Intramolecularly Coordinated Bis(crown ether)-Substituted Organotin Halides as Ditopic Salt Receptors

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Supporting Information

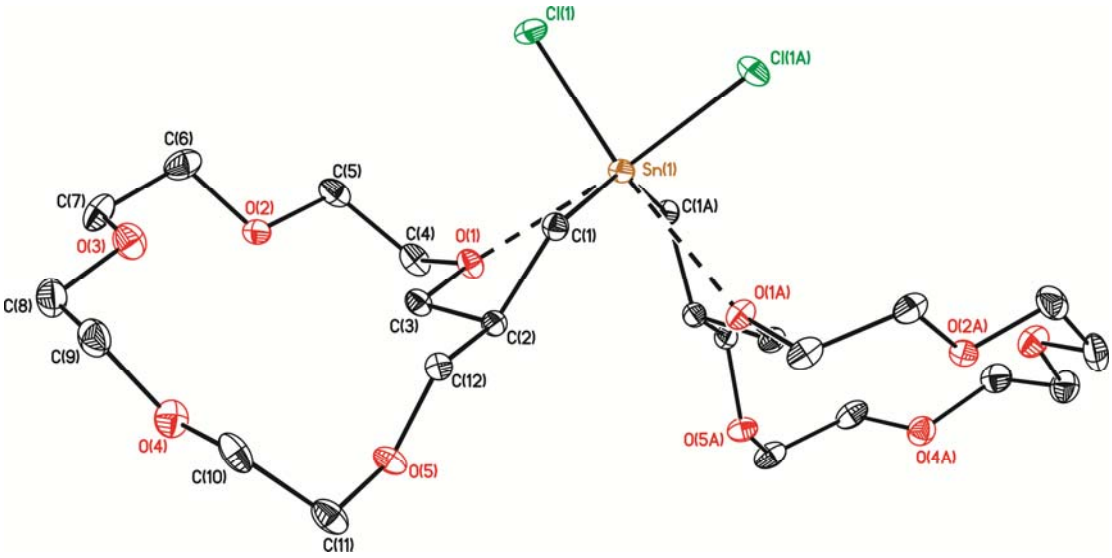

Figure S1. Molecular structure of diorganodichloridostannane **5** showing 30% probability displacement ellipsoides and crystallographic numbering scheme. The hydrogen atoms are omitted for clarity. Symmetry code a: 1–x+1,y,–z+3/2.

