Electronic Supplementary Material (ESI) for RSC Advances

Supporting Information

Aqueous Biphasic Systems: A Benign Route using Cholinium-Based Ionic Liquids

Shahla Shahriari,[Υ,a,b] Liliana C. Tomé,[a,c] João M. M. Araújo,[Υ,c] Luís Paulo N. Rebelo,[c] João A. P. Coutinho[a], Isabel M. Marrucho[*a,c] and Mara G. Freire*[a,c]

[a]Departamento de Química, CICECO (www.ciceco.ua.pt), Universidade de Aveiro, 3810-193 Aveiro, Portugal.

[b]Department of Chemical Engineering, Islamic Azad University, Shahr-e-Qods Branch, Tehran, Iran.

[c]Instituto de Tecnologia Química e Biológica, ITQB2, Universidade Nova de Lisboa (www.itqb.unl.pt), Av. República, Apartado 127, 2780-901 Oeiras, Portugal.

[Υ]Equally contributing authors.

*Corresponding author

Tel: +351 214469441; Fax: +351 214411277; E-mail address: imarrucho@itqb.unl.pt, maragfreire@ua.pt.

Table S1. Weight fraction data for the ternary system composed of [Ch]Cl (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[Ch]Cl			
100 w_1	100 w_2	100 w_1	100 w_2
31.0807	18.1274	11.2800	33.4940
27.4307	21.1342	10.9914	33.8145
24.7816	22.6430	10.6994	34.2173
22.9733	23.7823	10.4137	34.5027
22.1106	24.3677	10.1464	34.8022
21.3207	24.9459	9.9269	34.9697
20.4478	25.6316	9.6793	35.1943
19.8120	26.0186	9.3999	35.5710
18.8014	27.0334	9.1613	35.8192
18.2768	27.3941	8.7645	35.9955
17.7600	27.7600	8.4962	36.3929
16.9308	28.5547	25.3290	22.3926
16.5120	28.7739	27.3644	20.3549
15.8399	29.4370	29.5319	18.9057
15.2173	30.1102	30.7425	17.9073
14.8100	30.4219	31.8267	17.0329
14.3945	30.7688	34.3204	15.7149
14.0467	31.0182	37.0261	14.3675
13.7073	31.2917	37.6994	13.4084
13.3728	31.5385	40.1490	12.2682
12.9115	32.0163	42.2652	11.0472
12.6228	32.1945	44.4110	10.0112
12.2358	32.6255	50.3648	7.8594
11.9758	32.7806	54.0834	6.1509
11.6359	33.1351		

Table S2. Weight fraction data for the ternary system composed of [Ch][Lev] (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[Ch][Lev]			
100 w_1	100 w_2	100 w_1	100 w_2
9.8373	33.7379	29.2488	16.6588
10.8128	32.4690	29.9132	16.2310
11.8218	31.4440	30.0675	15.9353
14.1676	29.8203	31.4628	15.3719
15.1268	29.0729	32.1408	14.9663
16.8214	27.7232	32.6066	14.3953
18.2691	26.5052	32.9402	14.0666
18.6510	25.8648	33.7109	13.7024
19.2679	25.1564	35.5511	13.1167
20.5059	24.1747	35.5892	12.4803
21.6411	23.0555	36.9547	11.8730
23.2572	21.7217	37.5496	11.2938
24.4018	20.5951	38.7510	10.6214
24.8108	20.1060	39.6219	10.0631
25.7246	19.5962	40.4251	9.6564
26.6152	19.0014	41.7899	9.1856
26.9407	18.6130	42.2028	8.8137
27.8019	18.0566	42.4678	8.5827
27.9056	17.6890	43.4454	7.8415
28.5959	17.1518	45.1992	7.1801

Table S3. Weight fraction data for the ternary system composed of [Ch][Glu] (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[Ch][Glu]			
100 w_1	100 w_2	100 w_1	100 w_2
16.3777	31.1019	12.0810	34.0495
16.0130	31.2440	11.5445	34.7626
15.7501	31.4553	11.1362	34.8785
15.2380	31.8482	10.7793	34.9143
14.7366	32.2227	10.2796	35.3874
14.3147	32.5577	9.3993	36.7562
13.8786	32.9221	21.8122	27.9730
13.4975	33.2454	30.5284	23.3701
13.0570	33.3136	35.0579	20.3596
12.3754	33.8418		

