Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Roburic Acid, a Triterpene 3,4-Seco Acid

TING-TING JONG* AND CHI-TAIN CHEN

Department of Chemistry, National Chung-Hsing University, Taichung, Taiwan

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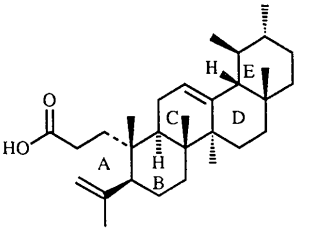
Abstract

The structure and stereochemistry of roburic acid [3,4-secoursa-4(23),12-dien-3-oic acid, $C_{30}H_{48}O_2$], isolated as the main component from the Chinese drug 'Ch'in-Chiao' (*Gentiana macrophylla pall.*) has been established.

Comment

The title compound, (1), has been isolated and its chemical structure verified by partial synthesis from α-amyrin *via* peracid oxidation (Mangoni & Belardini, 1963). The compound was obtained by silica-gel chromatography of the chloroform extract of *G. macrophylla*. The mass spectrum showed the base peak at 218, indicating a 12-ursane or oleanane-type triterpenoid. The IR spectrum showed no evidence of a 3-hydroxyl group, so a 3,4-seco acid was assumed. This X-ray analysis establishes the molecular structure and conformation of the compound.



(1)

The triterpenoid ring *A* has been opened at the C(3)—C(4) bond to give a carboxylic acid group at C(3) and an olefin at C(4). Rings *B*, *D* and *E* have chair forms, the *B*/*C* rings are *trans* fused while the

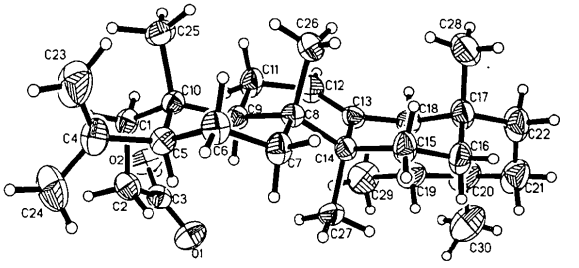


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of $C_{30}H_{48}O_2$.

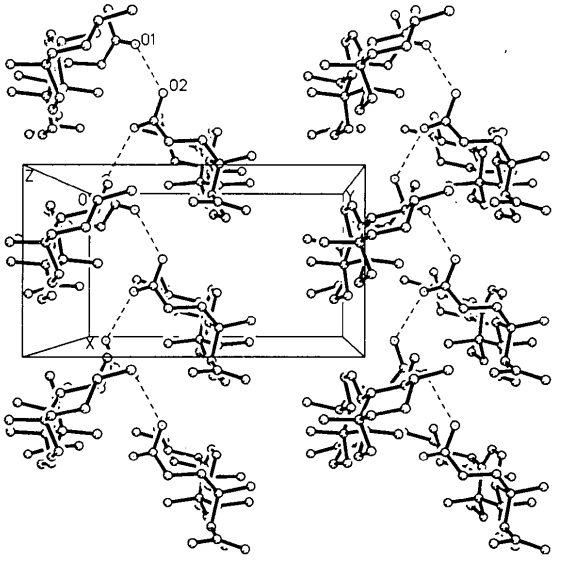


Fig. 2. A view of the unit cell along the *c* axis with O(1)—H···O(2)-type hydrogen bonding.

D/*E* rings are *cis* fused; the *C* ring adopts a half-chair conformation. Hydrogen bonds [O(1)—H···O(2)(at $-\frac{1}{2}+x, -\frac{1}{2}-y, -z$) 2.735 (3) Å] link the molecules in chains around twofold screw axes parallel to *a*. The molecular conformation and numbering scheme are shown in Fig. 1 and a view of the molecular packing is shown in Fig. 2.

Experimental

The title compound was isolated as the main component of the Chinese drug 'Ch'in-Chiao' from a chloroform/hexane mixture (m.p. 464–466 K).

