Crystal structure of bis(tetrabutylammonium) oxopentathiocyanato-molybdate(V) — ethanol (1:0.5), $[(C_4H_9)_4N]_2[MoO(NCS)_5] \cdot 0.5C_2H_5OH$

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Abstract

$C_{38}H_{75}MoN_7O_{1.5}S_5$, triclinic, $P\bar{1}$ (no. 2), $a = 16.282(3)$ Å, $b = 17.261(4)$ Å, $c = 21.234(4)$ Å, $\alpha = 111.27(3)^\circ$, $\beta = 112.36(3)^\circ$, $\gamma = 92.36(3)^\circ$, $V = 5030.8$ Å³, $Z = 4$, $R_{\text{int}}(F) = 0.040$, $wR_{\text{ref}}(F^2) = 0.107$, $T = 293$ K.

Source of material

The precursor $(Bu_4N)_2[Mo_6O_{18}(NC_6H_4Br)]$ was prepared according to the procedure [1]. $(Bu_4N)_2[Mo_6O_{18}(NC_6H_4Br)]$ (1 mmol, 1.52 g) and KSCN (10 mmol, 0.97 g) were resolved in 20 ml acetonitrile to obtain a dark red solution. On evaporation, the product deposited from the filtrate as plate-like crystals. X-ray quality crystals were recrystallized from acetonitrile solution.

Discussion

The title compound, $[Bu_4N]_2[MoO(NCS)_5]$, where the Bu_4N^+ cation is tetrabutylammonium, consists of two Bu_4N^+ cations and a molybdenum(V) complex anion $[MoO(NCS)_5]^{2-}$. The central Mo is coordinated by one oxygen atom and five NCS ligands (via Mo—N bonds), which construct an octahedron. There are two crystallographically independent $[MoO(NCS)_5]^{2-}$ anion in the unit cell, and both possess C_1 symmetry in the solid state. The most obvious difference between them is their axial C—N—Mo angles at 171.7° and 150.9° . The approximate symmetry of the anion is C_{4v} , but the nonlinear unit C—N—Mo brakes the axial symmetry. The dihedral angles between axial NCS and the closest equatorial NCS are $24.2(9)^\circ$ (Mo1 anion) and $29.3(3)^\circ$ (Mo2 anion), respectively, which deviates from 0° or 45° , so the mirror

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symmetry is broken further. The bond lengths of Mo1—N5 and Mo2—N10, which are mutually *trans* to Mo—O bonds are 2.22 and 2.21 Å, respectively. The shorter Mo—N bond length (2.056(9) Å) in the equatorial plane, leads to an obviously distorted polyhedron [MoN₅O] caused by the *trans* effect of the terminal oxygen [2,3]. The Mo—O bonds reveal substantial double-bond character, so that the bond lengths of 1.668(1) and 1.669(1) Å are very close to the bond lengths in [MoOCl₅]²⁻ (1.647(5) Å) [4] and MoO(ttp)NCS (1.673(3) Å) [5]. The four equatorial NCS ligands are nearly in a plane in both anions. The bond angles between these equatorial ligands and O—Mo bonds are 95.9±1.4°. The reason for such distortion is that the O—Mo double bonds are more repellant than the *trans* Mo—N single bonds and push equatorial NCS away from the oxygen atoms. The equatorial C—N—Mo angles are close to 180° (172.7° - 177.7°), which suggest an *sp* hybridization of these N atoms [6]. Oppositely, the *axial* C—N—Mo angles present larger deviation from linearity (171.7° and 150.9°). In one of four crystallographically independent Bu₄N⁺ cations, the atom C26A/C26B at end of one butyl is disordered with occupancies of 50 %, respectively. This kind of disorder is very common in the crystal of tetrabutylammonium salts because *n*-butyl is a flexible chain. What's more, the only solvent molecule in the crystal structure, an ethanol, is disordered with same occupancy of 50 %.

Table 1. Data collection and handling.