Table S4. Weight fraction data for the ternary system composed of [Ch][Suc] (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[Ch][Suc]			
100 w_1	100 w_2	100 w_1	100 w_2
12.7599	33.1821	8.7412	37.0507
12.2553	33.7777	8.4731	37.0584
11.6729	34.2480	8.0773	37.7527
11.2753	34.3653	7.6119	38.2467
10.7041	34.9487	7.1680	38.5260
10.3546	35.3603	9.8334	36.0308
9.6568	35.9372	13.9719	32.8335
9.2611	36.5555	29.5240	23.3187

Table S5. Weight fraction data for the ternary system composed of [Ch][Ac] (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[Ch][Ac]			
100 w_1	100 w_2	100 w_1	100 w_2
31.6476	18.3572	14.2371	29.8045
29.6005	19.2785	13.8845	30.0448
27.8208	20.1399	13.5077	30.3409
26.6496	20.8964	13.1741	30.5847
25.8706	21.2578	12.9135	30.7058
24.9365	21.8259	12.6245	30.9277
23.9054	22.6236	12.2885	31.2506
23.1927	22.9956	12.0853	31.3420
22.4218	23.5432	11.7772	31.6313
21.5986	24.1576	11.5784	31.7321
20.7941	24.7302	11.4210	31.8897
19.9479	25.4675	11.2505	31.9525
19.2317	25.9997	11.1412	32.0856
18.5387	26.5233	10.9110	32.2369
17.9108	27.0230	10.6581	32.4341
17.5158	27.2564	10.4529	32.5997
16.7793	27.8888	10.2410	32.7507
16.2651	28.2890	9.9978	32.9825
15.8012	28.6487	9.7893	33.1480
15.4713	28.8569	9.5570	33.3709
15.1412	29.1116	9.3246	33.6081
14.6432	29.4792		

Table S6. Weight fraction data for the ternary system composed of [Ch][Sal] (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[Ch][Sal]			
100 w_1	100 w_2	100 w_1	100 w_2
76.2431	1.9687	21.7720	18.9174
63.6986	3.3666	20.2539	19.7512
54.5508	4.9457	18.9609	20.5343
51.6854	5.5305	17.7784	21.2220
50.1838	6.3404	16.7355	21.8011
46.4232	7.7320	15.6094	22.5132
45.5155	7.5084	13.9205	23.5769
43.3698	8.8926	13.2748	23.9272
42.8357	8.1883	11.9115	24.9074
39.3379	9.8034	10.9015	25.5524
38.1139	10.4157	10.4195	25.8998
34.8756	12.0234	9.9986	26.2043
34.5809	11.7913	9.1091	26.9015
32.2066	12.8155	8.8328	27.0956
31.9328	13.4358	8.4230	27.4559
30.1948	13.7483	7.9960	27.8379
29.4974	14.6243	7.2774	28.5126
29.3698	14.2374	6.6277	29.1566
28.5010	14.6608	5.6444	30.2529
27.4032	15.6451	4.9555	31.1175
25.0612	17.0961	4.3869	31.8268
23.5047	17.8358	3.6636	32.9181

Table S7. Weight fraction data for the ternary system composed of [BCh]Cl (1) + K_3PO_4 (2) + H_2O (3) at 298 K.

[BCh]Cl			
100 w_1	100 w_2	100 w_1	100 w_2
66.7029	1.8377	14.7708	25.2346
53.9583	4.8519	14.3820	25.3951
51.8869	5.3601	13.9172	25.6354
48.8018	6.2236	12.5024	26.8381
46.0772	6.8129	12.1721	27.0108
44.3195	7.3867	11.8818	27.1388
39.1718	10.5973	11.6184	27.8624
37.1387	11.1209	11.5702	27.2891
35.7791	11.6813	11.4092	27.9340
34.6459	12.1438	11.1352	28.0782
33.1914	12.8580	10.9391	28.1456
32.0684	13.3498	10.4937	28.3266
27.1624	16.9826	8.6755	30.0776
26.3222	17.2949	7.9554	30.7711
25.4641	17.6456	7.6729	31.0091
24.4040	18.1612	7.5115	31.0678
23.4029	18.6732	7.4002	31.1034
22.4889	19.1455	7.2607	31.1668
21.6512	19.5859	7.0376	31.3158
18.3653	22.3223	6.7650	31.4366
17.7925	22.6025	6.6482	31.4977
17.7240	23.0905	6.4126	31.6271
17.2038	23.3238	6.3266	31.6638
16.7328	23.5465	6.2132	31.7192
16.1753	23.8367	6.0211	31.8085
15.7281	24.0220	5.8881	31.8884

