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NOTE

Study on Novel Structure of 4,4'-Bipyridine *bis*(salicylato)borate Hydrate

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4,4'-Bipyridine *bis*(salicylato)borate hydrate with the molecular formula $(C_{10}H_{10}N_2)[B(C_7H_4O_3)_2]_2(H_2O)_4$, is prepared by a hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. The compound crystallize in monoclinic, system with space group and cell parameters, C2/c, a = 24.847 Å, b = 7.4833 Å, c = 21.055 Å, $\alpha = \gamma = 90°$, $\beta = 112.738°$, V = 3610.7 Å^3. The crystal packing is stabilized by O-H···O and O-H···N hydrogen bonding interaction.

Key Words: 4,4'-Bipyridine *bis*(salicylato)borate, Structure analysis, Hydrogen bonding.

Borate compounds have been the intensive subjects of crystallographic study for the last century. There is a great interest preparing the anhydrous main group or transition metal borate compounds[1-3]. Alkali-metals *bis*(salicylato)borates have also received the most attention, since the lithium organoborates had been considered as the lithium battery electrolytes[4]. Studies of organic base *bis*(salicylato)- borates have been less extensive. Piperidinium *bis*(salicylato)borate is reported in the literature[5]. In this paper, the 4,4'-bipyridine *bis*(salicylato)borate dihydrate is reported.

All commercially obtained reagent-grade chemicals were used without further purification. A mixture of $SnCl_2$ (0.1 mmol, 0.019 g), dilute HCl (0.2 mL), 4,4'-bipyridine (0.1 mmol, 0.016 g), salicylic acid (0.1 mmol, 0.014 g) and boric acid (0.1 mmol, 0.006 g) were added into 20 mL water with 10 % ethanol and heated for 6 h at 394 K. The solution was obtained by filtration after cooling the reaction to room temperature. Colourless block single crystals suitable for X-ray measurements were obtained after a few weeks.

The crystal structure of 4,4'-bipyridine *bis*(salicylato)borate hydrate (Fig. 1) is built up of 4,4'-bipyridine cation, *bis*(salicylato)-borate anion and water molecules. The crystal data and structure refinement is shown in Table-1. Boron atom is coordinated by four O atoms from two salicylic acid. The distance d (B-O) are in the range of 1.441-1.486 Å. The angles of O-B-O are in the range of 106.0-112.8°. The torsion angles of C1-O1-B1-O6, C3-O3-B1-O4 and C10-O6-B1-O1 are 99.0,143.8 and 90.9°, respectively. Selected bond lengths and bond angles are shown in Table-2.

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT FOR THE TITLE COMPLEX

Empirical formula	$C_{38}H_{34}B_2N_2O_{16}$
Formula weight	796.29
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 24.847(5) Å α = 90° b = 7.4833(15) Å β = 112.738(2)° c = 21.055(4) Å γ = 90°.
Volume	3610.7(13) Å^3
Z, Calculated density	4, 1.465 mg/m^3
Absorption coefficient	0.114 mm^{-1}
$F_{(000)}$	1656
Crystal size (mm)	0.32 × 0.30 × 0.25
Theta range for data collection	3.17-25.01°
Limiting indices	-29 ⇐ h ⇐ 29, -8 ⇐ k ⇐ 8, -25 ⇐ l ⇐ 25
Reflections collected/unique	13632/3183 [$R_{(int)}$ = 0.1453]
Completeness to theta = 25.01	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9720 and 0.9644
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3183/0/299
Goodness-of-fit on F^2	1.047
Final R indices [I > 2σ(I)]	R_1 = 0.0709, wR_2 = 0.1699
R indices (all data)	R_1 = 0.1207, wR_2 = 0.2047
Largest diff. peak and hole	0.498 and -0.440 e Å^{-3}

The crystal packing is stabilized by O-H···O and O-H···N hydrogen bonding interaction.

TABLE-2
SELECT BOND LENGTHS [Å] AND
ANGLES [°] FOR THE TITLE COMPLEX

N(1)-C(19)	1.288(13)	O(3)-B(1)-O(6)	109.3(3)
N(1)-C(15)	1.33(2)	O(3)-B(1)-O(1)	112.8(3)
O(1)-C(1)	1.316(4)	O(6)-B(1)-O(1)	110.3(3)
O(1)-B(1)	1.484(5)	O(3)-B(1)-O(4)	106.3(3)
O(3)-B(1)	1.441(5)	O(6)-B(1)-O(4)	112.0(3)
O(4)-C(8)	1.311(4)	O(1)-B(1)-O(4)	106.0(3)
O(4)-B(1)	1.486(5)	N(1)-C(15)-C(16)	121(2)
O(6)-C(10)	1.350(5)	N(1)-C(15)-H(15)	119.7
O(6)-B(1)	1.462(6)	N(1)-C(19)-C(18)	121.3(10)
C(1)-O(1)-B(1)	123.3(3)	N(1)-C(19)-H(19)	119.3
C(3)-O(3)-B(1)	119.7(3)	N(1)-C(15')-H(15')	119.2
C(8)-O(4)-B(1)	123.9(3)	C(18')-C(19')-N(1)	117.8(6)
C(10)-O(6)-B(1)	119.9(3)	N(1)-C(19')-H(19')	121.1

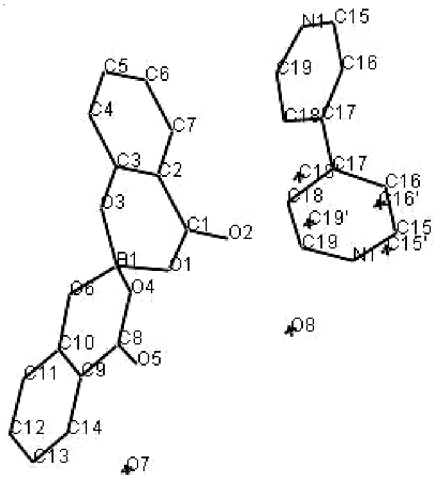

Fig. 1. Molecular structure of $C_{38}H_{34}B_2N_2O_{16}$

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