

## SUPPLEMENTARY INFORMATION

### SILP materials based on TiO<sub>2</sub>-SiO<sub>2</sub> and TiO<sub>2</sub>-SiO<sub>2</sub>/lignin supports as new catalytic materials for hydrosilylation reaction – synthesis, physicochemical characterization and catalysis

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## 1. Materials and Techniques

### *Materials*

Reagents used in catalytic tests, such as 1-octene, allyl glycidyl ether, octafluoropentyl ether, 1,1,1,3,5,5,5-heptamethyltrisiloxane (HMST), triethylsilane, triethoxysilane (TriEOS) and *n*-decan were supplied by Sigma Aldrich and used as received. Ionic liquids: 1-butyl-3-methylimidazolium chloride, 1-butyl-4-methylpyridinium chloride, tributyltetradecylphosphonium chloride, triethylsulfonium bis(trifluoromethanesulfonyl)amide, trimethylsulfonium methylsulfate and lithium bis(trifluoromethanesulfonyl)amide, dimethyl sulfate and lithium bis(trifluoromethylsulfonyl)-imide were purchased from Iolitec. The reagents used to obtain the supports and their modification, i.e. isopropanol, methanol, dioxane, ammonia solution (25%), tetraethoxysilane, titanium (IV) isopropoxide (TIPP), Kraft lignin, 1,4-dioxane, sodium(meta)periodate, 3-(trimethoxysilyl)propyl isocyanate were purchased from Sigma Aldrich and did not require additional purification. The solvents used to prepare the SILP, such as: acetonitrile and dichloromethane were purchased from Fisher Chemicals.

### *Techniques*

The reaction yield was determined by using Clarus 680 gas chromatograph (Perkin Elmer) equipped with a 30 m capillary column Agilent VF-5ms and TCD detector. The measurement were carried out by using temperature program: 60 °C (3 min.), 10°C min<sup>-1</sup>, 290°C (5min.). Obtained hydrosilylation reaction product and synthesized ionic liquids were subjected to NMR analysis. NMR spectra were made with Bruker BioSpin (400Hz) spectrometer using acetonitrile-d<sub>3</sub> and chloroform-d as solvents. Ion chromatography experiments were carried out using Metrohm Eco IC system (Metrohm, Herisau, Switzerland) equipped with 863 Compact IC Autosampler, 10.0 µl injection loop and conductometric detector (maintained at room temperature). Self-regenerating Suppressor Module (MSM) (Metrohm, Herisau, Switzerland), regenerated with distilled water and sulfuric acid, was used to separate and determine anions in ionic liquids. FT-IR *in situ* tests were implemented by using Mettler Toledo ReactIR 15 instrument. During the experiment spectra with 256 scans for 1 at 30 second interval with resolution of 1 cm<sup>-1</sup> were recorded. The course of the reaction was determined on the basis of the disappearance of the band at 903 cm<sup>-1</sup>, corresponding to the Si-H bond. The IR spectra were recorded in the Bruker Tensor 27 apparatus in the range 500–4000 cm<sup>-1</sup> of wave number. XRD analysis of inorganic supports was performed by using X-ray diffractometer Bruker D8 Advance and standard measuring cuvette. The measurement was carried out for 1 hour, at an 2theta angle length 0 – 75. Catalyst leaching from the SILP materials was tested by using ICP-MS method, carried out on Perkin Elmer Nexion 300D inductively coupled mass plasma spectrometer. Thermogravimetric analysis was performed by TA Instruments analyser, model TG Q50 at a linear heating rate 10°C/min. under nitrogen (50ml/min.). For supports samples the analysis was conducted at 1000°C, samples of ionic liquids and SILP materials were analyzed at 600°C. Surface area and porosity characteristic was performed by using Micromeritics ASAP 2010 sorptometer. The analysed samples were first degassed at 393K for 20h. The BET (Brunauer-Emmet-Teller) surface area of supports and SILP materials were determined by N<sub>2</sub> adsorption at 77K. The total pore volumes and average pore diameters were determined by applying the Barrett–Joyner–Halenda (BJH) method to the isotherm desorption branch. Indication of the percentage of N, C, H, S in the samples was performed by elemental analysis. For this purpose Elementar Analyser Vario EL III was used. The samples were burned at a temperature of 1200°C, and then the generated gases were separated on adsorption columns and detected by the difference in thermal conductivity. SEM-EDX technique enabled to confirm presence of Mg, Si, O, S, P and C in the analysed samples, for supports and for SILP materials. For this analysis scanning electron microscope (SEM) Hitachi S-3500 N equipped with an energy-dispersive X-ray (EDX) detector Ultra Dry Silicon Drift X-ray Detector made by Thermo Scientific was used. The particle size measurement analysis was performed using the Zetasizer Nano ZS analyser by Malvern Instruments Ltd. The device analyses particles in the range of 0.6 - 6000 nm using the backscatter intensity beam of light. The analysed samples were powder dispersion in isopropanol or water, which was additionally destabilized in an ultrasonic bath.

## 2. Synthesis of ionic liquids

### *Synthesis of ionic liquids with Ntf<sub>2</sub><sup>-</sup>*

Synthesis of ionic liquids with bis(trifluoromethanesulfonyl)amide anion was carried out by metathesis reaction based on a procedure described in the literature<sup>1</sup>. For this purpose 1 equivalent of corresponding chloride (1-butyl-3-methylimidazolium chloride, 1-butyl-4-methylpyridinium chloride or tributyltetradecylphosphonium chloride) was dissolved in 20 ml of distilled water and placed into separating funnel. Then 20 ml of dichloromethane and 1.02 equivalent of 80% aqueous solution of lithium bis(trifluoromethanesulfonyl)amide (LiNtf<sub>2</sub>) were added. The content of the separating funnel was shaken vigorously for about 10 minutes. Then the water layer was removed and the organic layer was washed twice with distilled water and once with water and small amount of (0.02 eq) aqueous solution of LiNtf<sub>2</sub>. The whole procedure was repeated until Cl<sup>-</sup> anions in the aqueous layer disappeared. The presence or absence of chloride anions was confirmed by the test with AgNO<sub>3</sub>. The received organic phase was transferred to a rotary evaporator to remove the solvent, and then dried under vacuum to obtain a pure ionic liquid with the Ntf<sub>2</sub><sup>-</sup> anion. The complete exchange of Cl<sup>-</sup> to Ntf<sub>2</sub><sup>-</sup> anions was confirmed by ion chromatography (ESI). The structure of the obtained ionic liquids was confirmed by IR spectra and NMR analysis.

#### *1-butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide [BMIM][Ntf<sub>2</sub>]*

<sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.97 (t, *J*=7.4 Hz, 3H, -C-CH<sub>3</sub>); 1.37 (m, 2H, -C-C-CH<sub>2</sub>-); 1.87 (m, 2H, -C-CH<sub>2</sub>-C); 3.95 (s, 3H, N-CH<sub>3</sub>); 4.17 (t, *J*=7.5 Hz, 2H, N-CH<sub>2</sub>-); 7.28 (m, 1H, -HC-N-); 7.32 (m, 1H, -C=C-), 8.70 (s, 1H, N=CH-N).

IR: 3157-3122 cm<sup>-1</sup> (NH), 2880-2967 cm<sup>-1</sup> (CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>), 1467cm<sup>-1</sup>, 1572cm<sup>-1</sup> (C=C), 1330-1347 cm<sup>-1</sup>(-SO<sub>2</sub>-N), 1177 cm<sup>-1</sup> (-CF<sub>3</sub>), 1133 cm<sup>-1</sup> (-SO<sub>2</sub>), 1056 cm<sup>-1</sup> (-SO<sub>2</sub>), 612 cm<sup>-1</sup>, 513 cm<sup>-1</sup>, 568 cm<sup>-1</sup> (-CF<sub>3</sub>).

#### *1-butyl-4-methylpyridinium bis(trifluoromethanesulfonyl)amide [BMPy][Ntf<sub>2</sub>]*

<sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.96 (t, *J*=7.4 Hz, 3H, CH<sub>3</sub>-C-C-); 1.39 (m, 2H, -CH<sub>2</sub>-C-); 1.96 (m, 2H, -C-CH<sub>2</sub>-); 2.67 (s, 3H, -C-CH<sub>3</sub>); 4.51 (t, *J*=7.6 Hz, 2H, CH<sub>2</sub>-N-); 7.82 (d, *J*=6.4 Hz, 2H, -CH=C-); 8.63 (d, 2H, *J*=6.6 Hz, N=CH-C-).

IR: 3068 cm<sup>-1</sup> (NH), 2968-2880 cm<sup>-1</sup> (CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>), 1644 cm<sup>-1</sup> (C-C), 1577 – 1472 cm<sup>-1</sup> (C=C), 1346 – 1132 cm<sup>-1</sup> (-SO<sub>2</sub>-N), 1051 cm<sup>-1</sup> (-SO<sub>2</sub>), 612 cm<sup>-1</sup>, 600 cm<sup>-1</sup>, 569 cm<sup>-1</sup>, 510 cm<sup>-1</sup> (-CF<sub>3</sub>).

#### *Tributyltetradecylphosphonium bis(trifluoromethanesulfonyl)amide [P<sub>44414</sub>][Ntf<sub>2</sub>]*

<sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.88 (m, 3H, -C-CH<sub>3</sub>), 1.00 (m, 9H, C-CH<sub>3</sub>); 1.33 (m, 28H, -C-CH<sub>2</sub>-); 1.54 (m, 8H, -C-CH<sub>2</sub>-); 2.14 (m, 8H, P-CH<sub>2</sub>-).

IR: 2961 cm<sup>-1</sup>, 2855 cm<sup>-1</sup> (-CH<sub>2</sub>-CH<sub>2</sub>-), 2926 cm<sup>-1</sup> (P-CH<sub>2</sub>-), 1466 cm<sup>-1</sup> (C-C-C), 1348 – 1331 cm<sup>-1</sup> (-SO<sub>2</sub>-N), 1180 cm<sup>-1</sup> (-CF<sub>3</sub>), 1135 cm<sup>-1</sup>, 1054 cm<sup>-1</sup> (-SO<sub>2</sub>), 615 cm<sup>-1</sup>, 600 cm<sup>-1</sup>, 569 cm<sup>-1</sup>, 510 cm<sup>-1</sup> (-CF<sub>3</sub>).

### *Synthesis of ionic liquids with MeSO<sub>4</sub><sup>-</sup>*

To the round flask the solution of ionic liquid (1 eq.) in 10 cm<sup>3</sup> of acetonitrile (1-butyl-3-methylimidazolium chloride or tributyltetradecylphosphonium chloride) or 10 cm<sup>3</sup> of dichloromethane (1-butyl-4-methylpyridinium chloride) was placed and stirred. Then dimethyl sulfate (1 eq.) was dispensed dropwise for 5 minutes. After dosing the entire portion of methylsulfate precursor, the flask content was stirred for additional 30 minutes. Then the solvent was evaporated and obtained ionic liquid with MeSO<sub>4</sub><sup>-</sup> anion was dried under vacuum. The complete exchange of Cl<sup>-</sup> to MeSO<sub>4</sub><sup>-</sup> anion was confirmed by the test with AgNO<sub>3</sub>, where formation of AgCl wasn't observed<sup>2</sup>. The structure of the obtained ionic liquids was confirmed by IR spectra and NMR analysis.

#### *1-butyl-3-methylimidazolium methyl sulfate [BMIM][MeSO<sub>4</sub>]*

<sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.95 (t, *J*=7.4 Hz, 3H, CH<sub>3</sub>-C-C-); 1.35 (m, 2H, -CH<sub>2</sub>-C-); 1.86 (m, 2H, -C-CH<sub>2</sub>-); 3.71 (s, 3H, H<sub>3</sub>C-O-); 3.97 (s, 3H, -CH<sub>3</sub>); 4.21 (t, *J*=7.4 Hz, 2H, -H<sub>2</sub>C-N-); 7.42 (s, 1H, -C=C-), 7.48 (s, 1H, -C=C-); 9.41 (s, 1H, -N=C-).

IR: 3104-3150 cm<sup>-1</sup> (NH), 2875-2961 cm<sup>-1</sup> (CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>), 1466cm<sup>-1</sup>, 1573cm<sup>-1</sup> (N-C, C=C, C-C), 1384 cm<sup>-1</sup> (-CH<sub>3</sub>), 1220 - 980 cm<sup>-1</sup> (S=O), 1220 cm<sup>-1</sup> (-C-O), 1168 cm<sup>-1</sup> (-CH<sub>3</sub>, -N-C), 1006-1059 cm<sup>-1</sup> (C-C), 730 cm<sup>-1</sup> (NH<sub>2</sub>), 608 - 653 cm<sup>-1</sup> (-CH), 551 - 576 cm<sup>-1</sup> (-SO<sub>4</sub>).

#### *1-butyl-4-methylpyridinium methyl sulfate [BMPy][MeSO<sub>4</sub>]*

<sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.96 (t, *J*=7.4 Hz, 3H, CH<sub>3</sub>-C-C-); 1.39 (m, 2H, -CH<sub>2</sub>-C-); 1.97 (m, 2H, -C-CH<sub>2</sub>-); 2.65 (s, 3H, -C-CH<sub>3</sub>); 3.72 (s, 3H, H<sub>3</sub>C-O-); 4.89 (t, 2H, *J*=7.4 Hz, -CH<sub>2</sub>-N-); 7.86 (d, 2H, *J*=6.6 Hz, -C=C-); 9.02 (d, 2H, *J*=6.6 Hz, -C-N-).

