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1,5-Dimethyl citrate monohydrate from *Dioscorea opposita* Thunb.

Mingjing Li,[a] Yong Wang,[a] Pengtao Ma,[b] Dexue Fu[c]* and Xiuhua Liu[a]

[a]Institute of Natural Products and Medicinal Chemistry, College of Chemistry and Chemical Engineering, Henan University, Kaifeng 475004, People's Republic of China, [b]Institute of Molecular and Crystal Engineering, College of Chemistry and Chemical Engineering, Henan University, Kaifeng 475004, People's Republic of China, and [c]College of Chemical Engineering, JiaoZuo University, Jiaozuo 454100, People's Republic of China
Correspondence e-mail: mptonline@163.com

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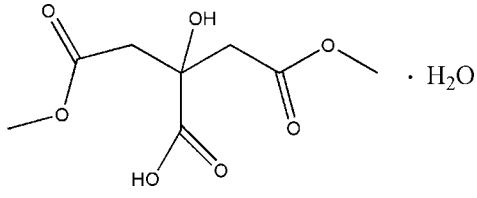
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 12.7.

The hydrated title compound, $C_8H_{12}O_7\cdot H_2O$, was isolated from the Chinese yam *Dioscorea opposita* Thunb. An intramolecular O—H···O hydrogen bond occurs in the organic molecule. In the crystal structure, the molecules are linked by intermolecular O—H···O hydrogen bonds, thereby forming chains propagating in [010].

Related literature

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



Experimental

Crystal data

$C_8H_{12}O_7\cdot H_2O$
M_r = 238.19
Triclinic, $P\overline{1}$
a = 7.8872 (5) Å
b = 8.1304 (5) Å
c = 9.5891 (6) Å
α = 94.569 (1)°
β = 110.122 (1)°
γ = 106.957 (1)°
V = 541.07 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 296 (2) K
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.977, T_{max} = 0.984
2932 measured reflections
1993 independent reflections
1729 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.089
S = 1.07
1993 reflections
157 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1W	0.82	1.74	2.5523 (15)	173
O5—H5···O4	0.82	2.17	2.6530 (13)	118
O5—H5···O2^{i}	0.82	2.15	2.8487 (14)	144
O1W—H1AW···O5^{ii}	0.82 (2)	1.98 (2)	2.7869 (16)	167 (2)
O1W—H1BW···O4^{iii}	0.85 (3)	1.99 (3)	2.8111 (17)	160 (2)

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

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: HB2628).

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supplementary materials

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1,5-Dimethyl citrate monohydrate from *Dioscorea opposita* Thunb.

M. Li, Y. Wang, P. Ma, D. Fu and X. Liu

Comment

The Chinese yam Dioscorea opposite Thunb is widely used in traditional medicine for the treatment of anorexia, chronic diarrhoea, diabetes, seminal emission and excessive leucorrhea. Up to now, many components have been successfully isolated and indentified from Dioscorea opposite Thumb. (Sautour *et al.*, 2004). Herein we report the title compound, (I), isolated from the same plant (Fig. 1).

Within the organic molecule, an intra-molecular O—H···O hydrogen bond occurs (Table 1), thus constructing an S(5) ring. In the crystal, the 1,5-dimethyl citrate molecules and water molecules through intermolecular O—H···O hydrogen bonds and finally construct a one-dimensional chain (Fig. 2).

Experimental

Dried Dioscorea opposite Thumb (3 kg) was pulverized and extracted with EtOH three times, 3 h each time. Concentration of the EtOH extract was performed and further fractionated by addition of light petroleum, EtOAc and n-BuOH, sequentially. The n-BuOH-soluble fraction was then separated and purified on silica gel column using $CHCl_3$–MeOH mixtures with increasing polarity: 90:10; 85:15; 80:20; 75:25; 60:40; 50:50 to yield twelve fractions. After one week, colourless blocks of (I) were obtained from the seventh fraction.

Refinement

The water H atoms were located in difference Fourier maps and freely refined. The other H atoms were positioned geometrically (O—H = 0.82 Å, C—H = 0.96–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O or methyl-C).

