Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

ELECTRONIC SUPPLEMENTARY MATERIAL

Phase Behaviour and Conductivity of Supporting Electrolytes in Supercritical Difluoromethane and 1,1-Difluoroethane

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Synthetic details

Instrumentation

$[N(^nC_4H_9)_4]Cl$ and $Li[NTf_2]$ (from Sigma Aldrich) and $[N(CH_3)_4][FAP]$ (from VWR) were stored in a glove box and used as received. [EMIM]Cl (Aldrich) was dried at 100 °C in vacuo for 4 hours.[1] $Li[Al(OC_4F_9)_4]$ was synthesised according to a literature procedure.[2] ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded in CD_2Cl_2 solution at 298 K using Bruker AV-300 or DPX-400 spectrometers and are referenced to the residual solvent resonance. $^{19}F\{^1H\}$ and $^{31}P\{^1H\}$ spectra were obtained from CD_2Cl_2 solution on a Bruker AV-300 spectrometer at 298 K and referenced to external $CFCl_3$ and 85% H_3PO_4 respectively. ^{27}Al NMR spectra were obtained from $CDCl_3$ solution at 298 K on a Bruker DPX-400 spectrometer and referenced to external $[Al(H_2O)_6]^{3+}$. Microanalyses were undertaken at London Metropolitan University.

Tetramethylammonium trifluoro-tris(pentafluoroethyl)phosphate, $[N(CH_3)_4][FAP]$

Crystallised by slow evaporation of a saturated CH_2Cl_2 solution. ^1H NMR (300.1 MHz, CD_2Cl_2): 3.16 (12H, s) ppm. $^{13}C\{^1H\}$ NMR (75.5 MHz, CD_2Cl_2): 56.96 (t, $J_{C–N}$ = 3.8 Hz, NMe_4) ppm. $^{19}F\{^1H\}$ NMR (282.4 MHz, CD_2Cl_2): -44.87 (dm, $^1J_{F–P}$ = 890 Hz, $^2J_{F–F}$ = 17.2 Hz, $^3J_{F–F}$ = 12.9 Hz, PF), -80.82 (m, $^3J_{F–F}$ = 12.9, 8.6 Hz, CF_3), -82.46 (m, $^3J_{F–F}$ = 12.9, 8.6 Hz, $2CF_3$), -88.54 (dm, $^1J_{F–P}$ = 900 Hz, $^2J_{F–F}$ = 17.2 Hz, $^3J_{F–F}$ = 12.9, 8.6 Hz, 2PF), -116.16 (br d, $^2J_{F–P}$ = 99 Hz, CF_2), -116.69 (dm, $^2J_{F–P}$ = 99 Hz, $^3J_{F–F}$ = 8.6 Hz, $2CF_2$) ppm. $^{31}P\{^1H\}$ NMR (121.5 MHz, CD_2Cl_2): -146.51 (dtpt, $^1J_{P–F}$ = 890, 900 Hz, $^2J_{P–F}$ = 99, 14 Hz) ppm.