IR: 3052cm<sup>-1</sup> (NH), 2961-2875 cm<sup>-1</sup> (-CH<sub>2</sub>), 1642 cm<sup>-1</sup> (C-C), 1520 – 1574 cm<sup>-1</sup> (C-C, C=C), 1471cm<sup>-1</sup> (N-C), 1383 cm<sup>-1</sup> (-CH<sub>3</sub>), 1222 cm<sup>-1</sup> (S=O, C-O), 1173 cm<sup>-1</sup> (-CH<sub>3</sub>, N-C), 1007- 1058 cm<sup>-1</sup> (C-C) 729 cm<sup>-1</sup> (N-H), 551 - 576 cm<sup>-1</sup> (-SO<sub>4</sub>).

Tributhyltetradecylphosphonium methyl sulfate [P<sub>44414</sub>][MeSO<sub>4</sub>]

<sup>1</sup>H NMR (CDCl<sub>3</sub>) δ (ppm): 0.87 (m, 3H, -C-CH<sub>3</sub>), 0.93 (m, 9H, C-CH<sub>3</sub>); 1.24 (m, 20H, -C-CH<sub>2</sub>-); 1.93 (m, 16H, -C-CH<sub>2</sub>-); 2.26 (m, 8H, P-CH<sub>2</sub>-); 3.70 (s, 3H, H<sub>3</sub>C-O-).

IR: 2954 cm<sup>-1</sup>, 2855 cm<sup>-1</sup> (CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>), 2924 cm<sup>-1</sup> (P-CH<sub>2</sub>-), 1464, 1413 cm<sup>-1</sup> (C-C-C-), 1381 cm<sup>-1</sup> (-CH<sub>3</sub>), 1250 cm<sup>-1</sup> (-S=O, C-O), 1224 cm<sup>-1</sup> (C-O), 1018 cm<sup>-1</sup>, 1069 cm<sup>-1</sup>, 1099 cm<sup>-1</sup> (-C-C-), 607 cm<sup>-1</sup> (-CH<sub>2</sub>-) 550 - 576 cm<sup>-1</sup> (-SO<sub>4</sub>).

### 3. Characteristic of obtained ionic liquids

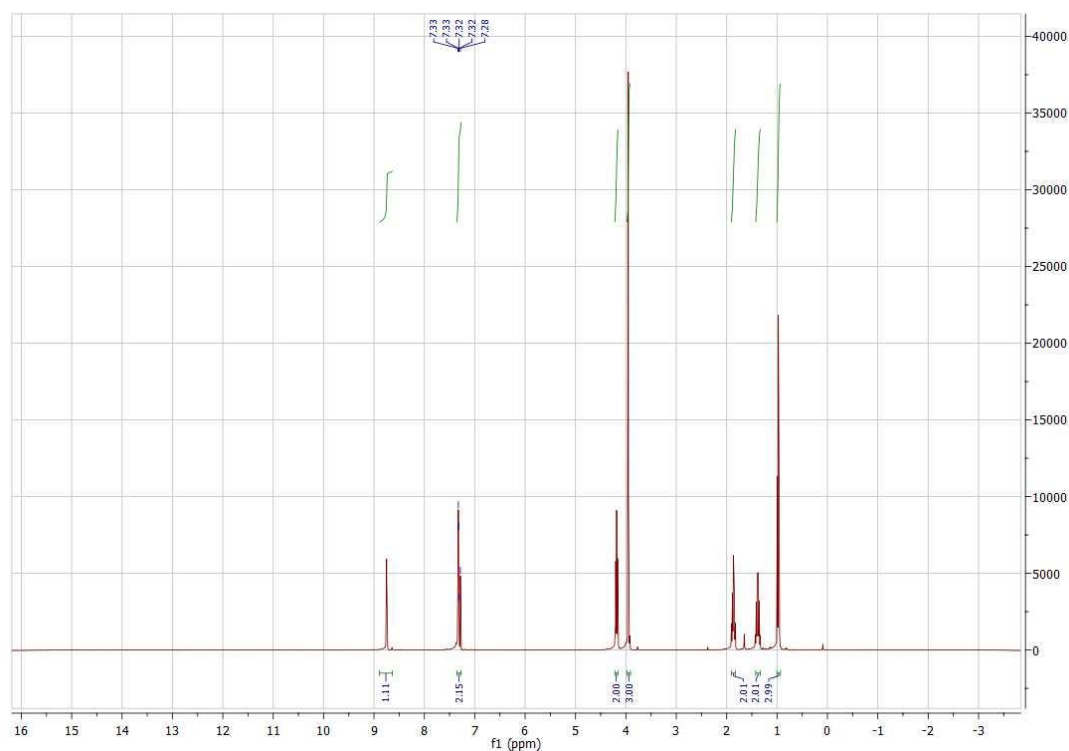
#### 3.1. Thermogravimetric analysis

**Table S1.** Thermogravimetric analysis of ionic liquids applied in the SILP materials

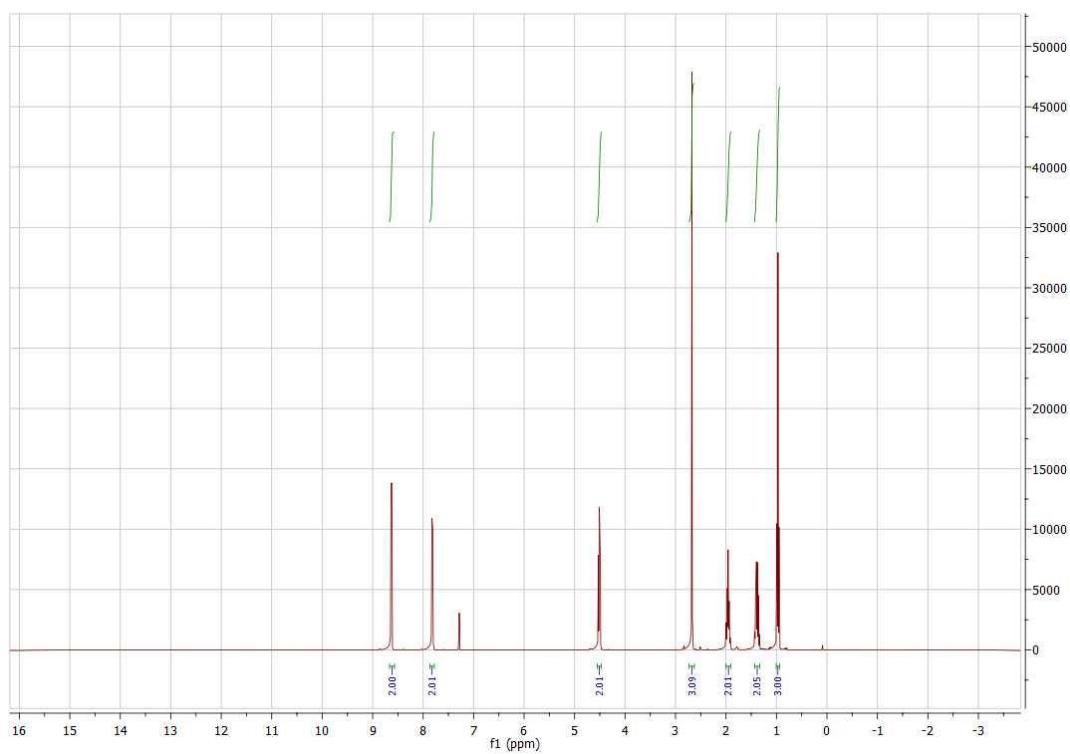
Ionic liquid	T <sub>onset</sub> [°C]	5% weight loss [°C]
[BMIM][Ntf <sub>2</sub> ]	447.19	423.45
[BMPy][Ntf <sub>2</sub> ]	431.37	419.90
[P <sub>44414</sub> ][Ntf <sub>2</sub> ]	440.94	417.02
[S <sub>222</sub> ][Ntf <sub>2</sub> ]	293.74	283.75
[BMIM][MeSO <sub>4</sub> ]	369.62	297.40
[BMPy][MeSO <sub>4</sub> ]	347.61	294.66
[P <sub>44414</sub> ][MeSO <sub>4</sub> ]	351.52	337.50
[S <sub>111</sub> ][MeSO <sub>4</sub> ]	291.00	259.72

#### 3.2. Spectroscopic characteristic

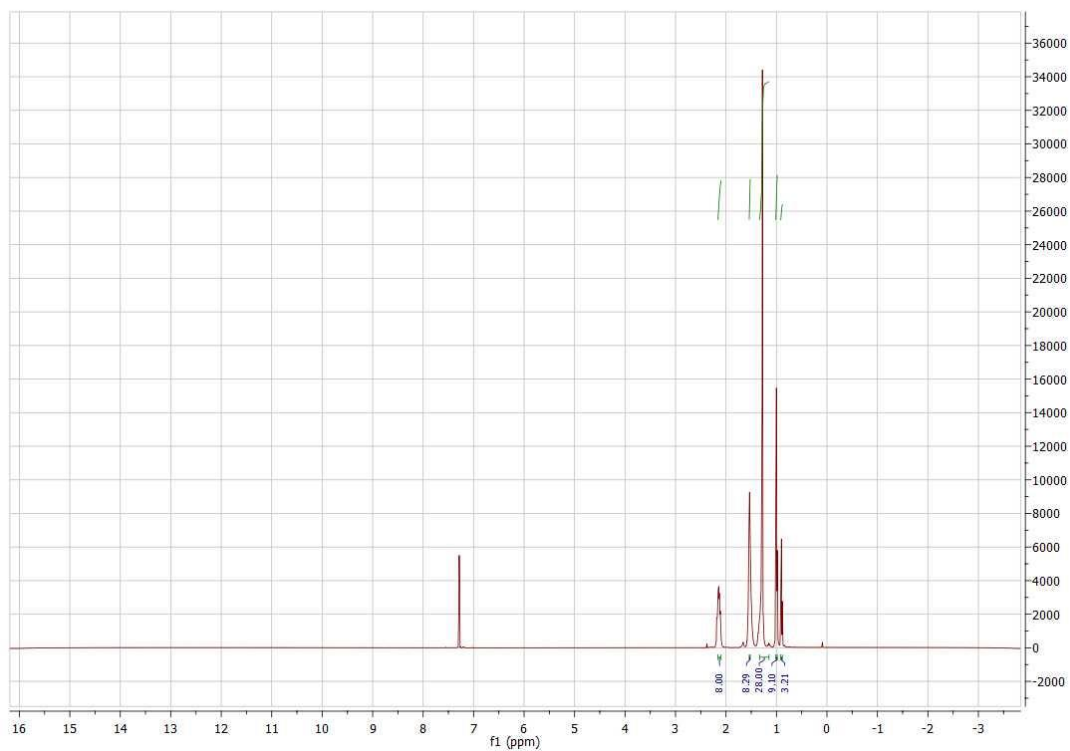
##### NMR spectra



**Fig. S1.** <sup>1</sup>H NMR spectrum of 1-butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide.



**Fig. S2.**  $^1\text{H}$  NMR spectrum of 1-butyl-4-methylpyridinium bis(trifluoromethanesulfonyl)amide.



**Fig. S3.**  $^1\text{H}$  NMR spectrum of tributyltetradecylphosphonium bis(trifluoromethanesulfonyl)amide.

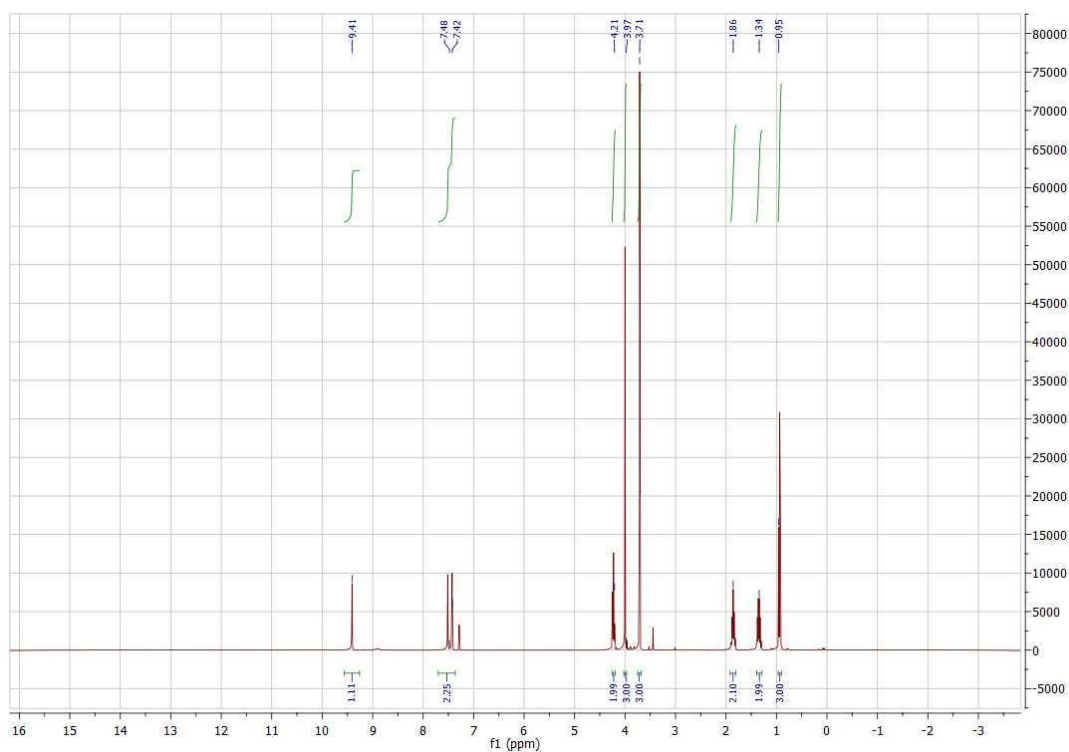


Fig. S4. <sup>1</sup>H NMR spectrum of 1-butyl-3-methylimidazolium methylsulfate.

