

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors (\AA^2) with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
O(1)	0.1762 (2)	0.2957 (2)	0.6906 (1)	0.0251
O(2)	0.2936 (2)	0.1747 (2)	0.5382 (1)	0.0282
N(1)	-0.0399 (2)	0.4862 (2)	0.8453 (1)	0.0257
C(1)	0.1673 (2)	0.2368 (2)	0.5901 (1)	0.0181
C(2)	0.0000	0.2500	0.5254 (2)	0.0162
C(3)	0.0159 (3)	0.3401 (2)	0.4109 (2)	0.0260
H(1a)	-0.098 (3)	0.424 (3)	0.899 (2)	0.051 (3)†
H(1b)	-0.136 (3)	0.558 (3)	0.811 (2)	0.051 (3)†
H(1c)	0.045 (3)	0.549 (3)	0.879 (2)	0.051 (3)†
H(1d)	0.010 (3)	0.421 (3)	0.782 (2)	0.051 (3)†
H(3a)	0.138 (3)	0.381 (3)	0.391 (2)	0.051 (3)†
H(3b)	-0.080 (3)	0.420 (3)	0.390 (2)	0.051 (3)†

† Isotropic U .

Table 2. Interatomic distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

O(1)—C(1)	1.259 (2)	C(2)—C(3)	1.517 (3)
O(2)—C(1)	1.256 (2)	C(3)—C(3')	1.474 (4)
C(1)—C(2)	1.505 (2)		
O(2)—C(1)—O(1)	123.3 (2)	C(3)—C(2)—C(1')	118.1 (1)
C(2)—C(1)—O(1)	118.7 (2)	C(3)—C(2)—C(1)	113.4 (1)
C(2)—C(1)—O(2)	117.9 (1)	C(3)—C(2)—C(3')	58.1 (2)
C(1)—C(2)—C(1')	120.4 (2)	C(3)—C(3')—C(2)	60.93 (8)
Hydrogen bonds			
O(1)⋯N(1)	2.897 (2)	O(2)⋯N(1 ⁱⁱⁱ)	2.890 (2)
O(1)⋯H(1d)	1.95 (3)	O(2)⋯H(1d ⁱⁱⁱ)	1.99 (3)
O(1)⋯H(1d)—N(1)	161 (1)	O(2)⋯H(1d ⁱⁱⁱ)—N(1 ⁱⁱⁱ)	168 (2)
O(1)⋯N(1 ⁱⁱ)	2.855 (2)	O(2)⋯N(1 ⁱⁱ)	2.794 (2)
O(1)⋯H(1b ⁱⁱ)	1.88 (3)	O(2)⋯H(1d ⁱⁱ)	1.87 (3)
O(1)⋯H(1b ⁱⁱ)—N(1 ⁱⁱ)	158 (2)	O(2)⋯H(1d ⁱⁱ)—N(1 ⁱⁱ)	175 (2)

Symmetry codes: (i) $-x, \frac{1}{2}-y, z$; (ii) $\frac{1}{2}+x, 1-y, \frac{3}{2}-z$; (iii) $\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$; (iv) $\frac{1}{2}-y, y-\frac{1}{2}, \frac{3}{2}-z$.

Colourless needles of the compound were crystallized from water/ethanol at 277 K. The structure was solved by direct methods (SHELXS86; Sheldrick, 1986) and refined by full-matrix least squares.

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Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55269 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1005]

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Structure of the 1:1 Complex of 6,6'-Diquinolyl Ether with 5,5-Diethylbarbituric Acid

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Abstract

The crystal structure shows that barbital (5,5-diethylbarbituric acid) is bonded to 6,6'-diquinolyl ether by two short $\text{NH} \cdots \text{N}$ hydrogen bonds with $\text{N} \cdots \text{N}$ 2.88 Å. This arrangement is similar to those reported for hydrogen bonds in the molecular complexes of barbiturates.

Comment

Barbiturates are widely used medicinally and are among the most frequently encountered drugs for the treatment of epilepsy. This class of antiepileptic drug is of special interest since many of them and their active metabolites form metal complexes. Various metal complexes of barbiturates have been synthesized and studied (Caira, Fazarkey, Linder & Nassimbeni, 1973; Pezeshk, Greenaway & Sorenson, 1983). A series of crystal structure determinations of molecular complexes of barbiturates with other molecules, such as 9-ethyladenine (Voet & Rich, 1972; Voet, 1972), caffeine (Craven & Gartland, 1970), aminopyrine (Kiryu, 1971), urea (Gartland & Craven, 1974), acetamide (Hsu & Craven, 1974a), salicylamide (Hsu & Craven, 1974b) and imidazole (Hsu & Craven, 1974c), have been reported. In crystal complexes of barbiturates with these ligands there are stronger $\text{NH} \cdots \text{O} \cdots \text{C}$ and $\text{NH} \cdots \text{N}$ hydrogen bonds between the two components than are found in the crystal structures of the individual components.