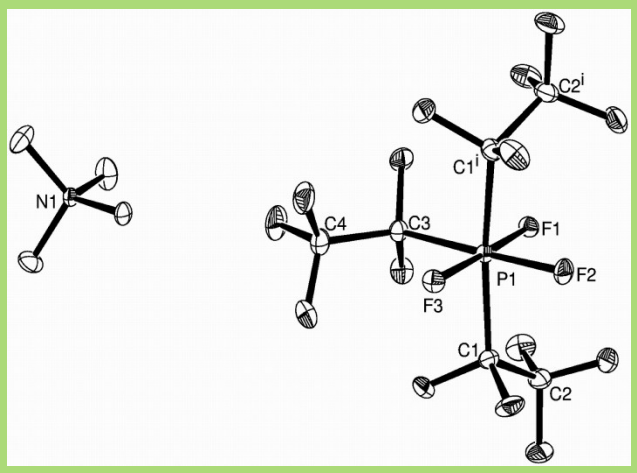

Figure S1: ORTEP of $[N(CH_3)_4][FAP]$. Thermal ellipsoids at 50% probability, hydrogens omitted for clarity. Selected bond lengths (Å) and angles (°): P1–F1 1.621(1), P1–F2 1.620(1), P1–F3 1.631(1), P1–C1 1.940(2), P1–C3 1.951(2); F1–P1–F3 177.51(7), C1–P1–$C1^i$ 169.2(1), F2–P1–C3 176.44(9), F1–P1–F2 90.21(8), F2–P1–F3 92.28(7), F1–P1–C3 86.22(9), F1–P1–C1 92.49(5). Symmetry code: x, 0.5 – y, z.

Tetrakis-*n*butylammonium trifluoro-tris(pentafluoroethyl)phosphate, $[N(^nC_4H_9)_4][FAP]$

A solution of $[N(^nC_4H_9)_4]Cl$ (1.39 g, 5.0 mmol) in MeCN (5 mL) was added dropwise to a stirred solution of $[N(CH_3)_4][FAP]$ (2.60 g, 5.0 mmol) in MeCN (5 mL). A white solid immediately precipitated and the reaction was stirred for 24 hours. After this time it was filtered through Celite® and all volatiles removed. The solid was extracted into CH_2Cl_2, filtered through a plug of silica and concentrated to ~5 mL, then pentane (50 mL) was added to precipitate a colourless oil. The supernatant was decanted away and a white solid formed upon removal of all volatiles. Yield 3.30 g (96%). Crystallisation occurred from the slow evaporation of a CH_2Cl_2 solution. Anal. calc. for $C_{22}H_{36}NF_{18}P$ (687.23): C 38.42; H 5.28; N 2.04. Found C 38.25; H 5.68; N 2.15. ^{1}H NMR (400.1 MHz, CD_2Cl_2): 3.06 (8H, m, NCH_2), 1.59 (8H, m, $NCH_2C\underline{H}_2$), 1.41 (8H, sextet, J = 7.4 Hz, $C\underline{H}_2CH_3$), 1.01 (12H, t, J = 7.3 Hz, CH_3) ppm. ^{13}C{^{1}H} NMR (100.6 MHz, CD_2Cl_2): 59.34 (NCH_2), 24.26, 20.05 (CH_2), 13.69 (CH_3) ppm. ^{19}F{^{1}H} NMR (282.4 MHz, CD_2Cl_2): -45.22 (dm, $^1J_{F-P}$ = 890 Hz, $^2J_{F-F}$ = 17.2 Hz, $^3J_{F-F}$ = 12.9 Hz, PF), -80.77 (m, $^3J_{F-F}$ = 12.9, 8.6 Hz, CF_3), -82.45 (m, $^3J_{F-F}$ = 12.9, 8.6 Hz, $2CF_3$), -88.56 (dm, $^1J_{F-P}$ = 900 Hz, $^2J_{F-F}$ = 17.2 Hz, $^3J_{F-F}$ = 12.9, 8.6 Hz, 2PF), -116.22 (br d, $^2J_{F-P}$ = 99 Hz, CF_2), -116.75 (dm, $^2J_{F-P}$ = 99 Hz, $^3J_{F-F}$ = 8.6 Hz, $2CF_2$) ppm. ^{31}P{^{1}H} NMR (121.5 MHz, CD_2Cl_2): -146.87 (dtpt, $^1J_{P-F}$ = 890, 900 Hz, $^2J_{P-F}$ = 99, 14 Hz) ppm.