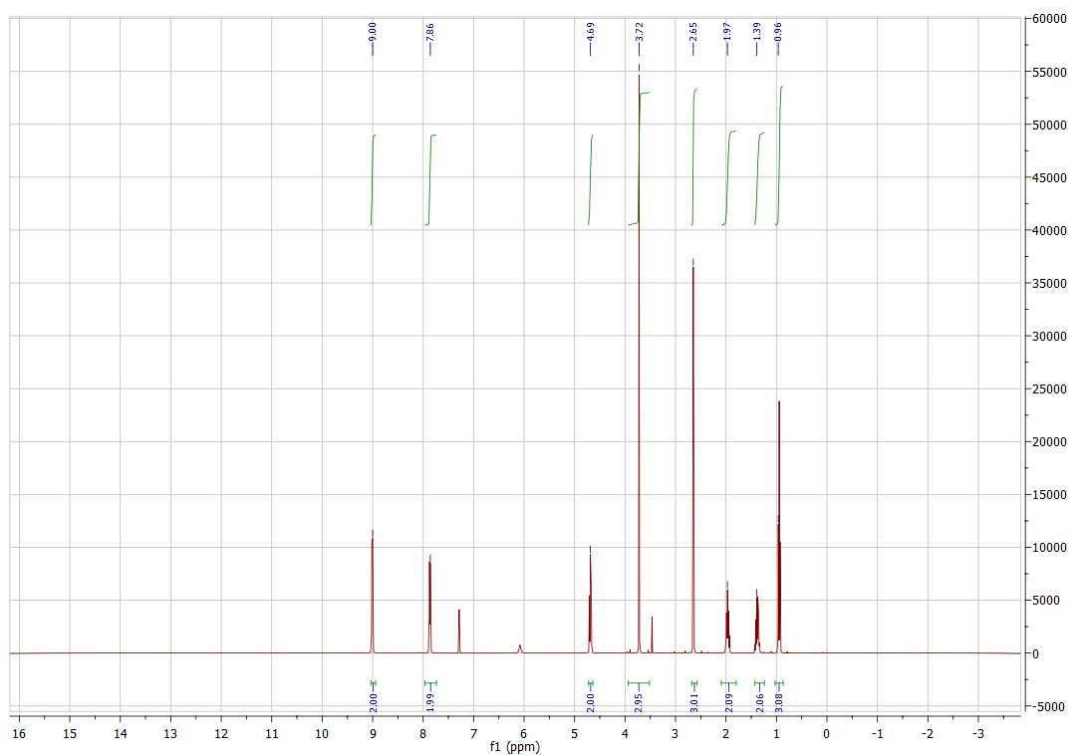
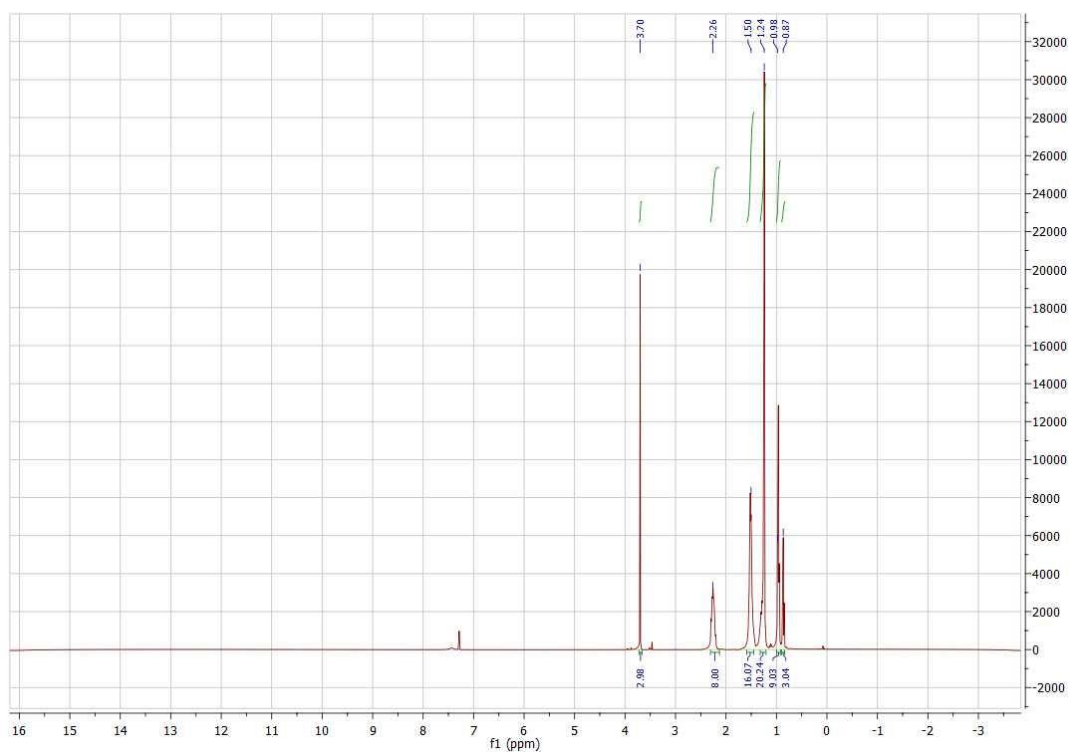
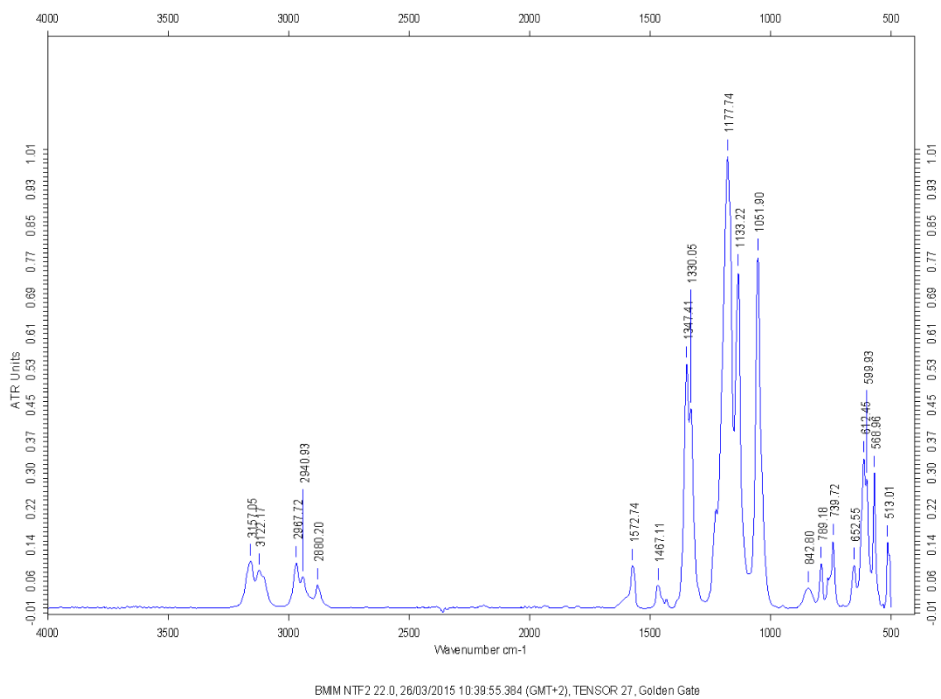


Fig. S5. <sup>1</sup>H NMR spectrum of 1-butyl-4-methylpyridinium methylsulfate.

