sensors

Supplementary

Study of Room Temperature Ionic Liquids as Gas Sensing Materials in Quartz Crystal Microbalances.

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A1. RTILs used in the study

Table S1. Details of the sensing materials used in the study. All data at 293 K and 101.325 Pa except (a) at 298, (b) 373 and (c) 303.15 K.

Sensing material			Sensor thickness			
Identifier	CAS #	Full name	Density (kg/m³)	Ref.	Viscosity (Pa s)	Ref.
[C2mim][otf]	145022-44-2	1-Ethyl-3-methylimidazolium triflate	1386	[1]	0.04	[2]
[C2mim][Tf2N]	174899-82-2	1-Ethyl-3-methylimidazolium bis(trifluoromethylsulphonyl)imide	1526	[3]	0.03	[4]
[C2mim][Ac]	143314-17-4	1-Ethyl-3-methylimidazolium acetate	1103	[5]	0.20	[1]
[C2mim][dca]	370865-89-7	1-Ethyl-3-methylimidazolium dicyanamide	1107	[6]	0.02	[6]
[C4mim][dca]	448245-52-1	1-butyl-3-methylimidazolium dicyanamide	1064	[7]	0.04	[8]
[C4mim][otf]	174899-66-2	1-Butyl-3-methylimidazolium triflate	1306	[9]	0.10	[10]
[C4mim][Ac]	284049-75-8	1-Butyl-3-methylimidazolium acetate	1055	[11]	0.55	[12]
[C4mim][Cl]	79917-90-1	1-butyl-3-methylimidazolium chloride	1074 (a)	[13]	41.00	[14]
[C4mim][Tf2N]	174899-83-3	1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide	1442	[7]	0.06	[15]
[C4mim][Br]	85100-77-2	1-butyl-3-methylimidazolium bromide	1300 (a)	[16]	0.06 (b)	[17]
[C6mim][Tf2N]	382150-50-7	1-hexyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imide	1371	[18]	0.09	[18]
[C6mim][otf]	460345-16-8	1-hexyl-3-methylimidazolium trifluoromethanesulfonate	1235	[19]	0.12 (c)	[10]
[C6mim][PF6]	304680-35-1	1-hexyl-3-methylimidazolium hexafluorophosphate	1295	[20]	0.587	[20]
[C6mim][Br]	85100-78-3	3-hexyl-1-methyl-1H-imidazolium bromide	1228	[21]	6.94	[21]
[C6mim][Cl]	171058-17-6	1-hexyl-3-methylimidazolium chloride	1034	[22]	18.00	[14]
[C8mim][PF6]	304680-36-2	1-octyl-3-methylimidazolium Hexafluorophosphate	1235	[23]	0.98	[24]
[C8mim][Tf2N]	178631-04-4	1-octyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]imide	1328	[25]	0.12	[4]

[C10mim][BF4]	244193-56-4	1-Decyl-3-methylimidazolium tetrafluoroborate	1072	[25]	0.93	[14]
[BmPY][Tf2N]	623580-02-9	1-Butyl-1-methylpiperidinium bis(trifluoromethylsulphonyl)imide	1387	[26]	0.26	[26]
[N4111][Tf2N]	324575-10-2	Butyltrimethylammonium bis(trifluoromethanesulfonyl)imide	1397	[27]	0.14	[27]
[Py41][Tf2N]	223437-11-4	1-Butyl-1-methylpyrrolidinium bis(trifluoromethanesulfonyl)imide	1400	[5]	0.10	[28]
[P4441][Tf2N]	324575-10-2	Tributylmethylphosphonium bis(trifluoromethanesulfonyl)imide	1258	[29]	0.30	[29]
[N1888][Tf2N]	258273-75-5	Methyltrioctylammonium bis(trifluoromethylsulphonyl)imide	1105	[30]	0.68	[30]
TCP	1330-78-5	Tricresyl phosphate	1160	[31]	0.72	[31]
APIEZON-L	8009-03-08	Petrolatum (Vaseline)	896	[32]	64.00	[32]
PEG 1k	25322-68-3	Poly(ethylene glycol) 1,000	1200	[33]	Solid	[34]
OV-17	63148-58-3	Methyl Phenyl Silicone Oil	1092	[35]	0.10	[36]
SIPONATE DS-10	25155-30-0	Sodium dodecylbenzenesulfonate	1000	[37]	Solid	

