Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

3,5-Dihydroxy-2-methyl-4*H*-pyran-4-one

Cheng-Ming Dong,[a] Shou-Cheng Pu[b] and Wen-Yuan Gao[b]*

[a]Department of Pharmaceutical Science, Henan College of Traditional Chinese Medicine, Zhengzhou 450008, People's Republic of China, and [b]School of Pharmaceutical Science & Technology, Tianjin University, Tianjin 300072, People's Republic of China
Correspondence e-mail: pharmgao@tju.edu.cn

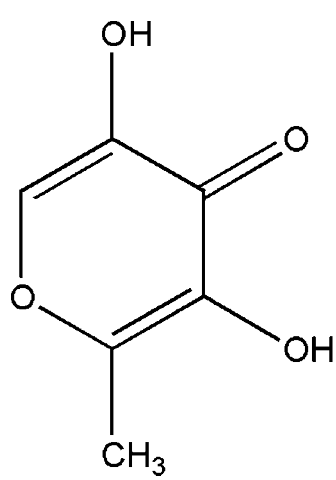
Received 18 March 2008; accepted 19 April 2008

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.097; data-to-parameter ratio = 12.0.

In the title compound, $C_6H_6O_4$, inter- and intramolecular hydrogen bonds are observed which help to establish the crystal structure. There are weak π–π interactions between pyran rings separated by 3.5692 (9) Å.

Related literature

For general background, see: Shinoda *et al.* (2004). For related structures, see: Yao *et al.* (2005); Gibbons *et al.* (2000).



Experimental

Crystal data

$C_6H_6O_4$
M_r = 142.11
Monoclinic, $P2_1/n$
a = 6.9400 (14) Å
b = 6.0648 (12) Å
c = 14.008 (3) Å
β = 92.77 (3)°
V = 588.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 113 (2) K
0.14 × 0.12 × 0.10 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.981, T_{max} = 0.986
3970 measured reflections
1381 independent reflections
1166 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.096
S = 1.10
1381 reflections
115 parameters
All H-ataom parameters refined
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H6···O3^{i}	0.838 (18)	1.89 (2)	2.6902 (12)	159.6 (13)
O2—H5···O3^{ii}	0.94 (2)	1.75 (2)	2.6596 (12)	162.6 (17)
O4—H6···O3	0.838 (18)	2.44 (2)	2.7820 (12)	105.4 (10)
C1—H3···O4	1.005 (15)	2.537 (14)	2.8957 (15)	100.5 (9)
C6—H4···O2^{iii}	0.936 (14)	2.412 (13)	3.3354 (14)	169.4 (12)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2074).

References

Gibbons, S., Denny, B. J., Ali-Amine, S., Mathew, K. T., Skelton, B. W., White, A. H. & Gray, A. I. (2000). *J. Nat. Prod.* **63**, 839–840.
Rigaku/MSC. (2005). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shinoda, Y., Murata, M., Homma, S. & Komura, H. (2004). *Biosci. Biotechnol. Biochem.* **68**, 529–536.
Yao, G.-M., Wang, Y.-B., Wang, L.-Q. & Qin, G.-W. (2005). *Acta Cryst.* E**61**, o1403–o1405.

supporting information

Acta Cryst. (2008). E**64**, o1032 [doi:10.1107/S1600536808010957]

3,5-Dihydroxy-2-methyl-4*H*-pyran-4-one

Cheng-Ming Dong, Shou-Cheng Pu and Wen-Yuan Gao

S1. Comment

The title compound, 3,5-dihydroxy-2-methyl-pyran-4-one, (I) was identified as a decomposition product in the stored solution of orange juice (Shinoda, *et al.*, 2004). We report here the crystal structure of the title compound (Fig. 1) which was isolated from Hydrocotyle sibthorpoioides Lam. The structure of (I) is stabilized by two strong intermolecular hydrogen bonds of the type O—H···O and a weak intermolecular interaction of the type C—H···O. Intramolecular interactions are also observed which result in five membered rings; details are given in Table 1. There is indication of *π*-*π* interactions between the pyran rings lying about inversion centers with minimum separation of 3.5692 (9) Å. The crystal structures of 2-hydroxymethyl analogue (Yao *et al.*, 2005) and 5-hydroxy-3-methoxy-pyran-4-one (Gibbons *et al.*, 2000) have been reported.

S2. Experimental

Dried powder of Hydrocotyle sibthorpoioides Lam was exacted with EtOH and the extract was concentrated *in vacuo*. The residue was subjected to silical-gel coloumn chromatography. Elution with chloroform-methanol (95:5 *v*/*v*) yielded the title compound. Crystals suitable for XRD study were grwon from a solution of methanol at room temperature by slow evaporation.

S3. Refinement

All H atoms were located from difference map and allowed to refine freely.