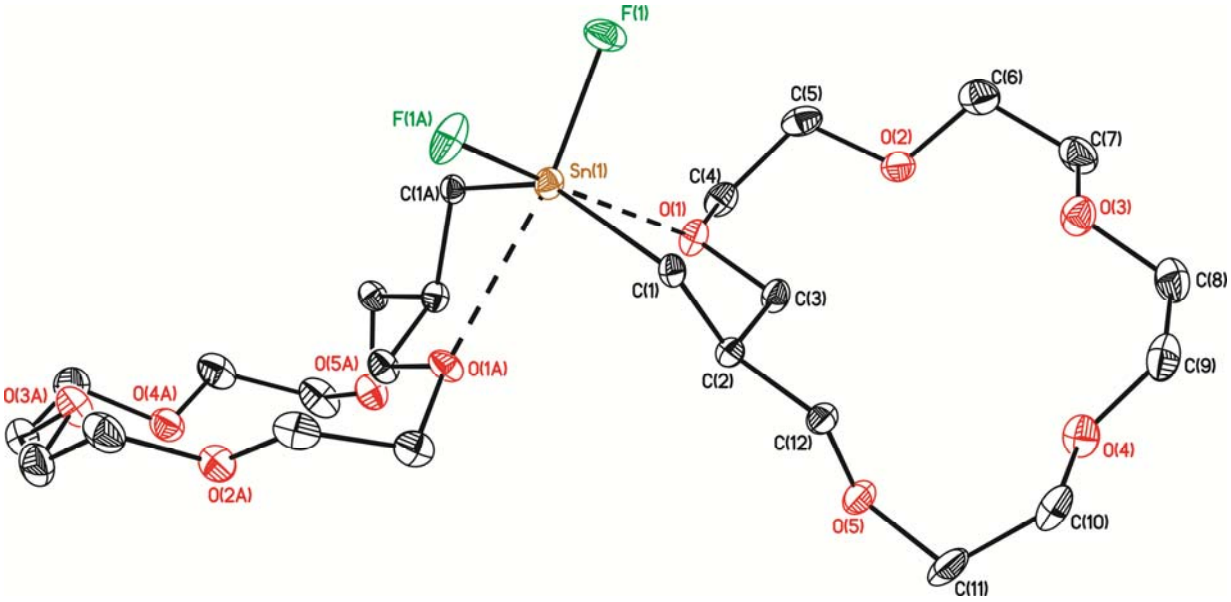

Figure S2. Molecular structure of diorganodifluoridostannane **6** showing 30% probability displacement ellipsoides and crystallographic numbering scheme. The hydrogen atoms are omitted for clarity. Symmetry code a: –x,y,–z+1/2.

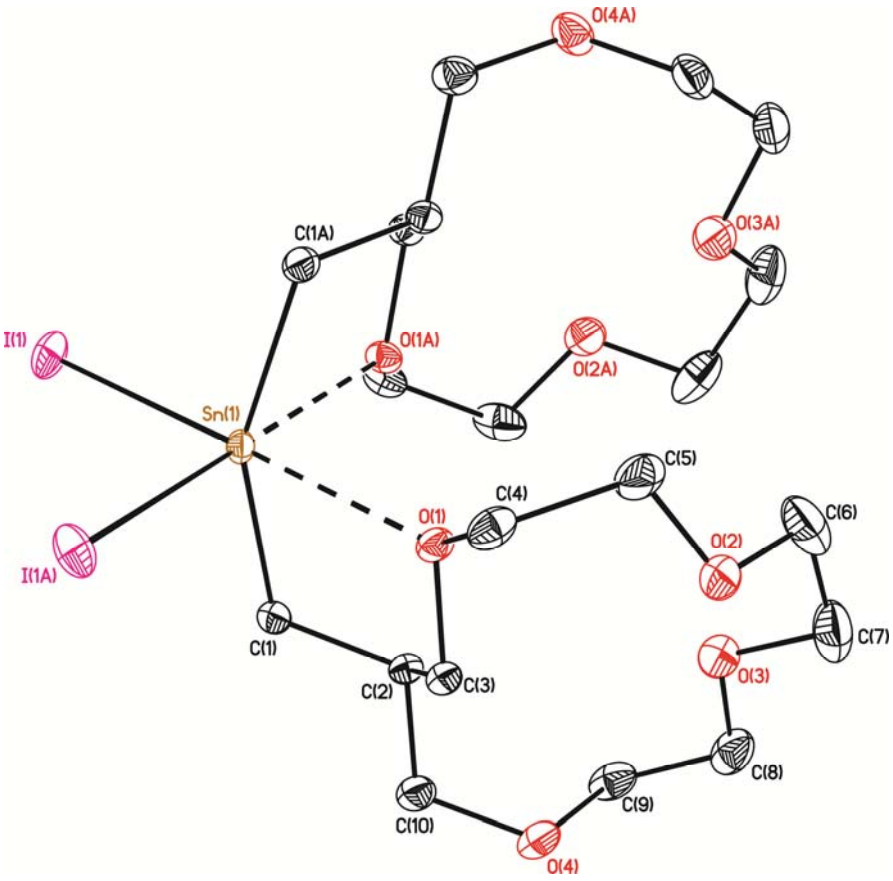

Figure S3. Molecular structure of diorganodiiodidostannane **10** showing 30% probability displacement ellipsoides and crystallographic numbering scheme. The hydrogen atoms are omitted for clarity. Symmetry code a: –x+1,y,–z+1/2.