Table S2 detail the deposition parameters used for each sensor. The materials used in the study were bought from the following companies:

1. Solvents: acetone (99.5%) from Junsei, chloroform (99% with ethanol as stabilizer 0.3% to 1%) and ethanol (99.5%) from wako, methanol (99.8%) from TCI.
2. The VOCs used (hexanol, 2-hexanone, hexanoic acid, and buty acetate) were al bough from TCI.
3. Ionic liquids: all bought from TCI except [C4mim][ac] bought from Sigma Aldrich and [BmPY][T2fN] that was bought from IOLITEC.
4. The conventional films were bought from Shimazdu Corporation except TCP that was bought from TCI.

Table 2. Details of the sensors used in the study.

Coating Solution				Deposition thickness		
Sensing material	Solvent name	Concentration	Deep Coating pull-up Speed	Δ Fs	Δ M	Thickness
		(μg/mL)	(μm/s)	(Hz)	kg	(nm)
[C2mim][otf]	Ethanol	9.59	100	1688	1.39E-09	66.64
[C2mim][T2fN]	Ethanol	9.87	100	1184	1.23E-09	48.11
[C2mim][Ac]	Ethanol	6.71	200	1584	1.04E-09	49.98
[C2mim][dca]	Ethanol	9.38	100	1514	1.08E-09	51.07
[C4mim][dca]	Acetone	12.95	70	1300	1.38E-09	56.04
[C4mim][otf]	Acetone	12.52	50	1149	1.26E-09	48.24
[C4mim][Ac]	Acetone	8.42	500	967	1.24E-09	57.01
[C4mim][Cl]	Chloroform	5.93	200	1006	1.26E-09	46.45
[C4mim][Tf2N]	Acetone	9.74	500	1509	1.62E-09	57.07
[C4mim][Br]	Chloroform	9.43	600	1200	1.28E-09	47.31
[C6mim][Tf2N]	Acetone	9.39	1500	1194	1.67E-09	60.93
[C6mim][otf]	Acetone	10.93	500	1268	1.25E-09	45.32
[C6mim][PF6]	Acetone	9.45	100	1042	1.7E-09	78.71
[C6mim][Br]	Chloroform	9.41	800	1119	1.62E-09	74.21
[C6mim][Cl]	Chloroform	10.40	600	1300	1.81E-09	66.20
[C8mim][PF6]	Acetone	9.35	100	1438	1.27E-09	42.33
[C8mim][Tf2N]	Acetone	9.50	100	1175	1.2E-09	49.65
[C10mim][BF4]	Methanol	9.40	150	1055	1.39E-09	68.58
[BmPY][Tf2N]	Acetone	9.65	100	1182	1.28E-09	50.25
[N4111][Tf2N]	Acetone	9.89	600	1561	1.36E-09	55.98
[Py41][Tf2N]	Acetone	9.55	600	1163	1.45E-09	61.70
[P4441][Tf2N]	Acetone	9.26	100	1293	1.44E-09	81.61
[N1888][Tf2N]	Acetone	9.79	150	1156	1.25E-09	53.14
TCP	Ethanol	10.21	100	1352	1.26E-09	58.90
APIEZON-L	Chloroform	10.55	1600	1339	1.32E-09	67.09
PEG 1k	Chloroform	10.31	1500	1169	1.12E-09	43.62
OV-17	Chloroform	10.06	1500	1179	1.54E-09	63.54
SIPONATE DS-10	Chloroform	8.77	1500	1230	1.13E-09	53.70

A2. Calibration

A2.1. Measurements

A series of calibration experiments were used to analyze the sensitivity of every sensor to the four gases. In these measurements, each sensor was exposed to the four different gases at concentrations of 100%, 50%, 25%, and 10% of the maximum concentration. With these data, the slope, i.e., sensor's sensitivity to that gas, was calculated using a linear least squares method. For some sensors, more measurements were made in order of test the repeatability. Figures S1 to S4 show these measurements. The repeatability was calculated as the root mean squares and it is represented in Table S3. The sensitivities for all gases in frequency shift and resistance shift are in Tables S4 and S5, respectively.

