Electronic Supplementary Material (ESI) for ChemComm.

Supporting information

Evidence for anion-binding of all-cis hexafluorocyclohexane in solution and solid state

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Contents

General Experimental Section

All commercially available reagents were purchased from Sigma Aldrich, Alfa Aesar, Acros Organics or TCI and were used without further purification unless otherwise stated. Hydrogenation reactions were carried out in a 150mL Roth High Pressure Autoclave. Deuterated solvents were purchased from Sigma Aldrich. All solvents except acetone were dried over molecular sieves for at least 24 hours. For titrations with TBAF acetone-d_6 was distilled from Drierite™ and immediately used. NMR spectra were recorded on Bruker Avance 400 (^{1}H: 400 MHz) spectrometers at 293 K and referenced to the residual solvent peak (^{1}H: acetone-d_6, 2.05 ppm; ^{13}C: acetone-d_6, 206.68, 29.92 ppm) ^{19}F NMR spectra were referenced to hexafluorobenzene as internal standard (^{19}F: C_6F_6, -164.9 ppm). Coupling constants (J) are denoted in Hz and chemical shifts (δ) in ppm. Mass spectra were obtained on a Bruker SolariX (HRMS-ESI$^+$, solvent: acetonitrile) instrument.

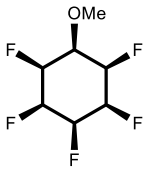
Synthesis of the Catalyst

[Rh(CAAC)(COD)Cl] was prepared according to the procedure reported by Wiesenfeldt et al. and references therein.[S1]

Synthesis of all-cis 1,2,3,4,5,6-hexafluorocyclohexane (1)

All-cis 1,2,3,4,5,6-hexafluorocyclohexane was prepared according to the procedure reported by Wiesenfeldt et al.[S1]

Synthesis of all-cis 1-methoxy-2,3,4,5,6-pentafluorocyclohexane (2)



In a dry 22 mL screw-cap vial containing a stirring bar, were placed Rh(CAAC)(COD)Cl catalyst (6.23 mg, 11 µmol, 0.01 equiv.) and dry silica gel (605 mg). The vial was closed with a septum containing a needle and the vial was placed in a Schlenk tube. High vacuum (<0.1 mbar) was applied for at least 20 minutes. 2,3,4,5,6-Pentafluoroanisol (216.12 mg, 0.135 mL, 1.09 mmol, 1 equiv.) and anhydrous hexane (17 mL) were added subsequently under argon. The reaction mixture was subjected to 5 quick vacuum/argon cycles to remove residual air and the vial was transferred to the 150 mL autoclave under argon which was carefully evacuated again after transferring. The autoclave was pressurized and depressurized 3 times to 30 bars with hydrogen gas and then the pressure was finally set to 75 bars. The reaction mixture was stirred at this pressure for 14 h at room temperature after which the autoclave was carefully depressurized, silica gel was filtered off and thoroughly washed with acetone. The solvent was removed under reduced pressure and the crude product was purified via flash column chromatography (DCM/acetone, 95/5 v/v) to give 99.7 mg (46%) of the desired compound as a colorless solid.

^{1}H NMR (400 MHz, acetone-d_6): δ 5.46 - 5.26 (m, 3H), 5.07 - 4.84 (m, 2H), 3.67 (t, J = 29.2 Hz, 1H), 3.51 (s, 1H). ^{19}F NMR (377 MHz, acetone-d_6): δ -211.6 (s, 2F), -217.18 (d, J = 25 Hz, 2F), -218.06 (tt, J = 24.6, 9.9 Hz, 1F). ^{13}C NMR (101 MHz, acetone-d_6): δ 88.27 (d, J = 18.6 Hz), 87.24, 86.32 (d, J = 18.4 Hz), 85.29 (m), 74.58 (m), 57.23.

HRMS (ESI$^+$): m/z = 431.1041 $[2M+Na]^+$ (calculated 431.1039 for $C_{14}H_{18}F_{10}O_2Na^+$).

NMR Titration Data

All salts and hosts were dried under high vacuum (10^{-3} bar) over phosphorus pentoxide overnight prior to titrations. As a source of fluoride, we used a 1.0 M anhydrous solution of tetrabutylammonium fluoride in THF. ^{19}F NMR titrations were performed via addition of aliquots of the guest anion in the tetrabutylammonium form dissolved in a stock solution of the host, to the solution of the host, containing hexafluorobenzene as an internal standard. Spectra were recorded using a Bruker Avance 400 spectrometer and spectra were referenced to the peak of hexafluorobenzene (^{19}F: C_6F_6, -164.9 ppm). All titrations were repeated 3 times. Titration data was fitted using supramolecular.org. All raw data, calculated fits and related data can be accessed via supramolecular.org.[S2]

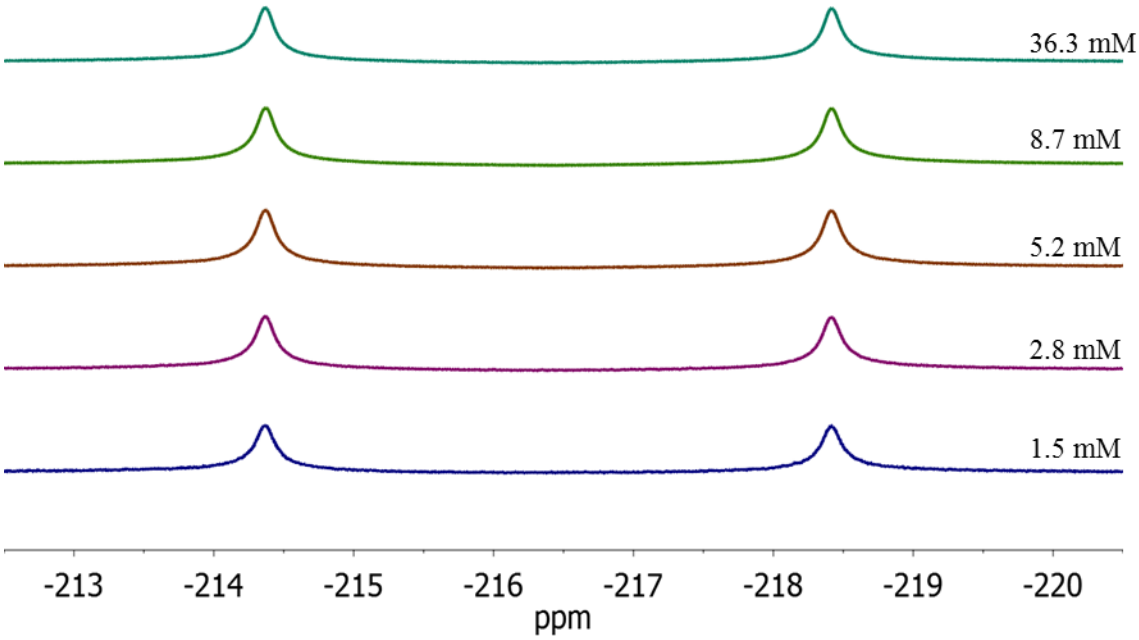


Figure S1. Investigation of possible aggregation: ^{19}F NMR stacked plot of **1** at different concentrations (acetone-d_6, 293 K).

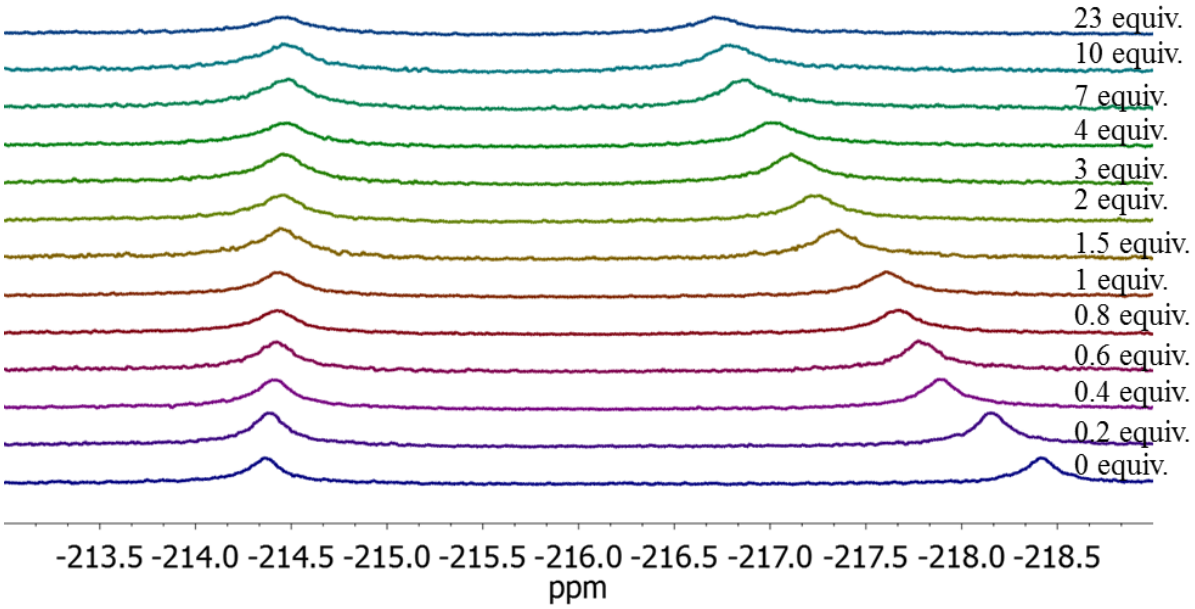


Figure S2. ^{19}F NMR stack plot for titration of **1** (5 mM) with TBACl in acetone-d_6 at 293 K. The signal at -218.4 ppm was used to obtain binding isotherms.

