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Two New Dibenzobicyclic Penta- and Hexacoordinated Tin Compounds

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ABSTRACT: *Two new dibenzobicyclic penta- and hexacoordinated tin compounds (*1–2*) were prepared, derived from bis(2-hydroxy-3,5-di-tert-butylphenol)imine and dichloro-dimethylstannane and trichloro-n-butylstannane, respectively. Each compound is an example of two different coordination forms of the ligand. The crystalline complexes have been characterized by magnetic moment, infrared and mass spectrometry, elemental analysis, and X-ray diffraction studies. Compound* 1 *crystallized in the monoclinic system, space group $P2_1/n$; it contains a five-coordinated trigonal bipyramidal tin atom with methyl groups in equatorial positions and a planar dibenzobicyclic ring. It is a paramagnetic compound ($\mu_{eff} = 1.59\ \mu_B$) due to the radical dianion nature of the ligand. Compound* 2 *crystallized also in the monoclinic system, space group $P2_1/c$; it contains a six-coordinated tin atom with an octahedral geometry; the dibenzobicyclic ring is planar, and the chlorine atoms are in trans positions. It is diamagnetic with a delocalized monoanion structure of the ligand.* © 1998 John Wiley & Sons, Inc. Heteroatom Chem 9:321–326, 1998

INTRODUCTION

In previous reports, we have described the synthesis and structures of pentacoordinated phosphorus heterocycles derived from diphenolamine [1–4]. These dibenzophosphoranes of type **A**, have a trigonal bipyramidal geometry in a rigid planar tetracyclic framework. They are more stable than their aliphatic analogs [5,6], Figure 1. Also, we have reported on the preparation of tetracoordinated boron compounds of type **B**, derived from dibenzobicyclic structures [7]. And, we have employed the bis[4,6-di-*tert*-butyl-iminophenolate]zinc complex [8–10], of type **C**, in order to prepare the phosphorus heterocycles, **D**. It was reported that the 4,6-di-*tert*-butyl-iminophenolate ligand may coordinate to the metals in either the monoanionic form, **E**, or in the radical dianion form, **F**, both with an important charge delocalization [11,12].

A reaction was reported recently of tin(II)chloride with the ligand LH_3 that afforded the hexacoordinated tin compound of type **C** bonded to two identical ligands with a very distorted octahedral symmetry due to the constraints produced by the ligand geometry [13] (Figure 1).

The structure of this paramagnetic compound is a delocalized frame; one localized form can be de-

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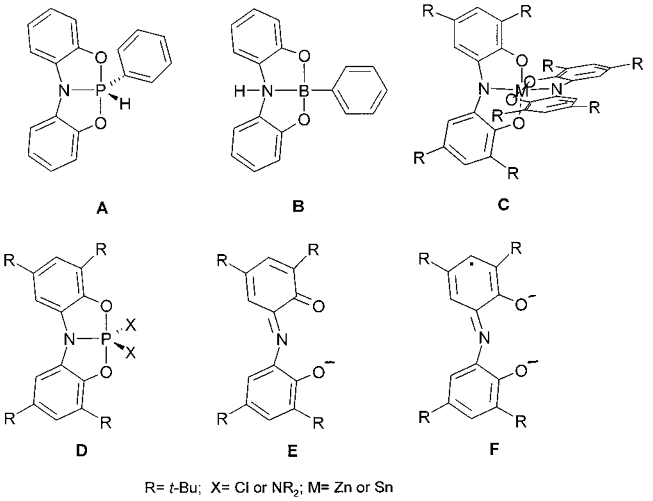
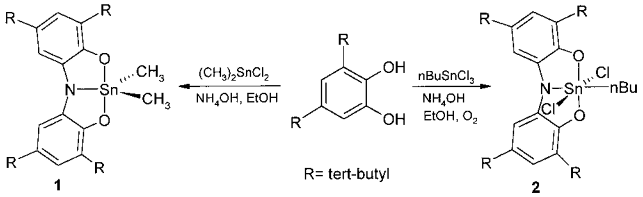


