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# 3,4-Dihydroxy-9*H*-xanthen-9-one trihydrate

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**Key indicators**

Single-crystal X-ray study
$T$ = 291 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.050
$wR$ factor = 0.151
Data-to-parameter ratio = 8.8

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

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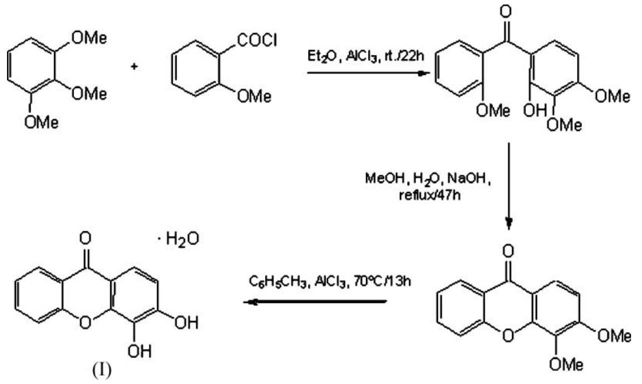
The xanthone skeleton of the title compound, $C_{13}H_8O_4\cdot3H_2O$, exhibits a planar conformation, with the hydroxy H atoms lying in the plane. In the crystal structure, the molecules are packed into columns with their planar skeletons parallel to one another.

## Comment

Structural elucidation of xanthones is essential for understanding the wide range of biological and pharmacological activities described for these compounds. The structure determination of 3,4-dihydroxyxanthone is part of our ongoing research concerning the structural properties of xanthones (Kijjoa *et al.*, 1998; Gales *et al.*, 2001). The title compound, (I), is a key precursor for the synthesis of more complex xanthonic derivatives (Sousa *et al.*, 2002). This simple oxygenated xanthone has been assessed for its capacity to inhibit, *in vitro*, the growth of three human cancer lines, *viz.* MCF-7 (breast cancer), TK-10 (renal cancer) and UACC-62 (melanoma) with $GI_{50}$ values in the range $22 < GI_{50} < 60$ µ*M* (Pedro *et al.*, 2002). 3,4-Dihydroxyxanthone was found to possess a pronounced inhibitory activity on the mitogenic response of human lymphocytes to PHA ($IC_{50}$ = 12.2 ± 1.3 µ*M*). During elucidation of the mechanism of the antiproliferative activity, the title compound revealed an effect compatible with PKC inhibition (Saraiva *et al.*, 2003). For some isoforms, 3,4-dihydroxyxanthone showed potencies even higher than those presented by standard PKC inhibitors, chelerythrine and NPC 15437, especially for isoforms $\eta$ and $\zeta$. The potencies presented by the title compound on different PKC isoforms have suggested the potential of the xanthone framework for the development of new isoform-selective PKC inhibitors.



In the crystal structure, the three-ring system is approximately planar (r.m.s. deviation from the least-squares plane is 0.028 Å). The maximum deviation from the xanthone skeleton

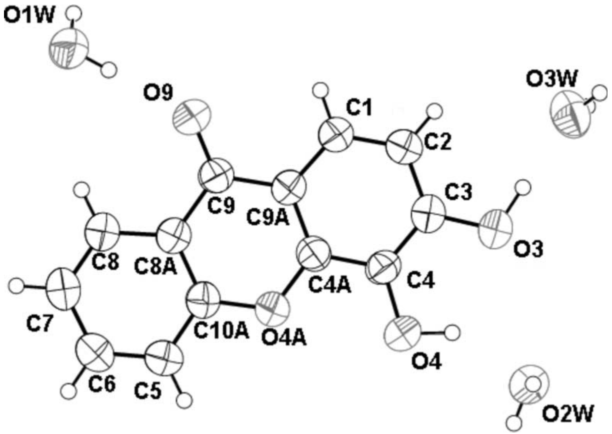


**Figure 1**
View of the molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

plane is 0.069 (3) Å for atom O9. The two OH substituents lie in the aromatic plane (Fig. 1), which is in agreement with the crystal structure of another xanthone with OH groups at C3 and C4 (Ishiguro *et al.*, 1993).

Atoms O3, O4 and O9 are hydrogen bonded to three distinct water molecules. The crystallographic packing reveals that the water molecules interact with each other, making a network of hydrogen bonds that mediate stabilizing interactions between the different molecules in the crystal structure (Figs. 2 and 3, and Table 1).

## Experimental

The title compound was prepared according to the procedure of Gottlieb *et al.* (1970). The compound crystallizes as thin plates.