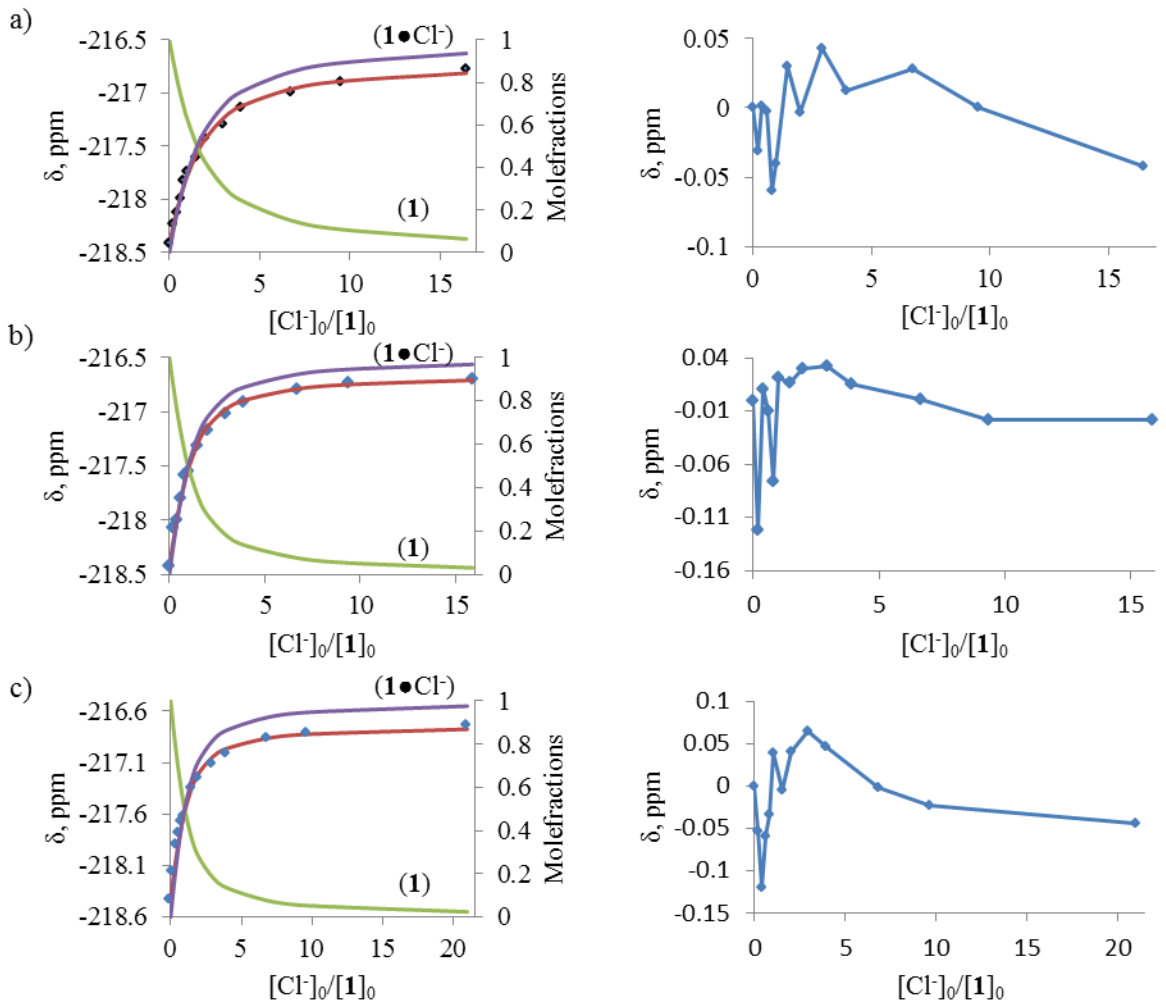


Figure S3. Left column: binding isotherms and species concentration plots for titration of **1** ($[\mathbf{1}]_0$: a) 2 mM, b) and c) 5 mM) with TBACl in acetone-d_6 at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

Table S1. Association constants obtained for the titration of **1** with TBACl in acetone-d_6 at 293 K and fitted with 2:1 (Nelder-Mead) and 1:1 models.

		H_2G + HG model				HG model	
	C, mM	K_1, M^{-1}	Fit error, $\pm$ M^{-1}	K_2, M^{-1}	Fit error, $\pm$ M^{-1}	K_a, M^{-1}	Fit error, $\pm$ M^{-1}
TBACl	2	700	400	600	700	400	40
	5	90	40	100	40	390	60
	5	20	5	1000	200	420	70
K_a (mean)		300 M^{-1}		600 M^{-1}		400 M^{-1}	
$s/\sqrt{n}$		400 M^{-1}		300 M^{-1}		10 M^{-1}	
$\pm t_{(0.05,\,2)}\, s/\sqrt{n}$		2000 M^{-1}		1000 M^{-1}		40 M^{-1}	

To estimate a confidence interval at 95% probability for $n = 3$ repeat measurements standard uncertainty $u = s/\sqrt{n}$, where s is the sample standard deviation, was multiplied by the Student t-value for the degrees of freedom $(n - 1)$.[S3]

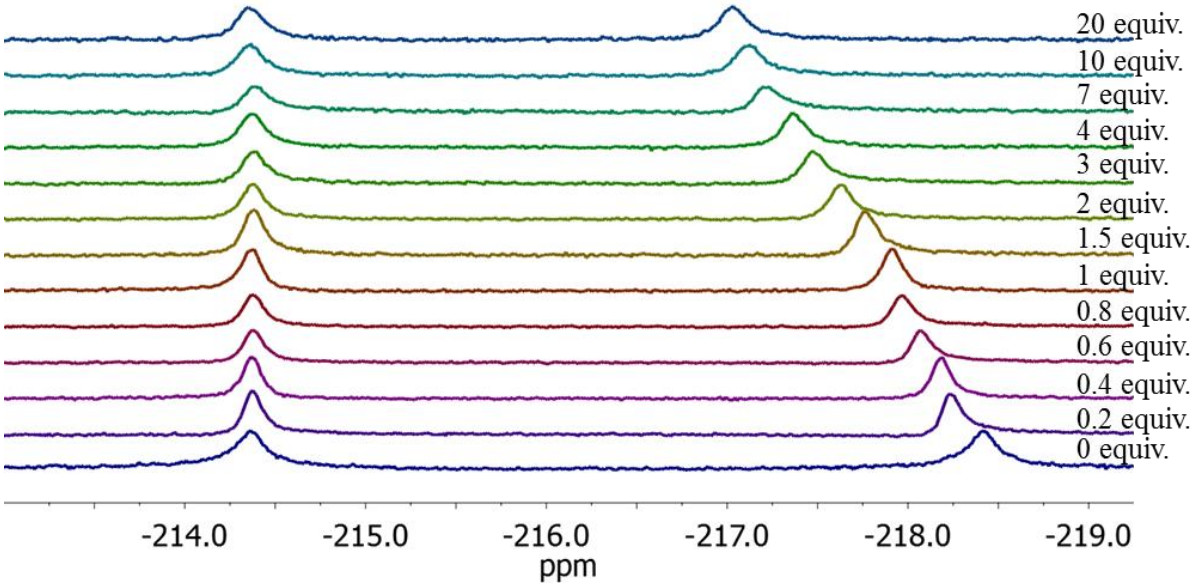


Figure S4. ^{19}F NMR stack plot for titration of **1** (5 mM) with TBABr in acetone-d_6 at 293 K. Signal at -218.4 ppm was used to obtain binding isotherms.

