

## Synthesis and Crystal Structure of Di-*n*-butyltin(IV) Complex with 2-Oxo-propionic Acid (4-Pyridinecarbonyl) Hydrazone

LIU, Hong-Wen<sup>a</sup>(刘宏文) LU, Wen-Guan<sup>\*a</sup>(卢文贯) TAO, Jia-Xun<sup>b</sup>(陶家洵)

<sup>a</sup> Department of Chemistry, Shaoguan University, Shaoguan, Guangdong 512005, China

<sup>b</sup> Department of Chemistry, Tsinghua University, Beijing 100084, China

The novel complex di-*n*-butyltin(IV) 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone, (*n*-C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>Sn[O<sub>2</sub>CC(CH<sub>3</sub>)=N—N=C(—O)C<sub>5</sub>H<sub>4</sub>N-4] · (H<sub>2</sub>O) has been synthesized and its structure has been determined by X-ray diffraction analysis. The complex crystallizes in orthorhombic system with space group *Pca*2<sub>1</sub>. Crystal data: *a*=2.7540(9) nm, *b*=0.9676(3) nm, *c*=1.5750(5) nm, *V*=4.197(2) nm<sup>3</sup>, *D<sub>c</sub>*=1.444 g/cm<sup>3</sup>, *Z*=8, *μ*=1.241 mm<sup>-1</sup>, *F*(000)=1856, *R*<sub>1</sub>=0.0462 and *wR*<sub>2</sub>=0.1001. In the crystals of the title complex, the tin atom is in six-coordination with a distorted octahedral geometry, three oxygen atoms [O(1), O(3) and O(4)] and one nitrogen atom N(1) forming the equatorial plane and C(10)—Sn(1)—C(14) being the axis. Two molecules form a dimer with weak interactions of Sn···O bonding and hydrogen bonds.

**Keywords** diorganotin(IV) carboxylate, tridentate hydrazone, crystal structure

### Introduction

The studies of diorganotin(IV) complexes are of current interest owing to their wide range of applications such as biocides and homogeneous catalysts in industry.<sup>1</sup> In recent years there have been more and more reports on the synthesis, antitumour activities and structural elucidation of various diorganotin(IV) derivatives of carboxylic acid.<sup>2-5</sup> In particular, people take considerable interest in structural studies of diorganotin(IV) complexes of carboxylic acid, because there are many possible bonding interactions between the oxygen atom of carboxyl group and tin atom. Studies on diorganotin(IV) complexes of carboxylic acid having carboxylate ligands with additional donor atom, such as nitrogen, revealed new structural types which may lead to complexes with different activities.<sup>5-10</sup> The ligand 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone C<sub>5</sub>H<sub>4</sub>N-4-C(=O)NH—N=C(CH<sub>3</sub>)CO<sub>2</sub>H which functions as a monobasic tridentate chelate to bind transition metal halides was studied.<sup>11,12</sup> This ligand was not previously used to yield diorganotin(IV) derivatives. In this paper, we report the condensation of this ligand with di-*n*-butyltin(IV) oxide yielding the di-*n*-butyltin(IV) 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone complex (*n*-C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>Sn[O<sub>2</sub>CC(CH<sub>3</sub>)=N—N=C(—O)C<sub>5</sub>H<sub>4</sub>N-4] · (H<sub>2</sub>O) and its crystal structure analysis.

### Experimental

#### Materials and instruments

All reagents were of analytical grade and used with-

out further purification. The ligand 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone C<sub>5</sub>H<sub>4</sub>N-4-C(=O)NH—N=C(CH<sub>3</sub>)CO<sub>2</sub>H was synthesized according to the reported method.<sup>12</sup> Elemental analyses (C, H and N) were performed on a Vario EL CHNS-O elemental analyzer. Infrared spectra were recorded as KBr discs on a Nicolet AVATAR 360 FT-IR spectrophotometer. The <sup>1</sup>H NMR, <sup>13</sup>C NMR and <sup>119</sup>Sn NMR spectra were recorded on a Varian UNITY-200 instrument, and the chemical shifts were measured relative to internal TMS for <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra and to external (*n*-C<sub>4</sub>H<sub>9</sub>)<sub>4</sub>Sn (δ -6.60) for <sup>119</sup>Sn NMR spectra, respectively. The melting points were measured with a digital melting point apparatus.