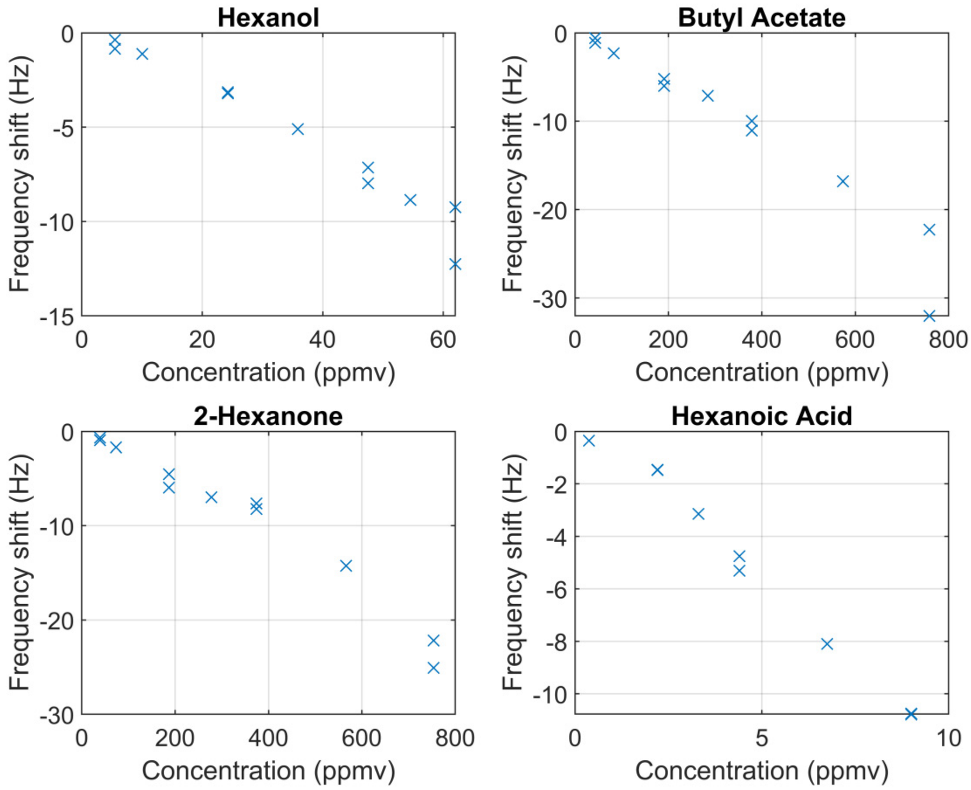

Figure S2. Repeatability measurement for [BmPY][Tf2N]