R= *t*-Bu; X= Cl or NR$_2$; M= Zn or Sn

FIGURE 1



R= tert-butyl

SCHEME 1 Preparation of compounds **1–2**.

scribed as one ligand being a diradical monoanion and the other a monoanion [13]. In order to avoid the introduction of two ligands, reactions with mono-and dialkyl tin compounds were performed with the aim to introduce only one 4,6-di-*tert*-butyl-iminophenolate ligand and to find out which coordination a tin center may achieve and which behavior of the ligand can be induced, depending on the tin substitution [11–13].

Therefore, the dibenzobicyclic penta- and hexacoordinated tin compounds (**1**–**2**) were prepared by reactions of 3,5-di-*tert*-butyl-1,2-catechol, ammonia, and dichloro-dimethylstannane and trichloro-*n*-butylstannane, respectively, crystalline products being obtained that were studied in different ways (Scheme 1).

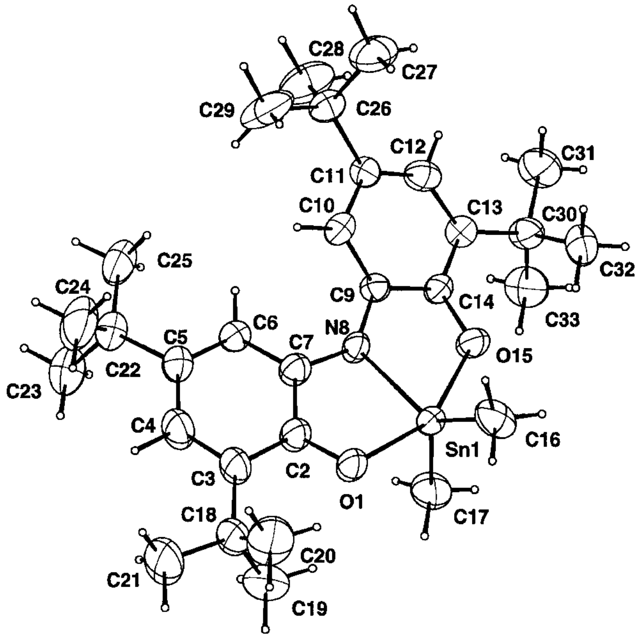


FIGURE 2 X-ray diffraction structure of compound **1**.

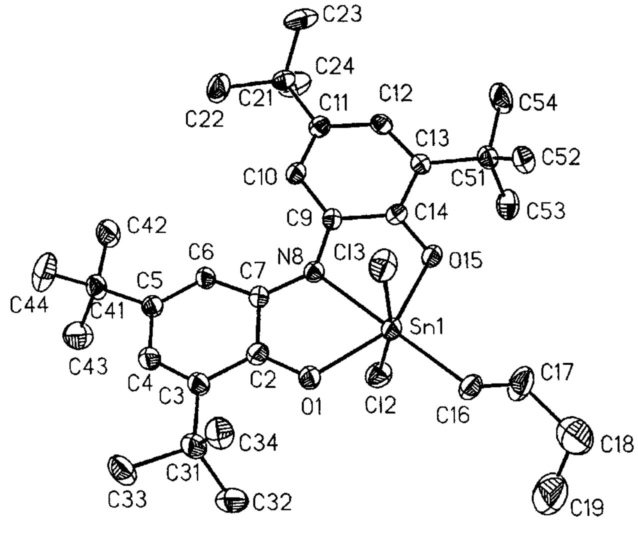


FIGURE 3 X-ray diffraction structure of compound **2**.

