droxy and carbonyl O atoms [O17···O11^{i} 2.967 (2), H17O···O11^{i} 2.15 (3) Å and O17—H17O···O11^{i} 164 (2)°; symmetry code: (i) $1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$].

Experimental

Ring opening of the compound 4-methyl-7,10-dihydro-8,9-epoxy-7,8-benzocoumarin with thiophenyl resulted in the title compound (Sriraghavan, 1997). Single crystals were grown by slow evaporation of the solvent from a solution of the compound in chloroform–methanol.

Crystal data

$C_{20}H_{18}O_3S$
$M_r = 338.40$
Monoclinic
$P2_1/c$
$a = 5.3861\ (6)$ Å
$b = 11.1410\ (11)$ Å
$c = 27.411\ (3)$ Å
$\beta = 93.433\ (8)°$
$V = 1641.9\ (4)$ Å^3
$Z = 4$
$D_x = 1.369$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 37 reflections
$\theta = 5.29–12.48°$
$\mu = 0.212$ mm^{-1}
$T = 293\ (2)$ K
Thick plate
0.66 × 0.38 × 0.14 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4309 measured reflections
2899 independent reflections
2236 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$

$\theta_{\rm max} = 25°$
$h = -1 \rightarrow 6$
$k = -1 \rightarrow 13$
$l = -32 \rightarrow 32$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.118$
$S = 1.032$
2898 reflections
289 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.194$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.185$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C12—C7—C8—C15	3.7 (3)	C12—C13—C14—C15	−63.2 (2)
C8—C7—C12—C13	−16.7 (3)	C7—C8—C15—C14	−20.7 (3)
C7—C12—C13—C14	46.0 (2)	C13—C14—C15—C8	49.8 (2)

Data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994). Structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Structure refinement: *SHELXL*93 (Sheldrick, 1993). Geometrical calculations: *PARST* (Nardelli, 1983*b*).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1193). Services for accessing these data are described at the back of the journal.

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5-Amino-1,6-dimethyl-1,2-dihydroquinolin-2-one Monohydrate

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Abstract

The quinolone ring system of the title compound, $C_{11}H_{12}N_2O.H_2O$, is essentially planar and the water molecule links neighbouring molecules *via* hydrogen bonds.

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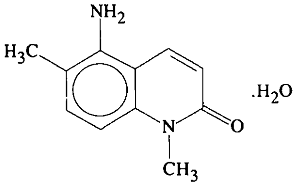
Comment

Carbostyril derivatives typically emit laser radiation in the spectral region from 400 to 430 nm (Maeda, 1984). Substituent effects play a vital role in the lasing action as they can be exploited in order to control laser efficiency, tunability and photostability (Hammond, Fletcher, Henry & Atkins, 1975). With this in view, the title compound, (I), was synthesized as a potential laser dye.



(I)

Bond lengths and valence angles are normal with the C3—C4 bond length of 1.341 (2) Å (Fig. 1) indicating a localized double bond (Chinnakali, Sivakumar, Natarajan, McGuire & Clearfield, 1991; Sudha, Subramanian, Sivaraman, Sriraghavan & Steiner, 1995). The molecule is planar within 0.034 (1) Å. The amino N13 atom is coplanar with the quinolone moiety, whereas methyl atoms C12 and C14 deviate by 0.113 (2) and 0.111 (2) Å, respectively. Water molecules form O—H···O, N—H···O and C—H···O hydrogen bonds between neighbouring molecules (Table 1).

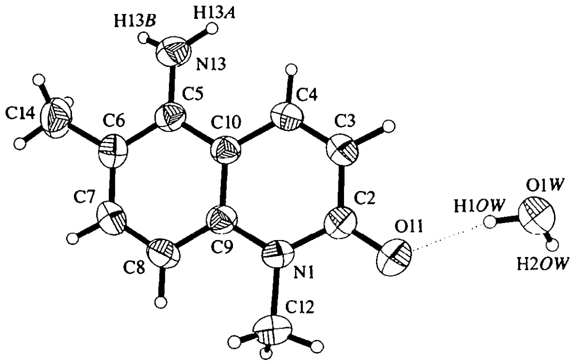


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as small spheres of arbitrary radii.

Experimental

Nitration of 1,6-dimethylquinol-2-one using urea nitrate–H_2SO_4 furnished 1,6-dimethyl-5-nitroquinol-2-one, which, on reduction with iron–acetic acid, furnished the title compound (Sriraghavan & Ramakrashnan, 1997). Single crystals were grown by slow evaporation of the compound from a 1:1 chloroform and methanol solvent system.