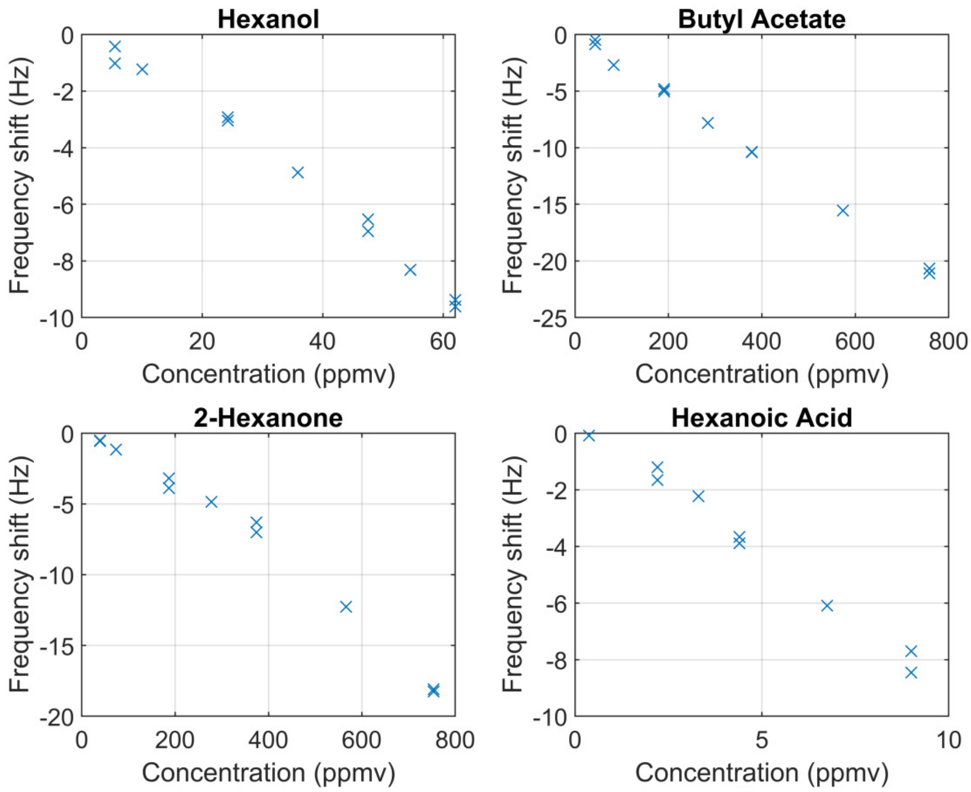

Figure S3. Repeatability measurement for [N1888][Tf2N]

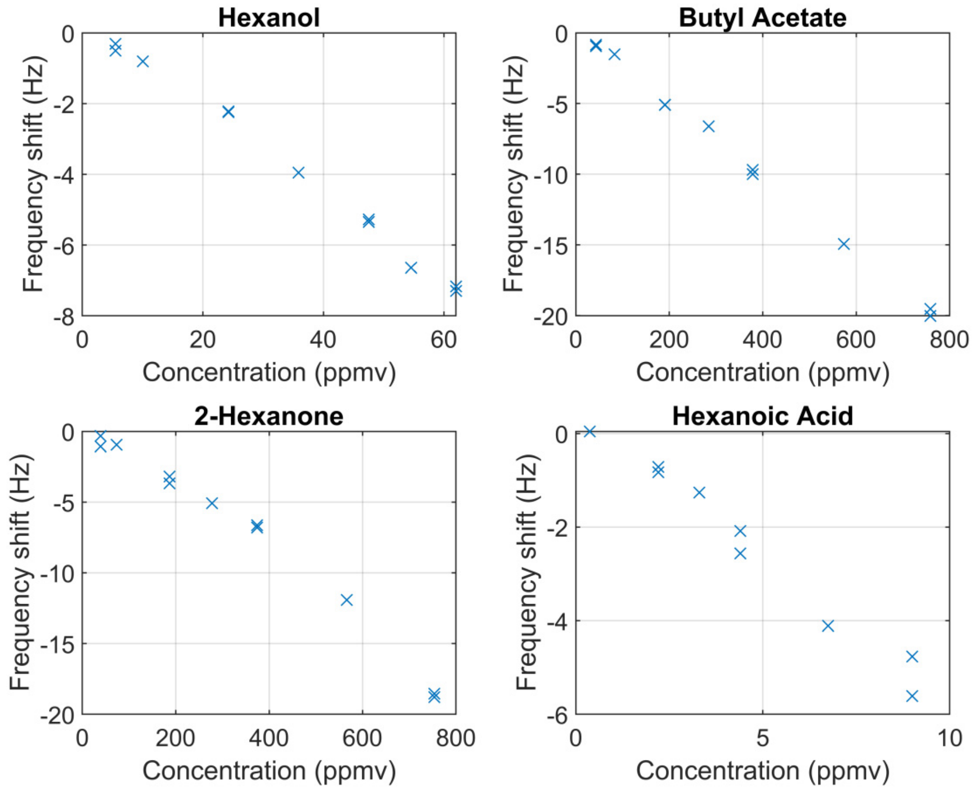

Figure S4. Repeatability measurement for [C8mim][Tf2N]