MOLECULAR STRUCTURES

Only one ligand had been introduced in **1** and **2**, as was deduced from the mass spectra of both compounds. The ligand may chelate the tin atom in different charged forms [11–13]. The structure of the ligand can be deduced from the magnetic moment that was $\mu_{\text{eff}} = 1.59\mu_{\text{B}}$ for **1** and corresponds to an electron. Compound **2** was diamagnetic $\mu_{\text{eff}} = 0\mu_{\text{B}}$ [11–13]; in spite of that, we were unable to record good NMR spectra. An explanation could be that the solution was contaminated with some free radical quinone or that the ligand has a very weak radical character. Both compounds supplied single crystals suitable for X-ray diffraction studies (Figures 2 and 3, and Tables 1–7).

The molecular structure of compound **1** was shown to be a tetracyclic compound with the tin

TABLE 1 Bond Lengths (Å) for Compound **1**

Sn1–O1	2.111(4)	Sn1–O15	2.115(4)
Sn1–N8	2.146(4)	Sn1–C16	2.099(6)
Sn1–C17	2.083(6)	O1–C2	1.310(7)
O15–C14	1.310(6)	N8–C7	1.365(8)
N8–C9	1.364(7)	C2–C3	1.417(9)
C2–C7	1.424(8)	C5–C6	1.350(9)
C3–C4	1.395(9)	C9–C14	1.444(8)
C4–C5	1.420(9)	C11–C12	1.365(9)
C6–C7	1.413(8)	C13–C14	1.423(8)
C9–C10	1.410(7)	C12–C13	1.383(8)
C10–C11	1.409(8)		

TABLE 2 Bond Lengths (Å) for Compound **2**

Sn1–O15	2.090(4)	Sn1–O1	2.116(4)
Sn1–C16	2.132(6)	Sn1–N8	2.221(5)
Sn1–C13	2.436(3)	Sn1–C12	2.455(3)
O1–C2	1.289(7)	C2–C3	1.428(8)
C2–C7	1.477(8)	C3–C4	1.354(8)
C3–C31	1.544(8)	C4–C5	1.439(8)
C5–C6	1.355(8)	C7–N8	1.336(7)
C6–C7	1.419(8)	C9–C10	1.403(7)
N8–C9	1.365(7)	C10–C11	1.358(8)
C9–C14	1.459(7)	C13–C14	1.429(8)
C11–C12	1.429(8)	C14–O15	1.293(6)
C12–C13	1.370(8)	C17–C18	1.60(2)
C16–C17	1.485(10)	C18–C19	1.36(2)

TABLE 3 Bond Angles (°) for Compound **1**

O1-Sn1-O15	151.6(1)	O1-Sn1-N8	75.6(2)
O15-Sn1-N8	76.2(2)	O1-Sn1-C16	96.6(2)
O15-Sn1-C16	98.1(2)	N8-Sn1-C16	116.4(3)
O1-Sn1-C17	94.0(3)	O15-Sn1-C17	96.8(2)
N8-Sn1-C17	117.6(2)	C16-Sn1-C17	125.9(3)
Sn1-O1-C2	114.4(4)	Sn1-O15-C14	115.3(3)
Sn1-N8-C7	114.4(4)	Sn1-N8-C9	114.8(3)
C7-N8-C9	130.5(4)	N8-C7-C6	126.4(5)
N8-C7-C2	135.9(5)	N8-C9-C14	113.6(5)
N8-C9-C10	125.9(5)		

TABLE 4 Bond Angles (°) for Compound **2**

O15-Sn1-O1	150.29(14)	O15-Sn1-C16	103.2(2)
O1-Sn1-Cl6	106.5(2)	O15-Sn1-N8	75.4(2)
O15-Sn1-N8	74.9(2)	C16-Sn1-N8	177.2(2)
O15-Sn1-Cl3	90.5(2)	O1-Sn1-Cl13	87.14(14)
C16-Sn1-Cl3	97.9(2)	N8-Sn1-Cl3	84.59(14)
O15-Sn1-Cl2	88.2(2)	O1-Sn1-Cl2	87.66(14)
C16-Sn1-Cl2	94.8(2)	N8-Sn1-Cl2	82.75(14)
Cl3-Sn1-Cl2	167.19(6)	C2-O1-Sn1	116.8(4)
O1-C2-C3	121.4(5)	O1-C2-C7	119.7(5)
N8-C7-C2	112.7(5)	N8-C7-C6	128.2(5)
C7-N8-C9	131.0(5)	C6-C7-C2	119.1(5)
C9-N8-Sn1	114.1(3)	C7-N8-Sn1	114.9(4)
N8-C9-C14	112.3(5)	N8-C9-C10	128.1(5)
O15-C14-C13	120.2(5)	O15-C14-C9	120.5(5)
C17-C16-Sn1	114.1(5)	C14-O15-Sn1	117.0(3)

