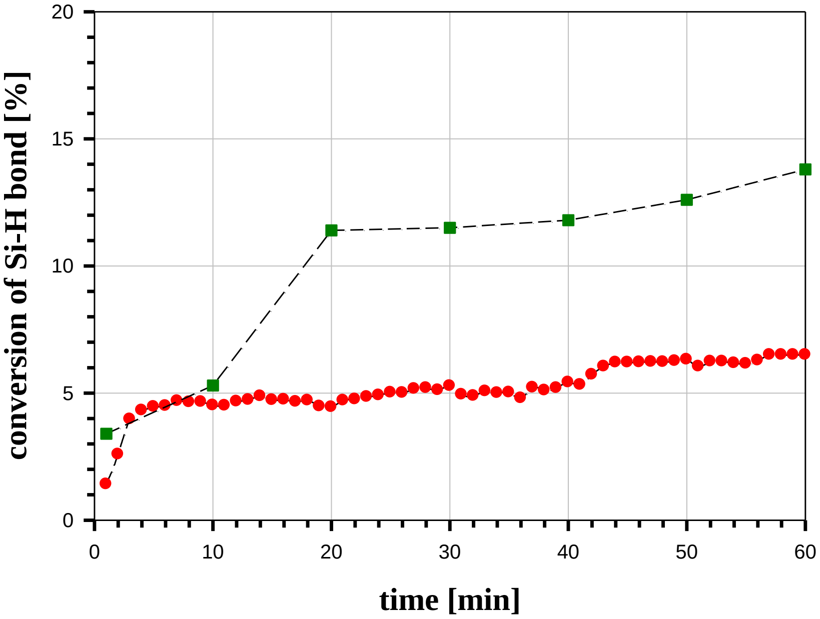

Figure S3. Conversion of Si-H bond (at 913 cm^{-1}) – comparison data obtained from GC and IR for reaction between HMTS and 3-allyloxy-1,2-propanediol performed at 80^{o}C and with concentration of catalyst 1x10^{-6} mol Pt/mol Si-H (■ – data from GC; ●- data from *in situ* IR).

Table S2. Comparison of data obtained from GC and *in situ* IR (conversion of Si-H bond, in percent) for the reaction between HMTS and 3-allyloxy-1,2-propanediol in batch.

T, conc. of the catalyst [°C; mol Pt/mol Si-H]	**conversion of Si-H bond [%]**													
	1 min		**10 min**		**20 min**		**30 min**		**40 min**		**50 min**		**60 min**	
	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR
80^{o}C, 1x10^{-4}	100	0	100	100	100	100	100	100	100	100	100	100	100	100
80^{o}C, 1x10^{-5}	95.3	0.1	100	60.2	100	76.0	100	87.9	100	94.3	100	98.0	100	100
80^{o}C, 1x10^{-6}	3.4	0.0	5.3	4.5	11.4	4.7	11.5	5.2	11.8	5.4	12.6	6.2	13.8	6.5
65^{o}C, 1x10^{-4}	100	0	100	100	100	100	100	100	100	100	100	100	100	100
65^{o}C, 1x10^{-5}	88.8	0.2	94.4	55.4	100	71.0	100	73.4	100	80.6	100	90.7	100	98.1
65^{o}C, 1x10^{-6}	0	0	5.5	0.2	5.7	0.2	5.8	0.2	5.9	0.2	8.4	0.2	8.8	0.2

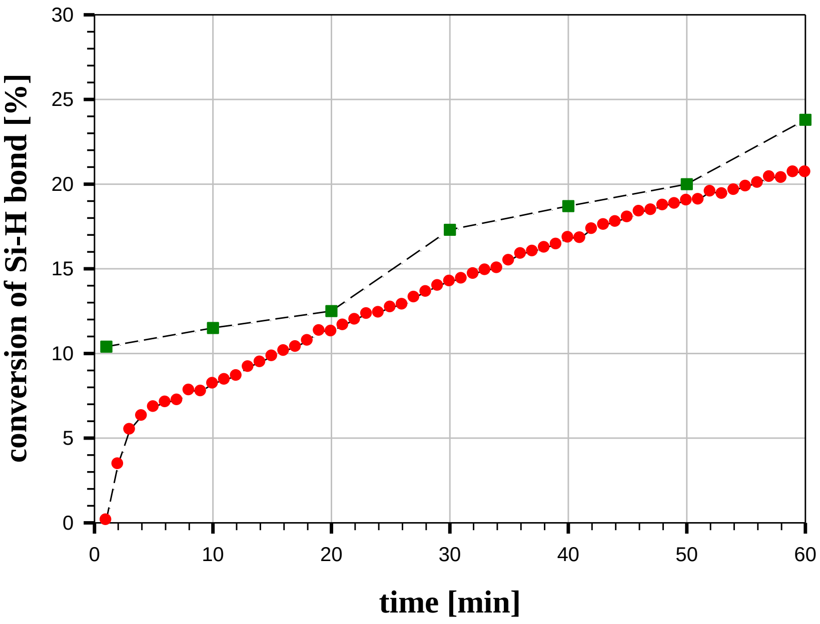

Figure S4. Conversion of Si-H bond (at 913 cm^{-1}) – comparison data obtained from GC and IR for reaction HMTS and allyl glycidyl ether performed at 65^{o}C and with concentration of catalyst $1x10^{-5}$ mol Pt/mol Si-H (■ – data from GC; ● - data from *in situ* IR).

