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# 3-Hydroxytropolone (2,7-dihydroxycyclohepta-2,4,6-trien-1-one)

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**Key indicators**

Single-crystal X-ray study
*T* = 153 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.036
*wR* factor = 0.107
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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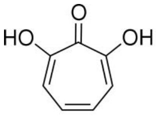
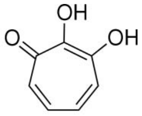
The title compound, $C_7H_6O_3$, exists in the crystalline state as the 2,7-dihydroxycyclohepta-2,4,6-trien-1-one tautomer rather than as 2,3-dihydroxycyclohepta-2,4,6-trien-1-one. Intermolecular O—H···O hydrogen bonds link the molecules into centrosymmetric dimers. The crystal packing is further stabilized by π–π interactions and weak intermolecular C—H···O hydrogen bonds.

## Comment

Crystal engineering is the planning and construction of the structure and properties of crystalline materials by designing molecular building blocks (Steed & Atwood, 2000; Matsumoto *et al.*, 2002). Troponoids have been an important building block for the construction of liquid crystals and organogels (Kubo *et al.*, 2004, 2005). Tropolone (Shimanouchi & Sasada, 1973) and its 5-substituted derivatives (Kubo *et al.*, 2001, 2006*a*,*b*) form centrosymmetric O—H···O hydrogen-bonded dimers, while 3-hydroxytropolone exhibits two tautomers, *viz*. 2,7-dihydroxycyclohepta-2,4,6-trien-1-one, (I), and 2,3-dihydroxycyclohepta-2,4,6-trien-1-one, (I′) (Tsuji *et al.*, 1999). The crystal structure determination of the title compound, (I), has been undertaken to determine its major tautomer and hydrogen-bonding scheme.


(I)


(I′)

In (I) (Fig. 1), the C—C bond lengths are normal and typical for tropolone (Shimanouchi & Sasada, 1973). The C═O [1.2629 (15) Å] and C—O [1.3466 (16) and 1.3553 (14) Å] bond lengths are similar to those observed in the structure of unsubsituted tropolone [1.261 (3) and 1.333 (3) Å for C═O and C—O, respectively; Shimanouchi & Sasada, 1973]. The longer C2—O2 and C7—O3 bond lengths and free refinement of two hydroxyl H atoms atoms bonded to O2 and O3 make the assignment of 7-hydroxytropolone, (I), rather than 3-hydroxytropolone, (I′), unambiguous. Both hydroxyl H atoms are involved in the formation of intramolecular O—H···O hydrogen bonds (Table 1).

Intermolecular O—H···O hydrogen bonds (Table 1) link molecules into centrosymmetric dimers with an intermolecular O1···O2 distance of 2.7144 (14) Å, which is close to that found in tropolone (2.746 Å; Shimanouchi & Sasada, 1973). The short C1···C5$^{vi}$ distance of 3.3554 (16) Å [symmetry code: (vi) $x - 1$, $y$, $z$] indicates the presence of π–π interactions;

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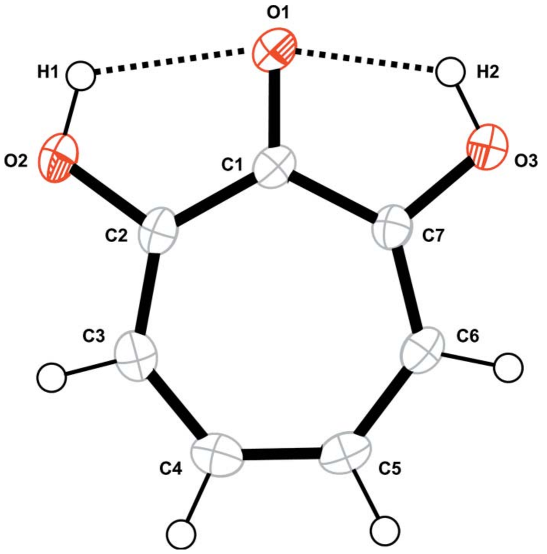

**Figure 1**
The molecular structure of (I), showing 50% probability displacement ellipsoids. The hydrogen bonds are indicated by dotted lines.

