Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 8-hydroxy-(*S*)-3-methyl-1-oxoisochromane-5-carboxylate (5-methoxycarbonylmellein)

Jian-Feng Wang,[a] Yong-Jie Zhang,[a] Yao-Jian Huang,[a] Wen-Jin Su,[a] Yang Lu,[b] Yu-Fen Zhao[b] and Seik Weng Ng[c]*

[a]Department of Biology, Xiamen University, Xiamen 361005, People's Republic of China, [b]Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.051
wR factor = 0.123
Data-to-parameter ratio = 7.0

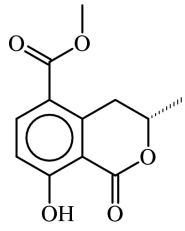
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 July 2003
Accepted 25 July 2003
Online 31 July 2003

The title compound, $C_{12}H_{12}O_5$, exists as two independent, relatively planar molecules in the asymmetric unit; these differ in the orientation of the ester group.

Comment

Methyl 8-hydroxy-3-methyl-1-oxo-isochromane-5-carboxylate, (I) (Fig. 1), another dihydroisocoumarin, was isolated from *Tubercularia sp*., and the formulation differs from that of carboxymellein in having an ester group instead of an acid group (Wang *et al.*, 2003). The compound crystallizes as two independent molecules per asymmetric unit; these differ in the orientation of the $-CO_2CH_3$ group with respect to the relatively planar dihydroisocoumarin system.



(I)

Experimental

The title compound was isolated from an endophytic fungus, *Tubercularia sp*., under conditions somewhat different from those used for isolating carboxymellein. Needle-shaped crystals were grown from an ethyl acetate solution.

