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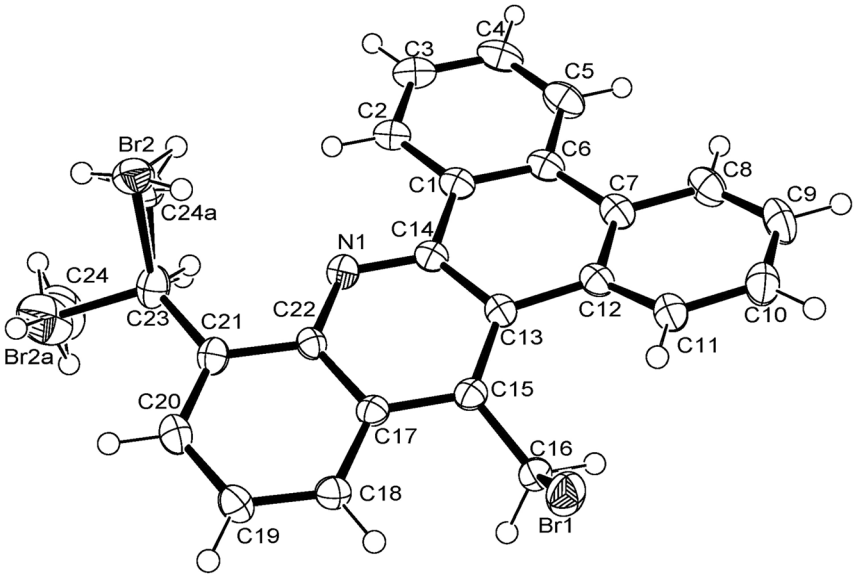
The crystal structure of 10-(1-bromoethyl)-14-(bromomethyl)dibenzo[*a*, *c*]acridine, $C_{24}H_{17}NBr_2$



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Abstract

$C_{24}H_{17}NBr_2$, monoclinic, $P2_1/n$ (no. 14), a = 9.6466(19) Å, b = 14.030(3) Å, c = 13.894(3) Å, β = 100.95(3)°, V = 1846.2(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0440, $wR_{ref}(F^2)$ = 0.0970, T = 173(2) K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.29 × 0.19 × 0.09 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	4.40 mm^{-1}
Diffractometer, scan mode:	Rigaku, ω scan
θ_{max}, completeness:	27.5°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12088, 4156, 0.059
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,808
$N(param)_{refined}$:	265
Programs:	Rigaku,[1] SHELX[2,3]

1 Source of materials

The title compound was synthesized *via* 3-step reactions with commercially available 9,10-phenanthrenequinone and 2,6-diethylaniline as starting materials according to our previous report.[4] The 10-ethyl-14-methyldibenzo[*a*,*c*]acridine (synthesized by our previous report[4]) (3.00 g, 9.34 mmol) was dissolved in CCl_4 (100 mL) and *N*-bromosuccinimide (3.32 g, 18.68 mmol) and dibenzoyl peroxide (0.23 g, 10 mol %) were added to the above solutions. The suspension was stirred and heated to reflux in a vessel open to the air for 6 h. On completion of the reaction, the mixture was filtered whilst hot. The filtrate was collected and the solvent removed under

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reduced pressure. The residue was recrystallized from dichloromethane and heptane forming the title compound as a yellow solid (4.25 g, 95 %). Melting point: 174–175 °C. Yellow crystals of the title compound were obtained in a mixture of dichloromethane and diethyl ether.

2 Experimental details

The crystal data was collected, refined and reduced by CrystalClear (Rigaku Inc., 2007).[1] The crystal structure was solved using the SHELXTL[2] structure solution program and refined with the SHELXL[3] refinement package. All hydrogen atoms were placed in calculated positions and refined as riding on their parent atom. It is noted that there exists position disorder in 1-bromoethyl connected to the C21 of benzene ring due to the rotation of the terminal ethyl group. To solve this problem, the C24 and Br2 atoms from 1-bromoethyl were split to two sets with the site occupancy factors of 0.91766 (C24 and Br2) and 0.08234 (C24A and Br2A) and the related SIMU, ISOR and DFIX orders were used in the refinement.

3 Comment

The 9,10-phenanthrenequinone-based α-diimine compounds are an important class of organic ligands and used to synthesize the corresponding α–diimine nickel complexes for ethylene polymerization[5–7] or pincer palladium complexes for catalyzing coupling reactions.[4] In this paper, a molecule and its synthesis by 3-step reactions using commercially available 9,10-phenanthrenequinone and 2,6-diethylaniline as raw materials is described.[4] The title molecule features some geometric similarities to the aforementioned α-diimines.

The asymmetric unit of the title compound contains one independent molecule. It is a pentacyclic skeleton composing of phenanthrene-based fused quinoline heterocycle. There are 1-bromomethyl and bromoethyl in the 3- and 7-position of the quinoline ring, respectively. The bond lengths of N1–C14 and N1–C22 are 1.329(4) and 1.350(4) Å, respectively, which is typical for carbon-nitrogen double bond.[8] In addition, the bond lengths of C16–Br1 and C23–Br2 are 1.983(3) and 1.976(4) Å, respectively. The bond lengths and bond angels of the title structure lie in the normal range and are in accordance with the previous literatures.[4,9,10] The dihedral angle between the plane (C1/C2/C3/C4/C5/C6) of benzene ring and the plane (C13/C14/N1/C22/C17/C15) of pyridine ring is 7.15°. The dihedral angle of the plane (C7/C8/C9/C10/C11/C12) of benzene ring and the plane (C13/C14/N1/C22/C17/C15) of pyridine ring is 17.73°. The complete set of X-ray diffraction data for the title compound was deposited to the Cambridge Crystallographic Data Center (CCDC entry no. 2418020).

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