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Garcinia lactone

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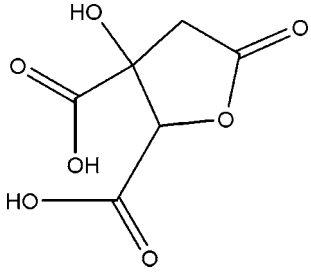
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.066; data-to-parameter ratio = 7.1.

The title compound, $C_6H_6O_7$, garcinia lactone [systematic name: (2*S*,3*S*)-3-hydroxy-5-oxo-2,3,4,5-tetrahydrofuran-2,3-dicarboxylic acid] was isolated from the rind of *Garcinia cambogia*. The five-membered ring adopts an envelope conformation and the packing is stabilized by O—H···O and C—H···O interactions.

Related literature

For background literature, see: Sullivan *et al.* (1974); Clouatre & Robenbaum (1994). For the extraction procedure, see: Balasubramanyam *et al.*, (2000).



Experimental

Crystal data

$C_6H_6O_7$
M_r = 190.11
Orthorhombic, $P2_12_12_1$
a = 6.2657 (10) Å
b = 8.6591 (14) Å
c = 13.504 (2) Å
V = 732.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.935, T_{max} = 0.984
5568 measured reflections
836 independent reflections
810 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.066
S = 1.10
836 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	1.95	2.764 (2)	173
O4—H4···O6^{ii}	0.82	1.90	2.721 (2)	174
O7—H7···O3^{iii}	0.82	1.96	2.697 (2)	150
C4—H4*A*···O2^{iv}	0.98	2.46	3.268 (2)	139
C2—H2*A*···O4	0.97	2.40	2.808 (3)	104
C2—H2*A*···O5^{v}	0.97	2.49	3.163 (2)	127

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2498).

References

Balasubramanyam, K., Chandrasekhar, B., Ramadoss, C. S. & Rao, P. V. S. (2000). US Patent 6 160 172 A.
Bruker (2000). *SHELXTL*. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *SMART* (Version 5.628) and *SAINT* (Version 6.45a). Bruker AXS Inc., Madison, Wisconsin, USA.
Clouatre, D. & Robenbaum, M. E. (1994). *The Diet and Health Benefits of HCA*. New Canaan, Connecticut: Keats Publishing.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Sullivan, A. C., Triscari, J., Hamilton, J. G., Miller, O. N. & Wheatley, V. R. (1974). *Lipids*, **9**, 121–128.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

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supplementary materials

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Garcinia lactone

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Comment

The main active ingredient of Garcinia cambogia fruit rind is (-)hydroxycitric acid (HCA) along with Garcinia lactone and citric acid (Clouatre & Robenbaum, 1994). In India the fruit is known as kokum, the extract of which is used as a souring agent in cooking. In Indian medicine, Garcinia is considered to be one of the prime herbs that is beneficial for heart. Garcinia has also received worldwide attention as a nutraceutical for effective obesity control (Sullivan *et al.*, 1974; Clouatre & Robenbaum, 1994). The title compound, (I), (Fig. 1) was extracted by a novel procedure (Balasubramanyam *et al.*,2000).

The five membered C1/C2/C3/C4/O2 lactone ring in (I) adopts an envelope conformation with C3 deviating by 0.539 (1) Å from the plane of the other four atoms. Selected torsion angles for O2/C4/C5/C6 and C2/C3/C6/O5 are −20.88 (1)° and −154.45 (1)°, respectively.

The crystal structure of (I) is stabilized by intermolecular O—H···O and intra- and intermolecular C—H···O hydrogen bonds (Table 1, Fig. 2).

Experimental

Crushed and dried Garcinia cambogia fruit rinds (200 g) were immersed in 250 ml hot water for 10 h. Water was decanted and the process was repeated three times. The combined water extracts were concentrated to get a thick syrupy mass to which acetone was added. The precipitated mass was filtered off and washed with acetone. On evaporation, the acetone layer gave a gummy mass which was extracted with ethyl acetate. The ethyl acetate was charcolized, dried over anhydrous sodium sulfate, which on concentration gave crude garcinia lactone. This material was recrystallized using ethyl acetate and n-hexane to yield the title compound (29.0 g) in high purity. Colourless plates of (I) were grown from ethyl acetate and n-hexane (1:1 *v*/*v*) for data collection.

