Crystal structure of (4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo[8.8.8]-hexacosane-κ^8N_2,O_6) potassium-di-(1,4,7,10,13,16-hexaoxacyclo-octadecane-κ^6O) potassium potassium nonastannide ethylenediamine sesquisolvate, $[K([2.2.2]crypt)][K(18\text{-crown-}6)]_2[KSn_9](en)_{1.5}$, $C_{45}H_{96}K_4N_5O_{18}Sn_9$

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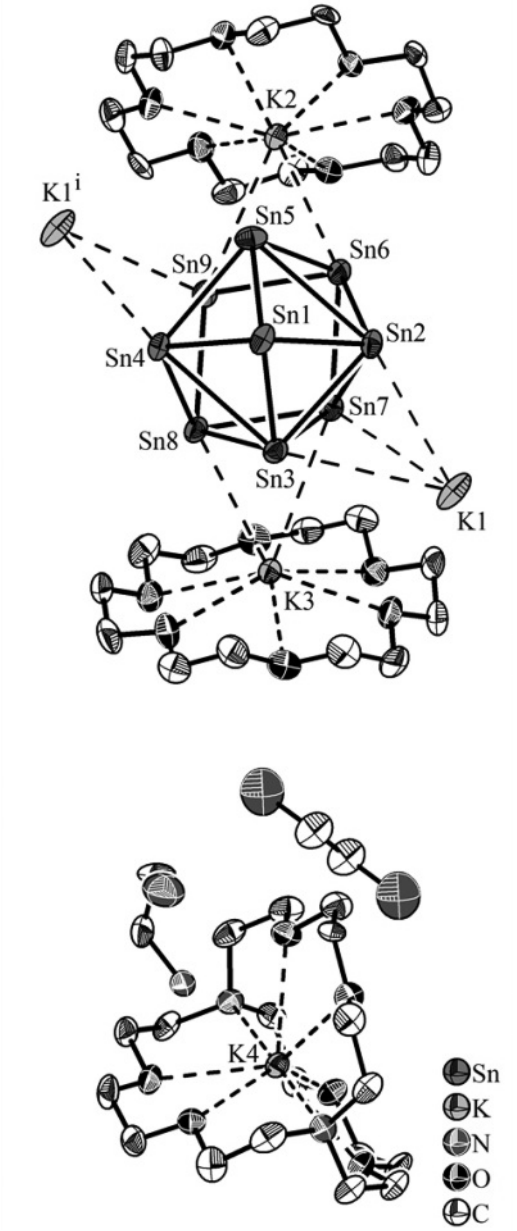

Abstract

$C_{45}H_{96}K_4N_5O_{18}Sn_9$, triclinic, $P\bar{1}$ (no. 2), a = 10.1654(3) Å, b = 18.509(1) Å, c = 20.3683(9) Å, α = 83.232(4)°, β = 81.475(3)°, γ = 82.427(3)°, V = 3737.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.0687, $wR_{ref}(F^2)$ = 0.1527, T = 123 K.

Table 1. Data collection and handling.

Crystal:	brown blocks, size 0.02×0.02×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	32.34 cm^{-1}
Diffractometer, scan mode:	Oxford Xcalibur 3, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	30342, 1357
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 7444
$N(param)_{refined}$:	730
Programs:	Crysalis CCD [7], SHELX [8], DIAMOND [9]

Source of material

50 mg K_4Sn_9 (0.041 mmol) and 6 mg [K(18-crown-6)-(THF)$_2$][Co(anthracene)$_2$]·THF [1] (0.007 mmol) were dissolved in 1.5 mL ethylenediamine. The reaction mixture was stirred at room temperature for 2 h, filtered into a Schlenk tube and layered with a solution of 40 mg [2.2.2]cryptand (0.106 mmol) in 3 mL toluene. After diffusion brown crystals of the title compound besides those of $[K([2.2.2]crypt)]_3[KSn_9]$ [2] were isolated and determined by single crystal X-ray diffraction.

Experimental details

All H atoms of crown ether, cryptand, and ethylenediamine molecules were included to calculated positions and were refined using a riding model with U_{iso} set to 1.2 U_{eq}(C) and 1.5 U_{eq}(N), respectively. The anisotropic displacement parameters of one C and two N atoms of the solvent molecules had to be restrained using the ISOR option of the SHELX program [8].