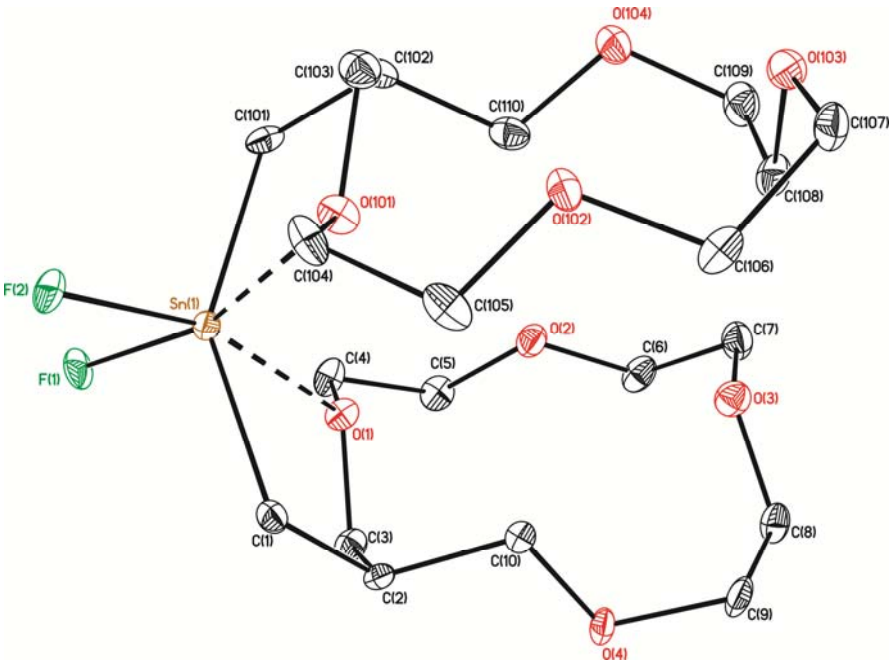

Figure S4. Molecular structure of diorganodifluoridostannane **12** showing 30% probability displacement ellipsoides and crystallographic numbering scheme. The hydrogen atoms are omitted for clarity.

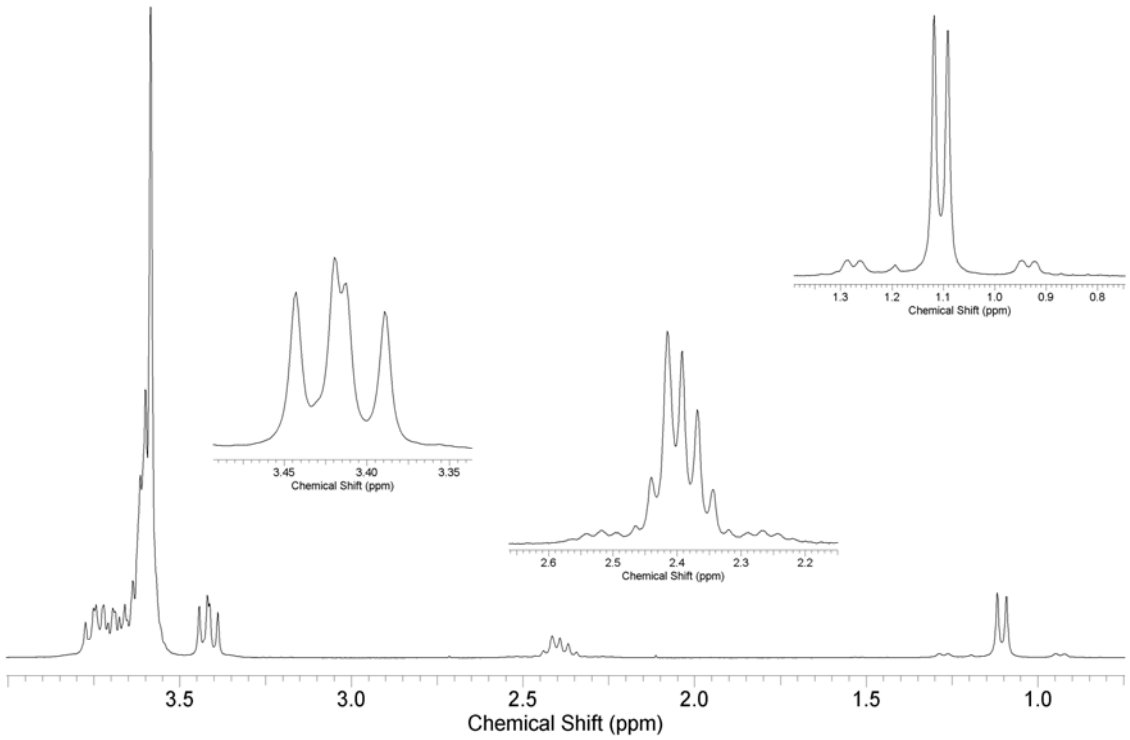

Figure S5. ^{1}H NMR spectrum of diorganodifluoridostannane **6** in $CDCl_3$ (300 MHz).

