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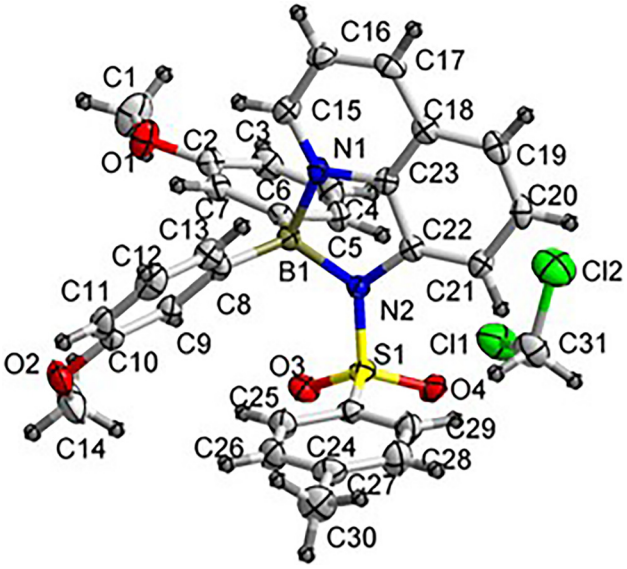
The crystal structure of 2,2-bis(3-methoxyphenyl)-1-tosyl-1,2-dihydro- 2λ⁴,3λ⁴ -[1,3,2]diazaborolo [4,5,1-*ij*]quinoline - dichloromethane (1/1)



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Abstract

$C_{31}H_{29}N_2BO_4Cl_2$, triclinic, $P\bar{1}$ (no. 1), a = 8.7365(3) Å, b = 11.9533(4) Å, c = 14.3566(5) Å, α = 85.7280(10)°, β = 74.8450(10)°, γ = 85.1990(10)°, V = 1439.88(9) Å^3, Z = 2, $R_{gt}(F)$ = 0.0478, w$R_{ref}(F^2)$ = 0.1176, T = 298 K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Block
Size:	0.13 × 0.12 × 0.1 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.34 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, ω and φ scans
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,743, 6597, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4883
$N(param)_{refined}$:	373
Programs:	Bruker programs [1], SHELX [2–4]

1 Source of materials

The title complex was synthesized from a mixture of quinolin-8-amine, 4-methylbenzenesulfonyl chloride and potassium trifluoro(3-methoxylphenyl)-λ^4-borane suspended in MeCN under air atomsphere at 130 °C for 24 h. Until the completion of this reaction, the resulting residue was adsorbed onto silica gel of a DCM solution, loaded directly onto a silica gel column, and purified by eluting first with EtOAc:PE 1:5 to get the target product as yellow solid.

2 Experimental details

Semi-empirical absorption corrections were applied using SADABS program. The structure was solved with SHELXT-2018, and refined using the SHELXL-2019 software package. All the hydrogen atoms were introduced at the calculated positions.

3 Comment

Four-coordinate organboron compounds, which contain π-conjugated chelated backbones, have been under constant investigation over the past decades [5]. They have been documented to express unique properties [6–11]. Therefore, their design [12] and synthesis have also attracted substantial interst.

Single-crystal X-ray diffraction analysis revealed that each asymmetric unit consisted of one B-containing molecule and