Quinoline is one of the ligands often used in coordination chemistry. Zhang and co-workers synthesized 6,6'-diquinolyl ether and described its structure (Zhang, Tan & Li, 1986; Wang, Zhang & Kong, 1988). It was found that 6,6'-diquinolyl ether is an important ligand in the syntheses of some complexes with biomedical activity. The present paper reports an X-ray diffraction study of the crystal structure of the molecular addition compound of 6,6'-diquinolyl ether with barbital (Fig. 1).

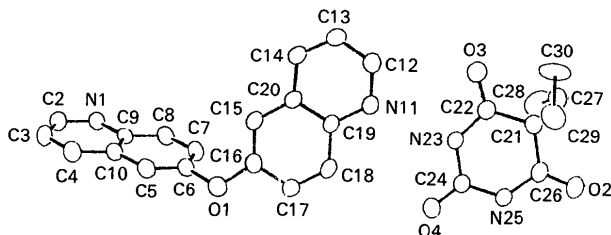


Fig. 1. An ORTEP (Johnson, 1965) drawing of the molecular structure.

In the quinolyl ring, the C—C bond lengths are between 1.352 and 1.419 Å, average 1.392 Å. The C—N bond lengths are between 1.314 and 1.367 Å, average 1.340 Å. The C—H bond lengths are in the expected range 0.93–1.01 Å. The average value of the C—O bond length is 1.394 Å; the C6—O—C16 bond angle is 117.5°. The above results are similar to those for the 6,6'-diquinolyl ether hydrate (Wang, Zhang & Kong, 1988). All atoms for each quinolyl ring are coplanar.

The 6,6'-diquinolyl ether molecule adopts a conformation with a dihedral angle of 108.8° between the two quinolyl rings.

The C30 atom in the ethyl group of the barbital molecule has abnormally high thermal parameters suggesting that it is disordered.

Strong intermolecular N1...H—N25 (N...N 2.88, N...H 1.90, H—N 0.99 Å, N...H—N 178°) and N11...H—N23 (N...N 2.88, N...H 1.95, H—N 0.94 Å, N...H—N 173°) hydrogen bonds occur. N11 is hydrogen bonded to N23 of the barbital of the same asymmetric unit while N1 is hydrogen bonded to N25 of the barbital of the asymmetric unit at $\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$. Thus barbital is linked with 6,6'-diquinolyl ether to form chains by the NH...N hydrogen bonds in which the barbital NH groups are donors and 6,6'-diquinolyl ether N atoms are acceptors (Hsu & Craven, 1974a). The dihedral angles between the planes of the two quinolyl rings and the barbital molecule of the same asymmetric unit are 105.12 and 5.23°, respectively.

Experimental

Crystal data

C₂₆H₂₄N₄O₄
M_r = 456.51
 Monoclinic

Z = 8
D_x = 1.315 Mg m⁻³
 Mo Kα radiation

C2/c

a = 24.565 (2) Å
b = 17.750 (2) Å
c = 10.959 (3) Å
 β = 105.24 (3)°
V = 4610.4 Å³

Data collection

CAD-4 four-circle diffractometer
 $\omega/2\theta$ scans
 Absorption correction: empirical
T_{min} = 0.925, *T_{max}* = 1.000
 5384 measured reflections
 4548 independent reflections

Refinement

Refinement on *F*
 Final *R* = 0.052
wR = 0.047
S = 3.75
 2657 reflections
 379 parameters
 (Δ/σ)_{max} = 0.60

λ = 0.7107 Å

Cell parameters from 25 reflections
 θ = 10–20°
 μ = 0.0846 mm⁻¹
T = 294 K
 0.40 × 0.25 × 0.25 mm

2657 observed reflections
 [*I* > 2σ(*I*)]
R_{int} = 0.017
 θ _{max} = 27°
h = 0 → 32
k = 0 → 23
l = -14 → 14
 3 standard reflections
 frequency: 90 min
 intensity variation: 1.5%