Crystal:	red block, size 0.35 × 0.35 × 0.45 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	5.03 cm ⁻¹
Diffractionmeter, scan mode:	Nonius Kappa CCD, CCD
2θ _{max} :	52°
N(<i>hkl</i>) _{measured} , N(<i>hkl</i>) _{unique} :	37529, 19666
Criterion for I _{obs} , N(<i>hkl</i>) _{gt} :	I _{obs} > 2 σ(I _{obs}), 13727
N(<i>param</i>) _{refined} :	979
Programs:	SHELXS-97, SHELXL-97, SHELXTL [7]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U _{iso}
H(11A)	2i		0.0333	0.5886	0.8458	0.055
H(11B)	2i		0.0984	0.6775	0.8844	0.055
H(12A)	2i		-0.0020	0.6188	0.9467	0.065
H(12B)	2i		0.0552	0.7120	0.9846	0.065
H(13A)	2i		0.1347	0.5681	0.9530	0.063
H(13B)	2i		0.1887	0.6625	0.9994	0.063
H(14A)	2i		0.1991	0.5970	1.0794	0.110
H(14B)	2i		0.0931	0.5764	1.0487	0.110
H(14C)	2i		0.1469	0.6709	1.0950	0.110
H(15A)	2i		0.0222	0.6840	0.7664	0.061
H(15B)	2i		-0.0438	0.5978	0.7371	0.061
H(16A)	2i		-0.1008	0.7463	0.7267	0.078
H(16B)	2i		-0.1683	0.6611	0.6992	0.078
H(17A)	2i		-0.0438	0.6784	0.6379	0.113
H(17B)	2i		-0.1068	0.5916	0.6121	0.113
H(18A)	2i		-0.1659	0.6488	0.5269	0.143
H(18B)	2i		-0.1691	0.7313	0.5888	0.143
H(18C)	2i		-0.2325	0.6446	0.5634	0.143
H(19A)	2i		-0.1212	0.6600	0.8802	0.060
H(19B)	2i		-0.1720	0.6720	0.8064	0.060
H(20A)	2i		-0.1027	0.5246	0.7972	0.068
H(20B)	2i		-0.1838	0.5368	0.7342	0.068
H(21A)	2i		-0.2363	0.4515	0.7810	0.087
H(21B)	2i		-0.1928	0.5257	0.8608	0.087
H(22A)	2i		-0.3467	0.5185	0.8068	0.118
H(22B)	2i		-0.3394	0.5299	0.7390	0.118
H(22C)	2i		-0.2954	0.6050	0.8185	0.118

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(23A)	2i		-0.0201	0.7903	0.9261	0.071
H(23B)	2i		-0.0707	0.7980	0.8503	0.071
H(24A)	2i		0.0673	0.8213	0.8430	0.111
H(24B)	2i		0.1188	0.8117	0.9182	0.111
H(25C)	2i	0.50	0.1337	0.9537	0.9458	0.109
H(25D)	2i	0.50	0.0727	0.9322	0.9806	0.109
H(25A)	2i	0.50	0.0205	0.9415	0.9098	0.109
H(25B)	2i	0.50	0.1247	0.9562	0.9342	0.109
H(26A)	2i	0.50	0.1365	0.9163	1.0292	0.139
H(26B)	2i	0.50	0.0349	0.9238	1.0093	0.139
H(26C)	2i	0.50	0.1090	1.0041	1.0365	0.139
C(26B)	2i	0.50	0.0125(8)	0.9628(7)	0.8994(6)	0.125(3)
H(26D)	2i	0.50	0.0070	0.9474	0.8495	0.187
H(26E)	2i	0.50	0.0318	1.0234	0.9266	0.187
H(26F)	2i	0.50	-0.0452	0.9448	0.8976	0.187
H(27A)	2i		0.1000	-0.2178	0.2546	0.050
H(27B)	2i		0.0548	-0.1422	0.2825	0.050
H(28A)	2i		0.1510	-0.1520	0.1949	0.066
H(28B)	2i		0.1163	-0.0702	0.2284	0.066
H(29A)	2i		0.0155	-0.1583	0.1021	0.076
H(29B)	2i		-0.0018	-0.2296	0.1276	0.076
H(30A)	2i		-0.1200	-0.1587	0.1140	0.171
H(30B)	2i		-0.0522	-0.0721	0.1715	0.171