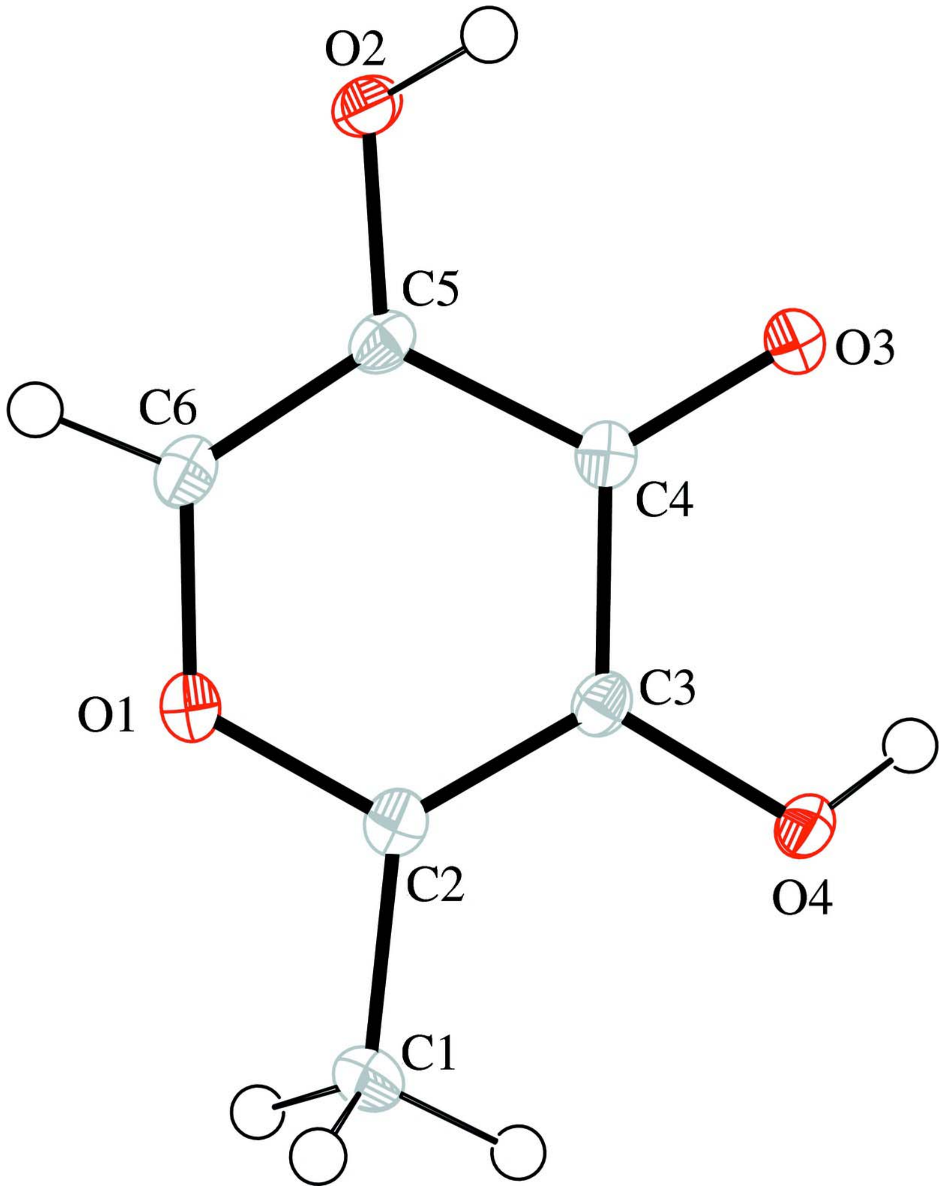


Figure 1

A view of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

3,5-Dihydroxy-2-methyl-4H-pyran-4-one

Crystal data

$C_6H_6O_4$
M_r = 142.11
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 6.9400 (14) Å
b = 6.0648 (12) Å
c = 14.008 (3) Å
β = 92.77 (3)°
V = 588.9 (2) Å^3
Z = 4
F(000) = 296
D_x = 1.603 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1620 reflections
θ = 1.5–27.9°
μ = 0.14 mm^{-1}

T = 113 K
Block, colorless
0.14 × 0.12 × 0.10 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.981, T_{max} = 0.986
3970 measured reflections
1381 independent reflections
1166 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.9°, θ_{min} = 2.9°
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 7$
$l = -10 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.096
S = 1.11
1381 reflections
115 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O3	0.27997 (10)	1.01120 (11)	0.27250 (5)	0.0149 (2)
O1	0.76427 (10)	1.05517 (12)	0.44524 (5)	0.0157 (2)
O4	0.56909 (12)	0.69576 (12)	0.26153 (5)	0.0171 (2)
O2	0.32648 (11)	1.36372 (12)	0.40092 (5)	0.0173 (2)
C4	0.43152 (15)	1.02588 (15)	0.32615 (7)	0.0124 (2)
C5	0.46324 (15)	1.20540 (16)	0.39217 (7)	0.0132 (2)
C3	0.58183 (15)	0.86536 (16)	0.32479 (7)	0.0127 (2)
C2	0.74196 (15)	0.88197 (16)	0.38483 (7)	0.0139 (2)
C6	0.62774 (16)	1.21333 (17)	0.44749 (7)	0.0157 (2)
C1	0.90601 (15)	0.72547 (19)	0.39113 (8)	0.0174 (3)
H4	0.656 (2)	1.323 (2)	0.4929 (10)	0.021 (3)*
H3	0.871 (2)	0.582 (2)	0.3585 (10)	0.028 (3)*
H1	1.017 (2)	0.782 (2)	0.3616 (11)	0.037 (4)*
H2	0.940 (2)	0.689 (2)	0.4591 (10)	0.025 (3)*