Crystal data

$C_{30}H_{48}O_2$	Mo $K\alpha$ radiation
M_r = 440.7	λ = 0.71069 Å
Orthorhombic	Cell parameters from 38 reflections
$P2_12_12_1$	θ = 1.5–25°
a = 7.554 (2) Å	μ = 0.068 mm^{-1}
b = 13.523 (2) Å	T = 293 K
c = 25.524 (4) Å	Prism
V = 2607.2 (9) Å^3	0.6 × 0.4 × 0.4 mm
Z = 4	Colourless
D_x = 1.123 Mg m^{-3}	

Data collection

Siemens *R3m/V* diffractometer	θ_{max} = 25°
2θ scans	h = 0 → 9
Absorption correction: none	k = 0 → 17
3427 measured reflections	l = 0 → 33
3392 independent reflections	2 standard reflections monitored every 100 reflections
2747 observed reflections [$F > 4.0\sigma(F)$]	intensity variation: not significant
R_{int} = 0.049	

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.001F^2]$
R = 0.0469	$(\Delta/\sigma)_{max}$ = 0.001
wR = 0.0596	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 1.40	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
2747 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
296 parameters	
H-atom parameters not refined	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O(1)	0.3271 (3)	−0.2516 (2)	0.0041 (1)	0.048 (1)
O(2)	0.5245 (3)	−0.1492 (2)	−0.0306 (1)	0.048 (1)
C(1)	0.2373 (3)	−0.0130 (2)	−0.0547 (1)	0.032 (1)
C(2)	0.2283 (3)	−0.1267 (2)	−0.0560 (1)	0.037 (1)
C(3)	0.3618 (3)	−0.1817 (2)	−0.0236 (1)	0.034 (1)
C(4)	−0.1371 (4)	0.0422 (2)	−0.0909 (1)	0.043 (1)
C(5)	−0.0882 (3)	0.0189 (2)	−0.0341 (1)	0.031 (1)
C(6)	−0.2194 (3)	0.0638 (2)	0.0045 (1)	0.037 (1)
C(7)	−0.1954 (3)	0.0184 (2)	0.0584 (1)	0.038 (1)
C(8)	−0.0090 (3)	0.0336 (2)	0.0812 (1)	0.029 (1)
C(9)	0.1347 (3)	0.0058 (2)	0.0399 (1)	0.028 (1)
C(10)	0.1073 (3)	0.0428 (2)	−0.0181 (1)	0.028 (1)
C(11)	0.3176 (3)	0.0302 (2)	0.0619 (1)	0.040 (1)
C(12)	0.3367 (3)	0.0094 (2)	0.1195 (1)	0.036 (1)
C(13)	0.2098 (3)	−0.0206 (2)	0.1516 (1)	0.031 (1)
C(14)	0.0209 (3)	−0.0355 (2)	0.1312 (1)	0.030 (1)
C(15)	−0.1175 (3)	−0.0094 (2)	0.1737 (1)	0.041 (1)
C(16)	−0.0684 (4)	−0.0445 (2)	0.2288 (1)	0.046 (1)
C(17)	0.1120 (4)	−0.0054 (2)	0.2466 (1)	0.044 (1)
C(18)	0.2557 (3)	−0.0454 (2)	0.2084 (1)	0.036 (1)
C(19)	0.3010 (4)	−0.1569 (2)	0.2174 (1)	0.045 (1)
C(20)	0.3455 (5)	−0.1757 (3)	0.2756 (1)	0.056 (1)
C(21)	0.1914 (5)	−0.1451 (3)	0.3102 (1)	0.063 (1)