H(30C)	2i		-0.0685	-0.1425	0.1982	0.171
H(31A)	2i		0.1737	-0.2141	0.3732	0.053
H(31B)	2i		0.1264	-0.1411	0.4026	0.053
H(32A)	2i		0.2760	-0.0589	0.4945	0.071
H(32B)	2i		0.3122	-0.1426	0.4699	0.071
H(33A)	2i		0.2095	-0.2086	0.5014	0.083
H(33B)	2i		0.2883	-0.1370	0.5707	0.083
H(34A)	2i		0.1520	-0.1241	0.5800	0.139
H(34B)	2i		0.1109	-0.1146	0.5040	0.139
H(34C)	2i		0.1898	-0.0434	0.5739	0.139
H(35A)	2i		0.2580	0.0042	0.4230	0.058
H(35B)	2i		0.2066	0.0013	0.3430	0.058
H(36A)	2i		0.0706	-0.0026	0.3533	0.064
H(36B)	2i		0.1162	-0.0101	0.4300	0.064
H(37A)	2i		0.1615	0.1325	0.4059	0.100
H(37B)	2i		0.2081	0.1249	0.4824	0.100
H(38A)	2i		0.1010	0.2090	0.4865	0.121
H(38B)	2i		0.0713	0.1268	0.4945	0.121
H(38C)	2i		0.0246	0.1343	0.4179	0.121
H(39A)	2i		0.2822	-0.1031	0.3026	0.055
H(39B)	2i		0.3285	-0.1044	0.3814	0.055
H(40A)	2i		0.2326	-0.2512	0.2484	0.057
H(40B)	2i		0.2792	-0.2526	0.3278	0.057
H(41A)	2i		0.3770	-0.1920	0.2635	0.064
H(41B)	2i		0.4220	-0.1991	0.3403	0.064
H(42A)	2i		0.4421	-0.3095	0.2499	0.105
H(42B)	2i		0.3368	-0.3390	0.2023	0.105
H(42C)	2i		0.3816	-0.3462	0.2791	0.105
H(43A)	2i		0.3835	0.4840	0.5636	0.054
H(43B)	2i		0.4681	0.5210	0.6409	0.054
H(44A)	2i		0.3035	0.4304	0.6166	0.067
H(44B)	2i		0.3926	0.4622	0.6909	0.067
H(45A)	2i		0.4644	0.3681	0.6310	0.072
H(45B)	2i		0.3792	0.3392	0.5544	0.072
H(46A)	2i		0.3798	0.2380	0.6037	0.121
H(46B)	2i		0.2943	0.2784	0.5992	0.121
H(46C)	2i		0.3795	0.3073	0.6759	0.121
H(47A)	2i		0.3895	0.7059	0.6374	0.051
H(47B)	2i		0.4764	0.6670	0.6518	0.051
H(48A)	2i		0.3298	0.6048	0.5103	0.052
H(48B)	2i		0.4262	0.5824	0.5282	0.052
H(49A)	2i		0.3900	0.7438	0.5384	0.061
H(49B)	2i		0.4883	0.7259	0.5625	0.061
H(50A)	2i		0.4363	0.7259	0.4452	0.109
H(50B)	2i		0.3536	0.6524	0.4157	0.109
H(50C)	2i		0.4524	0.6353	0.4400	0.109
H(51A)	2i		0.2500	0.5344	0.5457	0.056
H(51B)	2i		0.2406	0.5363	0.6167	0.056
H(52A)	2i		0.2375	0.6746	0.5764	0.062
H(52B)	2i		0.2367	0.6822	0.6522	0.062

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(53A)	2i		0.1002	0.5673	0.5117	0.070
H(53B)	2i		0.0978	0.5854	0.5888	0.070
H(54A)	2i		0.0008	0.6573	0.5287	0.109
H(54B)	2i		0.0825	0.7062	0.5268	0.109
H(54C)	2i		0.0788	0.7233	0.6032	0.109
H(55A)	2i		0.3771	0.6839	0.7371	0.061
H(55B)	2i		0.3579	0.5930	0.7345	0.061
H(56A)	2i		0.5295	0.6731	0.7619	0.081
H(56B)	2i		0.5088	0.5853	0.7647	0.081
H(57A)	2i		0.4705	0.6587	0.8669	0.095
H(57B)	2i		0.5739	0.6786	0.8858	0.095
H(58A)	2i		0.5268	0.8003	0.9335	0.139
H(58B)	2i		0.4555	0.7841	0.8531	0.139