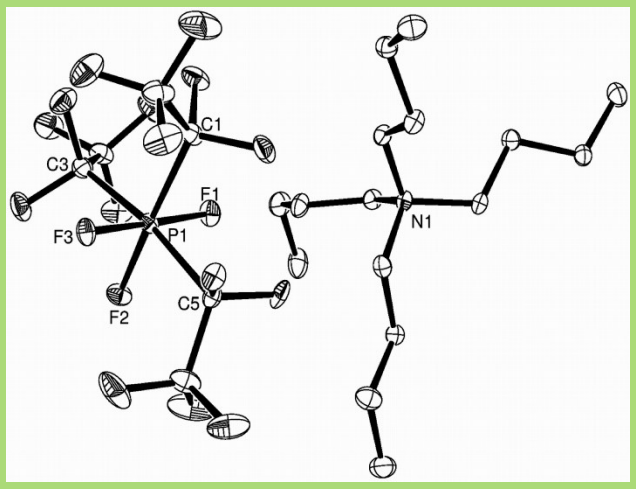

Figure S2 ORTEP of $[N(^nC_4H_9)_4][FAP]$ showing one orientation of the positional disorder in the anion. Thermal ellipsoids at 50% probability, hydrogens and positional disorder omitted for clarity. Selected bond lengths (Å) and angles (°): P1–F1 1.624(2), P1–F2 1.629(3), P1–F3 1.626(2), P1–C1 1.956(4), P1–C3 1.948(4), P1–C5 1.979(5); F1–P1–F3 177.8(2), C3–P1–C5 171.4(2), F2–P1–C1 177.0(2), F1–P1–F2 90.3(1), F2–P1–F3 91.8(2), C1–P1–C3 95.0(2), C1–P1–C5 93.3(2).

Tetrakis-*n*butylammonium bis-(trifluoromethanesulfonyl)amide, $[N(^nC_4H_9)_4][NTf_2]$

$[N(^nC_4H_9)_4]Cl$ (1.39 g, 5.0 mmol) and $Li[NTf_2]$ (1.44 g, 5.0 mmol) were dissolved in CH_2Cl_2 (20 mL). A white solid immediately precipitated then the reaction was stirred for 24 hours. After this time it was filtered and all volatiles removed. Yield 2.45 g (94%). Crystallisation occurred from vapour diffusion of hexane into a concentrated CH_2Cl_2 solution. Anal. calc. for $C_{18}H_{36}N_2O_4F_6S_2$ (522.20): C 41.36; H

6.95; N 5.36. Found C 41.35; H 7.01; N 5.41. ^{1}H NMR (400.1 MHz, CD_2Cl_2): 3.11 (8H, m, NCH_2), 1.60 (8H, m, $NCH_2C\underline{H}_2$), 1.41 (8H, sextet, J = 7.3 Hz, $C\underline{H}_2CH_3$), 1.00 (12H, t, J = 7.3 Hz, CH_3) ppm. ^{13}C{^{1}H} NMR (100.6 MHz, CD_2Cl_2): 120.53 (q, J_{C-F} = 321 Hz, CF_3), 59.33 (NCH_2), 24.32, 20.11 (CH_2), 13.77 (CH_3) ppm. ^{19}F{^{1}H} NMR (376.5 MHz, CD_2Cl_2): -79.68 ppm.

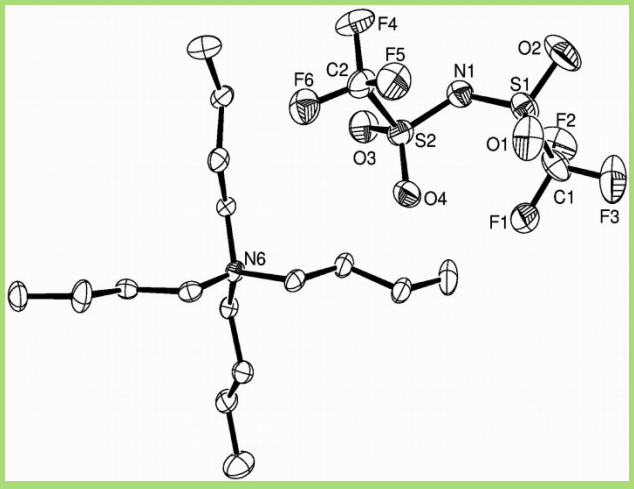

Figure S3 ORTEP of $[N(^{n}C_4H_9)_4][NTf_2]$ showing one of four molecules in the asymmetric unit. Thermal ellipsoids at 50% probability, hydrogens omitted for clarity. Selected ranges of bond lengths (Å) and angles (°): N–S 1.49(1) – 1.66(1), S–O 1.37(1) – 1.47(1); S–N–S 120.6(6) – 126.3(7).

