Crystal structure of bis[potassium 18-crown-6-1,2-bis(dimethylfluorostannyl) benzene fluoride] pentahydrate, $2\{[K{\cdot}18\text{-Crown-6}]^+[O\text{-}C_6H_4(SnMe_2F)_2{\cdot}F]^-\} \cdot 5H_2O$

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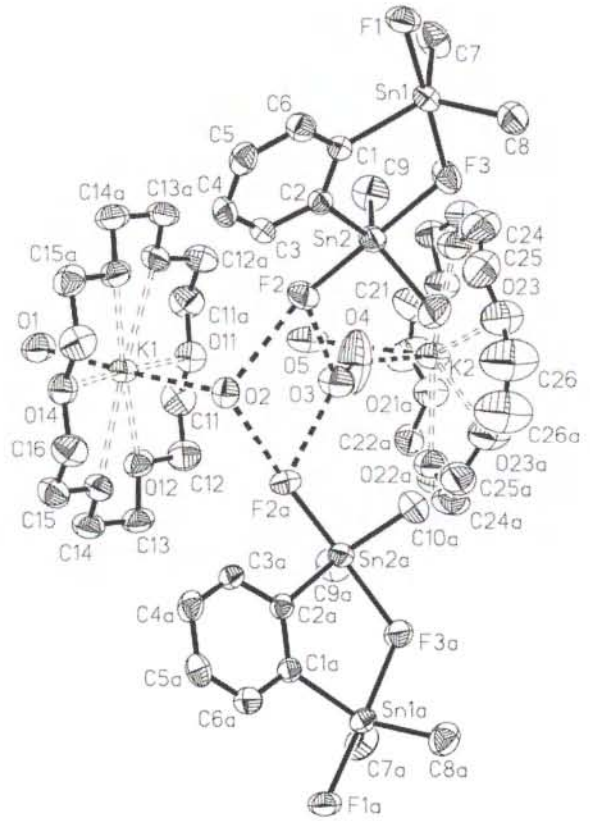

Abstract

$C_{44}H_{90}F_6K_2O_{17}Sn_4$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 17.007(1)$ Å, $b = 21.319(1)$ Å, $c = 18.343(1)$ Å, $V = 6650.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.030$, $wR_{ref}(F^2) = 0.074$, $T = 291$ K.

Source of material

Slow evaporation at room temperature and on contact with air moisture of a dichloromethane solution of $[o\text{-}C_6H_4(SnMe_2F)_2{\cdot}F]^-$ $[K{\cdot}18\text{-crown-6}]^+$, which was prepared according to [1], provided single crystals of the title compound.