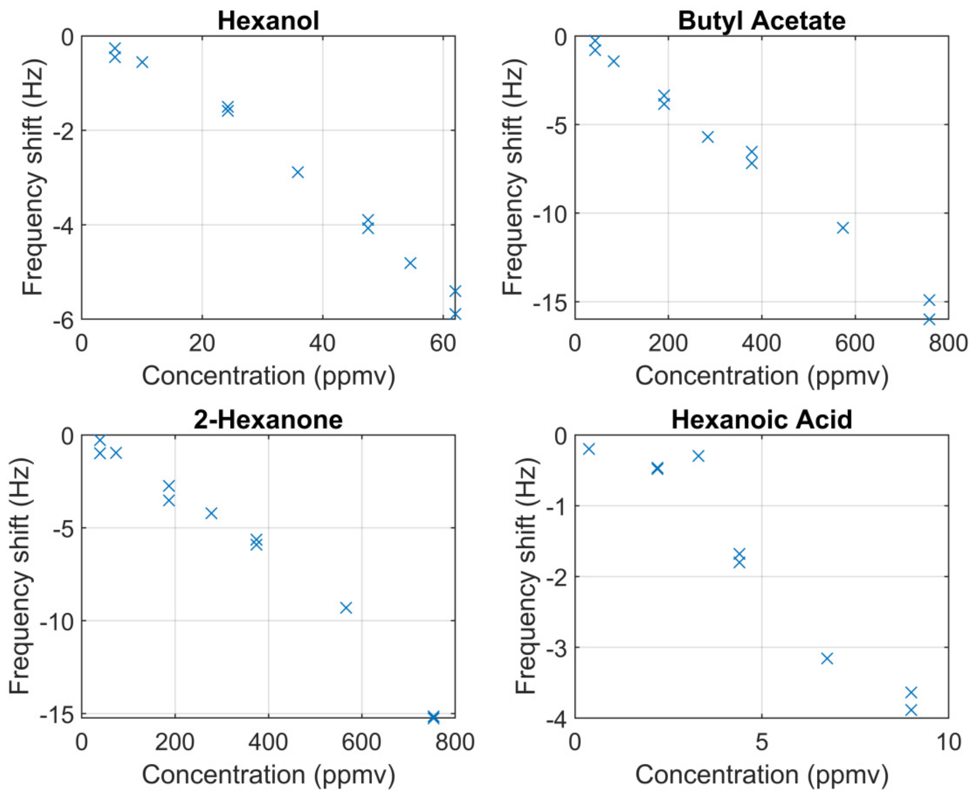

Figure S5. Repeatability measurement for [P4441][Tf2N]

Table S3 shows the repeatability calculated as the root mean square of the differences on frequency response for the same concentration, then divided by the max frequency to obtain the relative root mean square (RRMS). The maximum error was 15% and the average was 5.3 %

Table S3. Measurement repeatability in RRMS (%) of frequency shift for sensors [P4441][Tf2N], [C8mim][Tf2N], [N1888][Tf2N], and [BmPY][Tf2N].

	Hexanol	Butyl Acetate	2-Hexanone	Hexanoic Acid
[P4441][Tf2N]	4.71	1.67	4.04	12.90
[C8mim][Tf2N]	4.54	1.48	1.45	15.39
[N1888][Tf2N]	3.59	2.50	2.74	6.56
[BmPY][Tf2N]	4.11	10.08	6.21	2.97

Table S4 shows all the sensitivities in frequency shift and their R^2 for the linear adjustments. The sensitivity was transformed from Hz/% to Hz/ppmv by dividing by the max concentrations represented in Table 3 and multiplied by 100. Low R^2 could mean high deviation from linearity or poor measurement repetitivity (caused perhaps by slow sensor recovery or sensor drift). Higher sensitivities and high R^2 are desired, as they would increase the precision of the measurements.

Table S4. Sensor sensitivities to the four gases in frequency. The sensitivity to the different gases are marked in bars. Low R^2 are marked in red.

