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Abstract. A novel complex $C_{14}H_8BO_6$. ($C_5H_6ClN_2$). (H_2O) has been synthesized from a hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction. Orthorhombic, Pccn. a=16.6736(13) Å, b =29.447(3) Å, c =8.1366(6) Å, α=β=γ=90°. V=3995.0(6) Å^3. Z=8. R_1 =0.0567, wR_2 = 0.1624, T=293(2) K. The B atom is coordinated by four O atoms from two salicylic acid molecular. The molecular structure stabilized by the O-H…O and O-H…N hydrogen-bonding interactions.

Keywords. 4-amino-2-chloropyridine bis(salicylato)borate; structure analysis; hydrogen bonding.

1. Introduction

The increasing attention paid to boron containing compounds is associated with the possible concrete application of their physical, in particular, ferroelectric, ferromagnetic, nonlinear optical, semiconductor, and other specific properties in various devices. These compounds have high thermal, chemical, and mechanical stability and unique thermal conductivity. The unusual crystal chemistry of these compounds is illustrated by the different coordination of boron atoms resulting in various structural rearrangements whose study allows a better understanding of the genesis of these compounds and the establishment of the relation between their composition, structure, and properties [1-5]. The study of organic base bis(salicylato)-borates has been less extensive. Piperidinium and 4,4'-bipyridine bis(salicylato)borate are reported in the literature [6, 7]. In this paper, the 4-amino-2-chloropyridine bis(salicylato)borate hydrate is reported.

2. Experimental section

All commercially obtained reagent-grade chemicals were used without further purication. A mixture of $RbNO_3$ (0.01 mmol, 0.002g), 4-amino-2-chloropyridine (0.1mmol, 0.013g), boric acid (0.2mmol, 0.012g) and salicylic acid (0.1mmol, 0.014g) were added into 10 mL water with 10%(v/v) ethanol and heated for 7h at 403K. The solution was obtained by filtration after cooling the reaction to room temperature. Colorless single crystals suitable for X-ray measurements were obtained after a few weeks.

The crystal data was collected on a Bruker smart CCD Area Detector.

A colorless block single crystal with dimensions of 0.38 x 0.35 x 0.34 mm was selected for measurement. Diffraction data of the single crystal were collected by ψ-ω scan mode using a graphite-

monochromatic Mo *K*α radiation (λ=0.71073 Å) at 293(2) K on a Bruker Smart Apex CCD diffractometer.

3. Results and discussion

The title crystal structure (Fig.1) is built up of 4-amino-2-chloropyridine cation, bis(salicylato)borate anion and water molecules. The crystal data and structure refinement are shown in Table 1. The B atom is coordinated by four O atoms from two salicylic acid molecular. The distance d (B-O) are in the range of 1.446-1.475 Å. The length d(Cl-C) is 1.699 Å. The angles of O-B-O are in the range of 106.1-113.1°. The torsion angles of C10-O6-B1-O3, C1-O1-B1-O3 and C1-O1-B1-O4 are 92.2, 32.8 and 148.8°, respectively. Selected bond lengths and bond angles are shown in Table 2.