#### Synthesis of the title complex

The ligand 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone C<sub>5</sub>H<sub>4</sub>N-4-C(=O)NH—N=C(CH<sub>3</sub>)CO<sub>2</sub>H (1.0 mmol) and di-*n*-butyltin(IV) oxide (*n*-C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>SnO (1.1 mmol) were mixed with a solvent mixture of 30 mL of benzene and 10 mL of ethanol in a round-bottomed flask. The mixture was refluxed for 4 h, and the water produced during the reaction was removed azeotropically using a Dean-Stark apparatus. After reflux, the reaction mixture was filtrated when it was still hot. The slow evaporation of solvent from the filtrate gave orange yellow-crystals, which were recrystallized in a mixed solvent of 30 mL of benzene and 10 mL of ethanol to give orange-yellow crystals suitable for X-ray structure analysis. Yield 85%, m.p. 130—132 °C (dec.); <sup>1</sup>H NMR [(CD<sub>3</sub>)<sub>2</sub>SO, 200 MHz ] δ: 2.40 (s, 3H, CH<sub>3</sub>), 3.45 (s, 2H, H<sub>2</sub>O), 7.98 [d, *J*=5.80 Hz, 2H, pyridine (3,

\* E-mail: lwg@sgu.edu.cn

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5)], 8.75 [d, 2H,  $J=5.80$  Hz, pyridine (2, 6)],  $n\text{-C}_4\text{H}_9$ : 1.15—1.38 [m, 8H,  $\text{Sn}(\text{CH}_2)_2$  ( $\alpha$ ,  $\beta$ )], 1.41—1.52 [m, 4H,  $\text{CH}_2$  ( $\gamma$ )], 0.76 (t,  $J=7.24$  Hz, 6H,  $\text{CH}_3$ );  $^{13}\text{C}$  { $^1\text{H}$ } NMR  $\delta$ : 141.77 [pyridine (4)], 121.91 [pyridine (3, 5)], 150.23 [pyridine (2, 6)], 154.85 (N=CCH<sub>3</sub>), 164.95 (N=CO), 171.29 (CO<sub>2</sub>),  $n\text{-C}_4\text{H}_9$ : 25.76, 26.71, 28.83 [ $\text{Sn}(\text{CH}_2)_3$  ( $\alpha$ ,  $\beta$ ,  $\gamma$ )], 13.39 (CH<sub>3</sub>C=N), 13.53 (CH<sub>3</sub> in  $n\text{-C}_4\text{H}_9$ );  $^{119}\text{Sn}$  { $^1\text{H}$ } NMR  $\delta$ : 466.44; IR (KBr)  $\nu$ : 3381 (m, H<sub>2</sub>O), 2953—2853 (s, C—H), 1613, 1341 (s, CO<sub>2</sub>), 1636 (s, C=N), 1596 (s, C=N—N=C), 1202 (s, C—O), 687 (m, Sn—O), 485 (w, Sn—N)  $\text{cm}^{-1}$ . Anal. calcd for  $\text{C}_{17}\text{H}_{27}\text{N}_3\text{O}_4\text{Sn}$ : C 43.24, H 6.08, N 9.46; found C 43.21, H 6.09, N 9.33.

### Crystal structure determination

An orange-yellow crystal fragment of dimensions 0.34 mm  $\times$  0.15 mm  $\times$  0.08 mm was mounted on a Bruker SMART 1000 CCD diffractometer. The diffraction data were measured at room temperature (293 K) using graphite monochromated Mo K $\alpha$  ( $\lambda=0.071073$  nm) radiation. A total of 20584 independent reflections were collected by  $2\theta$ - $\omega$  scan technique in the range of  $1.48^\circ \leq \theta \leq 25.03^\circ$ , and 6713 reflections were independent ( $R_{\text{int}}=0.0665$ ), of which 3666 reflections were observed [ $I > 2\sigma(I)$ ]. The semi-empirical absorption correction was applied to the data by using SADABS program with  $T_{\text{max}}=0.9072$  and  $T_{\text{min}}=0.6776$ . The structure was solved by direct method and refined by using full-matrix least-squares method on  $F^2$  with anisotropic displacement parameters for all non-hydrogen atoms. The positions of hydrogen atoms were calculated by using idealized geometry except that those of coordination water were obtained from successive Fourier syntheses. All calculations were performed using SHELXTL-97.<sup>13</sup> The crystal and refinement data for the title complex are listed in Table 1.

