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7,8-Dihydroxy-3-methyl-10-oxo-1*H*,10*H*-pyrano[4,3-*b*]chromene-9-carboxylic acid

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.119
Data-to-parameter ratio = 13.0

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

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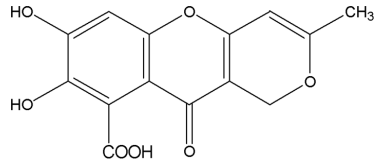
The structure of the title compound, anhydrofulvic acid, $C_{14}H_{10}O_7$, a yellow acidic metabolite isolated from *Paecilomyces sp*. was determined by X-ray analysis. The chromone ring system is essentially planar, with the carboxylic acid group coplanar with the ring.

Comment

The title compound, (I), had been prepared by dehydration of a natural product, fulvic acid [3,7,8-trihydroxy-3-methyl-10-oxo-4,10-dihydro-1*H*,3*H*-pyrano[4,3-*b*]chromene-9-carboxylic acid, (II)], isolated from several fungi (Dean *et al.*, 1957). In this study, (I) was isolated from the fermentation broth of *Paecilomyces sp*., an endophytic fungus of *Cephalataxus fortunei*, and its structure was determined by X-ray analysis.



(I)

The chromone ring system of (I) is essentially planar, with the hydroxyl and carboxylic acid groups coplanar with the benzene ring. There is one intermolecular hydrogen bond and three intramolecular hydrogen bonds in the crystal structure (Table 2), rendering the crystal very stable (m.p. 516–518 K).

Experimental

The title compound, (I), was isolated from the organic extract of the liquid culture of *Paecilomyces sp*. Recrystallization from ethyl acetate afforded green crystals suitable for X-ray analysis. The molecular formula of (I) was deduced from the high resolution ESI–MS spectrum as $C_{14}H_{10}O_7$, showing an accurate mass at *m*/*z* 291.0501 $[M + H]^+$. The ^{13}C NMR analysis revealed 14 C atoms: δ (p.p.m.) = 20.0 (C13), 64.3(C12), 94.5 (C10), 101.3 (C4), 103.358 (C8), 113.0 (C6), 118.0 (C1), 143.5 (C2), 149.8 (C3), 152.0 (C5), 158.6 (C9), 167.6 (C11), 168.8 (C14) and 171.3 (C7).

Crystal data

$C_{14}H_{10}O_7$
M_r = 290.22
Monoclinic, $P2_1/c$
a = 7.814 (5) Å
b = 10.085 (5) Å
c = 15.124 (5) Å
β = 90.178 (5)°
V = 1191.8 (10) Å^3
Z = 4
D_x = 1.617 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2225 reflections
θ = 2.4–27.5°
μ = 0.13 mm^{-1}
T = 296 (2) K
Chunk, green
0.20 × 0.18 × 0.10 mm

Data collection

Bruker AXS SMART area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.974, T_{max} = 0.987
10 074 measured reflections
2615 independent reflections
2225 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.119
S = 1.09
2615 reflections
201 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0758P)^2 + 0.0975P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.013 (3)

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.3410 (16)	O4—C14	1.2473 (16)
O1—C5	1.3671 (14)	C4—C5	1.3922 (18)
C1—C2	1.3972 (18)	O5—C14	1.2704 (18)
C1—C6	1.4468 (15)	C5—C6	1.4046 (16)
C1—C14	1.5057 (16)	C6—C7	1.4594 (17)
O2—C11	1.3531 (18)	C7—C8	1.4044 (16)
O2—C12	1.4362 (16)	C8—C9	1.3585 (18)
C2—O6	1.3329 (14)	C8—C12	1.5034 (18)
C2—C3	1.4257 (18)	C9—C10	1.4192 (16)
O3—C7	1.2802 (15)	C10—C11	1.344 (2)
C3—O7	1.3391 (16)	C11—C13	1.4838 (17)
C3—C4	1.3613 (16)		
C9—O1—C5	119.65 (9)	C9—C8—C7	121.37 (11)
C2—C1—C6	118.46 (10)	C9—C8—C12	116.98 (11)
O6—C2—C1	125.17 (11)	C7—C8—C12	121.50 (11)
O6—C2—C3	112.45 (11)	O1—C9—C8	121.83 (11)
C1—C2—C3	122.38 (10)	O1—C9—C10	116.77 (11)
O7—C3—C4	120.34 (11)	C8—C9—C10	121.35 (11)
O7—C3—C2	120.28 (11)	C2—C1—C14	115.70 (10)
C4—C3—C2	119.37 (11)	C6—C1—C14	125.84 (11)
C3—C4—C5	118.80 (11)	C11—O2—C12	117.25 (10)
O1—C5—C4	112.05 (10)	C11—C10—C9	118.01 (12)
O1—C5—C6	123.10 (10)	C10—C11—O2	122.64 (11)
C4—C5—C6	124.85 (10)	C10—C11—C13	124.90 (13)
C5—C6—C1	116.12 (11)	O2—C11—C13	112.29 (12)
C5—C6—C7	116.07 (10)	O2—C12—C8	112.12 (11)
C1—C6—C7	127.81 (10)	O4—C14—O5	119.47 (11)
O3—C7—C8	117.51 (11)	O4—C14—C1	118.04 (13)
O3—C7—C6	124.58 (11)	O5—C14—C1	122.49 (11)
C8—C7—C6	117.90 (10)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O3	1.18 (1)	1.19 (1)	2.3696 (15)	177 (2)
O6—H6···O4	0.93 (2)	1.56 (2)	2.4395 (18)	155.4 (18)
O7—H7···O6	0.84 (2)	2.17 (2)	2.6108 (16)	113.0 (19)
O7—H7···O4^{i}	0.84 (2)	1.94 (2)	2.6794 (15)	147 (2)

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

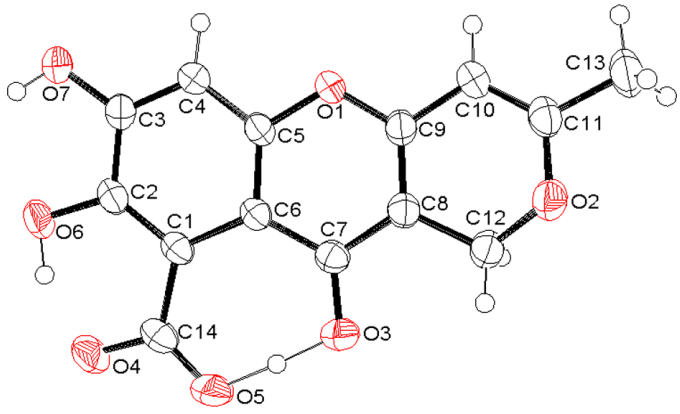


Figure 1
*ORTEP*3 (Farrugia, 1997) plot of the structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

O-bound H atoms were located in a difference Fourier synthesis, and their coordinates were refined. C-bound H atoms were placed at calculated positions (C—H = 0.93, 0.96 or 0.97 Å) and were included in the refinement in the riding-model approximation. Their displacement parameters were set at 1.2 or 1.5 times U_{eq} of the parent C or O atoms, respectively.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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