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6-Methyl citrate from *Dioscorea opposita* Thunb.

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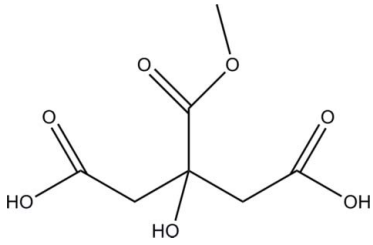
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.072; data-to-parameter ratio = 8.1.

The title compound (systematic name: methyl 3-carboxy-2-carboxymethyl-2-hydroxypropanoate), $C_7H_{10}O_7$, was isolated from the Chinese yam *Dioscorea opposita* Thunb. In the crystal structure, the molecules present a two-dimensional structure arising from intermolecular O—H···O hydrogen bonds.

Related literature

For related literature, see: Sautour *et al.* (2004).



Experimental

Crystal data

$C_7H_{10}O_7$
M_r = 206.15
Orthorhombic, $P2_12_12_1$
a = 5.8826 (14) Å
b = 11.927 (3) Å
c = 12.752 (3) Å
V = 894.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 296 (2) K
0.16 × 0.14 × 0.11 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.978, T_{max} = 0.985
4979 measured reflections
1047 independent reflections
1019 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.072
S = 1.14
1047 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O6^{i}	0.82	1.89	2.7011 (19)	171
O3—H2···O2^{ii}	0.82	2.03	2.8164 (18)	162
O4—H4···O5^{iii}	0.82	1.87	2.6854 (19)	176

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2611).

References

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supplementary materials

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6-Methyl citrate from *Dioscorea opposita* Thunb.

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Comment

Some medicinal components have been successfully isolated and indentified from the Dioscorea opposite Thumb for the treatment of anorexia and chronic diarrhoea (Sautour *et al.*, 2004). Herein we report the title compound, (I), isolated from the same plant.

Compound (I) consists of a citric acid skeleton, upon which one of the carboxyl units is methylated (Fig. 1). In the crystal, the molecules are connected with each other by intermolecular O—H···O hydrogen bonds and thus construct a two-dimensional network (Fig. 2 and Table 1).

Experimental

Dried Dioscorea opposite Thumb (3 kg) was extracted three times with EtOH, 3 h every time. The solvent was removed under reduced pressure to give a crude extract (80 g) which was then further fractionated by liquid-liquid partition between water and light petroleum, EtOAc and n-BuOH, sequentially. The n-BuOH soluble fraction was separated and purified on silica gel column eluted with $CHCl_3$—MeOH (90:10, 85:15, 80:20, 75:25, 60:40, 50:50) to yield twelve fractions. After a week, colorless crystals of (I) was crystallized from the ninth fraction.

Refinement

All H atoms were positioned geometrically, with O—H = 0.82 Å, C—H = 0.96 and 0.97Å for methyl and methylene, respectively, and were refined as riding with $U_{iso}(H)=1.2U_{eq}(C_{methylene})$ and $U_{iso}(H)=1.5U_{eq}(O \text{ or } C_{methyl})$.

Figures

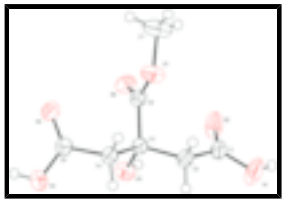

Fig. 1. The molecular structure of (I), with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level.

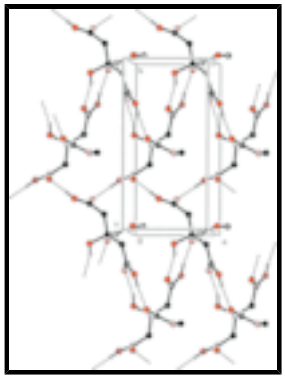

Fig. 2. Two-dimensional structure of (I), For clarity, H atoms not involved in hydrogen bonds are omitted.

methyl 3-carboxy-2-carboxymethyl-2-hydroxypropanoate

Crystal data

$C_7H_{10}O_7$	$F_{000} = 432$
$M_r = 206.15$	$D_x = 1.531$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 4374 reflections
$a = 5.8826$ (14) Å	$\theta = 3.2$–$28.3°$
$b = 11.927$ (3) Å	$\mu = 0.14$ mm^{-1}
$c = 12.752$ (3) Å	$T = 296$ (2) K
$V = 894.7$ (4) Å^3	Block, colourless
$Z = 4$	0.16 × 0.14 × 0.11 mm

