Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1318). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 172–174

trans-1,2-Bis(8-hydroxy-2-quinolinyl)-ethene: Comparison with *trans*-Stilbene

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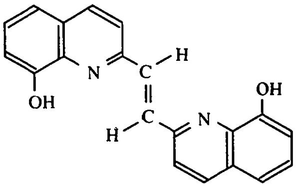
Abstract

The title compound, (*E*)-1,2-ethylenediylbis(2-quinolin-8-ol), $C_{20}H_{14}N_2O_2$, was isolated chromatographically from the Wittig reaction of 8-hydroxy-2-quinolinecarbaldehyde, triphenyl(2-methylene-8-hydroxyquinolinyl)-phosphonium chloride and *n*-butyllithium. The structure is almost planar with C_2 symmetry in the crystal. The olefinic C═C distance is 1.319 (3) Å and the structure consists of crystallographically equivalent molecules stacked along the **b** direction. These molecules are linked *via* zigzag hydrogen bonding between the quinolinol O atom of one molecule and the hydroxy H atom of another molecule. Such systematic hydrogen bonding on both sites of the molecule characterizes a novel backbone network.

Comment

Recently, *trans*-stilbene derivatives have attracted considerable interest because of their unusually short C═C bond distance (Ogawa, 1993). X-ray crystal structure reports of *trans*-1,2-disubstituted ethenes incorporating heteroaromatic compounds are known but limited in number. We describe herein a facile synthesis of *trans*-1,2-bis(8-hydroxy-2-quinolinyl)ethene, (I), *via* the Wittig reaction, and its structural characterization.



(I)

As shown in Fig. 1, the molecular structure of the title compound possesses an almost planar geometry, with torsion angles C9—C10—C10′—C9′ of 180° and N1—C9—C10—C10′ of 2.9 (3)°, the latter of which is small compared to the corresponding value found in *trans*-stilbene (Tirado-Rives, Fronczek & Gandour, 1985). The C10═C10′ double-bond distance of 1.319 (3) Å is in the range typical of ethylene C═C bond lengths [1.318 (5)–1.326 (3) Å] reported for *trans*-stilbene (Finder, Newton & Allinger, 1974; Bernstein, 1975; Boustra, Schouten & Kroon, 1984) and slightly shorter than the value of 1.332 (4) Å reported for *trans*-1,2-bis(2-methyl-4-quinazolinyl)ethylene (Wolfgang & Mehmet, 1990). The C9···C9′ distance is 3.821 Å in this compound and 3.873 Å in *trans*-stilbene, and the respective C9—C10—C10′ angles have comparable values of 124.4 (2) and 126.7 (2)°. These results can be explained by the steric effect of the intermolecular hydrogen bond [O1—H1···O 3.043 (4) Å] of the title compound. Fig. 2 shows the packing diagram viewed down the **a** direction of the molecule. Hydrogen bonds on both hydroxy groups of one molecule cause alignment of the zigzag molecules [O1—H1···O = 132 (2)°]. Thus, the structure exhibits a specific crystalline network in which the crystallographically equivalent molecules stack in a unique way, with molecules superimposed as shown in Fig. 2, similar to the situation seen in coronene (Robertson & White, 1944). The uniform stacking of planar π-donor molecules is ideal for both the design of molecular metals and the study of charge-transfer complexes.

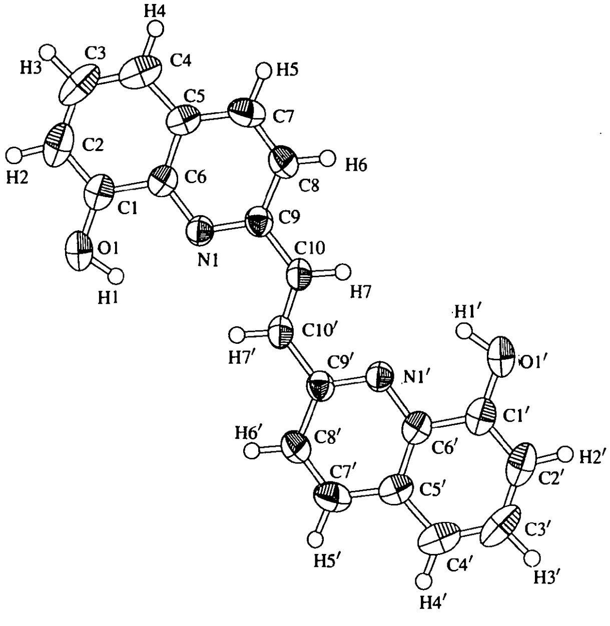


Fig. 1. Molecular structure of *trans*-1,2-bis(8-hydroxy-2-quinolinyl)-ethene, showing the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

