9,10-Dimethoxy-4-methyl-1,2-dihydro-1-azaanthracen-2-one (kalasinamide), a new azaanthracene alkaloid

Pongchan Thinapong *et al.*

Electronic paper

This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

9,10-Dimethoxy-4-methyl-1,2-di-hydro-1-azaanthracen-2-one (kalasin-amide), a new azaanthracene alkaloid

Pongchan Thinapong,* Orapin Rangsiman, Patoomratana Tuchinda, Bamroong Munyoo, Manat Pohmakotr and Vichai Reutrakul

Department of Chemistry, Faculty of Science, Mahidol University, Rama 6 Road, Bangkok 10400, Thailand
Correspondence e-mail: scpth@mahidol.ac.th

A new azaanthracene alkaloid, namely 9,10-dimethoxy-4-methyl-1,2-dihydro-1-azaanthracen-2-one (kalasinamide), $C_{16}H_{15}NO_3$, has been isolated from the stems of *Polyalthia suberosa* collected in the northeastern part of Thailand. Each of the aromatic rings in the molecule is planar within ±0.021 (2) Å. Molecules are linked to form centrosymmetric dimers by N—H···O hydrogen bonds [N···O 2.941 (4) Å].

Comment

Polyalthia suberosa (Roxb.), a shrubby tree native to southeast Asia and South China, has been widely investigated since 1985. Various parts of the tree, leaves, stems and barks have been investigated. The compounds identified are coumarin, sterols (Dan *et al.*, 1985; Goyal & Gupta, 1986), alkaloids (Ferdous *et al.*, 1992; Sahai *et al.*, 1996), triterpenes (Goyal & Gupta, 1986; Li *et al.*, 1993) and nitrogen heterocyclic compounds (Sahai *et al.*, 1996, Tuchinda *et al.*, 2000). Studies of suberosa has shown an anti-HIV principle (Li *et al.*, 1993).

The title compound, (I), extracted from the stem of the tree from Kalasin Province in the northeastern part of Thailand was found to be a new azaanthracene alkaloid (Tuchinda *et al.*, 2000). We therefore report the structure of the title compound as this might lead to a new anti-HIV active compound.

(I)
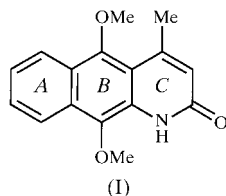
Bond lengths and angles within the anthracene rings system of (I) are in good agreement with literature values (Brown *et al.*, 1964). However, the C2=O2 bond length of 1.237 (6) Å is slightly longer than that normally found in ketones. Also, the N1—C2 and N1—C91 mean distance of 1.373 (6) Å is slightly shorter than the C—N bond length in heteroaromatic systems (Allen *et al.*, 1987). Appreciable electron delocalizations therefore occur here. Noticable electron delocalizations also occur at O1 and O3; the C9—O1 and O1—C12 bond lengths are 1.373 (6) and 1.435 (1) Å, and C10—O3 and O3—C13 are 1.369 (5) and 1.430 (2) Å, respectively. Mean planes through each of the three fused six-membered rings show that they are planar within ±0.02 Å. The dihedral angles *A/B* 3.00, *B/C* 2.2 and *A/C* 5.15° [*A* = C5—C6—C7—C8—C81—C101, *B* = C81—C9—C91—C41—C10—C101 and *C* = C91—N1—C2—C3—C4—C41] indicate that the planes are not quite coplanar. Mean-plane calculation through the 14-membered fused *A/B/C* ring shows a total puckering amplitude of 0.166 (3) Å. The torsion angles C13—O3—C10—C41 and C12—O1—C9—C91 of 102.5 (3) and 94.8 (3)°, respectively, are as expected. The methyl group at C4 has a steric effect on the methoxy group at C10. Molecules are linked by N—H···O hydrogen bonds about inversion centres to form centrosymmetric dimers with an N···O distance of 2.941 (4) Å.

Experimental

The orange title compound was crystallized from EtOH–CH$_2$Cl$_2$.

Crystal data

$C_{16}H_{15}NO_3$	$D_x = 1.380$ Mg m^{-3}
$M_r = 269.300$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 9
$a = 10.005$ (5) Å	reflections
$b = 16.449$ (5) Å	$\theta = 8.8300–11.7400°$
$c = 8.186$ (5) Å	$\mu = 0.096$ mm^{-1}
$\beta = 105.8$ (2)°	$T = 298$ K
$V = 1297.0$ (10) Å^3	Needle, orange
$Z = 4$	$0.250 \times 0.040 \times 0.025$ mm

Data collection

Enraf–Nonius CAD-4 diffract-ometer	$\theta_{max} = 24.93°$
ω–2θ scans	$h = -11 \rightarrow 11$
9602 measured reflections	$k = -19 \rightarrow 19$
2272 independent reflections	$l = -9 \rightarrow 9$
1785 reflections with $I > 2\sigma(I)$	3 standard reflections
$R_{int} = 0.066$	frequency: 2608 min
	intensity decay: 0.81%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2$
$R(F) = 0.053$	$+ 0.7373P]$
$wR(F^2) = 0.163$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.069$	$(\Delta/\sigma)_{max} = 0.001$
2272 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
184 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^i	0.86	2.10	2.941 (4)	164

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Based on a statistical analysis of intensity distribution, the space group was determined to be $P2_1/a$ and has been transformed to the conventional space group of $P2_1/c$. Friedel opposites were collected and merged. The H atoms were allowed in the refinement at geometrically idealized positions.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *maXus* (Mackay *et al.*, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer. L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Brown, D. S., Wallwork, S. C. & Wilson, A. (1964). *Acta Cryst.* **17**, 168–176.

Dan, S., Dan, S. S., Mukhopadhyay, P. & Mukherjee, M. K. (1985). *Int. J. Crude Drug Res.* **23**(2), 73–76.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Ferdous, A. J., Islam, M. O. & Hasan, C. M. (1992). *J. Bangladesh Acad. Sci.* **16**, 99–103.

Goyal, M. M. & Gupta, A. (1986). *Acta Cienc. Indica Chem.* **12**, 152–154.

Li, H. Y., Sun, N.-J., Kashiwada, Y., Sun, L., Snider, J. V., Cosentino, L. M. & Lee, K. H. (1993). *J. Nat. Prod.* **56**, 1130–1133.

Mackay, S., Edwards, C., Henderson, A., Gilmore, C., Stewart, N., Shankland, K. & Donald, A. (1999). *maXus*. University of Glasgow, Scotland.

Sahai, M., Srivastava, A., Jamal, P., Sinha, S., Singh A. P. & Fujimoto, Y. (1996). *Indian J. Chem. Ser. B*, **35**, 510–511.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Tuchinda, P., Pohmakotr, M., Munyoo, B., Reutrakul, V. & Santisuk, T. (2000). *Phytochemistry*, **53**, 1079–1082.