H(58C)	2i		0.5596	0.8042	0.8740	0.139
H(59A)	2i		0.3884	0.0723	0.8626	0.058
H(59B)	2i		0.3326	0.1223	0.9034	0.058
H(60A)	2i		0.2646	0.0252	0.7490	0.064
H(60B)	2i		0.2083	0.0820	0.7855	0.064
H(61A)	2i		0.2729	-0.0522	0.8232	0.073
H(61B)	2i		0.2109	0.0019	0.8541	0.073
H(62A)	2i		0.1232	-0.1285	0.7599	0.137
H(62B)	2i		0.1534	-0.1111	0.7036	0.137
H(62C)	2i		0.0915	-0.0563	0.7340	0.137
H(63A)	2i		0.3336	0.2942	0.8499	0.058
H(63B)	2i		0.2534	0.2194	0.8223	0.058
H(64A)	2i		0.3677	0.3203	0.9739	0.070
H(64B)	2i		0.2899	0.2430	0.9474	0.070
H(65A)	2i		0.2578	0.3878	0.9146	0.082
H(65B)	2i		0.1807	0.3112	0.8900	0.082
H(66A)	2i		0.1862	0.4233	0.9945	0.145
H(66B)	2i		0.2882	0.4235	1.0395	0.145

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(66C)	2i		0.2118	0.3465	1.0153	0.145
H(67A)	2i		0.3174	0.1452	0.7408	0.055
H(67B)	2i		0.4016	0.2197	0.7793	0.055
H(68A)	2i		0.4191	0.0521	0.7616	0.058
H(68B)	2i		0.4998	0.1278	0.7907	0.058
H(69A)	2i		0.3573	0.0618	0.6451	0.063
H(69B)	2i		0.4395	0.1357	0.6742	0.063
H(70A)	2i		0.4624	0.0087	0.6004	0.088
H(70B)	2i		0.4563	-0.0325	0.6536	0.088
H(70C)	2i		0.5382	0.0414	0.6822	0.088
H(71A)	2i		0.4663	0.2305	0.9638	0.061
H(71B)	2i		0.5087	0.1811	0.9102	0.061
H(72A)	2i		0.4804	0.3463	0.9300	0.069
H(72B)	2i		0.5348	0.2971	0.8867	0.069
H(73A)	2i		0.5922	0.3471	1.0428	0.077
H(73B)	2i		0.6474	0.3015	0.9989	0.077
H(74A)	2i		0.7094	0.4468	1.0617	0.123
H(74B)	2i		0.6162	0.4662	1.0188	0.123
H(74C)	2i		0.6718	0.4207	0.9754	0.123
H(3)	2i	0.50	0.5190	0.0568	0.1013	0.127
H(75A)	2i	0.50	0.3632	-0.0129	0.0848	0.171
H(75B)	2i	0.50	0.3572	0.0373	0.0338	0.171
H(76A)	2i	0.50	0.2985	-0.1080	-0.0431	0.142
H(76B)	2i	0.50	0.3898	-0.1284	0.0021	0.142
H(76C)	2i	0.50	0.3881	-0.0788	-0.0472	0.142
H(4)	2i	0.50	0.4959	-0.0433	0.0060	0.175
H(77A)	2i	0.50	0.4136	-0.0376	0.0953	0.089
H(77B)	2i	0.50	0.4403	0.0395	0.0787	0.089
H(78A)	2i	0.50	0.2945	-0.0183	0.0139	0.310
H(78B)	2i	0.50	0.3186	-0.1055	-0.0229	0.310
H(78C)	2i	0.50	0.3441	-0.0275	-0.0381	0.310

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Mo(1)	2i		0.29692(2)	0.12401(1)	0.26177(1)	0.0405(1)	0.0370(1)	0.0396(1)	0.0059(1)	0.0152(1)	0.0155(1)
Mo(2)	2i		0.14514(2)	0.42025(2)	0.69695(2)	0.0430(2)	0.0540(2)	0.0588(2)	0.0057(1)	0.0251(1)	0.0264(1)
S(1)	2i		0.44565(6)	0.09979(5)	0.49637(4)	0.0609(5)	0.0687(5)	0.0420(4)	0.0066(4)	0.0137(4)	0.0251(4)
S(2)	2i		0.37304(5)	0.42818(5)	0.39832(5)	0.0563(5)	0.0382(4)	0.0710(5)	0.0084(3)	0.0299(4)	0.0092(4)
S(3)	2i		0.28590(7)	-0.16780(5)	0.11566(6)	0.0955(7)	0.0435(4)	0.0766(6)	0.0086(4)	0.0421(6)	0.0067(4)