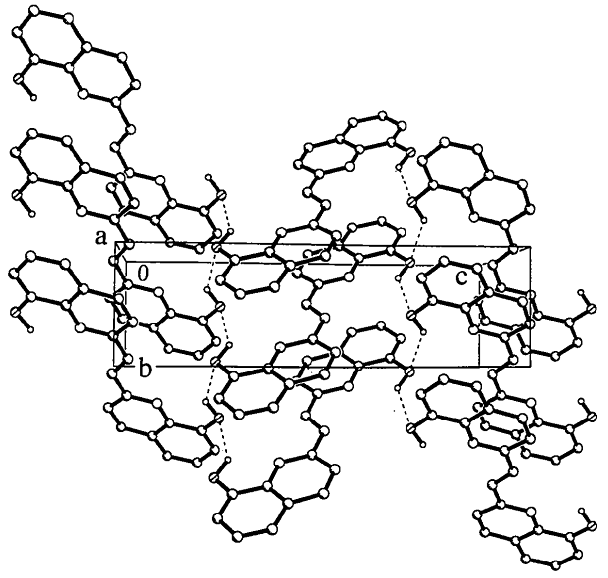


Fig. 2. The crystal packing, viewed approximately down the *a* axis, showing the hydrogen bonds.

Experimental

To a solution of butyllithium (34 mmol) in dry benzene (300 ml) was added triphenyl(2-methylene-8-hydroxy-quinolinyl)phosphonium chloride (26 mmol) over a period of 20 min. After stirring for 3 h under a nitrogen atmosphere, a solution of 8-hydroxy-2-quinolinecarbaldehyde (26 mmol) in dry benzene (200 ml) was added dropwise. The mixture was refluxed for 19 h whereupon a small amount of 0.1 M HCl was added to the mixture to afford a weak acidity. The crude product was purified by column chromatography (EtOAc/hexane 1:1 *v*/*v*) and recrystallized from $CHCl_3$/hexane solution to give the title compound [(I), BQOE; yield 23%, 6.3 mmol, m.p. 535 K] as yellow crystals. FT-IR (KBr disc): 3360 (OH), 1593, 1567 (C=C), 989 (*trans*-CH=CH) cm^{-1}. ^1H NMR (d_6-acetone) δ 8.93 (*s*, OH, 2H), 8.37 (*d*, 4-quiH, J = 8.6 Hz, 2H), 8.27 (*s*, CH=CH, 2H), 7.88 (*d*, 3-quiH, J = 8.6 Hz, 2H), 7.47 (*t*, 6-quiH, J = 7.2 Hz, 2H), 7.42 (*d*, 5-quiH, J = 8.2 Hz, 2H), 7.15 (*d*, 7-quiH, J = 7.2 Hz, 2H). Mass spectrum (*m*/*e*): 314 (M^+). Analysis calculated for $C_{20}H_{14}N_2O_2$: C 76.42, H 4.49, N 8.91%; found: C 76.30, H 4.48, N 8.81%.

Crystal data

$C_{20}H_{14}N_2O_2$
$M_r = 314.34$
Monoclinic
$P2_1/c$
$a = 10.380\,(7)$ Å
$b = 4.650\,(4)$ Å
$c = 16.04\,(1)$ Å
$\beta = 92.25\,(7)°$
$V = 773.6\,(9)$ Å^3
$Z = 2$
$D_x = 1.349$ Mg m^{-3}
$D_m = 1.346$ Mg m^{-3}
D_m measured by pycnometry

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.05–14.90°$
$\mu = 0.089$ mm^{-1}
$T = 293$ K
Prismatic
0.70 × 0.50 × 0.30 mm
Yellow

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1833 measured reflections
1740 independent reflections
1078 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.010$

$\theta_{max} = 27.5°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 6$
$l = -21 \rightarrow 21$
3 standard reflections monitored every 150 reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R(F) = 0.036$
$wR(F^2) = 0.032$
$S = 2.45$
1078 reflections
138 parameters
Only H-atom *U*'s refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.48$