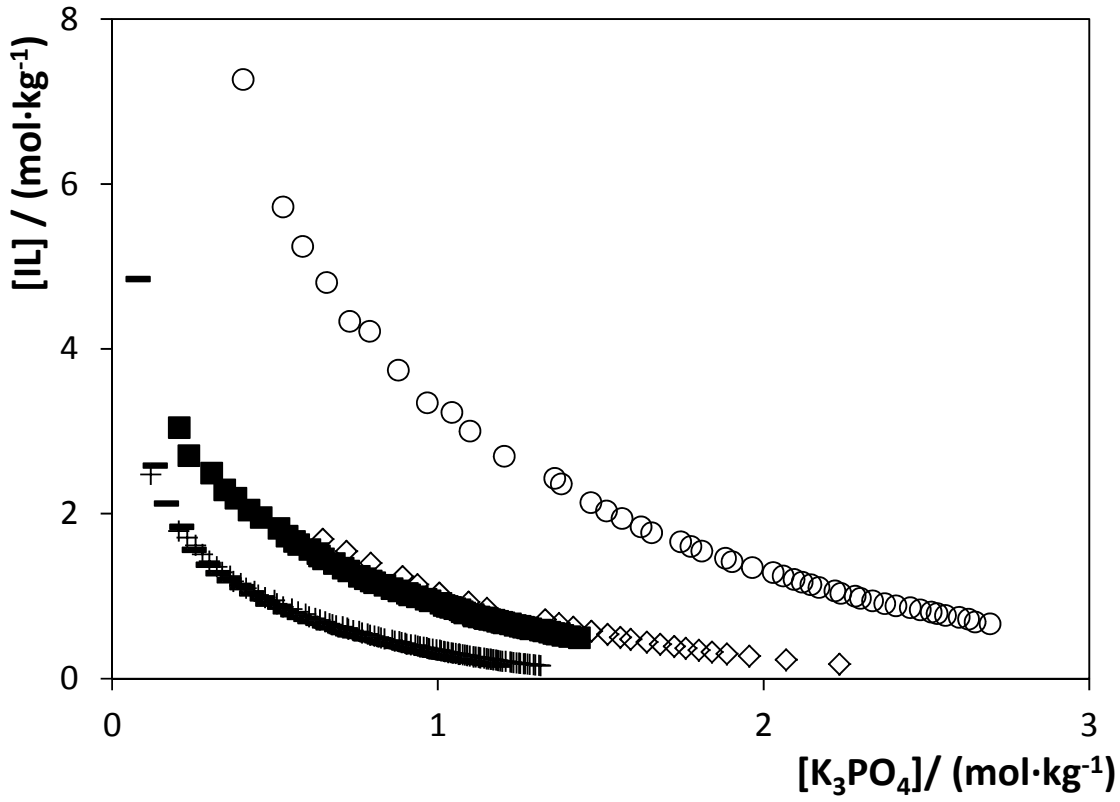

Fig. S1. Phase diagrams for the ternary systems composed of K_3PO_4 + chloride-based ionic liquid + H_2O at 298 K: (○) [Ch]Cl; (◊) [C_4mim]Cl; (■) [C_4mpyr]Cl; (+) [N_{4444}]Cl; (—) [P_{4444}]Cl.[30]

