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8*H*-Benzo[*g*][1,3]benzodioxolo[6,5,4-*de*]-quinolin-8-one (oxocrebanine)

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Key indicators

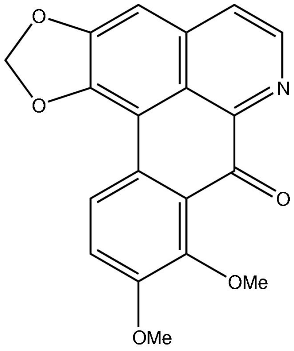
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.051
wR factor = 0.144
Data-to-parameter ratio = 12.8

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

In the title compound, $C_{19}H_{13}NO_5$, the five fused rings are essentially coplanar, while one of the O—Me bonds lies almost perpendicular to that plane. The molecule is stabilized by C—H···O intermolecular hydrogen bonds, which form zigzag polymeric chains parallel to the *b* axis.

Comment

Oxocrebanine (Fig. 1) is an oxoaporphine alkaloid found in plants such as *Stepania venosa* (Guinaudeau *et al.*, 1981), *Fissistigma glaucessens* (Wu *et al.*, 1990), *Goniothalamus amuyen* (Lu *et al.*, 1985) and *Illegera pentaphylla* (Ross *et al.*, 1985). The preferential activity of this compound towards the DNA repair-deficient mutant yeast strain RAD52Y and RS 321, compared with the wild-type DNA repair-proficient strain RAD$^+$ (Wijeratne *et al.*, 1996), implies that it may have anticancer activity. In the present study, oxocrebanine (I) was isolated from a plant of the genus Polyalthia.



(I)

The set of fused five- and six-membered rings in the parent moiety $C_{17}O_3N$ is essentially planar. The dioxoloisoquinoline fragment N1/C6*A*/C1*B*/C1*A*/C1/O1/C12/O2/C2/C3/C3*A*/C4/C5 shows a maximum deviation from planarity at C12 [0.072 (2) Å], the corresponding deviation for the C1*A*/C1*B*/C6*A*/C7/C7*A*/C8/C9/C10/C11/C11*A* ring system being 0.079 (2) Å at C6*A*. The dihedral angle between these two segments of the molecule is 5.39 (5)°. The O5/C14 methoxy group lies in the plane of the molecular ring systems [C8—C9—O5—C14 = 4.1 (3)°], while the O4—C13 bond is almost perpendicular to that plane [C13—O4—C8—C7*A* = 97.7 (2)°]. There is a weak intramolecular interaction, C11—H1*A*···O1, which may influence the planarity of the molecule. The solid-state structure is further stabilized by C12—H12*A*···O4^{iii} (Table 2) intermolecular hydrogen bonds to form dimers. These are further connected by C4—H4*A*···O3^{ii} interactions to form zigzag polymeric chains parallel to the *b* axis (Fig. 2).

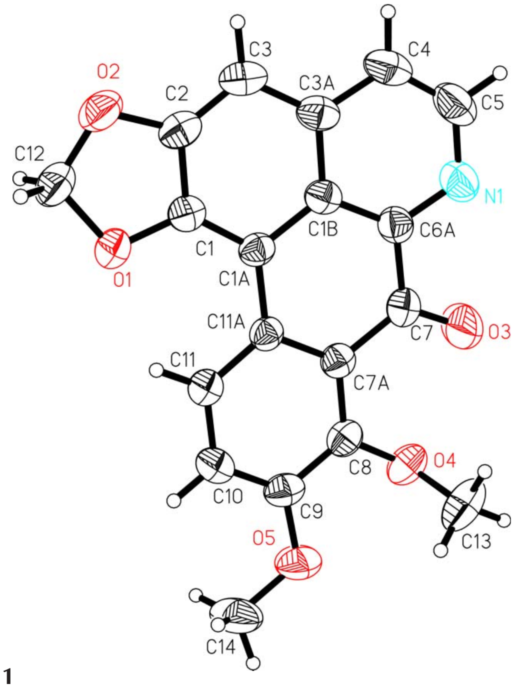


Figure 1
Molecular structure of the title compound, (I), with 50% probability displacement ellipsoids.

