Crystal structure of 5-methoxy-6-hydroxy-2,3-dihydro-7*H*-dibenzo[*de*,*h*]-quinolin-7-one, $C_{17}H_{13}NO_3$

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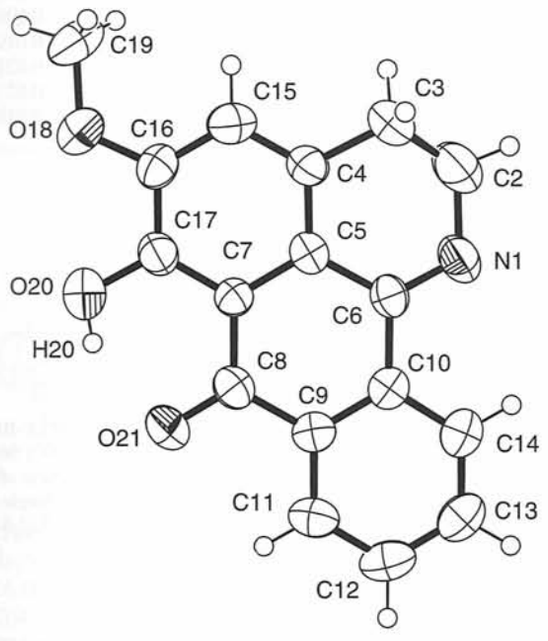

Abstract

$C_{17}H_{13}NO_3$, orthorhombic, $P2_1nb$ (No. 33), a = 8.7927(8) Å, b = 30.165(2) Å, c = 4.9136(6) Å, V = 1303.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.053, $wR_{ref}(F^2)$ = 0.161, T = 293 K.

Source of material

To a solution of phthalaldehydic acid (23 g, 153 mmol) in toluene (150 mL) was added homoveratrylamine (25 mL, 153 mmol) and the mixture was refluxed with stirring under a Dean-Stark trap for 2 hours at 413 K. The reaction mixture was then treated with polyphosphoric acid (80 g) and kept at 373 K for 10 minutes. The red mixture was dissolved in 100 mL water, neutralized with NH_3 and extracted with chloroform (700 ml). The chloroform extract was dried over anhydrous sodium sulphate, concentrated to dryness, and the residue subjected to flash chromatography on silica gel, eluting with hexane-ethyl acetate 95:5 (v/v) to give 5-methoxy-6-hydroxy-2,3-dihydro-7*H*-dibenzo[*de*,*h*]quinolin-7-one (3.83 g, yield 9%) crystallized in MeOH as red needles.

Experimental details

The low ratio of $N(hkl)_{gt}/N(param)$ of about 5 is due to the low quality of the crystal and to its partial descomposition.

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Discussion

The 5-methoxy-6-hydroxy-2,3-dihydro-oxoisoaporphine molecule is largely planar with two methylene carbons, C2 and C3, forming a torsion angle C6–N1–C2–C3 of 25.9(10)°. The methoxyl group carbon is coplanar with the aromatic ring with a C16–O18–C19 angle of 117.5(4)°. An intramolecular hydrogen bond is also observed d(O21···H20) = 1.824 Å since the bond d(H20—O20) is considerably shorter (0.820Å).

Table 1. Data collection and handling.

Crystal:	red prism, size 0.12 × 0.16 × 0.56 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	8.05 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	146.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1493, 1472
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1093
$N(param)_{refined}$:	193
Programs:	SIR-97 [1], SHELXL-97 [2], ORTEP-3 [3], WinGX [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2A)	4*a*	0.2483	0.5856	0.4957	0.120
H(2B)	4*a*	0.3582	0.5696	0.7246	0.120
H(3A)	4*a*	0.3147	0.6348	0.8385	0.103
H(3B)	4*a*	0.3096	0.6521	0.5385	0.103
H(11)	4*a*	1.0544	0.5850	–0.0316	0.066
H(12)	4*a*	0.9909	0.5268	–0.3196	0.074
H(13)	4*a*	0.7483	0.4970	–0.3005	0.071
H(14)	4*a*	0.5739	0.5240	0.0006	0.066
H(15)	4*a*	0.4926	0.6976	0.9669	0.067
H(19A)	4*a*	0.6042	0.7304	1.3022	0.109
H(19B)	4*a*	0.5892	0.7710	1.1048	0.109
H(19C)	4*a*	0.7131	0.7712	1.3343	0.109
H(20)	4*a*	1.0205	0.6820	0.6045	0.122