Data collection

Bruker SMART APEX CCD diffractometer	1047 independent reflections
Radiation source: fine-focus sealed tube	1019 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.021$
$T = 296(2)$ K	$\theta_{max} = 26.0°$
ω scans	$\theta_{min} = 2.3°$
Absorption correction: Multi-scan (SADABS; Sheldrick, 2001)	$h = -7 \rightarrow 7$
$T_{min} = 0.978$, $T_{max} = 0.985$	$k = -14 \rightarrow 14$
4979 measured reflections	$l = -15 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.072$	$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.1459P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.14	$(\Delta/\sigma)_{max} < 0.001$
1047 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
129 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.4925 (3)	0.69161 (15)	0.39585 (14)	0.0343 (4)
C2	0.4378 (4)	0.57648 (14)	0.35749 (13)	0.0322 (4)
H2A	0.5755	0.5318	0.3576	0.039*
H2B	0.3842	0.5813	0.2857	0.039*
C3	0.2577 (3)	0.51732 (12)	0.42384 (12)	0.0248 (3)
C4	0.2234 (3)	0.40041 (13)	0.37674 (12)	0.0273 (4)
H4A	0.1832	0.4077	0.3033	0.033*
H4B	0.3651	0.3590	0.3809	0.033*
C5	0.0418 (3)	0.33632 (12)	0.43216 (13)	0.0276 (4)
C6	0.3374 (3)	0.50941 (12)	0.53815 (12)	0.0257 (3)
C7	0.6422 (4)	0.4600 (2)	0.64713 (16)	0.0538 (6)
H7A	0.7871	0.4232	0.6430	0.081*
H7B	0.5460	0.4203	0.6953	0.081*
H7C	0.6630	0.5356	0.6713	0.081*
O1	0.5924 (4)	0.75236 (12)	0.32416 (11)	0.0669 (6)
H1	0.6223	0.8143	0.3483	0.100*
O2	0.4556 (3)	0.72368 (11)	0.48306 (11)	0.0486 (4)
O3	0.0456 (2)	0.57004 (10)	0.41776 (10)	0.0326 (3)
H2	0.0493	0.6290	0.4508	0.049*
O4	−0.1301 (3)	0.30863 (13)	0.37240 (11)	0.0473 (4)
H4	−0.2223	0.2724	0.4067	0.071*
O5	0.0531 (2)	0.30970 (10)	0.52392 (9)	0.0346 (3)

O6	0.2305 (2)	0.54263 (10)	0.61229 (8)	0.0323 (3)
O7	0.5378 (2)	0.46140 (12)	0.54464 (9)	0.0378 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0390 (10)	0.0313 (9)	0.0326 (8)	−0.0110 (9)	−0.0006 (8)	0.0030 (7)
C2	0.0421 (10)	0.0301 (8)	0.0245 (7)	−0.0086 (8)	0.0040 (8)	−0.0009 (7)
C3	0.0285 (8)	0.0221 (7)	0.0237 (7)	−0.0004 (7)	0.0000 (7)	−0.0014 (6)
C4	0.0334 (9)	0.0226 (7)	0.0259 (7)	−0.0017 (7)	0.0023 (7)	−0.0026 (6)
C5	0.0310 (9)	0.0199 (6)	0.0319 (8)	0.0013 (7)	0.0014 (8)	−0.0019 (6)
C6	0.0287 (8)	0.0228 (7)	0.0256 (7)	−0.0008 (7)	0.0012 (7)	−0.0006 (6)
C7	0.0449 (12)	0.0809 (15)	0.0358 (10)	0.0199 (13)	−0.0125 (9)	−0.0083 (11)
O1	0.1153 (16)	0.0492 (8)	0.0362 (7)	−0.0478 (11)	0.0083 (10)	0.0014 (6)
O2	0.0634 (10)	0.0381 (7)	0.0442 (8)	−0.0201 (8)	0.0179 (8)	−0.0127 (6)
O3	0.0347 (7)	0.0272 (6)	0.0360 (6)	0.0057 (5)	−0.0054 (6)	−0.0053 (5)
O4	0.0419 (8)	0.0582 (9)	0.0418 (8)	−0.0217 (8)	−0.0052 (6)	0.0073 (6)
O5	0.0352 (7)	0.0349 (6)	0.0337 (6)	0.0011 (6)	0.0025 (6)	0.0075 (5)
O6	0.0400 (7)	0.0321 (6)	0.0247 (5)	0.0070 (6)	0.0048 (5)	−0.0010 (5)
O7	0.0319 (7)	0.0532 (8)	0.0282 (6)	0.0120 (7)	−0.0024 (5)	−0.0060 (6)