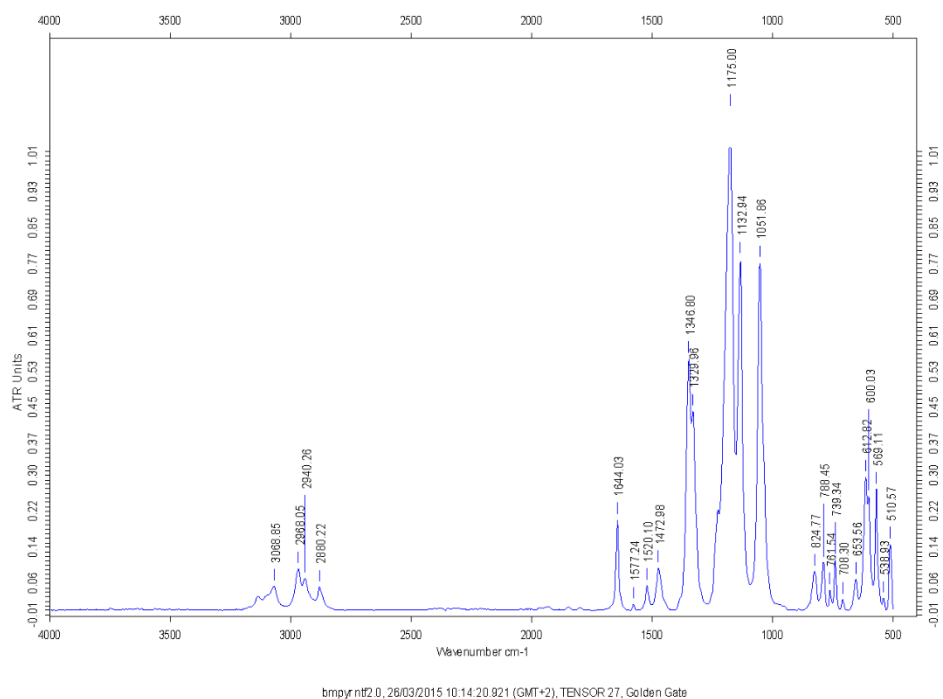


**Fig. S6.**  $^1\text{H}$  NMR spectrum of tributyltetradecylphosphonium methylsulfate.

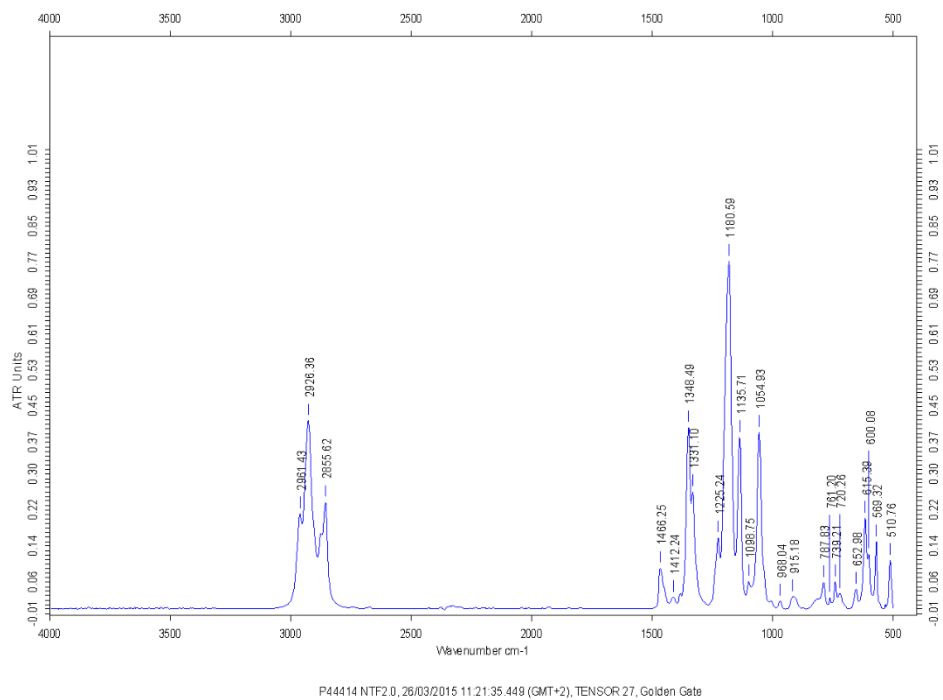
*IR spectra of ionic liquids*



**Fig. S7.** IR spectrum of 1-butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide.

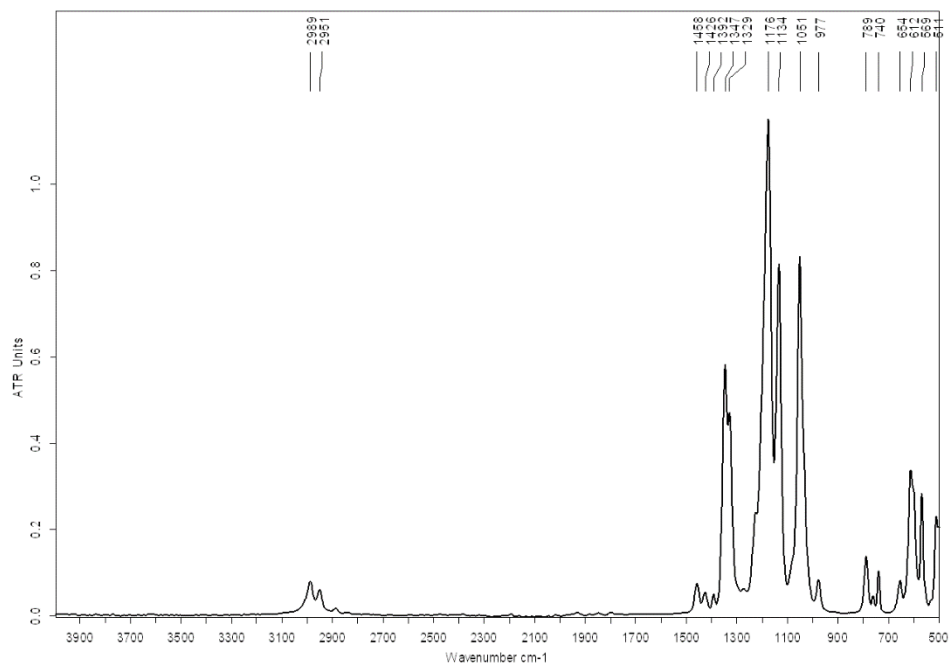


**Fig. S8.** IR spectrum of 1-butyl-4-methylpyridinium bis(trifluoromethanesulfonyl)amide.



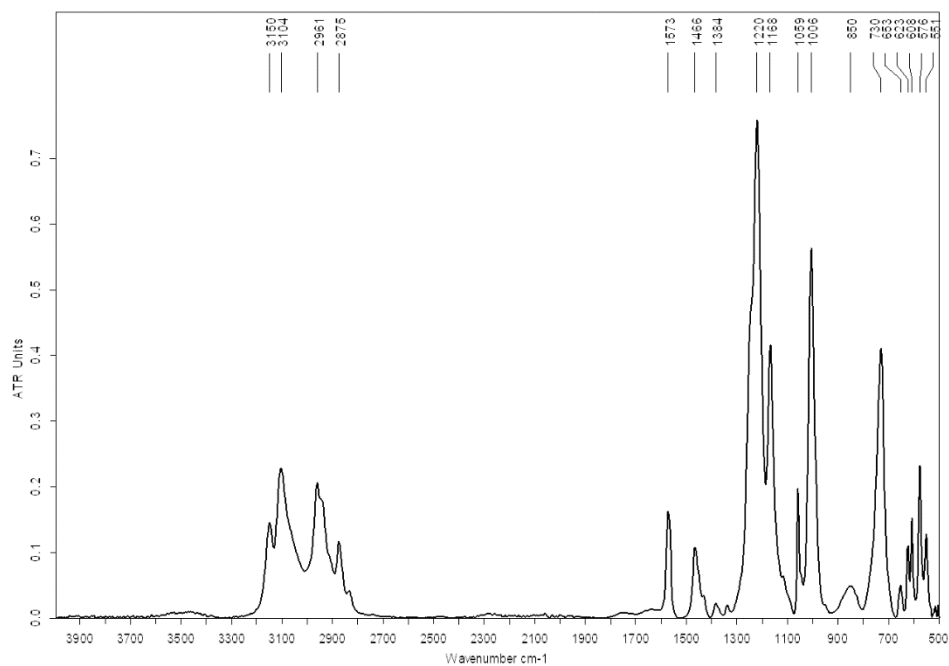
**Fig. S9.** IR spectrum of tributyltetradecylphosphonium bis(trifluoromethanesulfonyl)amide.





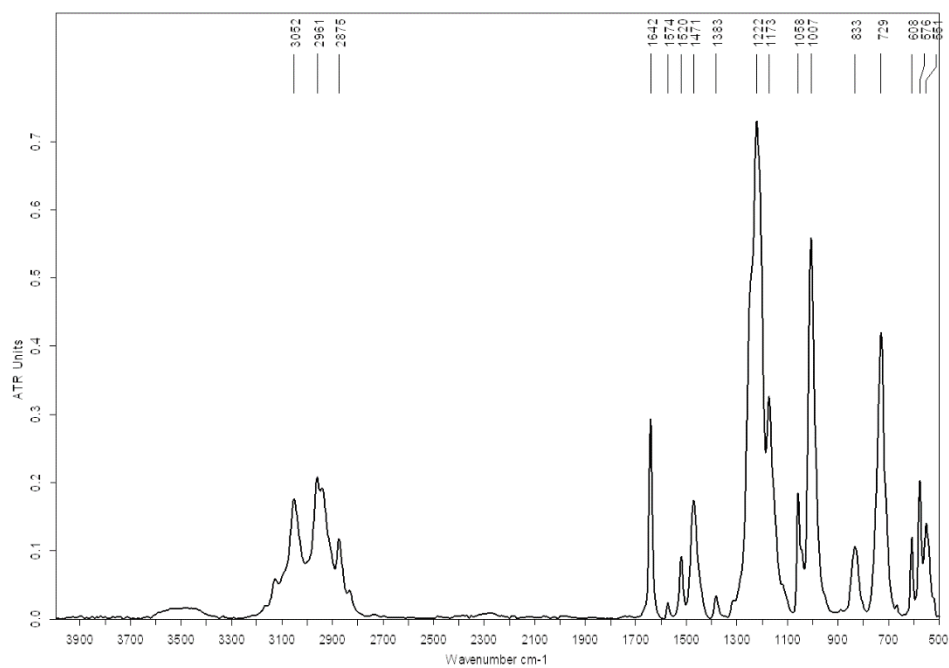
S222NIF2.0, 16/09/2020 08:48:11.549 (GMT+2), TENSOR 27, Golden Gate

**Fig. S10.** IR spectrum of triethylsulfonyl bis(trifluoromethanesulfonyl)amide.



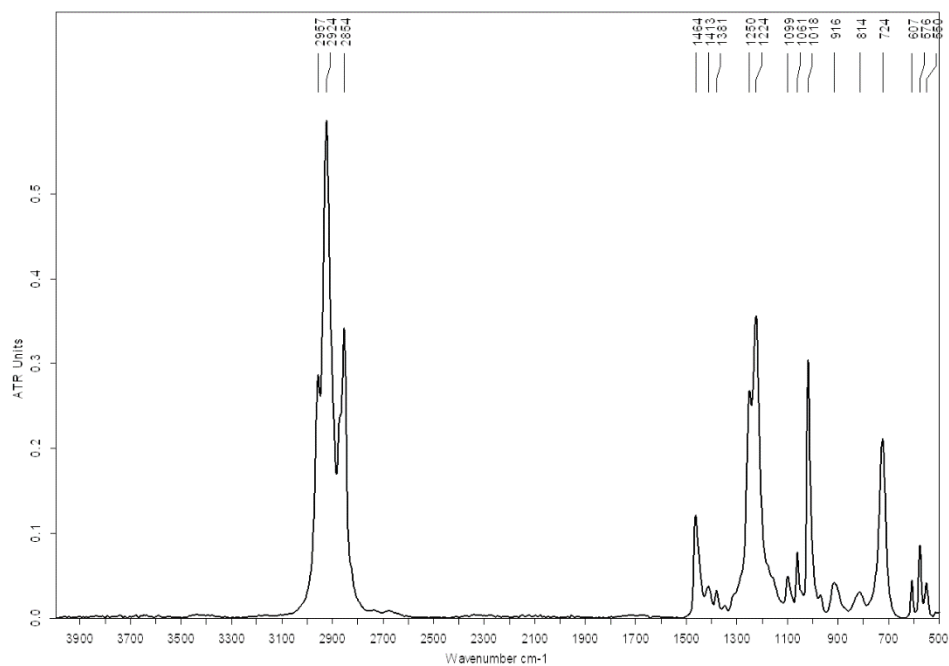
BMIM MeSO4.1, 30/11/2020 11:56:18.810 (GMT+1), TENSOR 27, Golden Gate

**Fig. S11.** IR spectrum of 1-butyl-3-methylimidazolium methylsulfate.



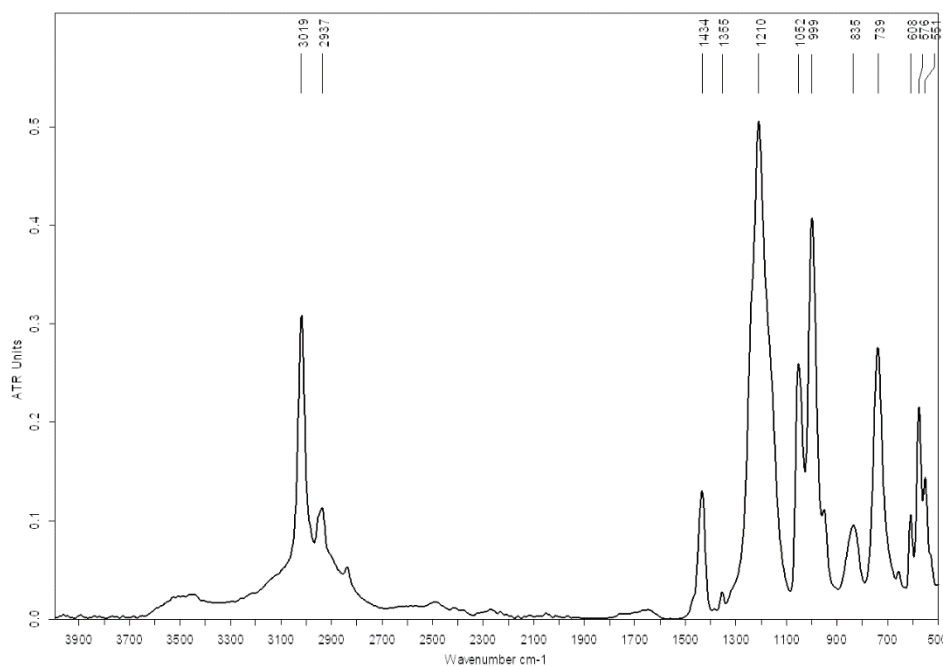
BMPy MeSO4.0, 30/11/2020 11:52:04.019 (GMT+1), TENSOR 27, Golden Gate

**Fig. S12.** IR spectrum of 1-butyl-4-methylpyridinium methylsulfate.



P44414 MeSO4.0, 30/11/2020 11:53:09.783 (GMT+1), TENSOR 27, Golden Gate

**Fig. S13.** IR spectrum of tributyltetradecylphosphonium methylsulfate.



S111 MeSO4.0, 30/11/2020 11:54:34.587 (GMT+1), TENSOR 27, Golden Gate

**Fig. S14.** IR spectrum of trimethylsulfonyl methylsulfate.

#### 4. Synthesis of inorganic support

##### *Synthesis of titanium dioxide – silica support (TiO<sub>2</sub>-SiO<sub>2</sub>)*

The synthesis of the inorganic oxide system TiO<sub>2</sub>-SiO<sub>2</sub> was carried out by the sol-gel method<sup>3</sup>. The reaction was carried out in a 2 L three-necked round bottom flask with mechanical stirrer, at room temperature. In the first stage, 6.55 mol of propan-2-ol and 0.125 mol of titanium (IV) isopropoxide (TIPP), as a precursor of TiO<sub>2</sub>, were introduced into the flask. Then 0.375 mol tetraethoxysilane (TEOS) as an organic precursor of SiO<sub>2</sub>, was introduced into the reaction mixture, at a rate of 6 cm<sup>3</sup>/min by using a peristaltic pump. The content of the flask was intensively stirred all the time. After dosed of all amount of TEOS, the reaction mixture was stirred for 10 min. Next, 1.97 mol of ammonia (25% aqueous solution), as a precursor of hydrolysis, by using peristaltic pump and with the same rate (6 cm<sup>3</sup>/min) was dosed. Obtained colloidal suspension was additionally stirred for 1 h. Then resulting alcogel was dried at 105°C for 24 h, and then the impurities have been removed by washing precipitate several times with distilled water and filtration. Obtained TiO<sub>2</sub>-SiO<sub>2</sub> was dried at 105°C for 6 h, calcinated at 600°C for 4 h and classified on sieves with a mesh size of 200 μm. In the presented research, in an analogous manner, the synthesis of oxide systems with different amounts of TiO<sub>2</sub> and SiO<sub>2</sub> precursors was carried out. The systems were prepared using an equimolar ratio of the individual precursors (0.25 mol TIPP and 0.25 mol TEOS) and with a significant excess of TiO<sub>2</sub> precursor (0.375 mol TIPP and 0.125 mol TEOS).

## 5. Synthesis of inorganic-organic support

### *Modification of TiO<sub>2</sub>-SiO<sub>2</sub> surface*

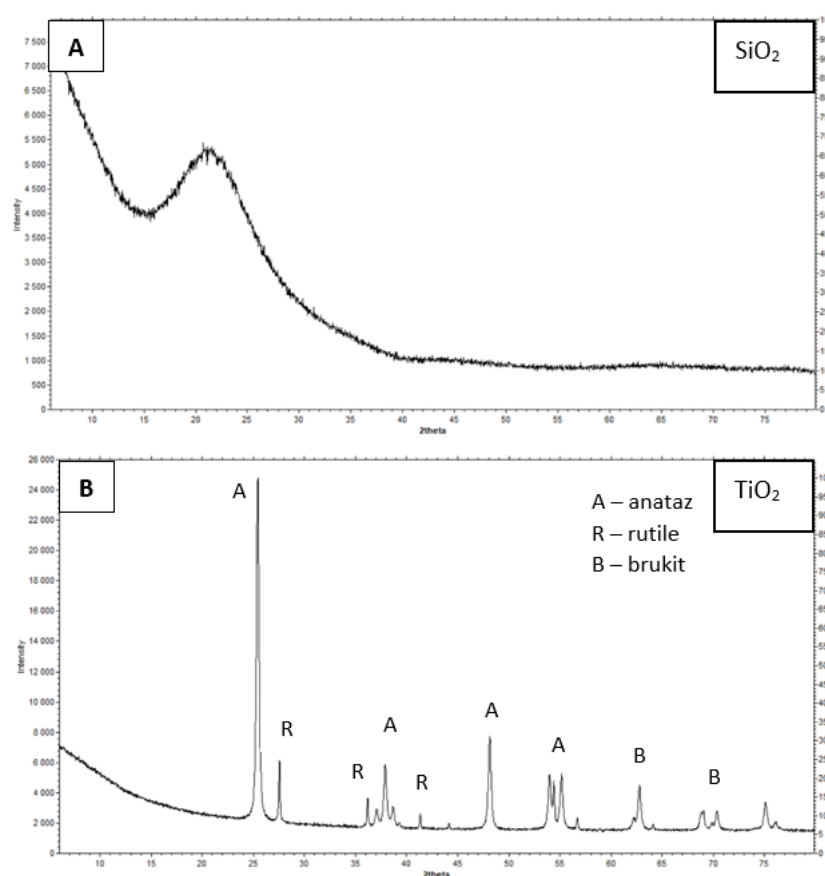
The TiO<sub>2</sub>-SiO<sub>2</sub> surface was grafted by using dry method. For this purpose, a mixture of 8 ml of methanol and 2 ml of water was prepared and then used for hydrolysed 1.25 g of 3-(trimethoxysilyl)propyl isocyanate. To the round bottom flask 5 g of TiO<sub>2</sub>-SiO<sub>2</sub> was added and previously prepared mixture was dosed by using peristaltic pump, with the rate 2 ml/min. The flask content was stirred all the time by magnetic stirrer. After dosing the entire modifier solution, the system was stirred for additional 60 minutes and then the suspension was filtrated and dried at 105°C for 24 h<sup>4</sup>.