Crystal data

$C_{12}H_{12}O_5$
M_r = 236.22
Monoclinic, $P2_1$
a = 10.9633 (7) Å
b = 7.1890 (5) Å
c = 14.425 (1) Å
β = 99.088 (1)°
V = 1122.7 (1) Å^3
Z = 4
D_x = 1.398 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2648 reflections
θ = 2.5–23.7°
μ = 0.11 mm^{-1}
T = 298 (2) K
Needle, colorless
0.53 × 0.19 × 0.11 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
8222 measured reflections
2154 independent reflections
1700 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 25.0°
$h = -12 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.123
S = 1.02
2154 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.069P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1*a*—C2*a*	1.319 (5)	O1*b*—C2*b*	1.318 (5)
O1*a*—C1*a*	1.443 (5)	O1*b*—C1*b*	1.444 (5)
O2*a*—C2*a*	1.182 (5)	O2*b*—C2*b*	1.191 (5)
O3*a*—C6*a*	1.332 (5)	O3*b*—C6*b*	1.348 (5)
O4*a*—C12*a*	1.219 (4)	O4*b*—C12*b*	1.220 (5)
O5*a*—C12*a*	1.320 (5)	O5*b*—C12*b*	1.319 (5)
O5*a*—C10*a*	1.458 (5)	O5*b*—C10*b*	1.465 (6)
C2*a*—C3*a*	1.481 (5)	C2*b*—C3*b*	1.471 (5)
C3*a*—C4*a*	1.387 (6)	C3*b*—C4*b*	1.388 (5)
C3*a*—C8*a*	1.396 (5)	C3*b*—C8*b*	1.394 (5)
C4*a*—C5*a*	1.358 (6)	C4*b*—C5*b*	1.359 (6)
C5*a*—C6*a*	1.387 (6)	C5*b*—C6*b*	1.377 (6)
C6*a*—C7*a*	1.401 (5)	C6*b*—C7*b*	1.401 (5)
C7*a*—C8*a*	1.409 (5)	C7*b*—C8*b*	1.396 (5)
C7*a*—C12*a*	1.473 (5)	C7*b*—C12*b*	1.467 (6)
C8*a*—C9*a*	1.501 (5)	C8*b*—C9*b*	1.499 (5)
C9*a*—C10*a*	1.491 (5)	C9*b*—C10*b*	1.501 (5)
C10*a*—C11*a*	1.500 (5)	C10*b*—C11*b*	1.498 (6)
C2*a*—O1*a*—C1*a*	118.0 (3)	C2*b*—O1*b*—C1*b*	116.8 (3)
C12*a*—O5*a*—C10*a*	118.0 (3)	C12*b*—O5*b*—C10*b*	118.9 (3)
O1*a*—C2*a*—O2*a*	121.9 (4)	O1*b*—C2*b*—O2*b*	120.3 (4)
O1*a*—C2*a*—C3*a*	115.0 (3)	O1*b*—C2*b*—C3*b*	112.7 (3)
O2*a*—C2*a*—C3*a*	123.1 (5)	O2*b*—C2*b*—C3*b*	126.9 (4)
C4*a*—C3*a*—C8*a*	118.7 (4)	C4*b*—C3*b*—C8*b*	118.0 (4)
C4*a*—C3*a*—C2*a*	114.8 (4)	C4*b*—C3*b*—C2*b*	120.0 (3)
C8*a*—C3*a*—C2*a*	126.5 (4)	C8*b*—C3*b*—C2*b*	122.0 (3)
C5*a*—C4*a*—C3*a*	122.8 (4)	C5*b*—C4*b*—C3*b*	123.1 (4)
C4*a*—C5*a*—C6*a*	120.0 (4)	C4*b*—C5*b*—C6*b*	119.4 (4)
O3*a*—C6*a*—C5*a*	117.1 (4)	O3*b*—C6*b*—C5*b*	117.9 (4)
O3*a*—C6*a*—C7*a*	124.2 (4)	O3*b*—C6*b*—C7*b*	122.5 (4)
C5*a*—C6*a*—C7*a*	118.7 (4)	C5*b*—C6*b*—C7*b*	119.5 (4)
C6*a*—C7*a*—C8*a*	121.0 (3)	C8*b*—C7*b*—C6*b*	120.4 (3)
C6*a*—C7*a*—C12*a*	117.6 (4)	C8*b*—C7*b*—C12*b*	120.8 (4)
C8*a*—C7*a*—C12*a*	121.4 (4)	C6*b*—C7*b*—C12*b*	118.8 (4)
C3*a*—C8*a*—C7*a*	118.8 (3)	C3*b*—C8*b*—C7*b*	119.6 (3)
C3*a*—C8*a*—C9*a*	125.8 (3)	C3*b*—C8*b*—C9*b*	124.5 (4)
C7*a*—C8*a*—C9*a*	115.4 (3)	C7*b*—C8*b*—C9*b*	115.9 (3)
C10*a*—C9*a*—C8*a*	112.4 (3)	C8*b*—C9*b*—C10*b*	111.6 (3)
O5*a*—C10*a*—C9*a*	110.9 (3)	O5*b*—C10*b*—C11*b*	106.5 (4)
O5*a*—C10*a*—C11*a*	106.2 (3)	O5*b*—C10*b*—C9*b*	109.5 (4)
C9*a*—C10*a*—C11*a*	113.9 (4)	C11*b*—C10*b*—C9*b*	113.5 (4)
O4*a*—C12*a*—O5*a*	117.3 (3)	O4*b*—C12*b*—O5*b*	118.2 (4)
O4*a*—C12*a*—C7*a*	123.2 (4)	O4*b*—C12*b*—C7*b*	122.2 (4)
O5*a*—C12*a*—C7*a*	119.5 (4)	O5*b*—C12*b*—C7*b*	119.6 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3*a*—H3*a*···O4*a*	0.82	1.86	2.578 (4)	145
O3*b*—H3*b*···O4*b*	0.82	1.84	2.565 (4)	146

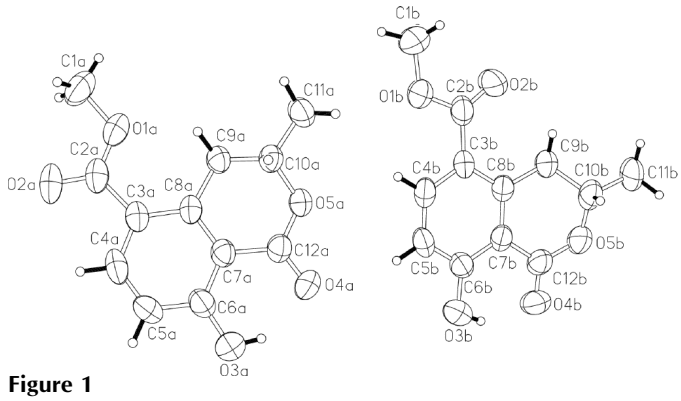


Figure 1
*ORTEP*II (Johnson, 1976) plot of the asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

The H atoms were positioned geometrically and were included in the refinement in the riding-model approximation, including torsional freedom of OH groups. The C—H distances were set to 0.93–0.98 Å and the O—H distance to 0.82 Å, with U_{iso} values for H atoms of 1.2 or 1.5 (methyl H) times U_{eq} of the parent atom. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; 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. 2001AA620401) and the University of Malaya for supporting this work.

References

Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M.(1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Wang, J. F., Fang, M.-J., Zhao, Y.-F., Huang, Y. J., Su, W.-J. & Ng, S. W. (2003). *Acta Cryst.* E**59**, o1233–o1234.