S(4)	2i		0.20748(8)	0.17609(7)	0.03945(6)	0.1184(9)	0.0956(7)	0.0634(6)	0.0371(6)	0.0438(6)	0.0520(6)
S(5)	2i		0.60407(5)	0.12855(5)	0.26103(5)	0.0513(4)	0.0471(4)	0.0679(5)	0.0139(3)	0.0342(4)	0.0234(4)
S(6)	2i		0.22927(7)	0.72426(6)	0.81376(7)	0.0832(7)	0.0549(5)	0.133(1)	0.0069(5)	0.0747(7)	0.0111(6)
S(7)	2i		-0.17654(6)	0.39314(7)	0.60733(6)	0.0458(5)	0.1135(8)	0.0664(6)	0.0211(5)	0.0179(5)	0.0146(6)
S(8)	2i		0.09599(6)	0.12383(5)	0.65201(5)	0.0707(6)	0.0573(5)	0.0752(6)	0.0051(4)	0.0233(5)	0.0347(5)
S(9)	2i		0.46262(6)	0.46297(7)	0.85178(6)	0.0536(5)	0.1067(8)	0.0888(7)	0.0225(5)	0.0167(5)	0.0611(6)
S(10)	2i		-0.00406(6)	0.39889(5)	0.84938(5)	0.0722(6)	0.0519(5)	0.0855(6)	0.0107(4)	0.0505(5)	0.0287(4)
O(1)	2i		0.1937(1)	0.1108(1)	0.2597(1)	0.043(1)	0.064(1)	0.066(1)	0.007(1)	0.022(1)	0.025(1)
O(2)	2i		0.1579(1)	0.4062(1)	0.6195(1)	0.054(1)	0.081(2)	0.062(1)	0.003(1)	0.028(1)	0.032(1)
N(1)	2i		0.3624(2)	0.1126(1)	0.3603(1)	0.048(1)	0.044(1)	0.043(1)	0.007(1)	0.018(1)	0.020(1)
N(2)	2i		0.3321(2)	0.2538(1)	0.3195(1)	0.047(1)	0.040(1)	0.045(1)	0.011(1)	0.017(1)	0.016(1)
N(3)	2i		0.2882(2)	-0.0027(1)	0.2030(1)	0.056(2)	0.035(1)	0.045(1)	0.002(1)	0.017(1)	0.013(1)
N(4)	2i		0.2567(2)	0.1426(1)	0.1651(1)	0.055(2)	0.050(1)	0.040(1)	0.010(1)	0.012(1)	0.021(1)
N(5)	2i		0.4337(2)	0.1360(1)	0.2626(1)	0.046(1)	0.039(1)	0.043(1)	0.008(1)	0.021(1)	0.016(1)
N(6)	2i		0.1690(2)	0.5507(2)	0.7402(2)	0.052(2)	0.057(2)	0.079(2)	0.008(1)	0.028(2)	0.030(2)
N(7)	2i		0.0076(2)	0.4133(2)	0.6480(2)	0.045(2)	0.070(2)	0.067(2)	0.005(1)	0.022(1)	0.030(2)
N(8)	2i		0.1228(2)	0.2925(2)	0.6745(2)	0.059(2)	0.055(2)	0.074(2)	0.007(1)	0.035(2)	0.026(1)
N(9)	2i		0.2779(2)	0.4323(2)	0.7657(2)	0.048(2)	0.065(2)	0.064(2)	0.011(1)	0.026(2)	0.029(1)
N(10)	2i		0.1136(2)	0.4320(2)	0.7920(2)	0.057(2)	0.063(2)	0.060(2)	0.013(1)	0.030(1)	0.028(1)
N(11)	2i		-0.0350(2)	0.6834(1)	0.8366(1)	0.041(1)	0.045(1)	0.062(2)	0.015(1)	0.031(1)	0.022(1)
N(12)	2i		0.1918(1)	-0.1156(1)	0.3449(1)	0.040(1)	0.037(1)	0.043(1)	0.0022(9)	0.017(1)	0.016(1)
N(13)	2i		0.3681(1)	0.5897(1)	0.6397(1)	0.034(1)	0.039(1)	0.044(1)	0.0086(9)	0.022(1)	0.016(1)
N(14)	2i		0.3771(2)	0.1858(1)	0.8541(1)	0.046(1)	0.044(1)	0.047(1)	0.013(1)	0.019(1)	0.026(1)
C(1)	2i		0.3986(2)	0.1084(2)	0.4180(2)	0.043(2)	0.036(1)	0.049(2)	0.003(1)	0.024(1)	0.013(1)
C(2)	2i		0.3495(2)	0.3268(2)	0.3527(2)	0.031(1)	0.049(2)	0.040(2)	0.012(1)	0.015(1)	0.020(1)
C(3)	2i		0.2880(2)	-0.0712(2)	0.1661(2)	0.049(2)	0.047(2)	0.044(2)	0.002(1)	0.018(1)	0.020(1)
