Crystal structure of tris[([2.2.2]crypt)potassium] nonastannide ethylenediamine hemisolvate, $[K(C_{18}H_{36}N_2O_6)]_3Sn_9 \cdot \frac{1}{2}C_2H_8N_2$

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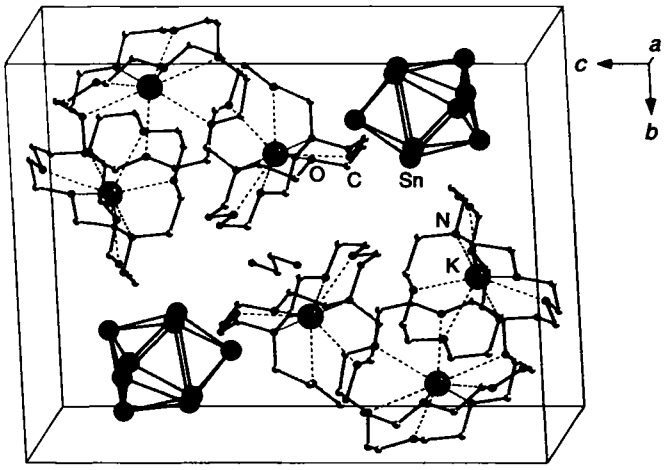

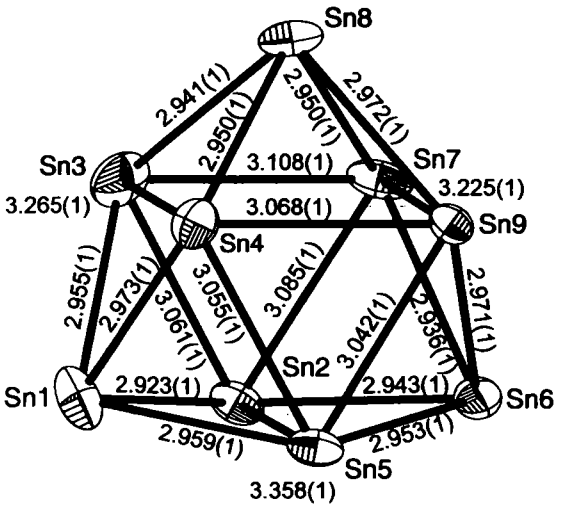

Abstract

$C_{55}H_{112}K_3N_7O_{18}Sn_9$, triclinic, $P\bar{1}$ (no. 2), a = 14.8163(1) Å, b = 16.1125(1) Å, c = 20.1823(1) Å, α = 92.7499(3)°, β = 96.5942(3)°, γ = 111.6478(2)°, V = 4427.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.106, T = 120 K.

Source of material

All experiments were carried out under argon atmosphere using a glove-box or Schlenk technique. The alloy K_4Sn_9 (200 mg, 0.164 mmol) and [2.2.2]crypt (246 mg, 0.65 mmol) were dissolved in ethylenediamine (en, ca. 2 ml) in 25 ml an Schlenk tube and stirred for 5 min to yield a dark red solution. Then Hg (200 mg, 1.0 mmol) was added dropwise to the Schlenk tube and stirred for another 2 hours at room temperature to yield a reddish-brown solution. Afterwards the reaction mixture was treated for 20 min in an ultrasonic bath, and then filtered. The filtrate was layered with toluene. After 48 hours, dark plate-like of crystals of $[K([2.2.2]crypt)]_3Sn_9 \cdot \frac{1}{2}$ en were obtained.