Type	Name	Hexanol		Butyl Acetate		2-Hexanone		Hexanoic Acid	
		Sensitivity (Hz/ppmv)	R^2	Sensitivity (Hz/ppmv)	R^2	Sensitivity (Hz/ppmv)	R^2	Sensitivity (Hz/ppmv)	R^2
RTIL	[C2mim][otf]	-0.1489	0.950	-1.2927	0.930	-1.4606	0.983	-0.0395	0.987
	[C2mim][Tf2N]	-0.0071	0.926	-0.1086	0.972	-0.0791	0.970	-0.0004	0.899
	[C2mim][Ac]	-0.7010	0.755	-10.2167	0.777	-3.7312	0.932	-0.0195	0.970
	[C2mim][dca]	-0.0286	0.995	-0.3317	0.986	-0.2260	0.972	-0.0023	0.880
	[C4mim][dca]	-0.0321	0.999	-0.9557	0.998	-0.9752	0.976	-0.0031	0.998
	[C4mim][otf]	-0.0273	0.997	-0.8313	0.999	-0.8058	0.979	-0.0019	0.997
	[C4mim][Ac]	-0.0241	0.996	-0.8711	1.000	-0.9113	0.981	-0.0015	0.984
	[C4mim][Cl]	-0.0309	0.984	-1.0873	1.000	-1.1356	0.983	-0.0024	0.992
	[C4mim][Tf2N]	-0.1804	0.996	-2.0745	0.944	-1.3524	0.998	-0.0136	0.968
	[C4mim][Br]	-0.3101	0.999	-1.1668	0.819	-1.9565	0.999	-0.0570	0.989
	[C6mim][Tf2N]	-0.1961	0.976	-2.4044	0.967	-1.9088	0.996	-0.0279	0.988
	[C6mim][otf]	-0.1014	0.681	-1.5802	0.986	-1.1638	0.958	0.0446	0.955
	[C6mim][Br]	-0.3609	0.897	-2.2214	0.912	-2.3198	1.000	-0.0134	0.964
	[C6mim][Cl]	-0.1550	0.963	-0.6525	0.881	-0.9212	1.000	-0.0181	0.992
	[C6mim][PF6]	-0.0192	0.956	-0.4743	0.998	-0.4694	0.985	-0.0021	0.993
	[C8mim][PF6]	-0.0436	0.988	-0.7331	0.990	-0.8275	0.987	-0.0070	0.979
	[C8mim][Tf2N]	-0.0274	0.994	-0.5796	0.998	-0.5414	0.978	-0.0025	0.975
	[C10mim][BF4]	-0.1043	0.966	-0.7098	0.994	-0.7659	0.997	-0.0133	0.845
	[BmPY][Tf2N]	-0.0396	0.960	-0.7801	0.929	-0.6660	0.958	-0.0050	0.989
	[N4111][Tf2N]	-0.1011	0.950	-1.3395	0.870	-0.7758	0.999	-0.0201	0.989
	[Py41][Tf2N]	-0.4551	0.916	-2.5746	0.929	-1.7941	0.958	-0.1068	0.990
	[P4441][Tf2N]	-0.0210	0.986	-0.4479	0.993	-0.4330	0.976	-0.0018	0.936
	[N1888][Tf2N]	-0.0346	0.990	-0.6096	0.998	-0.5312	0.981	-0.0037	0.990
Conventional films	TCP	-0.0094	0.870	-0.2335	0.982	-0.1059	0.954	-0.0007	0.992
	Apiezon-L	-0.0067	0.951	-0.3538	0.943	-0.1836	0.984	0.0003	0.976
	PEG-1k	-0.0353	0.992	-0.3721	0.907	-0.2331	0.986	-0.0015	0.971
	OV-17	-0.0080	0.970	-0.1911	0.761	-0.0891	0.907	-0.0003	0.778
	Siponate DS-10	-0.0117	0.693	0.3250	0.981	0.2273	0.957	-0.0019	0.955

In general, the sensitivity of the RTIL is higher than that of the conventional films, indicating they are a good choice for gas sensors. From these measurements, [C2mim][Ac], [Py41][Tf2N], [C4mim][Br], [C6mim][Br], [C6mim][Tf2N], and [C2mim][otf] seem appropriate sensors, as their sensitivity and R^2 are both high. [C2mim][Ac] particularly shows a very high response, even if the R^2 is not very good. Some sensors have a small response: [C2mim][Tf2N] and [C2mim][dca]. Other sensors have a different behavior depending on the gases; [C4mim][dca], [C4mim][otf], [C4mim][Ac], [C4mim][Cl], [C8mim][Tf2N], and [P4441][Tf2N] respond well to 2-hexanone and butyl acetate but little to hexanoic acid and hexanol; the opposite happens to [C4mim][Tf2N], [Py41][Tf2N] and the conventional films. No conclusion could be drawn about the effect of the anions size or the cations size on the sensitivity.

