Orthorhombic
$P2_12_12_1$
$a = 8.792\,(2)$ Å
$b = 12.481\,(2)$ Å
$c = 18.440\,(4)$ Å
$V = 2023.5\,(6)$ Å^3
$Z = 4$
$D_x = 1.282$ Mg m^{-3}
D_m not measured

Cell parameters from 25
reflections
$\theta = 10.1–11.9°$
$\mu = 0.195$ mm^{-1}
$T = 293$ K
Cubic fragment from a prism
$0.40 \times 0.38 \times 0.32$ mm
Colourless

Data collection

Enraf–Nonius CAD-4
diffractometer
ω scans
Absorption correction: none
3346 measured reflections
3346 independent reflections
2449 reflections with
$I > 2\sigma(I)$

$\theta_{max} = 29.96°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 25$
2 standard reflections
every 120 reflections
intensity decay: -4.61%

Refinement

Refinement on F
$R = 0.040$
$wR = 0.045$
$S = 1.854$
2449 reflections
236 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o) + 0.00022|F_o|^2]$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction:
Zachariasen (1967, 1968)
Extinction coefficient:
$1.3\,(1) \times 10^{-6}$
Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—O1	1.460 (2)	C2—C3	1.525 (3)
S1—O2	1.640 (1)	C3—C4	1.512 (3)
S1—C1	1.803 (2)	C4—C5	1.504 (3)
O2—C7	1.448 (2)	C5—C6	1.538 (3)
C1—C2	1.513 (3)	C7—C8	1.519 (3)
C1—C6	1.517 (3)	C7—C13	1.521 (3)
O1—S1—O2	107.3 (1)	C2—C3—C4	111.3 (2)
O1—S1—C1	106.56 (9)	C3—C4—C5	111.8 (2)
O2—S1—C1	94.89 (8)	C4—C5—C6	111.8 (2)
S1—O2—C7	114.8 (1)	C1—C6—C5	110.0 (2)
S1—C1—C2	113.5 (1)	O2—C7—C8	107.5 (1)
S1—C1—C6	110.6 (1)	O2—C7—C13	110.6 (1)
C2—C1—C6	111.9 (2)	C8—C7—C13	101.4 (2)
C1—C2—C3	109.4 (2)		

H atoms were located from difference Fourier syntheses but were not refined.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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A 2:1 co-crystal of 5-(2-pyridyl)-1H-1,2,4-triazole and 1,4-dihydroxybenzene

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Abstract

In the 2:1 co-crystal of 5-(2-pyridyl)-1H-1,2,4-triazole (pyt) and 1,4-dihydroxybenzene (dhb), $2C_7H_6N_4\cdot C_6H_6O_2$, the pyt molecules form two independent N—H$\cdots$N hydrogen-bonded chains. The dhb molecules act as hydrogen-bonding bridges between the two pyt chains *via* two O—H$\cdots$N hydrogen bonds. The complex is further stabilized by a number of weaker interactions, including C—H$\cdots$N, C—H$\cdots$O and C—H$\cdots\pi$ interactions.

Comment

Recently, the crystal structure of a co-crystal between 1,4-dihydroxybenzene (dhb; hydroquinone) and