### *Synthesis of TiO<sub>2</sub>-SiO<sub>2</sub>/lignin material*

The process of obtaining the TiO<sub>2</sub>-SiO<sub>2</sub>/lignin system was carried out in two stages. The first stage involved the activation of lignin. To the 250 ml three-necked flask added 67.5 ml of dioxane and 7.5 ml of distilled water. The system was stirred by using a mechanical stirrer a rate of 550 rpm. Then 1 g of lignin was added and the system was mixed for approx. 30 minutes. After that time, the solution consisting of 1.5 g of sodium periodate and 30 ml of water was dosed to the flask by using a peristaltic pump at the rate of 3 ml/min. The flask was darkened throughout the reaction to prevent light oxidation of the lignin. After the entire solution had been dispensed, the contents of the flask were stirred for another 30 minutes. Then 5 g of the previously modified support were added to the system and stirred for another hour. After this time, the dioxane was evaporated on a vacuum evaporator and the obtained precipitate was dried at 105 °C for 24 h<sup>5</sup>.

## 6. Physicochemical characteristic of synthesized supports

### 6.1. XRD



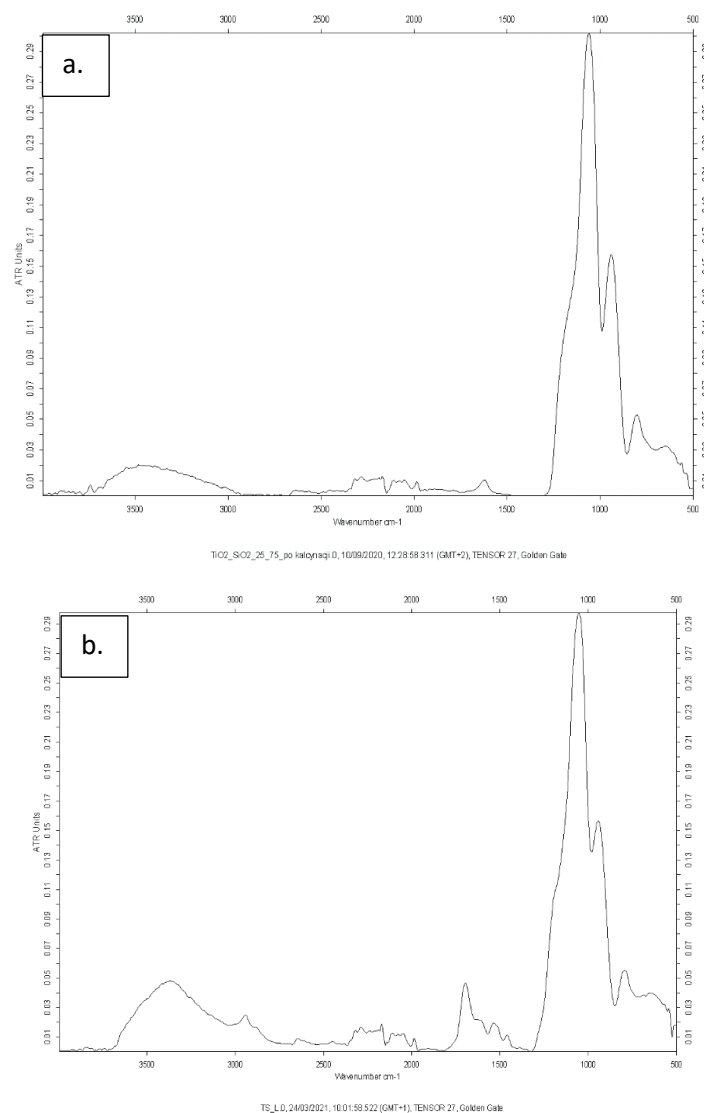
**Fig. S15.** X-rays diffraction patterns of SiO<sub>2</sub> (A) and TiO<sub>2</sub> (B).

## 6.2. Surface area and porosity characteristic

**Table S2.** Surface area and porosity characteristic of obtained SiO<sub>2</sub>, TiO<sub>2</sub> and TiO<sub>2</sub>-SiO<sub>2</sub> supports.

<i>Inorganic Support</i>	<i>BET Surface Area [m<sup>2</sup>/g]</i>	<i>Total Pore Volume [cm<sup>3</sup>/g]</i>	<i>Average Pore Diameter [nm]</i>
SiO <sub>2</sub>	8	0.02	5.97
TiO <sub>2</sub>	3	0.13	13.08
TiO <sub>2</sub> -SiO <sub>2</sub> (0.25:0.75)	328	0.91	10.95
TiO <sub>2</sub> -SiO <sub>2</sub> (0.50:0.50)	193	0.29	5.59
TiO <sub>2</sub> -SiO <sub>2</sub> (0.75:25)	183	0.29	5.21

## 6.3. IR spectra



**Fig. S16.** IR spectra of TiO<sub>2</sub>-SiO<sub>2</sub> (a) and TiO<sub>2</sub>-SiO<sub>2</sub>/lignin (b) supports

## 7. Preparation of Rh-SILP and Pt-SILP materials

New SILP materials were prepared using physical impregnation method. For this purpose 0.2g of proper ionic liquid (with [BMIM]<sup>+</sup>, [BMPy]<sup>+</sup>, [P<sub>44414</sub>]<sup>+</sup>, [S<sub>222</sub>]<sup>+</sup> or [S<sub>111</sub>]<sup>+</sup> cation and Ntf<sub>2</sub><sup>-</sup> or MeSO<sub>4</sub><sup>-</sup> anion ) and rhodium or platinum catalyst (0.68 mg of Rh(PPh<sub>3</sub>)<sub>3</sub> or 0.14 mg of Pt(cod)Cl<sub>2</sub>) were placed in a flask and dissolved in 20 cm<sup>3</sup> dichloromethane or acetonitrile (for [S<sub>111</sub>][MeSO<sub>4</sub>]). Then to obtained mixture 2 g of previously obtained inorganic support – TiO<sub>2</sub>-SiO<sub>2</sub> was added and stirred for 30 minutes. The process of preparation of

Rh-SILP and Pt-SILP was conducted at room temperature. Next the flask was transferred to the rotary evaporator to removed solvent. The evaporation of dichloromethane was carried out very slowly to obtained even layer of ionic liquid. Then the obtained SILP material was dried under vacuum for 48 h at 40°C. Obtained materials included 10% (w/w) of ionic liquid and  $4 \cdot 10^{-3}$  % of rhodium or platinum<sup>6</sup>. SILP systems with 15% and 20% (w/w) of ionic liquid were prepared in analogous way and only change was the amount of added ionic liquid corresponding respectively to 0.3 g and 0.4 g.

## 8. Physicochemical characteristic of obtained SILP materials

### 8.1. Thermogravimetric analysis (TG)

*Table S3. Thermogravimetric analysis of obtained SILP materials.*

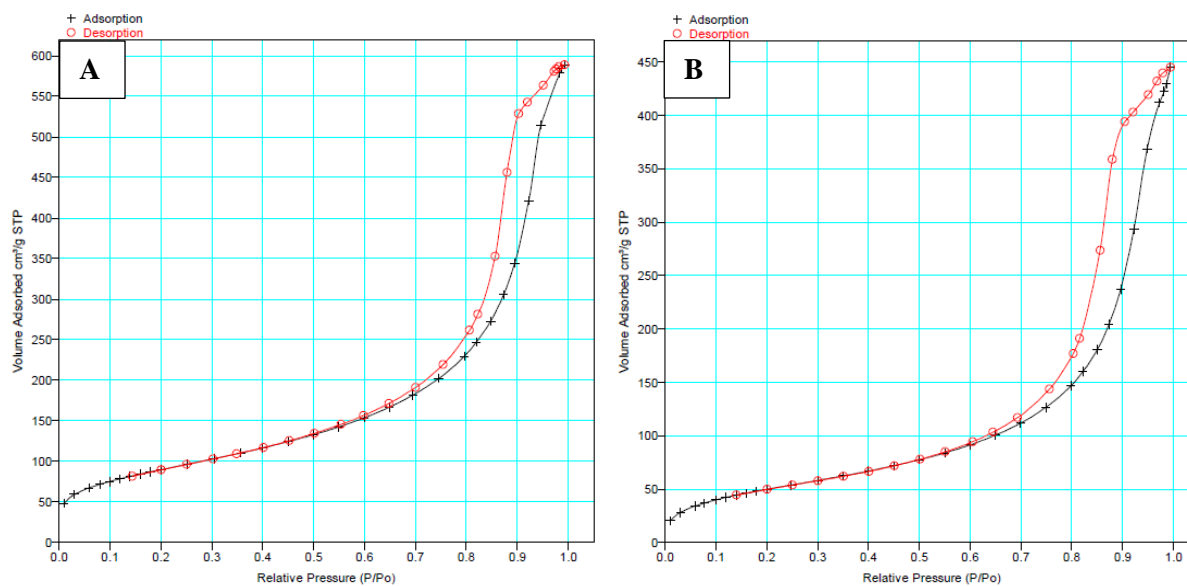
Sample	T <sub>onset</sub> [°C]	5% weight loss [°C]	10% weight loss [°C]
TS	248.01	197.73	991.35
TS_L	221.71	230.74	320.94
TS_1.1_Pt	377.82	307.09	405.48
TS_1.1_Rh	375.16	338.09	405.40
TS_2.1_Pt	358.16	268.98	384.92
TS_2.1_Rh	378.80	243.94	391.01
TS_3.1_Pt	355.64	304.07	418.97
TS_3.1_Rh	376.72	383.54	430.73
TS_4.1_Pt	270.30	257.88	294.49
TS_4.1_Rh	270.98	263.93	296.29
TS_L_3.1_Pt	278.94	262.56	354.53
TS_L_3.1_Rh	273.68	261.96	350.05
TS_L_4.1_Pt	208.47	228.47	280.89
TS_L_4.1_Rh	200.11	224.38	284.27
TS_1.2_Pt	380.55	385.53	442.25
TS_1.2_Rh	376.13	382.25	439.80
TS_2.2_Pt	344.11	351.91	436.16
TS_2.2_Rh	340.65	365.06	480.17
TS_3.2_Pt	358.36	365.43	465.81
TS_3.2_Rh	363.19	381.88	470.12
TS_4.2_Pt	200.21	245.61	418.45
TS_4.2_Rh	200.95	256.98	447.56
TS_L_3.2_Pt	239.51	270.77	347.96
TS_L_3.2_Rh	237.42	270.98	345.74
TS_L_4.2_Pt	198.96	219.43	269.77
TS_L_4.2_Rh	199.27	222.00	277.66

## 8.2. Surface area and porosity characteristic of supports

**Table S4.** Surface area and porosity characteristic of obtained SILP materials

<b>Sample</b>	<b>BET Surface Area [m<sup>2</sup>/g]</b>	<b>Total Pore Volume [cm<sup>3</sup>/g]</b>	<b>Average Pore Diameter [nm]</b>	<b><sup>1</sup><math>\alpha</math></b>	<b><sup>2</sup>Layer thickness [nm]</b>
TS	328	0.91	10.95	-	-
TS <sub>0</sub>	464	0.93	7.97	-	-
TS_L	176	0.51	11.43	-	-
TS_1.1_Rh	213	0.71	10.95	0.22	0.65
TS_2.1_Rh	166	0.55	10.19	0.39	0.51
TS_3.1_Rh	154	0.61	12.49	0.33	0.47
TS_4.1_Rh	209	0.69	10.58	0.23	0.64
TS_1.1_Pt	214	0.65	11.93	0.28	0.65
TS_2.1_Pt	204	0.69	11.25	0.24	0.62
TS_3.1_Pt	189	0.69	12.36	0.24	0.57
TS_4.1_Pt	210	0.69	11.38	0.24	0.64
TS_L_3.1_Rh	82	0.36	14.27	0.29	0.46
TS_L_4.1_Rh	81	0.37	13.66	0.27	0.46
TS_L_3.1_Pt	82	0.36	13.71	0.29	0.46
TS_L_4.1_Pt	90	0.38	13.43	0.25	0.51
TS_1.2_Rh	230	0.66	11.21	0.27	0.70
TS_2.2_Rh	241	0.70	10.95	0.20	0.70
TS_3.2_Rh	211	0.645	10.24	0.26	0.61
TS_4.2_Rh	255	0.72	10.24	0.19	0.74
TS_1.2_Pt	224	0.66	9.92	0.27	0.68
TS_2.2_Pt	241	0.69	9.75	0.20	0.70
TS_3.2_Pt	222	0.67	10.26	0.23	0.65
TS_4.2_Pt	246	0.72	10.37	0.18	0.72
TS_L_3.2_Rh	97	0.39	12.73	0.23	0.55
TS_L_4.2_Rh	116	0.45	13.01	0.11	0.66
TS_L_3.2_Pt	106	0.41	13.95	0.19	0.60
TS_L_4.2_Pt	127	0.45	12.22	0.11	0.72

<sup>1</sup>Pore filling degree of support as the ratio IL volume/support pore volume. <sup>2</sup>Ratio of the IL volume used for coating and the initial surface area.