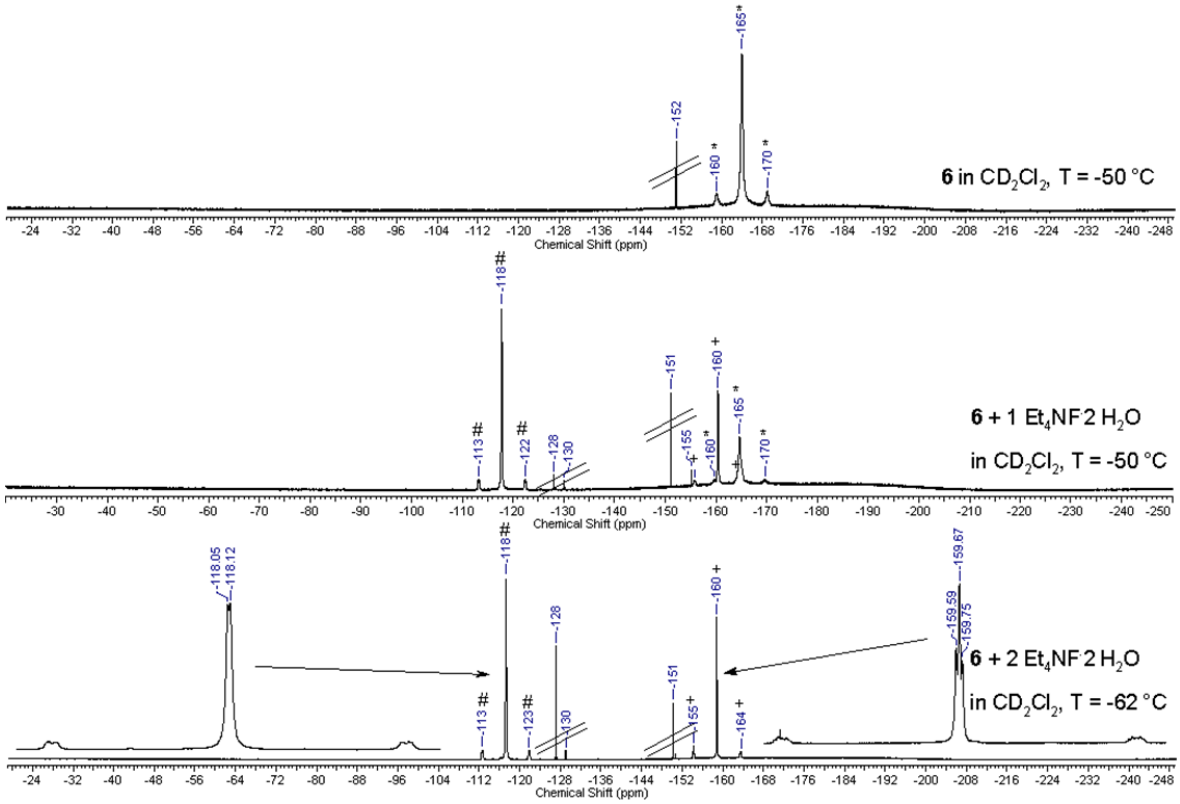

Figure S6. ^{19}F NMR spectra of solutions of diorganodifluoridostannane **6** with varying amounts of fluoride anions: a) pure compound **6**; b) compound **6** with one equivalent of fluoride; c) compound **6** with two equivalents of fluoride.

