Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,3,10,11-Tetramethoxy-15,16-diazapentaphene trihydrate

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Received 21 November 2006
Accepted 7 December 2006

Key indicators

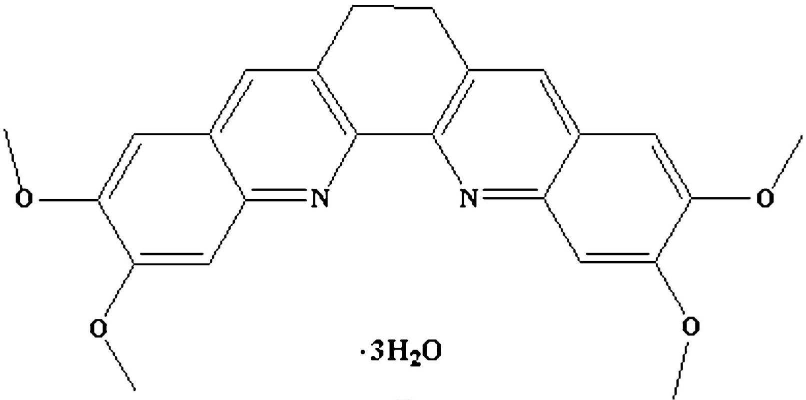
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.146
Data-to-parameter ratio = 16.9

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

In the title compound, $C_{24}H_{22}N_2O_4\cdot3H_2O$, the two quinoline ring systems are twisted with respect to each other; the dihedral angle is 31.9 (2)°. Adjacent heterocyclic molecules are linked *via* weak C—H···O hydrogen bonds.

Comment

Friedlander annulation, an acid- or base-catalysed condensation followed by a cyclodehydration between an aromatic 2-aminoaldehyde or ketone and a carbonyl compound containing a reactive α-methylene group, is one of the most simple and straightforward approaches for the synthesis of polysubstituted quinolines (Arisawa *et al.*, 2001; Cho *et al.*, 2000; Theoclitou & Robinson, 2002). Here we report the crystal structure of the title compound, (I), which was synthesized *via* Friedlander condensation.



(I)

The asymmetric unit of (I) is shown in Fig. 1. The title molecule is non-planar. The C17-containing ring adopts a half-chair conformation. The two quinoline ring systems are twisted with respect to each other; the dihedral angle is 31.9 (2)°. Adjacent molecules are linked *via* weak C—H···O hydrogen bonds (Table 1).

Experimental

To a mixture of 2-amino-4,5-dimethoxybenzaldehyde (0.88 mmol, 0.159 g) and 1,2-cyclohexanedione (0.44 mmol, 0.049 g) in absolute ethanol (5 ml) under N_2, sodium ethoxide (0.06 g, 0.88 mmol) in ethanol (5 ml) was added dropwise, and the mixture was refluxed for 7 h. After filtration, the filtrate was purified by chromatography on silica gel, eluting with ethanol–ethyl acetate (1:6 *v*/*v*). Recrystallization from an ethanol–ethyl acetate solution (1:6 *v*/*v*) gave single crystals of (I) (yield 0.108 g, 61%).

doi:10.1107/S1600536806053098

Crystal data

$C_{24}H_{22}N_2O_4 \cdot 3H_2O$
$M_r = 456.48$
Triclinic, $P\overline{1}$
$a = 7.9814\ (8)$ Å
$b = 11.3775\ (11)$ Å
$c = 13.1359\ (12)$ Å
$\alpha = 74.362\ (7)^\circ$
$\beta = 81.933\ (7)^\circ$
$\gamma = 77.998\ (7)^\circ$
$V = 1119.08\ (19)$ Å^3
$Z = 2$
$D_x = 1.355$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 298\ (2)$ K
Prism, brown
$0.12 \times 0.10 \times 0.07$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
9347 measured reflections
5092 independent reflections
3217 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\rm max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.146$
$S = 1.05$
5092 reflections
302 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.29$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O5—H5$A\cdots$O6	0.83	2.05	2.872 (2)	169
O5—H5$B\cdots$O7$^{\rm i}$	0.84	2.08	2.877 (3)	159
O6—H6$B\cdots$N1	0.84	2.16	2.987 (3)	167
O7—H7$A\cdots$O1$^{\rm ii}$	0.85	2.34	3.113 (2)	152
O7—H7$A\cdots$O2$^{\rm ii}$	0.85	2.24	2.944 (2)	140
O7—H7$B\cdots$N2	0.85	2.12	2.938 (2)	161
C18—H18$A\cdots$O4$^{\rm iii}$	0.97	2.42	3.282 (2)	147

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z$; (iii) $-x, -y, -z+1$.

Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(O). Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C). Other H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined in riding mode with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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: *SHELXTL*.

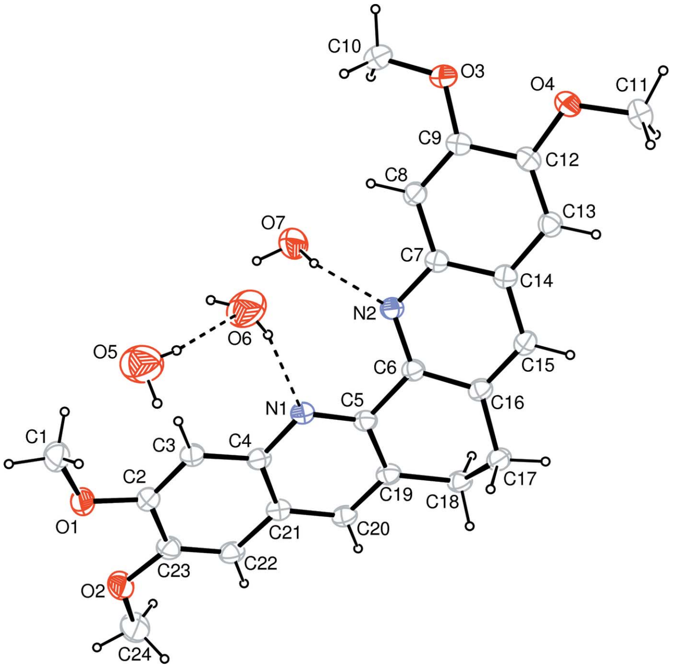


Figure 1
The asymmetric unit of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate hydrogen bonds.

The work was supported by the National Natural Science Foundation of China, and the Natural Science Foundation of Guangdong Province, China.

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