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Crystal structure of 8a,8a''-oxybis(8aH-8,9-dioxo-3a¹λ⁴-aza-8aλ⁴-borabenzofg]tetracene), C₃₄H₂₂B₂N₂O₅

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.31 × 0.09 × 0.06 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.10 mm ⁻¹
Diffractometer, scan mode:	APEX2, φ and ω
θ _{max} , completeness:	25.0°, >99%
N(hkl) _{measured} , N(hkl) _{unique} , R _{int} :	13538, 4470, 0.039
Criterion for I _{obs} , N(hkl) _{gt} :	I _{obs} > 2 σ(I _{obs}), 3245
N(param) _{refined} :	388
Programs:	SHELX [1–3], Bruker [4]

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Abstract

C₃₄H₂₂B₂N₂O₅, monoclinic, *P*2₁/*c* (no. 14), *a* = 9.9886(3) Å, *b* = 17.8995(6) Å, *c* = 14.3437(5) Å, β = 98.141(3)°, *V* = 2538.68(15) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0391, *wR*_{ref}(*F*²) = 0.1022, *T* = 100 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title complex was synthesized from a mixture of 2,6-bis(2-methoxyphenyl)pyridine (0.5 mmol) which was dissolved

in anhydrous DCM (10 mL), then cooled to −78 °C, BBr₃ (4 mmol) in anhydrous DCM was added slowly. The mixture was stirred until room temperature was reached. The yellow solid was filtered under a nitrogen atmosphere, the filter cake was washed using anhydrous hexane.

Experimental details

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

Comment

Phenol-pyridyl boron complexes, which have polydentate chelating sites, are one of the hotspots of coordination chemists. They are versatile building blocks in functional molecules, therefore, new methods for their preparation have attracted interests. Among this large family the well-known members are 1,6-bis(2-hydroxy-5-methylphenyl)pyridine ((mdppy)BF) [5, 6] and 1,6-bis(2-hydroxy-5-methylphenyl)pyridylboron substituted benzene [7, 8], which are used in OLEDs and fluoride ion sensors [5–8]. The change of functional groups attached to the boron atom have a great influence on their activities [9, 10].

In the asymmetric unit, there is only one molecule (see the Figure). The electron-deficient boron atom has the ability to reach the quartet coordination, meanwhile the nitrogen atom can offer its lone pair electrons to boron forming a coordination bond. In this compound B1, O3, O4, O5 and the N1 constitute a distorted tetrahedral configuration, together with another distorted tetrahedral configuration consisting of