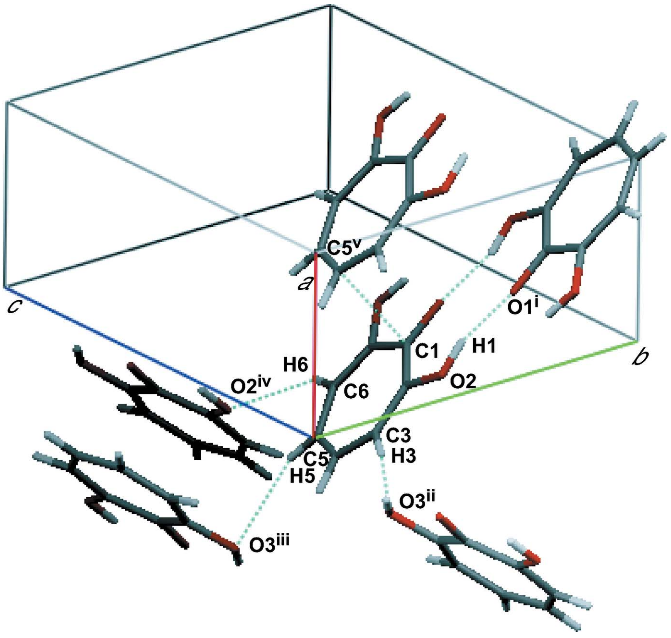

**Figure 2**
A portion of the crystal packing, showing intermolecular O—H$\cdots$O, C—H$\cdots$O and $\pi$–$\pi$ interactions as dotted lines [symmetry codes: (i) $1 - x$, $1 - y$, $-z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $-\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (v) $x - 1, y, z$].

these are associated with the distance range 3.3–3.8 Å (Prout *et al.*, 1973; Kubo *et al.*, 2001). The $\pi$–$\pi$ interactions and weak intermolecular C—H$\cdots$O hydrogen bonds (Table 1) stabilize the crystal packing (Fig. 2).

## Experimental

Compound (I) was prepared by hydrolysis of 2,7-diacetoxytropone with aqueous 50% acetic acid (Takeshita *et al.*, 1987). Crystals of (I) were grown from a chloroform solution by slow evaporation.

### *Crystal data*

$C_7H_6O_3$
$M_r = 138.12$
Monoclinic, $P2_1/n$
$a = 4.9981$ (19) Å
$b = 9.395$ (3) Å
$c = 13.279$ (5) Å
$\beta = 96.9657$ (19)°
$V = 618.9$ (4) Å$^3$

$Z = 4$
$D_x = 1.482$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.12$ mm$^{-1}$
$T = 153.1$ K
Prism, colourless
0.18 × 0.15 × 0.10 mm

### *Data collection*

Rigaku Saturn diffractometer
$\omega$ scans
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
$T_{min} = 0.920$, $T_{max} = 0.988$

4741 measured reflections
1364 independent reflections
1109 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.029$
$\theta_{max} = 27.5°$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.107$
$S = 1.01$
1364 reflections
103 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[0.0011F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O2—H1$\cdots$O1 | 0.923 (18) | 2.121 (18) | 2.6212 (13) | 112.8 (15) |
| O2—H1$\cdots$O1$^{i}$ | 0.923 (18) | 1.910 (19) | 2.7144 (14) | 144.5 (16) |
| O3—H2$\cdots$O1 | 0.91 (2) | 1.93 (2) | 2.5406 (13) | 122.4 (16) |
| C3—H3$\cdots$O3$^{ii}$ | 0.95 | 2.62 | 3.5640 (15) | 176 |
| C5—H5$\cdots$O3$^{iii}$ | 0.95 | 2.57 | 3.3805 (16) | 143 |
| C6—H6$\cdots$O2$^{iv}$ | 0.95 | 2.47 | 3.2021 (16) | 134 |

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

The H atom of the OH groups were located in a difference map, and their positions and $U_{iso}$(H) values were freely refined. The C-bound H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 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; Macrae *et al.*, 2006); software used to prepare material for publication: *CrystalStructure* 3.8 (Rigaku/MSC, 2006).

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