### Results and discussion

The X-ray structure of the title complex is shown in Figure 1, the packing of the molecule in unit cell is shown in Figure 2. The atomic coordinates and equivalent isotropic displacement parameters are listed in Table 2 and the selected bond lengths and bond angles are shown in Table 3.

The condensation of ligand 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone  $\text{C}_5\text{H}_4\text{N}-4\text{-C}(=\text{O})\text{NH}-\text{N}=\text{C}(\text{CH}_3)\text{CO}_2\text{H}$  with di- $n$ -butyltin(IV) oxide ( $n\text{-C}_4\text{H}_9$ )<sub>2</sub>SnO yields an O, N, O-chelated title complex di- $n$ -butyltin(IV) 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone complex ( $n\text{-C}_4\text{H}_9$ )<sub>2</sub>Sn[O<sub>2</sub>CC(CH<sub>3</sub>)=N—N=C(—O)C<sub>5</sub>H<sub>4</sub>N-4]  $\cdot$  (H<sub>2</sub>O). In this complex, the Sn atom exists in a distorted octahedral coordination environment in which one water molecule, one tridentate 2-oxo-propionic acid (4-pyridinecarbonyl) hydrazone ligand, and two *trans*  $n$ -butyl groups coordinate to each Sn center. The atoms O(1), O(3), N(1) and O(4) are coplanar within  $\pm 0.00050$  nm, which form the equatorial plane. Furthermore, the angle of the axial C(10)—Sn(1)—C(14) is  $164.9(5)^\circ$ , which deviates from

**Table 1** Crystal data and structure refinement for the title complex

Empirical formula	$\text{C}_{17}\text{H}_{27}\text{N}_3\text{O}_4\text{Sn}$
Formula weight	456.11
Wavelength/nm	0.071073
Crystal system	Orthorhombic
Space group	$Pca2_1$
$a/\text{nm}$	2.7540(9)
$b/\text{nm}$	0.9676(3)
$c/\text{nm}$	1.5750(5)
$V/\text{nm}^3, Z$	4.197(2), 8
$D_c/(\text{g} \cdot \text{cm}^{-3})$	1.444
Crystal size/ $\text{mm}^3$	0.34 $\times$ 0.15 $\times$ 0.08
$\theta$ range for data collection	$1.48^\circ$ to $25.03^\circ$
$F(000)$	1856
Absorption coefficient $\mu(\text{Mo K}\alpha)/\text{mm}^{-1}$	1.241
Limiting indices	$-27 \leq h \leq 32, -11 \leq k \leq 10,$ $-18 \leq l \leq 16$
Reflections collected	20584
Independent reflections	6713 ( $R_{\text{int}}=0.0665$ )
Reflections with $I > 2\sigma(I)$	3666
Data/restraints/parameters	6713/95/467
Goodness-of-fit on $F^2$	0.890
Refinement method	full-matrix least-squares on $F^2$
Final $R$ indices [ $I > 2\sigma(I)$ ]	$R_1=0.0462, wR_2=0.1001$
$R$ indices (all data)	$R_1=0.0989, wR_2=0.1272$
Largest diff. peak and hole/ ( $\text{e} \cdot \text{nm}^{-3}$ )	1032 and $-559$

the linear angle of  $180^\circ$ . These data indicate that the tin atom of this complex is in a distorted octahedral configuration. The O(1) atom of the carboxylate residue also binds the other tin atom, Sn(2), generating a Sn<sub>2</sub>O<sub>2</sub> four-membered ring. The distances of Sn(1)—O(5) (0.2621 nm) and Sn(2)—O(1) (0.2640 nm) are relatively longer than those of Sn(1)—O(1) and Sn(2)—O(5), but are comparable with those found in related seven-coordinate diorganotin systems.<sup>5,9</sup> Thereby the structure of the title complex can be described as a dimer with weak interactions of Sn $\cdots$ O bonding, and the coordination geometry of tin can be also described as a *trans*-C<sub>2</sub>SnO<sub>4</sub>N pentagonal bipyramid with the two  $n$ -butyl groups occupying *trans* positions [C(10)—Sn(1)—C(14)  $164.9(5)^\circ$  and C(27)—Sn(2)—C(31)  $163.8(6)^\circ$ ]. The bond distances of Sn(1)—O(4) and Sn(2)—O(8) are 0.2367(9) nm and 0.2363(9) nm, respectively, which are relatively longer than those in the analogous,<sup>5,9</sup> due to the formation of intradimeric hydrogen bonds, O(4) $\cdots$ O(6) (0.2668 nm) and O(8) $\cdots$ O(2) (0.2721 nm),