Tetrakis-*n*butylammonium tetrakis(perfluoro-tert-butoxy)aluminate, $[N(^{n}C_4H_9)_4][Al(OC(CF_3)_3)_4]$

$[N(^{n}C_4H_9)_4]Cl$ (1.46 g, 5.3 mmol) and $Li[Al(OC(CF_3)_3)_4]$ (4.87 g, 5.0 mmol) were dissolved in CH_2Cl_2 (30 mL). A white solid immediately precipitated then the reaction was stirred for 10 minutes. After this time it was filtered and all volatiles removed. The solid was triturated with water (250 mL) to remove excess $[N(^{n}C_4H_9)_4]Cl$, then the suspension was filtered and dried *in vacuo*. Yield 5.80 g (96%). Anal. calc. for $C_{32}H_{36}NO_4F_{36}Al$ (1209.54): C 31.78; H 3.00; N 1.16. Found C 31.03; H 2.45; N 1.16. ^{1}H NMR (300.1 MHz, $CDCl_3$): 3.10 ppm (8H, m, NCH_2), 1.50 (8H, m, $NCH_2C\underline{H}_2$), 1.39 (8H, m, $C\underline{H}_2CH_3$), 0.95 (12H, t, J = 7.0 Hz, CH_3). ^{19}F{^{1}H} NMR (282.4 MHz, $CDCl_3$): -75.7 ppm. ^{27}Al NMR (104.3 MHz, $CDCl_3$): 34.1 ppm.

Crystallography

Crystals were obtained as described above. Details of the crystallographic data collection and refinement are in Table S1. Diffractometer: Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum rotating anode generator (λ_1 = 0.71073 Å) with VHF Varimax optics (70 or 100 µm focus). Cell determination, data collection, data reduction, cell refinement and absorption correction: CrystalClear-SM Expert 2.1 b2.9.[3] Structure solution and refinement were carried out using WinGX and software packages within.[4] The anion in $[N(^{n}C_4H_9)_4][FAP]$ was positionally disordered across two positions but

it was satisfactorily modelled using suitable restraints. $[N(^nC_4H_9)_4][NTf_2]$ crystallised with Z' of 4, with one cation and one anion containing slight positional disorder. H atoms attached to C atoms were placed in geometrically assigned positions, with C—H distances of 0.98 Å (CH_3) or 0.99 Å (CH_2) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) (CH_2) or 1.5U_{eq}(C) (CH_3). enCIFer was used to prepare CIFs for publication.[5] CCDC reference numbers 1432360 ($[N(CH_3)_4][FAP]$, 1432361 ($[N(^nC_4H_9)_4][FAP]$) and 1432362 ($[N(^nC_4H_9)_4][NTf_2]$ contain crystallographic data in CIF format.

References

1. P. N. Bartlett, C. Y. Cummings, W. Levason, D. Pugh and G. Reid, *Chem. - Eur. J.*, 2014, 20, 5019-5027.
2. I. Raabe, K. Wagner, K. Guttsche, M. Wang, M. Graetzel, G. Santiso-Quinones and I. Krossing, *Chem. - Eur. J.*, 2009, 15, 1966-1976.
3. CrystalClear-SM Expert 2.1 b29, Rigaku Corporation, Tokyo, Japan, 2013.
4. L. J. Farrugia, *J. Appl. Crystallogr.*, 1999, 32, 837-838.
5. F. H. Allen, O. Johnson, G. P. Shields, B. R. Smith and M. Towler, *J. Appl. Crystallogr.*, 2004, 37, 335-338.

Table S1 crystallographic data for compounds reported in this paper. Common items: T = 100(2) K.