Discussion

Interest in synthesis, isolation and characterization of homoatomic-element polyhedra of the type E_n^{x-} (E = Group 14 element) from solution can be dated back to the 19th century [1]. The breakthrough in structural characterization of E_n^{x-} ions was the introduction of the sequestering agent [2.2.2]crypt by Corbett [2]. The extraction of an alloy K_4Sn_9 with ethylenediamine and addition of [2.2.2]crypt and toluene leads to a paramagnetic Sn_9^{3-} cluster $[K([2.2.2]crypt)]_3Sn_9 \cdot \frac{1}{2}$ en [3]. Later the isolation of Ge_9^{3-} and Pb_9^{3-} ions were also realized using a similar method [4]. Recently we found that the reaction of K_4Sn_9 with elementary Hg in the presence of [2.2.2]crypt gives the compound $[K([2.2.2]crypt)]_3Sn_9 \cdot \frac{1}{2}$ en with explicit new unit cell, which also features a Sn_9^{3-} ion. Comparing with the reported compound $[K([2.2.2]crypt)]_3Sn_9 \cdot \frac{1}{2}$ en (a = 15.002(1) Å, b = 21.794(1) Å, c = 14.060(1) Å, α = 99.77(1)°, β = 101.60(1)°, γ = 89.69(1)°, V = 4435.9(7) Å^3, Z = 2), the unit cell volume of the title compound is smaller. The quality of the crystal is much better than the reported one [$R_{gt}(F)$ = 0.085, $wR_{gt}(F^2)$ = 0.125 with $I > 3\sigma(I)$]. In addition, the en molecule in the title compound is also well ordered, and is thus quite different from the sesquisolvate [3].

In crystals of the title compound, three $[K([2.2.2]crypt)]^+$ cations are present per nonastannide cluster in addition of an en molecule, located around an crystallographic inversion center. This indicates clearly a charge allocation of –3 for the Sn_9 polyanion. Though there are three $[K([2.2.2]crypt)]^+$ units surround the Sn_9 cluster, all of them are quite far away from the polyanion and do not show any coordination to the cluster anion. The shortest Sn—K distance is 7.165 Å. The Sn_9^{3-} anion varies little from an ideal tricapped trigonal prism with D_{3h} symmetry (*closo* type) (figure, with thermal ellipsoid at 90 % probability level). The average Sn—Sn bond length is 2.998 Å, ranging from 2.923 Å to 3.108 Å, which is slightly longer than the reported one (2.982 Å). The height of the trigonal prisms (Sn3—Sn4, Sn2—Sn5 and Sn7—Sn9) are found to be elongated comparing to all other intramolecular Sn—Sn distances. For nine-atom clusters, the ratio of height (h) to edge (e) of the trigonal prism provides the best correlation of configuration with electron count [3,4]. The Sn_9^{3-} cluster ion has average values of 3.283 Å, 3.070 Å for h and e, respectively. The ratio 1.07 of h to e of Sn_9^{3-} cluster ion in the title compound is a little bit smaller than the reported one (1.08) for the Sn_9^{3-} ion in $[K([2.2.2]crypt)]_3Sn_9 \cdot \frac{1}{2}$ en [3].

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Table 1. Data collection and handling.

Crystal:	dark brown plate, size 0.20 × 0.40 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	26.91 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	50.82°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	102624, 16285
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 13197
$N(param)_{refined}$:	829
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.9078	0.6917	0.0640	0.048
H(1B)	2*i*	0.9496	0.7271	−0.0033	0.048
H(2A)	2*i*	1.0081	0.8765	0.0466	0.051
H(2B)	2*i*	0.8916	0.8290	0.0453	0.051
H(3A)	2*i*	0.8923	0.9242	0.1381	0.051
H(3B)	2*i*	1.0081	0.9776	0.1399	0.051
H(4A)	2*i*	0.9563	0.9905	0.2448	0.047
H(4B)	2*i*	0.9234	0.8842	0.2465	0.047
H(5A)	2*i*	1.0408	0.9349	0.3518	0.043
H(5B)	2*i*	1.0825	1.0376	0.3353	0.043
H(6A)	2*i*	1.2372	1.0291	0.3287	0.045
H(6B)	2*i*	1.2062	1.0141	0.4018	0.045
H(7A)	2*i*	1.1130	0.6392	0.0805	0.044
H(7B)	2*i*	1.0151	0.6093	0.0276	0.044
H(8A)	2*i*	0.9218	0.5941	0.1180	0.048
H(8B)	2*i*	0.9798	0.5274	0.1191	0.048
H(9A)	2*i*	0.9496	0.5271	0.2318	0.044
H(9B)	2*i*	0.9226	0.6146	0.2379	0.044
H(10A)	2*i*	0.9987	0.5915	0.3426	0.040
H(10B)	2*i*	1.0955	0.6034	0.3094	0.040
H(11A)	2*i*	1.1964	0.7377	0.3810	0.046
H(11B)	2*i*	1.1023	0.7106	0.4197	0.046
H(12A)	2*i*	1.0906	0.8512	0.4025	0.046
H(12B)	2*i*	1.1930	0.8638	0.4459	0.046
H(13A)	2*i*	1.1257	0.8423	0.0238	0.045
H(13B)	2*i*	1.1100	0.7490	−0.0163	0.045
H(14A)	2*i*	1.2364	0.7356	0.0591	0.044
H(14B)	2*i*	1.2754	0.8247	0.0213	0.044
H(15A)	2*i*	1.4038	0.8792	0.1086	0.057
H(15B)	2*i*	1.3619	0.8017	0.1568	0.057
H(16A)	2*i*	1.4657	0.9404	0.2187	0.057
H(16B)	2*i*	1.3902	0.9847	0.1899	0.057
H(17A)	2*i*	1.3642	1.0141	0.3013	0.049
H(17B)	2*i*	1.4417	0.9705	0.3271	0.049
H(18A)	2*i*	1.3194	0.8620	0.3779	0.046
H(18B)	2*i*	1.3393	0.9613	0.4090	0.046
H(19A)	2*i*	1.0585	0.2872	0.5423	0.048
H(19B)	2*i*	1.0790	0.2007	0.5179	0.048
H(20A)	2*i*	1.0706	0.2902	0.4289	0.047
H(20B)	2*i*	1.1647	0.3713	0.4682	0.047