H5	0.271 (3)	1.393 (3)	0.3397 (14)	0.054 (5)*
H6	0.454 (3)	0.669 (2)	0.2453 (11)	0.037 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0141 (4)	0.0150 (4)	0.0153 (4)	0.0004 (3)	−0.0022 (3)	−0.0013 (3)
O1	0.0155 (4)	0.0165 (4)	0.0148 (4)	−0.0005 (3)	−0.0020 (3)	−0.0020 (3)
O4	0.0136 (4)	0.0163 (4)	0.0211 (4)	0.0004 (3)	−0.0008 (3)	−0.0080 (3)
O2	0.0233 (4)	0.0142 (4)	0.0142 (4)	0.0058 (3)	−0.0018 (3)	−0.0018 (3)
C4	0.0142 (5)	0.0125 (5)	0.0105 (4)	−0.0022 (4)	0.0016 (4)	0.0017 (3)
C5	0.0177 (5)	0.0106 (5)	0.0116 (4)	0.0007 (4)	0.0021 (4)	0.0008 (3)
C3	0.0138 (5)	0.0118 (5)	0.0128 (5)	−0.0019 (4)	0.0023 (4)	−0.0011 (3)
C2	0.0145 (5)	0.0141 (5)	0.0132 (4)	−0.0017 (4)	0.0024 (4)	−0.0003 (3)
C6	0.0200 (6)	0.0133 (5)	0.0139 (5)	−0.0012 (4)	0.0006 (4)	−0.0020 (4)
C1	0.0126 (5)	0.0200 (6)	0.0195 (5)	0.0013 (4)	0.0001 (4)	−0.0010 (4)

Geometric parameters (Å, º)

O3—C4	1.2659 (13)	C4—C5	1.4386 (13)
O1—C6	1.3497 (13)	C5—C6	1.3494 (16)
O1—C2	1.3531 (12)	C3—C2	1.3646 (15)
O4—C3	1.3577 (12)	C2—C1	1.4816 (15)
O4—H6	0.838 (18)	C6—H4	0.936 (14)
O2—C5	1.3598 (12)	C1—H3	1.005 (15)
O2—H5	0.94 (2)	C1—H1	0.956 (17)
C4—C3	1.4276 (14)	C1—H2	0.996 (15)
C6—O1—C2	120.47 (8)	O1—C2—C3	120.53 (9)
C3—O4—H6	110.7 (11)	O1—C2—C1	113.31 (9)
C5—O2—H5	107.9 (12)	C3—C2—C1	126.15 (9)
O3—C4—C3	122.06 (9)	C5—C6—O1	122.45 (9)
O3—C4—C5	122.13 (9)	C5—C6—H4	124.0 (8)
C3—C4—C5	115.82 (9)	O1—C6—H4	113.5 (8)
C6—C5—O2	119.86 (9)	C2—C1—H3	111.1 (8)
C6—C5—C4	119.68 (10)	C2—C1—H1	112.2 (9)
O2—C5—C4	120.44 (9)	H3—C1—H1	107.1 (13)
O4—C3—C2	118.92 (9)	C2—C1—H2	110.3 (8)
O4—C3—C4	120.04 (9)	H3—C1—H2	106.4 (12)
C2—C3—C4	121.01 (9)	H1—C1—H2	109.5 (13)
O3—C4—C5—C6	−179.79 (9)	C6—O1—C2—C1	179.47 (9)
C3—C4—C5—C6	0.12 (14)	O4—C3—C2—O1	176.47 (9)
O3—C4—C5—O2	1.94 (15)	C4—C3—C2—O1	−1.93 (15)
C3—C4—C5—O2	−178.15 (8)	O4—C3—C2—C1	−2.39 (16)
O3—C4—C3—O4	3.12 (15)	C4—C3—C2—C1	179.21 (9)
C5—C4—C3—O4	−176.78 (8)	O2—C5—C6—O1	176.71 (9)
O3—C4—C3—C2	−178.50 (9)	C4—C5—C6—O1	−1.58 (15)

C5—C4—C3—C2	1.59 (14)	C2—O1—C6—C5	1.32 (15)
C6—O1—C2—C3	0.47 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H6···O3^{i}	0.838 (18)	1.89 (2)	2.6902 (12)	159.6 (13)
O2—H5···O3^{ii}	0.94 (2)	1.75 (2)	2.6596 (12)	162.6 (17)
O4—H6···O3	0.838 (18)	2.44 (2)	2.7820 (12)	105.4 (10)
C1—H3···O4	1.005 (15)	2.537 (14)	2.8957 (15)	100.5 (9)
C6—H4···O2^{iii}	0.936 (14)	2.412 (13)	3.3354 (14)	169.4 (12)

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iii) −*x*+1, −*y*+3, −*z*+1.