Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

12,13-Dihydro-12-oxo-13-phenylbenzo[*b*]-naphth[*f*][1,4]oxazepine-2,3-dicarbonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.076
Data-to-parameter ratio = 8.2

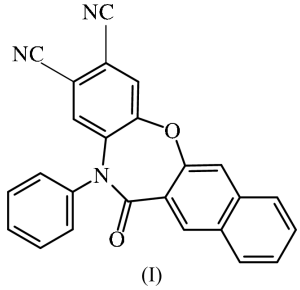
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $C_{25}H_{13}N_3O_2$, is stabilized only by weak van der Waals interactions. The seven-membered ring has a boat conformation.

Received 26 October 2004
Accepted 11 January 2005
Online 29 January 2005

Comment

4,5-Disubstituted phthalonitriles have been used as starting materials for peripherally substituted phthalocyanines and subphthalocyanines (McKeown, 1998). Phthalocyanines are among the most extensively investigated chemical species because of their uses in chemical sensors, low-dimensional conductors, non-linear optics and liquid crystals, as well as their applications as catalysts and dyes (Leznoff & Lever, 1989–1996). The production of phthalocyanines for use as dyes and pigments is *ca.* 80 000 tons per year (Wöhrle, 2001).



(I)

The seven-membered oxazepine ring of (I) adopts a boat conformation. The C ≡N bond lengths (Table 1) are in good agreement with literature values (Petek *et al.*, 2004).

Experimental

3-Hydroxy-2-naphthoic acid anilide (1.34 g, 5.09 mmol) and 4,5-dichloro-1,2-dicyanobenzene (1.00 g, 5.08 mmol) were heated to 348 K

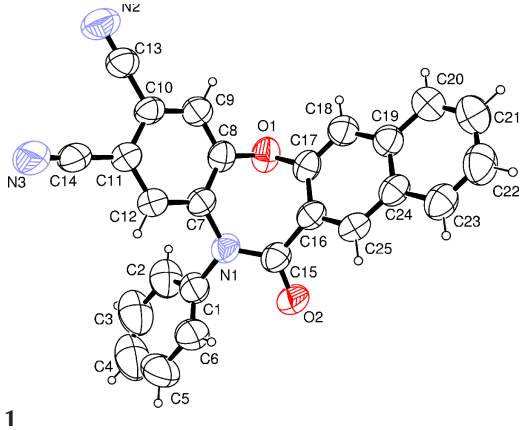


Figure 1
An *ORTEP-3* view (Farrugia, 1997) of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids.

in dry dimethylformamide (50 ml) with stirring under N_2. Dry fine-powdered potassium carbonate (2.10 g, 15.22 mmol) was added in portions (12 × 1 mmol) every 10 min. The mixture was heated for a further 48 h and, after cooling, was poured into ice–water (200 g). The product was filtered off and washed with (10% *w*/*w*) NaOH solution and water until the filtrate was neutral. Recrystallization from ethanol gave a white product (yield 0.70 g, 35.53%). Single crystals were obtained from ethanol at room temperature *via* slow evaporation (m.p. 525 K).

Crystal data

$C_{25}H_{13}N_3O_2$
M_r = 387.38
Orthorhombic, $Pca2_1$
a = 12.2243 (6) Å
b = 19.1241 (10) Å
c = 8.5207 (19) Å
V = 1992.0 (5) Å^3
Z = 4
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7142 reflections
θ = 2.1–27.2°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colourless
0.55 × 0.30 × 0.20 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.970, T_{max} = 0.983
8228 measured reflections
2244 independent reflections
1413 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.0°
$h = -8 \rightarrow 15$
$k = -24 \rightarrow 24$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.076
S = 0.88
2244 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.09 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0149 (15)

Table 1
Selected bond lengths (Å).

C13—N2	1.149 (3)	C14—N3	1.141 (4)

All H atoms were treated using a riding model, with C—H = 0.93 Å. The U_{iso} values for H atoms were assigned as $1.2U_{eq}$(carrier atom). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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