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1-Ethyl-2-oxo-5-phenyl-1,6-dihydro-2*H*-oxazolo[4′,5′:5,6]pyrido[3,4-*b*]indole methanol hemisolvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.052
wR factor = 0.147
Data-to-parameter ratio = 12.0

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

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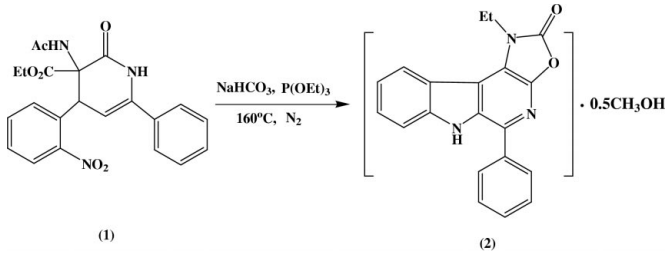
The title compound, $C_{20}H_{15}N_3O_2\cdot0.5CH_3OH$, has been synthesized from the ene-lactam with $P(OEt)_3$ under an N_2 atmosphere. The title indole molecules are linked *via* N—H···O hydrogen bonds, forming one-dimensional chains.

Comment

Azaelliptitoxin (azatoxin) as a potent inhibitor of DNA topoisomerase II and tubulin polymerization have attracted much attention (Tepe *et al.*, 1996). However, the crystal structures of azaelliptitoxin and its analogues have rarely been reported (Abu-Safieh *et al.*, 2002). Conveniently, we have synthesized the title compound, (2), similar to azaelliptitoxin in one pot from the ene-lactam (1) with $P(OEt)_3$ under an N_2 atmosphere (Yu *et al.*, 2004).



(1) (2)

The asymmetric unit of (2) comprises one title indole molecule and one-half of a methanol molecule of crystal-

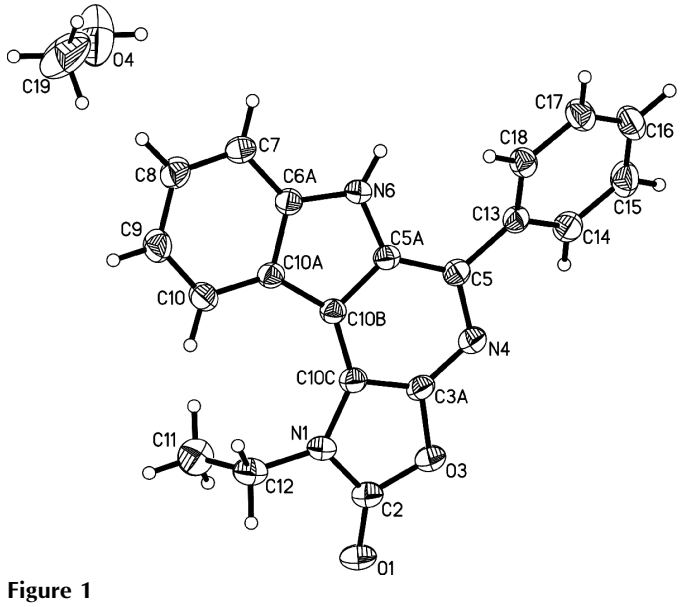


Figure 1
The molecular structure of (2) with 30% probability displacement ellipsoids. The methanol atom O4 shows positional disorder and one of two possible positions has been omitted for clarity.

lization (Fig. 1). The title indole molecules are linked by N—H···O hydrogen bonds (Table 1), forming one-dimensional chains (Fig. 2). Neighbouring molecular chains are parallel to one another, forming layers (Fig. 3); neighboring layers are cross-linked to each other through solvent methanol molecules, leading to the three-dimensional supermolecular framework.