atom in a distorted trigonal bipyramidal geometry, the oxygen atoms being in apical positions and nitrogen and methyl groups in equatorial positions. The angle C-Sn-C is slightly opened 125.7(4)°. The two angles C-Sn-N do not differ significantly [N-Sn-C34 116.8(3) and N-Sn-C33 117.4(3)°].

In compound **2**, the tin atom is hexacoordinated and is present in a distorted octahedral array. These characteristics can be seen from the O1-Sn1-O15 bond angle of 150.3(1)° and the C12-Sn1-C13 bond angle of 167.19(6)°, while the N8-Sn1-C16 bond angle is 177.2(2)°. The chlorine atoms in **2** are trans. The angle Cl-Sn-Cl is 167.19(6)°, the Sn–O bonds [2.090(4) and 2.116 (4) Å] have similar values to those reported [13], and Sn–N is slightly longer in **1**, 2.221(5), than in **2**, 2.146(4) Å.

In both compounds, the C–O and C–N bond lengths are short, (C–O bonds are C6–O30, 1.309(7) and C8–O31, 1.310(6) Å for **1** and C14–O15, 1.293(6) and C2–O1, 1.289(7) Å for **2**, and C–N bonds are C9–N8, 1.364(8) and C7–N8, 1.365(8) for **1** and C9–N8, 1.365(7) and C7–N8, 1.336(7) for **2**. Therefore, there is some asymmetry in the C–N bond lengths of **2** compared to **1**. Moreover, we note that the C–C bond lengths of the benzene rings show an alternation [C–C bond lengths ranging from 1.383(8) to 1.444(8) for **1** and from 1.354(8) to 1.477(8) for **2**], indicating a quinone type of structure, where the bonds C3–C4, C5–C6 as well as C10–C11 and C12–C13 are short, while C2–C3, C4–C5, C6–C7, C9–C10, C11–C12, and C13–C14 are clearly longer. These structures clearly contrast with X-ray diffraction data of the phosphorus compounds in which the ligand is neutral and perfectly aromatic [2–4] (C–N, 1.409 and C–O, 1.375 Å, and all C–C bonds in the rings are around 1.40 Å).

In both compounds, the three bonds of the nitrogen atoms are in a plane, and the tetracyclic framework is slightly folded. The angle O-Sn-O in **1** is 150.3(1)° and 150.29(14)° in **2**. This angle was related to a weak interaction between the metal acceptor and the nitrogen donor [12]. Compound **1** has an interplanar angle of 7° between the two five-membered rings. The same folding was found in the tin hexacoordinated compound bearing two dibenzobicyclic ligands reported by McGarvey et al. [13]. This contrasts with the structure of the analogous phosphorus heterocycle **A** that has an angle O-P-O near 165.9(1)° [2–4] and the angle O-B-O 110.3(3)° in the boron compound **B** [7], Figure 1.

