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(2*α*,3*α*,5*α*,20*S*,22*S*)-2,3,5,14,20,20,24-Heptahydroxycholest-7-en-6-one monohydrate

Yuan-Jiang Pan,[a]* Hui Zhou,[a] Hao Shi,[a] Shu-Ming Lv[a] and Zhi-Ming Jin[b]

[a]Department of Chemistry, Zhejiang University, Hangzhou 310027, People's Republic of China, and [b]College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: cheyjpan@public.zju.edu.cn

Key indicators

Single-crystal X-ray study
T = 291 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.041
wR factor = 0.064
Data-to-parameter ratio = 9.2

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

The crystal structure of the title compound consists of discrete $C_{27}H_{44}O_8$ and H_2O molecules, linked by hydrogen bonding.

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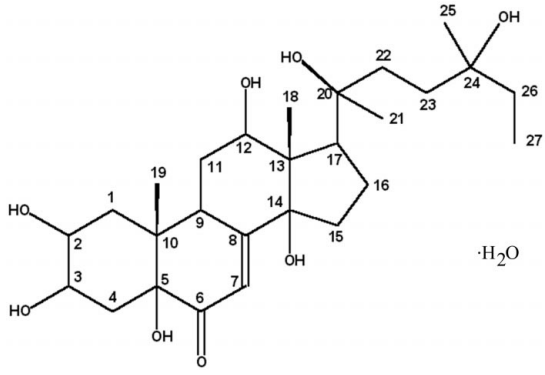
Comment

The title compound, (I), has been isolated from *Solanum nigrum* L. (De Souza *et al.*, 1970; Baltaev, 1995; Kissmer & Wichtl, 1987; Nishimoto *et al.*, 1987). It was isolated from Solanaceae for the first time, its structure was established from the spectroscopic and chemical evidence, and confirmed by this X-ray diffraction study.



(I)

The title compound (Fig. 1) is a typical example of a sterone. Its main skeleton is composed of four rings. The *A* and *C* rings exist in chair conformations. Because of carbonyl and double-bond planarity, the *B* ring is in a distorted chair conformation. Ring *D* has a half-chair conformation. The Csp^3-Csp^3 bond lengths C1—C2, C2—C3, C5—C10, C9—C10, C9—C11, C13—C17, C20—C22, C22—C23, C24—C25, C25—C26 and C25—C27 show some significant deviations from standard values. The bond angles C7—C6—C5, C8—C7—C6, C7—C8—C9, C7—C8—C14, C14—C13—C17, C15—C14—C8 and C20—C17—C13 show significant deviations from the ideal tetrahedral value of 109.5°. These deviations are common in steroids as a result of the strain induced by ring junctions, side chains and unsaturated bonds. The C7—C8 bond length indicates double-bond nature. The title compound is a hydrate; the water molecule is involved in hydrogen bonding, as both donor and acceptor (Table 2).

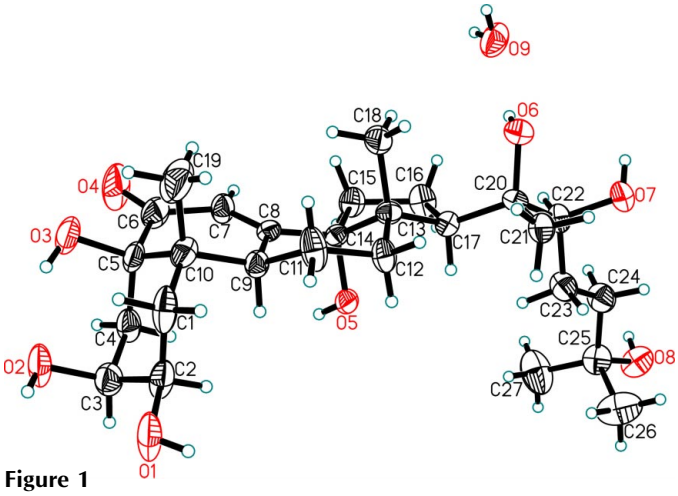


Figure 1
The asymmetric unit of the title compound with atom labels, showing 50% probability displacement ellipsoids.

Experimental

The title compound was separated from a *n*-butanol fraction of *Solanum nigrum* L. by $CHCl_3$–CH_3OH–H_2O. Crystals were grown from methanol at room temperature by slow evaporation of the solvent.

Crystal data

$C_{27}H_{44}O_8{\cdot}H_2O$
M_r = 514.64
Orthorhombic, $P2_12_12_1$
a = 7.153 (1) Å
b = 10.290 (2) Å
c = 37.220 (8) Å
V = 2739.6 (9) Å^3
Z = 4
D_x = 1.248 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 2.9–15.0°
μ = 0.09 mm^{-1}
T = 291 (2) K
Prism, colourless
0.36 × 0.30 × 0.24 mm

Data collection

Siemens $P4$ diffractometer
ω scans
3426 measured reflections
3185 independent reflections
1635 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.3°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = -1 \rightarrow 46$
3 standard reflections
every 97 reflections
intensity decay: 3.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.064
S = 0.80
3185 reflections
346 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0182P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0046 (3)