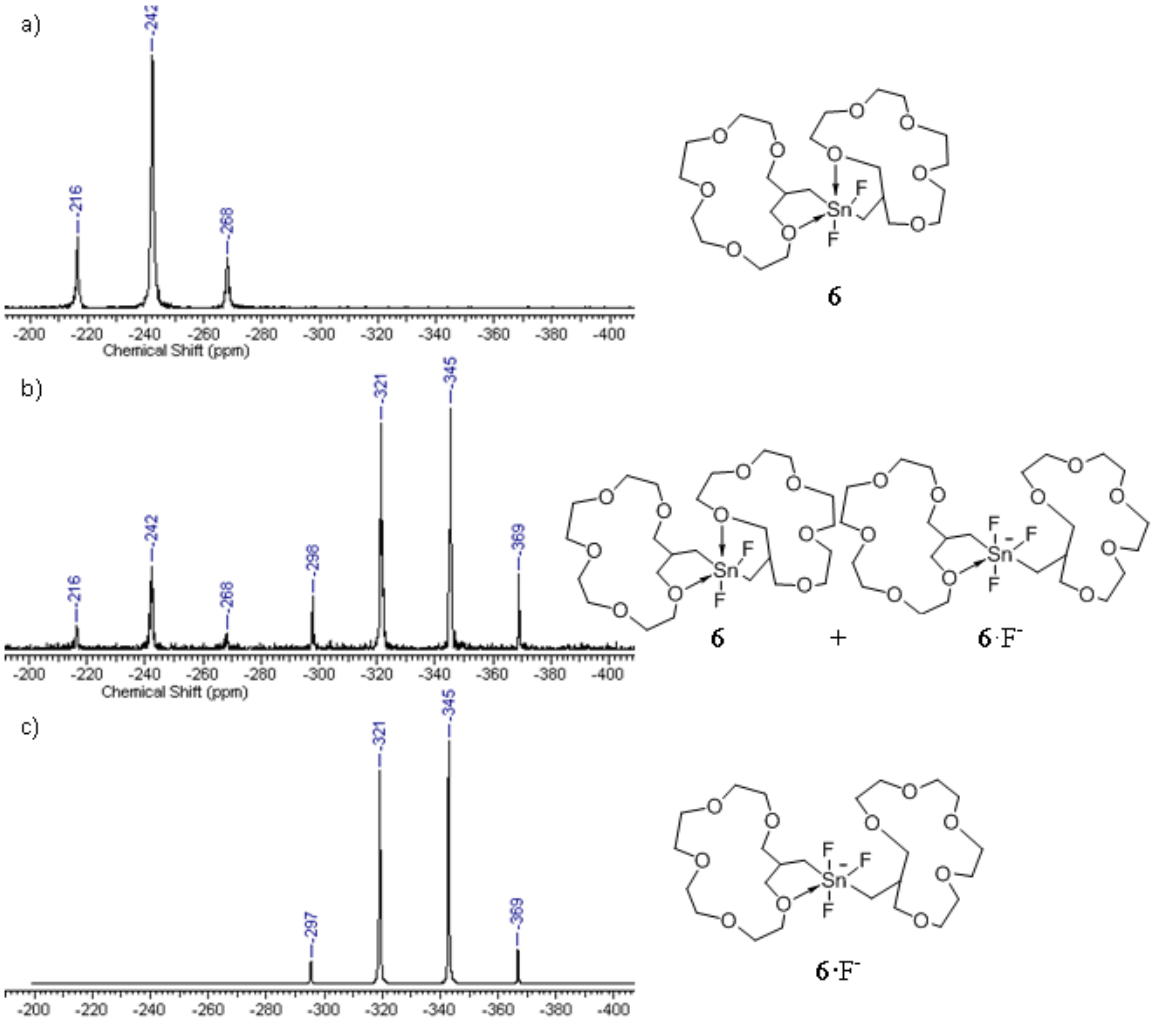

Figure S7. ^{119}Sn NMR spectra of a) diorganodifluorido stannane **6** (CD_2Cl_2, –50°C, 111.92 MHz); b) a mixture of diorganodifluoridostannane **6** and the diorganotrifluorido stannate complex **6**·F^- after one equivalent of $Et_4NF{\cdot}2\ H_2O$ had been added to a solution of **6** in CD_2Cl_2 (–50°C, 111.92 MHz); c) the diorganotrifluorido stannate complex **6**·F^- after two equivalents of $Et_4NF{\cdot}2\ H_2O$ had been added to a solution of **6** in CD_2Cl_2 (–50°C, 111.92 MHz).