$\Delta\rho$ _{max} = 0.32 e Å⁻³
 $\Delta\rho$ _{min} = -0.40 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 thermal parameters (Å²)

$$B_{\text{eq}} = (4/3)[a^2B(1,1)+b^2B(2,2)+c^2B(3,3)+ab(\cos \gamma)B(1,2)+ac(\cos \beta)B(1,3)+bc(\cos \alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
O1	0.54456 (9)	0.1666 (2)	0.0807 (2)	5.66 (6)
O2	0.0831 (1)	0.1581 (2)	0.0483 (3)	7.56 (7)
O3	0.1826 (1)	0.0204 (2)	-0.1932 (3)	7.96 (8)
O4	0.2717 (1)	0.1735 (2)	0.1222 (3)	7.99 (8)
N1	0.6912 (1)	0.2277 (2)	-0.2129 (2)	4.27 (6)
N11	0.3260 (1)	0.0670 (2)	-0.1173 (2)	3.99 (6)
N23	0.2259 (1)	0.0959 (2)	-0.0341 (3)	5.05 (7)
N25	0.1769 (1)	0.1643 (2)	0.0853 (3)	5.05 (7)
C2	0.7431 (1)	0.2018 (2)	-0.1774 (3)	4.77 (8)
C3	0.7635 (1)	0.1567 (2)	-0.0702 (4)	5.17 (9)
C4	0.7293 (1)	0.1392 (2)	0.0050 (3)	4.87 (9)
C5	0.6341 (1)	0.1508 (2)	0.0427 (3)	4.48 (8)
C6	0.5810 (1)	0.1783 (2)	0.0037 (3)	4.17 (7)
C7	0.5631 (1)	0.2225 (2)	-0.1061 (3)	4.53 (8)
C8	0.5998 (1)	0.2376 (2)	-0.1768 (3)	4.43 (8)
C9	0.6555 (1)	0.2100 (2)	-0.1403 (3)	3.73 (7)
C10	0.6730 (1)	0.1661 (2)	-0.0294 (3)	3.92 (7)
C12	0.3134 (1)	0.0238 (2)	-0.2182 (3)	4.39 (8)
C13	0.3530 (2)	-0.0017 (2)	-0.2801 (3)	4.84 (8)
C14	0.4078 (1)	0.0181 (2)	-0.2337 (3)	4.62 (8)
C15	0.4796 (1)	0.0910 (2)	-0.0764 (3)	4.24 (7)
C16	0.4910 (1)	0.1390 (2)	0.0236 (3)	4.03 (7)
C17	0.4488 (1)	0.1634 (2)	0.0799 (3)	4.52 (8)
C18	0.3949 (1)	0.1394 (2)	0.0324 (3)	4.52 (8)
C19	0.3808 (1)	0.0896 (2)	-0.0724 (3)	3.58 (7)
C20	0.4240 (1)	0.0659 (2)	-0.1274 (3)	3.65 (7)
C21	0.1228 (1)	0.0762 (2)	-0.0782 (3)	4.65 (8)
C22	0.1789 (1)	0.0619 (2)	-0.1072 (3)	5.09 (9)
C24	0.2276 (1)	0.1464 (2)	0.0618 (3)	5.16 (9)
C26	0.1254 (1)	0.1363 (2)	0.0223 (3)	4.95 (9)
C27	0.0800 (2)	0.1000 (3)	-0.1991 (4)	5.7 (1)
C28	0.0968 (2)	0.1685 (3)	-0.2608 (5)	8.1 (1)
C29	0.1054 (2)	0.0019 (3)	-0.0205 (4)	6.9 (1)
C30	0.0957 (6)	-0.0606 (6)	-0.104 (1)	34.6 (6)

Table 2. Geometric parameters (Å, °)