Refinement

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. Therefore the absolute structure of (I) is indeterminate from this experiment (in the arbitrarily chosen model used here, C3 and C4 have S conformation). All the H atoms were positioned geometrically (C—H = 0.97–0.98 Å, O—H = 0.82 Å) and refined as riding, with U_{iso}(H) = 1.5U_{eq}(C) or 1.5U_{eq}(O).

Figures

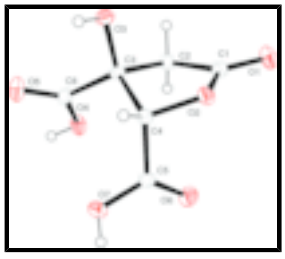

Fig. 1. View of (I) with 30% probability displacement ellipsoids (arbitrary spheres for the H atoms).

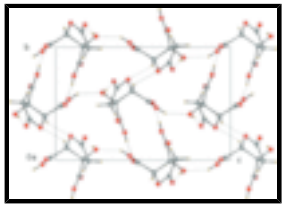

Fig. 2. Molecular chains along *a* axis in (I) due to O—H···O hydrogen bonds and C—H···O interactions (dashed lines).

(2*S*,3*S*)-3-hydroxy-5-oxo-2,3,4,5-tetrahydrofuran-2,3-dicarboxylic acid

Crystal data

$C_6H_6O_7$	D_x = 1.723 Mg m^{-3}
M_r = 190.11	Melting point: 449-451 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 545 reflections
a = 6.2657 (10) Å	θ = 1.0–28.0º
b = 8.6591 (14) Å	μ = 0.16 mm^{-1}
c = 13.504 (2) Å	T = 292 (2) K
V = 732.7 (2) Å^3	Plate, colourless
Z = 4	0.30 × 0.20 × 0.10 mm
F_{000} = 392	

Data collection

Bruker SMART APEX CCD diffractometer	836 independent reflections
Radiation source: fine-focus sealed tube	810 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 292(2) K	θ_{max} = 25.7º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −7→7
T_{min} = 0.935, T_{max} = 0.984	k = −10→10
5568 measured reflections	l = −16→15

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.066$

$S = 1.10$

836 reflections

118 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 0.2219P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.21$ e Å^{-3}

$\Delta\rho_{min} = -0.17$ e Å^{-3}

Extinction correction: none

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 $F^2 > 2$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}
O5	1.0046 (2)	−0.15092 (19)	0.14395 (13)	0.0390 (4)
O6	0.4251 (2)	0.00593 (19)	−0.05515 (12)	0.0350 (4)
O3	0.7966 (2)	0.09450 (18)	0.23129 (10)	0.0281 (4)
H3	0.9172	0.1169	0.2126	0.042*
O2	0.4978 (2)	0.21068 (16)	0.08633 (10)	0.0259 (3)
O7	0.7760 (3)	−0.03680 (19)	−0.07537 (11)	0.0336 (4)
H7	0.7377	−0.0840	−0.1249	0.050*
O4	0.6882 (2)	−0.25433 (17)	0.10495 (12)	0.0339 (4)
H4	0.7592	−0.3321	0.0942	0.051*
O1	0.1911 (2)	0.19661 (19)	0.16734 (13)	0.0371 (4)
C6	0.8148 (3)	−0.1423 (2)	0.13577 (15)	0.0254 (5)
C2	0.4591 (3)	−0.0075 (2)	0.18784 (15)	0.0250 (5)
H2A	0.3931	−0.0972	0.1576	0.030*
H2B	0.4451	−0.0149	0.2592	0.030*
C1	0.3614 (3)	0.1399 (2)	0.14948 (15)	0.0252 (4)
C3	0.6927 (3)	0.0069 (2)	0.15732 (14)	0.0213 (4)