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*a*	0.4574(4)	0.5726(1)	0.360(1)	0.045(2)	0.063(2)	0.101(4)	–0.010(2)	0.009(3)	–0.014(3)
C(2)	4*a*	0.3503(5)	0.5891(2)	0.568(2)	0.058(3)	0.092(4)	0.150(8)	–0.020(3)	0.028(4)	–0.031(5)
C(3)	4*a*	0.3639(5)	0.6327(2)	0.662(2)	0.045(2)	0.111(4)	0.100(5)	–0.010(3)	0.012(3)	–0.036(4)
C(4)	4*a*	0.5239(4)	0.6493(1)	0.690(1)	0.039(2)	0.063(2)	0.052(3)	0.001(2)	0.001(2)	–0.007(2)
C(5)	4*a*	0.6324(4)	0.6292(1)	0.521(1)	0.039(2)	0.042(2)	0.043(2)	–0.001(1)	–0.003(2)	0.008(2)
C(6)	4*a*	0.5878(4)	0.5914(1)	0.350(1)	0.039(2)	0.049(2)	0.048(2)	–0.001(2)	–0.006(2)	0.002(2)
C(7)	4*a*	0.7818(4)	0.6456(1)	0.5189(9)	0.039(2)	0.038(2)	0.041(2)	0.002(1)	–0.001(2)	0.001(2)
C(8)	4*a*	0.8949(4)	0.6268(1)	0.336(1)	0.044(2)	0.048(2)	0.051(3)	–0.002(2)	0.000(2)	0.006(2)
C(9)	4*a*	0.8504(4)	0.5902(1)	0.1522(9)	0.049(2)	0.040(2)	0.043(2)	0.006(2)	0.001(2)	0.004(2)
C(10)	4*a*	0.7034(4)	0.5726(1)	0.1618(9)	0.049(2)	0.040(2)	0.041(2)	0.003(2)	–0.004(2)	0.004(2)
C(11)	4*a*	0.9569(5)	0.5730(1)	–0.027(1)	0.056(2)	0.055(2)	0.054(3)	0.007(2)	0.014(2)	0.007(2)
C(12)	4*a*	0.9195(6)	0.5381(1)	–0.198(1)	0.080(3)	0.054(2)	0.052(3)	0.017(2)	0.014(3)	0.000(2)
C(13)	4*a*	0.7746(5)	0.5203(1)	–0.186(1)	0.079(3)	0.047(2)	0.051(3)	0.006(2)	–0.006(3)	–0.004(2)
C(14)	4*a*	0.6699(5)	0.5369(1)	–0.008(1)	0.062(2)	0.048(2)	0.054(3)	–0.001(2)	–0.008(3)	–0.001(2)
C(15)	4*a*	0.5656(5)	0.6842(1)	0.857(1)	0.056(2)	0.061(3)	0.052(3)	0.010(2)	0.004(3)	–0.004(2)
C(16)	4*a*	0.7129(5)	0.6994(1)	0.864(1)	0.055(2)	0.049(2)	0.047(2)	0.001(2)	–0.007(2)	–0.005(2)
C(17)	4*a*	0.8219(4)	0.6805(1)	0.692(1)	0.043(2)	0.050(2)	0.055(3)	–0.003(2)	0.000(2)	0.000(2)
O(18)	4*a*	0.7648(3)	0.7329(1)	1.0245(9)	0.073(2)	0.062(2)	0.066(2)	0.000(2)	0.001(2)	–0.019(2)
C(19)	4*a*	0.6592(6)	0.7530(2)	1.206(1)	0.092(4)	0.065(3)	0.060(3)	0.022(3)	–0.005(3)	–0.021(3)
O(20)	4*a*	0.9639(4)	0.6977(1)	0.696(1)	0.067(2)	0.087(2)	0.091(3)	–0.022(2)	0.006(2)	–0.027(2)
O(21)	4*a*	1.0283(3)	0.6412(1)	0.331(1)	0.045(2)	0.075(2)	0.091(3)	–0.010(1)	0.014(2)	–0.018(2)

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