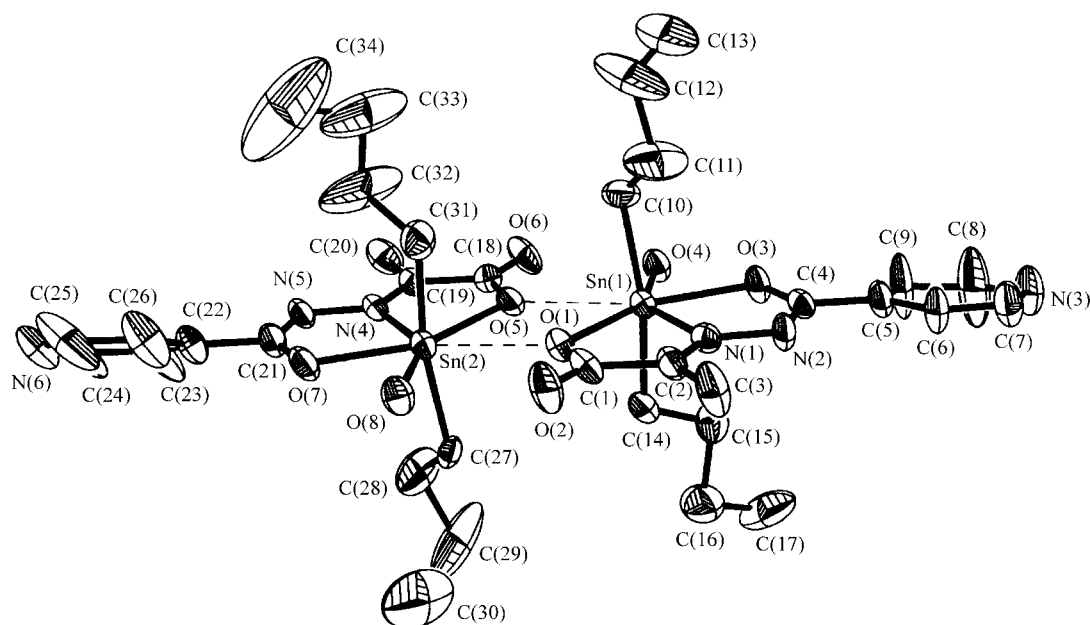


Figure 1 Molecular structure of the title complex.

Table 2 Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{nm}^2 \times 10^5$ ) for the title complex

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^a$	Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^a$
Sn(1)	-585(1)	6927(1)	6257(1)	49(1)	Sn(2)	7955(1)	8147(1)	6208(1)	52(1)
N(1)	-499(3)	5915(10)	7556(6)	52(3)	N(4)	7885(3)	9191(9)	4922(7)	51(3)
N(2)	-47(3)	5372(10)	7772(7)	62(3)	N(5)	7429(3)	9757(10)	4705(7)	54(3)
N(3)	1658(4)	3816(14)	7404(10)	100(4)	N(6)	5719(4)	11388(17)	5057(10)	114(5)
O(1)	-1314(2)	7007(9)	7035(5)	53(2)	O(5)	8699(3)	8120(9)	5436(6)	58(2)
O(2)	-1658(3)	6527(10)	8290(6)	87(3)	O(6)	9074(3)	8807(10)	4248(6)	82(3)
O(3)	162(3)	6257(8)	6446(5)	63(3)	O(7)	7206(3)	8851(8)	6031(7)	76(3)
O(4)	-196(3)	7401(10)	4949(6)	63(2)	O(8)	7551(3)	7583(9)	7485(6)	62(2)
C(1)	-1311(4)	6485(13)	7792(9)	60(3)	C(18)	8709(5)	8714(12)	4713(9)	54(3)
C(2)	-839(5)	5839(13)	8084(9)	63(4)	C(19)	8244(4)	9319(12)	4407(8)	54(3)
C(3)	-816(6)	5181(18)	8955(9)	95(6)	C(20)	8225(5)	10064(14)	3563(10)	88(5)
C(4)	247(4)	5610(12)	7134(9)	51(3)	C(21)	7132(4)	9547(14)	5311(10)	60(4)
C(5)	739(4)	5013(14)	7265(10)	71(4)	C(22)	6640(5)	10125(14)	5216(9)	66(4)
C(6)	900(5)	4409(15)	7990(10)	74(4)	C(23)	6414(8)	10360(30)	4507(17)	164(9)
C(7)	1351(5)	3917(15)	8023(9)	82(5)	C(24)	5946(8)	11100(20)	4416(14)	152(8)
C(8)	1474(8)	4310(20)	6683(11)	143(7)	C(25)	5913(9)	11240(30)	5760(14)	158(9)
C(9)	1030(6)	4951(17)	6600(11)	108(5)	C(26)	6371(6)	10585(18)	5876(12)	122(6)
C(10)	-427(5)	9005(12)	6593(9)	83(5)	C(27)	7820(4)	6077(11)	5793(8)	68(4)
C(11)	-251(7)	9240(16)	7470(10)	137(8)	C(28)	7646(7)	5956(14)	4897(10)	118(7)
C(12)	-162(8)	10810(20)	7637(13)	187(13)	C(29)	7666(8)	4401(19)	4622(14)	178(10)
C(13)	272(9)	11220(20)	7217(19)	241(16)	C(30)	7256(9)	3740(20)	4946(16)	188(10)
C(14)	-829(4)	5088(12)	5633(9)	69(4)	C(31)	8183(5)	9892(13)	6902(11)	87(5)
C(15)	-415(5)	4173(13)	5410(10)	92(5)	C(32)	8022(8)	11251(18)	6648(14)	196(11)
C(16)	-691(6)	2750(15)	4897(13)	126(7)	C(33)	8267(7)	12427(18)	7176(15)	171(9)
C(17)	-245(8)	1937(17)	4608(16)	162(10)	C(34)	7827(10)	12960(30)	7627(19)	270(17)