Compound	$[N(CH_3)_4][FAP]$	$[N(^nC_4H_9)_4][FAP]$	$[N(^nC_4H_9)_4][NTf_2]$
Formula	$C_{10}H_{12}F_{18}NP$	$C_{22}H_{36}F_{18}NP$	$C_{18}H_{36}F_6N_2O_4S_2$
M/g mol^{-1}	519.18	687.49	522.61
Crystal system	orthorhombic	orthorhombic	monoclinic
Space group (No.)	*Pnma* (62)	*Pbca* (61)	$P2_1$ (4)
a/Å	18.972(2)	17.591(3)	13.312(6)
b/Å	11.865(1)	17.971(3)	29.00(1)
c/Å	7.1966(9)	18.534(3)	13.523(6)
β/°	90	90	94.059(9)
U/Å^3	1782.1(3)	5859(2)	5207(4)
Z	4	8	8
μ(Mo-Kα) /mm^{-1}	0.332	0.223	0.272
F(000)	1024	2816	2208
Total reflections	6166	35369	21991
Unique reflections	2130	6685	17489
R_{int}	0.015	0.075	0.035
Goodness-of-fit on F^2	1.100	1.185	1.097
R_1[b] [$I_o > 2\sigma(I_o)$]	0.039	0.075	0.095
R_1 (all data)	0.042	0.097	0.128
wR_2[b] [$I_o > 2\sigma(I_o)$]	0.114	0.117	0.182
wR_2 (all data)	0.118	0.124	0.212

Table S2 The phase boundary data of all eight binary mixtures, electrolytes (1) + CH_2F_2 (2)

$x_1(\times10^{-3})$	[BMIM][NTf_2]										
0.39	T/K	299.7	303.4	312.9	322.8	332.8	342.8	353.1	363.9	368.9	373.6
	p/MPa	1.83	2.02	2.58	3.28	4.12	5.18	6.28	7.63	8.09	8.42
2.01	T/K	299.1	303.3	312.8	322.7	332.6	342.7	352.4	365.0	368.1	373.0
	p/MPa	1.87	2.10	2.61	3.27	4.07	5.13	6.92	8.67	9.24	10.00
4.13	T/K	298.7	303.3	313.0	323.1	332.8	343.1	357.5	363.0	367.9	372.6
	p/MPa	1.91	2.13	2.68	3.34	4.16	5.12	8.60	9.71	10.62	11.51
[EMIM][NTf_2]											
0.19	T/K	293.3	303.0	313.0	323.0	333.0	343.2	353.5	363.7	368.4	
	p/MPa	1.62	2.10	2.65	3.36	4.17	5.20	6.28	7.84	8.58	
0.64	T/K	296.4	303.1	312.9	322.9	333.0	343.3	354.0	363.0	368.2	372.9
	p/MPa	1.81	2.13	2.70	3.37	4.19	5.18	6.57	8.33	9.27	10.09
1.72	T/K	293.7	303.0	312.8	323.0	333.0	342.9	353.4	363.2	367.9	372.8
	p/MPa	1.62	2.09	2.63	3.26	4.16	5.27	6.85	8.93	9.72	10.62
2.81	T/K	294.1	303.1	312.9	323.0	333.1	344.2	353.6	363.0	368.1	373.6
	p/MPa	1.79	2.21	2.77	3.43	4.31	5.28	7.63	9.86	11.04	12.19
4.25	T/K	295.4	303.2	313.5	323.0	333.1	343.3	353.0	363.0	367.9	373.1
	p/MPa	1.65	2.03	2.56	3.21	4.04	5.01	7.68	10.28	11.48	12.74
[EMIM]Cl											
0.23	T/K	294.0	303.2	313.5	324.3	334.0	343.8	352.7	363.5	369.0	
	p/MPa	1.81	2.24	2.79	3.50	4.31	5.32	6.96	8.53	9.64	
0.47	T/K	298.8	303.3	312.9	322.9	332.8	344.2	352.7	358.2	364.0	368.2
	p/MPa	1.90	2.12	2.68	3.36	4.16	5.17	7.35	8.60	9.94	10.89
0.76	T/K	293.1	302.8	313.0	322.0	332.9	343.5	352.9	362.6		
	p/MPa	1.79	2.24	2.77	3.38	4.22	5.30	9.86	13.69		
[$N(^nC_4H_9)_4$]Cl											
0.49	T/K	293.8	303.1	313.1	323.0	333.0	344.3	353.3	363.7	368.5	373.6
	p/MPa	1.68	2.12	2.67	3.35	4.25	5.22	6.20	7.82	8.39	9.09
0.93	T/K	293.9	303.1	313.6	323.6	333.3	343.2	353.2	363.1		
	p/MPa	1.65	2.09	2.69	3.37	4.20	5.16	6.57	8.09		
1.90	T/K	293.5	303.1	313.4	323.2	333.2	343.7	353.7	362.9		
	p/MPa	1.70	2.16	2.71	3.41	4.19	5.18	6.98	8.90		
2.95	T/K	297.9	302.9	312.8	322.8	333.0	342.8	353.3	358.5	363.0	367.9
	p/MPa	1.90	2.15	2.72	3.36	4.23	5.18	7.16	8.48	9.43	10.42
3.84	T/K	294.1	303.2	313.1	323.0	333.0	343.6	353.1	363.0	368.3	373.3
	p/MPa	1.70	2.12	2.66	3.37	4.17	5.18	7.34	9.74	10.69	11.81
[$N(^nC_4H_9)_4$][NTf_2]											
0.52	T/K	293.6	302.9	312.9	323.0	332.8	342.9	353.0	362.8	367.9	372.7
	p/MPa	1.63	2.05	2.59	3.25	4.05	5.02	6.21	7.73	8.42	9.07
[$N(^nC_4H_9)_4$][FAP]											
0.48	T/K	298.8	303.1	312.9	322.8	332.9	342.9	352.9	363.5	368.1	372.8
	p/MPa	1.85	2.10	2.63	3.32	4.16	5.16	6.38	7.98	8.68	9.36
1.05	T/K	298.7	303.2	312.7	322.9	333.0	343.1	353.6	363.6	373.1	
	p/MPa	1.99	2.21	2.76	3.44	4.27	5.27	6.56	8.10	9.72	
2.00	T/K	298.8	303.1	312.8	322.8	333.0	343.0	353.3	363.2	372.8	