### *Crystal data*

$C_{13}H_8O_4{\cdot}3H_2O$
$M_r = 282.24$
Triclinic, $P\overline{1}$
$a = 6.838$ (3) Å
$b = 9.946$ (4) Å
$c = 10.087$ (4) Å
$\alpha = 105.94$ (5)°
$\beta = 96.54$ (5)°
$\gamma = 103.44$ (5)°
$V = 629.9$ (5) Å$^3$
$Z = 2$
$D_x = 1.488$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 34 reflections
$\theta = 6.5$–16.7°
$\mu = 0.12$ mm$^{-1}$
$T = 291$ (2) K
Plate, colorless
0.4 × 0.15 × 0.08 mm

### *Data collection*

Stoe IPDS-1 diffractometer
$\varphi$ scans
Absorption correction: none
4054 measured reflections
1882 independent reflections
1270 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\rm max} = 24.2°$
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 11$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.151$
$S = 1.05$
1882 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2 + 0.0203P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.19$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.16$ e Å$^{-3}$

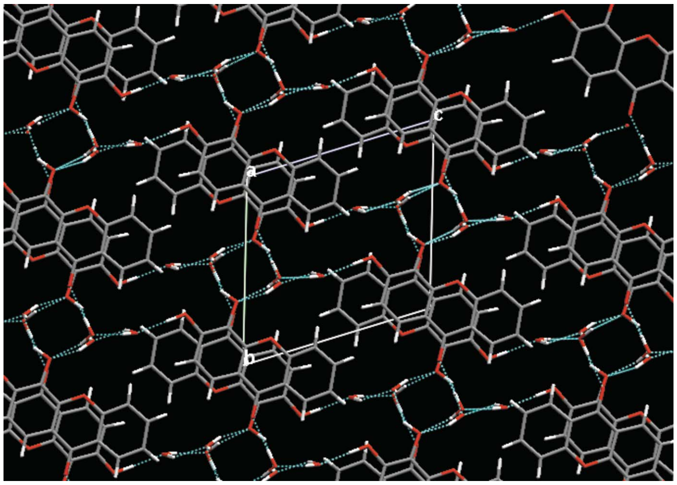


**Figure 2**
The molecular packing viewed along the *a* axis. The stabilizing hydrogen-bond interactions are indicated by dashed lines.

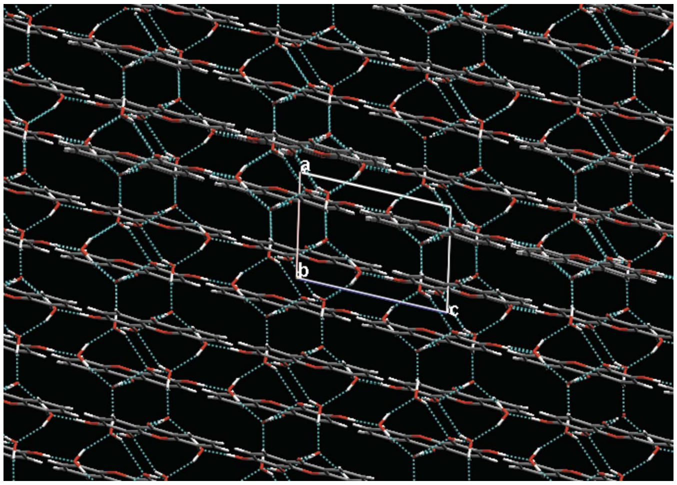


**Figure 3**
The molecular packing viewed along the *b* axis. Hydrogen-bond interactions are indicated by dashed lines.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O3$W$ | 0.99 (4) | 1.73 (4) | 2.717 (3) | 177 (3) |
| O4—H4···O2$W$ | 0.98 (5) | 1.84 (5) | 2.702 (3) | 145 (4) |
| O1$W$—H11$W$···O4$^{\rm i}$ | 0.85 (5) | 2.17 (5) | 2.999 (4) | 168 (4) |
| O1$W$—H12$W$···O9 | 0.92 (5) | 1.89 (5) | 2.805 (3) | 174 (4) |
| O2$W$—H21$W$···O1$W^{\rm ii}$ | 0.98 (5) | 1.95 (5) | 2.915 (5) | 170 (3) |
| O2$W$—H22$W$···O4$^{\rm iii}$ | 0.84 (6) | 2.66 (6) | 3.343 (4) | 141 (5) |
| O3$W$—H31$W$···O2$W^{\rm iv}$ | 0.85 (6) | 2.21 (6) | 2.994 (4) | 152 (5) |
| O3$W$—H32$W$···O1$W^{\rm v}$ | 0.89 (6) | 2.09 (6) | 2.968 (4) | 172 (5) |

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

C-bound H atoms were positioned with idealized geometry and refined riding on their parent C atom at distances of 0.93 Å, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The hydroxy and water H atoms were refined freely with isotropic displacement parameters. Because of the low scattering power of the small crystals, reflections were only measured up to $\theta_{\rm max} = 24.2°$.

**o2214** Gales *et al.* • $C_{13}H_8O_4{\cdot}3H_2O$ *Acta Cryst.* (2005). E**61**, o2213–o2215

Data collection: *IPDS1* (Stoe & Cie, 1994); cell refinement: *IPDS1*; data reduction: *IPDS1*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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*Acta Cryst.* (2005). E**61**, o2213–o2215 Gales *et al.* • $C_{13}H_8O_4{\cdot}3H_2O$ **o2215**