H(21A)	2*i*	1.2298	0.3502	0.3625	0.042
H(21B)	2*i*	1.1362	0.2595	0.3388	0.042
H(22A)	2*i*	1.2409	0.1785	0.3443	0.044
H(22B)	2*i*	1.2659	0.2503	0.2902	0.044
H(23A)	2*i*	1.4327	0.2603	0.2989	0.046
H(23B)	2*i*	1.4019	0.1819	0.3478	0.046
H(24A)	2*i*	1.5752	0.2690	0.3637	0.043
H(24B)	2*i*	1.5527	0.3543	0.3885	0.043
H(25A)	2*i*	1.1704	0.3872	0.6351	0.043
H(25B)	2*i*	1.2154	0.4226	0.5689	0.043
H(26A)	2*i*	1.3297	0.4899	0.6654	0.045
H(26B)	2*i*	1.3257	0.3930	0.6841	0.045
H(27A)	2*i*	1.4983	0.4349	0.6730	0.038
H(27B)	2*i*	1.4963	0.5321	0.6617	0.038
H(28A)	2*i*	1.5475	0.5229	0.5554	0.038
H(28B)	2*i*	1.6273	0.5195	0.6150	0.038

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(29A)	2*i*	1.6911	0.4505	0.5430	0.040
H(29B)	2*i*	1.6117	0.4419	0.4792	0.040
H(30A)	2*i*	1.6796	0.3300	0.4709	0.039
H(30B)	2*i*	1.6365	0.2926	0.5372	0.039
H(31A)	2*i*	1.1117	0.2306	0.6467	0.049
H(31B)	2*i*	1.2269	0.2596	0.6699	0.049
H(32A)	2*i*	1.1503	0.1016	0.6485	0.054
H(32B)	2*i*	1.1119	0.1113	0.5728	0.054
H(33A)	2*i*	1.2066	0.0365	0.5213	0.044
H(33B)	2*i*	1.2133	0.0091	0.5966	0.044
H(34A)	2*i*	1.3868	0.0731	0.6066	0.046
H(34B)	2*i*	1.3412	−0.0080	0.5482	0.046
H(35A)	2*i*	1.4973	0.0660	0.5158	0.038
H(35B)	2*i*	1.5301	0.1627	0.5568	0.038
H(36A)	2*i*	1.5945	0.1687	0.4532	0.038
H(36B)	2*i*	1.4847	0.1333	0.4146	0.038
H(37A)	2*i*	1.2232	0.2509	0.8520	0.079
H(37B)	2*i*	1.2832	0.2766	0.7901	0.079
H(38A)	2*i*	1.2379	0.3928	0.8262	0.075
H(38B)	2*i*	1.2961	0.3992	0.8995	0.075
H(39A)	2*i*	1.4142	0.5480	0.8983	0.059
H(39B)	2*i*	1.3401	0.5475	0.8336	0.059
H(40A)	2*i*	1.4687	0.5807	0.7695	0.052
H(40B)	2*i*	1.4905	0.6578	0.8293	0.052
H(41A)	2*i*	1.6567	0.7029	0.8316	0.094
H(41B)	2*i*	1.6413	0.6293	0.7705	0.094
H(42A)	2*i*	1.8019	0.6747	0.8355	0.087
H(42B)	2*i*	1.7513	0.6365	0.8994	0.087
H(43A)	2*i*	1.2945	0.1396	0.8530	0.076
H(43B)	2*i*	1.4078	0.1670	0.8814	0.076
H(44A)	2*i*	1.3771	0.1147	0.7675	0.091
H(44B)	2*i*	1.3366	0.1916	0.7495	0.091
H(45A)	2*i*	1.4670	0.2634	0.6809	0.103
H(45B)	2*i*	1.4928	0.1762	0.6911	0.103
H(46A)	2*i*	1.6524	0.2670	0.7378	0.102
H(46B)	2*i*	1.6276	0.2909	0.6634	0.102
H(47A)	2*i*	1.7414	0.4367	0.6822	0.083
H(47B)	2*i*	1.7763	0.4137	0.7548	0.083
H(48A)	2*i*	1.8186	0.5698	0.7511	0.075
H(48B)	2*i*	1.7051	0.5534	0.7327	0.075
H(49A)	2*i*	1.3584	0.3331	0.9626	0.079
H(49B)	2*i*	1.3049	0.2264	0.9574	0.079
H(50A)	2*i*	1.4426	0.2728	1.0366	0.085
