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6,8-Dihydroxy-3-methylisocoumarin

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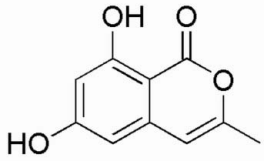
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 12.1.

The title compound, $C_{10}H_8O_4$, was isolated from the fermentation culture of the endophytic fungus *Cephalosporium* sp. In the crystal structure, molecules are connected into a one-dimensional chain along [101] by intermolecular O—H···O hydrogen bonds involving the hydroxyl and carbonyl functionalities. The chains are linked by non-classical C—H···O interactions, forming extended two-dimensional layers approximately parallel to (11$\bar{2}$).

Related literature

For new bioactive secondary metabolites from marine fungi, see: Shao *et al.* (2007). For the investigation of an endophytic strain *Cephalosporium* sp., see: Wei *et al.* (2008); Hemingway *et al.* (1977); Kendall *et al.* (1989). For crystal structures with non-classical C—H···O interactions, see: Nangia (2002).



Experimental

Crystal data

$C_{10}H_8O_4$
M_r = 192.16
Monoclinic, $P2_1/c$
a = 3.8201 (7) Å
b = 15.710 (3) Å
c = 14.196 (2) Å
β = 92.668 (2)°
V = 851.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 291 K
0.27 × 0.20 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.969, T_{max} = 0.978
4781 measured reflections
1586 independent reflections
1272 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.04
1586 reflections
131 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2^{i}	0.82	1.96	2.7225 (15)	155
C5—H5···O4^{ii}	0.93	2.60	3.4659 (19)	155

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick 2008); molecular graphics: *SHELXTL* (Sheldrick 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2219).

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supplementary materials

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6,8-Dihydroxy-3-methylisocoumarin

C. Shao, L. Han, C. Li, Z. Liu and C. Wang

Comment

Endophytic fungi have proven to be a rich source of novel structural compounds with interesting biological activities and a high level of biodiversity. In the course of our search for new or bioactive secondary metabolites from the marine fungi (Shao *et al.*, 2007). we have investigated an endophytic strain *Cephalosporium sp*. (Wei *et al.*, 2008). The title compound was previously isolated from the organic extracts of the fungus *Ceratocystis minor* (Hemingway *et al.*, 1977), and elucidated on the basic of spectroscopic analysis (Kendall *et al.*, 1989). Herein, the title compound was isolated from the fermentation culture of the endophytic fungus *Cephalosporium sp*., and its crystal structure is reported.

The asymmetric unit of the title compound contains one independent molecule (Fig. 1), in which the bond lengths and angles are within the expected ranges. The structural analysis reveals that the most relevant feature is the arrangement of the molecules, which are connected to form a one-dimensional chain along the [101] direction, by the formation of intermolecular O—H···O hydrogen bonds. Furthermore, weak non-conventional intermolecular C—H···O interactions are observed (Nangia, 2002), in which C5—H5 is a donor and O4 is an acceptor. These interactions consolidate the crystal packing. Details of hydrogen bonds are given in Table 1.

Experimental

A strain of fungus *Cephalosporium sp*. (No. 2090) was deposited in the School of Chemistry and Chemical Engineering, Sun Yat-sen University, Guangzhou, People's Republic of China. Culture conditions: GYT medium (glucose 10 g/L, peptone 2 g/L, yeast extract 1 g/L, NaCl 2.5 g/L) and incubation at 298 K for 4 weeks. The cultures (70 L) were filtered through cheesecloth. The filtrate was concentrated to 3 L below 323 K, extracted five times by shaking with an equal volume of ethyl acetate. The extract was evaporated under reduced pressure below 323 K. The combined organic extracts were chromatographed on silica-gel, eluting with petroleum ether/ethyl acetate, to yield the title compound. Crystals were obtained by evaporation of an ethyl acetate solution.

