acknowledged for support of this research. CHM also wishes to thank PRF/ACS for a Summer Research Faculty Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1169). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bentley, K. W. (1971). *The Alkaloids*, Vol. 13, edited by R. H. F. Manske, pp. 1–163. New York: Academic Press.

Bye, E. (1976). *Acta Chem. Scand. Ser. B*, **30**, 549–554.

Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Gylbert, L. (1973). *Acta Cryst.* B**29**, 1630–1635.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Karle, I. L. (1974). *Acta Cryst.* B**30**, 1682–1686.

Kartha, G., Ahmed, F. R. & Barnes, W. H. (1962). *Acta Cryst.* **15**, 326–333.

Klein, C. L., Majeste, R. J. & Stevens, E. D. (1987). *J. Am. Chem. Soc.* **109**, 6675–6681.

Lentz, G. R., Evans, S. M., Walter, D. E. & Hopfinger, A. J. (1986). *Opiates*. New York: Academic Press.

Mackay, M. & Hodgkin, D. C. (1955). *J. Chem. Soc.* pp. 3261–3267.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1996). C**52**, 3195–3196

(*Z*)-*N*-Formylnornuciferin Isolated from *Piper argyrophylum*

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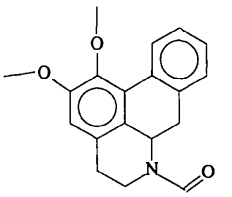
Abstract

The title compound (systematic name: 4,5,6a,7-tetrahydro-1,2-dimethoxy-6*H*-dibenzo[*de*,*g*]quinoline-6-carboxaldehyde), $C_{19}H_{19}NO_3$, was isolated as a minor component from the methanol extract of stems of *P. argyrophylum Miq.* The molecules are non-planar and the two benzene ring planes are at an angle of 21.9 (1)°. The *N*-formyl group and the two neighbouring C atoms constitute a planar fragment. The C—O—C planes of the two methoxy groups are nearly perpendicular to each other.

Comment

In a phytochemical investigation of the methanol extract of stems from *P. argyrophylum Miq.*, 25 compounds were isolated (Singh, Prasad, Olsen, Jha, Jain, Parmar & Wengel, 1996). One of the isolated compounds was the title compound (I) which could not be identified



(I)

immediately from spectroscopic data and so a structure determination by X-ray diffraction was undertaken. The identity of (I) was confirmed further by NMR spectroscopy (Pachaly, Adnan & Will, 1992). Compound (I) has three planar domains, the two benzene rings (C1–C4, C16, C17 and C5–C10) and the *N*-formyl group with the two neighbouring atoms (O20, C20, N13, C12, C14). The Csp^3 atoms C11, C12, C14 and C15 force the molecule to be non-planar; this is reflected in the torsion angles [C10—C11—C12—C17 59.1 (4), C12—N13—C14—C15 −58.9 (4)°] and in the angle of 21.9 (1)° between the least-squares planes of the benzene rings. The C—O—C planes of the two methoxy groups are nearly perpendicular to each other, with torsion angles

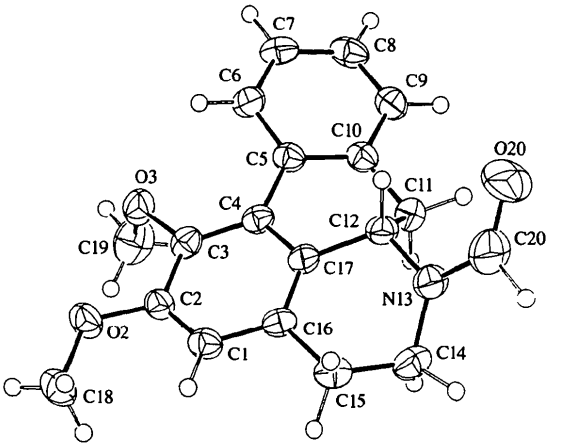


Fig. 1. Displacement-ellipsoid plot of (I) with ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of an arbitrary radius.

C18—O2—C2—C3 176.3 (3) and C19—O3—C3—C2 −83.9 (4)°. The dimensions of the *N*-formyl group (Table 2) are in agreement with those found in related compounds (van Koningsveld & Olieman, 1981; Pelletier, Desai, Finer-Moore & Moody, 1982; Roche, Roche, Nagel & McPhail, 1984). The absolute configuration of (I) was not determined.

Experimental

The title compound was purified by repetitive preparative TLC (Singh, Prasad, Olsen, Jha, Jain, Parmar & Wengel, 1996). Crystals suitable for X-ray investigation were obtained by evaporation of the solvent at room temperature from a methoxyethanol solution.