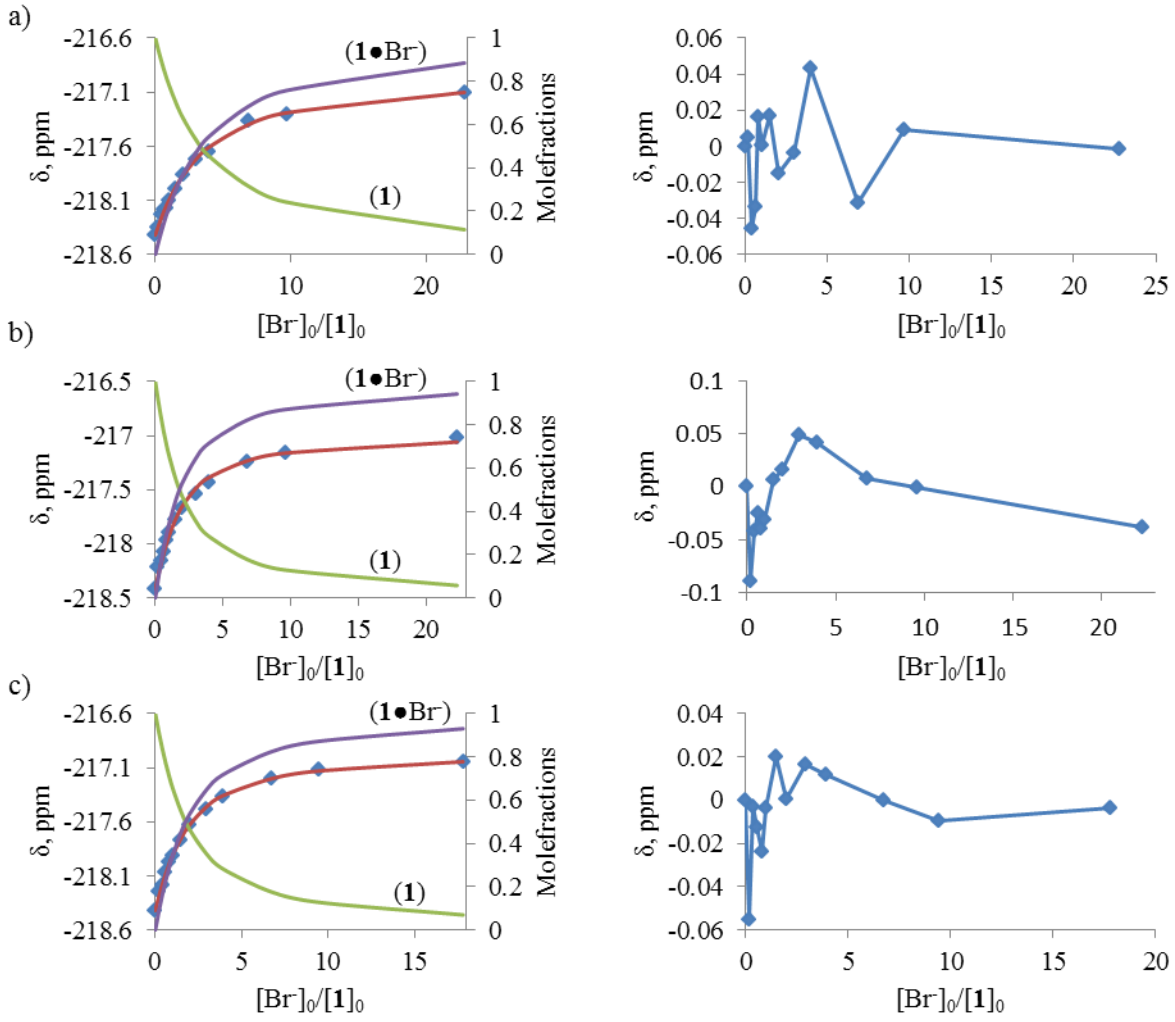


Figure S5. Left column: binding isotherms and species concentration plots for titration of **1** ([**1**]$_0$: a) 2 mM, b) and c) 5 mM) with TBABr in acetone-d_6 at 293 K. Blue diamonds: experimental points, red

line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

Table S2. Association constants obtained for the titration of **1** with TBABr in acetone-d_6 at 293 K and fitted with 2:1 (Nelder-Mead) and 1:1 models.

	H_2G + HG model					HG model	
	C, mM	K_1, M^{-1}	Fit error, $\pm M^{-1}$	K_2, M^{-1}	Fit error, $\pm M^{-1}$	K_a, M^{-1}	Fit error, $\pm M^{-1}$
TBABr	2	3	1	5000	500	160	10
	5	17	2	350	30	160	10
	5	1	0.3	8000	2000	140	20
K_a (mean)		7 M^{-1}		4600 M^{-1}		150 M^{-1}	
$s/\sqrt{n}$		5 M^{-1}		2300 M^{-1}		10 M^{-1}	
$\pm t_{(0.05,\,2)}\, s/\sqrt{n}$		20 M^{-1}		10000 M^{-1}		40 M^{-1}	

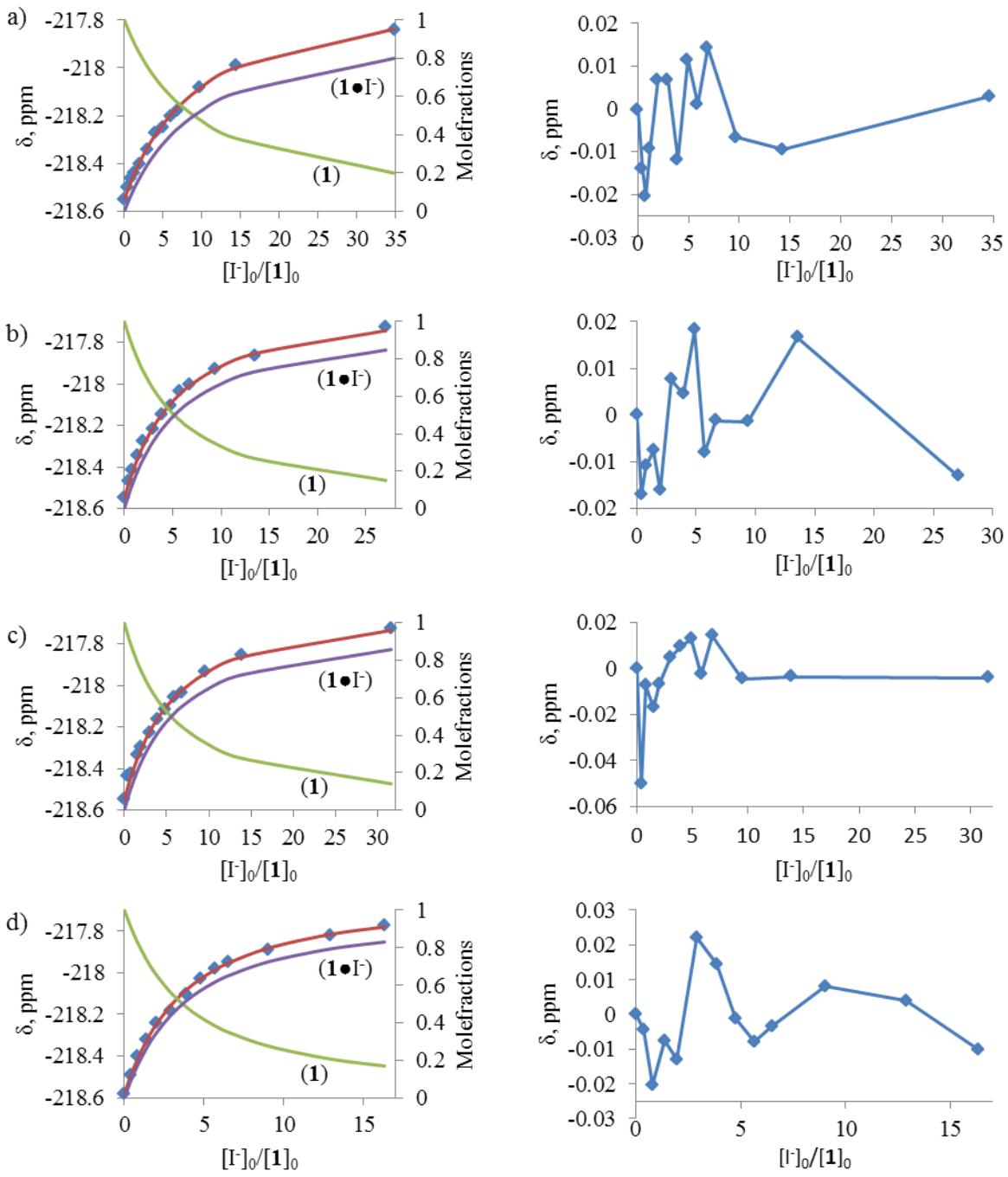


Figure S6. Left column: binding isotherms and species concentration plots for titration of **1** ([**1**]$_0$: a) 2 mM, b) and c) 5 mM, d) – 10 mM) with TBAI in acetone-d_6/DCM-d_2 (88/12 w/w) at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