H(50B)	2*i*	1.4557	0.2122	0.9760	0.085
H(51A)	2*i*	1.6273	0.2762	0.9846	0.054
H(51B)	2*i*	1.6143	0.3156	1.0558	0.054
H(52A)	2*i*	1.7021	0.4618	1.0300	0.060
H(52B)	2*i*	1.7674	0.4016	1.0313	0.060
H(53A)	2*i*	1.8689	0.5029	0.9642	0.056
H(53B)	2*i*	1.7995	0.5594	0.9616	0.056
H(54A)	2*i*	1.8863	0.5791	0.8684	0.059
H(54B)	2*i*	1.8358	0.4736	0.8463	0.059
H(55A)	2*i*	1.0725	0.5077	0.5558	0.041
H(55B)	2*i*	1.0194	0.4196	0.5046	0.041
H(71)	2*i*	1.1448	0.5802	0.4707	0.064
H(72)	2*i*	1.0953	0.4982	0.4231	0.064

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}
C(1)	2*i*	0.9609(4)	0.7391(4)	0.0462(3)	0.030(3)	0.042(4)	0.044(4)	0.011(3)	0.000(3)	−0.001(3)
C(2)	2*i*	0.9559(5)	0.8286(4)	0.0647(3)	0.034(3)	0.046(4)	0.048(4)	0.015(3)	0.007(3)	0.010(3)
C(3)	2*i*	0.9580(5)	0.9264(4)	0.1568(3)	0.033(3)	0.031(3)	0.065(4)	0.013(3)	0.009(3)	0.004(3)
C(4)	2*i*	0.9705(4)	0.9377(4)	0.2299(3)	0.026(3)	0.038(3)	0.061(4)	0.019(3)	0.014(3)	0.005(3)
C(5)	2*i*	1.0889(4)	0.9789(4)	0.3279(3)	0.039(3)	0.028(3)	0.047(4)	0.016(3)	0.020(3)	0.002(3)
C(6)	2*i*	1.1906(4)	0.9872(4)	0.3547(3)	0.041(4)	0.028(3)	0.042(3)	0.012(3)	0.011(3)	−0.005(3)
C(7)	2*i*	1.0465(5)	0.6409(4)	0.0725(3)	0.035(3)	0.029(3)	0.045(4)	0.011(3)	0.004(3)	−0.005(3)
C(8)	2*i*	0.9878(4)	0.5912(4)	0.1243(3)	0.033(3)	0.024(3)	0.053(4)	0.003(3)	−0.002(3)	−0.005(3)
C(9)	2*i*	0.9775(4)	0.5934(4)	0.2400(3)	0.031(3)	0.028(3)	0.052(4)	0.009(3)	0.012(3)	0.011(3)
C(10)	2*i*	1.0383(4)	0.6214(4)	0.3081(3)	0.036(3)	0.023(3)	0.045(3)	0.014(3)	0.012(3)	0.009(2)
C(11)	2*i*	1.1340(5)	0.7460(4)	0.3842(3)	0.038(4)	0.036(3)	0.040(3)	0.013(3)	0.008(3)	0.007(3)
C(12)	2*i*	1.1538(5)	0.8439(4)	0.4009(3)	0.041(4)	0.039(3)	0.035(3)	0.014(3)	0.012(3)	−0.003(3)
C(13)	2*i*	1.1288(4)	0.7825(4)	0.0288(3)	0.040(4)	0.044(4)	0.031(3)	0.017(3)	0.011(3)	0.002(3)
C(14)	2*i*	1.2320(4)	0.7949(4)	0.0540(3)	0.037(3)	0.039(3)	0.039(3)	0.016(3)	0.017(3)	0.002(3)
C(15)	2*i*	1.3604(5)	0.8595(5)	0.1433(3)	0.031(3)	0.083(5)	0.035(3)	0.026(3)	0.014(3)	0.012(3)
C(16)	2*i*	1.3958(4)	0.9280(5)	0.2026(3)	0.023(3)	0.072(5)	0.046(4)	0.010(3)	0.015(3)	0.024(3)
C(17)	2*i*	1.3714(4)	0.9574(4)	0.3122(3)	0.031(3)	0.040(4)	0.049(4)	0.009(3)	0.009(3)	0.001(3)
C(18)	2*i*	1.3119(4)	0.9189(4)	0.3680(3)	0.032(3)	0.035(3)	0.043(3)	0.010(3)	0.000(3)	−0.006(3)
C(19)	2*i*	1.1082(4)	0.2670(4)	0.5259(3)	0.025(3)	0.048(4)	0.053(4)	0.017(3)	0.015(3)	0.011(3)
