Thio-, seleno- and telluro-ether complexes of aluminium(III) halides: synthesis, structures and properties

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[*o*-$C_6H_4CH_2Se(Me)CH_2$][$AlCl_4$]:

o-$C_6H_4(CH_2SeMe)_2$ (0.12 g, 0.38 mmol) was added dropwise to a suspension of $AlCl_3$ (0.10 g, 0.75 mmol) in anhydrous CH_2Cl_2 (10 mL) with stirring to give a yellow solution. After 30 minutes all solvent was removed *in vacuo* to yield an orange oil. On standing, crystals suitable for X-ray diffraction formed in the oil. Yield 0.17 g, 78%. ^{27}Al NMR ($CDCl_3$, 295 K): 103.0. ^{77}Se NMR ($CDCl_3$, 295 K): 383. IR (cm^{-1}, Nujol): 491(s), 348(w). ES^+ MS (MeCN): *m/z* = 199 [*o*-$C_6H_4CH_2Se(Me)CH_2]^+$; ES^- MS (MeCN): *m/z* = 169 $[AlCl_4]^-$.

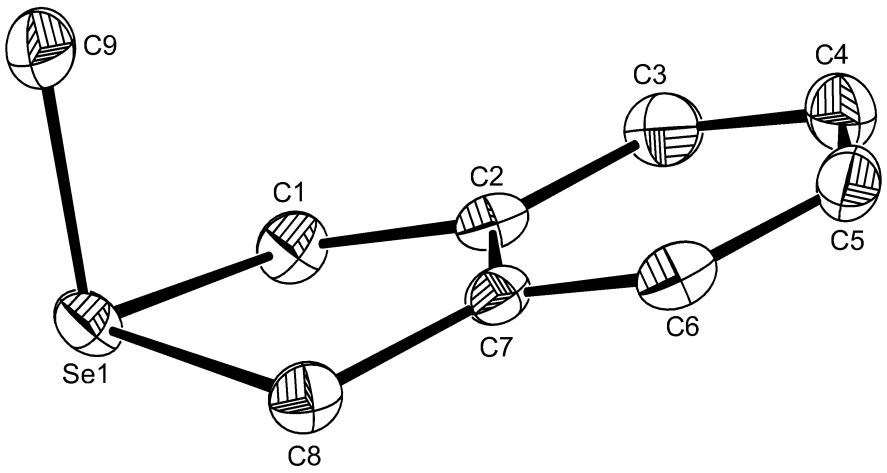

Fig S1. The structure of the cation in [$C_9H_{11}Se$][$AlCl_4$] showing the atom numbering scheme. Ellipsoids are drawn at the 50% probability level and H atoms are omitted for clarity. The molecule has 2-fold symmetry. Symmetry operation: a = –x, y, 1/2 – z.

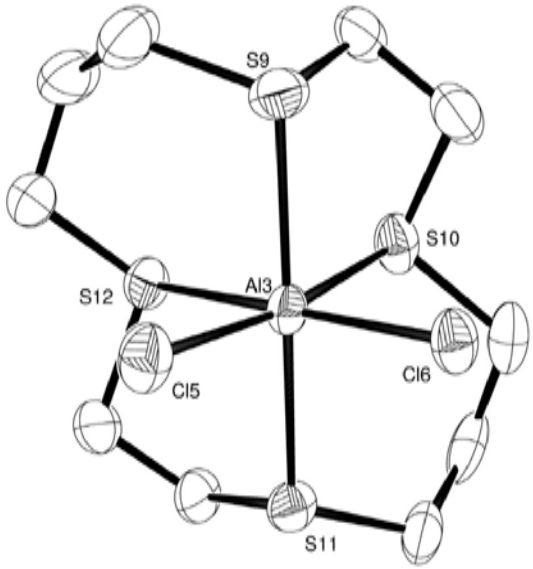

Fig S2. View of one of the cations in $[AlCl_2([14]aneS_4)][AlCl_4]$ showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are omitted for clarity.

