

# Methyl 8-hydroxy-(S)-3-methyl-1-oxoiso-chromane-5-carboxylate (5-methoxy-carbonylmellein)

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## Key indicators

Single-crystal X-ray study

$T = 298$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.006$  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>.

The title compound,  $\text{C}_{12}\text{H}_{12}\text{O}_5$ , exists as two independent, relatively planar molecules in the asymmetric unit; these differ in the orientation of the ester group.

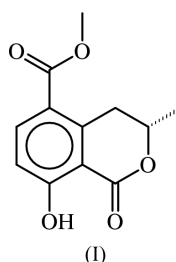
Received 23 July 2003

Accepted 25 July 2003

Online 31 July 2003

## 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  $-\text{CO}_2\text{CH}_3$  group with respect to the relatively planar dihydroisocoumarin system.



## 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

$\text{C}_{12}\text{H}_{12}\text{O}_5$

$M_r = 236.22$

Monoclinic,  $P2_1$

$a = 10.9633$  (7) Å

$b = 7.1890$  (5) Å

$c = 14.425$  (1) Å

$\beta = 99.088$  (1)°

$V = 1122.7$  (1) Å<sup>3</sup>

$Z = 4$

$D_x = 1.398$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

Cell parameters from 2648

reflections

$\theta = 2.5$ – $23.7^\circ$

$\mu = 0.11$  mm<sup>-1</sup>

$T = 298$  (2) K

Needle, colorless

$0.53 \times 0.19 \times 0.11$  mm

### Data collection

Bruker SMART area-detector diffractometer

$\varphi$  and  $\omega$  scans

Absorption correction: none

8222 measured reflections

2154 independent reflections

1700 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\text{max}} = 25.0^\circ$

$h = -12 \rightarrow 13$

$k = -8 \rightarrow 8$

$l = -16 \rightarrow 17$

### 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  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup>

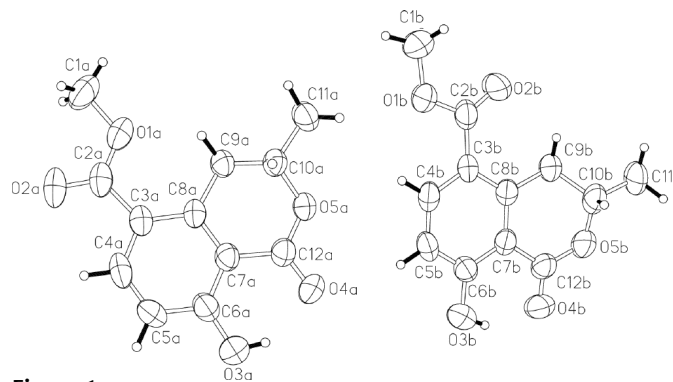
$\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

**Table 1**  
Selected geometric parameters (Å, °).

O1a—C2a	1.319 (5)	O1b—C2b	1.318 (5)
O1a—C1a	1.443 (5)	O1b—C1b	1.444 (5)
O2a—C2a	1.182 (5)	O2b—C2b	1.191 (5)
O3a—C6a	1.332 (5)	O3b—C6b	1.348 (5)
O4a—C12a	1.219 (4)	O4b—C12b	1.220 (5)
O5a—C12a	1.320 (5)	O5b—C12b	1.319 (5)
O5a—C10a	1.458 (5)	O5b—C10b	1.465 (6)
C2a—C3a	1.481 (5)	C2b—C3b	1.471 (5)
C3a—C4a	1.387 (6)	C3b—C4b	1.388 (5)
C3a—C8a	1.396 (5)	C3b—C8b	1.394 (5)
C4a—C5a	1.358 (6)	C4b—C5b	1.359 (6)
C5a—C6a	1.387 (6)	C5b—C6b	1.377 (6)
C6a—C7a	1.401 (5)	C6b—C7b	1.401 (5)
C7a—C8a	1.409 (5)	C7b—C8b	1.396 (5)
C7a—C12a	1.473 (5)	C7b—C12b	1.467 (6)
C8a—C9a	1.501 (5)	C8b—C9b	1.499 (5)
C9a—C10a	1.491 (5)	C9b—C10b	1.501 (5)
C10a—C11a	1.500 (5)	C10b—C11b	1.498 (6)
C2a—O1a—C1a	118.0 (3)	C2b—O1b—C1b	116.8 (3)
C12a—O5a—C10a	118.0 (3)	C12b—O5b—C10b	118.9 (3)
O1a—C2a—O2a	121.9 (4)	O1b—C2b—O2b	120.3 (4)
O1a—C2a—C3a	115.0 (3)	O1b—C2b—C3b	112.7 (3)
O2a—C2a—C3a	123.1 (5)	O2b—C2b—C3b	126.9 (4)
C4a—C3a—C8a	118.7 (4)	C4b—C3b—C8b	118.0 (4)
C4a—C3a—C2a	114.8 (4)	C4b—C3b—C2b	120.0 (3)
C8a—C3a—C2a	126.5 (4)	C8b—C3b—C2b	122.0 (3)
C5a—C4a—C3a	122.8 (4)	C5b—C4b—C3b	123.1 (4)
C4a—C5a—C6a	120.0 (4)	C4b—C5b—C6b	119.4 (4)
O3a—C6a—C5a	117.1 (4)	O3b—C6b—C5b	117.9 (4)
O3a—C6a—C7a	124.2 (4)	O3b—C6b—C7b	122.5 (4)
C5a—C6a—C7a	118.7 (4)	C5b—C6b—C7b	119.5 (4)
C6a—C7a—C8a	121.0 (3)	C6b—C7b—C8b	120.4 (3)
C6a—C7a—C12a	117.6 (4)	C6b—C7b—C12b	120.8 (4)
C8a—C7a—C12a	121.4 (4)	C6b—C7b—C12b	118.8 (4)
C3a—C8a—C7a	118.8 (3)	C3b—C8b—C7b	119.6 (3)
C3a—C8a—C9a	125.8 (3)	C3b—C8b—C9b	124.5 (4)
C7a—C8a—C9a	115.4 (3)	C7b—C8b—C9b	115.9 (3)
C10a—C9a—C8a	112.4 (3)	C8b—C9b—C10b	111.6 (3)
O5a—C10a—C9a	110.9 (3)	O5b—C10b—C11b	106.5 (4)
O5a—C10a—C11a	106.2 (3)	O5b—C10b—C9b	109.5 (4)
C9a—C10a—C11a	113.9 (4)	C11b—C10b—C9b	113.5 (4)
O4a—C12a—O5a	117.3 (3)	O4b—C12b—O5b	118.2 (4)
O4a—C12a—C7a	123.2 (4)	O4b—C12b—C7b	122.2 (4)
O5a—C12a—C7a	119.5 (4)	O5b—C12b—C7b	119.6 (4)

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

D—H...A	D—H	H...A	D...A	D—H...A
O3a—H3a...O4a	0.82	1.86	2.578 (4)	145
O3b—H3b...O4b	0.82	1.84	2.565 (4)	146



**Figure 1**  
ORTEPII (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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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.

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