C4	0.6711 (3)	0.1092 (2)	0.06284 (14)	0.0216 (4)
H4A	0.8023	0.1681	0.0515	0.026*
C5	0.6089 (3)	0.0196 (2)	−0.03010 (14)	0.0243 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.0218 (8)	0.0381 (9)	0.0570 (10)	0.0038 (7)	−0.0013 (8)	0.0039 (9)
O6	0.0283 (9)	0.0377 (9)	0.0389 (9)	−0.0021 (8)	−0.0084 (7)	−0.0072 (7)
O3	0.0240 (7)	0.0391 (9)	0.0212 (7)	−0.0047 (7)	−0.0007 (6)	−0.0034 (6)
O2	0.0271 (8)	0.0240 (7)	0.0265 (7)	0.0046 (6)	0.0039 (7)	0.0023 (6)
O7	0.0336 (9)	0.0420 (9)	0.0253 (7)	0.0052 (7)	0.0015 (7)	−0.0095 (7)
O4	0.0287 (8)	0.0251 (7)	0.0480 (10)	0.0020 (7)	−0.0003 (7)	−0.0072 (7)
O1	0.0246 (8)	0.0359 (9)	0.0510 (10)	0.0055 (8)	0.0084 (8)	−0.0037 (8)
C6	0.0244 (10)	0.0287 (11)	0.0230 (10)	0.0010 (9)	0.0000 (9)	0.0061 (9)
C2	0.0221 (10)	0.0274 (10)	0.0254 (10)	0.0003 (9)	0.0036 (8)	0.0016 (9)
C1	0.0228 (10)	0.0264 (10)	0.0263 (10)	−0.0015 (9)	−0.0008 (9)	−0.0039 (9)
C3	0.0207 (10)	0.0260 (10)	0.0173 (9)	−0.0030 (10)	−0.0003 (8)	0.0010 (8)
C4	0.0199 (9)	0.0239 (9)	0.0209 (10)	−0.0009 (9)	0.0016 (8)	0.0006 (8)
C5	0.0279 (11)	0.0233 (10)	0.0215 (10)	0.0005 (9)	0.0002 (8)	0.0019 (8)

Geometric parameters (Å, °)

O5—C6	1.197 (2)	O1—C1	1.199 (3)
O6—C5	1.206 (3)	C6—C3	1.530 (3)
O3—C3	1.413 (2)	C2—C1	1.507 (3)
O3—H3	0.8200	C2—C3	1.526 (3)
O2—C1	1.354 (2)	C2—H2A	0.9700
O2—C4	1.433 (2)	C2—H2B	0.9700
O7—C5	1.307 (3)	C3—C4	1.559 (3)
O7—H7	0.8200	C4—C5	1.526 (3)
O4—C6	1.320 (3)	C4—H4A	0.9800
O4—H4	0.8200		
C3—O3—H3	109.5	O3—C3—C2	107.16 (16)
C1—O2—C4	109.88 (15)	O3—C3—C6	110.94 (16)
C5—O7—H7	109.5	C2—C3—C6	117.51 (17)
C6—O4—H4	109.5	O3—C3—C4	108.30 (16)
O5—C6—O4	125.5 (2)	C2—C3—C4	100.64 (15)
O5—C6—C3	122.1 (2)	C6—C3—C4	111.56 (15)
O4—C6—C3	112.34 (16)	O2—C4—C5	107.47 (15)
C1—C2—C3	103.13 (17)	O2—C4—C3	103.47 (15)
C1—C2—H2A	111.1	C5—C4—C3	114.01 (16)
C3—C2—H2A	111.1	O2—C4—H4A	110.5
C1—C2—H2B	111.1	C5—C4—H4A	110.5
C3—C2—H2B	111.1	C3—C4—H4A	110.5
H2A—C2—H2B	109.1	O6—C5—O7	126.6 (2)
O1—C1—O2	120.2 (2)	O6—C5—C4	121.64 (18)
O1—C1—C2	129.7 (2)	O7—C5—C4	111.72 (18)