**Fig. S17.** Examples of adsorption and desorption isotherms for  $\text{TiO}_2\text{-SiO}_2$  (A) and the SILP system  $\text{TS}_{3.1}\text{-Pt}$  (B).

### 8.3. SEM-EDX analysis

**Table S5.** SEM-EDX analysis of SILP samples with higher catalyst concentration.

Sample	Element content [wt %]								
	C	O	F	Si	P	S	Ti	Rh	Pt
TS_4.1_Rh_H	3.38	45.03	2.77	27.62	0.36	2.03	18.39	0.25	-
TS_4.1_Pt_H	2.46	43.87	2.16	28.57	-	1.95	20.18	-	0.29

### 8.4. Elemental analysis

**Table S6.** Elemental analysis of obtained SILP materials.

Sample	N [%]	C [%]	H [%]	S [%]	Coverage rate P [ $\mu\text{mol}/\text{m}^2$ ]
TS	0.023	0.036	0.664	0.112	-
TS_4.1_Pt	0.351	4.083	1.171	2.449	1.54
TS_4.2_Pt	0.018	2.253	0.992	3.073	2.12
TS_L	1.033	10.800	2.510	0.174	-
TS_L_4.1_Pt	1.379	13.095	2.195	4.239	1.51



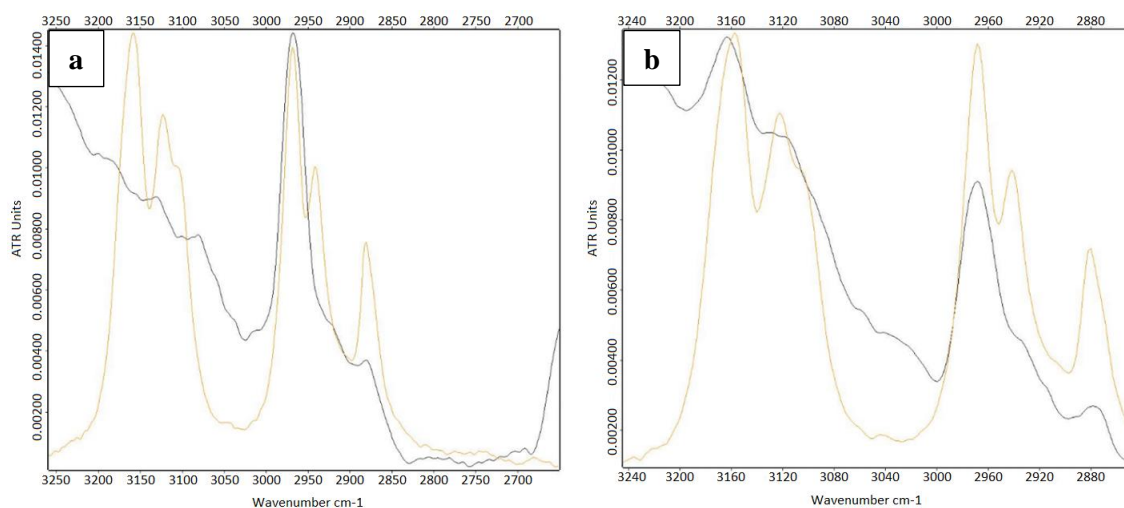
## 8.5. Particle size analysis

**Table S7.** Particle size distribution and maximum volumetric contribution of particles in the supports and SILP materials.

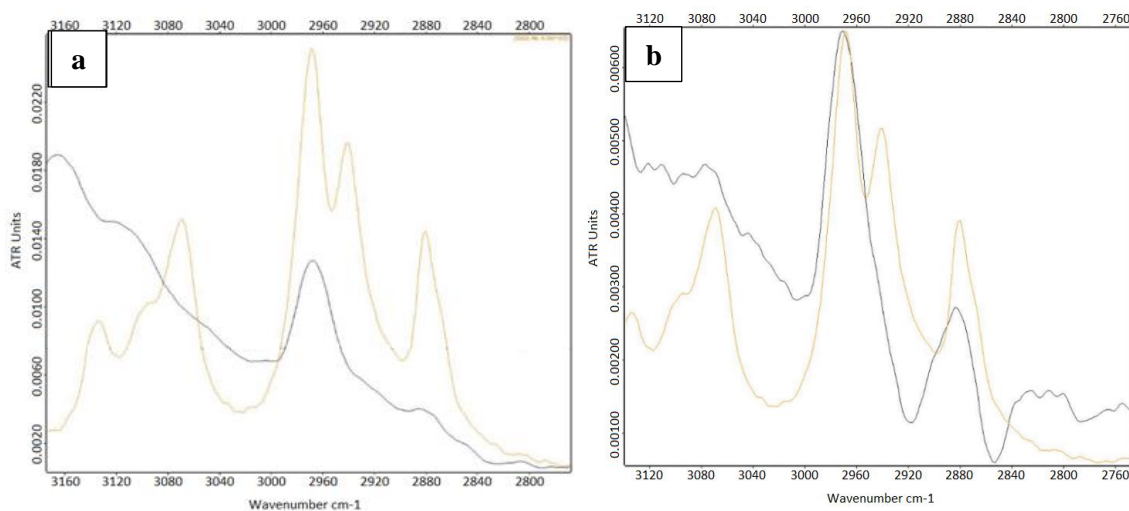
Sample	Particles Size Range [nm]	Average Particle Size [nm]	Maximum Volumetric Particles Participation [%]	PdI <sup>1</sup>
TS	122 – 396 nm	337 nm	220 nm – 33% 255 nm – 32%	0.600
TS_4.1_Pt	59 – 164 nm 342 – 825 nm	295 nm	106 nm – 3% 531 nm – 33%	0.864
TS_L	59 – 142 nm 220 – 715 nm	606 nm	106 nm – 13 % 255 nm – 12 %	0.606
TS_L_4.1_Pt	91– 220 nm 396 – 955 nm	264 nm	142 nm – 3 % 712 nm – 33 %	0.654

<sup>1</sup>Polydispersity Index

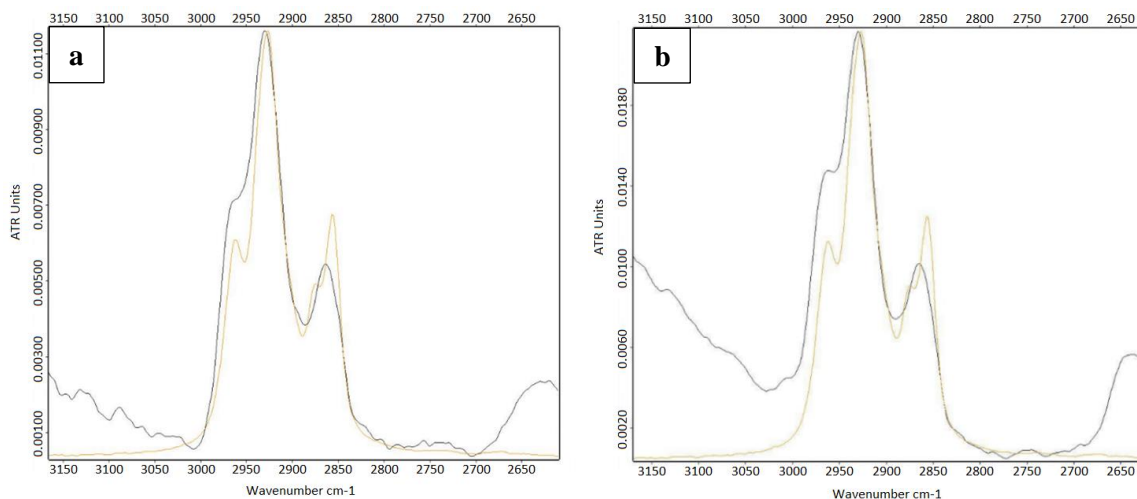
## 8.6. IR spectra of obtained SILP materials



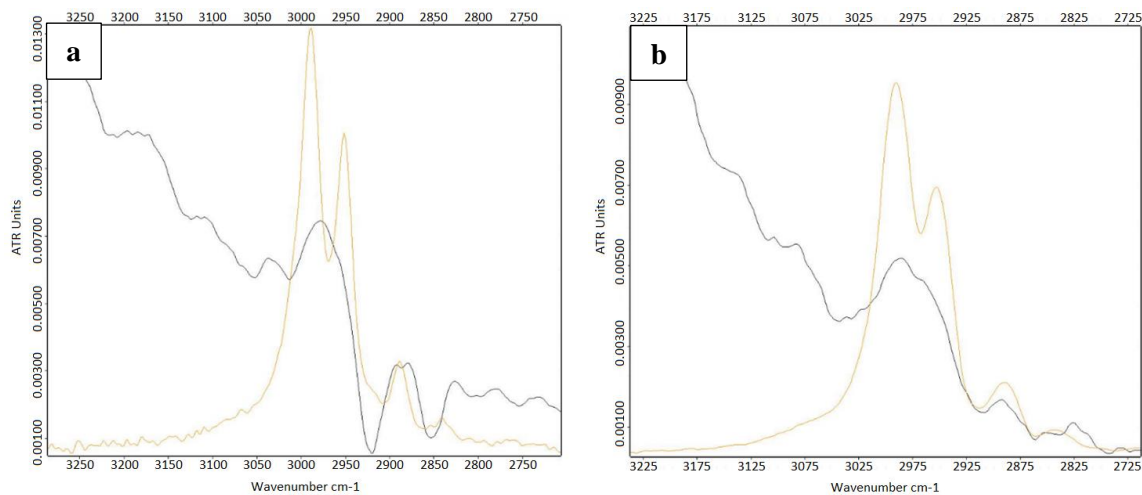
**Fig. S18.** IR spectra of the obtained SILP materials TS\_1.1\_Rh – (a) and TS\_1.1\_Pt (b) (black) and ionic liquid [BMIM][Ntf<sub>2</sub>] (orange).



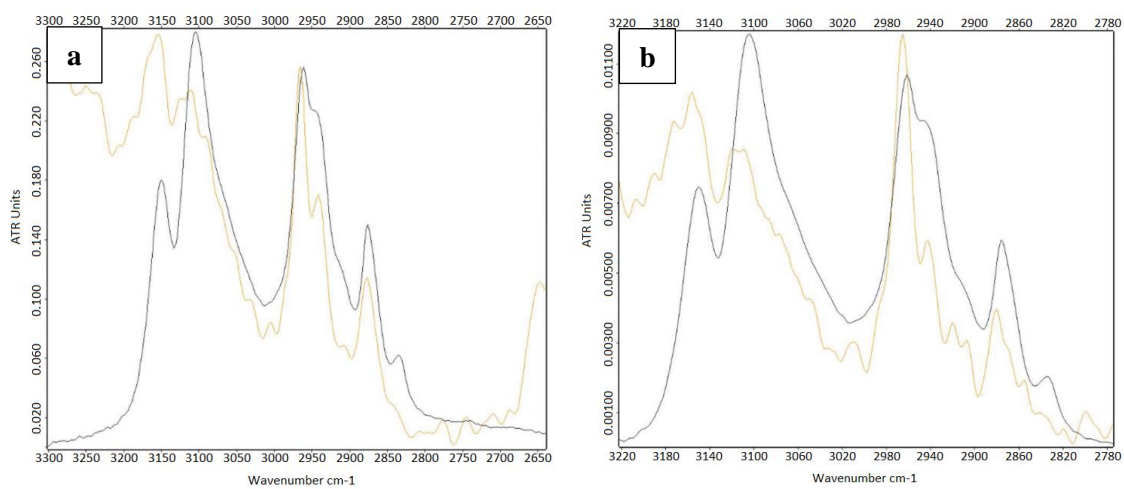
**Fig. S19.** IR spectra of the obtained SILP materials TS\_2.1\_Rh (a) and TS\_2.1\_Pt (b) (black) and ionic liquid [BMPy][Ntf<sub>2</sub>] (orange).



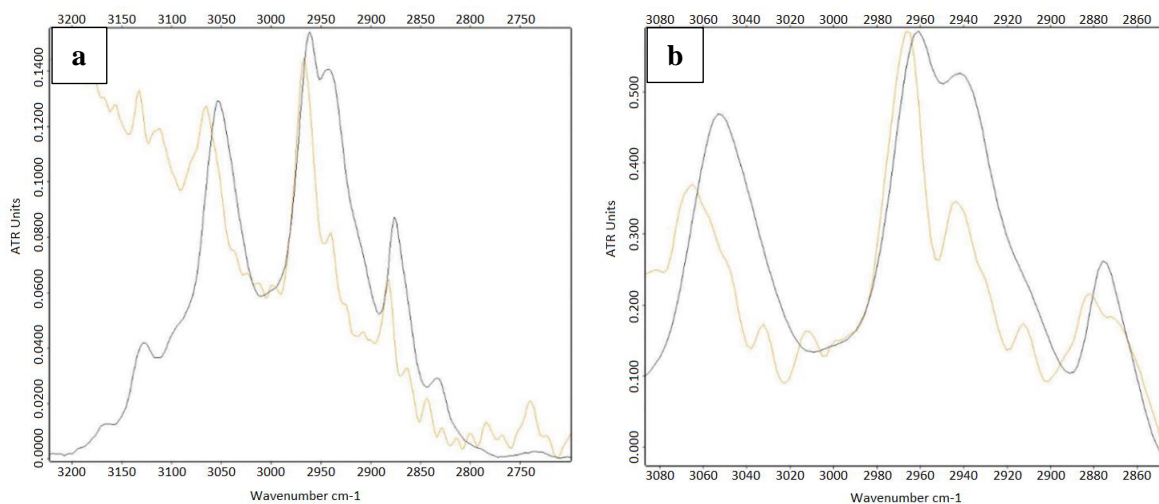
**Fig. S20.** IR spectra of the obtained SILP materials TS\_3.1\_Rh (a) and TS\_3.1\_Pt (b) (black) and ionic liquid [P<sub>44414</sub>][Ntf<sub>2</sub>] (orange).