C(4)	2i		0.2378(2)	0.1564(2)	0.1123(2)	0.051(2)	0.041(2)	0.053(2)	0.011(1)	0.023(2)	0.018(1)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(5)	2i		0.5040(2)	0.1319(2)	0.2612(1)	0.054(2)	0.030(1)	0.035(2)	0.007(1)	0.019(1)	0.014(1)
C(6)	2i		0.1944(2)	0.6238(2)	0.7714(2)	0.042(2)	0.066(2)	0.071(2)	0.014(2)	0.035(2)	0.031(2)
C(7)	2i		−0.0693(2)	0.4057(2)	0.6314(2)	0.056(2)	0.054(2)	0.047(2)	0.008(2)	0.021(2)	0.015(1)
C(8)	2i		0.1117(2)	0.2219(2)	0.6654(2)	0.043(2)	0.058(2)	0.051(2)	0.005(1)	0.021(1)	0.025(2)
C(9)	2i		0.3556(2)	0.4453(2)	0.8027(2)	0.063(2)	0.056(2)	0.060(2)	0.016(2)	0.032(2)	0.034(2)
C(10)	2i		0.0641(2)	0.4172(2)	0.8156(2)	0.053(2)	0.037(2)	0.051(2)	0.009(1)	0.021(2)	0.017(1)
C(11)	2i		0.0416(2)	0.6479(2)	0.8776(2)	0.042(2)	0.049(2)	0.051(2)	0.015(1)	0.025(1)	0.018(1)
C(12)	2i		0.0510(2)	0.6537(2)	0.9527(2)	0.056(2)	0.058(2)	0.053(2)	0.009(1)	0.031(2)	0.019(2)
C(13)	2i		0.1358(2)	0.6233(2)	0.9884(2)	0.054(2)	0.048(2)	0.044(2)	0.003(1)	0.018(2)	0.011(1)
C(14)	2i		0.1445(3)	0.6162(2)	1.0593(2)	0.082(3)	0.082(2)	0.056(2)	0.011(2)	0.029(2)	0.031(2)
C(15)	2i		−0.0365(2)	0.6591(2)	0.7599(2)	0.046(2)	0.065(2)	0.057(2)	0.018(1)	0.031(2)	0.031(2)
C(16)	2i		−0.1088(2)	0.6849(2)	0.7063(2)	0.051(2)	0.085(2)	0.081(2)	0.026(2)	0.033(2)	0.052(2)
C(17)	2i		−0.1027(3)	0.6526(3)	0.6302(2)	0.065(2)	0.171(4)	0.094(3)	0.042(3)	0.043(2)	0.093(3)
C(18)	2i		−0.1738(3)	0.6709(3)	0.5722(2)	0.097(3)	0.119(3)	0.097(3)	0.022(3)	0.046(3)	0.066(3)
C(19)	2i		−0.1255(2)	0.6456(2)	0.8303(2)	0.047(2)	0.052(2)	0.063(2)	0.016(1)	0.035(2)	0.023(2)
C(20)	2i		−0.1555(2)	0.5503(2)	0.7872(2)	0.050(2)	0.055(2)	0.067(2)	0.006(1)	0.029(2)	0.021(2)
C(21)	2i		−0.2220(2)	0.5128(2)	0.8079(2)	0.062(2)	0.062(2)	0.097(3)	0.010(2)	0.039(2)	0.032(2)
C(22)	2i		−0.3086(2)	0.5444(2)	0.7916(2)	0.058(2)	0.090(3)	0.097(3)	0.016(2)	0.038(2)	0.043(2)
C(23)	2i		−0.0198(2)	0.7786(2)	0.8779(2)	0.058(2)	0.042(2)	0.085(2)	0.016(1)	0.044(2)	0.021(2)
C(24)	2i		0.0670(3)	0.8304(2)	0.8907(3)	0.080(3)	0.052(2)	0.150(4)	0.008(2)	0.071(3)	0.024(2)
C(25)	2i		0.0749(3)	0.9244(2)	0.9342(3)	0.076(3)	0.056(2)	0.142(4)	0.010(2)	0.053(3)	0.036(3)
C(26A)	2i	0.50	0.0900(6)	0.9437(5)	1.0081(5)	0.093(6)	0.058(5)	0.082(6)	−0.005(4)	0.014(5)	0.008(4)
C(27)	2i		0.1067(2)	−0.1565(2)	0.2723(2)	0.034(1)	0.044(2)	0.043(2)	0.002(1)	0.013(1)	0.018(1)
C(28)	2i		0.1040(2)	−0.1316(2)	0.2101(2)	0.052(2)	0.066(2)	0.056(2)	0.008(2)	0.025(2)	0.032(2)
