Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-Hydroxy-(*S*)-3-methyl-1-oxoisochromane-5-carboxylic acid (5-carboxymellein)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.056
wR factor = 0.127
Data-to-parameter ratio = 5.9

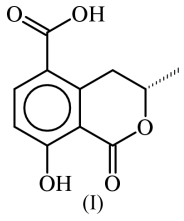
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecules of the title compound, $C_{11}H_{10}O_5$, are linked by a hydrogen bond involving the acid H and the carbonyl O atom of the dihydroisocoumarin unit into a linear chain running along the *b* axis of the monoclinic unit cell.

Received 22 July 2003
Accepted 23 July 2003
Online 31 July 2003

Comment

8-Hydroxy-3-methyl-1-oxoisochromane-5-carboxylic acid (5-carboxymellein), (I) (Fig. 1), a dihydroisocoumarin isolated from *Tubercularia sp*., an endophytic fungus of *Taxus mairei* that is found in Fujian Province, China, yields compounds that are cytotoxic to KB and HL60 cancer cell lines (Wang *et al.*, 2000). The structure has been assigned on the basis of two-dimensional NMR studies (Chinworrungsee *et al.*, 2001); the crystal structure shows that adjacent molecules are linked by a short hydrogen bond involving the carboxylic acid group and the double-bond O atom of the dihydroisocoumarin ring of an adjacent molecule [O···O = 2.702 (5) Å] to furnish a linear chain running along the *b* axis of the unit cell. The structure is similar to that of 5-methylmellein, which shows only weak bioactivity (Krohn *et al.*, 1997).



(I)

Experimental

The title compound was isolated from an endophytic fungus, *Tubercularia sp*., which was found in the inner bark of *Taxus mairei* of Fujian Province, China. Crystals were grown from a solution in ethyl acetate.

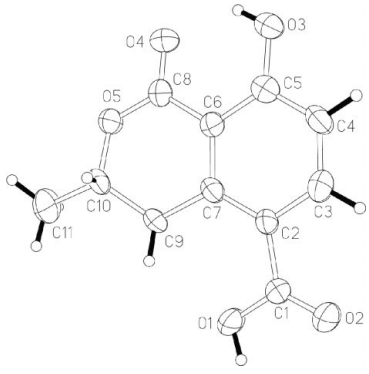


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Crystal data

$C_{11}H_{10}O_5$	D_x = 1.530 Mg m^{-3}
M_r = 222.19	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 634 reflections
a = 7.3351 (4) Å	θ = 2.8–21.7°
b = 9.0510 (5) Å	μ = 0.12 mm^{-1}
c = 7.4211 (5) Å	T = 298 (2) K
β = 101.723 (3)°	Plate, colorless
V = 482.41 (5) Å^3	0.15 × 0.12 × 0.06 mm
Z = 2	

Data collection

Bruker APEX area-detector diffractometer	726 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.040
Absorption correction: none	θ_{max} = 25.0°
2438 measured reflections	$h = -7 \rightarrow 8$
897 independent reflections	$k = -10 \rightarrow 10$
	$l = -8 \rightarrow 6$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.056	$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.127	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.05	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
897 reflections	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
151 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.323 (6)	C3—C4	1.366 (8)
O2—C1	1.183 (6)	C4—C5	1.375 (8)
O3—C5	1.330 (7)	C5—C6	1.398 (7)
O4—C8	1.217 (7)	C6—C7	1.409 (8)
O5—C8	1.298 (6)	C6—C8	1.467 (8)
O5—C10	1.467 (6)	C7—C9	1.508 (7)
C1—C2	1.479 (8)	C9—C10	1.502 (7)
C2—C7	1.390 (7)	C10—C11	1.493 (7)
C2—C3	1.402 (7)		
C8—O5—C10	117.9 (4)	C5—C6—C8	119.3 (5)
O2—C1—O1	122.8 (5)	C7—C6—C8	119.7 (5)
O2—C1—C2	122.2 (5)	C2—C7—C6	118.8 (5)
O1—C1—C2	115.0 (5)	C2—C7—C9	125.0 (5)
C7—C2—C3	118.9 (5)	C6—C7—C9	116.2 (4)
C7—C2—C1	126.6 (5)	O4—C8—O5	118.0 (5)
C3—C2—C1	114.5 (5)	O4—C8—C6	120.9 (6)
C4—C3—C2	121.9 (5)	O5—C8—C6	121.1 (5)
C3—C4—C5	119.9 (5)	C10—C9—C7	111.2 (4)
O3—C5—C4	116.0 (5)	O5—C10—C11	106.0 (5)
O3—C5—C6	124.5 (5)	O5—C10—C9	110.2 (4)
C4—C5—C6	119.4 (5)	C11—C10—C9	113.2 (5)
C5—C6—C7	120.8 (5)		

The acid and hydroxyl H atoms were located and refined subject to O—H = 0.85 (1) Å. Their displacement parameters were set to 1.2 times U_{eq} of their parent atoms. The C-bound H atoms were positioned geometrically and were included in the refinement in the riding-model approximation; U_{iso}(H) = 1.2U_{eq}(C,O) for H atoms on secondary and tertiary C atoms and on O atoms, and U_{iso} = 1.5U_{eq}(C) for methyl H atoms. The configuration was that taken from the NMR study of 5-carboxylemmein isolated from the marine fungus *Halorosellinia oceanica* (Chinworrungsee *et al.*, 2001); the report did not, however, mention how the configuration was assigned. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

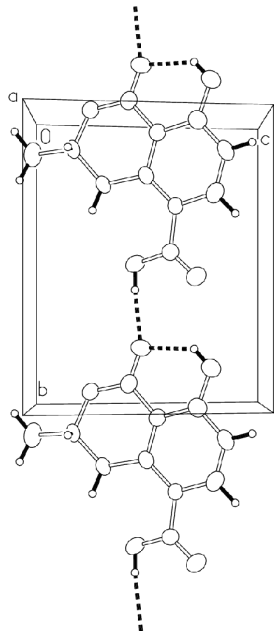


Figure 2
*ORTEP*II (Johnson, 1976) plot showing the hydrogen-bonded chain running along the *b* axis of the cell. [O1···O4^{i} = 2.702 (5) Å; symmetry code: (i) x, 1 + y, z].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

The authors thank the Natural Science Foundation of Fujian Province, China (grant No. C0110002), the Key Foundation of Science & Technology Project of Fujian Province, China (grant No. 2002H011), the National High Technology Research & Development Program of China (863 Program, No. 2002AA620401) and the University of Malaya for supporting this work.

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