Refinement

All H atoms were positioned geometrically and treated as riding, with C—H bond lengths constrained to 0.93 (aromatic CH), 0.96 (methyl CH_3), and 0.82 Å (hydroxyl OH), and with U_{iso}(H) = 1.5Ueq(carrier atom) or for CH_3 and OH groups and U_{iso}(H) = 1.2Ueq(carrier C) otherwise.

Figures

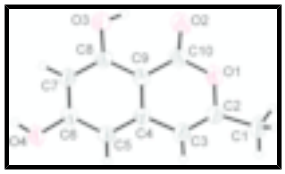

Fig. 1. View of the title molecule with atom numbering scheme and 30% probability displacement ellipsoids for non-hydrogen atoms.

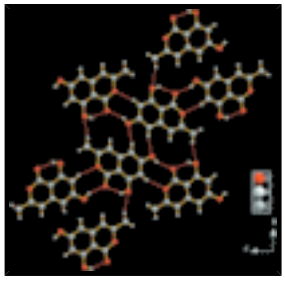

Fig. 2. View of the 2D layers formed by intermolecular O—H···O hydrogen bonds and weak non-conventional intermolecular C—H···O interactions.

6,8-Dihydroxy-3-methylisocoumarin

Crystal data

$C_{10}H_8O_4$	F_{000} = 400
M_r = 192.16	D_x = 1.500 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1738 reflections
a = 3.8201 (7) Å	θ = 2.6–25.5º
b = 15.710 (3) Å	μ = 0.12 mm^{-1}
c = 14.196 (2) Å	T = 291 K
β = 92.668 (2)º	Block, yellow
V = 851.1 (3) Å^3	0.27 × 0.20 × 0.19 mm
Z = 4	

Data collection

Bruker APEXII CCD diffractometer	1586 independent reflections
Radiation source: fine-focus sealed tube	1272 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 291 K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −4→4
T_{min} = 0.969, T_{max} = 0.978	k = −19→13
4781 measured reflections	l = −17→17

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.034$	$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.1957P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.094$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
1586 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
131 parameters	Extinction correction: SHELXL97 (Sheldrick 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.015 (3)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3482 (3)	0.32453 (7)	0.81217 (7)	0.0451 (3)
O2	0.3544 (3)	0.19366 (7)	0.75748 (8)	0.0572 (4)
O3	0.0718 (3)	0.15766 (7)	0.58873 (8)	0.0552 (4)
H3	0.1709	0.1476	0.6400	0.083*
O4	−0.4021 (3)	0.39231 (7)	0.41364 (7)	0.0487 (3)
H4	−0.4514	0.3541	0.3759	0.073*
C1	0.3853 (4)	0.45742 (12)	0.89102 (11)	0.0516 (4)
H1A	0.3148	0.5160	0.8859	0.077*
H1B	0.6359	0.4542	0.8987	0.077*
H1C	0.2819	0.4321	0.9446	0.077*
C2	0.2659 (4)	0.41080 (10)	0.80372 (10)	0.0395 (4)
C3	0.1028 (4)	0.44079 (10)	0.72629 (10)	0.0379 (4)
H3A	0.0505	0.4985	0.7224	0.046*
C4	0.0042 (3)	0.38590 (9)	0.64774 (9)	0.0318 (3)
C5	−0.1641 (4)	0.41528 (9)	0.56587 (10)	0.0355 (3)
H5	−0.2230	0.4725	0.5598	0.043*
C6	−0.2453 (4)	0.35866 (10)	0.49244 (10)	0.0354 (3)
C7	−0.1659 (4)	0.27235 (9)	0.50066 (10)	0.0378 (4)
H7	−0.2235	0.2354	0.4512	0.045*
C8	−0.0016 (4)	0.24192 (9)	0.58233 (10)	0.0368 (3)
C9	0.0880 (4)	0.29837 (9)	0.65755 (9)	0.0337 (3)
C10	0.2651 (4)	0.26818 (10)	0.74166 (10)	0.0401 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0531 (7)	0.0503 (7)	0.0308 (5)	0.0018 (5)	−0.0102 (5)	−0.0008 (5)
O2	0.0802 (9)	0.0448 (7)	0.0447 (7)	0.0086 (6)	−0.0182 (6)	0.0060 (5)