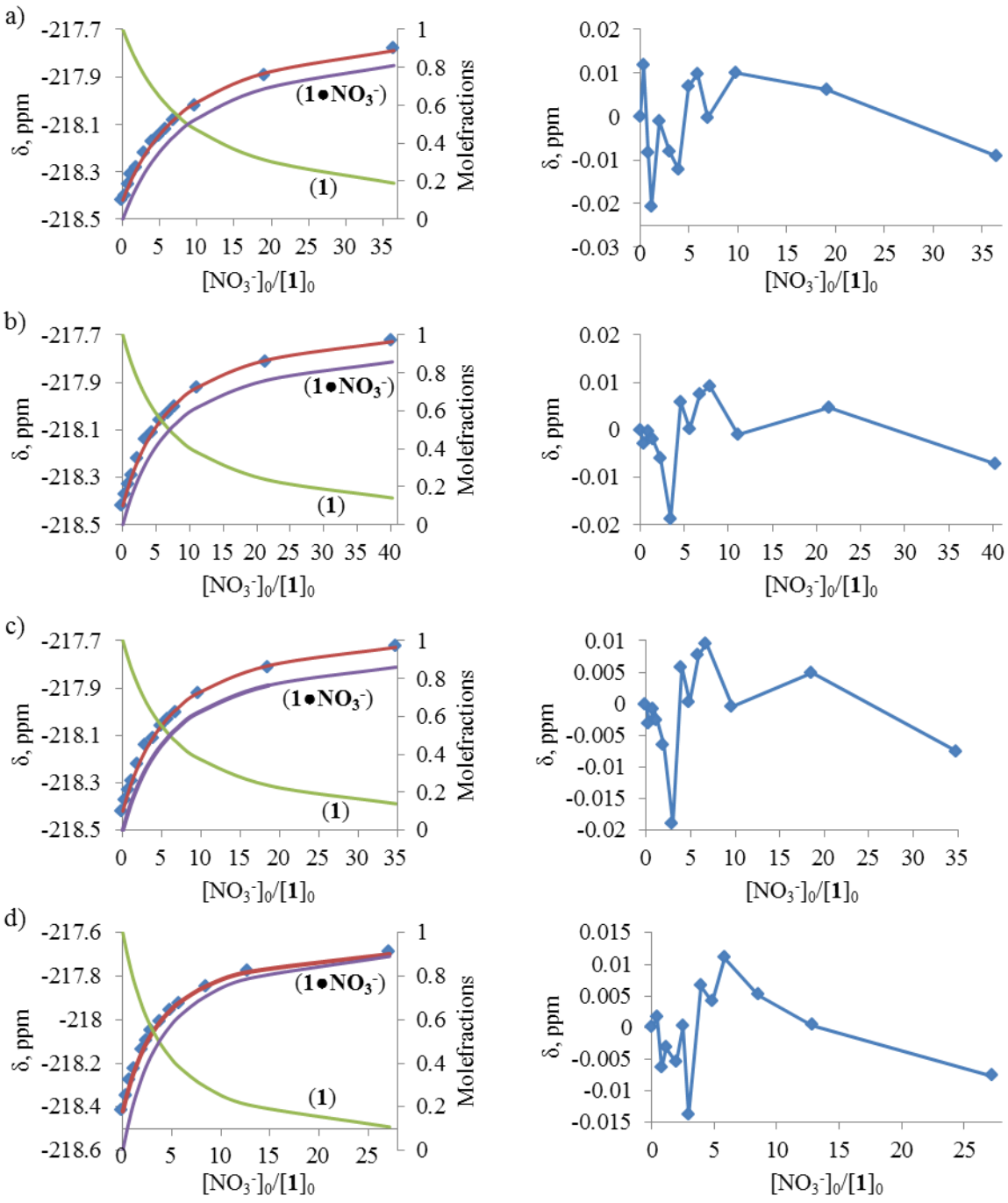


Figure S7. Left column: binding isotherms and species concentration plots for titration of **1** ($[\mathbf{1}]_0$: a) 2 mM, b) and c) 5 mM, d) – 10 mM) with $TBANO_3$ in acetone-d_6 at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

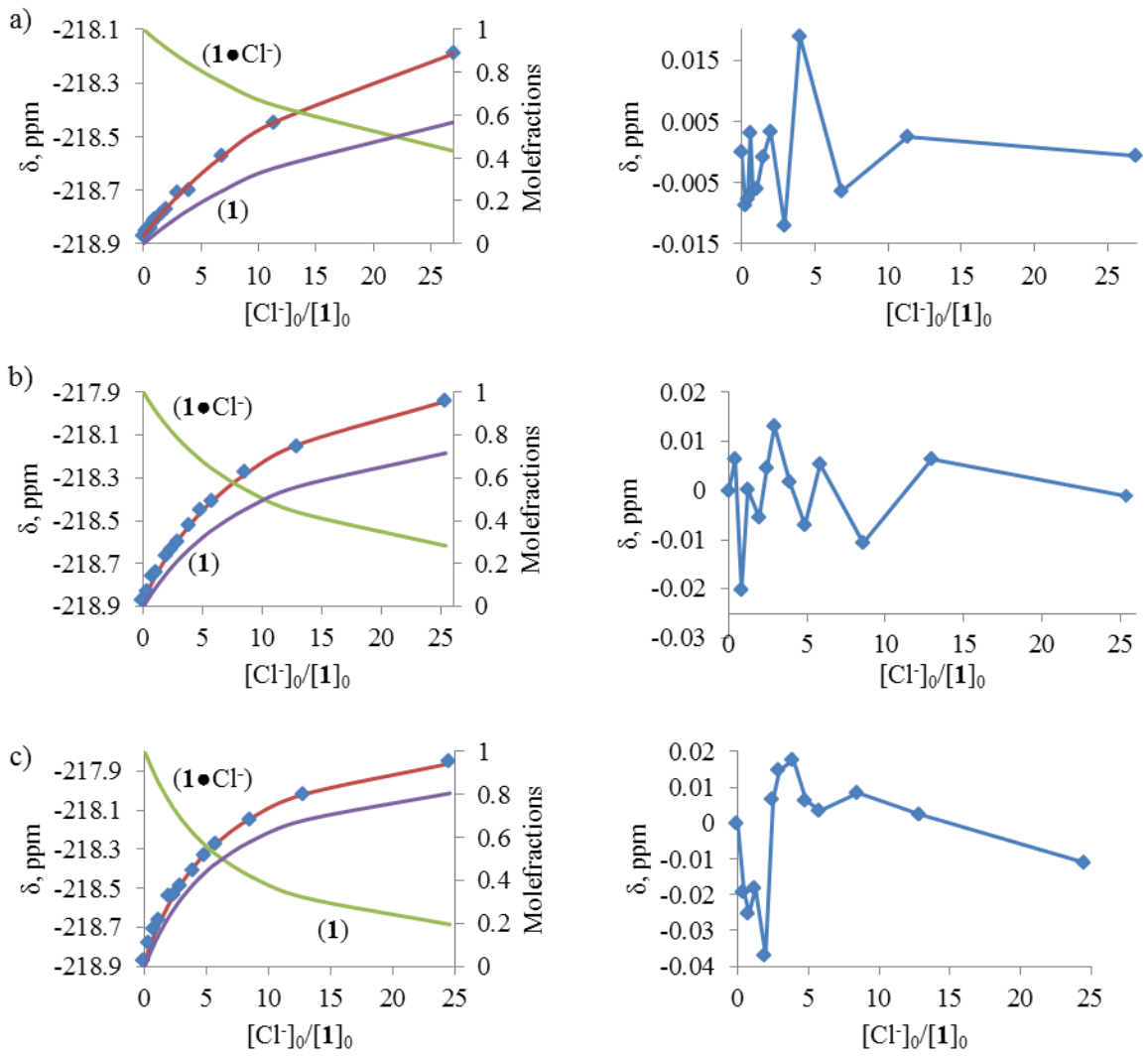


Figure S8. Left column: binding isotherms and species concentration plots for titration of **1** ($[\mathbf{1}]_0$: a) 2 mM, b) 5 mM, c) 10 mM) with TBACl in acetonitrile-d_3 at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

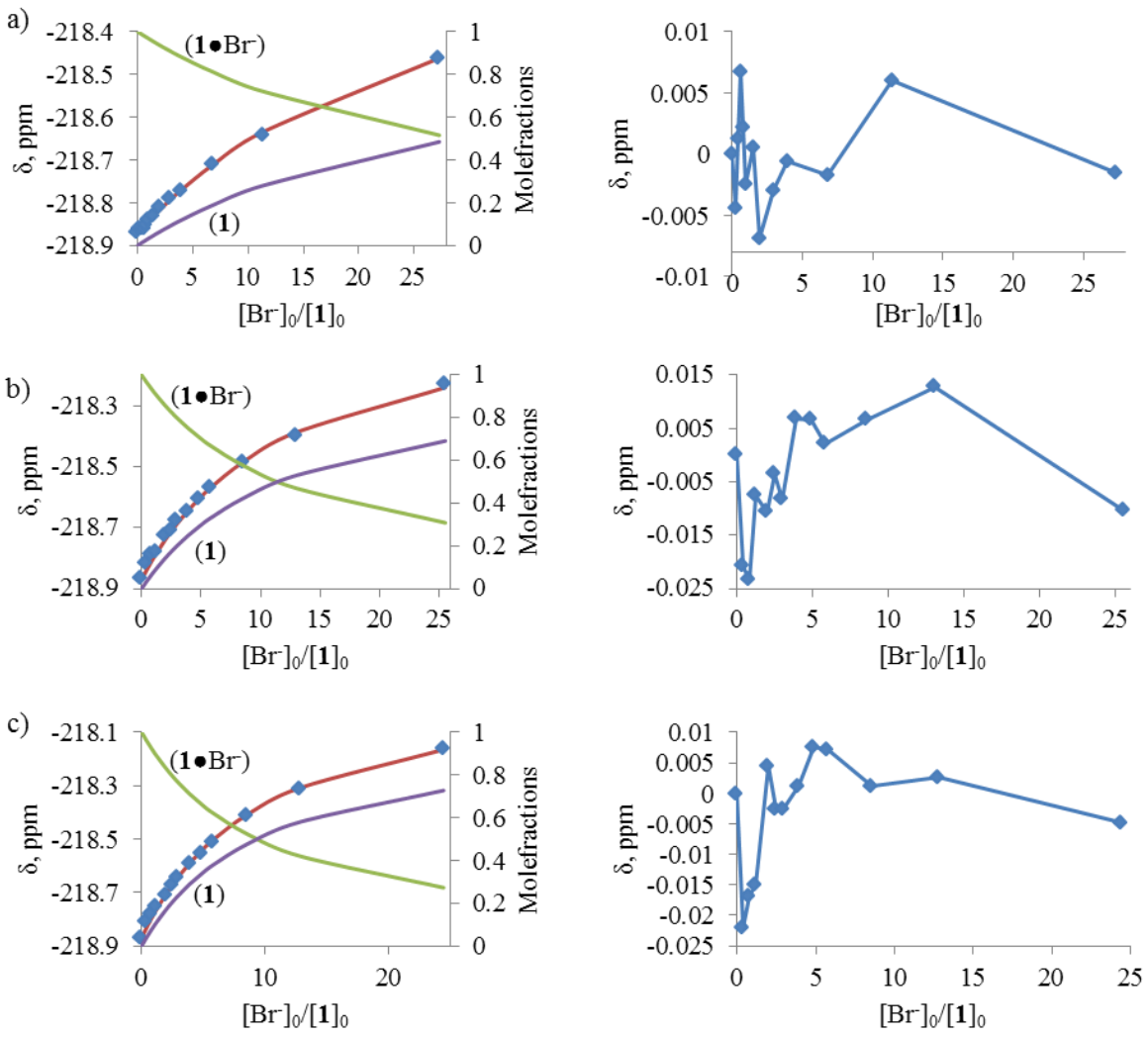


Figure S9. Left column: binding isotherms and species concentration plots for titration of **1** ([**1**]$_0$: a) 2 mM, b) 5 mM, c) 10 mM) with TBABr in acetonitrile-d_3 at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