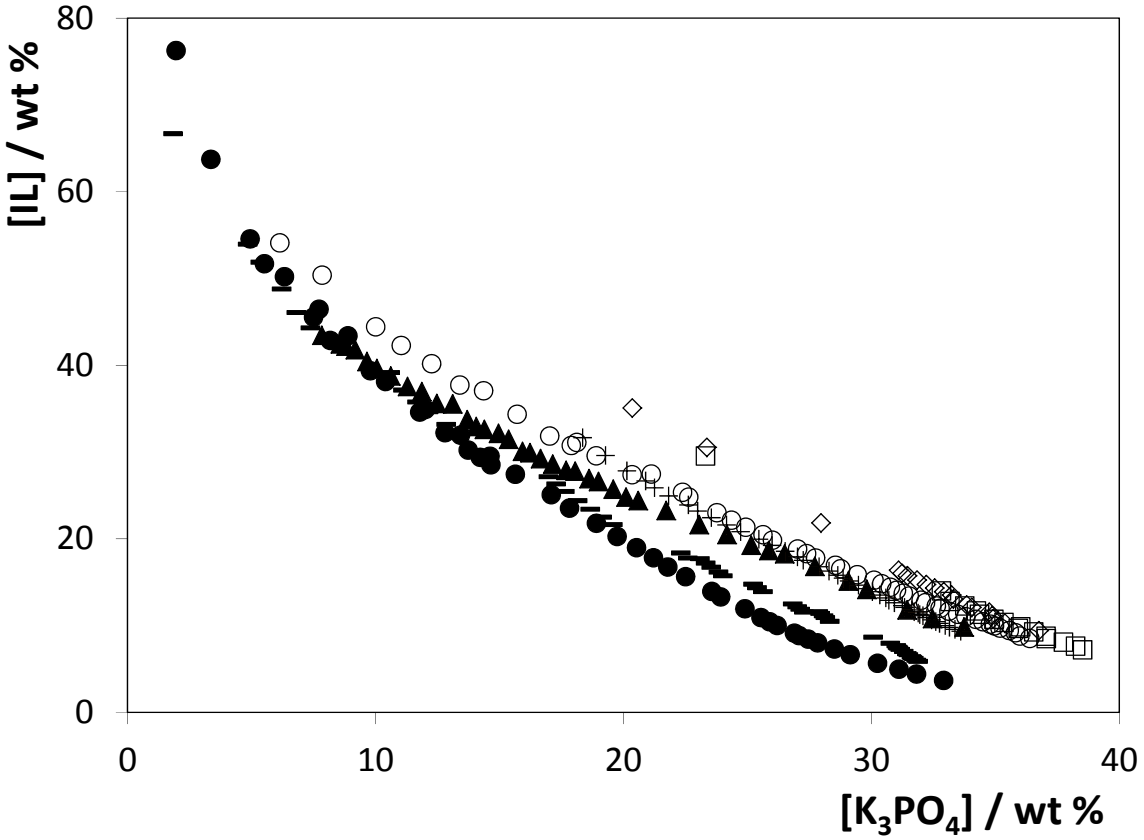

Fig. S2. Phase diagrams of the ternary systems composed of K_3PO_4 + cholinium-based ionic liquid + H_2O at 298 K (percentage weight fraction): (○) [Ch]Cl; (+) [Ch]Ac; (□) [Ch][Suc]; (◊) [Ch][Glu]; (▲) [Ch][Lev]; (▬) [BCh]Cl; (●) [Ch][Sal].

Table S8. Viscosity (η) and density (ρ) of the coexisting phases in ABS composed of cholinium-based ionic liquids + K_3PO_4 + H_2O at various temperatures.

Ionic liquid	T/ K	K_3PO_4-rich phase (bottom phase)		Cholinium-rich phase (top phase)	
		η / (mPa.s)	ρ / (g/cm^3)	η / (mPa.s)	ρ / (g/cm^3)
[Ch]Cl	298.15	8.616	1.493	5.859	1.126
	308.15	6.422	1.486	4.478	1.121
	318.15	4.995	1.480	3.537	1.116
	328.15	4.017	1.474	2.865	1.111
[Ch][Ac]	298.15	9.271	1.487	6.012	1.142
	308.15	6.889	1.480	4.444	1.136
	318.15	5.325	1.474	3.392	1.131
	328.15	4.245	1.468	2.695	1.125
[Ch][Lev]	298.15	10.233	1.522	8.219	1.123
	308.15	7.551	1.515	5.894	1.117
	318.15	5.810	1.509	4.413	1.111
	328.15	4.620	1.503	3.422	1.105
[Ch][Glu]	298.15	28.342	1.620	14.929	1.223
	308.15	19.062	1.618	10.567	1.218
	318.15	13.539	1.608	7.700	1.213
	328.15	10.073	1.602	5.934	1.207
[Ch][Sal]	298.15	4.866	1.382	7.497	1.148
	308.15	3.785	1.376	5.500	1.142
	318.15	3.055	1.370	4.197	1.136
	328.15	2.513	1.364	3.283	1.130
[Ch][Suc]	298.15	20.874	1.538	14.308	1.289
	308.15	14.562	1.532	10.233	1.285
	318.15	10.703	1.526	7.662	1.279
	328.15	8.211	1.521	5.946	1.274
[BCh]Cl	298.15	4.019	1.390	11.472	1.122
	308.15	3.188	1.384	7.975	1.116
	318.15	2.604	1.378	5.826	1.110
	328.15	2.179	1.372	4.432	1.104

Table S9. Partition coefficients (K), respective standard deviations (σ) and mixture compositions at 298 K.