C(29)	2i		0.0120(2)	−0.1685(2)	0.1431(2)	0.068(2)	0.073(2)	0.050(2)	0.018(2)	0.022(2)	0.030(2)
C(30)	2i		−0.0640(3)	−0.1322(4)	0.1580(2)	0.086(3)	0.188(5)	0.077(3)	0.053(3)	0.028(3)	0.066(3)
C(31)	2i		0.1815(2)	−0.1529(2)	0.3968(2)	0.050(2)	0.043(2)	0.043(2)	0.006(1)	0.021(1)	0.020(1)
C(32)	2i		0.2598(2)	−0.1204(2)	0.4738(2)	0.055(2)	0.071(2)	0.047(2)	0.010(2)	0.019(2)	0.025(2)
C(33)	2i		0.2338(3)	−0.1479(2)	0.5259(2)	0.082(3)	0.076(2)	0.049(2)	0.016(2)	0.024(2)	0.030(2)
C(34)	2i		0.1655(3)	−0.1035(3)	0.5479(2)	0.102(3)	0.128(4)	0.076(3)	0.030(3)	0.057(3)	0.049(3)
C(35)	2i		0.2013(2)	−0.0198(2)	0.3782(2)	0.047(2)	0.036(1)	0.057(2)	−0.000(1)	0.022(2)	0.016(1)
C(36)	2i		0.1264(2)	0.0141(2)	0.3983(2)	0.054(2)	0.042(2)	0.065(2)	0.007(1)	0.027(2)	0.020(2)
C(37)	2i		0.1514(3)	0.1091(2)	0.4382(3)	0.087(3)	0.046(2)	0.123(3)	0.015(2)	0.061(3)	0.021(2)
C(38)	2i		0.0806(3)	0.1484(2)	0.4614(2)	0.093(3)	0.065(2)	0.094(3)	0.030(2)	0.053(2)	0.029(2)
C(39)	2i		0.2767(2)	−0.1314(2)	0.3333(2)	0.037(2)	0.049(2)	0.049(2)	0.002(1)	0.018(1)	0.017(1)
C(40)	2i		0.2828(2)	−0.2230(2)	0.2975(2)	0.039(2)	0.050(2)	0.045(2)	0.005(1)	0.013(1)	0.017(1)
C(41)	2i		0.3726(2)	−0.2255(2)	0.2906(2)	0.043(2)	0.060(2)	0.054(2)	0.009(1)	0.020(2)	0.022(2)
C(42)	2i		0.3843(2)	−0.3129(2)	0.2520(2)	0.052(2)	0.072(2)	0.072(2)	0.014(2)	0.025(2)	0.016(2)
C(43)	2i		0.4023(2)	0.5076(2)	0.6171(2)	0.043(2)	0.043(2)	0.051(2)	0.013(1)	0.025(1)	0.018(1)
C(44)	2i		0.3693(2)	0.4410(2)	0.6370(2)	0.059(2)	0.053(2)	0.070(2)	0.018(2)	0.035(2)	0.030(2)
C(45)	2i		0.3986(2)	0.3583(2)	0.6078(2)	0.065(2)	0.057(2)	0.073(2)	0.029(2)	0.036(2)	0.034(2)
C(46)	2i		0.3594(3)	0.2891(2)	0.6231(2)	0.095(3)	0.053(2)	0.105(3)	0.027(2)	0.046(3)	0.037(2)
C(47)	2i		0.4113(2)	0.6539(2)	0.6219(2)	0.039(2)	0.039(1)	0.052(2)	0.005(1)	0.023(1)	0.017(1)
C(48)	2i		0.3942(2)	0.6273(2)	0.5418(2)	0.034(2)	0.049(2)	0.045(2)	0.002(1)	0.017(1)	0.017(1)
C(49)	2i		0.4252(2)	0.7008(2)	0.5280(2)	0.043(2)	0.056(2)	0.057(2)	0.006(1)	0.019(2)	0.030(2)
C(50)	2i		0.4160(2)	0.6764(2)	0.4503(2)	0.065(2)	0.094(3)	0.071(2)	−0.000(2)	0.034(2)	0.042(2)
C(51)	2i		0.2656(2)	0.5698(2)	0.5977(2)	0.039(2)	0.049(2)	0.060(2)	0.008(1)	0.025(2)	0.026(1)
C(52)	2i		0.2192(2)	0.6437(2)	0.6006(2)	0.045(2)	0.058(2)	0.066(2)	0.017(1)	0.031(2)	0.031(2)
C(53)	2i		0.1161(2)	0.6110(2)	0.5612(2)	0.045(2)	0.072(2)	0.074(2)	0.020(2)	0.032(2)	0.039(2)
C(54)	2i		0.0649(2)	0.6808(2)	0.5543(2)	0.060(2)	0.086(2)	0.089(3)	0.035(2)	0.040(2)	0.042(2)
C(55)	2i		0.3938(2)	0.6289(2)	0.7241(2)	0.061(2)	0.052(2)	0.050(2)	0.017(1)	0.035(2)	0.020(1)