C(20)	2*i*	1.1317(4)	0.3051(5)	0.4607(3)	0.030(3)	0.053(4)	0.043(3)	0.023(3)	0.004(3)	0.008(3)
C(21)	2*i*	1.2023(4)	0.2846(4)	0.3656(3)	0.023(3)	0.045(4)	0.035(3)	0.012(3)	−0.002(2)	0.013(3)
C(22)	2*i*	1.2664(4)	0.2434(4)	0.3387(3)	0.035(3)	0.039(3)	0.026(3)	0.004(3)	−0.002(2)	0.005(2)
C(23)	2*i*	1.4286(5)	0.2478(4)	0.3463(3)	0.047(4)	0.041(4)	0.034(3)	0.022(3)	0.014(3)	0.006(3)
C(24)	2*i*	1.5295(4)	0.2881(4)	0.3867(3)	0.036(3)	0.041(3)	0.042(3)	0.021(3)	0.026(3)	0.015(3)
C(25)	2*i*	1.2198(4)	0.3860(4)	0.6063(3)	0.035(3)	0.042(3)	0.044(3)	0.026(3)	0.016(3)	0.005(3)
C(26)	2*i*	1.3196(5)	0.4287(4)	0.6465(3)	0.047(4)	0.039(3)	0.037(3)	0.025(3)	0.015(3)	−0.003(3)
C(27)	2*i*	1.4876(4)	0.4735(4)	0.6387(3)	0.036(3)	0.031(3)	0.028(3)	0.018(3)	−0.007(2)	−0.008(2)
C(28)	2*i*	1.5602(4)	0.4867(4)	0.5910(3)	0.028(3)	0.022(3)	0.041(3)	0.007(2)	−0.003(2)	−0.006(2)
C(29)	2*i*	1.6256(4)	0.4123(4)	0.5188(3)	0.019(3)	0.027(3)	0.050(4)	0.004(2)	0.009(3)	0.004(3)
C(30)	2*i*	1.6247(4)	0.3219(4)	0.4969(3)	0.023(3)	0.029(3)	0.052(4)	0.014(2)	0.013(3)	0.009(3)
C(31)	2*i*	1.1739(5)	0.2337(4)	0.6314(3)	0.041(4)	0.055(4)	0.038(3)	0.025(3)	0.025(3)	0.016(3)
C(32)	2*i*	1.1658(5)	0.1387(4)	0.6106(3)	0.039(4)	0.050(4)	0.051(4)	0.017(3)	0.027(3)	0.022(3)
C(33)	2*i*	1.2465(5)	0.0535(4)	0.5662(3)	0.045(4)	0.023(3)	0.040(3)	0.006(3)	0.018(3)	0.010(2)
C(34)	2*i*	1.3460(5)	0.0535(4)	0.5621(3)	0.059(4)	0.025(3)	0.041(3)	0.018(3)	0.026(3)	0.018(3)
C(35)	2*i*	1.4909(4)	0.1250(4)	0.5156(3)	0.041(3)	0.027(3)	0.036(3)	0.022(3)	0.006(3)	0.004(2)
C(36)	2*i*	1.5281(4)	0.1688(4)	0.4554(3)	0.039(3)	0.028(3)	0.037(3)	0.019(3)	0.014(3)	0.001(2)
C(37)	2*i*	1.2859(5)	0.2907(5)	0.8388(5)	0.032(4)	0.054(5)	0.111(7)	0.011(3)	0.017(4)	0.029(5)
C(38)	2*i*	1.2952(5)	0.3848(5)	0.8511(4)	0.038(4)	0.072(5)	0.092(6)	0.030(4)	0.024(4)	0.024(4)
C(39)	2*i*	1.3998(5)	0.5357(5)	0.8489(4)	0.052(4)	0.059(4)	0.053(4)	0.039(4)	0.012(3)	0.007(3)
C(40)	2*i*	1.4820(5)	0.5944(4)	0.8188(3)	0.048(4)	0.041(4)	0.052(4)	0.027(3)	0.015(3)	0.007(3)
C(41)	2*i*	1.6507(5)	0.6405(5)	0.8200(6)	0.041(4)	0.035(4)	0.151(9)	0.008(3)	−0.002(5)	0.027(5)
C(42)	2*i*	1.7443(5)	0.6282(5)	0.8499(5)	0.036(4)	0.032(4)	0.145(8)	0.006(3)	0.003(5)	0.032(4)
C(43)	2*i*	1.3613(6)	0.1837(5)	0.8510(4)	0.045(4)	0.040(4)	0.092(6)	0.001(3)	0.006(4)	0.015(4)
C(44)	2*i*	1.3840(6)	0.1770(5)	0.7807(5)	0.067(6)	0.033(4)	0.112(8)	0.016(4)	−0.028(5)	−0.017(4)
