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5-Methyltropolone

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.109
Data-to-parameter ratio = 15.0

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

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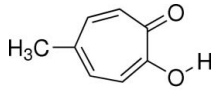
The title compound [systematic name: 2-hydroxy-5-methyl-2,4,6-cycloheptatrien-1-one], $C_8H_8O_2$, has two independent molecules in the asymmetric unit, which are linked into a dimer by a pair of intermolecular O—H···O hydrogen bonds. The crystal packing is consolidated by π–π, C—H···π and C—H···O interactions.

Comment

Troponoids, being a remarkable class of non-benzenoid π-conjugated systems, have been used as ligands and building blocks of various molecular assemblies such as liquid crystals and organogelators (Kubo *et al.*, 2004; Kubo, Takahashi & Takechi, 2006). Recently, we have prepared liquid crystals with a tropone and tropolone core (Kubo *et al.*, 2004).

The crystal structures of tropolone (Shimanouchi & Sasada, 1973), 5-cyano- and 5-nitrotropolones (Kubo *et al.*, 2001), and 5-methoxytropolone (Kubo, Yamamoto & Mori, 2006) have been reported. We now report the structure of the title compound, (I), with the aim of contributing to a deeper understanding of troponoids and their molecular assemblies.



(I)

The asymmetric unit of (I) contains two independent molecules (Table 1 and Fig. 1) linked into dimers *via* intermolecular O—H···O hydrogen bonds involving the OH group and the carbonyl O atom. Two intramolecular O—H···O bonds are also present (Table 2 and Fig. 1). The intermolecular O1···O4 and O2···O3 distances of 2.6976 (15) and 2.7606 (15) Å, respectively, are close to that found in tropolone (2.746 Å; Shimanouchi & Sasada, 1973) where a similar dimerization occurs. An intermolecular π–π interaction occurs in (I) between symmetry-related C1–C7 rings at (*x*, *y*, *z*) and $(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$, as indicated by the C···C separations

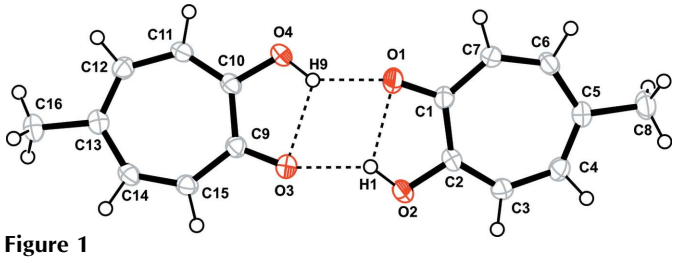


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids. Hydrogen bonds are indicated by dashed lines.

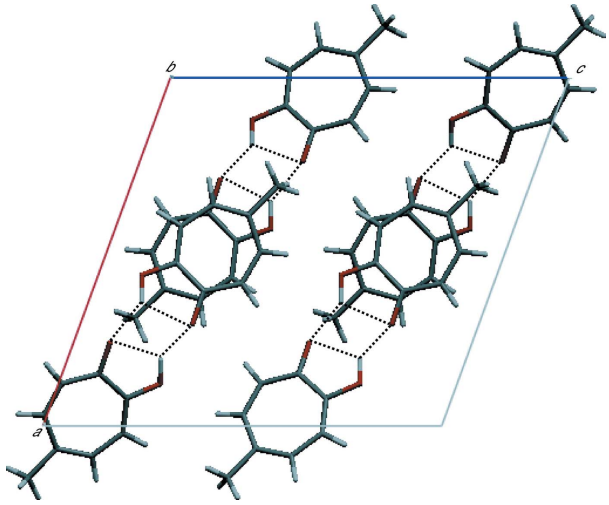


Figure 2
A packing diagram for (I), viewed down the *b* axis, with hydrogen bonds shown as dotted lines.

ranging from 3.431 (2) to 3.962 (2) Å and by the centroid–centroid distance of 3.679 (2) Å.

Intermolecular C—H···π and C—H···O interactions are observed in the crystal structure (Table 2), with distances typical for these types of interactions: C—H···π = 2.8–3.1 Å (Kubo, Fukeda *et al.*, 2006; Kubo, Yamamoto & Mori, 2006), C—H···O = 2.5–2.7 Å (Kubo, Fukeda *et al.*, 2006; Kubo, Yamamoto & Mori, 2006). The combination of all of the above interactions builds up a three-dimensional network.

Experimental

Compound (I) was prepared by the reduction of 3,7-dibromo-5-morpholinomethyltropolone with sodium hydroxide and palladium–carbon under hydrogen (Seto & Ogura, 1959). Crystals of (I) were grown from a hexane solution by slow evaporation of the solvent.

Crystal data

$C_8H_8O_2$
M_r = 136.15
Monoclinic, $P2_1/c$
a = 13.628 (3) Å
b = 7.1352 (14) Å
c = 14.663 (3) Å
β = 110.1031 (11)°
V = 1338.9 (5) Å^3
Z = 8
D_x = 1.351 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 153.1 K
Prism, colourless
0.18 × 0.15 × 0.12 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.939, T_{max} = 0.988
10387 measured reflections
3054 independent reflections
2143 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.036
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.01
3054 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[0.0007F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.2571 (17)	C5—C8	1.508 (2)
O2—C2	1.344 (2)	C6—C7	1.362 (2)
O3—C9	1.2545 (17)	C9—C10	1.4606 (16)
O4—C10	1.3429 (19)	C9—C15	1.431 (2)
C1—C2	1.4540 (16)	C10—C11	1.3662 (19)
C1—C7	1.431 (2)	C11—C12	1.411 (2)
C2—C3	1.3674 (18)	C12—C13	1.368 (2)
C3—C4	1.407 (2)	C13—C14	1.4175 (16)
C4—C5	1.370 (2)	C13—C16	1.506 (2)
C5—C6	1.4162 (17)	C14—C15	1.361 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O1	0.97 (2)	2.04 (2)	2.5877 (12)	113.7 (19)
O2—H1···O3	0.97 (2)	1.93 (2)	2.7606 (15)	142.3 (19)
O4—H9···O1	0.94 (2)	1.89 (2)	2.6976 (15)	142.4 (19)
O4—H9···O3	0.94 (2)	2.09 (2)	2.5989 (12)	112.6 (19)
C6—H4···O2^{i}	0.95	2.60	3.4528 (17)	150
C12—H11···O1^{ii}	0.95	2.56	3.4369 (16)	153
C14—H12···O4^{iii}	0.95	2.39	3.2589 (15)	152

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

The H atoms of the OH groups were located in a difference map and freely refined. H atoms bonded to C atoms were included in the refinement at calculated positions as riding atoms, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.3U_{eq}(methyl C).

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku Corporation, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Version 1.3; Bruno *et al.*, 2002); software used to prepare material for publication: *CrystalStructure*.

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