Table S1. Crystal data and structure refinement for compound **4**, **5** and **6**.

	4	**5**	**6**
Empirical formula	$C_{24}H_{46}Br_2O_{10}Sn$	$C_{24}H_{46}Cl_2O_{10}Sn$	$C_{24}H_{46}F_2O_{10}Sn$
Temperature /K	173(2)	173(2)	173(2)
Wavelength /Å	0.71073	0.71073	0.71073
Crystal System	monoclinic	monoclinic	monoclinic
Crystal size, mm	0.50x0.37x0.30	0.17x0.12x0.04	0.18x0.05x0.04
Space group	*I*2/a	*I*2/a	*I*2/a
a / Å	16.1335(19)	15.948(4)	14.586(6)
b / Å	11.5894(7)	11.4212(6)	12.2579(13)
c / Å	16.415(3)	16.389(2)	16.169(4)
α / °	90	90	90
β / °	100.304(18)	100.19(2)	102.09(4)
γ / °	90	90	90
Volume / Å³	3019.7(7)	2938.1(8)	2826.7(14)
Z	4	4	4
D_c /(g/cm³)	1.701	1.547	1.530
Absorption coefficient /mm^{-1}	3.542	1.102	0.968
F(000)	1560	1416	1352
θ range for data collection	2.52-25.50	2.18-25.50	2.19-25.49
Reflections collected	9403	12808	7918
Completenes to θ_{max} / %	99.9	100.0	100.0
Independent reflections / $R_{int.}$	2814/ 0.0294	2745 /0.0502	2629/ 0.0467
Parameter / restraints	168/ 0	168/ 0	168/ 0
GooF (F^2)	0.922	0.810	0.807
R_1(F) [$I > 2\sigma(I)$]	0.0239	0.0238	0.0329
$wR_2(F^2)$ (alle Daten)	0.0505	0.0360	0.0305
Largest diff. peak and hole, e$Å^{-3}$	0.595 / -0.428	0.662 / –0.292	1.096 / -0.547

Table S2. Crystal data and structure refinement for compound **10**, **11** and **12**.

	10	**11**	**12**
Empirical formula	$C_{20}H_{38}I_2O_8Sn$	$C_{20}H_{38}Br_2O_8Sn$	$C_{20}H_{38}F_2O_8Sn$
Temperature /K	173(2)	173(2)	173(2)
Wavelength /Å	0.71073	0.71073	0.71073
Crystal System	monoclinic	monoclinic	orthorhombic
Crystal size, mm	0.25x0.24x0.14	0.35x0.30x0.20	0.50x0.11x0.08
Space group	*I*2/a	*I*2/a	*Pbca*
a / Å	14.020(5)	13.743(3)	16.6098(7)
b / Å	11.6934(7)	11.334(2)	14.2088(7)
c / Å	17.380(3)	17.683(4)	19.5728(10)
α / °	90	90	90
β / °	101.20(4)	97.82(3)	90
γ / °	90	90	90
Volume / Å³	2795.0(12)	2728.7(9)	4619.3(4)
Z	4	4	8
D_c /(g/cm³)	1.851	1.623	1.620
Absorption coefficient /mm^{-1}	3.160	3.901	1.165
F(000)	1512	1296	2320
θ range for data collection	2.29 – 25.50	2.14 – 25.50	2.42 – 25.49
Reflections collected	6981	25024	14098
Completenes to θ_{max} / %	100.0	99.9	100.0
Independent reflections / $R_{int.}$	2605/ 0.0362	2537/ 0.0376	4297/ 0.0487
Parameter / restraints	141/ 0	148/ 0	280/ 0
GooF (F^2)	0.913	1.122	0.852
R_1(F) [$I > 2\sigma(I)$]	0.0258	0.0420	0.0292
$wR_2(F^2)$ (alle Daten)	0.0551	0.1482	0.0521
Largest diff. peak and hole, e$Å^{-3}$	0.829 / -0.621	1.942 / -1.518	0.841/ –0.487