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The crystal structure of 8-methoxy-2,2-diphenyl-tosyl-1,2-dihydro-2 λ^4 ,3 λ^4 -[1,3,2]diazaborolo [4,5,1-*ig*]quinoline, C₂₉H₂₅BN₂O₃S

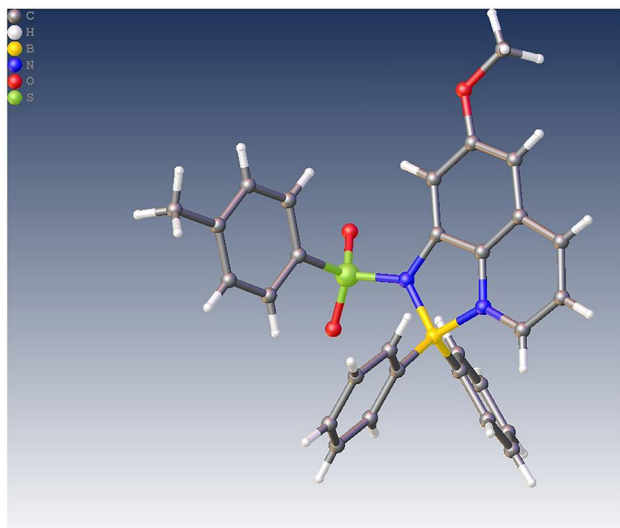


Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.15 × 0.14 mm
Wavelength:	MoK α radiation (0.71073 Å)
μ :	0.17 mm ⁻¹
Diffractionmeter, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	29.9°, 99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	26,032, 7065, 0.076
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 5174
$N(\text{param})_{\text{refined}}$:	327
Programs:	BRUKER [1], SHELX [2, 4, 5], OLEX2 [3]

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Abstract

C₂₉H₂₅BN₂O₃S, monoclinic, $P2_1/c$ (no. 14), $a = 14.4345(6)$ Å, $b = 9.7073(5)$ Å, $c = 18.3873(8)$ Å, $\beta = 107.766^\circ$, $V = 2453.56(19)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0587$, $wR_{\text{ref}}(F^2) = 0.1592$, $T = 193$ K.

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The molecular 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.

1 Source of materials

The title complex was synthesized from a mixture of 6-methoxyl-quinolin-8-amine, 4-methylbenzenesulfonyl

chloride and potassium trifluoro(phenyl)- λ^4 -borane suspended in MeCN under air atmosphere at 130 °C for 24 h. After the completion of this reaction, the resulting residue was adsorbed onto silica gel by rotary evaporation of a DCM solution, loaded directly onto a silica gel column, and purified by rotary evaporation by eluting first with EtOAc:PE 1:5 to get the target product as yellow solid. Crystals of this target product formed and were obtained in a mixed system of *n*-hexane and dichloromethane (DCM as the good solvent and *n*-hexane as the poor solvent).

2 Experimental details

All the H atoms on the benzene rings were placed geometrically and refined without any constraints or restraints.

3 Comment

Four-coordinated organoboron complexes have been widely used as organic fluorescent materials, but the synthesis of N,N-chelated B,B-diaryl tetra-coordinated boron complexes is greatly limited due to the use of organometallic reagents which are extremely sensitive to air and water. A one-pot three-component synthetic strategy of N,N-chelated-B,B-diaryl tetra-coordinated boron complexes is reported. 8-Aminoquinoline is used as the precursor of N,N-chelated ligands, and potassium aryltrifluoroborates (ArBF₃K), which are chemically stable and available in the market, are used