C(56)	2i		0.4927(2)	0.6405(2)	0.7742(2)	0.066(2)	0.082(2)	0.048(2)	0.014(2)	0.021(2)	0.024(2)
C(57)	2i		0.5145(3)	0.6857(2)	0.8567(2)	0.099(3)	0.082(3)	0.053(2)	0.012(2)	0.030(2)	0.027(2)
C(58)	2i		0.5141(3)	0.7763(2)	0.8814(2)	0.124(4)	0.081(3)	0.062(2)	0.013(2)	0.036(2)	0.022(2)
C(59)	2i		0.3416(2)	0.1056(2)	0.8581(2)	0.052(2)	0.053(2)	0.055(2)	0.018(1)	0.026(2)	0.035(2)
C(60)	2i		0.2549(2)	0.0490(2)	0.7940(2)	0.058(2)	0.051(2)	0.057(2)	0.011(1)	0.025(2)	0.029(2)
C(61)	2i		0.2239(2)	−0.0224(2)	0.8109(2)	0.070(2)	0.058(2)	0.069(2)	0.013(2)	0.036(2)	0.035(2)
C(62)	2i		0.1404(3)	−0.0853(2)	0.7462(2)	0.106(3)	0.070(2)	0.088(3)	−0.015(2)	0.032(3)	0.035(2)
C(63)	2i		0.3141(2)	0.2484(2)	0.8606(2)	0.050(2)	0.049(2)	0.054(2)	0.020(1)	0.021(2)	0.029(1)
C(64)	2i		0.3086(2)	0.2874(2)	0.9349(2)	0.068(2)	0.064(2)	0.057(2)	0.028(2)	0.031(2)	0.033(2)
C(65)	2i		0.2395(3)	0.3450(2)	0.9289(2)	0.081(2)	0.066(2)	0.071(2)	0.039(2)	0.040(2)	0.032(2)
C(66)	2i		0.2306(3)	0.3885(3)	1.0011(2)	0.144(4)	0.088(3)	0.101(3)	0.066(3)	0.080(3)	0.051(3)
C(67)	2i		0.3792(2)	0.1667(2)	0.7792(2)	0.050(2)	0.047(2)	0.048(2)	0.011(1)	0.021(1)	0.026(1)
C(68)	2i		0.4361(2)	0.1039(2)	0.7574(2)	0.051(2)	0.046(2)	0.056(2)	0.013(1)	0.025(2)	0.025(1)
C(69)	2i		0.4215(2)	0.0837(2)	0.6777(2)	0.054(2)	0.057(2)	0.060(2)	0.016(1)	0.031(2)	0.032(2)
C(70)	2i		0.4744(2)	0.0195(2)	0.6510(2)	0.073(2)	0.056(2)	0.063(2)	0.020(2)	0.038(2)	0.030(2)
C(71)	2i		0.4712(2)	0.2227(2)	0.9179(2)	0.048(2)	0.056(2)	0.049(2)	0.015(1)	0.016(2)	0.026(2)
C(72)	2i		0.5198(2)	0.3057(2)	0.9285(2)	0.052(2)	0.059(2)	0.062(2)	0.010(2)	0.023(2)	0.028(2)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(73)	2i		0.6072(2)	0.3413(2)	1.0014(2)	0.053(2)	0.070(2)	0.065(2)	0.011(2)	0.025(2)	0.023(2)
C(74)	2i		0.6555(2)	0.4265(2)	1.0156(2)	0.058(2)	0.072(2)	0.096(3)	0.005(2)	0.028(2)	0.020(2)
O(3)	2i	0.50	0.4953(4)	0.0062(4)	0.0761(3)	0.090(5)	0.084(4)	0.069(4)	0.032(3)	0.029(3)	0.024(3)
C(75)	2i	0.50	0.382(2)	−0.007(1)	0.048(2)	0.19(3)	0.10(1)	0.18(3)	0.03(2)	0.15(3)	0.02(1)
C(76)	2i	0.50	0.3630(9)	−0.0874(7)	−0.0155(9)	0.089(9)	0.077(6)	0.13(1)	0.016(5)	0.046(8)	0.049(6)
O(4)	2i	0.50	0.4795(5)	−0.0679(6)	0.0270(5)	0.110(5)	0.153(7)	0.155(7)	0.076(5)	0.077(5)	0.109(6)
C(77)	2i	0.50	0.418(1)	−0.022(1)	0.057(1)	0.059(7)	0.067(8)	0.082(8)	−0.009(6)	0.033(6)	0.014(7)
C(78)	2i	0.50	0.338(1)	−0.045(2)	−0.002(1)	0.11(1)	0.35(5)	0.13(2)	0.06(2)	0.00(1)	0.13(3)

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