Discussion

Since the first reports about dissolution of elemental tin in alkali metal ammonia solutions by Ioannis 1891 [3], the Zintl compounds containing tetrel elements, particularly the remarkably stable nine-membered cluster compounds, have been investigated intensively, and plenty of chemical reactions like reduction, oligomerization, functionalization, and even filling of the nine-membered clusters with transition metal atoms, have succeeded [4]. To enable this extended variety in chemical applications, often dissolution of solid Zintl cluster compounds in organic solvents is helpful or necessary, where addition of se-

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questering agents like crown ethers or cryptands are needed. During our systematic investigations of Zintl cluster compounds, among others the experimental procedure introduced above has been applied, and single crystals of [K([2.2.2]crypt)][K(18-crown-6)]$_2$[KSn$_9$]·1.5en have been grown. The crystal structure consists of deltahedral Sn$_9$ clusters, ethylenediamine solvate molecules, and potassium cations, which are coordinated either by atoms of Sn$_9$ clusters or by N and O atoms of cryptand or crown ether molecules. As indicated by the composition of four K atoms per polyanion, the Sn$_9$ cluster bears a fourfold negative charge corresponding to a 22 skeleton electron cluster. Based on Wade-Mingos' electron counting rules for deltahedral clusters, such a $[Sn_9]^{4-}$ anion is predicted to exhibit a *nido* cage, a monocapped square antiprism with C_{4v} symmetry. The shape of the Sn$_9$ cluster in the title structure deviates slightly from this symmetry as the ratio of the diagonals of the open "square" spanned by atoms Sn6, Sn7, Sn8, and Sn9, is 1.25 instead of 1.0 for the perfect square, and the dihedral angle between the two triangle halves of this square is 14° instead of 0° for a planar face [4]. The resulting symmetry is approximately D_{3h}. Most of the Sn–Sn bonds in the cluster are in the narrow range between 2.91 and 3.02 Å, while the short edges of the capped rectangle are slightly longer (Sn2–Sn3 3.068 Å, Sn4–Sn5 3.041 Å). If the polyhedron is described as a tricapped trigonal prism, the central prism is distorted. The heights are 3.342 Å (Sn3–Sn4), 3.437 Å (Sn2–Sn5), and 3.643 Å (Sn7–Sn9). As an interesting feature, the crystal structure of the title compound contains K cations in three different chemical environments: cations K1, which are exclusively coordinated to atoms of Sn$_9$ clusters forming infinite $^1{}_\infty[KSn_9]^{3-}$ chains along the *a* axis, as well as cations located between coordinating crown ether molecules and $[Sn_9]^{4-}$ clusters (K2, K3), and cations completely separated by the cryptand (K4). The K atoms show three (K1) or two (K1 opposite side, K2, K3) shorter (3.56-3.68 Å) and medium (3.81-3.99 Å) contacts to the Sn$_9$ cage. Further contacts are larger than 4.25 Å. The arrangement of cations and Sn$_9$ clusters as one-dimensional [KSn$_9$] chains is similar to those found in the crystal structure of the by-product [K([2.2.2]crypt)]$_3$[KSn$_9$] [2] both realizing the molecular deficit of sequestering agents in the reaction mixture.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	2*i*	–0.1066	0.3943	0.2809	0.048
H(1B)	2*i*	–0.1386	0.4757	0.3033	0.048
H(2A)	2*i*	0.0679	0.4629	0.3428	0.046
H(2B)	2*i*	–0.0238	0.4025	0.3804	0.046
H(3A)	2*i*	0.1616	0.3164	0.4050	0.051
H(3B)	2*i*	0.2546	0.3793	0.3752	0.051