Figures

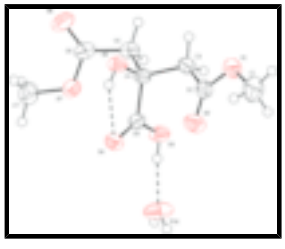

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. The hydrogen bonds are shown as dashed lines.

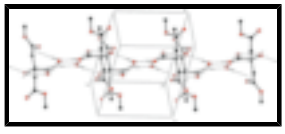

Fig. 2. One-dimensional structure of (I), Hydrogen bonds are shown as dashed lines. For clarity, H atoms not involved in hydrogen bonds are omitted.

1,5-Dimethyl citrate monohydrate

Crystal data

$C_8H_{12}O_7 \cdot H_2O$	$Z = 2$
$M_r = 238.19$	$F_{000} = 252$
Triclinic, $P\overline{1}$	$D_x = 1.462$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.8872$ (5) Å	Cell parameters from 1584 reflections
$b = 8.1304$ (5) Å	$\theta = 2.7$–28.2º
$c = 9.5891$ (6) Å	$\mu = 0.13$ mm^{-1}
$\alpha = 94.569$ (1)º	$T = 296$ (2) K
$\beta = 110.122$ (1)º	Block, colourless
$\gamma = 106.957$ (1)º	0.18 × 0.16 × 0.12 mm
$V = 541.07$ (6) Å^3	

Data collection

Bruker SMART CCD diffractometer	1993 independent reflections
Radiation source: fine-focus sealed tube	1729 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.011$
$T = 296$(2) K	$\theta_{max} = 25.5$º
ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -9 \rightarrow 9$
$T_{min} = 0.977$, $T_{max} = 0.984$	$k = -6 \rightarrow 9$
2932 measured reflections	$l = -11 \rightarrow 10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.1209P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1993 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
157 parameters	$\Delta\rho_{min} = -0.15$ 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}
O1	0.74007 (15)	1.12766 (14)	0.25191 (13)	0.0449 (3)
O2	0.66449 (16)	0.93114 (15)	0.39024 (12)	0.0463 (3)
O3	0.30961 (15)	0.57067 (13)	0.20264 (11)	0.0383 (3)
H3	0.3345	0.4988	0.2545	0.057*
O4	0.29657 (15)	0.70190 (13)	0.41154 (11)	0.0383 (3)
O5	0.24343 (15)	0.97566 (12)	0.29313 (11)	0.0355 (2)
H5	0.2747	0.9576	0.3798	0.053*
O6	−0.22748 (17)	0.79091 (17)	0.04817 (16)	0.0596 (4)
O7	−0.11049 (14)	0.59972 (14)	0.16700 (13)	0.0433 (3)
C1	0.9351 (2)	1.1961 (3)	0.3639 (2)	0.0605 (5)
H1A	0.9336	1.2305	0.4615	0.091*
H1B	1.0076	1.2961	0.3368	0.091*
H1C	0.9932	1.1071	0.3678	0.091*
C2	0.6206 (2)	0.99264 (18)	0.27822 (16)	0.0336 (3)
C3	0.4279 (2)	0.92440 (19)	0.15003 (15)	0.0346 (3)
H3A	0.4295	0.8363	0.0765	0.042*
H3B	0.4062	1.0200	0.1003	0.042*
C4	0.25973 (19)	0.84363 (16)	0.19763 (14)	0.0288 (3)
C5	0.0742 (2)	0.77648 (19)	0.05383 (15)	0.0347 (3)
H5A	0.0722	0.8670	−0.0062	0.042*
H5B	0.0745	0.6749	−0.0063	0.042*
C6	−0.1043 (2)	0.72700 (18)	0.08723 (16)	0.0365 (3)
C7	−0.2732 (3)	0.5434 (2)	0.2110 (2)	0.0578 (5)
H7A	−0.2883	0.6440	0.2578	0.087*
H7B	−0.2518	0.4669	0.2814	0.087*
H7C	−0.3872	0.4818	0.1228	0.087*
C8	0.29059 (18)	0.69683 (17)	0.28355 (15)	0.0293 (3)
O1W	0.4011 (2)	0.34048 (17)	0.34949 (16)	0.0595 (4)
H1AW	0.345 (3)	0.234 (3)	0.318 (2)	0.071 (7)*
H1BW	0.504 (4)	0.354 (3)	0.425 (3)	0.098 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0359 (6)	0.0441 (6)	0.0536 (7)	0.0080 (5)	0.0192 (5)	0.0163 (5)
O2	0.0452 (6)	0.0510 (7)	0.0368 (6)	0.0127 (5)	0.0103 (5)	0.0159 (5)
O3	0.0492 (6)	0.0325 (5)	0.0390 (6)	0.0223 (5)	0.0161 (5)	0.0105 (4)
O4	0.0468 (6)	0.0426 (6)	0.0342 (5)	0.0211 (5)	0.0190 (5)	0.0162 (4)
O5	0.0485 (6)	0.0310 (5)	0.0321 (5)	0.0194 (4)	0.0164 (5)	0.0081 (4)
O6	0.0473 (7)	0.0609 (8)	0.0844 (9)	0.0345 (6)	0.0243 (6)	0.0323 (7)
O7	0.0359 (6)	0.0428 (6)	0.0592 (7)	0.0182 (5)	0.0214 (5)	0.0211 (5)
C1	0.0342 (9)	0.0655 (12)	0.0714 (12)	0.0059 (8)	0.0181 (9)	0.0107 (10)
C2	0.0373 (8)	0.0324 (7)	0.0371 (8)	0.0137 (6)	0.0197 (6)	0.0079 (6)
C3	0.0387 (8)	0.0358 (7)	0.0318 (7)	0.0126 (6)	0.0151 (6)	0.0128 (6)
C4	0.0337 (7)	0.0261 (6)	0.0286 (7)	0.0123 (5)	0.0122 (6)	0.0071 (5)
C5	0.0384 (8)	0.0337 (7)	0.0318 (7)	0.0158 (6)	0.0097 (6)	0.0098 (6)
C6	0.0356 (8)	0.0314 (7)	0.0374 (8)	0.0141 (6)	0.0062 (6)	0.0049 (6)
C7	0.0453 (10)	0.0548 (10)	0.0816 (13)	0.0163 (8)	0.0328 (9)	0.0235 (10)
C8	0.0268 (6)	0.0285 (7)	0.0324 (7)	0.0103 (5)	0.0101 (5)	0.0079 (5)
O1W	0.0611 (8)	0.0325 (7)	0.0639 (9)	0.0151 (6)	−0.0009 (7)	0.0144 (6)