3,5-bis(2-pyridyl)-1,2,4-triazole (bpt) was reported (Nieuwenhuyzen et al., 1997). In this structure, substantial hydrogen bonding between the two components and a well defined long-range ordering of the dhb moieties are observed. We believe that these materials might have interesting electron- and proton-transfer properties, and we have therefore embarked on a systematic study of 1,4-dihydroxybenzene–pyridyltriazole co-crystals. In this contribution, we report the crystal structure of a co-crystal of dhb and 5-(2-pyridyl)-1H-1,2,4-triazole (pyt).
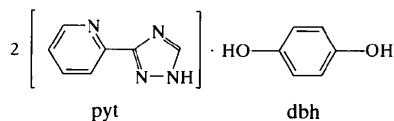
The asymmetric unit contains two independent pyt molecules and one dhb molecule. The molecular structure and atom-numbering scheme are illustrated in Fig. 1. The bond lengths and angles for the pyt moieties are consistent with those found in [Ru(bpy)$_2$(bpt)]PF$_6$·0.5H$_2$O (Hage et al., 1989), [Ru(bpy)$_2$(bpt)](CF$_3$SO$_3$)·4H$_2$O (Hage et al. 1990) and [Ru(bpy)$_2$(mpt)]PF$_6$·4H$_2$O (Buchanan et al., 1990), where bpy is 2,2′-bipyridine and mpt is 3-methyl-5-(2-pyridyl)-1,2,4-triazole.
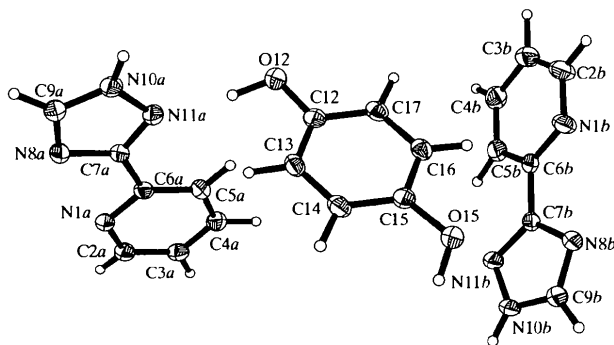
Fig. 1. View of the title compound with displacement ellipsoids at the 50% probability level.

Both of the pyt molecules form infinite hydrogen-bonded chains via a strong N—H···N (triazole-to-triazole) hydrogen bond and a weaker N—H···N (triazole-to-pyridyl) interaction (Fig. 2). This weaker interaction is hindered by the presence of a pyridyl-H atom (H2), contributing to the twist of the pyridyl rings out of the triazole plane. The degree of twist is different for the two pyridyl rings: 1.8 (1)° for molecule a and 11.6 (1)° for molecule b.

The chains of pyt molecules pack in a herring-bone fashion with respect to one another [87.45 (5)°

between the chains of molecule a and the chains of molecule b] and are involved in edge-to-face C—H···π interactions (3.5–3.9 Å) with adjacent hydrogen-bonded chains. This arrangement of chains results in the formation of channels in the [001] direction, in which the dhb molecules are situated. The dhb molecules provide a bridge between the independent chains (a and b) of the pyt molecules via strong O—H···N hydrogen bonds to the triazole N atom not involved in chain formation (Table 1). These strong hydrogen bonds are complemented by weaker C—H···O and C—H···π interactions between the pyt and dhb molecules which stabilize the hydrogen-bonded network (Fig. 2).
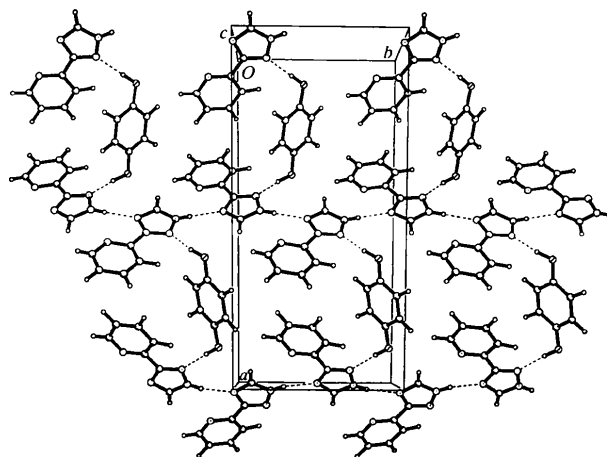
Fig. 2. View of the hydrogen-bonded layers of dhb and pyt molecules.