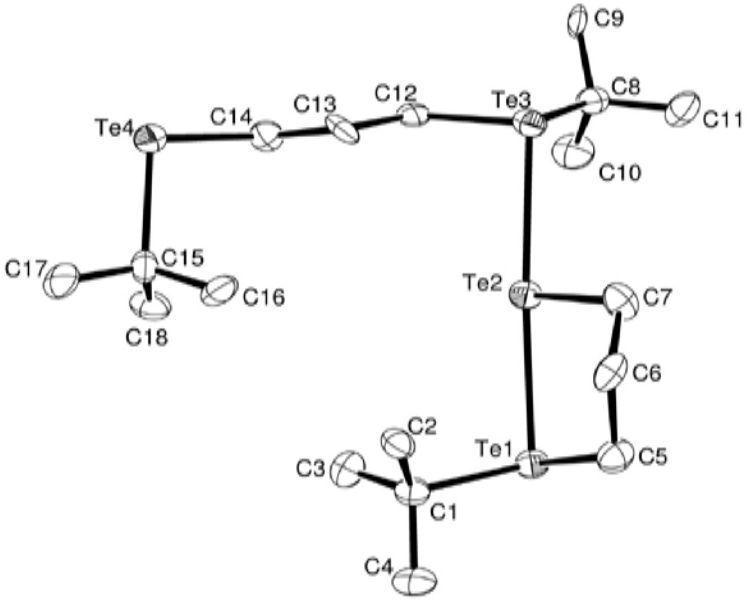

Fig S3 Structure of the cation in $[^tBuTe(CH_2)_3Te(^tBu)Te(CH_2)_3Te^tBu][AlCl_4]$. Ellipsoids are shown at the 40% probability level and H atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Te1-Te2 = 2.937(2), Te2-Te3 = 2.990(2), Te1-Te2-Te3 = 170.33(6).

Table S1 X-ray data for [*o*-$C_6H_4CH_2Se(Me)CH_2$][$AlCl_4$], [$AlCl_2$([14]aneS_4)] [$AlCl_4$] and [^{t}BuTe$(CH_2)_3$Te(^{t}Bu)Te $(CH_2)_3$Te^{t}Bu] [$AlCl_4$]

Compound	[*o*-$C_6H_4CH_2Se(Me)CH_2$] [$AlCl_4$]	[$AlCl_2$([14]aneS_4)] [$AlCl_4$]	[^{t}BuTe$(CH_2)_3$Te(^{t}Bu)Te $(CH_2)_3$Te^{t}Bu] [$AlCl_4$]
Formula	$C_9H_{11}AlCl_4Se$	$C_{10}H_{20}Al_2Cl_6S_4$	$C_{18}H_{39}AlCl_4Te_4$
M	366.92	535.19	934.67
crystal system	monoclinic	monoclinic	triclinic
Space group	$P2_1/c$ (no. 14)	$P2_1/n$ (no. 14)	P-1 (no. 2)
a [Å]	7.4218(19)	21.849(2)	10.016(1)
b [Å]	14.027(3)	11.869(1)	12.524(2)
c [Å]	13.481(3)	26.054(2)	14.049(2)
α [deg]	90	90	113.209(8)
([deg]	94.415(6)	100.82	90.417(6)
γ [deg]	90	90	105.465(7)
U [$Å^3$]	1399.2(6)	6636.3(9)	1548.3(3)
Z	4	4	2
μ(Mo Kα) [mm^{-1}]	3.477	1.226	4.109
total no. reflns	11574	21108	11817
unique reflns	5425	11544	5448
R_{int}	0.031	0.241	0.149
no. of params, restraints	137, 0	644, 765	253, 144
R_1[b] [$I_o > 2\sigma(I_o)$]	0.058	0.122	0.083
R_1 [all data]	0.078	0.305	0.188
wR_2[b] [$I_o > 2\sigma(I_o)$]	0.119	0.236	0.126
wR_2 [all data]	0.132	0.335	0.162