IL+ K_3PO_4+ H_2O system	mixture composition / wt %		$K \pm \sigma$
	Ionic liquid	K_3PO_4	
Tetracycline			
[Ch]Cl	29.70	19.80	22.01 ± 0.48
[Ch][Ac]	29.80	19.98	21.04 ± 0.01
[Ch][Lev]	30.15	20.22	21.03 ± 0.15
Tetracycline.HCl			
[Ch]Cl	30.02	19.99	100.36 ± 1.04
[Ch][Ac]	30.90	19.88	104.47 ± 0.25
[Ch][Lev]	30.15	20.22	53.70 ± 0.54
[Ch][Glu]	30.67	27.01	0.51 ± 0.17
[Ch][Suc]	30.19	26.98	7.79 ± 0.66
Ciprofloxacin.HCl			
[Ch]Cl	29.70	19.99	31.21 ± 0.78
[Ch][Ac]	30.90	19.88	39.61 ± 0.54
[Ch][Lev]	30.15	20.22	22.76 ± 0.68
[Ch][Glu]	30.32	27.24	0.47 ± 0.15
[Ch][Suc]	30.50	26.91	2.29 ± 0.04

Table S10. Partition coefficients (K), respective standard deviations (σ) and mixture compositions of the systems composed of [Ch]Cl + K_3PO_4 + H_2O at 298 K.

mixture composition / wt %		$K \pm \sigma$
[Ch]Cl	K_3PO_4	
Tetracycline.HCl		
20.46	27.51	100.40 ± 0.48
29.70	19.99	100.36 ± 1.04
20.82	29.80	137.10 ± 0.05
24.95	30.24	131.23 ± 0.96
20.65	34.33	Complete extraction
Ciprofloxacin.HCl		
20.32	27.52	31.21 ± 0.43
29.70	19.99	31.21 ± 0.78
20.12	29.91	126.28 ± 1.31
24.88	29.96	77.28 ± 0.27
20.28	34.95	177.41 ± 0.76

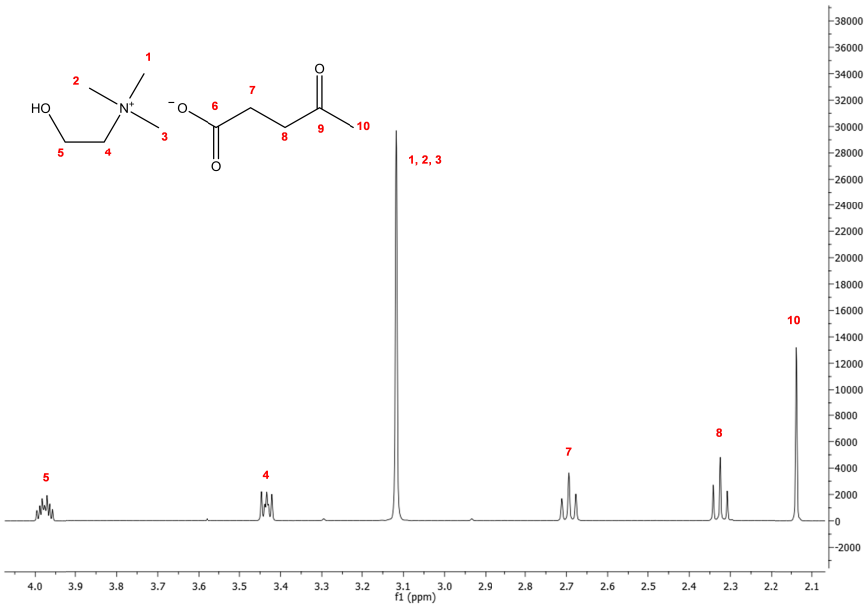

Fig. S3. ^{1}H NMR spectrum of [Ch][Lev] in D_2O.