Table S3. Comparison of data obtained from GC and *in situ* IR (conversion of Si-H bond, in percent) for the reaction between HMTS and allyl glycidyl ether in batch.

T, conc. of the catalyst [°C; mol Pt/mol Si-H]	**conversion of Si-H bond [%]**													
	1 min		**10 min**		**20 min**		**30 min**		**40 min**		**50 min**		**60 min**	
	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR	GC	IR
80°C, $1x10^{-4}$	90.2	0.6	100	100	100	100	100	100	100	100	100	100	100	0
80°C, $1x10^{-5}$	7.5	0	15.7	9.8	19.8	13.2	21.1	15.5	21.2	20.7	23.0	20.2	23.7	22.0
80°C, $1x10^{-6}$	~2	0	~2	1.1	~2	1.4	~2	1.7	~2	2.0	~2	2.2	~2	2.4
65°C, $1x10^{-4}$	90.4	0.9	92.1	100	100	100	100	100	100	100	100	100	100	100
65°C, $1x10^{-5}$	10.4	0.2	11.5	8.2	12.5	11.3	17.3	14.2	18.7	16.8	20.0	19.0	23.8	20.7
65°C, $1x10^{-6}$	~1	0	~1	0.9	~1	1.1	~1	1.4	~1	2.2	~1	1.9	~1	1.8

Table S4. Experimental conditions in microreactor system (1.4 volume) including flow rates of HMTS and olefins in different residence time.

Residence time [min]	Flow rate of 1-octene: HMTS [ml/min]	Flow rate of 3-allyloxy-1,2-propanediol: HMTS [ml/min]	Flow rate of allyl glicydyl ether: HMTS [ml/min]
1	0.510:0.890	0.440:0.960	0.430:0.970
10	-	0.044:0.096	0.043:0.097
20	-	0.022:0.048	0.022:0.049
30	-	0.015:0.032	0.014:0.032
40	-	0.011:0.024	0.011:0.024
50	-	0.009:0.019	0.086:0.019
60	-	0.007:0.016	0.007:0.016

Table S5. Experimental conditions in microreactor system (3 ml volume) including flow rates of HMTS and olefins in different residence time.

Residence time [min]	Flow rate of 1-octene: HMTS [ml/min]	Flow rate of 3-allyloxy-1,2-propanediol: HMTS [ml/min]	Flow rate of allyl glicydyl ether: HMTS [ml/min]
1	1.100:1.900	0.940:2.060	0.910:2.090
10	-	0.094:0.206	0.091:0.209
20	-	0.047:0.103	0.046:0.105
30	-	0.031:0.069	0.030:0.070
40	-	0.024:0.052	0.023:0.052
50	-	0.019:0.041	0.018:0.042
60	-	0.016:0.034	0.0150.035

Table S6. Experimental conditions in microreactor system (6 ml volume) including flow rates of HMTS and olefins in different residence time.

Residence time [min]	Flow rate of 1-octene [ml/min]	Flow rate of 3-allyloxy-1,2-propanediol [ml/min]	Flow rate of allyl glicydyl ether [ml/min]
1	2.200:3.800	1.880:4.120	1.820:4.180
10	-	0.188:0.412	0.182:0.418
20	-	0.094:0.206	0.091:0.209
30	-	0.063:0.137	0.061:0.139
40	-	0.047:0.103	0.046:0.105
50	-	0.038:0.082	0.036:0.084
60	-	0.031:0.069	0.030:0.070

Table S7. Experimental conditions in microreactor system (9 ml volume) including flow rates of HMTS and olefins in different residence time.

Residence time [min]	Flow rate of 1-octene [ml/min]	Flow rate of 3-allyloxy-1,2-propanediol [ml/min]	Flow rate of allyl glicydyl ether [ml/min]
1	-	2.820:6.180	2.730:6.270
10	-	0.282:0.618	0.273:0.627
20	-	0.141:0.309	0.138:0.315
30	-	0.093:0.207	0.090:0.210
40	-	0.072:0.156	0.069:0.156
50	-	0.057:0.123	0.054:0.126
60	-	0.048:0.102	0.045:0.105

Table S8. Experimental conditions in microreactor system (12 ml volume) including flow rates of HMTS and olefins in different residence time.

Residence time [min]	Flow rate of 1-octene [ml/min]	Flow rate of 3-allyloxy-1,2-propanediol [ml/min]	Flow rate of allyl glicydyl ether [ml/min]
1	-	3.760:8.240	3.640:8.360
10	-	0.376:0.824	0.364:0.836
20	-	0.188:0.412	0.182:0.418
30	-	0.126:0.274	0.122:0.178
40	-	0.094:0.206	0.092:0.210
50	-	0.076:0.164	0.072:0.168
60	-	0.62:0.138	0.060:0.140