Discussion

Potassium 18-crown-6-1,2-bis(dimethylfluorostannyl)benzene fluoride, $[o\text{-}C_6H_4(SnMe_2F)_2{\cdot}F]^-$ $[K{\cdot}18\text{-crown-6}]^+$, crystallizes in contact with air moisture together with 2.5 molar equivalents of H_2O which connect the anions with the cations as well as the anions among each other by a series of hydrogen bonding contacts. In the unit cell there are two different potassium atoms. The potassium atom K1 is displaced by 0.150(3) Å in direction of O2 out of the plane of the oxygen atoms of the crown ether building the equatorial plane of a hexagonal bipyramid. The oxygen atoms of two water molecules (O1, O2) occupy the axial positions. Because of the coordination of two H_2O molecules at the same side the potassium atom K2 is displaced by 0.654(4) Å out of the strongly distorted plane of the oxygen atoms of the crown ether. A similar coordination mode for potassium has been reported for $[Ph_2SiF_3]^-$ $[K{\cdot}18\text{-crown-6}]^+$ [2]. The crystal lattice of the title compound comprises discrete $[o\text{-}C_6H_4(SnMe_2F)_2{\cdot}F]^-$ anions and $[K{\cdot}18\text{-crown-6}]^+$ cations. The anion features a single fluoride bridge between the two trigonal bipyramidal coordinated tin atoms with the axial positions being occupied by the fluorine atoms. The carbon atoms C1, C7, C8 (for Sn1) and C2, C9, C10 (for Sn2) define the equatorial planes. The tin atoms are displaced by 0.079(4) Å (Sn1) and 0.107(3) Å (Sn2) in direction of the terminal fluoride atoms F1 and F2, respectively. The Sn1–F3–Sn2 bridge is asymmetric with d(Sn1—F3) = 2.168(3) Å and d(Sn2—F3) = 2.191(3) Å. The terminal distances d(Sn1—F1) = 2.073(3) Å and d(Sn2—F2) = 2.063(3) Å are shorter and in spite of hydrogen bonding contacts of F2 very similar. The F1, F2, Sn1 and Sn2 atoms lie within a plane from which the bridging F3 is displaced by 0.369(4) Å.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.30 × 0.30 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	16.82 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, 720 frames, $\Delta\omega = 0.5°$, 2 × 2s per frame
$2\theta_{max}$:	43.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	61110, 4165
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2787
$N(param)_{refined}$:	349
Programs:	SHELXS-97 [5], SHELXTL [6], SHELXL-97 [7]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	8*d*	–0.0078	0.3931	0.6451	0.065(8)
H(4)	8*d*	–0.1337	0.4285	0.6581	0.065(8)
H(5)	8*d*	–0.1571	0.5230	0.7179	0.065(8)
H(6)	8*d*	–0.0534	0.5791	0.7648	0.065(8)
H(7A)	8*d*	0.1765	0.6612	0.6988	0.162(5)
H(7B)	8*d*	0.2273	0.6009	0.6854	0.162(5)