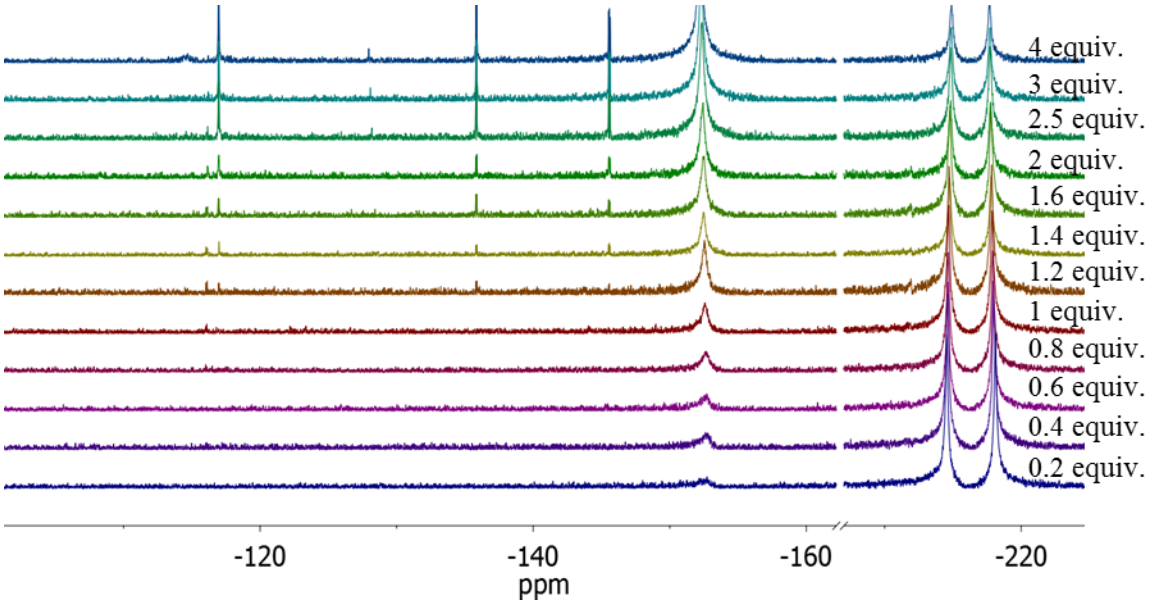


Figure S10. ^{19}F NMR stack plot for titration of **1** (5 mM) with TBAF in acetone-d_6 at 293 K. Signal at -218.4 ppm was used to obtain binding isotherms.

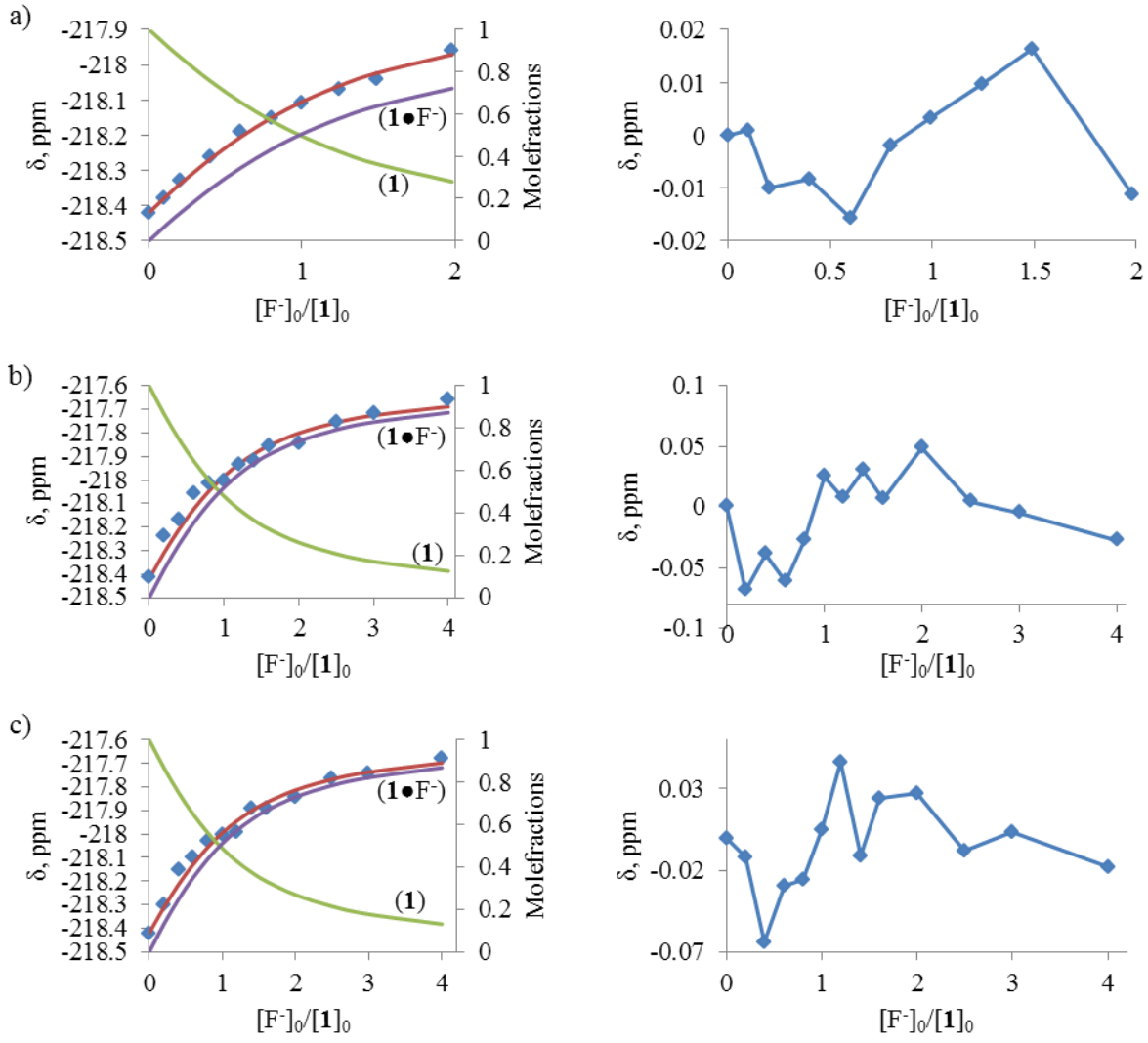


Figure S11. Left column: binding isotherms and species concentration plots for titration of **1** ([**1**]$_0$: a) 2 mM, b) and c) 5 mM) with TBAF in acetone-d_6 at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots.

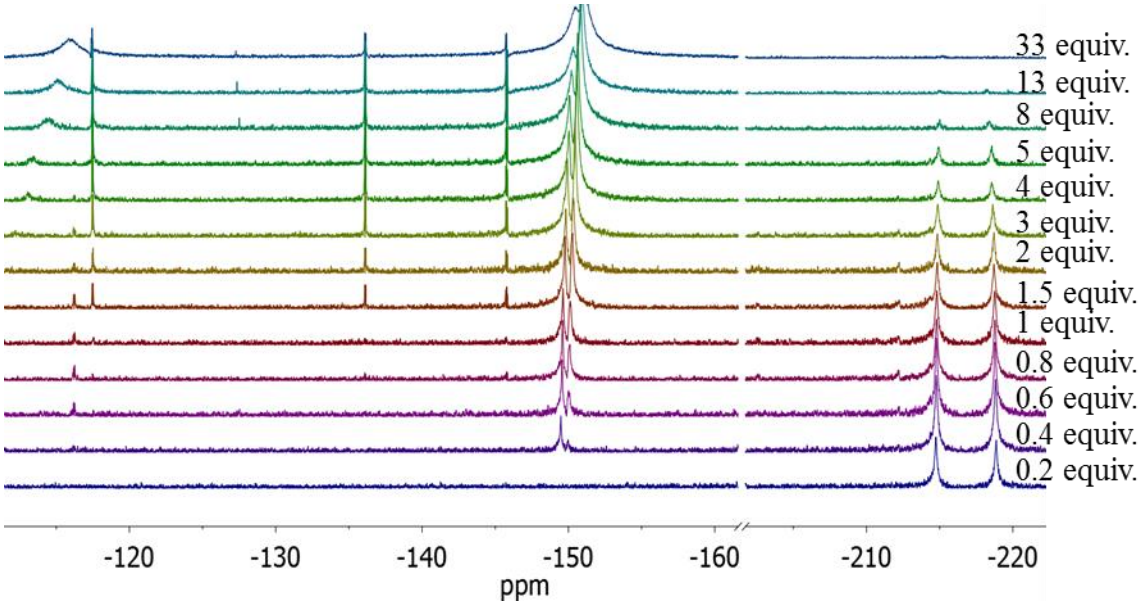


Figure S12. Decomposition of **1** (2 mM) during titration with TBAF in acetonitrile-d_3 at 293 K.