H(4A)	2*i*	0.3771	0.2600	0.3716	0.045
H(4B)	2*i*	0.2802	0.2410	0.3219	0.045
H(5A)	2*i*	0.4863	0.2277	0.2407	0.043
H(5B)	2*i*	0.5716	0.2548	0.2915	0.043
H(6A)	2*i*	0.6145	0.3584	0.2146	0.044
H(6B)	2*i*	0.6732	0.2837	0.1826	0.044
H(7A)	2*i*	0.6673	0.3471	0.0789	0.037
H(7B)	2*i*	0.5955	0.4217	0.1075	0.037
H(8A)	2*i*	0.5478	0.4101	–0.0029	0.037
H(8B)	2*i*	0.4701	0.3410	0.0279	0.037
H(9A)	2*i*	0.2587	0.4122	–0.0022	0.037
H(9B)	2*i*	0.3459	0.4775	–0.0330	0.037
H(10A)	2*i*	0.1293	0.5257	–0.0047	0.048
H(10B)	2*i*	0.2213	0.5524	0.0431	0.048
H(11A)	2*i*	–0.0595	0.5468	0.0832	0.040
H(11B)	2*i*	0.0388	0.5687	0.1307	0.040
H(12A)	2*i*	–0.1544	0.5206	0.1932	0.047
H(12B)	2*i*	–0.1069	0.4443	0.1619	0.047
H(13A)	2*i*	0.2310	0.7681	0.5970	0.048
H(13B)	2*i*	0.2874	0.7608	0.5199	0.048
H(14A)	2*i*	0.3076	0.8753	0.5494	0.057
H(14B)	2*i*	0.3989	0.8379	0.6047	0.057
H(15A)	2*i*	0.5747	0.9142	0.5351	0.049
H(15B)	2*i*	0.4658	0.9395	0.4850	0.049
H(16A)	2*i*	0.6085	0.8823	0.4006	0.059
H(16B)	2*i*	0.6785	0.9442	0.4270	0.059
H(17A)	2*i*	0.9002	0.8851	0.3939	0.052
H(17B)	2*i*	0.8283	0.8282	0.3616	0.052
H(18A)	2*i*	1.0463	0.7786	0.3916	0.060
H(18B)	2*i*	0.9833	0.7833	0.4683	0.060
H(19A)	2*i*	1.0157	0.6502	0.4890	0.050
H(19B)	2*i*	1.0847	0.6491	0.4128	0.050
H(20A)	2*i*	0.9060	0.5821	0.3940	0.051
H(20B)	2*i*	1.0061	0.5368	0.4423	0.051
H(21A)	2*i*	0.8132	0.4717	0.4865	0.043
H(21B)	2*i*	0.7100	0.5271	0.4470	0.043
H(22A)	2*i*	0.6026	0.4691	0.5443	0.047
H(22B)	2*i*	0.6922	0.5053	0.5873	0.047
H(23A)	2*i*	0.5054	0.5908	0.6414	0.054
H(23B)	2*i*	0.4267	0.5325	0.6151	0.054
H(24A)	2*i*	0.3187	0.6323	0.5480	0.053
H(24B)	2*i*	0.2783	0.6380	0.6267	0.053
H(25A)	2*i*	1.2080	0.3144	0.1082	0.047
H(25B)	2*i*	1.2521	0.2313	0.1349	0.047
H(26A)	2*i*	1.1152	0.2997	0.2170	0.048
H(26B)	2*i*	0.9938	0.3045	0.1738	0.048
H(27A)	2*i*	0.8821	0.2370	0.2721	0.049
H(27B)	2*i*	1.0152	0.2310	0.3072	0.049
H(28A)	2*i*	1.0361	0.0992	0.3029	0.039
H(28B)	2*i*	0.9087	0.1297	0.3527	0.039
H(29A)	2*i*	0.7894	0.0337	0.3436	0.049
H(29B)	2*i*	0.9174	–0.0026	0.2975	0.049
H(30A)	2*i*	0.7103	–0.0413	0.2786	0.042
H(30B)	2*i*	0.6622	0.0415	0.2530	0.042
H(31A)	2*i*	1.0875	0.3310	0.0053	0.038
H(31B)	2*i*	0.9853	0.3313	0.0732	0.038
H(32A)	2*i*	0.8782	0.3139	–0.0189	0.035
H(32B)	2*i*	0.9760	0.2398	–0.0293	0.035
H(33A)	2*i*	0.7751	0.1853	–0.0199	0.034
H(33B)	2*i*	0.6844	0.2592	–0.0005	0.034
H(34A)	2*i*	0.6049	0.1991	0.1023	0.046
H(34B)	2*i*	0.5696	0.1613	0.0413	0.046
H(35A)	2*i*	0.5492	0.0512	0.1020	0.044
H(35B)	2*i*	0.5802	0.0809	0.1683	0.044
H(36A)	2*i*	0.6347	–0.0488	0.1703	0.037
H(36B)	2*i*	0.7423	–0.0368	0.1053	0.037
H(37A)	2*i*	1.1848	0.2100	–0.0233	0.038
H(37B)	2*i*	1.2923	0.2474	0.0071	0.038
H(38A)	2*i*	1.3705	0.1271	0.0045	0.042
H(38B)	2*i*	1.3239	0.1396	0.0815	0.042
H(39A)	2*i*	1.3393	0.0024	0.0189	0.049
H(39B)	2*i*	1.3056	0.0083	0.0976	0.049
H(40A)	2*i*	1.1084	–0.0347	0.0281	0.047
H(40B)	2*i*	1.2128	–0.0902	0.0615	0.047
H(41A)	2*i*	1.0453	–0.1358	0.1331	0.052
H(41B)	2*i*	0.9361	–0.0885	0.0912	0.052
H(42A)	2*i*	0.9483	–0.0783	0.2286	0.049
H(42B)	2*i*	0.8420	–0.1206	0.2024	0.049
H(3D)	2*i*	0.7566	0.0853	0.6473	0.178
H(3E)	2*i*	0.6294	0.0936	0.6180	0.178
H(43A)	2*i*	0.7265	–0.0455	0.6429	0.127
H(43B)	2*i*	0.5755	–0.0087	0.6579	0.127
H(44A)	2*i*	0.6063	0.0198	0.7642	0.100
H(44B)	2*i*	0.6851	–0.0610	0.7599	0.100
H(4D)	2*i*	0.8475	–0.0093	0.7798	0.082
H(4E)	2*i*	0.7893	0.0659	0.7574	0.082
H(45A)	2*i*	0.9970	0.0209	0.4308	0.125
H(45B)	2*i*	0.9561	0.0759	0.4883	0.125
H(5C)	2*i*	0.8339	–0.0496	0.5194	0.237
H(5E)	2*i*	0.7667	0.0265	0.5219	0.237

