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A host–guest 1:2 inclusion compound of 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol and 4-methoxybenzaldehyde

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.124
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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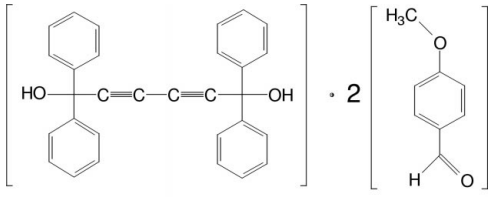
The inclusion method was used to isolate a single component from a volatile oil mixture (extracted from the natural medicine, *fructus foeniculi*) with 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol as the host molecule and 4-methoxybenzaldehyde as the guest (an oil mixture component). Crystals of the host–guest inclusion complex were obtained as $C_{30}H_{22}O_2\cdot 2C_8H_8O_2$, with the host molecule residing on a crystallographic inversion centre and connected to the guest molecule by an intermolecular hydrogen bond [O···O = 2.846 (5) Å]. The host–guest molecules form a layer-type structure, which extends along the *a* axis and periodically arranges along the *b* axis.

Comment

An important direction for pharmaceutical research is to produce bioactive lead compounds from natural products. Volatile oils exist in many natural products and they have important implications in pharmacodynamics research. Most of these oils are monoterpenoids, sesquiterpenoids and aromatic compounds, and they have functional groups such as alcohol, aldehyde, ketone and lactone. The similarity in chemical structure and volatility makes it more difficult to extract and isolate components from the mother oil.



(I)

The inclusion method used by us depends on two essential factors: (i) the similar geometric topology and (ii) intermolecular hydrogen bonding and van der Waals forces between the host–guest molecules (Lehn, 1988). We selected 4-methoxybenzaldehyde (anisaldehyde) in the volatile oil mixture as the guest molecule; this has antifungal bioactivity (Sun & Sheng, 1998). We selected 1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol as the host molecule; this can include many guest molecules, such as alcohols, aldehydes, ketones and lactones (Takumi *et al.*, 1996). Thus we obtained a crystal of the 1:2 host–guest complex 1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol–4-methoxybenzaldehyde (1/2), (I).

In (I), the inversion centre of the host molecule coincides with the crystallographic inversion centre (1/2,0,0) (Fig. 1). The asymmetric unit consistes of half of the host molecule and one guest molecule, *i.e.* in a 1:2 ratio. With van der Waals forces, the host–guest molecules form a layer-type structure,

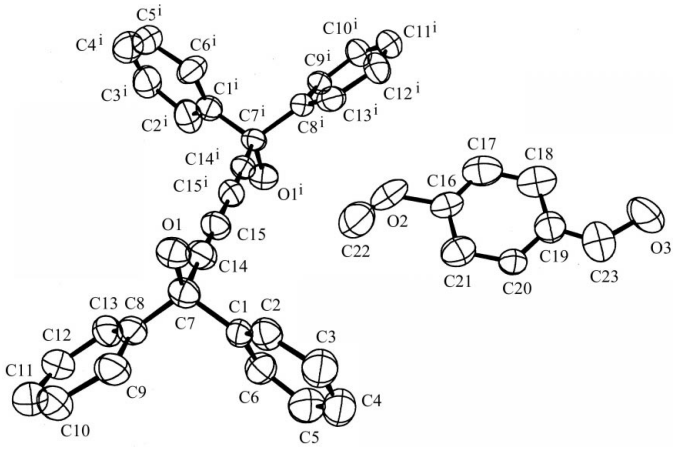


Figure 1
The molecular structure of inclusion complex (I), showing 50% probability displacement ellipsoids [symmetry code: (i) $1-x, -y, -z$]. H atoms have been omitted for clarity.

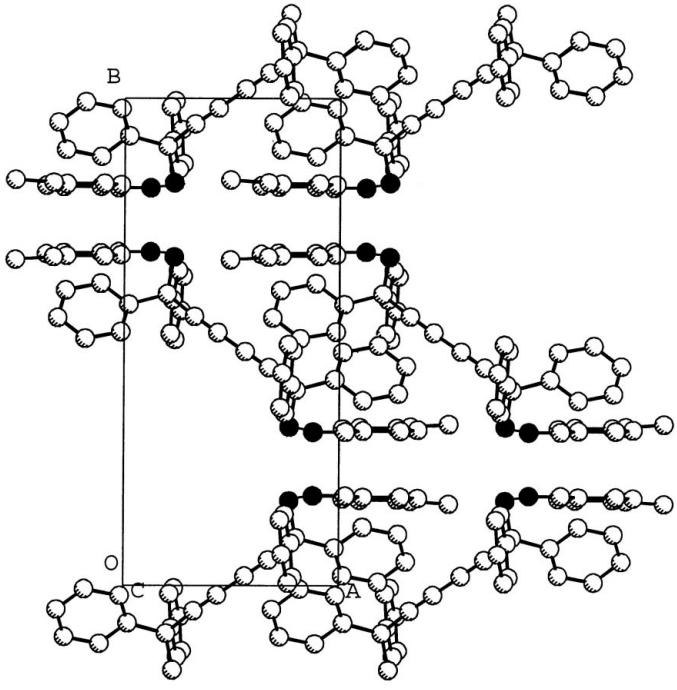


Figure 2
The crystal packing, viewed down the *c* axis. The black atoms are the O atoms involved in hydrogen bonds.

which extends along the *a* axis and periodically arranges along the *b* axis (Fig. 2). The guest molecule is planar, as expected. The hydroxyl group of the host molecule is associated with the aldehyde group of the guest molecule (details in Table 1).

Experimental

Fructus foeniculi (100 g) was ground and distilled with water vapor. The volatile oil was dehydrated with anhydrous Na_2SO_4 and 2 g of this material was obtained. 1,1,6,6–Tetraphenylhexa-2,4-diyne-1,6-diol (0.2 g) was added to a 10 ml conical flask and dissolved in ethyl ether (0.6 ml). The volatile oil (0.3 g) and petroleum ether (1 ml) were added to the host solution and the resulting solution stored at 298 K. After the crystals were found, recrystallization was conducted under the same conditions, yielding the title host–guest inclusion complex.

Crystal data

$C_{30}H_{22}O_2{\cdot}2C_8H_8O_2$
$M_r = 686.80$
Monoclinic, $P2_1/c$
$a = 8.593\ (1)$ Å
$b = 18.181\ (1)$ Å
$c = 12.649\ (1)$ Å
$\beta = 111.38\ (1)^\circ$
$V = 1840.2\ (3)$ Å^3
$Z = 2$
$D_x = 1.244$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3223 reflections
$\theta = 2$–25°
$\mu = 0.08$ mm^{-1}
$T = 296\ (1)$ K
Block, pale yellow
$0.40 \times 0.30 \times 0.22$ mm

Data collection

MAC DIP 2030K diffractometer
ω scans
Absorption correction: none
5896 measured reflections
3223 independent reflections
3101 reflections with $F^2 \geq 2\sigma(F^2)$
$R_{int} = 0.032$
$\theta_{max} = 25.0^\circ$
$h = 0 \rightarrow 11$
$k = -21 \rightarrow 20$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.124$
$S = 1.46$
3101 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1082P)^2 + 0.627P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{ii}	0.84	2.01	2.846 (5)	171

Symmetry code: (ii) $1+x, y, z-1$.

Data collection: *DENZO* (MacScience, 1996); cell refinement: *SCALE* (MacScience, 1996); data reduction: *SCALE*; program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

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