Geometric parameters (Å, °)

O1—C2	1.3269 (17)	C2—C3	1.497 (2)
O1—C1	1.448 (2)	C3—C4	1.5349 (18)
O2—C2	1.2060 (17)	C3—H3A	0.9700
O3—C8	1.3113 (16)	C3—H3B	0.9700
O3—H3	0.8200	C4—C8	1.5332 (18)
O4—C8	1.2088 (16)	C4—C5	1.5385 (19)
O5—C4	1.4186 (15)	C5—C6	1.499 (2)
O5—H5	0.8200	C5—H5A	0.9700
O6—C6	1.1956 (18)	C5—H5B	0.9700
O7—C6	1.3345 (18)	C7—H7A	0.9600
O7—C7	1.4469 (19)	C7—H7B	0.9600
C1—H1A	0.9600	C7—H7C	0.9600
C1—H1B	0.9600	O1W—H1AW	0.82 (2)
C1—H1C	0.9600	O1W—H1BW	0.85 (3)
C2—O1—C1	116.12 (13)	O5—C4—C5	107.74 (11)
C8—O3—H3	109.5	C8—C4—C5	111.81 (11)
C4—O5—H5	109.5	C3—C4—C5	107.94 (11)
C6—O7—C7	116.87 (12)	C6—C5—C4	113.12 (11)
O1—C1—H1A	109.5	C6—C5—H5A	109.0
O1—C1—H1B	109.5	C4—C5—H5A	109.0
H1A—C1—H1B	109.5	C6—C5—H5B	109.0
O1—C1—H1C	109.5	C4—C5—H5B	109.0
H1A—C1—H1C	109.5	H5A—C5—H5B	107.8
H1B—C1—H1C	109.5	O6—C6—O7	123.30 (14)
O2—C2—O1	123.76 (14)	O6—C6—C5	125.41 (14)

