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The crystal structure of 8a-methoxy 8a-methoxy-1,5,8a,9a-tetrahydro-4*H*-8,9-dioxo-3a¹λ⁴-aza-8aλ⁴, C₁₈H₁₄BNO₃

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.10 × 0.08 × 0.06 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	27.7°, 99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	13137, 3347, 0.032
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2468
<i>N</i> (<i>param</i>) _{refined} :	209
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

C₁₈H₁₄BNO₃, monoclinic, *P*2₁/*c* (no. 14), *a* = 9.2580(17) Å, *b* = 11.950(2) Å, *c* = 13.076(2) Å, β = 99.150(2)°, *V* = 1428.2(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0412, *wR*_{ref}(*F*²) = 0.1087, *T* = 100 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.

Source of material

The title complex was synthesized by dissolving 2,6-bis(2-methoxyphenyl)pyridine (0.5 mmol) in anhydrous DCM

(10 mL); the mixture was then cooled to −78 °C. BBr₃ (4 mmol) in anhydrous DCM was added slowly to the mixture, keeping stirring until warm to room temperature. Water was carefully added to the reaction mixture; DCM was used for extraction, the organic layer was washed with water and brine, then dried under anhydrous Na₂SO₄. 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 DCM followed by DCM:methanol 30:1 to get the target product as a yellow solid.

Experimental details

All H atoms were refined using the appropriate riding constraints for positions and *U*_{iso} parameters.

Comment

In coordination chemistry, polypyridines play an important role by virtue of their polydentate chelating properties. Among the numerous compounds, phenol-pyridyl boron complexes, which contain mixed pyridine-phenol donor sets, have attracted considerable attention. In recent years, much effort has been focussed in the design and synthesis of this kind of compounds, the most notable members of this family being 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 have the potential to be used in functional

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
B1	0.26248(17)	0.34954(13)	0.10402(13)	0.0157(3)
N1	0.39325(12)	0.42947(9)	0.15252(9)	0.0140(3)
O1	0.17603(11)	0.32183(8)	0.18445(8)	0.0185(2)
O2	0.18718(10)	0.41361(8)	0.02024(8)	0.0174(2)
O3	0.32106(10)	0.24341(8)	0.07481(8)	0.0171(2)
C1	0.44003(15)	0.24551(11)	0.02697(11)	0.0158(3)
C2	0.46325(16)	0.15561(12)	−0.03683(12)	0.0198(3)
H2	0.3938	0.0966	−0.0483	0.024*
C3	0.58731(17)	0.15263(12)	−0.08318(12)	0.0223(3)
H3	0.6029	0.0911	−0.1261	0.027*
C4	0.68976(16)	0.23893(12)	−0.06770(12)	0.0211(3)
H4	0.7741	0.2366	−0.1006	0.025*
C5	0.66799(16)	0.32800(12)	−0.00416(11)	0.0181(3)
H5	0.7376	0.3870	0.0060	0.022*
C6	0.54396(15)	0.33214(11)	0.04559(11)	0.0148(3)
C7	0.52473(15)	0.42296(11)	0.11846(11)	0.0149(3)
C8	0.63413(15)	0.50010(11)	0.15346(11)	0.0173(3)
H8	0.7269	0.4950	0.1315	0.021*
C9	0.60673(16)	0.58424(12)	0.22032(11)	0.0189(3)
H9	0.6794	0.6389	0.2426	0.023*
C10	0.47324(16)	0.58854(11)	0.25472(11)	0.0181(3)
H10	0.4549	0.6456	0.3015	0.022*
C11	0.36554(15)	0.50960(11)	0.22110(11)	0.0160(3)
C12	0.22369(15)	0.50471(12)	0.25807(11)	0.0173(3)
C13	0.17647(16)	0.59177(13)	0.31736(12)	0.0222(3)
H13	0.2335	0.6580	0.3291	0.027*
C14	0.04875(17)	0.58261(14)	0.35869(13)	0.0271(4)
H14	0.0178	0.6425	0.3978	0.033*
C15	−0.03451(17)	0.48536(14)	0.34282(13)	0.0263(4)
H15	−0.1212	0.4782	0.3726	0.032*
C16	0.00835(16)	0.39894(13)	0.28382(12)	0.0228(3)
H16	−0.0493	0.3330	0.2729	0.027*
C17	0.13589(16)	0.40844(12)	0.24038(11)	0.0180(3)
C18	0.06866(17)	0.36017(13)	−0.04342(13)	0.0282(4)
H18A	0.1061	0.3000	−0.0829	0.042*
H18B	0.0162	0.4149	−0.0914	0.042*
H18C	0.0017	0.3287	0.0001	0.042*

materials, including organic light-emitting devices (OLEDs), nonlinear optics, fluorescent and fluoride ion sensors [5–8]. Varying the types of substitution on the boron atom may lead to some different properties that may have a profound impact on their activity [9, 10]. Here we report the crystal structure of the title compound (*cf.* the Figure).

There is one molecule in the asymmetric unit (see the figure). The B₁, O₁, O₂, O₃ and the N₁ atoms are arranged in an asymmetrical tetrahedral configuration with an electron pair donation from N1 to B1 prior to (∼sp³) hybridisation, as well documented in standard inorganic chemistry literature. All bonds and angles in the crystal structure are within the normal range. Several C–H...O hydrogen bonds contribute to the structure stability. In conclusion, we have developed a facile process for the synthesis of the title compound.

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