O2—C1—C2	110.10 (17)		
C4—O2—C1—O1	−169.51 (18)	C1—O2—C4—C5	91.59 (18)
C4—O2—C1—C2	10.6 (2)	C1—O2—C4—C3	−29.35 (19)
C3—C2—C1—O1	−166.8 (2)	O3—C3—C4—O2	−76.88 (18)
C3—C2—C1—O2	13.1 (2)	C2—C3—C4—O2	35.34 (19)
C1—C2—C3—O3	84.37 (18)	C6—C3—C4—O2	160.76 (15)
C1—C2—C3—C6	−150.01 (17)	O3—C3—C4—C5	166.72 (15)
C1—C2—C3—C4	−28.73 (19)	C2—C3—C4—C5	−81.1 (2)
O5—C6—C3—O3	−30.7 (3)	C6—C3—C4—C5	44.4 (2)
O4—C6—C3—O3	151.34 (16)	O2—C4—C5—O6	−20.9 (3)
O5—C6—C3—C2	−154.5 (2)	C3—C4—C5—O6	93.2 (2)
O4—C6—C3—C2	27.6 (2)	O2—C4—C5—O7	159.30 (16)
O5—C6—C3—C4	90.1 (3)	C3—C4—C5—O7	−86.7 (2)
O4—C6—C3—C4	−87.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.82	1.95	2.764 (2)	173
O4—H4···O6^{ii}	0.82	1.90	2.721 (2)	174
O7—H7···O3^{iii}	0.82	1.96	2.697 (2)	150
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Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*+1/2, −*y*−1/2, −*z*; (iii) −*x*+3/2, −*y*, *z*−1/2; (iv) *x*+1/2, −*y*+1/2, −*z*; (v) *x*−1, *y*, *z*.

Fig. 1

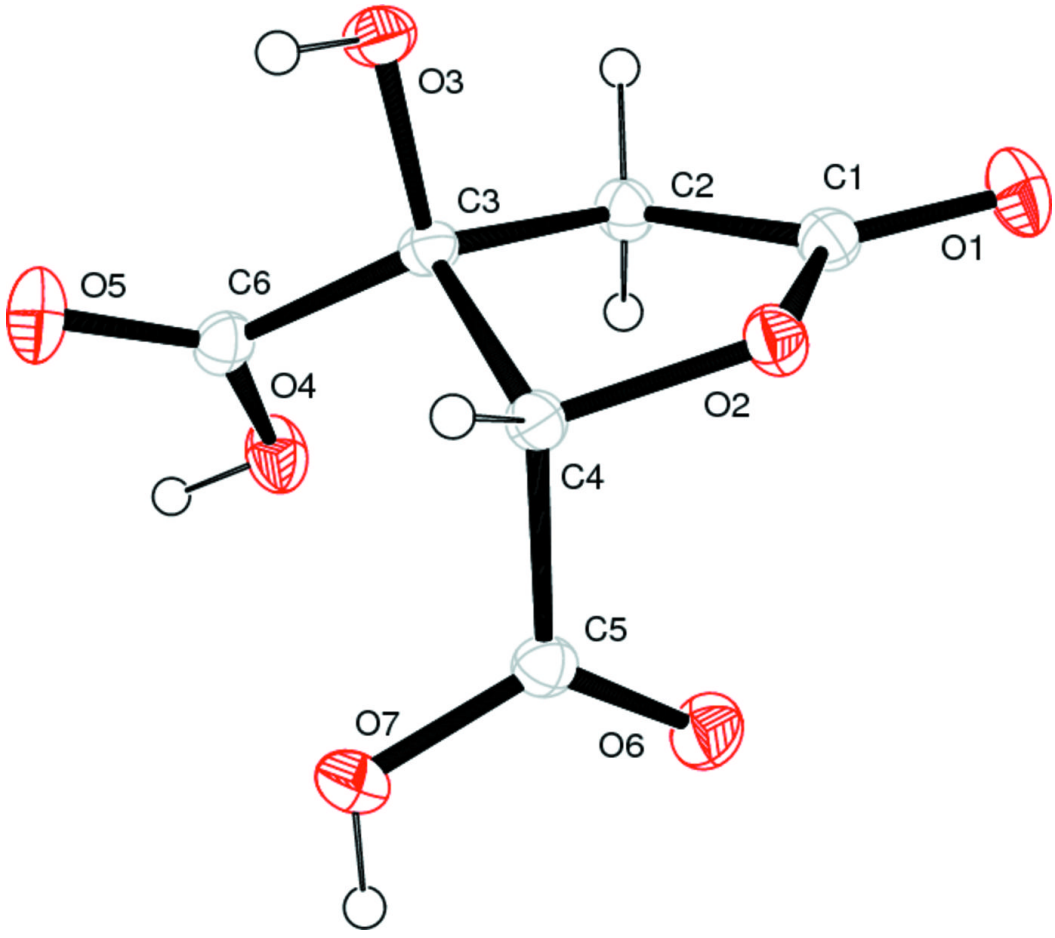

Fig. 2