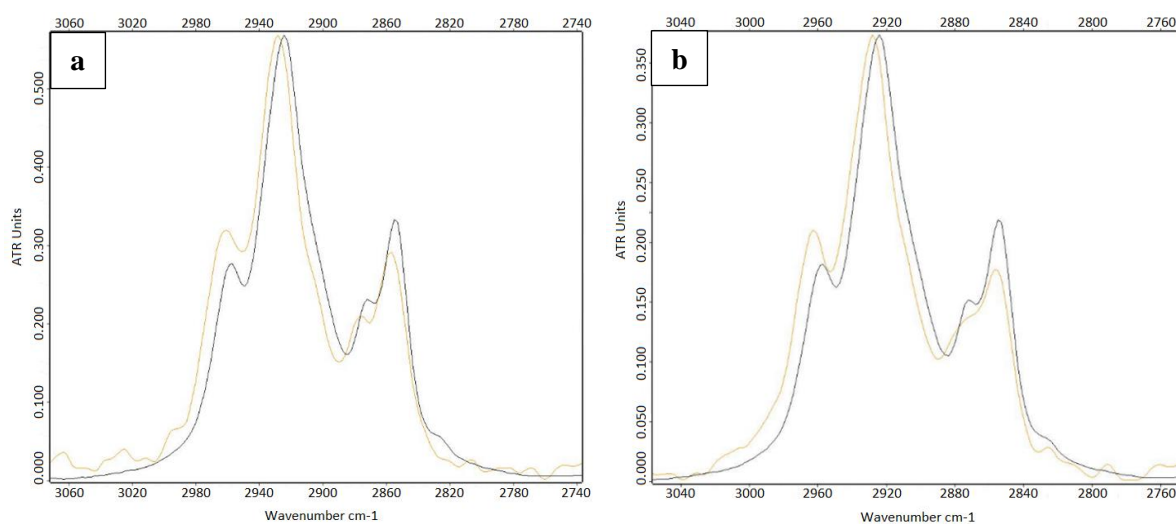
**Fig. S21.** IR spectra of the obtained SILP materials TS\_4.1\_Rh (a) and TS\_4.1\_Pt (b) (black) and ionic liquid [S<sub>222</sub>][Ntf<sub>2</sub>] (orange).



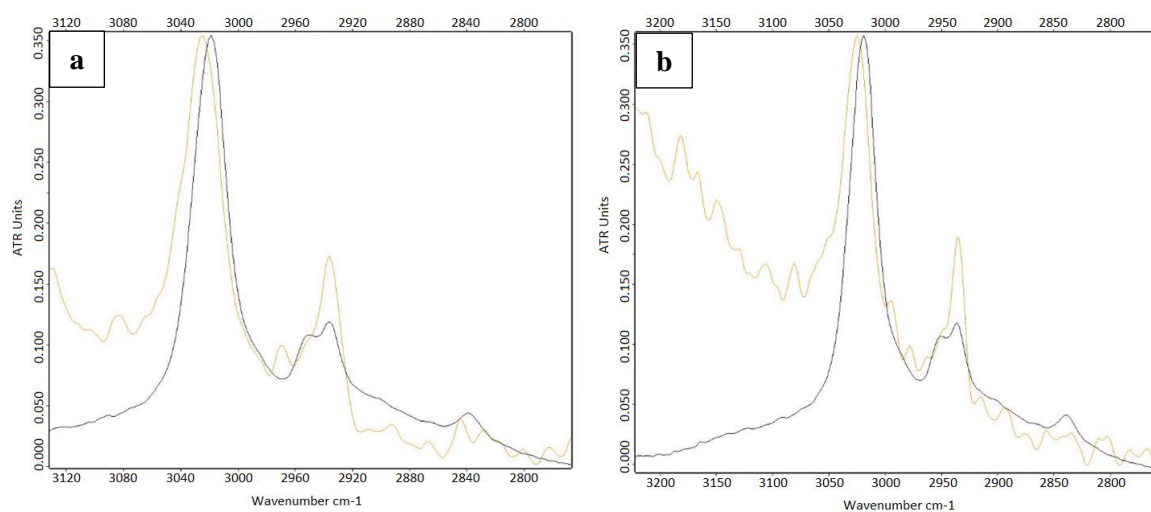
**Fig. S22.** IR spectra of the obtained SILP materials TS\_1.2\_Rh (a) and TS\_1.2\_Pt (b) (orange) and ionic liquid [BMIM][MeSO<sub>4</sub>] (black).



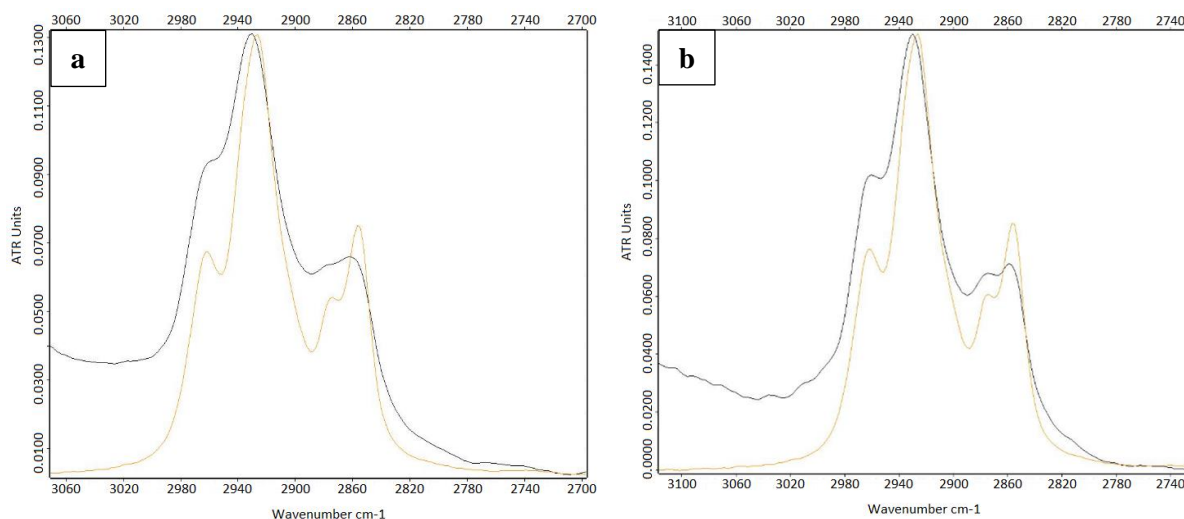
**Fig. S23.** IR spectra of the obtained SILP materials TS\_2.2\_Rh (a) and TS\_2.2\_Pt (b) (orange) and ionic liquid [BMPy][MeSO<sub>4</sub>] (black).



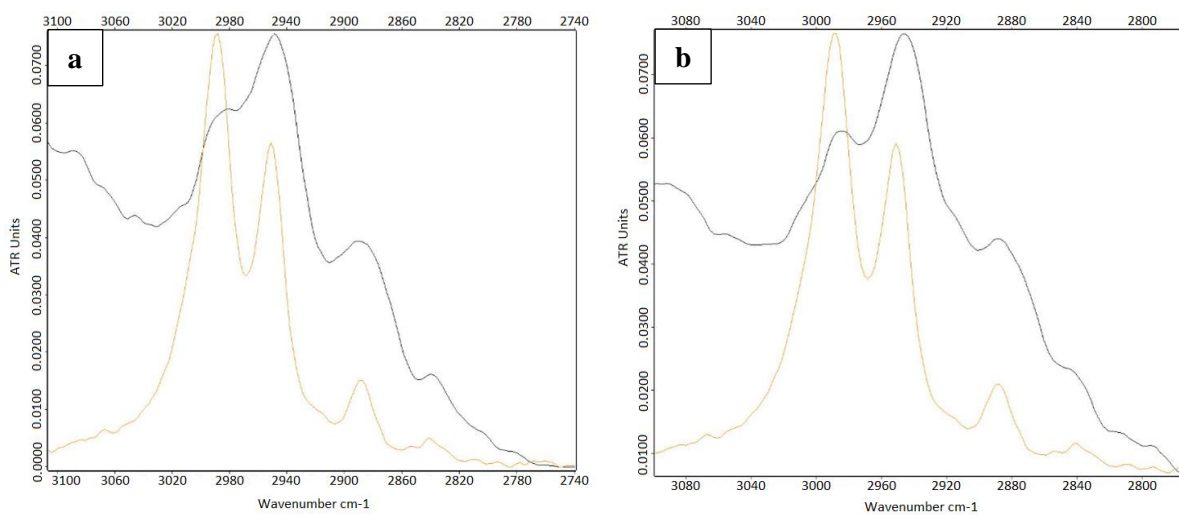
**Fig. S24.** IR spectra of the obtained SILP materials TS\_3.2\_Rh (a) and TS\_3.2\_Pt (b) (orange) and ionic liquid [P<sub>44414</sub>][MeSO<sub>4</sub>] (black).



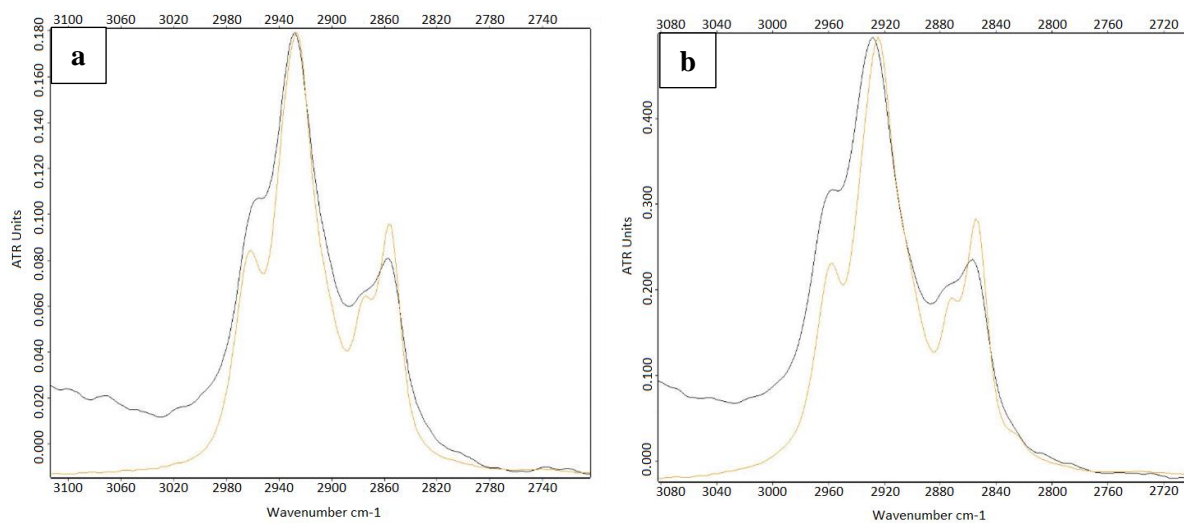
**Fig. S25.** IR spectra of the obtained SILP materials TS\_4.2\_Rh (a) and TS\_4.2\_Pt (b) (orange) and ionic liquid [S<sub>111</sub>][MeSO<sub>4</sub>] (black).



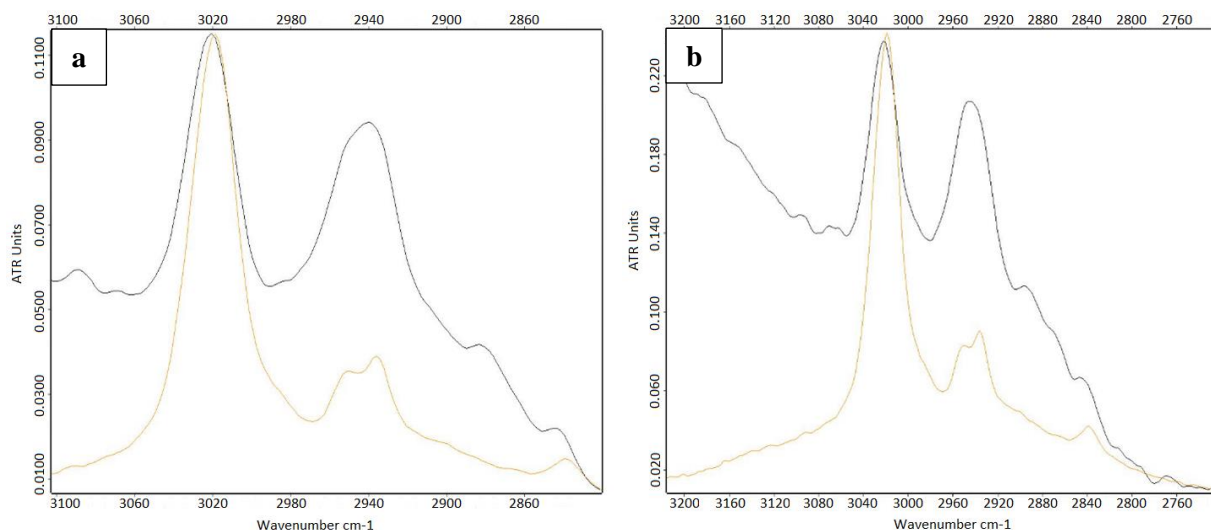
**Fig. S26.** IR spectra of the obtained SILP materials TS\_L\_3.1\_Rh (a) and TS\_L\_3.1\_Pt (b) (black) and ionic liquid [P<sub>44414</sub>][Ntf<sub>2</sub>] (orange).