C(22)	0.1514 (5)	−0.0373 (3)	0.3035 (1)	0.056 (1)
C(23)	−0.1883 (6)	0.1377 (3)	−0.1062 (1)	0.073 (1)
C(24)	−0.1428 (6)	−0.0375 (3)	−0.1271 (1)	0.068 (1)
C(25)	0.1508 (4)	0.1534 (2)	−0.0267 (1)	0.042 (1)
C(26)	0.0082 (4)	0.1441 (2)	0.0972 (1)	0.041 (1)
C(27)	−0.0027 (4)	−0.1466 (2)	0.1179 (1)	0.040 (1)
C(28)	0.1132 (5)	0.1084 (2)	0.2462 (1)	0.058 (1)
C(29)	0.4505 (5)	−0.1906 (3)	0.1816 (1)	0.062 (1)
C(30)	0.3975 (6)	−0.2833 (3)	0.2866 (2)	0.079 (1)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(3)	1.210 (3)	C(21)—C(22)	1.498 (5)
C(1)—C(2)	1.539 (3)	O(2)—C(3)	1.318 (3)
C(2)—C(3)	1.500 (4)	C(1)—C(10)	1.551 (3)
C(4)—C(23)	1.404 (5)	C(4)—C(5)	1.528 (3)
C(5)—C(6)	1.525 (3)	C(4)—C(24)	1.419 (5)
C(6)—C(7)	1.517 (4)	C(5)—C(10)	1.566 (3)
C(8)—C(9)	1.559 (3)	C(7)—C(8)	1.537 (3)
C(9)—C(11)	1.528 (3)	C(8)—C(14)	1.599 (3)
C(11)—C(12)	1.505 (4)	C(9)—C(10)	1.576 (3)
C(13)—C(14)	1.533 (3)	C(12)—C(13)	1.325 (3)
C(14)—C(15)	1.547 (3)	C(13)—C(18)	1.528 (4)
C(15)—C(16)	1.530 (4)	C(16)—C(17)	1.531 (4)
C(17)—C(28)	1.538 (4)	C(17)—C(22)	1.544 (4)
C(19)—C(20)	1.546 (4)	C(18)—C(19)	1.562 (5)
C(20)—C(21)	1.518 (5)		
C(2)—C(1)—C(10)	118.1 (2)	O(1)—C(3)—C(2)	124.3 (2)
O(1)—C(3)—O(2)	122.8 (2)	C(5)—C(4)—C(23)	121.3 (3)
O(2)—C(3)—C(2)	112.8 (2)	C(23)—C(4)—C(24)	120.5 (3)
C(5)—C(4)—C(24)	117.9 (3)	C(4)—C(5)—C(10)	115.7 (2)
C(4)—C(5)—C(6)	112.0 (2)	C(5)—C(6)—C(7)	110.3 (2)
C(6)—C(5)—C(10)	111.2 (2)	C(7)—C(8)—C(9)	110.4 (2)
C(6)—C(7)—C(8)	113.5 (2)	C(9)—C(8)—C(14)	107.5 (2)
C(7)—C(8)—C(14)	110.7 (2)	C(8)—C(9)—C(10)	117.9 (2)
C(8)—C(9)—C(11)	109.2 (2)	C(10)—C(9)—C(11)	113.3 (2)
C(1)—C(10)—C(5)	109.8 (2)	C(9)—C(10)—C(25)	114.3 (2)
C(5)—C(10)—C(9)	107.7 (2)	C(11)—C(12)—C(13)	126.4 (2)
C(9)—C(11)—C(12)	113.9 (2)	C(12)—C(13)—C(18)	119.4 (2)
C(12)—C(13)—C(14)	120.3 (2)	C(8)—C(14)—C(13)	109.0 (2)
C(14)—C(13)—C(18)	120.3 (2)	C(13)—C(14)—C(15)	111.2 (2)
C(8)—C(14)—C(15)	109.3 (2)	C(14)—C(15)—C(16)	114.1 (2)
C(15)—C(16)—C(17)	112.4 (2)	C(16)—C(17)—C(18)	108.3 (2)
C(16)—C(17)—C(22)	110.8 (2)	C(18)—C(17)—C(22)	110.9 (2)
C(13)—C(18)—C(19)	113.6 (2)	C(13)—C(18)—C(17)	111.1 (2)
C(18)—C(19)—C(20)	110.3 (2)	C(17)—C(18)—C(19)	113.4 (2)
C(20)—C(21)—C(22)	110.8 (3)	C(19)—C(20)—C(21)	110.3 (3)
C(1)—C(2)—C(3)	117.0 (2)	C(17)—C(22)—C(21)	114.7 (3)