H(7C)	8*d*	0.2448	0.6441	0.7527	0.162(5)
H(8A)	8*d*	0.0973	0.5590	0.9238	0.162(5)
H(8B)	8*d*	0.1677	0.6061	0.9163	0.162(5)
H(8C)	8*d*	0.1823	0.5338	0.9080	0.162(5)
H(9A)	8*d*	0.2126	0.4782	0.5769	0.162(5)
H(9B)	8*d*	0.2695	0.4217	0.5918	0.162(5)
H(9C)	8*d*	0.2776	0.4836	0.6373	0.162(5)
H(10A)	8*d*	0.1571	0.3272	0.7837	0.162(5)
H(10B)	8*d*	0.2184	0.3761	0.8129	0.162(5)
H(10C)	8*d*	0.2420	0.3296	0.7505	0.162(5)
H(11A)	8*d*	0.2185	0.1940	0.3724	0.162(5)
H(11B)	8*d*	0.1373	0.1935	0.3312	0.162(5)
H(12A)	8*d*	0.1576	0.1006	0.4050	0.162(5)
H(12B)	8*d*	0.1535	0.1439	0.4744	0.162(5)
H(13A)	8*d*	0.0408	0.0824	0.5101	0.162(5)
H(13B)	8*d*	0.0420	0.0445	0.4365	0.162(5)
H(14A)	8*d*	–0.0810	0.0878	0.4075	0.162(5)
H(14B)	8*d*	–0.0843	0.0453	0.4776	0.162(5)
H(15A)	8*d*	–0.1923	0.1015	0.5297	0.162(5)
H(15B)	8*d*	–0.1972	0.1430	0.4591	0.162(5)
H(16A)	8*d*	–0.2499	0.1940	0.5633	0.162(5)
H(16B)	8*d*	–0.1649	0.1942	0.5973	0.162(5)
H(21A)	8*d*	0.6021	0.2929	0.3425	0.162(5)
H(21B)	8*d*	0.6908	0.2929	0.3625	0.162(5)
H(22A)	8*d*	0.5543	0.3765	0.3968	0.162(5)
H(22B)	8*d*	0.6418	0.3989	0.4033	0.162(5)
H(23A)	8*d*	0.6275	0.4116	0.5271	0.162(5)
H(23B)	8*d*	0.5643	0.4518	0.4858	0.162(5)
H(24A)	8*d*	0.4685	0.4471	0.5807	0.162(5)
H(24B)	8*d*	0.5442	0.4249	0.6222	0.162(5)
H(25A)	8*d*	0.4385	0.3931	0.6845	0.162(5)
H(25B)	8*d*	0.4023	0.3620	0.6149	0.162(5)
H(26A)	8*d*	0.4638	0.2917	0.7554	0.162(5)
H(26B)	8*d*	0.39	0.2917	0.7055	0.162(5)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn(1)	8*d*	0.12458(2)	0.57427(2)	0.78575(2)	0.0593(3)	0.0514(2)	0.0634(3)	–0.0103(2)	–0.0009(2)	–0.0054(2)
Sn(2)	8*d*	0.16138(2)	0.41983(2)	0.69194(2)	0.0589(3)	0.0553(2)	0.0832(3)	0.0075(2)	–0.0080(2)	–0.0100(2)
K(1)	4*c*	–0.0201(1)	1/4	0.45389(9)	0.073(1)	0.0503(9)	0.066(1)	0	0.0034(9)	0
K(2)	4*c*	0.5094(1)	1/4	0.5275(1)	0.114(2)	0.072(1)	0.078(1)	0	0.028(1)	0
F(1)	8*d*	0.0516(2)	0.6513(1)	0.7988(2)	0.100(3)	0.049(2)	0.088(2)	0.002(2)	0.001(2)	–0.009(2)
F(2)	8*d*	0.1222(2)	0.3511(1)	0.6216(2)	0.101(3)	0.063(2)	0.101(3)	0.007(2)	–0.013(2)	–0.026(2)
F(3)	8*d*	0.1988(2)	0.4930(1)	0.7683(2)	0.054(2)	0.083(2)	0.130(3)	0.008(2)	–0.024(2)	–0.034(2)
O(1)	4*c*	–0.0288(5)	1/4	0.3026(3)	0.194(8)	0.079(4)	0.073(4)	0	–0.043(4)	0
O(2)	4*c*	0.0186(3)	1/4	0.6001(3)	0.087(4)	0.080(3)	0.073(4)	0	–0.012(3)	0
O(3)	4*c*	0.2228(4)	1/4	0.6034(4)	0.092(5)	0.089(4)	0.133(5)	0	0.012(4)	0
O(4)	4*c*	0.3521(5)	1/4	0.5204(4)	0.125(7)	0.44(2)	0.088(6)	0	0.006(5)	0
O(5)	4*c*	0.4085(4)	1/4	0.3825(3)	0.164(6)	0.071(3)	0.095(5)	0	0.059(4)	0
O(11)	4*c*	0.1387(3)	1/4	0.4173(3)	0.079(5)	0.093(4)	0.077(4)	0	0.015(3)	0
O(12)	8*d*	0.0528(3)	0.1360(2)	0.4229(2)	0.090(4)	0.065(2)	0.082(3)	0.019(2)	–0.006(2)	–0.004(2)