We have tried to find a correlation, as reported

TABLE 5 Crystal Data of **1** and **2**

	$C_{30}H_{46}ON_3SN$ (1)	$C_{32}H_{49}Cl_2O_6NSn$ (2)[a]
Fw (g/mol)	523.34	669.31
Space group	$P2_1$-*n*	$P2_1/c$
a(Å)	6.4613(7)	13.196(1)
b(Å)	46.853(4)	13.736(2)
c(Å)	10.3199(8)	20.028(2)
α(°)	90.0	90.0
β(°)	91.232(8)	109.20(2)
γ(°)	90.0	90.0
V(Å^3)	3123.4(5)	3428.4(7)
Z	4	4
Crystal dimensions	0.2 × 0.2 × 0.3	0.2 × 0.2 × 0.18
Crystal habit	dark violet fragment	dark green fragment
Diffractometer	CAD4-Enraf-Nonius	Siemens P4 CCD Detector
Radiation	Mo$K\alpha$(λ = 0.71069 Å)	Mo$K\alpha$(λ = 0.71073 Å)
Linear abs coeff	8.36 cm^{-1}	11.872 cm^{-1}
ρ (calc) g cm^{-3}	1.11	1.297
Scan type	$\omega/2\theta$	φ^b
θ limits (°)	2.06–25	1.63–28.95
Temperature of measurement	room temperature	243 K
Octants collected	−7, 0; −55, 0; −12, 12	
No. of data collected	5356	19,143
No. of unique data collected	5281	6,443
No. of unique data used	3787 $(Fo)^2 > 3\sigma(Fo)^2$	3,742 $(F > 4\sigma(F))$
R(int)	0.08	0.0882
$R = \Sigma(\|\|Fo\| - \|\|Fc\|)/\Sigma\|Fo\|$	0.035	0.0549
$Rw = [\Sigma w(\|Fo\| - \|Fc\|)^2/\Sigma wFo^2]^{1/2}$	0.043 w = 1	0.1192
Goodness of fit s	2.94	1.097
No. of variables	446	355
$\Delta\rho_{min}$ (e/Å^3)	−0.49	−0.651
$\Delta\rho_{max}$ (e/Å^3)	0.38	1.272

[a] Background measurement: stationary crystal and stationary counter at the beginning and end of scan, each for 25% of total scan time.
[b] 1200 frames, 10 s exposure, 0.3° in φ.

in the literature [11–13], between the bond lengths and the specific nature of the ligand, but we could not find it. Both structures have a quinone-like structure that is more important in **2**. Based on the magnetic properties, and since no hydrogen atoms could be localized at either the oxygen or the nitrogen atoms, it follows from the stoichiometries and the structures of **1** and **2** that the ligand in **1** must be a radical dianion. Whereas for **2**, a monoanion structure was found. Thus, it follows that these coordinations of the ligand are controlled by the tin center substitution.

EXPERIMENTAL

All chemicals were reagent grade and were used without further purification. Solvents were purified by the usual methods. Melting points were obtained with a Gallenkamp apparatus and are uncorrected. IR (KBr disc) spectra were determined on a Perkin Elmer 16FPC IR spectrometer. Mass spectra were recorded on a Hewlett-Packard HP 5989[a] instrument. Elemental analyses were performed at the Facultad de Química of the National University of Mexico.

Compound 1

In a 50 mL flask, an aqueous solution of ammonia (2N, 3.25 mL), a solution of 1.11 g of 3,5-di-*tert*-butylcatechol (5 mmol in 20 mL of 95% ethanol), and a solution of 0.55 g of dimethyltin dichloride (2.5 mmol in 10 mL of 95% ethanol) were placed in the presence of air; after some minutes, the mixture became violet. A violet crystalline solid was obtained after 24 hours. The suspension was filtered, and the insoluble product was washed with water (10 mL) and recrystallized from hexane/ethanol (0.22 g, 78%), mp 178–179°C. IR (KBr, ν_{max}/cm^{-1}) 2962, 1472, 1444, 1416, 1378, 1360, 1316, 1228, 1180, 1112, 1044, 1024, 1008. MS (EI, 70 ev) *m*/*e*, M^+ [564.25 (3), 566.25 (2), 567.40 (3), 568.25 (38), 569.25 (37), 570.25 (76), 571.25 (49), 572.25 (100), 574.25 (18), 576.25 (16)], 557.25 (10), 573.25 (31), 576.25 (16), 577.25 (6). Anal. calcd for $C_{30}H_{46}NO_2Sn$: C, 63.05; H, 8.05; N, 2.45. Found: C, 62.36; H, 8.10; N, 2.53.