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)	2*i*	0.45730(8)	0.80696(6)	0.14239(5)	0.0232(4)	0.0328(6)	0.0344(6)	–0.0043(4)	–0.0045(4)	0.0096(5)
Sn(2)	2*i*	0.66561(8)	0.68383(6)	0.16202(5)	0.0196(4)	0.0392(6)	0.0255(6)	–0.0007(4)	0.0010(4)	0.0015(5)
Sn(3)	2*i*	0.56457(8)	0.78622(5)	0.27138(5)	0.0247(4)	0.0272(6)	0.0344(6)	–0.0066(4)	–0.0045(4)	–0.0032(5)
Sn(4)	2*i*	0.24803(8)	0.77259(5)	0.25259(5)	0.0184(4)	0.0285(6)	0.0343(6)	–0.0002(4)	–0.0029(4)	0.0008(5)
Sn(5)	2*i*	0.3391(1)	0.66780(6)	0.14608(5)	0.0427(5)	0.0401(7)	0.0233(6)	–0.0110(5)	–0.0124(5)	0.0004(5)
Sn(6)	2*i*	0.52769(9)	0.55309(6)	0.20655(6)	0.0226(4)	0.0327(6)	0.0546(8)	–0.0036(4)	–0.0035(5)	–0.0182(5)
Sn(7)	2*i*	0.62349(8)	0.62518(5)	0.30660(5)	0.0253(4)	0.0295(6)	0.0266(6)	–0.0006(4)	–0.0056(4)	0.0012(5)
Sn(8)	2*i*	0.37604(8)	0.70516(6)	0.36816(5)	0.0202(4)	0.0521(7)	0.0237(6)	–0.0060(4)	–0.0014(4)	–0.0043(5)
Sn(9)	2*i*	0.27897(8)	0.60846(5)	0.28650(5)	0.0270(4)	0.0289(6)	0.0294(6)	–0.0069(4)	0.0008(4)	–0.0002(5)
K(1)	2*i*	0.9436(3)	0.6929(2)	0.2568(2)	0.028(2)	0.066(3)	0.075(3)	–0.016(2)	–0.020(2)	0.011(2)
K(2)	2*i*	0.2815(3)	0.4359(2)	0.1940(2)	0.022(1)	0.035(2)	0.033(2)	–0.006(1)	–0.001(1)	–0.005(2)
O(1)	2*i*	0.0068(8)	0.4660(5)	0.2256(5)	0.021(4)	0.041(6)	0.034(6)	–0.003(4)	0.001(4)	0.003(5)
O(2)	2*i*	0.1267(9)	0.3629(5)	0.3160(5)	0.032(5)	0.038(6)	0.038(7)	–0.006(5)	0.003(5)	–0.006(5)
O(3)	2*i*	0.3975(8)	0.3183(5)	0.2821(5)	0.034(5)	0.039(6)	0.032(6)	–0.004(5)	–0.005(4)	–0.009(5)
O(4)	2*i*	0.5114(8)	0.3335(5)	0.1481(5)	0.029(5)	0.039(6)	0.031(6)	–0.006(4)	–0.007(4)	0.001(5)
O(5)	2*i*	0.3782(8)	0.4369(5)	0.0585(4)	0.029(5)	0.037(6)	0.023(6)	0.000(4)	–0.001(4)	–0.008(5)
O(6)	2*i*	0.1036(8)	0.4773(5)	0.0894(5)	0.026(4)	0.023(5)	0.039(6)	–0.004(4)	–0.004(4)	0.001(5)
C(1)	2*i*	–0.066(1)	0.4382(9)	0.2876(7)	0.038(8)	0.04(1)	0.03(1)	–0.004(7)	0.012(7)	–0.004(8)
C(2)	2*i*	0.026(1)	0.4192(8)	0.3369(8)	0.040(8)	0.032(9)	0.04(1)	–0.015(7)	0.020(7)	–0.026(8)
C(3)	2*i*	0.213(1)	0.3378(9)	0.3638(7)	0.042(8)	0.06(1)	0.025(9)	–0.026(8)	–0.002(7)	–0.004(8)
C(4)	2*i*	0.320(1)	0.2810(9)	0.3370(7)	0.036(7)	0.06(1)	0.018(8)	–0.008(7)	–0.005(6)	0.010(7)
C(5)	2*i*	0.514(1)	0.2714(8)	0.2561(8)	0.039(8)	0.029(9)	0.04(1)	0.003(7)	–0.006(7)	–0.008(7)