O(13)	8*d*	–0.0905(3)	0.1363(2)	0.4994(2)	0.084(3)	0.060(2)	0.077(3)	–0.009(2)	–0.005(2)	–0.003(2)
O(14)	4*c*	–0.1754(3)	1/4	0.5112(3)	0.068(4)	0.082(4)	0.063(4)	0	0.000(3)	0
O(21)	8*d*	0.6235(3)	0.3160(2)	0.4397(3)	0.132(4)	0.105(4)	0.102(4)	–0.006(3)	0.023(3)	0.006(3)
O(22)	8*d*	0.5242(3)	0.3801(2)	0.5329(3)	0.131(5)	0.081(3)	0.116(4)	–0.002(3)	–0.002(4)	0.000(3)
O(23)	8*d*	0.4837(4)	0.3165(3)	0.6582(3)	0.211(7)	0.120(4)	0.096(4)	0.014(5)	0.036(4)	–0.005(3)
C(1)	8*d*	0.0334(3)	0.5198(2)	0.7363(3)	0.051(4)	0.047(3)	0.054(4)	–0.004(3)	–0.004(3)	0.003(2)
C(2)	8*d*	0.0480(3)	0.4617(2)	0.7009(3)	0.051(4)	0.052(3)	0.051(3)	–0.003(3)	–0.002(3)	0.002(3)
C(3)	8*d*	–0.0166(3)	0.4301(2)	0.6707(3)	0.064(4)	0.048(3)	0.064(4)	–0.007(3)	–0.010(3)	–0.003(3)
C(4)	8*d*	–0.0921(4)	0.4515(3)	0.6773(3)	0.052(4)	0.073(4)	0.079(4)	–0.018(3)	–0.014(3)	0.006(3)
C(5)	8*d*	–0.1061(3)	0.5079(3)	0.7131(3)	0.042(4)	0.084(4)	0.075(4)	0.005(3)	–0.002(3)	0.012(3)
C(6)	8*d*	–0.0437(3)	0.5412(2)	0.7415(3)	0.058(4)	0.060(3)	0.067(4)	–0.002(3)	0.004(3)	–0.002(3)
C(7)	8*d*	0.2042(4)	0.6274(3)	0.7219(3)	0.091(5)	0.106(5)	0.086(5)	–0.043(4)	0.012(4)	–0.002(4)
C(8)	8*d*	0.1459(4)	0.5674(2)	0.8989(3)	0.110(6)	0.081(4)	0.083(5)	–0.023(4)	–0.020(4)	0.007(3)
C(9)	8*d*	0.2412(4)	0.4557(3)	0.6138(3)	0.080(5)	0.113(5)	0.117(6)	0.000(4)	0.024(4)	–0.002(4)
C(10)	8*d*	0.2000(4)	0.3543(3)	0.7705(4)	0.111(6)	0.094(4)	0.122(6)	0.027(4)	–0.033(5)	–0.002(4)
C(11)	8*d*	0.1619(4)	0.1948(3)	0.3788(4)	0.088(5)	0.112(5)	0.111(6)	0.017(4)	0.023(4)	–0.021(5)
C(12)	8*d*	0.1354(5)	0.1390(3)	0.4245(4)	0.105(7)	0.079(4)	0.118(6)	0.028(4)	–0.001(5)	–0.018(4)
C(13)	8*d*	0.0229(4)	0.0824(3)	0.4598(3)	0.122(7)	0.050(3)	0.088(5)	0.013(4)	–0.009(4)	–0.002(3)
C(14)	8*d*	–0.0632(5)	0.0839(2)	0.4576(3)	0.127(7)	0.056(4)	0.085(5)	–0.008(4)	–0.011(4)	–0.006(3)
C(15)	8*d*	–0.1730(4)	0.1395(3)	0.5068(3)	0.083(6)	0.081(4)	0.091(5)	–0.022(4)	–0.003(4)	0.005(4)
C(16)	8*d*	–0.1942(4)	0.1948(3)	0.5519(3)	0.091(5)	0.093(5)	0.085(5)	–0.014(4)	0.019(4)	0.006(4)
C(21)	8*d*	0.6393(6)	0.2801(3)	0.3797(4)	0.26(1)	0.143(7)	0.102(6)	–0.066(6)	0.069(7)	–0.021(5)
C(22)	8*d*	0.6001(5)	0.3764(4)	0.4282(5)	0.139(8)	0.097(6)	0.125(8)	–0.029(5)	–0.027(6)	0.037(5)
C(23)	8*d*	0.5809(6)	0.4093(3)	0.4968(6)	0.157(9)	0.088(6)	0.151(9)	–0.012(6)	–0.020(7)	0.011(6)
C(24)	8*d*	0.4989(6)	0.4106(4)	0.5946(5)	0.22(1)	0.099(6)	0.112(8)	0.055(7)	0.000(7)	–0.026(6)
C(25)	8*d*	0.4513(6)	0.3705(5)	0.6401(5)	0.19(1)	0.133(8)	0.100(7)	0.056(8)	–0.003(7)	–0.028(6)
C(26)	8*d*	0.4448(7)	0.2792(3)	0.7077(5)	0.29(1)	0.18(1)	0.156(9)	0.004(8)	0.123(9)	0.010(7)

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