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
S1	0.25565 (3)	0.88064 (5)	0.56661 (3)	0.02659 (12)
O1	0.18385 (10)	0.85533 (16)	0.49422 (8)	0.0343 (3)
O2	0.11524 (12)	0.36235 (16)	0.48890 (8)	0.0438 (4)
O3	0.26754 (10)	1.01731 (14)	0.59740 (8)	0.0337 (3)
N1	0.22866 (10)	0.67298 (17)	0.74577 (8)	0.0257 (3)
N2	0.23168 (11)	0.78332 (16)	0.63043 (8)	0.0264 (3)
C1	0.20195 (12)	0.5838 (2)	0.68620 (10)	0.0252 (4)
C2	0.16825 (15)	0.9725 (2)	0.80142 (12)	0.0360 (4)
H2	0.219312	0.942358	0.844619	0.043*
C3	0.54709 (15)	0.7575 (2)	0.53841 (12)	0.0366 (5)
C4	0.20066 (13)	0.64640 (19)	0.61619 (10)	0.0267 (4)
C5	0.02071 (14)	1.0667 (2)	0.67797 (13)	0.0377 (5)
H5	−0.029726	1.099224	0.635027	0.045*
C6	0.36110 (13)	0.8641 (2)	0.76375 (10)	0.0276 (4)
C7	0.64428 (16)	0.7189 (3)	0.52936 (15)	0.0502 (6)
H7A	0.638489	0.630615	0.502523	0.075*
H7B	0.692673	0.710828	0.579852	0.075*
H7C	0.664762	0.790316	0.499827	0.075*
C8	0.36856 (13)	0.8293 (2)	0.55706 (10)	0.0281 (4)
C9	0.14494 (15)	0.4274 (2)	0.55769 (11)	0.0323 (4)
C10	0.16765 (13)	0.9288 (2)	0.72878 (11)	0.0281 (4)
C11	0.55795 (15)	0.9286 (2)	0.83610 (12)	0.0374 (5)
H11	0.624416	0.950915	0.859876	0.045*
C12	0.17854 (13)	0.4456 (2)	0.69296 (11)	0.0283 (4)
C13	0.08016 (19)	0.2237 (2)	0.48676 (14)	0.0473 (6)
H13A	0.061148	0.189698	0.434080	0.071*
H13B	0.023690	0.221780	0.505755	0.071*
H13C	0.131659	0.164947	0.518982	0.071*
C14	0.09128 (13)	0.9784 (2)	0.66746 (11)	0.0319 (4)
H14	0.087598	0.950794	0.617082	0.038*
C15	0.21256 (13)	0.4926 (2)	0.82749 (11)	0.0318 (4)
H15	0.216591	0.462716	0.877563	0.038*
C16	0.18560 (14)	0.4007 (2)	0.76779 (11)	0.0326 (4)
H16	0.171794	0.307596	0.776674	0.039*
C17	0.46201 (15)	0.6924 (2)	0.49596 (11)	0.0348 (4)
H17	0.464798	0.623441	0.460040	0.042*
C18	0.43366 (13)	0.7625 (2)	0.78185 (11)	0.0327 (4)
H18	0.415589	0.669227	0.769582	0.039*
C19	0.45249 (15)	0.8952 (2)	0.60006 (13)	0.0375 (5)
H19	0.449588	0.964189	0.635964	0.045*
C20	0.37326 (14)	0.7268 (2)	0.50531 (10)	0.0308 (4)
H20	0.315740	0.680550	0.476530	0.037*
C21	0.02359 (15)	1.1073 (2)	0.75067 (13)	0.0399 (5)
H21	−0.024273	1.168366	0.757947	0.048*
C22	0.17028 (14)	0.5672 (2)	0.55136 (11)	0.0298 (4)
H22	0.166271	0.605489	0.502935	0.036*
C23	0.54064 (15)	0.8594 (3)	0.59010 (14)	0.0423 (5)
H23	0.598123	0.905393	0.619162	0.051*
C24	0.39133 (15)	0.9992 (2)	0.78451 (11)	0.0323 (4)
H24	0.344414	1.071054	0.773699	0.039*
C25	0.53079 (14)	0.7935 (2)	0.81701 (12)	0.0379 (5)
H25	0.578339	0.722455	0.827920	0.045*
C26	0.48829 (15)	1.0308 (2)	0.82053 (11)	0.0364 (5)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H26	0.506773	1.123328	0.834543	0.044*
C27	0.09685 (16)	1.0582 (2)	0.81259 (13)	0.0399 (5)
H27	0.098382	1.083188	0.862907	0.048*
C28	0.14961 (15)	0.3649 (2)	0.62579 (11)	0.0338 (4)
H28	0.133868	0.270198	0.627685	0.041*
C29	0.23401 (13)	0.6289 (2)	0.81538 (11)	0.0299 (4)
H29	0.252643	0.690964	0.857264	0.036*
B1	0.24958 (15)	0.8251 (2)	0.71844 (12)	0.0262 (4)

as the source of diaryl groups on the boron centers. A series of these compounds are obtained in moderate to excellent yields. This reaction route has good substrate applicability and functional group compatibility, which provides a convenient and efficient reaction route for the synthesis of diarylboron complexes. The compound mentioned in this article was synthesized through this route in an excellent yield.

The integration of diffraction data and intensity corrections for the Lorentz and polarization effects were performed by using SAINT program. Semi-empirical absorption corrections were applied using SADABS program. All the hydrogen atoms were introduced at the calculated positions.

The asymmetric unit contains one title molecule. As exhibited in Figure, the central boron atom B1 is connected to two nitrogen atoms (N1, N2) from the quinoline unit and two carbon atoms (C6, C10) from two different benzene units to form a typical tetrahedral geometry. The B–N and B–C bond lengths range from 1.608(3) to 1.618(3) Å and the C/N–B–C/N bond angles are in the range of 95.42(13)°–116.42(16)°, which are comparable to some previously reported boron-containing compounds [5–9]. Due to the tetrahedral geometry of central B atom, the dihedral angles among the two benzene rings connected to the B atom and the quinoline unit are 64.50°, 68.44° and 75.98°, respectively. Moreover, the presence of the sulfanilamide group causes the methylbenzene unit closely perpendicular to the quinoline unit with the dihedral angles of 78.63° and the C–H... π interactions between the methylbenzene unit and one of the benzene rings leads to their dihedral angle of as low as 39.34°. In addition, the molecules are assembled by the intermolecular π ... π and C–H... π interactions among the quinoline units, benzene rings and methylbenzene units and C–H...O hydrogen bonds between the sulfanilamide oxygen atom and the methoxy group into two-dimensional (2D) supramolecular layers, which are further joined together by

the C–H $\cdots\pi$ interactions between the benzene rings and the methylbenzene units to form the final three-dimensional (3D) supramolecular structure.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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