**Fig. S27.** IR spectra of the obtained SILP materials TS\_L\_4.1\_Rh (a) and TS\_L\_4.1\_Pt (b) (black) and ionic liquid [S<sub>222</sub>][Ntf<sub>2</sub>] (orange).



**Fig. S28.** IR spectra of the obtained SILP materials TS\_L\_3.2\_Rh (a) and TS\_L\_3.2\_Pt (b) (black) and ionic liquid [P<sub>44414</sub>][MeSO<sub>4</sub>] (orange).



**Fig. S29.** IR spectra of the obtained SILP materials TS\_L\_4.2\_Rh (a) and TS\_L\_4.2\_Pt (b) (black) and ionic liquid [S<sub>111</sub>][MeSO<sub>4</sub>] (orange).

## 9. Catalytic tests

Catalytic activity of all obtained Rh-SILP and Pt-SILP materials was tested in the hydrosilylation reaction of 1-octene and 1,1,1,3,5,5,5-heptamethyltrisiloxane (HMTS). For this purpose to the 2 ml reaction vessel 0.1 g Rh or Pt-SILP material (corresponding quantities of 10<sup>-5</sup> mol of Rh or Pt per 1 mol of Si-H) was introduced. Then 3.68 mmol of 1-octene, 3.68 mmol of HMTS and 1 mmol of *n*-decane (as an internal standard) was added. Then the reaction mixture was heated to 100°C for 30 minutes, without stirring. After that time the reaction mixture was left for 10 minutes for better sedimentation process of the SILP particles. Then the reaction mixture was gently removed from the reaction vessel by a needle and syringe. The reaction mixture was then cooled and analysed by Gas Chromatograph. In the next catalytic cycle new portion of substrates was added to the catalytic material remaining in the reactor, without any regeneration. This activity was repeated until basis of GC analysis, the catalytic activity of the tested SILP material had disappeared. For the most active SILP materials hydrosilylation reactions with 1-octene, allyl-glycidyl ether, octafluoropentyl ether and HMTS, triethylsilan and triethoxysilan were additionally conducted. For reactions with ethers 4.41 mmol of olefine and 3.68 mmol of HMTS 1 mmol of *n*-decane was applied. The reaction conditions were analogous to the above. Obtained product was subjected to NMR or GC-MS analysis.

### 9.1. Results of catalytic tests with different amount of IL

**Table S8.** Catalytic activity of SILP materials with different amount of ionic liquid.

Catalytic cycle No	Catalytic cycle					
	TS_3.1_Rh_10%	TS_3.1_Pt_10%	TS_3.1_Rh_15%	TS_3.1_Pt_15%	TS_3.1_Rh_20%	TS_3.1_Pt_20%
1	99	99	99	99	99	99
2	99	99	99	99	99	99
3	88	99	99	99	99	72
4	86	99	76	99	41	63
5	73	99	60	99	21	21
	99-50%		49-30%		29-0%	

## 9.2. Catalytic test with SILP materials based on SiO<sub>2</sub> support

**Table S9.** Catalytic activity SILP materials with SiO<sub>2</sub> support in the hydrosilylation reaction.

SILP	Conversion of Si-H [%]										TON	TOF ·10 <sup>6</sup> h <sup>-1</sup>
	1	2	3	4	5	6	7	8	9	10		
S_4.1_Rh	99	32	25	0	-	-	-	-	-	-	156 000	0.31
S_4.1_Pt	99	85	80	78	72	54	49	33	25	10	585 000	1.17
S_4.2_Rh	83	45	18	0	-	-	-	-	-	-	146 000	0.30
S_4.2_Pt	99	68	25	22	0	-	-	-	-	-	214 000	0.43
99-50%				49-30%						29-0%		

## 10. Spectroscopic characteristic of obtained hydrosilylation reaction product

### 3-octyl-1,1,1,3,5,5,5-heptamethyltrisiloxane:

<sup>1</sup>H NMR (CDCl<sub>3</sub>)ppm: 0.02 (s, 3H, Si-CH<sub>3</sub>), 0.11 (m, 18H, Si-(CH<sub>3</sub>)<sub>3</sub>), 0.48 (t; 2H; Si-CH<sub>2</sub>), 0.9 (t; 3H; CH<sub>2</sub>-CH<sub>3</sub>), 1.36-1.27 (m; 12H; CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>);

<sup>13</sup>C NMR (CDCl<sub>3</sub>)ppm: 0.28 (O-Si-CH<sub>3</sub>), 1.89 (Si-CH<sub>3</sub>), 14.10 (C-CH<sub>3</sub>), 17.63 (C-Si), 22.70 (Si-C-C), 23.07 (C-CH<sub>3</sub>), 29.35, 29.27 (C-C-C), 31.95 (C-C-C), 33.25 (C-C-C);

<sup>29</sup>Si NMR (CDCl<sub>3</sub>)ppm: -2.19 (-O-Si-O-), 6.75 (OSi(CH<sub>3</sub>)<sub>3</sub>).

GC-MS: 319.1 (25%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>OSi(CH<sub>3</sub>)<sub>2</sub>SiCH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>CH<sub>3</sub>]<sup>+</sup>), 221.4 (100%, [OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>SiCH<sub>3</sub>]<sup>+</sup>), 207.7 (14%, [OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>3</sub>Si(CH<sub>3</sub>)<sub>2</sub>SiCH<sub>3</sub>]<sup>+</sup>), 150.3 (3%, [OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>3</sub>SiOCH<sub>3</sub>]<sup>+</sup>). Retention time: 11.380 min.

### 3-(3-glycidyloxypropyl)-1,1,1,3,5,5,5-heptamethyltrisiloxane:

<sup>1</sup>H NMR (CD<sub>3</sub>CN, δ,ppm): 0.05 (m, 3H, -SiCH<sub>3</sub>); 0.13 (m, 18H, -Si(CH<sub>3</sub>)<sub>3</sub>); 0.5 (m, 2H, -Si-CH<sub>2</sub>-); 1.59 (m, J=11,3 Hz, 2H, -Si-CH<sub>2</sub>-CH<sub>2</sub>-); 2.54 (m, J=5,1 Hz, 1H, HC-CH<sub>2</sub>-O); 2.74 (dd, J=5,1 Hz, 1H, HC-CH<sub>2</sub>-O); 3.08 (m, J=6,8 Hz, 1H, HC-O-CH<sub>2</sub>-); 3.27 (dd, J=11,5 Hz, 1H, -O-CH<sub>2</sub>-); 3.43 (m, 2H, -CH<sub>2</sub>-O-CH<sub>2</sub>-); 3.69 (m, J=17,1 Hz, 1H, -O-CH<sub>2</sub>-);

<sup>13</sup>C NMR (CD<sub>3</sub>CN, δ,ppm): -1.0 (-Si-CH<sub>3</sub>), 0.37-1.39 (-Si(CH<sub>3</sub>)<sub>3</sub>); 12.77 (-Si-C-); 23.16 (-Si-C-C-); 43.56 (-C-O-C-); 50.71 (-C-O-C-); 71.41 (-O-C-C-); 73.67 (-C-C-O-);

<sup>29</sup>Si NMR (CD<sub>3</sub>CN, δ,ppm): -20.52 (-O-Si-O-), 8.07 (OSi(CH<sub>3</sub>)<sub>3</sub>).

GC-MS: 294.6 (9%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>SiCH<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>]<sup>+</sup>), 250.5 (17%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>SiCH<sub>3</sub>(CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>)]<sup>+</sup>), 221.3 (100%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>SiCH<sub>3</sub>]<sup>+</sup>), 205.4 (45% [OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>3</sub>SiCH<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>]<sup>2+</sup>). Retention time: 12.325 min.

### 3-(octafluoropentyloxy)propyl-1,1,1,3,5,5,5-heptamethyltrisiloxane:

<sup>1</sup>H NMR (CD<sub>3</sub>CN, δ,ppm): 0.06 (m, 3H, -SiCH<sub>3</sub>), 0.14 (m, 18H, -Si(CH<sub>3</sub>)<sub>3</sub>), 0.51 (m, 2H, -Si-CH<sub>2</sub>-), 1.61 (m, 2H, -Si-CH<sub>2</sub>-CH<sub>2</sub>-), 3.57 (t, J=6.77 Hz, 2H, -CH<sub>2</sub>-O-), 4.01 (m, 2H, -O-CH<sub>2</sub>-CF<sub>2</sub>-), 6.42 (tt, 1H, -CF<sub>2</sub>H).

<sup>13</sup>C NMR (CD<sub>3</sub>CN, δ,ppm): -1.15 (-SiCH<sub>3</sub>), 0.12 – 0.90 (-Si-(CH<sub>3</sub>)<sub>3</sub>), 13.07 (Si-C-C), 23.16 (Si-C-), 66.73 (-O-C-), 74.89 (-C-O-), 108.04, 110.57, 113.52 (O-CH<sub>2</sub>-CF<sub>2</sub>-), 115.71 (-CF<sub>2</sub>H), 117.21 (C-CN).

<sup>29</sup>Si NMR (CD<sub>3</sub>CN, δ,ppm): -21.16 (-O-Si-O-), 7.43 (OSi(CH<sub>3</sub>)<sub>3</sub>).

GC-MS: 480.2 (2%, [(-OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>OSi(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CHF<sub>2</sub>]<sup>+</sup>), 406.4 (1%, [OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>3</sub>OSi(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CHF<sub>2</sub>]<sup>+</sup>), 294.6 (1%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>OCH<sub>3</sub>Si(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>]<sup>+</sup>), 263.1 (3%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>OCH<sub>3</sub>Si(CH<sub>2</sub>)<sub>3</sub>]<sup>+</sup>), 221.4 (100%, [(OSi(CH<sub>3</sub>)<sub>3</sub>)<sub>2</sub>OCH<sub>3</sub>Si]<sup>+</sup>).

Retention time: 10.984 min.

### triethyl-1-octylsilane:

GC-MS: 199.2 (100%, [(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>SiC<sub>8</sub>H<sub>15</sub>]<sup>+</sup>), 186.4 (4%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>5</sub>H<sub>11</sub>]<sup>+</sup>), 172.3 (3%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>4</sub>H<sub>8</sub>]<sup>+</sup>), 116.3 (2%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si]<sup>+</sup>). Retention time: 11.352 min.

### triethyl-1-(3-glycidoxypropyl)silane:

GC-MS: 215.2 (1% [(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>SiCH<sub>2</sub>((CH<sub>2</sub>)<sub>3</sub>OCHOCH<sub>2</sub>)]<sup>+</sup>), 200.4 (2%, [(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>Si((CH<sub>2</sub>)<sub>3</sub>OCHOCH<sub>2</sub>)]<sup>+</sup>), 171.2 (100%, [C<sub>2</sub>H<sub>5</sub>Si(CH<sub>2</sub>)<sub>3</sub>OCHOCH<sub>2</sub>]), 158.2 (2%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>3</sub>H<sub>6</sub>]<sup>+</sup>), 143.2 (46%, [(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>Si((CH<sub>2</sub>)<sub>3</sub>O)]<sup>+</sup>), 115.2 (87%, [(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>OCH<sub>2</sub>]<sup>+</sup>). Retention time: 12.333 min.

*triethyl-1-((octafluoropentyl)oxy)propyl)silane:*

**GC-MS:** 359.9 (1%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>3</sub>H<sub>6</sub>OCH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>H]<sup>+</sup>), 157.3 (3.5%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>3</sub>H<sub>6</sub>]<sup>+</sup>), 144.3 (1%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>2</sub>H<sub>4</sub>]<sup>+</sup>), 115.1 (100%, [(C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si]<sup>+</sup>). Retention time: 10.927 min.

*triethoxy(octyl)silane:*

**GC-MS:** 233.1 (32%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>2</sub>SiC<sub>8</sub>H<sub>17</sub>]<sup>+</sup>), 220.38 (2%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>4</sub>H<sub>8</sub>]<sup>+</sup>), 205.3 (41.5%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>3</sub>H<sub>6</sub>]<sup>+</sup>), 187.2 (43%, [OC<sub>2</sub>H<sub>5</sub>SiC<sub>8</sub>H<sub>17</sub>]<sup>+</sup>), 85.2 (100%, [C<sub>6</sub>H<sub>13</sub>]<sup>+</sup>). Retention time: 11.646 min.

*(3-glycidyloxypropyl)triethoxysilane:*

**GC-MS:** 278.1 (1%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CHOCH<sub>2</sub>]<sup>+</sup>), 249.1 (35%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>2</sub>SiO(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CHOCH<sub>2</sub>]<sup>+</sup>), 221.3 (1%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si(CH<sub>2</sub>)<sub>3</sub>O]<sup>+</sup>), 191.3 (4.8%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiCH<sub>2</sub>CH]<sup>+</sup>), 177.1 (48%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>2</sub>Si(CH<sub>2</sub>)<sub>3</sub>O]<sup>+</sup>), 163.1 (2%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si]<sup>+</sup>), 146.2 (2%, [Si(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CHOCH<sub>2</sub>]<sup>+</sup>), 131.2 (28%, [OC<sub>2</sub>H<sub>5</sub>Si(CH<sub>2</sub>)<sub>3</sub>O]<sup>+</sup>). Retention time: 12.392 min.

*(octafluoropentyl)propyl)triethoxysilane:*

**GC-MS:** 391.0 (100%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>2</sub>Si(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CHF<sub>2</sub>]<sup>+</sup>), 386.5 (3%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>]<sup>+</sup>), 348.3 (3%, [OC<sub>2</sub>H<sub>5</sub>Si(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CHF<sub>2</sub>]<sup>+</sup>), 205.2 (50.5%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>SiC<sub>3</sub>H<sub>6</sub>]<sup>+</sup>), 164.2 (4%, [(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>Si]<sup>+</sup>). Retention time: 10.921 min.

## 11. References

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