<sup>a</sup>  $U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

**Table 3** Selected bond lengths (nm) and angles (°) for the title complex

Sn(1)—C(10)	0.2124(11)	Sn(2)—C(31)	0.2107(12)
Sn(1)—C(14)	0.2140(11)	Sn(2)—C(27)	0.2140(10)
Sn(1)—O(3)	0.2178(7)	Sn(2)—O(7)	0.2191(8)
Sn(1)—N(1)	0.2281(10)	Sn(2)—N(4)	0.2272(10)
Sn(1)—O(1)	0.2353(7)	Sn(2)—O(5)	0.2383(8)
Sn(1)—O(4)	0.2367(9)	Sn(2)—O(8)	0.2363(9)
Sn(1)—O(5)	0.2621	Sn(2)—O(1)	0.2640
N(1)—C(2)	0.1255(14)	N(4)—C(19)	0.1285(14)
N(1)—N(2)	0.1393(12)	N(4)—N(5)	0.1411(12)
N(2)—C(4)	0.1312(15)	N(5)—C(21)	0.1272(16)
N(3)—C(7)	0.1294(16)	N(6)—C(25)	0.124(2)
N(3)—C(8)	0.1330(19)	N(6)—C(24)	0.122(2)
O(1)—C(1)	0.1294(14)	O(5)—C(18)	0.1275(14)
O(2)—C(1)	0.1237(14)	O(6)—C(18)	0.1248(14)
O(3)—C(4)	0.1273(14)	O(7)—C(21)	0.1334(16)
O(4)···O(6)	0.2668	O(8)···O(2)	0.2721
O(4)···N(6) <sup>#1</sup>	0.2783	O(8)···N(3) <sup>#2</sup>	0.2810
C(10)-Sn(1)-C(14)	164.9(5)	C(31)-Sn(2)-C(27)	163.8(6)
C(10)-Sn(1)-O(3)	93.1(5)	C(31)-Sn(2)-O(7)	95.6(4)
C(14)-Sn(1)-O(3)	96.5(4)	C(27)-Sn(2)-O(7)	95.0(4)
C(10)-Sn(1)-N(1)	99.3(4)	C(31)-Sn(2)-N(4)	97.6(5)
C(14)-Sn(1)-N(1)	95.0(5)	C(27)-Sn(2)-N(4)	97.4(4)
O(3)-Sn(1)-N(1)	69.6(3)	O(7)-Sn(2)-N(4)	70.6(4)
C(10)-Sn(1)-O(1)	90.7(4)	C(31)-Sn(2)-O(8)	83.4(5)
C(14)-Sn(1)-O(1)	89.9(4)	C(27)-Sn(2)-O(8)	87.8(4)
O(3)-Sn(1)-O(1)	138.1(3)	O(7)-Sn(2)-O(8)	74.7(4)
N(1)-Sn(1)-O(1)	68.6(3)	N(4)-Sn(2)-O(8)	145.2(3)
C(10)-Sn(1)-O(4)	86.6(4)	C(31)-Sn(2)-O(5)	91.0(5)
C(14)-Sn(1)-O(4)	84.5(4)	C(27)-Sn(2)-O(5)	89.1(4)
O(3)-Sn(1)-O(4)	75.5(3)	O(7)-Sn(2)-O(5)	138.4(4)
N(1)-Sn(1)-O(4)	144.8(3)	N(4)-Sn(2)-O(5)	67.8(3)
O(1)-Sn(1)-O(4)	146.5(3)	O(8)-Sn(2)-O(5)	146.9(3)
C(2)-N(1)-N(2)	118.8(10)	C(19)-N(4)-N(5)	119.7(11)
C(2)-N(1)-Sn(1)	122.9(9)	C(19)-N(4)-Sn(2)	122.7(8)
N(2)-N(1)-Sn(1)	118.3(7)	N(5)-N(4)-Sn(2)	117.6(7)
C(4)-N(2)-N(1)	107.4(10)	C(21)-N(5)-N(4)	109.2(11)
C(7)-N(3)-C(8)	111.6(13)	C(25)-N(6)-C(24)	119.5(17)
C(1)-O(1)-Sn(1)	117.5(7)	C(18)-O(5)-Sn(2)	117.9(7)
C(4)-O(3)-Sn(1)	115.9(7)	C(21)-O(7)-Sn(2)	114.1(8)
O(2)-C(1)-O(1)	124.5(12)	O(6)-C(18)-O(5)	125.0(11)
O(2)-C(1)-C(2)	118.9(13)	O(6)-C(18)-C(19)	118.3(12)
O(1)-C(1)-C(2)	116.5(11)	O(5)-C(18)-C(19)	116.7(12)
N(1)-C(2)-C(1)	114.5(12)	N(4)-C(19)-C(20)	125.0(11)
N(1)-C(2)-C(3)	126.4(12)	N(4)-C(19)-C(18)	114.8(12)