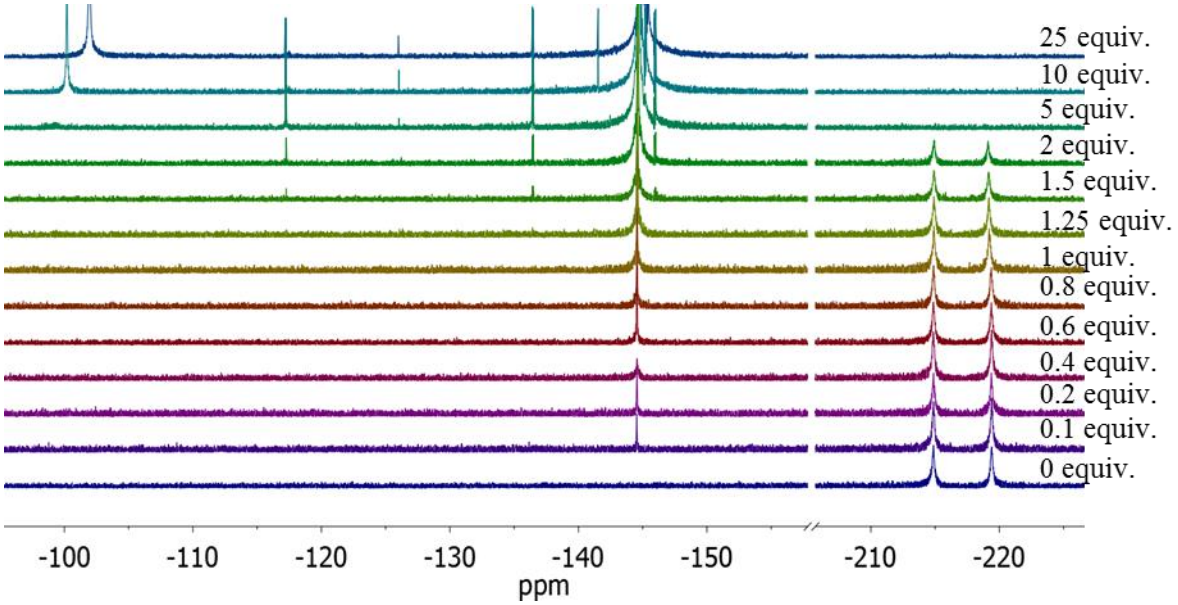


Figure S13. Decomposition of **1** (2 mM) during titration with TBAF in DMSO-d_6 at 293 K.

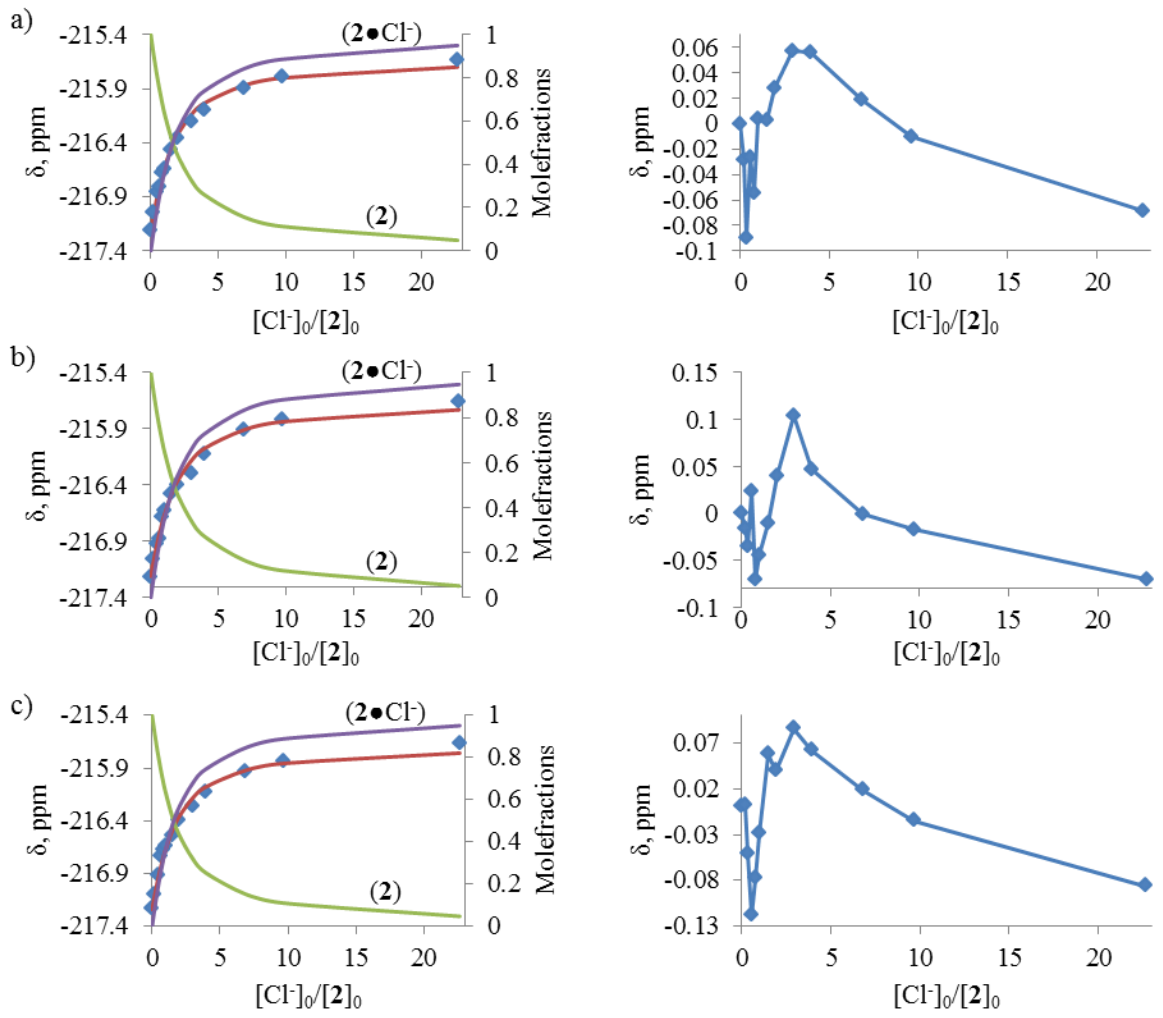


Figure S14. Left column: binding isotherms and species concentration plots for titration of **2** ([**2**]$_0$: 5 mM) with TBACl in acetone-d_6 at 293 K. Blue diamonds: experimental points, red line: fit according to 1:1 model, purple and green: mole fractions of corresponding species. Right column: residual plots. Chemical shift of only one fluorine was shown for clarity.

URLs to NMR Titrations

Table S3. URLs to the raw data, calculated fit and statistical information for the titration of **1** in acetone-d_6 at 293 K.

Salt	URL
TBAF	http://app.supramolecular.org/bindfit/view/7ea20f6e-20b4-4f03-abbd-573be2dbc4d2
	http://app.supramolecular.org/bindfit/view/a6144c0b-61d7-4a06-b524-fdd0c67561aa
	http://app.supramolecular.org/bindfit/view/853c7d7c-6e1d-4127-9ffc-0c7aba46ba38
TBACl	http://app.supramolecular.org/bindfit/view/ba5c0152-1b63-44bc-9fd6-d72b9c260660
	http://app.supramolecular.org/bindfit/view/f3127579-8411-4b61-94c5-df9914684a2d
	http://app.supramolecular.org/bindfit/view/f5f3822e-7224-433a-8218-f519cccc641a
TBABr	http://app.supramolecular.org/bindfit/view/ba9aab15-cc2a-4531-ba1e-dd5cfe07f653
	http://app.supramolecular.org/bindfit/view/f6147ca7-265b-4293-ae03-3045ed195d68

	http://app.supramolecular.org/bindfit/view/20591e77-a2ae-4326-abc1-4b692f3d9c74
TBAI	http://app.supramolecular.org/bindfit/view/4c42aede-b764-40d4-8ded-1e8cb188aaf0
	http://app.supramolecular.org/bindfit/view/987f6e84-497d-46d2-8f74-b90722895c3a
	http://app.supramolecular.org/bindfit/view/d3d769e8-333a-4a99-9e53-3f4e4189effe
	http://app.supramolecular.org/bindfit/view/b941fef1-1c8e-4813-8552-1bcd0c6bdf56
$TBANO_3$	http://app.supramolecular.org/bindfit/view/c22a80b8-4d48-4278-877c-4801dc4f6b32
	http://app.supramolecular.org/bindfit/view/1d19502b-057e-4349-8a86-69317d22c016
	http://app.supramolecular.org/bindfit/view/790f7df9-7a06-4ac8-8e3b-e639fdc174a2
	http://app.supramolecular.org/bindfit/view/dc963425-d231-4e92-a785-e1f4394201cd

Table S4. URLs to the raw data, calculated fit and statistical information for the titration of **1** in acetonitrile-d_3 at 293 K.