Experimental

The *Polyalthia bullata* (Annonaceae) species was collected from the Jambu Bongkok Reserved Forest, Terengganu, on the east coast of Peninsular Malaysia, and the voucher specimen (AZ 2004/2) was deposited at the herbarium of the Faculty of Science and Technology, Universiti Kebangsaan Malaysia. The air-dried, powdered roots of *Polyalthia bullata* (dry weight 1.0 kg) were successively Soxhlet extracted with petroleum ether, ethyl acetate and methanol. The ethyl acetate extract was subjected to acid–base extraction, and the alkaloidal fractions subjected to column chromatography over silica gel with a mixture of hexane and ethyl acetate as eluant. Early fractions were combined and further purified by chromatotron eluting with a mixture of chloroform and methanol. Orange–red crystals of (I) (0.15 g) were obtained upon evaporation of the purified solution. The melting point, 547.5–549.8 K (literature 546–548 K; Lu *et al.*, 1985), and the NMR data are in agreement with the values reported by Wijeratne *et al.* (1996).

Crystal data

$C_{19}H_{13}NO_5$
$M_r = 335.30$
Monoclinic, $P2_1/c$
$a = 8.370$ (2) Å
$b = 11.847$ (3) Å
$c = 14.825$ (4) Å
$\beta = 93.096$ (5)°
$V = 1467.8$ (7) Å^3
$Z = 4$

$D_x = 1.517$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 808 reflections
$\theta = 2.2$–1.5°
$\mu = 0.11$ mm^{-1}
$T = 298$ (2) K
Block, orange–red
0.39 × 0.31 × 0.28 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min} = 0.958$, $T_{\max} = 0.970$
8071 measured reflections

2894 independent reflections
2142 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 26.0$°
$h = -8 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$

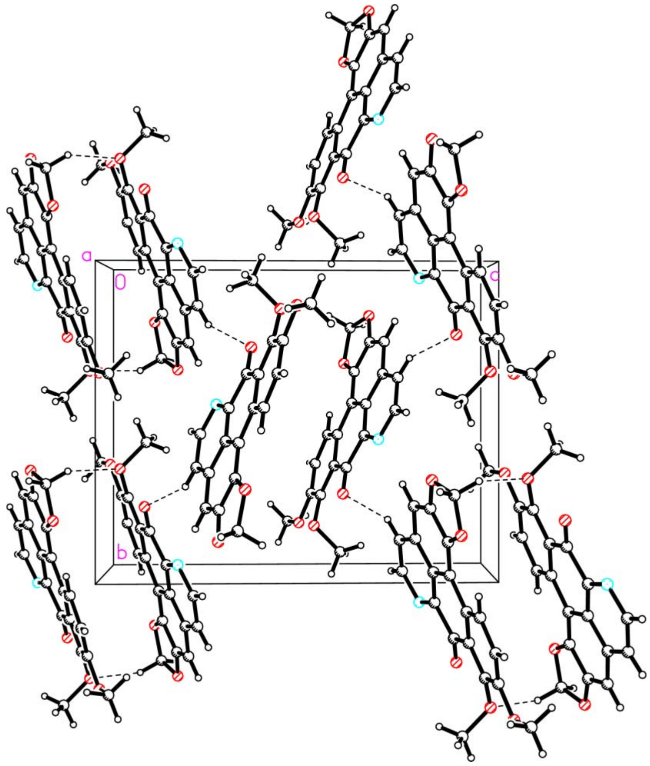


Figure 2
Packing diagram of the compound, viewed down the *b* axis. Dashed lines denote C—H···O hydrogen bonds.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.144$
$S = 1.06$
2894 reflections
226 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 0.1614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

O1—C1	1.364 (2)	C1—C1*A*	1.370 (2)
O1—C12	1.434 (2)	C2—C3	1.341 (3)
O2—C2	1.365 (2)	C4—C5	1.362 (3)
O2—C12	1.420 (2)	C8—C9	1.397 (3)
O3—C7	1.209 (2)	C9—O5	1.364 (2)
O4—C8	1.365 (2)	C9—C10	1.373 (3)
O4—C13	1.427 (2)	C10—C11	1.380 (2)
N1—C6*A*	1.327 (2)	C14—O5	1.424 (3)
N1—C5	1.347 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···O1$^{\rm i}$	0.93	2.18	2.874 (2)	131
C13—H13*B*···O5$^{\rm i}$	0.96	2.58	3.066 (3)	112
C4—H4*A*···O3$^{\rm ii}$	0.93	2.52	3.354 (3)	149
C12—H12*A*···O4$^{\rm iii}$	0.97	2.45	3.115 (3)	126

Symmetry codes: (i) x, y, z; (ii) $-x+2, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y+2, -z$.

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After their location in a difference map, all the H atoms were placed at ideal positions, with C—H = 0.93–0.97 Å, and allowed to ride on the parent C atom with $U_{\rm iso}$(H) set at $1.2U_{\rm eq}$ (CH and CH_2) and $1.5U_{\rm eq}$ (CH_3) of the parent atom.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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