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(3a*RS*,7a*RS*)-3a-Hydroxy-3,3a,7,7a-tetrahydrobenzofuran-2,6-dione

Xiaoyan Tian, Yunshan Wu, Ningbo Gong, Yang Lu and Weishuo Fang*

Institute of Materia Medica, Chinese Academy of Medical Sciences & Peking Union Medicinal College, Beijing 100050, People's Republic of China

Correspondence e-mail: wfang@imm.ac.cn

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Key indicators

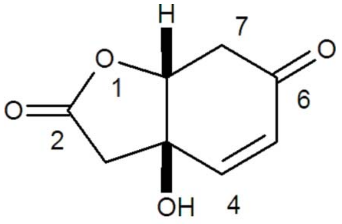
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.055
wR factor = 0.147
Data-to-parameter ratio = 13.9

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

Single crystals of senecio lactone, $C_8H_8O_4$, isolated from *Senecio scandens* Buch–Ham, were obtained from an acetone solution. In the crystal structure, molecules are linked to each other by intermolecular O—H···O hydrogen bonds involving a ketone group and the hydroxyl group.

Comment

Senecio lactone, (I), as an important reaction intermediate, was synthesized in order to explain whether atmospheric oxygen is incorporated during the process in which the enzyme *p*-hydroxyphenylpyruvate hydroxylase catalyses the conversion of *p*-hydroxyphenylpyruvic acid into homogentisic acid (Saito, Chujo *et al.*, 1975; Saito, Yamane *et al.*, 1975). As a patented anti-ulcer agent, it has been obtained by acidic hydrolysis of a natural mixture of quinol esters (Massanet *et al.*, 1993). Senecio lactone was first reported as a new natural product in 1993. In order to determine its relative configuration, we now report an unambiguous crystal structure for (I).



(I)

The structure of senecio lactone, (I), is based on *cis*-fused rings (Fig. 1). Bond lengths and angles (Table 1) are consistent with normal values. Both five-membered lactone (*A*) and six-membered cyclohexenone (*B*) rings have an envelope conformation. The C1—O1 and C5—C6 single bonds are slightly shortened because of their conjugation with neighbouring carbonyl groups. The presence of an intermolecular O—H···O hydrogen bond involving the hydroxyl group and the ketone goup C6═O4 (Table 2) contributes to the stability of the crystal structure (Fig. 2).

Experimental

The dried aerial parts (8.0 kg) of *S. scandens* were powdered and extracted with boiling ethanol three times (40 l). The combined alcohol extracts were concentrated *in vacuo* to yield a dark-brown residue (1.2 kg), which was chromatographed on silica gel (60–100 mesh, 4 kg), to provide fractions I (53 g), II (78 g), III (125 g), IV (353 g) and V (200 g). Fraction II was chromatographed on silica gel (2 kg), eluting with petroleum ether-acetone (75:25), to afford senecio lactone (500 mg) as a white solid, m.p. 382–384 K. $[\alpha]_D^{26} = 0$ (*c*

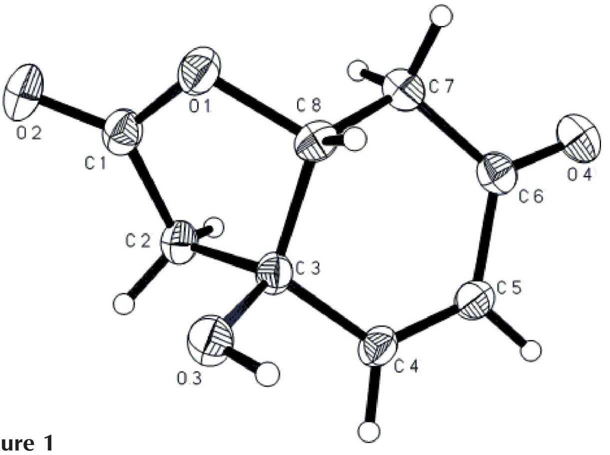


Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

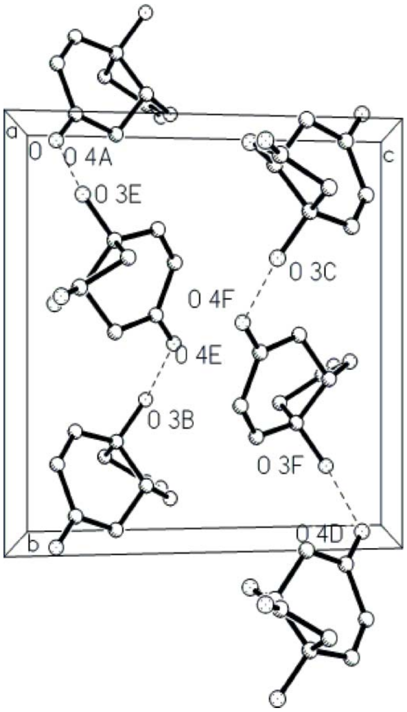


Figure 2
The crystal packing of (I), viewed down the [100] axis. Hydrogen bonds are shown as dashed lines. H atoms have been omitted.

6.33, acetone); EIMS (m/z): 168 [M+]. ^{1}H and ^{13}C NMR data are in agreement with the literature (Saito, Chujo *et al.*, 1975; Saito, Yamane *et al.*, 1975). Single crystals were obtained by slow evaporation of an acetone solution.

Crystal data

$C_8H_8O_4$
$M_r = 168.15$
Monoclinic, $P2_1/n$
$a = 6.7870$ (14) Å
$b = 10.711$ (2) Å
$c = 10.311$ (2) Å
$\beta = 102.06$ (3)°
$V = 733.0$ (3) Å^3
$Z = 4$

$D_x = 1.524$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2341 reflections
$\theta = 2.8$–25.0°
$\mu = 0.12$ mm^{-1}
$T = 295$ (2) K
Plate, colourless
0.50 × 0.40 × 0.10 mm

Data collection

MAC DIP-2030K diffractometer
ω scans
2341 measured reflections
1534 independent reflections
1533 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.032$
$\theta_{\rm max} = 25.0°$
$h = 0 \rightarrow 8$
$k = -12 \rightarrow 13$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.147$
$S = 1.27$
1534 reflections
110 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.3161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.122 (15)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.354 (3)	O4—C6	1.225 (2)
O1—C8	1.454 (2)	C4—C5	1.330 (3)
O2—C1	1.203 (2)	C5—C6	1.465 (3)
O3—C3	1.428 (2)		
C1—O1—C8	109.83 (15)	C4—C5—C6	122.18 (18)
C5—C4—C3	123.65 (19)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O4$^{\rm i}$	0.82	1.96	2.756 (2)	165

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

All H atoms were placed in calculated positions and refined as riding on their carrier atoms. Constrained distances and isotropic U parameters: methine CH: 0.98 Å and $U_{\rm iso}$= 1.2$U_{\rm eq}$(C); methylene CH$_2$: 0.97 Å and $U_{\rm iso}$= 1.2$U_{\rm eq}$(C); C(sp^2)H: 0.93 Å and $U_{\rm iso}$= 1.2$U_{\rm eq}$(C); OH: 0.82 Å and $U_{\rm iso}$= 1.5$U_{\rm eq}$(O).

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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