Salt	URL
TBACl	http://app.supramolecular.org/bindfit/view/fb0dd0cc-d8e9-43cd-9622-e42e42ca9fd2
	http://app.supramolecular.org/bindfit/view/05e6e248-8e8f-4477-b02e-e5df9b53dc99
	http://app.supramolecular.org/bindfit/view/503f5dca-a32f-4edb-ba80-8a4dd37af7b2
TBABr	http://app.supramolecular.org/bindfit/view/0a288146-6926-4708-b3d5-a22a39d0691d
	http://app.supramolecular.org/bindfit/view/62f19df1-1ff7-4bbe-964b-039a349a37b3
	http://app.supramolecular.org/bindfit/view/2b732ce3-1b2c-495e-b32f-768ffc68d996

Table S5. URLs to the raw data, calculated fit and statistical information for the titration of **2** in acetone-d_6 at 293 K.

Salt	URL
TBACl	http://app.supramolecular.org/bindfit/view/dd46e7f8-4c5b-4f8c-af30-ace29d420d7d
	http://app.supramolecular.org/bindfit/view/578ecf57-cc44-4de0-96c6-73b235571ede
	http://app.supramolecular.org/bindfit/view/d82fdca8-82c7-435c-b718-25baa95cbc9b

Crystallographic Data

All-cis 1,2,3,4,5,6-hexafluorocyclohexane (**1)** (5.2 mg, 0.027 mmol, 2 equiv.) and tetraphenylphosphonium chloride (5.1 mg, 0.0136 mmol, 1 equiv.) was dissolved in 1 mL of anhydrous acetone. To dissolve all tetraphenylphosphonium chloride, a small amount of chloroform was added. The obtained clear solution was filtered through a syringe filter and transferred into 1 mL clear vials (c.a. 0.1 mL in each). Solutions were frozen down in liquid nitrogen and anhydrous n-hexane was layered on top.

The cif-file was deposited in the Cambridge Structural Database under identifier CCDC 1826208.

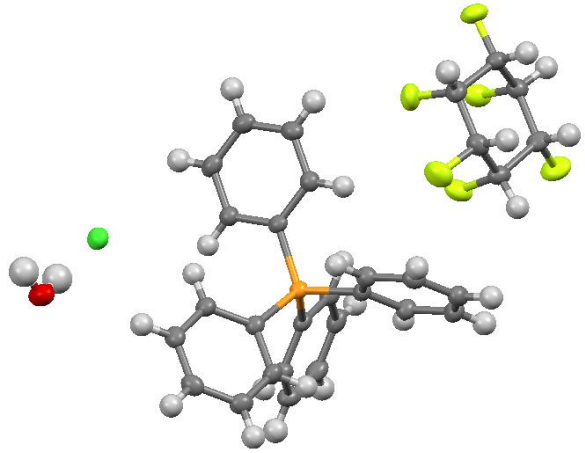


Figure S15. Co-crystal of **1** with tetraphenylphosphonium chloride. Thermal ellipsoids are shown at the 50% probability level.

Table S6. Crystal data and structure refinement for shy17098.[S4-S6]

Identification code	shy17098
Empirical formula	$C_{30}H_{28}ClF_6OP$
Formula weight	584.94
Temperature/K	150(2)
Crystal system	triclinic
Space group	P-1
a/Å	7.1352(3)
b/Å	13.5280(7)
c/Å	14.6245(8)
α/°	70.019(5)
β/°	86.507(4)
γ/°	89.808(4)
Volume/Å^3	1323.96(12)
Z	2
ρ_{calc}g/cm^3	1.467
μ/mm^{-1}	0.271
F(000)	604.0
Crystal size/mm^3	0.220 × 0.170 × 0.100
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.704 to 59.04
Index ranges	$-8 \leq h \leq 9$, $-18 \leq k \leq 18$, $-20 \leq l \leq 20$
Reflections collected	14548
Independent reflections	6333 [R_{int} = 0.0391, R_{sigma} = 0.0495]
Data/restraints/parameters	6333/0/355
Goodness-of-fit on F^2	1.044
Final R indexes [I>=2σ (I)]	R_1 = 0.0493, wR_2 = 0.1211

Table S7. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for shy17098. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	***x***	***y***	***z***	**U(eq)**
P001	909.1(7)	8252.5(3)	7791.6(3)	23.74(13)
Cl02	-1162.1(7)	6335.4(4)	5398.6(4)	34.62(14)
F003	-4670.7(17)	6943.6(10)	12329.2(9)	41.7(3)
F004	-1589.9(18)	7958.7(10)	11117.4(9)	44.1(3)
F005	-3898.1(19)	4894.9(9)	13375.7(10)	46.4(3)
F006	-2776.2(19)	8527.0(9)	12687.4(11)	49.3(4)
F007	-2242(2)	5772.1(11)	11553.2(10)	50.0(4)
F008	1275.3(19)	6599.3(11)	11277.5(10)	50.9(4)
O00	2445(2)	6089.1(12)	4183.1(12)	42.0(4)
C00A	2627(3)	8195.8(14)	6867.6(14)	25.7(4)
C00B	2171(3)	8355.0(13)	8784.1(14)	25.0(4)
C00C	-493(3)	9405.1(13)	7330.8(14)	24.6(4)
C00D	-652(3)	7131.6(13)	8194.9(14)	25.0(4)
C00E	2589(3)	7462.1(14)	6394.7(14)	28.7(4)
C00F	4105(3)	8943.6(15)	6631.1(15)	29.6(4)
C00G	-1760(3)	6950.7(14)	7507.0(14)	27.8(4)
C00H	2(3)	10202.7(14)	6454.9(15)	30.0(4)
C00I	1824(3)	9157.5(14)	9163.3(15)	28.3(4)
C00J	-854(3)	6486.5(14)	9172.4(14)	28.7(4)
C00K	-2068(3)	9502.7(14)	7906.5(15)	28.4(4)
C00L	5490(3)	8953.5(15)	5928.1(16)	32.2(4)
C00M	-3020(3)	6112.1(15)	7790.8(16)	32.2(4)
C00N	3996(3)	7479.1(14)	5694.2(15)	31.6(4)
C00O	-2143(3)	5648.6(15)	9448.6(15)	32.4(4)
C00P	5438(3)	8220.8(15)	5459.7(15)	32.0(4)
C00Q	4604(3)	7701.8(15)	9896.9(15)	31.9(4)
C00R	4254(3)	8501.6(15)	10277.2(15)	32.1(4)
C00S	-2514(3)	5667.0(14)	13215.9(15)	31.3(4)
C00T	3580(3)	7632.7(14)	9149.3(14)	28.8(4)
C00U	-3455(3)	6707.3(15)	13083.5(15)	31.0(4)
C00V	-3110(3)	10414.5(15)	7606.4(16)	32.3(4)
C00W	-1939(3)	7549.7(14)	12847.8(16)	32.5(4)
C00X	-3212(3)	5469.1(15)	8758.1(16)	32.9(4)
C00Y	2873(3)	9223.6(15)	9910.1(16)	33.7(5)
C00Z	-1214(3)	5663.5(15)	12362.8(16)	34.9(5)
C010	-632(3)	7622.4(15)	11972.5(15)	32.0(4)
C011	-1057(3)	11111.2(15)	6158.3(16)	35.9(5)
C012	-2596(3)	11213.3(15)	6740.4(17)	35.8(5)
C013	212(3)	6553.8(16)	12122.0(16)	33.6(4)