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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}
B1	−0.0857(2)	0.14033(12)	0.72871(14)	0.0142(5)
B2	0.0903(2)	0.03227(12)	0.73065(14)	0.0145(5)
C1	−0.05387(18)	−0.07591(10)	0.71221(13)	0.0177(4)
C2	−0.16145(19)	−0.11201(11)	0.74463(14)	0.0227(5)
H2	−0.178975	−0.103755	0.807198	0.027*
C3	−0.2430(2)	−0.15987(11)	0.68615(16)	0.0290(5)
H3	−0.315694	−0.184836	0.709039	0.035*
C4	−0.2195(2)	−0.17185(12)	0.59394(16)	0.0294(5)
H4	−0.277218	−0.203878	0.553515	0.035*
C5	−0.1121(2)	−0.13697(11)	0.56175(15)	0.0256(5)
H5	−0.095987	−0.145414	0.498902	0.031*
C6	−0.02587(19)	−0.08906(10)	0.62023(13)	0.0191(4)
C7	0.09438(19)	−0.05590(10)	0.58878(13)	0.0185(4)
C8	0.1477(2)	−0.07985(11)	0.50982(13)	0.0222(5)
H8	0.105811	−0.119634	0.472717	0.027*
C9	0.2613(2)	−0.04603(11)	0.48518(13)	0.0245(5)
H9	0.296875	−0.061795	0.430402	0.029*
C10	0.3232(2)	0.01065(11)	0.53997(13)	0.0216(5)
H10	0.402655	0.033452	0.523749	0.026*
C11	0.26934(18)	0.03458(11)	0.61927(12)	0.0165(4)
C12	0.32842(18)	0.09426(10)	0.68124(13)	0.0169(4)
C13	0.42473(19)	0.14379(11)	0.65402(14)	0.0207(5)
H13	0.444470	0.142622	0.591169	0.025*
C14	0.49085(19)	0.19395(11)	0.71692(14)	0.0232(5)
H14	0.554881	0.227660	0.697386	0.028*
C15	0.46361(19)	0.19512(11)	0.80930(14)	0.0219(5)
H15	0.511790	0.228340	0.853606	0.026*
C16	0.36710(18)	0.14833(10)	0.83683(13)	0.0180(4)
H16	0.348478	0.149913	0.899934	0.022*
C17	0.29685(17)	0.09897(10)	0.77345(13)	0.0149(4)
C18	−0.23826(19)	0.07202(10)	0.81526(13)	0.0169(4)
C19	−0.35159(19)	0.02666(11)	0.80997(14)	0.0225(5)
H19	−0.412361	0.023581	0.752818	0.027*
C20	−0.3764(2)	−0.01396(11)	0.88728(14)	0.0264(5)
H20	−0.455250	−0.044092	0.883625	0.032*
C21	−0.2865(2)	−0.01106(11)	0.97072(14)	0.0263(5)
H21	−0.301636	−0.040888	1.022999	0.032*
C22	−0.1754(2)	0.03526(11)	0.97714(14)	0.0225(5)
H22	−0.114336	0.037166	1.034241	0.027*
C23	−0.15100(18)	0.07971(10)	0.90063(13)	0.0173(4)
C24	−0.04681(18)	0.13796(10)	0.91036(13)	0.0170(4)
C25	0.01972(19)	0.16199(12)	0.99706(13)	0.0225(5)
H25	0.003340	0.137847	1.053340	0.027*
C26	0.10904(19)	0.22062(12)	1.00112(14)	0.0242(5)
H26	0.155363	0.236583	1.060191	0.029*
C27	0.13159(19)	0.25632(11)	0.91972(13)	0.0212(5)
H27	0.191109	0.297926	0.922791	0.025*
C28	0.06700(18)	0.23133(10)	0.83291(13)	0.0166(4)
C29	0.08544(18)	0.26571(10)	0.74273(13)	0.0162(4)
C30	0.19096(19)	0.31566(11)	0.73481(14)	0.0214(5)
H30	0.255144	0.325982	0.788708	0.026*
C31	0.2041(2)	0.35021(11)	0.65087(14)	0.0235(5)
H31	0.276722	0.383830	0.647047	0.028*
C32	0.11050(19)	0.33558(11)	0.57178(14)	0.0218(5)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H32	0.119079	0.359326	0.513682	0.026*
C33	0.00488(19)	0.28659(10)	0.57730(13)	0.0183(4)
H33	−0.059176	0.276997	0.523093	0.022*
C34	−0.00784(17)	0.25133(10)	0.66197(13)	0.0149(4)
N1	−0.02051(14)	0.17233(9)	0.83027(10)	0.0153(4)
N2	0.15603(14)	0.00083(8)	0.64192(10)	0.0154(4)
O1	0.02476(12)	−0.03014(7)	0.77155(8)	0.0173(3)
O2	0.20188(12)	0.05422(7)	0.80228(8)	0.0154(3)
O3	−0.21778(12)	0.11018(7)	0.73759(8)	0.0183(3)
O4	−0.11339(12)	0.20475(7)	0.66575(8)	0.0149(3)
O5	0.00512(12)	0.09099(7)	0.69360(8)	0.0149(3)

B2, O1, O2, O5 and N2. All bonds and angles in the crystal structure are within the normal range.

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