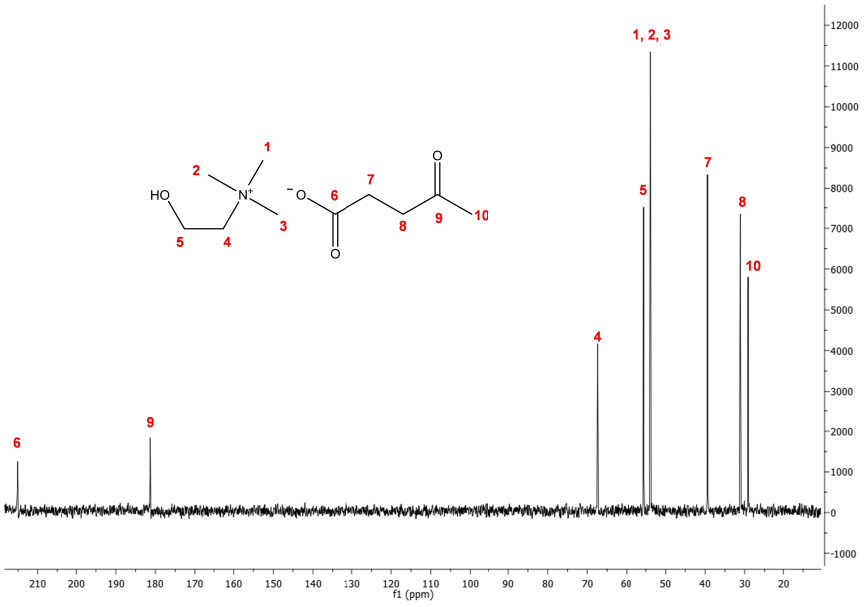

Fig. S4. ^{13}C NMR spectrum of [Ch][Lev] in D_2O.

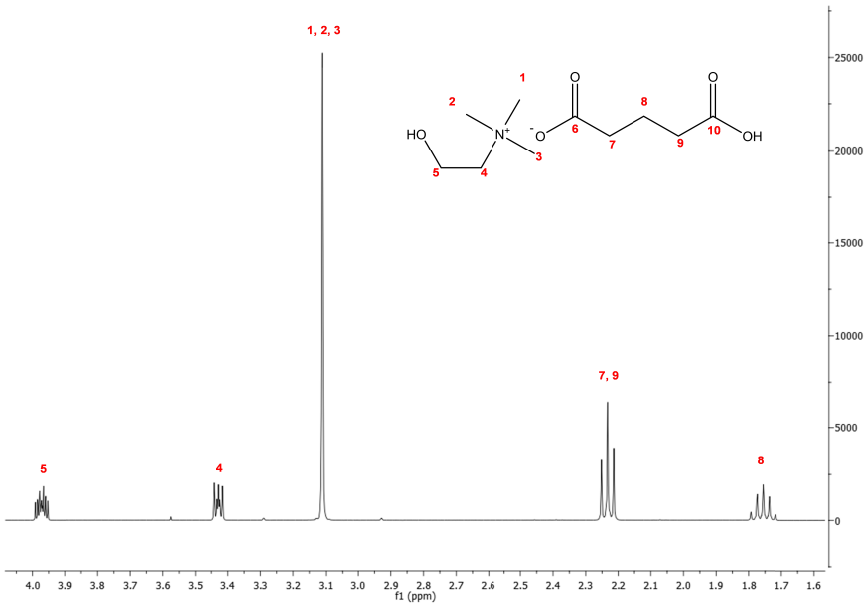

Fig. S5. ^{1}H NMR spectrum of [Ch][Glu] in D_2O.