O3	0.0804 (9)	0.0330 (6)	0.0503 (7)	0.0065 (5)	−0.0162 (6)	−0.0011 (5)
O4	0.0677 (8)	0.0425 (6)	0.0342 (6)	0.0033 (5)	−0.0174 (5)	−0.0004 (5)
C1	0.0488 (10)	0.0661 (11)	0.0390 (9)	−0.0024 (8)	−0.0060 (7)	−0.0131 (8)
C2	0.0382 (8)	0.0459 (9)	0.0344 (8)	−0.0026 (7)	0.0008 (6)	−0.0058 (6)
C3	0.0414 (8)	0.0372 (8)	0.0350 (8)	−0.0021 (6)	0.0002 (6)	−0.0034 (6)
C4	0.0311 (7)	0.0354 (8)	0.0291 (7)	−0.0033 (6)	0.0016 (6)	−0.0006 (6)
C5	0.0405 (8)	0.0314 (8)	0.0344 (7)	0.0001 (6)	−0.0011 (6)	0.0009 (6)
C6	0.0354 (8)	0.0407 (8)	0.0297 (7)	−0.0013 (6)	−0.0037 (6)	0.0029 (6)
C7	0.0437 (8)	0.0379 (9)	0.0312 (7)	−0.0045 (6)	−0.0047 (6)	−0.0053 (6)
C8	0.0412 (8)	0.0318 (8)	0.0370 (8)	−0.0010 (6)	−0.0011 (6)	−0.0003 (6)
C9	0.0344 (8)	0.0364 (8)	0.0301 (7)	−0.0011 (6)	−0.0005 (6)	0.0019 (6)
C10	0.0436 (9)	0.0422 (9)	0.0339 (8)	0.0007 (7)	−0.0030 (6)	0.0033 (6)

Geometric parameters (Å, °)

O1—C10	1.3626 (18)	C3—C4	1.4457 (19)
O1—C2	1.3951 (19)	C3—H3A	0.9300
O2—C10	1.2370 (18)	C4—C5	1.3813 (19)
O3—C8	1.3553 (18)	C4—C9	1.417 (2)
O3—H3	0.8200	C5—C6	1.394 (2)
O4—C6	1.3518 (17)	C5—H5	0.9300
O4—H4	0.8200	C6—C7	1.393 (2)
C1—C2	1.493 (2)	C7—C8	1.378 (2)
C1—H1A	0.9600	C7—H7	0.9300
C1—H1B	0.9600	C8—C9	1.418 (2)
C1—H1C	0.9600	C9—C10	1.426 (2)
C2—C3	1.325 (2)		
C10—O1—C2	121.61 (11)	C4—C5—C6	119.64 (13)
C8—O3—H3	109.5	C4—C5—H5	120.2
C6—O4—H4	109.5	C6—C5—H5	120.2
C2—C1—H1A	109.5	O4—C6—C7	122.43 (13)
C2—C1—H1B	109.5	O4—C6—C5	116.35 (13)
H1A—C1—H1B	109.5	C7—C6—C5	121.22 (13)
C2—C1—H1C	109.5	C8—C7—C6	119.81 (13)
H1A—C1—H1C	109.5	C8—C7—H7	120.1
H1B—C1—H1C	109.5	C6—C7—H7	120.1
C3—C2—O1	120.81 (13)	O3—C8—C7	118.69 (13)
C3—C2—C1	128.92 (15)	O3—C8—C9	121.23 (13)
O1—C2—C1	110.27 (13)	C7—C8—C9	120.08 (13)
C2—C3—C4	121.57 (14)	C4—C9—C8	119.18 (12)
C2—C3—H3A	119.2	C4—C9—C10	120.08 (13)
C4—C3—H3A	119.2	C8—C9—C10	120.74 (13)
C5—C4—C9	120.07 (12)	O2—C10—O1	115.39 (13)
C5—C4—C3	122.96 (13)	O2—C10—C9	125.66 (14)
C9—C4—C3	116.96 (12)	O1—C10—C9	118.96 (13)
C10—O1—C2—C3	0.1 (2)	C5—C4—C9—C8	0.3 (2)
C10—O1—C2—C1	179.78 (13)	C3—C4—C9—C8	−179.69 (13)
O1—C2—C3—C4	−0.1 (2)	C5—C4—C9—C10	179.32 (13)
C1—C2—C3—C4	−179.74 (14)	C3—C4—C9—C10	−0.66 (19)