Continued

N(2)-C(4)-O(3)	128.7(11)	N(5)-C(21)-O(7)	128.3(12)
O(3)-C(4)-C(5)	118.4(11)	O(7)-C(21)-C(22)	114.7(12)
N(2)-C(4)-C(5)	112.9(12)	N(5)-C(21)-C(22)	117.0(13)
N(3)-C(7)-C(6)	127.4(13)	N(6)-C(24)-C(23)	118(2)
N(3)-C(8)-C(9)	125.4(15)	N(6)-C(25)-C(26)	123.3(19)

Symmetry transformations used to generate equivalent atoms: #1:  $x-1/2, -y+2, z$ ; #2:  $x+1/2, -y+1, z$ .

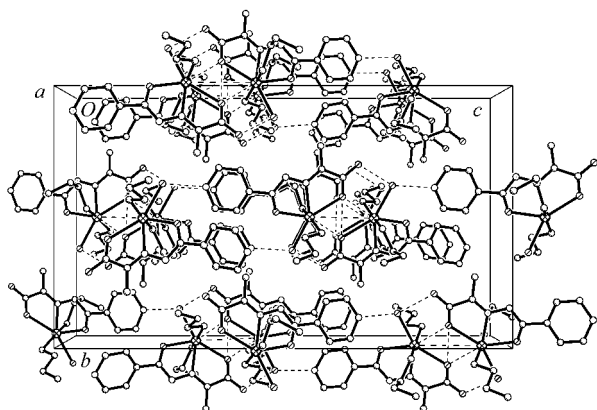


Figure 2 Packing of the molecules in a unit cell.

and interdimeric hydrogen bonds,  $O(4)\cdots N(6)^{\#1}$  (0.2783 nm) and  $O(8)\cdots N(3)^{\#2}$  (0.2810 nm) (symmetry transformations used to generate equivalent atoms: #1:  $x-1/2, -y+2, z$ ; #2:  $x+1/2, -y+1, z$ ). The atoms of the *n*-butyl chains and pyridyl rings show relatively high temperature factors owing to the flexibility of the *n*-butyl and pyridyl groups. Neighboring molecules are held together by hydrogen bonds  $O(4)\cdots N(6)^{\#1}$  and  $O(8)\cdots N(3)^{\#2}$ . These hydrogen bonds contribute to the crystal stability and compactness and result in a one-dimensional chain arrangement (Figure 2).

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