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

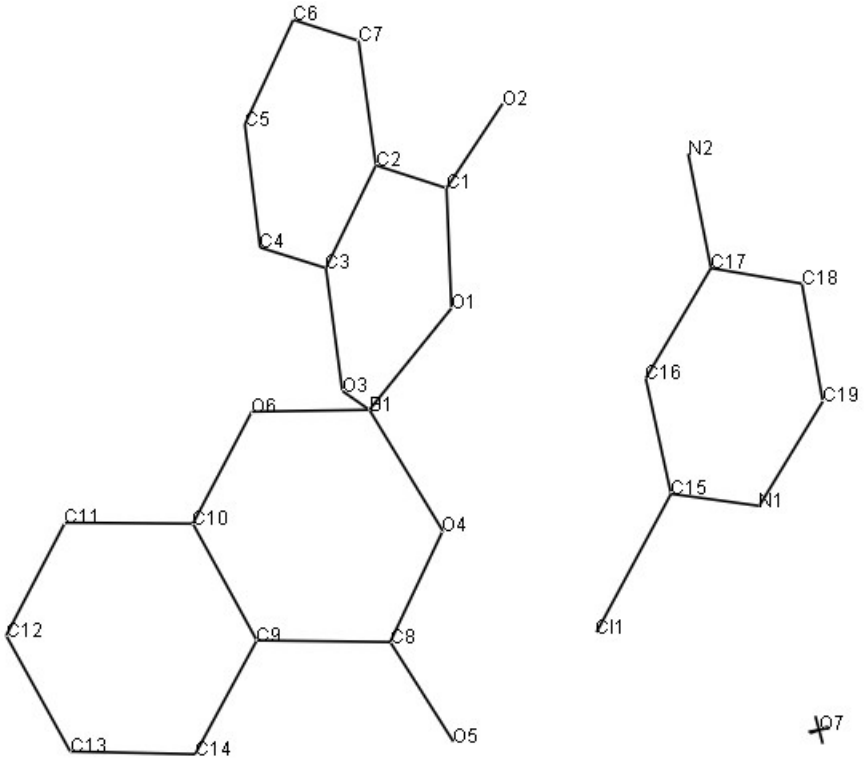

Fig.1 The molecular structure of $C_{19}H_{16}BClN_2O_7$

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

Empirical formula	$C_{19}H_{16}BClN_2O_7$
Formula weight	430.60
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pccn
Unit cell dimensions	a = 16.6736(13) Å alpha = 90°.
	b = 29.447(3) Å beta = 90°.
	c = 8.1366(6) Å gamma = 90°.
Volume	3995.0(6) $Å^3$
Z, Calculated density	8, 1.432 Mg/m^3
Absorption coefficient	0.236 mm^{-1}
F(000)	1776
Crystal size	0.38 x 0.35 x 0.34 mm
Theta range for data collection	3.11 to 25.02°.
Limiting indices	-19<=h<=19, -34<=k<=35, -9<=l<=9
Reflections collected / unique	28192 / 3519 [R(int) = 0.1091]
Completeness to theta = 25.02	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9240 and 0.9156
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3519 / 0 / 272
Goodness-of-fit on F^2	1.035
Final R indices [I>2sigma(I)]	R1 = 0.0567, wR2 = 0.1296
R indices (all data)	R1 = 0.1144, wR2 = 0.1624
Extinction coefficient	0.0076(11)
Largest diff. peak and hole	0.232 and -0.317 e. $Å^{-3}$

Table 2. Select bond lengths [Å] and angles [°] for the title complex.

Cl(1)-C(15)	1.699(4)
N(1)-C(19)	1.343(4)
N(1)-C(15)	1.345(4)
N(2)-C(17)	1.328(5)
O(1)-C(1)	1.323(4)
O(1)-B(1)	1.465(4)
O(2)-C(1)	1.224(3)
O(3)-C(3)	1.358(3)
O(3)-B(1)	1.454(4)
O(4)-C(8)	1.317(4)
O(4)-B(1)	1.475(4)

O(5)-C(8)	1.232(3)
O(6)-C(10)	1.338(4)
O(6)-B(1)	1.446(4)
C(1)-C(2)	1.458(4)
C(2)-C(3)	1.392(4)
C(2)-C(7)	1.396(4)
C(3)-C(4)	1.387(4)
C(4)-C(5)	1.364(4)
C(5)-C(6)	1.385(5)
C(6)-C(7)	1.372(4)
C(8)-C(9)	1.468(4)
C(9)-C(10)	1.390(4)
C(9)-C(14)	1.392(4)
C(10)-C(11)	1.396(4)
C(11)-C(12)	1.364(5)
C(12)-C(13)	1.378(5)
C(13)-C(14)	1.378(4)
C(15)-C(16)	1.353(5)
C(16)-C(17)	1.400(5)
C(17)-C(18)	1.404(4)
C(18)-C(19)	1.348(5)
C(19)-N(1)-C(15)	119.6(3)
C(1)-O(1)-B(1)	122.3(2)
C(3)-O(3)-B(1)	117.3(2)
C(8)-O(4)-B(1)	123.3(2)
C(10)-O(6)-B(1)	119.8(3)
O(6)-B(1)-O(3)	110.8(3)
O(6)-B(1)-O(1)	107.6(3)
O(3)-B(1)-O(1)	112.5(3)
O(6)-B(1)-O(4)	113.1(3)
O(3)-B(1)-O(4)	106.1(3)
O(1)-B(1)-O(4)	106.8(3)
O(2)-C(1)-O(1)	119.5(3)
O(2)-C(1)-C(2)	123.8(3)
O(1)-C(1)-C(2)	116.7(3)
C(3)-C(2)-C(7)	118.7(3)
C(3)-C(2)-C(1)	120.4(3)
C(7)-C(2)-C(1)	120.8(3)
O(3)-C(3)-C(4)	118.5(3)
O(3)-C(3)-C(2)	120.7(2)
C(4)-C(3)-C(2)	120.8(3)
C(5)-C(4)-C(3)	118.7(3)
C(4)-C(5)-C(6)	122.1(3)
C(7)-C(6)-C(5)	119.0(3)
C(6)-C(7)-C(2)	120.7(3)
O(5)-C(8)-O(4)	119.5(3)
O(5)-C(8)-C(9)	123.1(3)
O(4)-C(8)-C(9)	117.4(3)
C(10)-C(9)-C(14)	119.8(3)
C(10)-C(9)-C(8)	119.5(3)
C(14)-C(9)-C(8)	120.6(3)

O(6)-C(10)-C(9)	121.6(3)
O(6)-C(10)-C(11)	119.0(3)
C(9)-C(10)-C(11)	119.4(3)
C(12)-C(11)-C(10)	119.5(3)
C(11)-C(12)-C(13)	121.7(3)
C(14)-C(13)-C(12)	119.3(3)
C(13)-C(14)-C(9)	120.2(3)
N(1)-C(15)-C(16)	121.3(3)
N(1)-C(15)-Cl(1)	115.4(3)
C(16)-C(15)-Cl(1)	123.2(3)
C(15)-C(16)-C(17)	120.1(3)
N(2)-C(17)-C(16)	121.3(3)
N(2)-C(17)-C(18)	121.4(3)
C(16)-C(17)-C(18)	117.3(3)
C(19)-C(18)-C(17)	119.6(3)
N(1)-C(19)-C(18)	122.1(3)

4. Conclusion

A novel complex $C_{14}H_8BO_6$. ($C_5H_6ClN_2$). (H_2O) has been synthesized from a hydrothermal reaction and the crystal structure has been determined by means of single-crystal X-ray diffraction.

Acknowledgments

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