C(6)	2*i*	0.590(1)	0.3138(8)	0.1992(8)	0.025(7)	0.04(1)	0.04(1)	–0.002(7)	0.005(7)	0.001(8)
C(7)	2*i*	0.580(1)	0.3744(8)	0.0938(7)	0.031(7)	0.036(9)	0.026(9)	–0.013(6)	–0.005(6)	0.002(7)
C(8)	2*i*	0.496(1)	0.3878(8)	0.0379(7)	0.028(7)	0.033(9)	0.031(9)	–0.014(6)	0.016(6)	–0.013(7)
C(9)	2*i*	0.294(1)	0.4563(8)	0.0082(7)	0.039(7)	0.04(1)	0.016(8)	–0.012(7)	0.000(6)	–0.013(7)
C(10)	2*i*	0.185(1)	0.5092(9)	0.0314(7)	0.040(8)	0.05(1)	0.029(9)	–0.019(8)	–0.009(7)	0.009(8)
C(11)	2*i*	0.001(1)	0.5272(8)	0.1167(7)	0.030(7)	0.030(9)	0.034(9)	0.007(6)	0.002(7)	0.002(7)
C(12)	2*i*	–0.075(1)	0.4879(8)	0.1757(8)	0.021(6)	0.04(1)	0.05(1)	0.003(6)	0.004(7)	–0.008(8)
K(3)	2*i*	0.6319(3)	0.7043(2)	0.4704(2)	0.024(1)	0.029(2)	0.026(2)	–0.004(1)	–0.003(1)	–0.000(1)
O(7)	2*i*	0.4078(8)	0.7074(5)	0.5829(5)	0.028(5)	0.034(6)	0.042(7)	–0.008(5)	0.005(5)	–0.003(5)
O(8)	2*i*	0.4863(9)	0.8330(5)	0.5128(5)	0.035(5)	0.030(6)	0.045(7)	0.004(5)	0.007(5)	–0.002(5)
O(9)	2*i*	0.7504(9)	0.8414(5)	0.4584(5)	0.050(6)	0.035(6)	0.032(6)	–0.014(5)	–0.003(5)	0.002(5)
O(10)	2*i*	0.9077(8)	0.7096(6)	0.4237(5)	0.022(4)	0.041(7)	0.058(8)	–0.005(5)	0.001(5)	–0.010(6)
O(11)	2*i*	0.8274(8)	0.5762(5)	0.4920(5)	0.035(5)	0.033(6)	0.034(6)	0.000(5)	–0.006(5)	–0.002(5)
O(12)	2*i*	0.5594(9)	0.5779(5)	0.5437(5)	0.046(5)	0.028(6)	0.037(7)	–0.008(5)	–0.006(5)	0.005(5)
C(13)	2*i*	0.313(1)	0.7667(9)	0.5639(8)	0.025(7)	0.05(1)	0.04(1)	0.000(7)	0.004(7)	–0.012(8)
C(14)	2*i*	0.373(1)	0.8328(9)	0.5605(8)	0.043(9)	0.06(1)	0.03(1)	0.007(8)	0.011(8)	–0.010(9)
C(15)	2*i*	0.538(2)	0.9008(8)	0.4962(8)	0.052(9)	0.025(9)	0.04(1)	–0.001(7)	–0.009(8)	0.002(8)
C(16)	2*i*	0.644(2)	0.8961(9)	0.4395(9)	0.05(1)	0.03(1)	0.07(1)	0.001(8)	–0.007(9)	–0.013(9)
C(17)	2*i*	0.861(2)	0.8381(8)	0.4027(8)	0.053(9)	0.04(1)	0.04(1)	–0.026(8)	0.011(8)	0.005(8)
C(18)	2*i*	0.963(1)	0.7777(9)	0.4233(9)	0.027(7)	0.07(1)	0.05(1)	–0.029(8)	0.009(8)	–0.01(1)
C(19)	2*i*	0.999(1)	0.6484(8)	0.4426(8)	0.026(7)	0.04(1)	0.05(1)	0.003(7)	0.008(7)	–0.005(8)
C(20)	2*i*	0.939(1)	0.5802(9)	0.4376(8)	0.029(7)	0.05(1)	0.04(1)	0.019(7)	–0.014(7)	–0.006(8)
C(21)	2*i*	0.753(1)	0.5182(8)	0.4883(7)	0.053(9)	0.024(9)	0.031(9)	0.004(7)	–0.020(7)	0.004(7)
