Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methacryloxytropone

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.128
Data-to-parameter ratio = 12.4

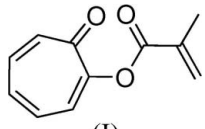
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 8 June 2006
Accepted 19 June 2006

The title compound, 7-oxo-1,3,5-cycloheptatrien-1-yl 2-methyl-2-propenoate, $C_{11}H_{10}O_3$, is known as an intermediate for the synthesis of biologically active polymers. In its crystal structure, the tropolone ring forms a dihedral angle of 69.76 (2)° with the ester plane, and intermolecular C—H···π and C—H···O interactions are observed.

Comment

Tropone and tropolones are important building blocks for constructing liquid crystals (Kubo, Mori *et al.*, 2005), organogelators (Kubo *et al.*, 2004), mercury(II) cation carriers (Mori *et al.*, 1996), chromo- and fluoroionophores (Yamamoto *et al.*, 2002; Yamamoto *et al.*, 2003), dyes (Kubo & Mori, 2001) and intermediates for drug synthesis (Vandecandelaere *et al.*, 1997). Tropone and tropolones are also known to have biological activity against bacteria, fungi and viruses (Trust & Bartlett, 1975). 2-Methacryloxytropone, in particular, has been employed as an intermediate for the synthesis of biologically active polymers (Cornell & Donaruma, 1965).



(I)

The crystal structures of both tropone and tropolone have been reported previously (Barrow *et al.*, 1973; Shimanouchi & Sasada, 1973). We have also reported the structures of 5-cyano- and 5-nitrotropolone (Kubo, Yamamoto & Mori, 2001), 2-butanoyloxy-5-nitrotropone (Kubo, Tsuruta & Mori, 2001), and 5-phenyltropone (Kubo, Matsumoto & Mori, 2005). We report here the crystal structure of 2-methacryloxytropone, (I), with the aim of contributing to a deeper understanding of the molecular structure, molecular assembly and biological activity of troponoids.

The seven-membered ring in (I) is nearly planar; the respective deviations of each atom from the least-squares plane defined by atoms C1–C7/O1/O2, are 0.0002 (13), 0.0584 (13), −0.0105 (13), −0.1031 (12), −0.0436 (13), 0.0959 (15), 0.1107 (13), −0.1134 (8) and 0.0424 (6) Å. The dihedral angle between the C1–C7/O1/O2 plane and the plane of the ester group (defined by atoms O2, O5 and C8) is 69.76 (2)°, similar to that in 2-butanoyloxy-5-nitrotropone (71.8°; Kubo, Tsuruta & Mori, 2001) and bis(5-hexyloxytropon-2-yl)-4,4-azobisbenzoate (71.8°; Kubo, Mori *et al.*, 2005). The C—C bond lengths of the seven-membered ring of (I) are different from those of tropolone (Shimanouchi &

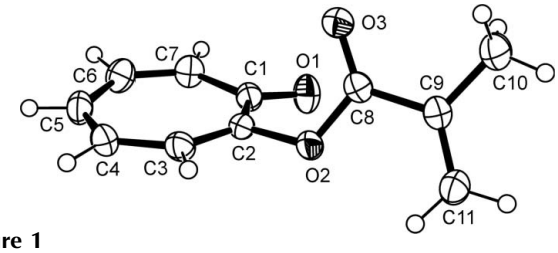


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms.

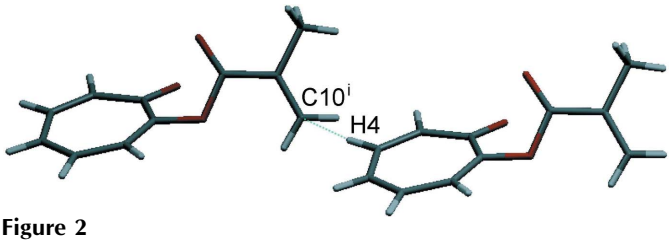


Figure 2
The C—H···π interaction (indicated by a dotted line) in (I) [symmetry code: (i) x, 1 + y, z].

Sasada, 1973) but similar to those of tropone (Barrow *et al.*, 1973).

Intermolecular C—H···π and C—H···O interactions are observed in the crystal structure (Table 2 and Fig. 2), with distances typical for these types of interactions: C—H···π 2.8–3.1 Å (Matsumoto *et al.*, 2002; Kubo, Matsumoto & Mori, 2005), C—H···O 2.5–2.7 Å (Kubo, Matsumoto & Mori, 2005; Takahashi *et al.*, 2006).

Experimental

Compound (I) was prepared by esterification of tropolone with methacryl chloride, according to a literature procedure (Cornell & Donaruma, 1965). Crystals of (I) were grown by slow evaporation of a hexane solution.

Crystal data

$C_{11}H_{10}O_3$
M_r = 190.20
Monoclinic, $P2_1/c$
a = 6.1152 (14) Å
b = 10.214 (2) Å
c = 15.113 (3) Å
β = 97.263 (15)°
V = 936.4 (3) Å^3
Z = 4
D_x = 1.349 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 0.82 mm^{-1}
T = 173.1 K
Block, colorless
0.30 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
φ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.643, T_{max} = 0.885
11901 measured reflections
1702 independent reflections
1441 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.025
θ_{max} = 68.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.128
S = 1.01
1702 reflections
137 parameters
H-atom parameters constrained
$w = 1/[0.0021F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2304 (19)	C3—C4	1.420 (2)
O2—C2	1.3962 (16)	C4—C5	1.355 (2)
C1—C2	1.470 (2)	C5—C6	1.422 (2)
C1—C7	1.4613 (18)	C6—C7	1.350 (2)
C2—C3	1.353 (2)		
C2—O2—C8—O3	14.13 (19)	O2—C8—C9—C10	−3.1 (2)
C2—O2—C8—C9	−167.73 (11)	O2—C8—C9—C11	176.49 (12)
C8—O2—C2—C1	65.47 (15)	O3—C8—C9—C10	174.94 (15)
C8—O2—C2—C3	−120.83 (13)	O3—C8—C9—C11	−5.4 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H4···C10^{i}	0.95	2.84	3.702 (2)	152
C3—H1···O3^{ii}	0.95	2.51	3.240 (2)	134
C7—H5···O1^{iii}	0.95	2.59	3.523 (2)	166
C10—H6···O2^{iv}	0.95	2.71	3.398 (2)	130
C10—H7···O1^{v}	0.95	2.49	3.325 (2)	147

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

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).

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*.

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