Compound 2

In a 50 mL flask, an aqueous solution of ammonia (2N, 5.0 mL), a solution of 1.11 g of 3,5-di-*tert*-bu-

TABLE 6 Fractional Atomic Coordinates of Compound **1**

Atom	*x/a*	*y/b*	*z/c*	*U* (*iso*)
SN (1)	0.68679(6)	0.163612(9)	0.03280(4)	0.0389
O (1)	0.7556(7)	0.12114(9)	0.0866(4)	0.0520
O (15)	0.5557(6)	0.19401(9)	−0.0963(4)	0.0485
N (8)	0.5342(7)	0.1381(1)	−0.1131(4)	0.0400
C (2)	0.7193(9)	0.1023(1)	−0.0047(6)	0.0477
C (3)	0.802(1)	0.0743(1)	0.0025(6)	0.0538
C (5)	0.625(1)	0.0631(1)	−0.2061(7)	0.0549
C (7)	0.5935(9)	0.1101(1)	−0.1138(5)	0.0465
C (9)	0.4090(8)	0.1530(1)	−0.1971(5)	0.0389
C (11)	0.1524(8)	0.1595(1)	−0.3692(5)	0.0437
C (13)	0.3262(8)	0.2012(1)	−0.2786(5)	0.0425
C (14)	0.4332(8)	0.1835(1)	−0.1872(5)	0.0388
C (18)	0.944(1)	0.0651(2)	0.1165(8)	0.0666
C (22)	0.587(1)	0.0404(2)	−0.3115(8)	0.0680
C (26)	−0.0107(9)	0.1467(2)	−0.4656(6)	0.0514
C (30)	0.361(1)	0.2336(1)	−0.2744(6)	0.0501
C (4)	0.750(1)	0.0553(2)	−0.0969(7)	0.0627
C (6)	0.5513(9)	0.0900(1)	−0.2130(6)	0.0637
C (10)	0.2616(8)	0.1411(1)	−0.2842(5)	0.0485
C (12)	0.1916(9)	0.1881(1)	−0.3659(5)	0.0499
C (16)	0.513(1)	0.1738(2)	0.1962(6)	0.0438
C (17)	0.9952(9)	0.1744(2)	0.0044(8)	0.0444
C (19)	1.135(1)	0.0855(2)	0.121(1)	0.0481
C (20)	0.826(2)	0.0672(2)	0.2427(8)	0.0501
C (21)	1.022(2)	0.0347(2)	0.104(1)	0.0629
C (23)	0.788(1)	0.0297(2)	−0.3668(9)	0.0689
C (24)	0.469(2)	0.0154(2)	−0.253(1)	0.0689
C (25)	0.454(2)	0.0525(2)	−0.4237(9)	0.0689
C (27)	−0.219(1)	0.1617(2)	−0.4430(8)	0.0652
C (28)	0.053(1)	0.1523(2)	−0.6018(6)	0.0644
C (29)	−0.046(1)	0.1151(2)	−0.4502(9)	0.0644
C (31)	0.242(1)	0.2487(2)	−0.3853(8)	0.0644
C (32)	0.281(1)	0.2455(2)	−0.1469(7)	0.0973
C (33)	0.591(1)	0.2409(2)	−0.2884(8)	0.1061