The space group, $P2_12_12_1$, was determined from the systematic absences: $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$. All computation was performed using *SHELXTL-Plus* (Sheldrick, 1991). Molecular graphics were obtained with *ORTEP*II (Johnson, 1976).

Support of this study by the National Sciences Council is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1063). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6-Amino-5-hydroxyiminomethyl-1,3-dimethyluracil

JOHN N. LOW

Department of Applied Physics and Electronic & Manufacturing Engineering, University of Dundee, Dundee DD1 4HN, Scotland

GEORGE FERGUSON

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

MIGUEL N. MORENO-CARRETERO AND FRANCISCO HUESO-UREÑA

Departamento de Quimica Inorganica, Facultad de Ciencias Experimentales, Universidad de Jaèn, 23071-Jaèn, Spain

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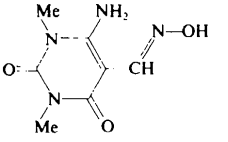
Abstract

The structure of the title compound [6-amino-5-hydroxyiminomethyl-1,3-dimethyl-2,4(1*H*,3*H*)-pyrimidinedione, $C_7H_{10}N_4O_3$] shows that there is extensive electronic delocalization in the uracil ring, as found in analogous uracil derivatives.

Comment

The bonds and angles of the title compound, (I), are very similar to those found for 6-amino-1,3-dimethyluracil (Ferguson, Gallagher, Low, Howie, Hueso-Ureña & Moreno Carretero, 1993). The bonds are longer than those found in 5-formyl and 5-nitroso derivatives (Low, Howie, Hueso-Ureña & Moreno-Carretero, 1992). Only the exocyclic C=O bonds show true double-bond character, and the C6—N6 distance is intermediate between that of a single and a double bond. The C4—C5 and C5—C6 distances in the present compound do show some difference due to the different substituents. The structure is fully hydrogen bonded.



(I)

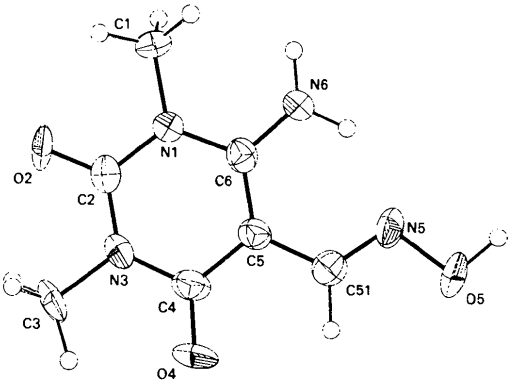


Fig. 1. Perspective view of the molecule. Displacement ellipsoids are shown at the 50% probability level.

Experimental

Crystal data

$C_7H_{10}N_4O_3$
M_r = 198.18
Tetragonal
$I4_1/a$
a = 23.0740 (19) Å
c = 6.9535 (9) Å
V = 3702.1 (6) Å^3
Z = 16
D_x = 1.422 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12.00–31.00°
μ = 0.11 mm^{-1}
T = 293 K
Prism
0.440 × 0.243 × 0.243 mm
Yellow

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4107 measured reflections
2010 independent reflections
1059 observed reflections [$I > 3.0\sigma(I)$]
R_{int} = 0.009
θ_{max} = 26.87°
$h = 0 \rightarrow 29$
$k = 0 \rightarrow 29$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 120 min
intensity variation: 2.5%

Refinement

Refinement on F
R = 0.042
wR = 0.056
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}