C(22)	2*i*	0.651(1)	0.5119(8)	0.5457(8)	0.047(8)	0.029(9)	0.05(1)	–0.002(7)	–0.021(8)	–0.002(8)
C(23)	2*i*	0.463(1)	0.5802(9)	0.6035(8)	0.047(9)	0.06(1)	0.03(1)	–0.023(9)	–0.007(8)	0.000(9)
C(24)	2*i*	0.353(1)	0.6391(9)	0.5896(8)	0.035(8)	0.05(1)	0.04(1)	–0.020(8)	0.001(7)	0.009(9)
K(4)	2*i*	0.9578(3)	0.1152(2)	0.1299(2)	0.023(1)	0.024(2)	0.031(2)	–0.004(1)	–0.004(1)	0.000(1)
N(1)	2*i*	1.118(1)	0.2410(6)	0.0690(6)	0.029(6)	0.037(8)	0.022(7)	–0.008(5)	–0.004(5)	–0.001(6)
N(2)	2*i*	0.800(1)	–0.0087(6)	0.1886(6)	0.031(6)	0.026(7)	0.025(7)	–0.007(5)	0.000(5)	0.003(5)
O(13)	2*i*	1.0499(9)	0.2036(5)	0.2135(5)	0.040(5)	0.035(6)	0.029(6)	–0.017(5)	0.003(5)	–0.006(5)
O(14)	2*i*	0.8615(8)	0.1022(5)	0.2675(5)	0.035(5)	0.037(6)	0.027(6)	–0.009(5)	–0.002(4)	0.002(5)
O(15)	2*i*	0.8416(8)	0.2333(5)	0.0502(5)	0.030(5)	0.033(6)	0.031(6)	–0.005(4)	–0.013(4)	0.005(5)
O(16)	2*i*	0.7103(8)	0.1049(5)	0.0878(5)	0.024(4)	0.033(6)	0.029(6)	–0.007(4)	–0.004(4)	0.008(5)
O(17)	2*i*	1.2010(8)	0.0871(5)	0.0427(5)	0.021(4)	0.039(7)	0.047(7)	–0.005(4)	0.001(4)	–0.009(5)
O(18)	2*i*	1.0690(8)	–0.0292(5)	0.1159(5)	0.033(5)	0.030(6)	0.029(6)	–0.004(4)	–0.006(4)	–0.002(5)
C(25)	2*i*	1.175(1)	0.2666(9)	0.1238(7)	0.035(7)	0.05(1)	0.04(1)	–0.022(7)	–0.008(7)	0.006(8)
C(26)	2*i*	1.077(1)	0.2747(8)	0.1850(7)	0.048(8)	0.04(1)	0.04(1)	–0.026(8)	–0.004(7)	0.004(8)
C(27)	2*i*	0.969(1)	0.2077(9)	0.2771(7)	0.044(8)	0.05(1)	0.028(9)	–0.014(8)	–0.003(7)	–0.002(8)
C(28)	2*i*	0.949(1)	0.1304(8)	0.3053(7)	0.035(7)	0.05(1)	0.019(8)	–0.004(7)	–0.008(6)	–0.013(7)
C(29)	2*i*	0.834(1)	0.0312(8)	0.2971(8)	0.044(8)	0.03(1)	0.04(1)	–0.021(7)	0.009(7)	0.006(8)
C(30)	2*i*	0.742(1)	0.0047(8)	0.2554(7)	0.032(7)	0.037(9)	0.04(1)	–0.006(7)	–0.013(7)	0.014(7)
C(31)	2*i*	1.032(1)	0.3002(8)	0.0385(7)	0.039(7)	0.025(8)	0.031(9)	–0.007(7)	–0.003(7)	–0.002(7)
C(32)	2*i*	0.930(1)	0.2721(8)	0.0045(7)	0.029(7)	0.029(9)	0.027(9)	0.002(6)	–0.007(6)	0.006(7)
C(33)	2*i*	0.735(1)	0.2140(7)	0.0178(7)	0.034(7)	0.021(8)	0.032(9)	–0.003(6)	–0.007(6)	–0.008(7)
C(34)	2*i*	0.644(1)	0.1708(8)	0.0643(8)	0.022(6)	0.030(9)	0.07(1)	–0.008(6)	–0.014(7)	–0.007(8)
C(35)	2*i*	0.622(1)	0.0581(8)	0.1275(8)	0.024(6)	0.05(1)	0.04(1)	–0.009(7)	–0.010(7)	0.005(8)