C2—C3—C4—C5	−179.60 (14)	O3—C8—C9—C4	−179.69 (13)
C2—C3—C4—C9	0.4 (2)	C7—C8—C9—C4	0.5 (2)
C9—C4—C5—C6	−1.1 (2)	O3—C8—C9—C10	1.3 (2)
C3—C4—C5—C6	178.85 (13)	C7—C8—C9—C10	−178.57 (13)
C4—C5—C6—O4	−178.42 (12)	C2—O1—C10—O2	179.49 (13)
C4—C5—C6—C7	1.3 (2)	C2—O1—C10—C9	−0.4 (2)
O4—C6—C7—C8	179.14 (14)	C4—C9—C10—O2	−179.17 (15)
C5—C6—C7—C8	−0.5 (2)	C8—C9—C10—O2	−0.2 (2)
C6—C7—C8—O3	179.79 (14)	C4—C9—C10—O1	0.7 (2)
C6—C7—C8—C9	−0.3 (2)	C8—C9—C10—O1	179.67 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2	0.82	1.92	2.6426 (16)	146
O4—H4···O2^{i}	0.82	1.96	2.7225 (15)	155
C5—H5···O4^{ii}	0.93	2.60	3.4659 (19)	155

Symmetry codes: (i) *x*−1, −*y*+1/2, *z*−1/2; (ii) −*x*−1, −*y*+1, −*z*+1.

Fig. 1

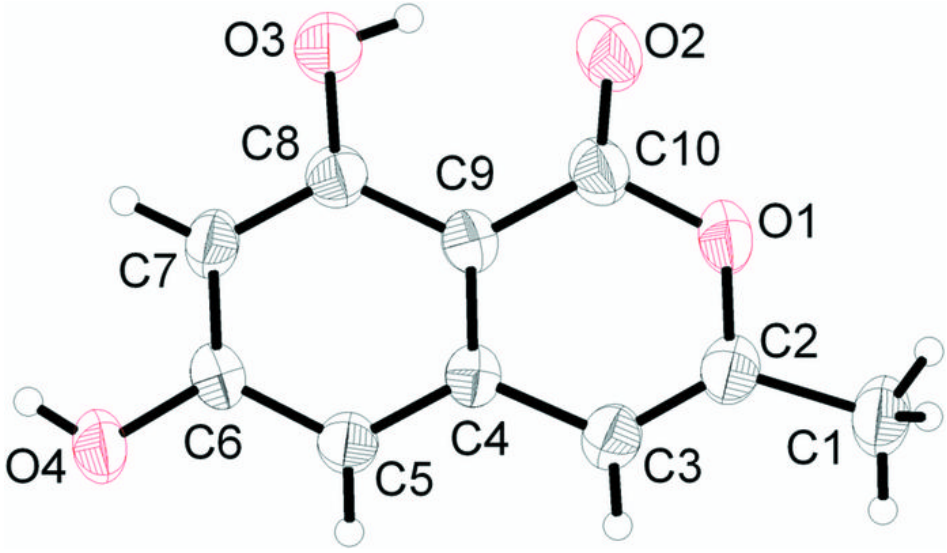

Fig. 2