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5-Methoxytropolone

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.081
Data-to-parameter ratio = 12.0

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

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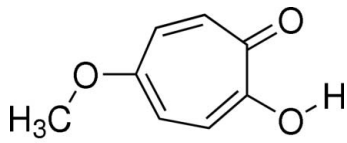
The title compound [systematic name: 2-hydroxy-5-methoxy-2,4,6-cycloheptatrien-1-one], $C_8H_8O_3$, contains intra- and intermolecular O—H···O hydrogen bonds, resulting in inversion-symmetry generated dimers. The crystal packing is consolidated by π–π, C—H···π and C—H···O interactions.

Comment

Troponoids, being a remarkable class of non-benzenoid π-conjugated systems, have been used as the building blocks of various molecular assemblies such as liquid crystals and organogelators (Kubo, 2004). Recently, we have prepared liquid crystals with a troponoid core, *e.g.* 5-alkoxytropolone (Mori *et al.*, 1990), 5-alkanoyloxytropolone (Mori *et al.*, 2001), 5-cyanotropolone (Hashimoto *et al.*, 2001) and 5-nitrotropolone (Kubo *et al.*, 2002). The crystal structures of tropolone (Shimanouchi & Sasada, 1973), 5-cyano- and 5-nitrotropolones (Kubo *et al.*, 2001), and 5-acetoxytropolone (Kubo, Yamamoto *et al.*, 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)

Compound (I) forms O—H···O hydrogen-bonded dimers about inversion centres, involving the OH group and an intermolecular carbonyl O1^{i} acceptor (see Table 1 for symmetry code). An intramolecular O—H···O bond to O1 occurs simultaneously (Table 1, Fig. 1). The intermolecular O2···O1^{i} distance of 2.710 (2) Å is close to that in tropolone (2.746 Å; Shimanouchi & Sasada, 1973), where a similar dimerization occurs.

Intermolecular π–π interactions occur in (I) between the tropolone ring planes, as shown in Fig. 2. The C3···C6^{v} [symmetry code: (v) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$] separation is 3.2559 (16) Å, which is within the range associated with π–π interactions (3.3–3.8 Å; Kubo *et al.*, 2001; Kubo *et al.*, 2002). This value is shorter than that of tropolone (3.418 Å; Shimanouchi & Sasada, 1973) and 5-(acetoxy)tropolone [3.455 (2) Å; Kubo, Yamamoto *et al.*, 2006].

Intermolecular C—H···π and C—H···O interactions are observed in the crystal structure of (I) (Table 1, Fig. 2), with distances typical for these types of interactions: C—H···π =

2.8–3.1 Å (Kubo, Fukeda *et al.*, 2006; Kubo, Yamamoto *et al.*, 2006) and C—H···O = 2.5–2.7 Å (Kubo, Fukeda *et al.*, 2006; Kubo, Matsumoto *et al.*, 2006; Kubo, Yamamoto *et al.*, 2006).

The combination of all of the above interactions builds up a three-dimensional network.

Experimental

Compound (I) was prepared by the reaction of 5-hydroxytropolone with dimethylsulfate (Nozoe *et al.*, 1953). Crystals of (I) were grown from a methanol solution by slow evaporation.

Crystal data

$C_8H_8O_3$
M_r = 152.15
Monoclinic, $P2_1/n$
a = 8.434 (2) Å
b = 7.265 (2) Å
c = 12.308 (3) Å
β = 106.976 (15)°
V = 721.3 (3) Å^3
Z = 4
D_x = 1.401 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 0.91 mm^{-1}
T = 153.1 K
Prism, colourless
0.38 × 0.19 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.573, T_{max} = 0.888
6663 measured reflections
1272 independent reflections
1169 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.036
θ_{max} = 68.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.081
S = 1.13
1272 reflections
106 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 0.1908P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick 1997)
Extinction coefficient: 0.0228 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O1	0.92 (2)	2.07 (2)	2.605 (1)	115 (1)
O2—H1···O1^{i}	0.92 (2)	1.96 (2)	2.710 (2)	138 (2)
C6—H4···O2^{ii}	0.95	2.44	3.267 (2)	146
C7—H5···O1^{iii}	0.95	2.61	3.540 (2)	165
C8—H6···C7^{iv}	0.98	2.89	3.530 (2)	124

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x, y-1, z$; (iii) $-x+2, -y, -z+1$; (iv) $-x+1, -y, -z+1$.

The H atom of the OH group was located in a difference map and its position and U_{iso} value were freely refined. All C-bound H atoms were placed in idealized locations (C—H = 0.95–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

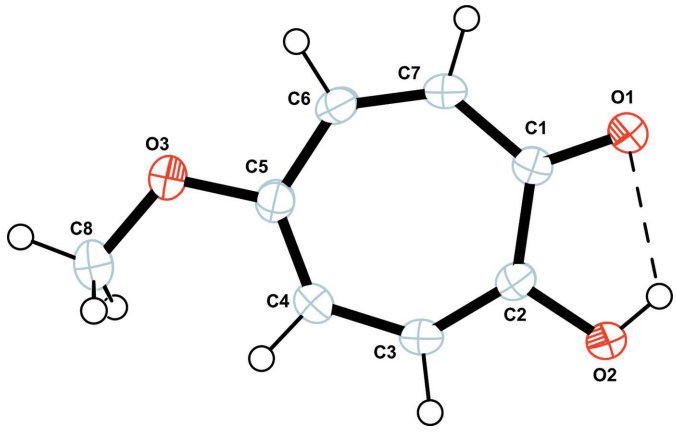


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. The hydrogen bond is indicated by a dashed line.

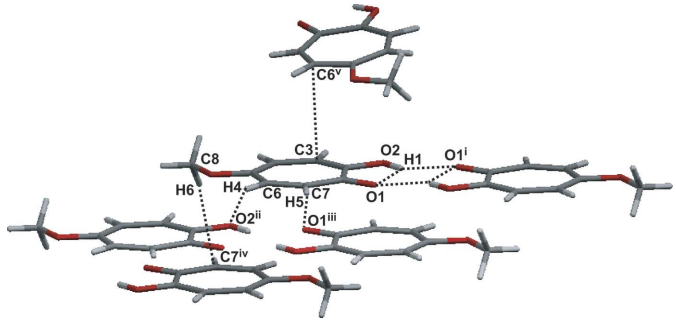


Figure 2
Intermolecular O—H···O, C—H···O, π–π and C—H···π interactions in (I). Dotted lines indicate these interactions. [Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $x, y-1, z$; (iii) $2-x, -y, 1-z$; (iv) $1-x, -y, 1-z$; (v) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$.]

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