Crystal data

$C_{11}H_{12}N_2O.H_2O$
$M_r = 206.24$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Monoclinic
$P2_1/c$
$a = 9.233\ (1)$ Å
$b = 15.283\ (1)$ Å
$c = 7.448\ (1)$ Å
$\beta = 98.08\ (1)°$
$V = 1040.5\ (2)$ Å^3
$Z = 4$
$D_x = 1.317$ Mg m^{-3}
D_m not measured
Cell parameters from 40 reflections
$\theta = 5.44–12.48°$
$\mu = 0.092$ mm^{-1}
$T = 293\ (2)$ K
Rectangular
$0.80 \times 0.60 \times 0.50$ mm
Orange

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3104 measured reflections
2367 independent reflections
1814 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\rm max} = 27.50°$
$h = -11 \rightarrow 11$
$k = -19 \rightarrow 1$
$l = -1 \rightarrow 9$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.127$
$S = 1.070$
2367 reflections
193 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.0588P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.021 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1OW···O11	0.86 (2)	1.94 (2)	2.786 (2)	167 (2)
O1W—H2OW···O11$^{\rm i}$	0.88 (2)	2.06 (2)	2.928 (2)	169 (2)
N13—H13A···O1$W^{\rm ii}$	0.93 (2)	2.15 (2)	3.026 (2)	156 (2)
N13—H13B···O1$W^{\rm iii}$	0.87 (2)	2.43 (2)	3.239 (2)	155 (2)

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

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically. Uncertainties on bond lengths are 0.002 Å or lower.

Data collection, cell refinement, data reduction: *XSCANS* (Siemens, 1994); structure solution, molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); structure refinement: *SHELXL*93 (Sheldrick, 1993); geometrical calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1174). Services for accessing these data are described at the back of the journal.

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Bis(3,4-dimethoxybenzyl) Ether and Tetramethoxy-4,4'-(2,3-dimethyltetramethylene)dipyrocatechol†

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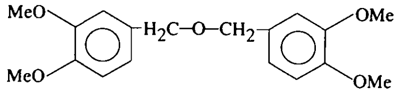
Abstract

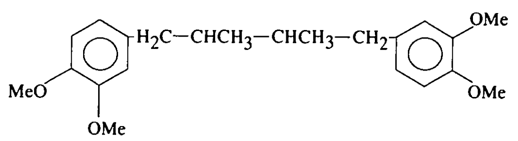
In molecules of both bis(3,4-dimethoxybenzyl) ether, $C_{18}H_{22}O_5$, (I), and *meso*-tetramethoxy-4,4'-(2,3-dimethyltetramethylene)dipyrocatechol, $C_{22}H_{30}O_4$, (II), the methoxy groups are almost coplanar with the attached benzene rings. In (I), the two benzene rings make a dihedral angle of 17.20(5)°, whereas in (II), they are almost perpendicular [94.8(4)°] to one another. The crystal structure is stabilized by van der Waals interactions in both compounds.

Comment

The structure determinations of the title compounds, (I) and (II), represent part of an investigation of a series of methoxybenzenes.



(I)



(II)

The mean C_{sp^2}—O [1.366(2) Å] and C_{sp^3}—O [1.423(3) Å] bond distances in the methoxy groups agree with values observed in other methoxybenzene derivatives (Fun, Chinnakali, Sivakumar, Sam & How, 1997; Bryan & White, 1982). The methoxy groups are almost coplanar with the attached benzene rings but significant displacements from their adjacent phenyl rings are observed for C15 (0.120 Å) and C18 (0.239 Å) in (I),

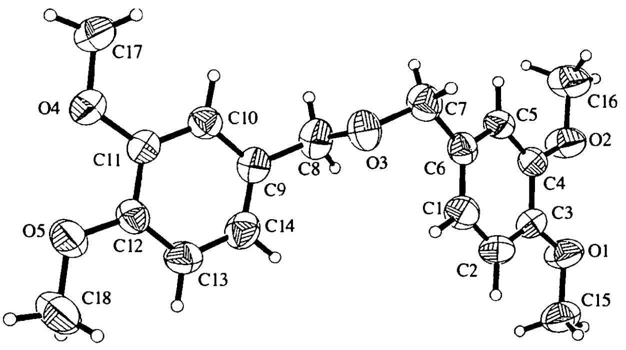


Fig. 1. View of (I) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

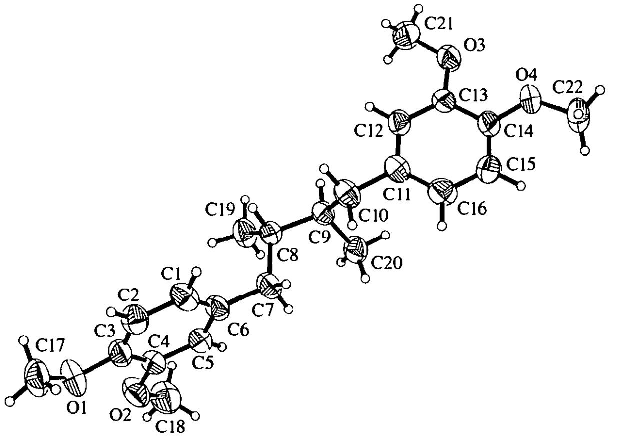


Fig. 2. View of (II) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

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† Alternative name: 1,1',2,2'-tetramethoxy-4,4'-(2,3-dimethyltetramethylene)dibenzene.

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