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Key indicators

Single-crystal X-ray study
 $T = 100\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
Disorder in solvent or counterion
 R factor = 0.052
 wR factor = 0.144
Data-to-parameter ratio = 17.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

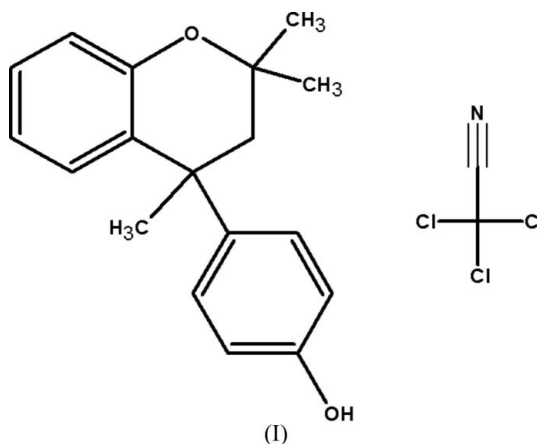
Dianin's compound trichloroacetonitrile clathrate

The the title compound [systematic name: 4-(2,2,4-trimethylchroman-4-yl)phenol-trichloroacetonitrile (6/1)], $6\text{C}_{18}\text{H}_{20}\text{O}_2 \cdot \text{C}_2\text{Cl}_3\text{N}$, the usual trigonal packing of the Dianin's compound host structure occurs. The guest is found in a host-guest ratio of 6:1. $\text{Cl} \cdots \pi$ interactions serve to order the guest, thus facilitating its structural characterization.

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Comment

As a continuation of our work on Dianin's compound and its clathration of various guests, we have prepared a new clathrate, (I), containing trichloroacetonitrile (CCl_3CN) (Fig. 1) (Esterhuysen *et al.*, 2005; Lloyd *et al.*, 2005; Lloyd & Bredenkamp, 2005; Lloyd, Alen, Jacobs *et al.*, 2006; de Vries *et al.*, 2005). Owing to the size of the CCl_3CN guest, a 6:1 host-guest ratio is both predicted and found.



Dianin's compound forms an isoskeletal series of clathrates (the host framework is essentially identical in all cases) (Lloyd, Alen, Bredenkamp *et al.*, 2006). Together with phenol, β -hydroquinol and a few chiral propargylic alcohols (a recent addition) (Hyacinth *et al.*, 2006), Dianin's compound belongs to the hexahost family of compounds. This means that six hydroxyl groups hydrogen-bond together to form a hexagonal cyclic arrangement (Table 1) (Flippen *et al.*, 1970).

A cavity of approximately 231 \AA^3 is created by the interdigitation of the six host hydrogen-bonded arrays stacked one on top of each other to form columns. The CCl_3CN guest in this cavity is disordered over two positions, corresponding to the molecule pointing up or down within the cavity. Therefore, unlike the carbon tetrachloride clathrate structures previously reported, the CCl_3CN guest of (I) is relatively ordered (Abriel *et al.*, 1990; Pang *et al.*, 1990). This can be accounted for by the strong $\text{Cl} \cdots \pi$ interaction between the Cl groups of the guest

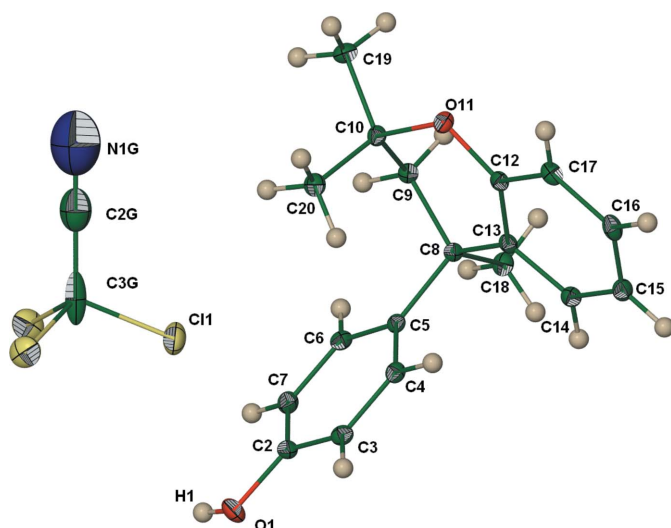


Figure 1
The molecular structure of (I), showing atom labels and 50% probability displacement ellipsoids.