Experimental

1,4-Dihydroxybenzene (Aldrich) was purified by recrystallization from ethanol and 5-(2-pyridyl)-1H-1,2,4-triazole was prepared as reported in the literature (Uda et al., 1976). Co-crystals were obtained by dissolving the two components in a 1:2 ratio in ethanol–water (1:1) or ethanol–acetone (2:1). Crystals were formed slowly upon standing at room temperature (m.p. 434 K)

Crystal data

$2C_7H_6N_4·C_6H_6O_2$	Mo $K\alpha$ radiation
$M_r = 402.42$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 35
$P2_1/c$	reflections
$a = 20.745 (2)$ Å	$\theta = 5.0–12.5°$
$b = 9.921 (1)$ Å	$\mu = 0.099$ mm^{-1}
$c = 9.232 (1)$ Å	$T = 153 (2)$ K
$\beta = 101.15 (1)°$	Colourless
$V = 1864.2 (3)$ Å^3	$0.65 \times 0.52 \times 0.36$ mm
$Z = 4$	Block
$D_x = 1.434$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens *P*4 diffractometer	$\theta_{max} = 25°$
ω scans	$h = -24 \rightarrow 24$
Absorption correction: none	$k = -11 \rightarrow 0$
3496 measured reflections	$l = 0 \rightarrow 10$
3269 independent reflections	3 standard reflections
2633 reflections with	every 97 reflections
$I > 2\sigma(I)$	intensity decay: 1%
$R_{int} = 0.015$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.003$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$\Delta\rho_{max} = 0.186$ e Å^{-3}
$wR(F^2) = 0.085$	$\Delta\rho_{min} = -0.305$ e Å^{-3}
$S = 1.062$	Extinction correction: none
3268 reflections	Scattering factors from
273 parameters	*International Tables for*
H atoms: see below	*Crystallography* (Vol. C)

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.616P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O12—H12O$\cdots$N11A	0.84	1.995	2.830 (2)	175
O15—H15O$\cdots$N11B^i	0.84	2.004	2.826 (2)	167
N10A—H10A$\cdots$N8A^{ii}	0.86	1.996	2.844 (2)	167
N10B—H10B$\cdots$N8B^{iii}	0.87	2.001	2.845 (2)	162

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

H atoms were placed in idealized positions and refined as riding.

Data collection: *XSCANS* (Fait, 1993). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1258). Services for accessing these data are described at the back of the journal.

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Isoavarol diacetate

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Abstract

The crystal and molecular structures of isoavarol diacetate, (1*R*, 2*S*, 4a*S*, 8a*S*)-2-(1,2,4a-trimethyl-5-methylene-perhydro-1-naphthylmethyl)-*p*-phenylene diacetate, $C_{25}H_{34}O_4$, have been determined. There are two molecules of this quinoid-containing sesquiterpene in the asymmetric unit. Each of these molecules participates in intermolecular C—H$\cdots$O contacts between the aromatic C—H groups and the carbonyl-O atoms of the acetyl groups. Isoavarol differs from avarol in both the exocyclic position of the double bond at C4 and the chair conformation of ring *A*. The acetyl groups in each molecule of the title compound are oriented on one side of the phenylene ring, on opposite sides of the bicyclic system of the molecule.

Comment

Isoavarol, a sesquiterpene hydroquinone isomer of the well known compound avarol (De Rosa *et al.*, 1976), has been isolated from the Pacific sponge *Dysidea sp.* and its structure, including its absolute configuration, has been studied using spectroscopic and chemical correlative methods (Shubina *et al.*, 1990). Simultaneously, the same compound was obtained from the Okinawian *Dysidea* (Iguchi *et al.*, 1990) and named as neoavarol. Current interest in structural investigations on isoavarol and its derivatives is stimulated by the high and diverse biological activities of avarol itself, as well as of some related prenylated sesquiterpene quinones (Müller *et al.*, 1987; De Giulio *et al.*, 1990; Pennings *et al.*, 1994).

Recently, crystal structures of avarol (Puliti *et al.*, 1994) and its diacetate (Puliti *et al.*, 1995) have been reported, but an X-ray analysis of isoavarol and its derivatives has not previously been reported. Certain long range effects are characteristic of natural products belonging to this series; for instance, a substitution of the hydroxyl group at C4 in illimaquinone results in the rotation of the aromatic residues around the C9—C15 bond (Ilyin *et al.*, 1991). To elucidate the spatial