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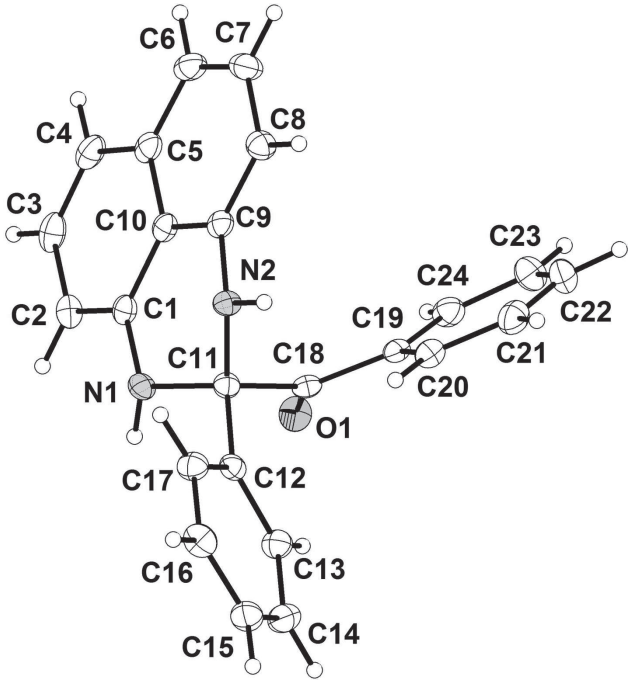
Crystal structure of phenyl(2-phenyl-2,3-dihydro-1*H*-perimidin-2-yl)methanone, $C_{24}H_{18}N_2O$



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Abstract

$C_{24}H_{18}N_2O$, monoclinic, $P2_1/n$ (no. 14), $a = 5.9227(7)$ Å, $b = 16.462(2)$ Å, $c = 18.150(2)$ Å, $\beta = 98.372(2)°$, $V = 1750.8(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0353$, $wR_{ref}(F^2) = 0.0942$, $T = 100(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.20 × 0.10 × 0.05 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	53°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	63640, 3704, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3277
$N(param)_{refined}$:	247
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3, 4], publCIF [5]

Source of material

Benzil (2.10 g; 10.0 mmol) and 1,8-diaminonaphthalene (1.58 g; 10.0 mmol) were dissolved in ethanol (75 mL). Concentrated HCl (aq) (4 drops) was added. The clear solution was heated to reflux for ~40 h during which a yellow precipitate formed. After cooling to room temperature, the yellow precipitate was removed by suction filtration, washed with ethanol (50 mL) and air-dried for ~20 h (1.94 g; 55%). The product was recrystallized from ethanol.

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Carbon-bound H atoms were fixed at 0.93 Å, with displacement parameters 1.2 times U_{eq}(C). Nitrogen-bound H atoms were fixed at 0.86 Å, with U_{iso}(H) set to 1.2U_{eq}(N).

Comment

Perimidines are an important class of compounds with attractive biological and physical properties [6–12]. The title compound [13–16] was prepared by condensation of 1,8-diaminonaphthalene and benzil with acid catalysis. One of the nitrogen atoms (N1) is coplanar with the ten carbon atoms of the naphthylene ring (C1—C10). The second nitrogen atom (N2) is slightly diplaced below this plane by 0.14 Å and the bridging carbon atom (C11) is displaced above this plane by 0.56 Å. The plane of the phenyl ring (C12—C17) intersects with the naphthyl plane (C1—C10) at an angle of 67.9° and the

