organic papers

11-Phenyl-7,8,9,10,10a,11,12,13,14,15-decahydro-5*H*-3,1-benzoxazino[2,1-e]acridine

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2-Hydroxy-8-phenyltricyclo[7.3.1.0^{2,7}]tridecan-13-one reacts with 2-aminobenzyl alcohol to form the title compound, $C_{26}H_{29}NO$, which crystallizes with two independent molecules in the asymmetric unit. The N atom shows a nearly planar coordination owing to the strain imposed on the fused-ring compound.

Comment

2-Hydroxy-8-phenyltricyclo[7.3.1.0^{2,7}]tridecan-13-one (trivial name: phenyl ketol) condenses through its carbonyl group with primary amines to form Schiff bases that undergo cyclization owing to the proximity of the hydroxy group, as noted with, for example, aniline and substituted anilines, which afford 9-phenyl-10-organyldecahydroacridines (Alekseev *et al.*, 1976; Kruglyakova *et al.*, 1977). The reaction of phenyl ketol with *o*-aminobenzyl alcohol probably yields as the initial product 10-(2-hydroxymethylphenyl)-1,2,3,4,5,-6,7,8,9,10-decahydro-9-phenylacridine; the –CH$_2$OH group then adds across one of the two double bonds, giving rise to the title compound, (I). The compound crystallizes with two independent molecules in the asymmetric unit; the bond dimensions in both are normal, and there are no important intermolecular interactions in the crystal structure. The compound is expected to undergo oxidative coupling with methylene-active compounds in the presence of manganese dioxide (Slabko *et al.*, 1990).

(I)
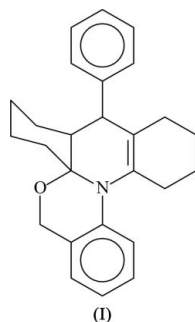
Experimental

Commercially available phenyl ketol (2.0 g, 7.0 mmol) was dissolved in *p*-xylene (50 ml) and to the solution was added *o*-aminobenzyl alcohol (1.0 g, 8.1 mmol). The reaction was catalysed by *p*-toluene-sulfonic acid (5 mg); the mixture was heated for 35 h in a Dean–Stark apparatus. On completion of the reaction, the solvent was removed under reduced pressure. The oily residue was triturated with ethanol (10 ml) to yield a solid compound that was purified by column chromatography on alumina, with *n*-hexane as eluant, in about 30% yield (0.81 g; m.p. 435–437 K). The molecular weight of the compound was established by mass spectrometry.
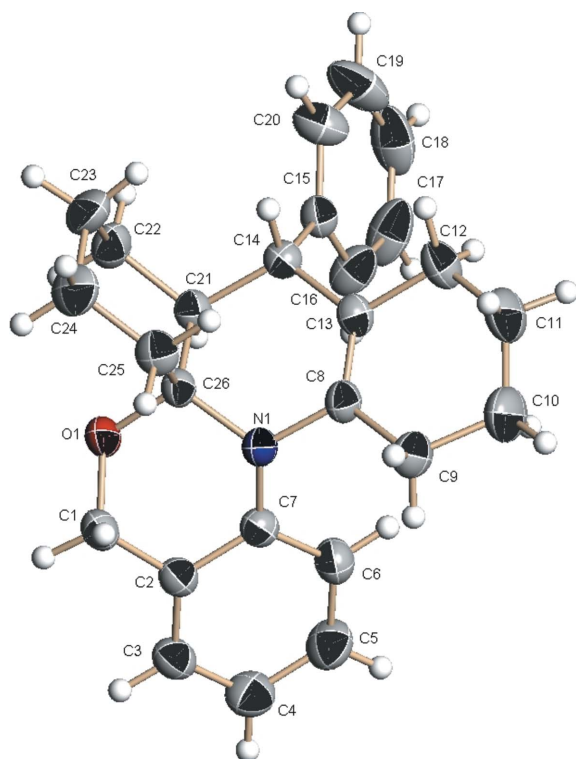
Figure 1
View of one of the two independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	6699 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.033$
φ and ω scans	$\theta_{max} = 27.5°$
Absorption correction: None	$h = -13 \rightarrow 13$
25549 measured reflections	$k = -14 \rightarrow 14$
9331 independent reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$+ 0.6554P]$
$wR(F^2) = 0.133$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
9331 reflections	$\Delta\rho_{max} = 0.49$ e Å^{-3}
505 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H-atom parameters constrained	

H atoms were placed at calculated positions [C—H = 0.93–0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$] and were included in the refinement in the riding-model approximation.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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Crystal data

$C_{26}H_{29}NO$	$Z = 4$
$M_r = 371.50$	$D_x = 1.211$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.653$ (1) Å	Cell parameters from 956
$b = 11.427$ (1) Å	reflections
$c = 17.337$ (2) Å	$\theta = 2.4–27.6°$
$\alpha = 91.124$ (2)°	$\mu = 0.07$ mm^{-1}
$\beta = 100.810$ (2)°	$T = 295$ (2) K
$\gamma = 100.191$ (2)°	Prism, colorless
$V = 2037.4$ (4) Å^3	$0.40 \times 0.35 \times 0.25$ mm