tylcatechol (5 mmol in 20 mL of 95% ethanol), and a solution of 0.71 g of *n*-butyl tin trichloride (2.5 mmol in 10 mL of 95% ethanol) were mixed and stirred. A slight oxygen stream was bubbled in the reaction mixture; it became green after some minutes. A dark green crystalline solid was obtained after 24 hours, which was removed by filtration, and this product was washed with water (10 mL) and recrystallized from ethanol/dimethyl chloride (0.25 g, 15%), mp 138–139°C. IR (KBr, ν_{max}/cm^{-1}) 3108, 2868, 1500, 1458, 1420, 1364, 1300, 1270, 1232, 1200, 1092, 1038, 1020, 998, 900. MS (EI, 70 ev) *m/e*, M$^+$ [665.20 (1), 666.20 (1), 667.20 (3), 668.20 (2), 669.20 (5)], 670.20 (2), 671.25 (3), 672.20 (1), 673.20 (1), 422.30 (17), 423.35 (10), 464.45 (70), 465.40 (24), 479.45 (100), 480.45 (36), 597.30 (11), 599.30 (13), 610.20 (19), 611.20 (14), 612.20 (31), 613.20 (13), 614.25 (17), Anal. calcd for $C_{32}H_{49}Cl_2NO_2Sn$: C, 57.45; H, 7.33; N, 2.09. Found: C, 57.23; H, 7.51; N, 1.80.

TABLE 7 Compound **2** Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (A^2 $\times$ 10^3)

	x	*y*	*z*	*U*(*eq*)
Sn(1)	2903.8(3)	6310.6(3)	2964.5(2)	48.4(2)
O(1)	3698(3)	5019(3)	3431(2)	52.6(10)
Cl(2)	3826.6(14)	6168.4(13)	2085.8(10)	69.9(5)
C(2)	3312(4)	4211(4)	3124(3)	49.0(14)
Cl(3)	1788(2)	6120.1(13)	3712.8(11)	75.6(5)
C(3)	3799(4)	3298(4)	3386(3)	49.1(14)
C(4)	3394(5)	2497(4)	2995(3)	51.6(15)
C(5)	2511(4)	2490(4)	2341(3)	45.2(13)
C(6)	2016(4)	3347(4)	2100(3)	47.2(14)
C(7)	2350(4)	4223(4)	2484(3)	45.6(13)
N(8)	1914(3)	5108(3)	2338(2)	42.5(11)
C(9)	968(4)	5406(4)	1856(3)	43.7(13)
C(10)	118(4)	4828(4)	1439(3)	46.9(14)
C(11)	−761(4)	5223(4)	953(3)	46.0(13)
C(12)	−805(4)	6260(4)	903(3)	47.1(14)
C(13)	−47(4)	6893(4)	1306(3)	46.9(14)
C(14)	885(4)	6464(4)	1801(3)	45.2(14)
O(15)	1646(3)	7009(3)	2195(2)	52.2(10)
C(16)	3866(5)	7490(4)	3520(3)	57(2)
C(17)	3877(8)	8347(6)	3070(5)	103(3)
C(18)	4731(10)	9144(9)	3491(10)	165(6)
C(19)	5729(12)	8769(10)	3599(10)	169(6)
C(21)	−1670(4)	4625(4)	471(3)	54(2)
C(22)	−1514(7)	3529(5)	617(6)	107(3)
C(23)	−2731(5)	4924(7)	541(4)	86(2)
C(24)	−1707(6)	4798(7)	−284(4)	85(2)
C(31)	4744(5)	3266(5)	4088(4)	59(2)
C(32)	5704(5)	3814(6)	4003(4)	80(2)
C(33)	5099(6)	2206(5)	4299(4)	75(2)
C(34)	4420(6)	3708(5)	4687(3)	67(2)
C(41)	2171(5)	1535(4)	1949(3)	56(2)
C(42)	1185(6)	1659(5)	1295(4)	81(2)
C(43)	3084(7)	1139(6)	1718(5)	96(3)
C(44)	1879(9)	814(6)	2439(5)	105(3)
C(51)	−163(5)	8010(4)	1240(3)	54(2)
C(52)	−55(6)	8469(5)	1957(4)	69(2)
C(53)	690(7)	8418(5)	961(4)	73(2)
C(54)	−1279(7)	8288(5)	728(5)	89(3)

$U_{(eq)}$ is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

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