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.03471(14)	0.48645(5)	0.21651(5)	0.0207(2)
N2	0.55029(16)	0.44201(6)	0.15019(6)	0.0143(2)
C19	0.38909(19)	0.47854(7)	0.29437(6)	0.0151(2)
C5	0.2403(2)	0.24648(7)	0.12042(6)	0.0181(2)
N1	0.18919(16)	0.46976(6)	0.08326(5)	0.0155(2)
H1	0.06885(16)	0.50168(6)	0.07585(5)	0.0185(2)*
C8	0.6578(2)	0.30623(7)	0.19638(7)	0.0188(2)
H8	0.7984(2)	0.32563(7)	0.22228(7)	0.0226(3)*
C9	0.50445(19)	0.35953(7)	0.15790(6)	0.0152(2)
C1	0.12935(19)	0.38666(7)	0.08258(6)	0.0154(2)
C3	−0.1226(2)	0.27446(8)	0.04448(7)	0.0221(3)
H3	−0.2632(2)	0.25544(8)	0.01822(7)	0.0265(3)*
C2	−0.0746(2)	0.35823(8)	0.04514(6)	0.0187(2)
H2a	−0.1825(2)	0.39510(8)	0.01984(6)	0.0224(3)*
C4	0.0290(2)	0.21959(8)	0.08091(7)	0.0212(3)
H4	−0.0077(2)	0.16339(8)	0.07965(7)	0.0254(3)*
C12	0.42314(19)	0.58393(7)	0.13764(6)	0.0148(2)
C10	0.29050(19)	0.33105(7)	0.12067(6)	0.0155(2)
C18	0.24208(19)	0.48786(7)	0.22045(6)	0.0145(2)
C13	0.3118(2)	0.64663(7)	0.16946(7)	0.0185(2)
H13	0.1904(2)	0.63435(7)	0.19655(7)	0.0223(3)*
C6	0.4038(2)	0.19305(7)	0.15972(7)	0.0213(3)
H6	0.3738(2)	0.13638(7)	0.16013(7)	0.0256(3)*
C7	0.6051(2)	0.22289(8)	0.19713(7)	0.0219(3)
H7	0.7110(2)	0.18649(8)	0.22407(7)	0.0263(3)*
C21	0.7325(2)	0.49885(8)	0.38178(7)	0.0237(3)
H21	0.8792(2)	0.52274(8)	0.39411(7)	0.0285(3)*
C14	0.3769(2)	0.72677(8)	0.16187(7)	0.0230(3)
H14	0.3004(2)	0.76907(8)	0.18388(7)	0.0276(3)*
C16	0.6621(2)	0.68333(8)	0.08849(7)	0.0212(3)
H16	0.7809(2)	0.69603(8)	0.06041(7)	0.0254(3)*
C17	0.5969(2)	0.60292(7)	0.09589(7)	0.0186(2)
H17	0.6705(2)	0.56083(7)	0.07257(7)	0.0223(3)*
C15	0.5536(2)	0.74529(8)	0.12218(7)	0.0217(3)
H15	0.6004(2)	0.80009(8)	0.11799(7)	0.0260(3)*
C23	0.4258(3)	0.41798(8)	0.41673(7)	0.0276(3)
H23	0.3649(3)	0.38535(8)	0.45228(7)	0.0331(4)*
C20	0.6072(2)	0.51182(7)	0.31190(7)	0.0183(2)
H20	0.6700(2)	0.54336(7)	0.27601(7)	0.0220(3)*
C24	0.2979(2)	0.43273(7)	0.34806(7)	0.0205(3)
H24	0.1474(2)	0.41169(7)	0.33721(7)	0.0246(3)*
C11	0.35390(19)	0.49572(7)	0.14733(6)	0.0141(2)
C22	0.6432(2)	0.45080(9)	0.43381(7)	0.0282(3)
H22	0.7310(2)	0.44050(9)	0.48097(7)	0.0339(4)*
H2	0.678(3)	0.4602(10)	0.1758(9)	0.025(4)*

planes described by the atoms N1—C11—N2 and C12—C11—C18 are almost orthogonal with an intersection angle of 89.8°. The crystal architecture is best viewed along the *a*-axis, exhibiting a head-to-tail arrangement of naphthyl-phenyl layers with interlocking benzoyl rings from the adjacent layer, maintained by a single hydrogen bond, N—H···O=C, and multiple weak Ar···H, Ar···Ar, C=O···H—C contacts.

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