Experimental

The ene-lactam (1) (1.06 g, 2.5 mmol) and $NaHCO_3$ (0.21 g, 2.5 mmol) in $P(OEt)_3$ (25 ml) were heated at 393 K under nitrogen. After 3 h, all ene-lactam (1) had disappeared (monitored by thin-layer chromatography). The solvent was removed *in vacuo*, then the title compound was collected by chromatography on silica gel with CH_2Cl_2–EtOAc–petroleum ether (1:10:10). Crystals of (2) suitable for X-ray analysis were obtained by recrystallization from MeOH.

Crystal data

$C_{20}H_{15}N_3O_2{\cdot}0.5CH_4O$
M_r = 345.37
Monoclinic, $C2/c$
a = 17.728 (4) Å
b = 8.5045 (19) Å
c = 22.575 (5) Å
β = 105.925 (4)°
V = 3273.0 (12) Å^3
Z = 8
D_x = 1.402 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 883 reflections
θ = 2.4–22.7°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.3 × 0.2 × 0.2 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.97, T_{max} = 0.98
7860 measured reflections
2884 independent reflections
2232 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 25.0°
$h = -17 \rightarrow 20$
$k = -10 \rightarrow 9$
$l = -26 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.147
S = 1.06
2884 reflections
241 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.081P)^2 + 0.3023P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.011
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O1^{i}	0.86	2.16	2.938 (2)	150

Symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}+y, z$.

Methanol atom C19 lies on a twofold axis parallel to b, and atom O4 shows positional disorder over two sites. All H atoms were positioned geometrically, with C—H = 0.93–0.97 Å, O—H = 0.85 Å and N—H = 0.86 Å, and were refined as riding, with U_{iso}(H) set at 1.2 or 1.5 times U_{eq} of the parent atom.

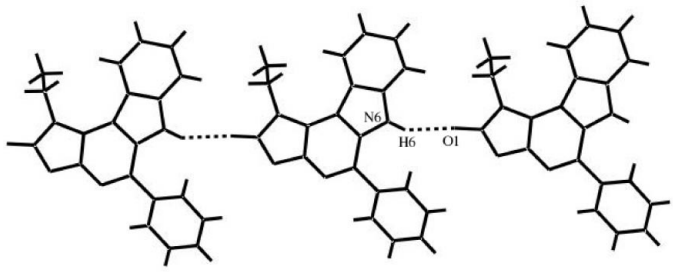


Figure 2
The one-dimensional chain of molecules linked by hydrogen bonds (dashed lines). Symmetry code of atom O1: $\frac{1}{2}+x, \frac{1}{2}+y, z$.

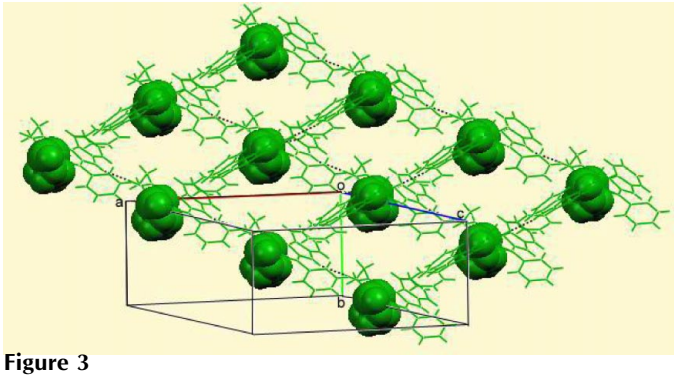


Figure 3
The crystal structure of (2).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Version 1.2.1; Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXTL*.

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References

Abu-Safieh, K. A., El-Abadelah, M. M., Sabri, S. S., Voelter, W. Mossmer, A. M. & Stroeble, M. (2002). *Z. Naturforsch. Teil B*, **57**, 1327–1332.
Bruker (2000). *SMART, SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Tepe, J. J., Madalengoitia, J. S., Slunt, K. M., Werbovetz, K. W., Spoors, P. G. & Macdonald, T. L. (1996). *J. Med. Chem.* **39**, 2188–2196.
Yu, G., Wang, S.-Z., Wang, K., Hu, Y.-F. & Hu, H.-W. (2004). *Synthesis*, **7**, 1021–1028.