The same analysis was made for the resistance measurements (Table S5). Sensors that have high overall resistance sensitivities and high R^2 (good linearity of the calibration and low noise) are: [BmPY][Tf2N], [N1888][Tf2N], [C8mim][Tf2N], [P4441][Tf2N], for RTILs, and TCP in conventional films. [C2mim][Tf2N], [C4mim][Ac], [C4mim][Br] have small responses.

Table S5. Sensor sensitivities to the four gases in resistance. The sensitivity to the different gases are marked in bars. Low R^2 are marked in red.

Type		Hexanol		Butyl Acetate		2-Hexanone		Hexanoic Acid	
		Sensitivity (Ω/ppmv)	R^2	Sensitivity (Ω/ppmv)	R^2	Sensitivity (Ω/ppmv)	R^2	Sensitivity (Ω/ppmv)	R^2
RTIL	[C2mim][otf]	0.0005	0.617	0.0132	0.891	0.0131	0.973	0.0001	0.881
	[C2mim][Tf2N]	-0.0001	0.204	0.0004	0.101	0.0005	0.171	0.0000	0.040
	[C2mim][Ac]	-0.0333	0.230	0.1881	0.463	0.0249	0.943	0.0001	0.819
	[C2mim][dca]	0.0011	0.938	0.0187	0.968	0.0119	0.971	0.0001	0.946
	[C4mim][dca]	0.0003	0.781	0.0080	0.999	0.0096	0.965	0.0000	0.792
	[C4mim][otf]	0.0002	0.902	0.0051	0.999	0.0063	0.998	0.0000	0.829
	[C4mim][Ac]	0.0000	0.009	0.0027	0.933	0.0039	0.874	0.0000	0.731
	[C4mim][Cl]	0.0003	0.997	0.0133	0.986	0.0174	0.982	0.0000	0.346
	[C4mim][Tf2N]	0.0002	0.754	0.0050	0.949	0.0030	0.821	0.0000	0.155
	[C4mim][Br]	0.0049	0.727	0.0004	0.000	0.0066	0.309	0.0007	0.319
	[C6mim][Tf2N]	0.0010	0.893	0.0148	0.997	0.0124	0.970	0.0001	0.959
	[C6mim][otf]	0.0194	0.623	0.0185	0.031	0.0275	0.663	0.0123	0.910
	[C6mim][Br]	0.0147	0.876	0.0970	0.988	0.0536	0.984	0.0001	0.980
	[C6mim][Cl]	0.0013	0.976	0.0127	0.814	0.0110	0.986	0.0002	0.988
	[C6mim][PF6]	0.0001	0.514	0.0048	0.901	0.0061	0.979	0.0000	0.843
	[C8mim][PF6]	0.0008	0.959	0.0189	0.985	0.0228	0.970	0.0001	0.985
	[C8mim][Tf2N]	-0.0274	0.994	-0.5796	0.998	-0.5414	0.978	-0.0025	0.975
	[C10mim][BF4]	0.0050	0.994	0.0179	0.066	0.0302	0.918	0.0020	0.936
	[BmPY][Tf2N]	-0.0396	0.960	-0.7801	0.929	-0.6660	0.958	-0.0050	0.989
	[N4111][Tf2N]	-0.0047	0.307	-0.0016	0.002	0.0085	0.704	0.0001	0.474
	[Py41][Tf2N]	0.0042	0.394	0.0198	0.053	-0.0237	0.738	-0.0002	0.535
	[P4441][Tf2N]	-0.0210	0.986	-0.4479	0.993	-0.4330	0.976	-0.0018	0.936
	[N1888][Tf2N]	-0.0346	0.990	-0.6096	0.998	-0.5312	0.981	-0.0037	0.990
Conventional films	TCP	0.0021	0.979	0.0420	1.000	0.0353	0.970	0.0001	0.961
	Apiezon-L	0.0002	0.658	0.0007	0.673	0.0011	0.575	0.0000	0.746
	PEG-1k	0.0009	0.960	0.0090	0.929	0.0066	0.990	0.0000	0.901
	OV-17	0.0000	0.053	0.0035	0.869	0.0015	0.924	0.0000	0.131
	Siponate DS-10	0.0001	0.627	0.0001	0.001	0.0028	0.952	0.0000	0.547

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