Geometric parameters (Å, °)

C1—O2	1.196 (2)	C5—O5	1.214 (2)
C1—O1	1.306 (2)	C5—O4	1.309 (2)
C1—C2	1.493 (2)	C6—O6	1.203 (2)
C2—C3	1.528 (2)	C6—O7	1.313 (2)
C2—H2A	0.9700	C7—O7	1.444 (2)
C2—H2B	0.9700	C7—H7A	0.9600
C3—O3	1.399 (2)	C7—H7B	0.9600
C3—C4	1.532 (2)	C7—H7C	0.9600
C3—C6	1.534 (2)	O1—H1	0.8200
C4—C5	1.492 (2)	O3—H2	0.8200
C4—H4A	0.9700	O4—H4	0.8200
C4—H4B	0.9700		
O2—C1—O1	123.71 (17)	C3—C4—H4B	109.2
O2—C1—C2	124.04 (16)	H4A—C4—H4B	107.9
O1—C1—C2	112.21 (16)	O5—C5—O4	122.53 (17)
C1—C2—C3	113.13 (15)	O5—C5—C4	123.44 (17)
C1—C2—H2A	109.0	O4—C5—C4	114.01 (14)
C3—C2—H2A	109.0	O6—C6—O7	124.30 (16)
C1—C2—H2B	109.0	O6—C6—C3	124.53 (16)
C3—C2—H2B	109.0	O7—C6—C3	111.18 (13)
H2A—C2—H2B	107.8	O7—C7—H7A	109.5
O3—C3—C2	112.32 (13)	O7—C7—H7B	109.5
O3—C3—C4	105.67 (13)	H7A—C7—H7B	109.5
C2—C3—C4	107.13 (13)	O7—C7—H7C	109.5
O3—C3—C6	110.67 (13)	H7A—C7—H7C	109.5

C2—C3—C6	110.02 (14)	H7B—C7—H7C	109.5
C4—C3—C6	110.91 (12)	C1—O1—H1	109.5
C5—C4—C3	112.03 (13)	C3—O3—H2	109.5
C5—C4—H4A	109.2	C5—O4—H4	109.5
C3—C4—H4A	109.2	C6—O7—C7	116.36 (14)
C5—C4—H4B	109.2		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O6^{i}	0.82	1.89	2.7011 (19)	171
O3—H2···O2^{ii}	0.82	2.03	2.8164 (18)	162
O4—H4···O5^{iii}	0.82	1.87	2.6854 (19)	176

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

Fig. 1

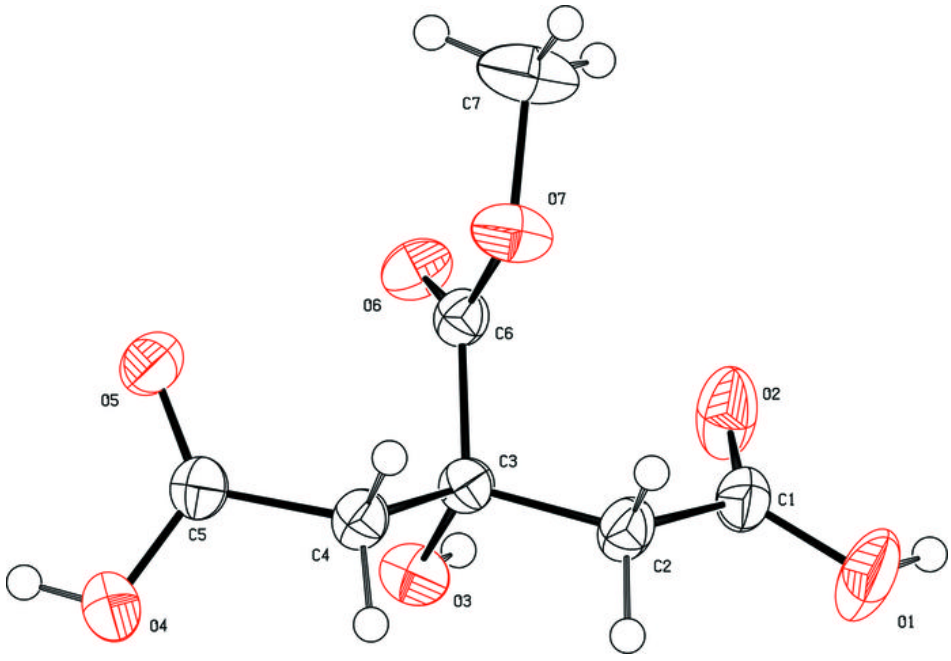

Fig. 2