$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: Lp secondary
Extinction coefficient: 3.68605×10^{-6}
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)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O1	0.4064 (1)	0.4602 (3)	1.25654 (7)	5.96 (4)
N1	0.3750 (1)	0.3303 (3)	1.09415 (7)	3.37 (3)
C1	0.3068 (2)	0.5840 (4)	1.2117 (1)	4.40 (4)

C2	0.2255 (2)	0.7702 (5)	1.2483 (1)	5.81 (6)
C3	0.1242 (2)	0.8970 (4)	1.2016 (2)	6.14 (6)
C4	0.1033 (2)	0.8350 (4)	1.1193 (2)	5.42 (5)
C5	0.1857 (1)	0.6421 (3)	1.0796 (1)	3.96 (4)
C6	0.2893 (1)	0.5164 (3)	1.12609 (9)	3.49 (3)
C7	0.1730 (2)	0.5604 (4)	0.9949 (1)	4.75 (5)
C8	0.2579 (2)	0.3727 (4)	0.9630 (1)	4.25 (4)
C9	0.3601 (1)	0.2605 (3)	1.01407 (9)	3.32 (3)
C10	0.4523 (1)	0.0627 (3)	0.97855 (9)	3.57 (4)

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

O1—C1	1.363 (2)	C10—C10′	1.319 (3)
N1—C6	1.355 (2)	N1—C9	1.328 (2)
C1—C2	1.358 (3)	C1—C6	1.414 (2)
C2—C3	1.397 (3)	C3—C4	1.360 (3)
C4—C5	1.410 (3)	C5—C6	1.411 (2)
C5—C7	1.412 (2)	C7—C8	1.355 (3)
C8—C9	1.413 (2)	C9—C10	1.459 (2)
O1—C1—C6	119.0 (2)	O1—C1—C2	120.8 (2)
C6—N1—C9	117.9 (1)	C2—C1—C6	120.2 (2)
N1—C9—C10	118.6 (1)	N1—C9—C8	121.7 (2)
C9—C10—C10′	124.4 (2)	C8—C9—C10	119.7 (1)
C10′—C10—H7	118.1 (8)	C9—C10—H7	117.4 (8)
O1—C1—C6—N1	−0.3 (2)	N1—C9—C10—C10′	2.9 (3)
C8—C9—C10—C10′	−177.1 (2)	C9—C10—C10′—C9′	180.0

Symmetry code: (′) $1 - x, -y, 2 - z$.

Non-H atoms were refined anisotropically and H atoms were refined isotropically.

Rigaku AFC-7*R* software was used for data collection, cell refinement and data reduction. The title structure was solved by direct methods using *SAPI*91 (Fan, 1991) and refined by expanded Fourier techniques using the full-matrix least-squares technique of *DIRDIF*92 (Beurskens *et al.*, 1992). All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1992).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KH1043). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 174–177

Low-Temperature Triphenylmethane

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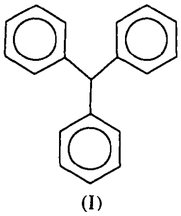
Abstract

The crystal structure of triphenylmethane, $C_{19}H_{16}$, has been redetermined at 150 K. It confirms the previously reported room-temperature study [Riche & Pascard-Billy (1974). *Acta Cryst.* B**30**, 1874–1876], while correcting for the erroneously reported space group from $P2_12_12_1$ to $Pna2_1$. The structure contains two crystallographically independent triphenylmethane molecules, both with a propeller configuration. The deformations from ideal phenyl-ring geometry conform with those expected for rings having electron-releasing substituents.

Comment

Commercially available triphenylchloromethane contains some triphenylmethane as an impurity (capillary GC–MS) and has to be purified by recrystallization prior to use. Good quality crystals of triphenylmethane, (I), were obtained as a by-product. The room-temperature X-ray structure of this compound has been reported previously by Riche & Pascard-Billy (1974). Unfortunately, the space group was reported incorrectly as $P2_12_12_1$ rather than the correct $Pna2_1$ (see below), which was presumably used in the study. This error appears to have propagated into the Cambridge Structural Database (Allen, Kennard & Taylor, 1983; Version 5, October 1994), where additional errors were introduced in the coordinates of atoms C36 and C37 on the basis of the incorrect space group symmetry, resulting in nonsensical connectivity. The present low-temperature study clarified the case and confirms the previously reported structural results with improved precision.



(I)

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