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.21869 (6)	0.72291 (4)	0.87012 (4)	0.02221 (13)
Cl1	0.05747 (9)	0.31422 (7)	0.83673 (5)	0.0545 (2)
Cl2	0.37115 (9)	0.33595 (7)	0.86335 (6)	0.0586 (2)
O3	0.09889 (18)	0.75753 (12)	0.82058 (11)	0.0295 (4)
O4	0.19962 (19)	0.62273 (12)	0.93308 (10)	0.0292 (4)
O1	0.2782 (2)	0.70862 (13)	0.37230 (11)	0.0357 (4)
N1	0.6067 (2)	0.73957 (14)	0.65325 (12)	0.0210 (4)
O2	0.0434 (2)	1.10077 (13)	0.64094 (13)	0.0419 (4)
N2	0.3843 (2)	0.70763 (14)	0.78747 (11)	0.0207 (4)
C22	0.5258 (2)	0.66114 (16)	0.80826 (14)	0.0199 (4)
C6	0.3556 (2)	0.68228 (16)	0.61039 (14)	0.0210 (4)
C7	0.3381 (3)	0.72363 (17)	0.51995 (14)	0.0222 (4)
H7	0.356247	0.800099	0.500351	0.027*
C23	0.6538 (2)	0.68270 (16)	0.72791 (14)	0.0203 (4)
C18	0.8137 (3)	0.65087 (16)	0.72405 (15)	0.0230 (4)
C2	0.2947 (3)	0.65559 (18)	0.45774 (15)	0.0252 (5)
C15	0.7149 (3)	0.76705 (18)	0.57275 (15)	0.0265 (5)
H15	0.681992	0.806336	0.520531	0.032*
C5	0.3294 (3)	0.56856 (17)	0.63568 (16)	0.0265 (5)
H5	0.339978	0.537432	0.696689	0.032*
C8	0.3718 (3)	0.89408 (16)	0.67044 (14)	0.0230 (4)
C19	0.8428 (3)	0.58887 (17)	0.80669 (16)	0.0284 (5)
H19	0.948297	0.564279	0.808479	0.034*
C24	0.2387 (3)	0.83354 (17)	0.93983 (14)	0.0239 (4)
C21	0.5586 (3)	0.60049 (17)	0.88676 (15)	0.0251 (5)
H21	0.475602	0.582799	0.942199	0.030*
C3	0.2700 (3)	0.54352 (18)	0.48349 (16)	0.0298 (5)
H3	0.241301	0.496631	0.441061	0.036*
C20	0.7186 (3)	0.56497 (17)	0.88321 (16)	0.0279 (5)
H20	0.740400	0.522093	0.937250	0.033*
C9	0.2250 (3)	0.93712 (17)	0.65596 (14)	0.0247 (5)
H9	0.152011	0.886931	0.646894	0.030*
C17	0.9260 (3)	0.68224 (18)	0.63844 (16)	0.0289 (5)
H17	1.036083	0.664207	0.632263	0.035*
C13	0.4758 (3)	0.97138 (18)	0.68232 (15)	0.0291 (5)
H13	0.576428	0.944825	0.691825	0.035*
C25	0.2040 (3)	0.94305 (18)	0.90964 (16)	0.0323 (5)
H25	0.167430	0.958167	0.852916	0.039*
C10	0.1846 (3)	1.05202 (18)	0.65468 (15)	0.0288 (5)
C16	0.8761 (3)	0.73907 (18)	0.56372 (16)	0.0298 (5)
H16	0.951891	0.759421	0.505560	0.036*
C4	0.2883 (3)	0.50116 (18)	0.57316 (17)	0.0323 (5)
H4	0.272131	0.424226	0.591789	0.039*
C29	0.2921 (3)	0.81138 (19)	1.02264 (16)	0.0332 (5)
H29	0.315142	0.736138	1.043778	0.040*
C27	0.2776 (3)	1.01072 (19)	1.04520 (16)	0.0320 (5)
C11	0.2904 (3)	1.12680 (18)	0.66683 (16)	0.0345 (6)
H11	0.262823	1.205381	0.665667	0.041*
C12	0.4350 (3)	1.08630 (19)	0.68054 (16)	0.0349 (6)
H12	0.507781	1.137125	0.688870	0.042*
C26	0.2230 (3)	1.03045 (19)	0.96274 (17)	0.0355 (6)
H26	0.198079	1.105600	0.942224	0.043*
C28	0.3114 (3)	0.9006 (2)	1.07421 (17)	0.0373 (6)
H28	0.348617	0.885604	1.130708	0.045*
C1	0.2295 (4)	0.6440 (2)	0.30698 (18)	0.0468 (7)
H1A	0.218159	0.692070	0.250507	0.070*
H1B	0.127320	0.613229	0.339329	0.070*
H1C	0.309474	0.582200	0.286153	0.070*
C30	0.2996 (4)	1.1075 (2)	1.10112 (19)	0.0440 (7)
H30A	0.347426	1.168132	1.055929	0.066*
H30B	0.369589	1.081913	1.142931	0.066*
H30C	0.196168	1.135170	1.140848	0.066*
B1	0.4162 (3)	0.76092 (19)	0.67822 (16)	0.0203 (5)
C31	0.1650 (3)	0.3598 (2)	0.91181 (19)	0.0427 (6)
H31A	0.133500	0.319552	0.976009	0.051*
H31B	0.138899	0.441143	0.920738	0.051*
C14	−0.0780 (4)	1.0289 (2)	0.6422 (3)	0.0599 (9)
H14A	−0.041911	0.978445	0.588939	0.090*
H14B	−0.173387	1.074208	0.634887	0.090*
H14C	−0.102797	0.984454	0.703825	0.090*

one free solvent CH_2Cl_2 molecule. As exhibited in the Figure, the central boron atom B1 adopted a typical tetrahedral coordination geometry to be bound to two nitrogen atoms (N1, N2) from the quinoline unit and two carbon atoms (C6, C8) from two methoxybenzene units. The B–N and B–C bond lengths are in the range of 1.609(3)–1.613(3) Å and the C/N–B–C/N angles are in the range of 95.54(14)–117.14(14)°, which are in accordance with reported boron compounds [13–15]. The methoxybenzene units are closely perpendicular to the quinoline unit with the dihedral angles of 80.98 and 70.89°, respectively. Meanwhile, the C–H$\cdots\pi$ interactions makes some contribution to the formation of the crystal. The solvent CH_2Cl_2 molecules are entrapped in the crystal.

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