Table S8. Anisotropic displacement parameters ($Å^2 \times 10^3$) for shy17098. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
P001	25.0(3)	20.2(2)	26.9(3)	-9.11(19)	-2.63(19)	1.69(17)
Cl02	37.1(3)	32.2(3)	37.7(3)	-16.8(2)	2.4(2)	0.8(2)
F003	30.8(7)	43.4(7)	43.2(7)	-3.8(6)	-8.7(5)	-0.2(5)
F004	41.7(7)	43.6(7)	34.8(7)	3.0(6)	-5.7(6)	0.2(6)
F005	51.6(8)	30.8(6)	53.3(8)	-10.7(6)	2.1(6)	-13.5(6)
F006	50.4(8)	25.3(6)	71.8(10)	-17.1(6)	1.0(7)	7.5(5)
F007	60.4(9)	58.8(8)	38.3(7)	-26.2(7)	-3.3(7)	-9.8(7)
F008	45.6(8)	56.0(8)	48.1(8)	-16.9(7)	13.9(6)	3.1(6)
O00	42.0(9)	34.4(8)	49.2(10)	-16.0(7)	9.3(7)	-2.2(7)
C00A	26.0(9)	23.1(8)	28.9(10)	-9.5(7)	-3.9(8)	3.9(7)
C00B	24.8(9)	23.8(8)	27.5(10)	-10.4(7)	-1.5(7)	-0.1(7)
C00C	25.0(9)	19.8(8)	30.8(10)	-10.2(7)	-5.8(8)	1.9(7)
C00D	25.5(9)	20.5(8)	30.4(10)	-10.9(7)	0.8(7)	1.5(7)
C00E	32.1(10)	22.9(9)	31.4(10)	-9.5(8)	-1.9(8)	0.4(7)
C00F	27.6(10)	28.4(9)	35.4(11)	-14.2(8)	-4.1(8)	-1.1(7)
C00G	29.7(10)	25.2(9)	29.2(10)	-10.1(8)	-3.1(8)	1.2(7)
C00H	28.3(10)	26.3(9)	33.8(11)	-8.0(8)	-2.7(8)	-0.7(7)
C00I	28.6(10)	25.1(9)	32.3(10)	-10.7(8)	-5.5(8)	4.1(7)
C00J	29.6(10)	28.0(9)	29.3(10)	-10.8(8)	-2.5(8)	2.8(7)
C00K	28.1(10)	25.8(9)	32.3(10)	-11.2(8)	-2.8(8)	0.3(7)
C00L	24.9(10)	31.7(10)	37.8(11)	-9.1(9)	-3.3(8)	1.0(8)
C00M	28.1(10)	31.7(10)	41.7(12)	-18.9(9)	-2.8(9)	-0.4(8)
C00N	38.7(11)	25.0(9)	32.5(11)	-11.7(8)	-2.2(9)	6.4(8)
C00O	36.4(11)	25.0(9)	32.7(11)	-6.8(8)	2.4(9)	1.1(8)
C00P	27.9(10)	33.5(10)	31.4(10)	-7.7(8)	2.0(8)	6.8(8)
C00Q	27.1(10)	29.6(10)	35.3(11)	-6.4(8)	-2.1(8)	3.1(8)
C00R	31.4(11)	34.1(10)	29.9(10)	-9.3(8)	-4.8(8)	-3.9(8)
C00S	33.4(11)	24.4(9)	34.7(11)	-8.3(8)	-2.7(9)	-4.0(8)
C00T	28.9(10)	26.4(9)	31.7(10)	-11.2(8)	-0.8(8)	3.3(7)
C00U	29.8(10)	30(1)	30.8(10)	-7.4(8)	-1.8(8)	2.0(8)
C00V	28.9(10)	32.3(10)	43.1(12)	-21.6(9)	-7.3(9)	6.1(8)
C00W	33.6(11)	21.8(9)	41.6(12)	-10.2(8)	-3.7(9)	4.9(8)
C00X	29.7(11)	26.3(9)	43.9(12)	-14.7(9)	4.5(9)	-1.4(8)
C00Y	38.4(12)	29.4(10)	36.6(11)	-15.2(9)	-3.1(9)	0.8(8)
C00Z	42.8(12)	27.9(10)	35.4(11)	-12.5(9)	-2.3(9)	3.0(8)
C010	29.9(10)	28.0(9)	34.3(11)	-5.3(8)	-5.0(8)	-2.0(8)
C011	39.3(12)	24.7(9)	39.4(12)	-4.3(8)	-10.2(10)	-0.1(8)
C012	36.3(11)	25.4(9)	48.9(13)	-15.3(9)	-13.1(10)	6.1(8)
C013	29.3(11)	36.6(11)	33.2(11)	-10.6(9)	1.6(8)	3.1(8)

Table S9. Bond lengths for shy17098.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
P001	C00A	1.789(2)	C00G	C00M	1.381(3)
P001	C00D	1.7909(18)	C00H	C011	1.392(3)
P001	C00B	1.7983(19)	C00I	C00Y	1.389(3)
P001	C00C	1.8000(18)	C00J	C00O	1.394(3)
F003	C00U	1.395(2)	C00K	C00V	1.390(3)
F004	C010	1.397(2)	C00L	C00P	1.386(3)
F005	C00S	1.390(2)	C00M	C00X	1.383(3)
F006	C00W	1.400(2)	C00N	C00P	1.383(3)
F007	C00Z	1.396(2)	C00O	C00X	1.387(3)
F008	C013	1.392(2)	C00Q	C00T	1.380(3)
C00A	C00E	1.391(3)	C00Q	C00R	1.391(3)
C00A	C00F	1.407(3)	C00R	C00Y	1.383(3)
C00B	C00I	1.393(3)	C00S	C00Z	1.510(3)
C00B	C00T	1.398(3)	C00S	C00U	1.514(3)
C00C	C00H	1.390(3)	C00U	C00W	1.511(3)
C00C	C00K	1.396(3)	C00V	C012	1.384(3)
C00D	C00J	1.396(3)	C00W	C010	1.512(3)
C00D	C00G	1.401(3)	C00Z	C013	1.512(3)
C00E	C00N	1.384(3)	C010	C013	1.515(3)
C00F	C00L	1.379(3)	C011	C012	1.384(3)

Table S10. Bond angles for shy17098.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C00A	P001	C00D	112.60(8)	C00X	C00O	C00J	120.05(19)
C00A	P001	C00B	106.91(9)	C00N	C00P	C00L	120.15(19)
C00D	P001	C00B	110.95(9)	C00T	C00Q	C00R	120.00(18)
C00A	P001	C00C	109.95(9)	C00Y	C00R	C00Q	119.98(18)
C00D	P001	C00C	107.60(8)	F005	C00S	C00Z	108.69(16)
C00B	P001	C00C	108.78(8)	F005	C00S	C00U	108.58(16)
C00E	C00A	C00F	119.46(18)	C00Z	C00S	C00U	114.35(17)
C00E	C00A	P001	124.22(15)	C00Q	C00T	C00B	120.11(17)
C00F	C00A	P001	116.31(14)	F003	C00U	C00W	109.92(16)
C00I	C00B	C00T	119.87(17)	F003	C00U	C00S	110.65(16)
C00I	C00B	P001	121.52(14)	C00W	C00U	C00S	108.02(16)
C00T	C00B	P001	118.56(14)	C012	C00V	C00K	120.16(19)
C00H	C00C	C00K	120.29(17)	F006	C00W	C00U	109.15(16)
C00H	C00C	P001	121.91(15)	F006	C00W	C010	108.61(16)
C00K	C00C	P001	117.74(14)	C00U	C00W	C010	114.62(17)
C00J	C00D	C00G	120.32(17)	C00M	C00X	C00O	120.92(18)
C00J	C00D	P001	121.66(14)	C00R	C00Y	C00I	120.57(18)
C00G	C00D	P001	117.92(14)	F007	C00Z	C00S	110.13(17)
C00N	C00E	C00A	119.78(18)	F007	C00Z	C013	109.89(17)
C00L	C00F	C00A	120.04(18)	C00S	C00Z	C013	109.74(16)
C00M	C00G	C00D	119.92(19)	F004	C010	C00W	110.84(16)
C00C	C00H	C011	119.90(19)	F004	C010	C013	110.52(17)
C00Y	C00I	C00B	119.46(18)	C00W	C010	C013	108.85(16)
C00O	C00J	C00D	119.05(18)	C012	C011	C00H	119.6(2)
C00V	C00K	C00C	119.36(18)	C011	C012	C00V	120.66(19)
C00F	C00L	C00P	120.06(18)	F008	C013	C00Z	108.73(16)
C00G	C00M	C00X	119.73(19)	F008	C013	C010	108.63(17)
C00P	C00N	C00E	120.51(18)	C00Z	C013	C010	114.32(17)

Table S11. Hydrogen atom coordinates (Å×10^4) and isotropic displacement parameters (Å^2×10^3) for shy17098.

Atom	***x***	***y***	***z***	**U(eq)**
H00A	1473.2	6132.61	4532.67	63
H00B	2466.79	5446.92	4225.46	63
H00E	1600.97	6951.09	6552.07	34
H00F	4148.73	9442.21	6955.9	35
H00G	-1644.21	7404.27	6846.26	33
H00H	1061.55	10127.92	6059.25	36
H00I	877.9	9655.64	8912.94	34
H00J	-123.57	6616.71	9642.59	34
H00K	-2423.9	8951.01	8497.68	34
H00L	6481.24	9463	5764.66	39
H00M	-3752.67	5977.53	7323.53	39
H00N	3969.99	6977.46	5372.05	38
H00O	-2289.61	5200.15	10110.36	39
H00P	6395.04	8228.15	4976.4	38
H00Q	5547.02	7203.15	10151.74	38
H00R	4964.16	8551.86	10789.05	39
H00S	-1770.72	5484.59	13807.64	38
H00T	3834.51	7093.41	8882.48	35
H00U	-4177.09	6668.74	13702.87	37
H00V	-4178.35	10489.94	7996.29	39
H00W	-1167.94	7398.33	13427.33	39
H00X	-4087.4	4896.48	8952.12	39
H00Y	2640.67	9769.38	10171.09	40
H00Z	-541.36	4980.5	12541.73	42
H010	401.51	8140.42	11918.54	38
H011	-724.3	11657.76	5559.93	43
H012	-3307.18	11837.74	6543.83	43
H013	1069.05	6395.2	12668.36	40

References

S1 M. P. Wiesenfeldt, Z. Nairoukh, W. Li and F. Glorius, *Science*, 2017, **357**, 908-912.

S2 http://supramolecular.org/

S3 D. B. Hibbert and P. Thordarson, *Chem. Commun.*, 2016, **52**, 12792-12805.

S4 V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Cryst.*, 2009, **42**, 339-341.

S5 G. M. Sheldrick, *Acta Cryst.* 2015, **A71**, 3-8.

S6 G. M. Sheldrick, *Acta Cryst*. 2015, **C71**, 3-8.