	p/MPa	1.93	2.14	2.73	3.41	4.25	5.23	6.58	8.62	10.47	
3.64	T/K	298.7	303.4	312.8	322.9	332.9	342.9	352.7	362.7	367.5	372.8
	p/MPa	2.01	2.24	2.80	3.45	4.30	5.27	7.07	8.91	9.99	11.15
$[N(CH_3)_4][FAP]$											
0.51	T/K	298.7	303.1	312.8	322.9	332.8	342.8	353.1	363.0	368.2	372.9
	p/MPa	1.91	2.12	2.71	3.40	4.22	5.20	6.38	7.90	8.69	9.38
1.03	T/K	298.8	303.0	312.6	322.6	332.5	343.5	352.9	362.8	372.4	
	p/MPa	1.99	2.20	2.75	3.42	4.24	5.18	6.53	8.24	9.84	
1.90	T/K	293.8	303.0	312.7	322.0	334.4	342.8	352.7	362.7	372.4	
	p/MPa	1.68	2.11	2.68	3.34	4.44	5.13	6.47	8.41	10.36	
3.05	T/K	298.6	302.9	312.7	322.8	332.8	342.9	353.1	363.2	372.8	
	p/MPa	1.97	2.19	2.75	3.43	4.25	5.23	6.87	8.91	10.81	
$[N(^nC_4H_9)_4][Al(OC(CF_3)_3)_4]$											
0.43	T/K	312.9	318.5	328.1	338.0	342.8	347.7	352.7	357.8	362.9	368.3
	p/MPa	2.76	3.16	3.85	4.68	5.22	5.90	6.58	7.22	8.13	9.20
1.29	T/K	298.3	308.0	318.0	328.0	338.0	347.9	352.9	357.8	362.9	368.8
	p/MPa	2.18	2.67	3.26	3.96	4.80	5.86	6.43	7.21	8.07	9.14
2.54	T/K	298.3	307.9	317.9	328.0	338.0	347.9	353.0	358.0	362.9	368.0
	p/MPa	2.15	2.62	3.19	3.92	4.81	5.76	6.44	7.35	8.11	9.15
3.77	T/K	298.3	308.1	318.2	328.2	338.1	348.4	353.0	358.2	362.9	368.5
	p/MPa	2.05	2.54	3.12	3.85	4.67	5.79	6.45	7.43	8.25	9.33
5.27	T/K	298.3	307.9	318.0	327.9	338.2	348.1	353.0	358.0	363.2	368.2
	p/MPa	1.97	2.47	3.16	3.79	4.74	5.85	6.67	7.59	8.55	9.45