O2—C2—C3	124.70 (13)	O7—C6—C5	111.29 (12)
O1—C2—C3	111.52 (12)	O7—C7—H7A	109.5
C2—C3—C4	114.33 (11)	O7—C7—H7B	109.5
C2—C3—H3A	108.7	H7A—C7—H7B	109.5
C4—C3—H3A	108.7	O7—C7—H7C	109.5
C2—C3—H3B	108.7	H7A—C7—H7C	109.5
C4—C3—H3B	108.7	H7B—C7—H7C	109.5
H3A—C3—H3B	107.6	O4—C8—O3	125.61 (12)
O5—C4—C8	108.85 (10)	O4—C8—C4	122.10 (12)
O5—C4—C3	109.34 (10)	O3—C8—C4	112.29 (11)
C8—C4—C3	111.07 (11)	H1AW—O1W—H1BW	107 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1W	0.82	1.74	2.5523 (15)	173
O5—H5···O4	0.82	2.17	2.6530 (13)	118
O5—H5···O2^{i}	0.82	2.15	2.8487 (14)	144
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O1W—H1BW···O4^{iii}	0.85 (3)	1.99 (3)	2.8111 (17)	160 (2)

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

Fig. 1

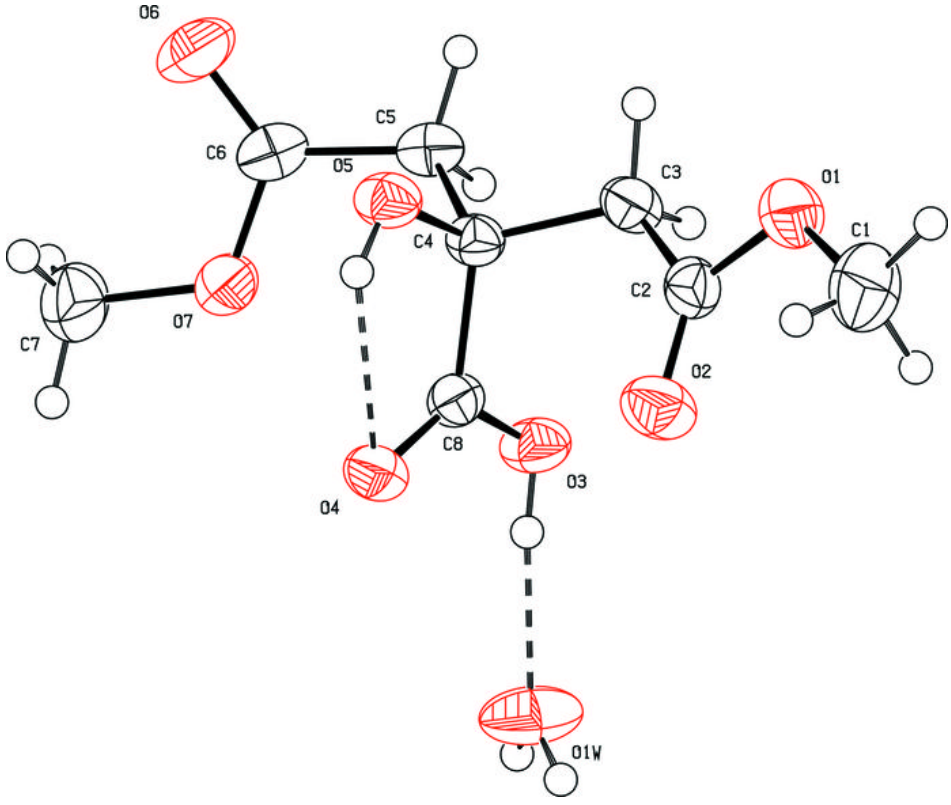

Fig. 2