Table 3. continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(36)	2*i*	0.698(1)	–0.0152(7)	0.1463(7)	0.031(7)	0.030(9)	0.032(9)	–0.014(6)	–0.011(6)	0.016(7)
C(37)	2*i*	1.225(1)	0.2125(8)	0.0179(7)	0.023(6)	0.04(1)	0.028(9)	–0.015(6)	–0.002(6)	0.012(7)
C(38)	2*i*	1.292(1)	0.1395(8)	0.0379(7)	0.025(7)	0.05(1)	0.031(9)	–0.007(7)	0.006(6)	–0.004(8)
C(39)	2*i*	1.266(1)	0.0128(8)	0.0555(9)	0.024(7)	0.028(9)	0.07(1)	0.008(6)	–0.001(7)	–0.007(8)
C(40)	2*i*	1.171(1)	–0.0400(8)	0.0606(8)	0.034(7)	0.029(9)	0.05(1)	0.007(7)	–0.004(7)	–0.012(8)
C(41)	2*i*	0.988(1)	–0.0884(8)	0.1287(8)	0.045(8)	0.029(9)	0.05(1)	–0.005(7)	0.001(8)	0.004(8)
C(42)	2*i*	0.895(1)	–0.0783(7)	0.1918(8)	0.050(9)	0.013(8)	0.06(1)	–0.013(7)	–0.024(8)	0.014(8)
N(3)	2*i*	0.706(2)	0.063(1)	0.624(1)	0.14(1)	0.13(1)	0.10(1)	0.006(9)	–0.043(8)	–0.035(8)
C(43)	2*i*	0.669(2)	–0.004(2)	0.663(1)	0.10(2)	0.16(3)	0.09(2)	–0.04(2)	–0.03(1)	–0.07(2)
C(44)	2*i*	0.683(2)	–0.009(1)	0.740(1)	0.04(1)	0.10(2)	0.11(2)	–0.01(1)	0.01(1)	–0.04(2)
N(4)	2*i*	0.808(1)	0.0189(8)	0.7468(7)	0.049(8)	0.06(1)	0.05(1)	0.005(7)	–0.011(7)	–0.018(8)
C(45)	2*i*	0.961(2)	0.025(1)	0.478(1)	0.09(1)	0.11(1)	0.11(1)	–0.017(9)	–0.027(8)	–0.003(9)
N(5)	2*i*	0.823(2)	–0.005(1)	0.496(1)	0.15(1)	0.16(1)	0.16(1)	–0.011(9)	–0.012(9)	–0.017(9)

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