Crystal data

$C_{19}H_{19}NO_3$
M_r = 309.36
Orthorhombic
$P2_12_12_1$
a = 6.276 (1) Å
b = 15.232 (1) Å
c = 16.616 (2) Å
V = 1588.4 (4) Å^3
Z = 4
D_x = 1.294 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 11.43–13.54°
μ = 0.08 mm^{-1}
T = 293 K
Needle
0.42 × 0.16 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4*F* diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration from crystal shape
T_{min} = 0.985, T_{max} = 0.994
2047 measured reflections
2047 independent reflections
1396 observed reflections [$I > 2.5\sigma(I)$]
θ_{max} = 27.96°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 21$
1 standard reflection
frequency: 90 min
intensity decay: 2.5%

Refinement

Refinement on F
R = 0.041
wR = 0.028
S = 2.484
1396 reflections
265 parameters
H atoms from $\Delta\rho$ map; only coordinates of H atoms refined, U_{iso}(fixed) = 0.035 Å^2
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = 0.23
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O2	0.7589 (4)	0.4088 (2)	0.4091 (1)	0.045 (2)
O3	0.4303 (4)	0.4926 (2)	0.4685 (1)	0.042 (2)
O20	0.3902 (5)	0.2311 (2)	0.8517 (2)	0.087 (3)
N13	0.6556 (5)	0.2921 (2)	0.7779 (2)	0.041 (2)
C1	0.8268 (6)	0.3338 (2)	0.5358 (2)	0.036 (2)
C2	0.7126 (6)	0.3898 (2)	0.4879 (2)	0.035 (2)
C3	0.5356 (6)	0.4355 (2)	0.5193 (2)	0.034 (2)
C4	0.4726 (6)	0.4229 (2)	0.5989 (2)	0.031 (2)
C5	0.2937 (6)	0.4722 (2)	0.6371 (2)	0.033 (2)
C6	0.1265 (6)	0.5110 (2)	0.5943 (2)	0.038 (2)
C7	−0.0313 (6)	0.5584 (3)	0.6324 (2)	0.043 (2)
C8	−0.0277 (6)	0.5668 (3)	0.7152 (3)	0.043 (2)
C9	0.1300 (6)	0.5272 (3)	0.7595 (2)	0.039 (2)
C10	0.2918 (6)	0.4798 (2)	0.7218 (2)	0.033 (2)
C11	0.4603 (6)	0.4338 (2)	0.7705 (2)	0.038 (2)
C12	0.5102 (6)	0.3462 (2)	0.7312 (2)	0.034 (2)
C14	0.8811 (6)	0.2940 (3)	0.7572 (3)	0.041 (2)
C15	0.9055 (6)	0.2654 (3)	0.6701 (2)	0.040 (2)
C16	0.7678 (6)	0.3212 (2)	0.6158 (2)	0.033 (2)
C17	0.5870 (6)	0.3625 (2)	0.6458 (2)	0.031 (2)
C18	0.9470 (8)	0.3665 (3)	0.3789 (3)	0.048 (3)
C19	0.5226 (9)	0.5778 (3)	0.4634 (3)	0.062 (3)
C20	0.5780 (8)	0.2379 (3)	0.8342 (3)	0.062 (3)

Table 2. *Selected geometric parameters (Å, °)*

O20—C20	1.218 (6)	N13—C14	1.457 (5)
N13—C12	1.454 (5)	N13—C20	1.340 (6)
C12—N13—C14	118.2 (3)	C14—N13—C20	122.0 (4)
C12—N13—C20	119.6 (3)	O20—C20—N13	124.7 (4)

Data collection: *CAD-4F Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4F Software*. Data reduction: Norrestam & Nielsen (1982) and *Xtal*3.2 (Hall, Flack & Stewart, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *Xtal*3.2. Molecular graphics: *Xtal*3.2. Software used to prepare material for publication: *Xtal*3.2.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and a data-comparison table have been deposited with the IUCr (Reference: BM1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1989). *CAD-4F Software*. Enraf–Nonius, Delft, The Netherlands.
Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal*3.2 *Reference Manual*. Universities of Western Australia, Australia, and Maryland, USA.
Koningsveld, H. van & Olieman, C. (1981). *Cryst. Struct. Commun.* **10**, 541–546.
Norrestam, R. & Nielsen, K. (1982). Technical University of Denmark. Private communication.
Pachaly, P., Adnan, A. Z. & Will, G. (1992). *Planta Med.* **58**, 184–187.
Pelletier, S. W., Desai, H. K., Finer-Moore, J. & Moody, N. V. (1982). *Tetrahedron Lett.* **23**, 4229–4232.
Roche, V. F., Roche, E. B., Nagel, D. L. & McPhail, A. T. (1984). *J. Org. Chem.* **49**, 3881–3887.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Singh, S. K., Prasad, A. K., Olsen, C. E., Jha, A., Jain, S. C., Parmar, V. S. & Wengel, J. (1996). *Phytochemistry*. In the press.