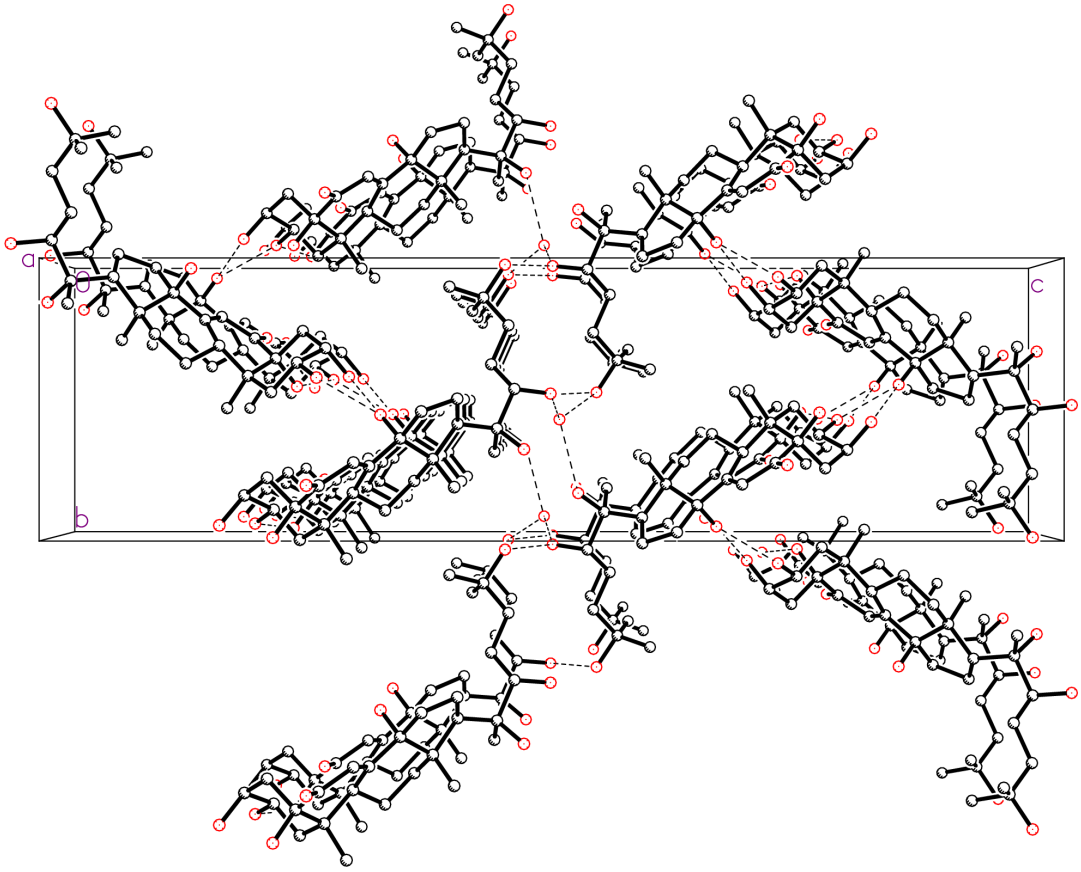


Figure 2
An illustration of the unit-cell packing of the title compound, viewed along the crystallographic *a* axis. H atoms are omitted for clarity. Hydrogen bonding is shown by dashed lines.

Table 1
Selected geometric parameters (Å, °).

O4—C6	1.217 (4)	C8—C9	1.498 (4)
C1—C2	1.511 (4)	C9—C11	1.551 (4)
C1—C10	1.543 (4)	C9—C10	1.558 (4)
C2—C3	1.507 (4)	C13—C17	1.554 (4)
C3—C4	1.526 (4)	C20—C22	1.558 (4)
C4—C5	1.525 (4)	C22—C23	1.520 (4)
C5—C6	1.518 (4)	C24—C25	1.518 (4)
C5—C10	1.549 (4)	C25—C26	1.507 (4)
C6—C7	1.459 (4)	C25—C27	1.508 (4)
C7—C8	1.335 (4)		
O1—C2—C3	110.7 (3)	C7—C8—C14	121.4 (3)
C2—C3—C4	107.2 (3)	C8—C9—C10	114.0 (3)
C5—C4—C3	113.0 (3)	C1—C10—C5	107.9 (3)
C6—C5—C10	110.2 (3)	C5—C10—C9	110.8 (3)
C4—C5—C10	112.3 (3)	C14—C13—C17	99.2 (2)
C7—C6—C5	118.2 (3)	C15—C14—C8	119.5 (3)
C8—C7—C6	121.7 (3)	C20—C17—C13	121.4 (3)
C7—C8—C9	123.2 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.82	2.07	2.850 (3)	160
O2—H2O···O5^{ii}	0.82	1.99	2.807 (3)	172
O3—H3O···O2	0.82	1.95	2.669 (3)	146
O5—H5···O1^{iii}	0.82	1.83	2.620 (3)	162
O6—H6···O9	0.82	2.37	3.146 (4)	158
O7—H7···O8^{iv}	0.82	2.02	2.817 (3)	165
O8—H8···O9^{v}	0.82	2.01	2.819 (4)	168
O9—H9O*B*···O6^{vi}	0.822 (10)	2.070 (11)	2.892 (4)	180 (6)
O9—H9O*A*···O7^{vii}	0.822 (10)	2.053 (14)	2.862 (4)	168 (4)

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

Atoms H90*A* and H90*B* were located by difference Fourier synthesis, the remaining H atoms were placed in geometrically calculated positions. All H atoms were included in the final refinement and allowed to ride on their parent atoms with U_{iso}(H) = $1.2U_{eq}$(parent). Friedel reflections were merged before the final refinement and only the relative stereochemistry is shown in the Scheme and Figures; the absolute configuration can not be determined reliably in this experiment.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL*/*PC* (Siemens, 1991); software used to prepare material for publication: *SHELXL*97.

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