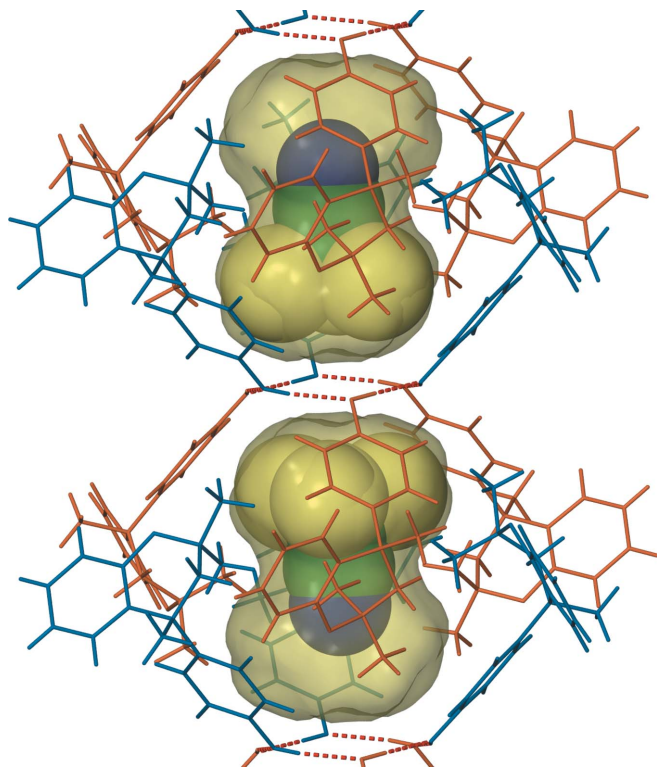


Figure 2
The encapsulation of CCl_3CN within the cavities of Dianin's compound. Void space is represented by a yellow semi-transparent surface. Chirally identical Dianin's compound molecules are shown in the same colour and in capped-stick representation (blue or orange). Guest molecules are shown in a space-filling representation.

and the benzene rings of the host, represented by the short $\text{Cl1}\cdots\text{C2}$ and $\text{Cl1}\cdots\text{C3}$ distances of 3.08 and 3.30 Å, respectively (Lloyd, Alen, Bredenkamp *et al.*, 2006; Williams & Hsu, 1985). This result represents an approach to reducing the problem of crystallographic disorder in host–guest chemistry

by using a guest that has the same or similar symmetry as the host structure.

Experimental

Dianin's compound was prepared according to the literature method of Lloyd *et al.* (2005). Single crystals of (I) suitable for X-ray analysis were then obtained by slow evaporation of a solution of Dianin's compound in trichloroacetonitrile.

Crystal data

$6\text{C}_{18}\text{H}_{20}\text{O}_2\cdot\text{C}_2\text{Cl}_3\text{N}$
 $M_r = 1754.42$
 Trigonal, $R\bar{3}$
 $a = 26.8141$ (13) Å
 $c = 10.8765$ (11) Å
 $V = 6772.5$ (8) Å³
 $Z = 3$

$D_x = 1.291$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.17$ mm⁻¹
 $T = 100$ (2) K
 Prism, colourless
 $0.35 \times 0.25 \times 0.23$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: none
 14334 measured reflections

3553 independent reflections
 2904 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$
 $\theta_{\text{max}} = 28.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.144$
 $S = 1.03$
 3553 reflections
 198 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.087P)^2 + 2.3737P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.008$
 $\Delta\rho_{\text{max}} = 0.57$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.45$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{O1---H1}\cdots\text{O1}^i$	0.84	1.98	2.803 (3)	166

Symmetry code: (i) $x - y, x, -z$.

All H atoms were positioned geometrically, with $\text{C---H} = 0.95, 0.99$ and 0.98 Å for aromatic C, CH_2 and CH_3 , respectively, and with $\text{O---H} = 0.84$ Å, and they were constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{O})$ for CH_2 , aromatic C and O---H groups, and $1.5U_{\text{eq}}(\text{C})$ for CH_3 groups.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *X-SEED*.

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