Table S3 The phase boundary data of the binary mixtures of $[N(^nC_4H_9)_4][BF_4]$(1)+ CHF_2CH_3(2)

$x_1(\times10^{-3})$	T/K	p/MPa	$x_1(\times10^{-3})$	T/K	p/MPa	$x_1(\times10^{-3})$	T/K	p/MPa	$x_1(\times10^{-3})$	T/K	p/MPa
1.67	333.0	2.01	4.01	313.2	1.57	8.12	323.1	1.88	15.8	323.1	1.87
1.67	343.0	2.40	4.01	323.0	1.82	8.12	333.0	2.19	15.8	333.2	2.17
1.67	352.9	2.89	4.01	333.0	2.10	8.12	343.0	2.54	15.8	342.9	2.52
1.67	363.2	3.43	4.01	342.9	2.47	8.12	353.3	3.01	15.8	353.0	2.97
1.67	373.0	4.03	4.01	353.0	2.94	8.12	363.2	3.57	15.8	362.7	3.52
1.67	378.4	4.39	4.01	363.0	3.48	8.12	373.0	4.94	15.8	368.5	4.57
1.67	383.4	5.16	4.01	373.0	4.08	8.12	378.4	6.10	15.8	373.2	5.52
1.67	388.5	6.06	4.01	378.3	4.92	8.12	383.2	7.14	15.8	378.0	6.72
1.67	393.4	6.87	4.01	383.0	5.77	8.12	388.2	8.20	15.8	383.0	7.88
1.67	397.5	7.56	4.01	387.5	6.79	8.12	392.7	9.01	15.8	388.2	9.01
			4.01	393.0	7.80		397.8	9.98	15.8	393.3	10.12
			4.01	398.0	8.76				15.8	398.1	11.19

Table S4 The phase boundary data of the binary mixtures of $[N(^nC_4H_9)_4][BF_4]$(1) + CH_2F_2(2)

$x_1(\times10^{-3})$	T/K	p/MPa
4.17	296.4	1.86 [a]
4.17	312.7	2.74 [a]
4.17	322.9	3.45 [a]
4.17	333.1	4.30 [a]
4.17	343.1	5.30 [a]
4.17	353.5	7.05 [a]
4.17	358.7	8.31 [a]
4.17	363.2	9.38
4.17	367.8	10.16
4.17	373.2	11.31
4.17	378.5	12.46
4.17	383.9	13.70
4.17	388.0	14.61
4.17	393.2	15.65
4.17	398.0	16.67

[a] Data reported graphically in the reference (P. N. Bartlett, D. C. Cook, M. W. George, J. Ke, W. Levason, G. Reid, W. Su and W. Zhang, *Phys. Chem. Chem. Phys.*, 2011, **13**, 190-198.).

Table S5 The phase boundary data of the binary mixtures of $[N(^nC_4H_9)_4][B\{3,5\text{-}C_6H_3(CF_3)_2\}_4]$ (1) + CHF_2CH_3(2)

$x_1(\times10^{-3})$	T/K	p/MPa
1.65	328.8	1.83
1.65	333.0	1.96
1.65	343.1	2.34
1.65	352.8	2.76
1.65	363.2	3.36
1.65	373.0	3.99
1.65	378.0	4.41
1.65	383.2	4.91
1.65	388.2	5.44
1.65	393.4	6.01