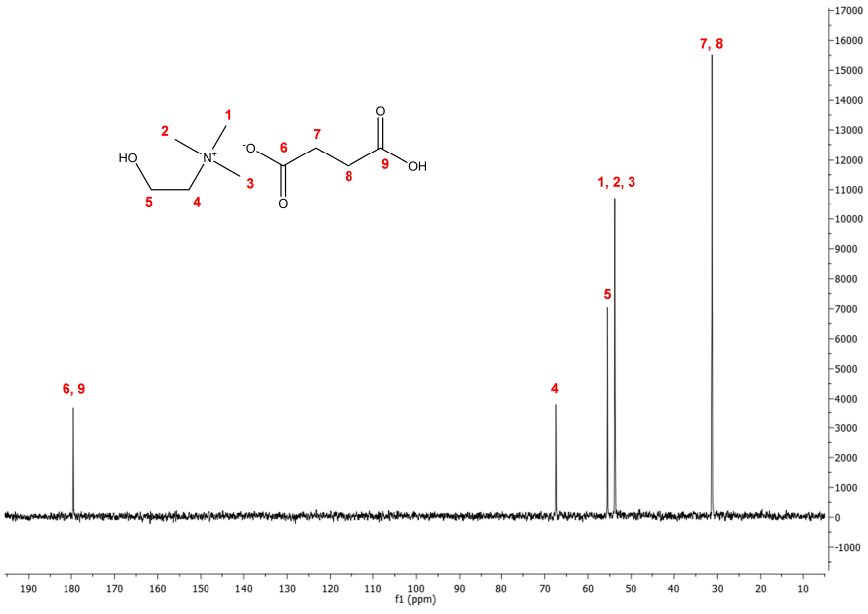

Fig. S6. ^{13}C NMR spectrum of [Ch][Glu] in D_2O.

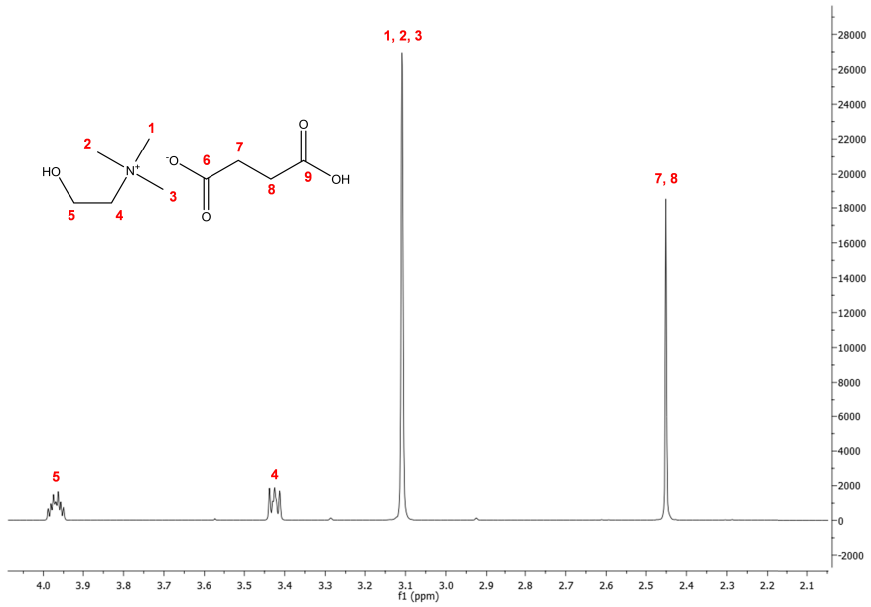

Fig. S7. ^{1}H NMR spectrum of [Ch][Suc] in D_2O.

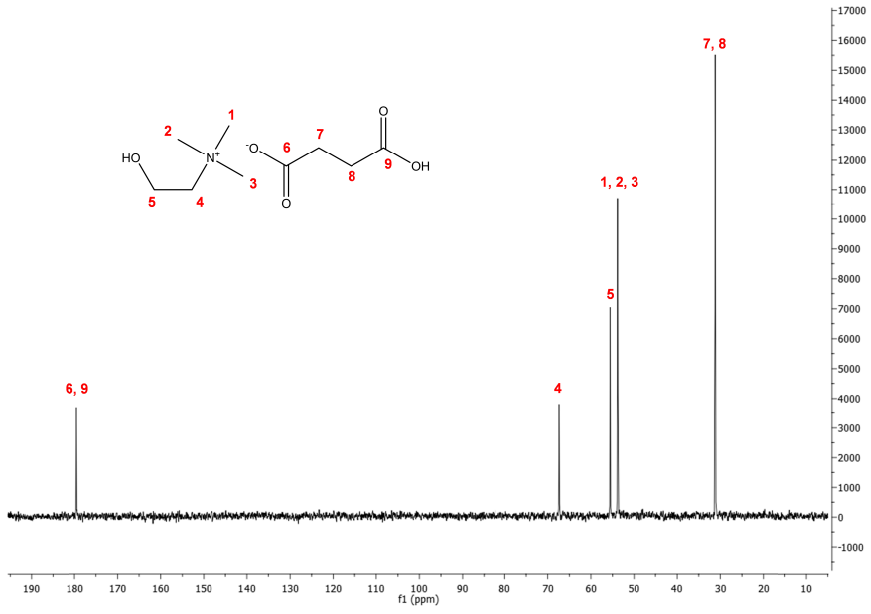

Fig. S8. ^{13}C NMR spectrum of [Ch][Suc] in D_2O.