O1—C6	1.399 (4)	C7—C8	1.362 (5)
O1—C16	1.388 (4)	C8—C9	1.409 (4)
O2—C26	1.211 (5)	C9—C10	1.413 (4)
O3—C22	1.219 (5)	C12—C13	1.400 (5)
O4—C24	1.212 (5)	C13—C14	1.355 (5)
N1—C2	1.315 (4)	C14—C20	1.411 (4)
N1—C9	1.367 (5)	C15—C16	1.358 (5)
N11—C12	1.314 (4)	C15—C20	1.407 (4)
N11—C19	1.365 (4)	C16—C17	1.407 (5)
N23—C22	1.360 (4)	C17—C18	1.358 (4)
N23—C24	1.375 (5)	C18—C19	1.419 (4)
N25—C24	1.373 (5)	C19—C20	1.414 (5)
N25—C26	1.366 (4)	C21—C22	1.516 (5)
C2—C3	1.399 (5)	C21—C26	1.522 (5)
C3—C4	1.359 (6)	C21—C27	1.519 (5)
C4—C10	1.419 (5)	C21—C29	1.569 (6)
C5—C6	1.352 (5)	C27—C28	1.501 (7)
C5—C10	1.416 (5)	C29—C30	1.41 (1)
C6—C7	1.406 (5)		
C6—O1—C16	117.5 (2)	C15—C16—C17	121.8 (3)
C2—N1—C9	118.0 (3)	C16—C17—C18	119.5 (3)
C12—N11—C19	117.9 (3)	C17—C18—C19	120.9 (3)
C22—N23—C24	126.2 (4)	N11—C19—C18	118.9 (3)
C24—N25—C26	125.9 (3)	N11—C19—C20	122.5 (3)
N1—C2—C3	123.6 (4)	C18—C19—C20	118.6 (3)
C2—C3—C4	119.8 (3)	C14—C20—C15	123.4 (3)
C3—C4—C10	118.6 (3)	C14—C20—C19	116.8 (3)
C6—C5—C10	119.3 (4)	C15—C20—C19	119.8 (3)
O1—C6—C5	118.2 (3)	C22—C21—C26	113.8 (3)
O1—C6—C7	119.5 (3)	C22—C21—C27	108.9 (4)
C5—C6—C7	122.1 (3)	C22—C21—C29	107.5 (3)
C6—C7—C8	119.4 (4)	C26—C21—C27	109.0 (4)
C7—C8—C9	120.7 (4)	C26—C21—C29	105.2 (3)
N1—C9—C8	118.6 (3)	C27—C21—C29	112.7 (3)
N1—C9—C10	122.1 (3)	O3—C22—N23	119.9 (3)
C8—C9—C10	119.3 (3)	O3—C22—C21	121.4 (4)
C4—C10—C5	122.8 (4)	N23—C22—C21	118.7 (3)
C4—C10—C9	118.0 (3)	O4—C24—N23	121.3 (3)
C5—C10—C9	119.3 (3)	O4—C24—N25	122.1 (4)
N11—C12—C13	123.7 (3)	N23—C24—N25	116.6 (3)
C12—C13—C14	118.9 (3)	O2—C26—N25	120.2 (3)
C13—C14—C20	120.1 (3)	O2—C26—C21	121.3 (3)
C16—C15—C20	119.5 (3)	N25—C26—C21	118.6 (3)
O1—C16—C15	123.7 (4)	C21—C27—C28	114.2 (3)
O1—C16—C17	114.4 (3)	C21—C29—C30	114.7 (7)

A solution of sodium barbital (1 mmol) in 10 ml H₂O was added dropwise to a solution of 6,6'-diquinolyl ether (1 mmol) in 15 ml absolute alcohol at room temperature (294 K), with constant stirring for 2 h. Brown stick-like or plate-like crystals were grown from the mixed solution after 10 d. The crystals were filtered, washed with H₂O and absolute alcohol. Analysis: calculated for C₂₆H₂₄N₄O₄: C 68.40, H 5.30, N 12.28%; found: C 68.18, H 5.12, N 12.40%.

Structure analysis was performed with the *SDP* program on a PDP11/44 computer. The crystal structure was solved by direct methods. The positions of 34 non-H atoms in the asymmetric unit were determined with *MULTAN82* (Main *et al.*, 1982). The positions of all H atoms were determined by difference Fourier syntheses. The refinements were carried out with unit weights by full-matrix least squares for the coordinates of all atoms and anisotropic thermal parameters for the non-H atoms (isotropic thermal parameters for H atoms were fixed at 5 Å²).

Lists of structure factors, anisotropic thermal parameters, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55380 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0411]

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(E)-Methyl 2-Phenyl-2-(3-pyridyl)vinyl Sulfide

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Abstract

The unambiguous identification by X-ray diffraction of the (*E*) configuration of the title compound confirms a previous tentative assignment by NMR. The two planar rings (phenyl and pyridyl) are at an angle of about 70°. Crystal cohesion is ensured by a dense network of van der Waals contacts.