C(45)	2*i*	1.5074(9)	0.2384(6)	0.7098(4)	0.17(1)	0.051(5)	0.037(4)	0.043(6)	0.008(5)	−0.009(4)
C(46)	2*i*	1.6117(9)	0.2924(6)	0.7096(4)	0.19(1)	0.071(6)	0.046(5)	0.090(7)	0.056(6)	0.023(4)
C(47)	2*i*	1.7308(6)	0.4370(7)	0.7297(4)	0.063(5)	0.138(8)	0.048(4)	0.071(6)	0.040(4)	0.047(5)
C(48)	2*i*	1.7514(5)	0.5310(6)	0.7579(4)	0.027(4)	0.096(6)	0.074(5)	0.025(4)	0.019(3)	0.056(5)
C(49)	2*i*	1.3643(6)	0.2760(6)	0.9481(4)	0.046(4)	0.083(6)	0.071(5)	0.018(4)	0.032(4)	0.031(4)
C(50)	2*i*	1.4506(6)	0.2703(6)	0.9886(4)	0.069(6)	0.072(5)	0.071(5)	0.017(4)	0.024(4)	0.044(4)
C(51)	2*i*	1.6210(5)	0.3283(5)	1.0088(3)	0.065(5)	0.062(4)	0.031(3)	0.043(4)	0.018(3)	0.019(3)
C(52)	2*i*	1.7098(5)	0.4097(5)	1.0069(3)	0.065(5)	0.071(5)	0.023(3)	0.040(4)	−0.003(3)	−0.003(3)
C(53)	2*i*	1.8101(5)	0.5083(4)	0.9393(3)	0.034(4)	0.036(4)	0.057(4)	0.006(3)	−0.015(3)	0.000(3)
C(54)	2*i*	1.8259(4)	0.5253(5)	0.8683(4)	0.024(3)	0.046(4)	0.071(5)	0.008(3)	0.001(3)	0.024(3)
C(55)	2*i*	1.0437(4)	0.4860(4)	0.5085(3)	0.026(3)	0.031(3)	0.043(3)	0.013(3)	−0.002(3)	−0.004(3)
N(1)	2*i*	1.0560(3)	0.7344(3)	0.0722(2)	0.029(3)	0.030(3)	0.037(3)	0.010(2)	0.007(2)	0.001(2)
N(2)	2*i*	1.2065(3)	0.9014(3)	0.3524(2)	0.026(2)	0.027(2)	0.036(3)	0.009(2)	0.008(2)	0.003(2)
N(3)	2*i*	1.1942(3)	0.2933(3)	0.5783(2)	0.027(3)	0.036(3)	0.037(3)	0.017(2)	0.011(2)	0.011(2)
N(4)	2*i*	1.5329(3)	0.2621(3)	0.4560(2)	0.026(2)	0.028(2)	0.033(2)	0.017(2)	0.011(2)	0.005(2)
N(5)	2*i*	1.3667(4)	0.2713(4)	0.8751(3)	0.034(3)	0.040(3)	0.076(4)	0.012(2)	0.016(3)	0.018(3)
N(6)	2*i*	1.7430(4)	0.5388(4)	0.8291(3)	0.025(3)	0.043(3)	0.069(4)	0.012(2)	0.007(3)	0.028(3)
N(7)	2*i*	1.1209(4)	0.5192(3)	0.4665(3)	0.027(3)	0.040(3)	0.062(3)	0.014(2)	0.006(2)	0.000(3)
O(1)	2*i*	0.9684(3)	0.8443(3)	0.1360(2)	0.028(2)	0.030(2)	0.058(3)	0.014(2)	0.006(2)	0.002(2)
O(2)	2*i*	1.0694(3)	0.9501(3)	0.2579(2)	0.040(2)	0.040(2)	0.043(2)	0.022(2)	0.018(2)	0.002(2)
O(3)	2*i*	1.0369(3)	0.6304(2)	0.1897(2)	0.034(2)	0.023(2)	0.041(2)	0.002(2)	0.006(2)	0.000(2)
O(4)	2*i*	1.0710(3)	0.7162(2)	0.3216(2)	0.034(2)	0.021(2)	0.042(2)	0.009(2)	0.008(2)	0.005(2)
O(5)	2*i*	1.2633(3)	0.8482(3)	0.1171(2)	0.026(2)	0.044(2)	0.045(2)	0.015(2)	0.009(2)	0.004(2)
O(6)	2*i*	1.3378(3)	0.8949(3)	0.2550(2)	0.026(2)	0.033(2)	0.041(2)	0.006(2)	0.010(2)	0.004(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(7)	2*i*	1.1954(3)	0.2671(3)	0.4336(2)	0.026(2)	0.046(2)	0.029(2)	0.020(2)	0.005(2)	0.012(2)
O(8)	2*i*	1.3653(3)	0.2849(2)	0.3727(2)	0.027(2)	0.030(2)	0.028(2)	0.008(2)	0.006(2)	0.002(2)
O(9)	2*i*	1.3918(3)	0.4330(2)	0.6037(2)	0.031(2)	0.035(2)	0.029(2)	0.020(2)	0.005(2)	0.001(2)
O(10)	2*i*	1.5526(3)	0.4018(2)	0.5619(2)	0.021(2)	0.024(2)	0.036(2)	0.008(2)	0.005(2)	−0.002(2)
O(11)	2*i*	1.2553(3)	0.1405(2)	0.5908(2)	0.031(2)	0.027(2)	0.039(2)	0.009(2)	0.017(2)	0.011(2)
O(12)	2*i*	1.3909(3)	0.1133(2)	0.5142(2)	0.038(2)	0.025(2)	0.032(2)	0.016(2)	0.012(2)	0.011(2)
O(13)	2*i*	1.3831(3)	0.4442(3)	0.8303(2)	0.038(2)	0.041(2)	0.055(3)	0.023(2)	0.017(2)	0.014(2)
O(14)	2*i*	1.5709(3)	0.5814(3)	0.8449(2)	0.042(3)	0.033(2)	0.060(3)	0.019(2)	0.007(2)	0.012(2)
O(15)	2*i*	1.4821(4)	0.2375(3)	0.7757(2)	0.074(4)	0.037(3)	0.043(3)	0.021(2)	−0.005(2)	−0.005(2)
O(16)	2*i*	1.6331(3)	0.3816(3)	0.7346(2)	0.059(3)	0.063(3)	0.036(2)	0.044(3)	0.019(2)	0.015(2)
O(17)	2*i*	1.5374(3)	0.3412(3)	0.9788(2)	0.055(3)	0.055(3)	0.041(2)	0.031(2)	0.023(2)	0.024(2)
O(18)	2*i*	1.7268(3)	0.4279(3)	0.9403(2)	0.041(2)	0.038(2)	0.026(2)	0.016(2)	−0.001(2)	0.003(2)
K(1)	2*i*	1.13029(8)	0.81794(8)	0.21083(6)	0.0236(6)	0.0228(6)	0.0406(7)	0.0078(5)	0.0103(5)	0.0010(5)
K(2)	2*i*	1.36093(8)	0.27715(7)	0.51460(5)	0.0190(5)	0.0249(6)	0.0246(6)	0.0105(5)	0.0063(4)	0.0056(4)
K(3)	2*i*	1.55457(8)	0.40413(8)	0.85249(6)	0.0246(6)	0.0279(6)	0.0263(6)	0.0111(5)	0.0068(5)	0.0064(5)
Sn(1)	2*i*	0.63282(3)	0.20147(3)	0.18173(2)	0.0293(2)	0.0452(3)	0.0441(3)	0.0071(2)	−0.0060(2)	0.0129(2)
Sn(2)	2*i*	0.67588(3)	0.12265(3)	0.30297(2)	0.0217(2)	0.0299(2)	0.0398(2)	0.0072(2)	0.0113(2)	0.0126(2)
Sn(3)	2*i*	0.75751(4)	0.09525(3)	0.17463(2)	0.0496(3)	0.0278(2)	0.0540(3)	0.0052(2)	0.0132(2)	−0.0111(2)
Sn(4)	2*i*	0.83624(3)	0.31036(3)	0.15980(2)	0.0374(2)	0.0324(2)	0.0232(2)	0.0126(2)	0.0055(2)	0.0084(2)
Sn(5)	2*i*	0.75773(3)	0.34390(2)	0.28772(2)	0.0285(2)	0.0277(2)	0.0410(2)	0.0173(2)	0.0127(2)	0.0085(2)
Sn(6)	2*i*	0.83529(3)	0.25967(3)	0.39507(2)	0.0278(2)	0.0465(2)	0.0237(2)	0.0139(2)	0.0092(2)	0.0063(2)
Sn(7)	2*i*	0.88995(3)	0.13537(3)	0.31263(2)	0.0321(2)	0.0335(2)	0.0508(3)	0.0223(2)	0.0204(2)	0.0229(2)
Sn(8)	2*i*	0.96652(3)	0.21216(3)	0.19134(2)	0.0419(2)	0.0367(2)	0.0426(2)	0.0212(2)	0.0257(2)	0.0086(2)
Sn(9)	2*i*	0.96549(3)	0.34741(2)	0.29640(2)	0.0187(2)	0.0245(2)	0.0288(2)	0.0062(2)	0.0052(2)	0.0051(2)

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