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6,8-Dihydroxy-3-methylisocoumarin

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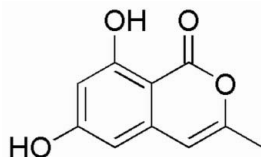
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 12.1.

The title compound, $\text{C}_{10}\text{H}_8\text{O}_4$, 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 $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds involving the hydroxyl and carbonyl functionalities. The chains are linked by non-classical $\text{C}-\text{H}\cdots\text{O}$ interactions, forming extended two-dimensional layers approximately parallel to (112).

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 $\text{C}-\text{H}\cdots\text{O}$ interactions, see: Nangia (2002).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{O}_4$
 $M_r = 192.16$
Monoclinic, $P2_1/c$
 $a = 3.8201$ (7) Å

$b = 15.710$ (3) Å
 $c = 14.196$ (2) Å
 $\beta = 92.668$ (2)°
 $V = 851.1$ (3) Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.12$ mm⁻¹

$T = 291$ K
 $0.27 \times 0.20 \times 0.19$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

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
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O4}-\text{H4}\cdots\text{O2}^{\text{i}}$	0.82	1.96	2.7225 (15)	155
$\text{C5}-\text{H5}\cdots\text{O4}^{\text{ii}}$	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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6,8-Dihydroxy-3-methylisocoumarin

Chang-lun Shao, Lei Han, Chun-yuan Li, Zhen Liu and Chang-yun Wang

S1. 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 basis 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.

S2. 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.

S3. 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 CH₃), and 0.82 Å (hydroxyl OH), and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{carrier atom})$ or for CH₃ and OH groups and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier C})$ otherwise.

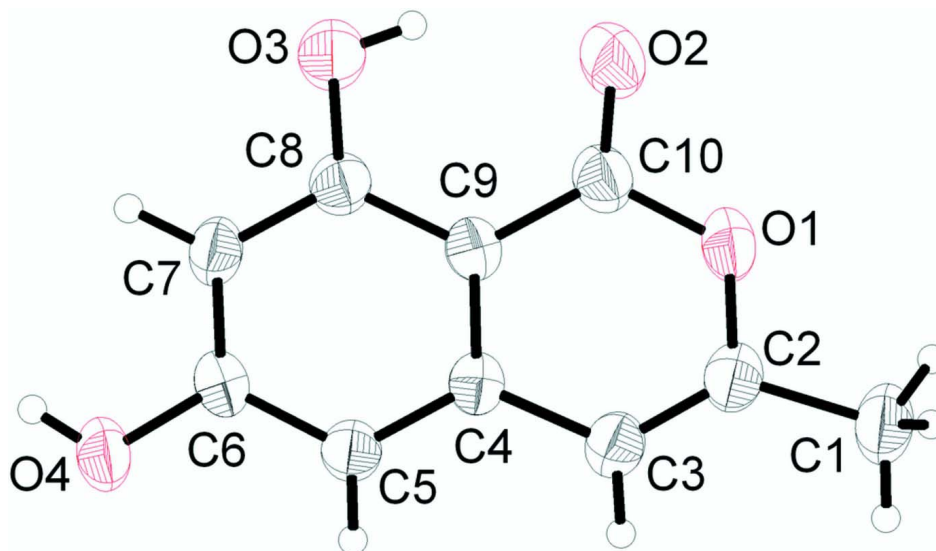


Figure 1

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

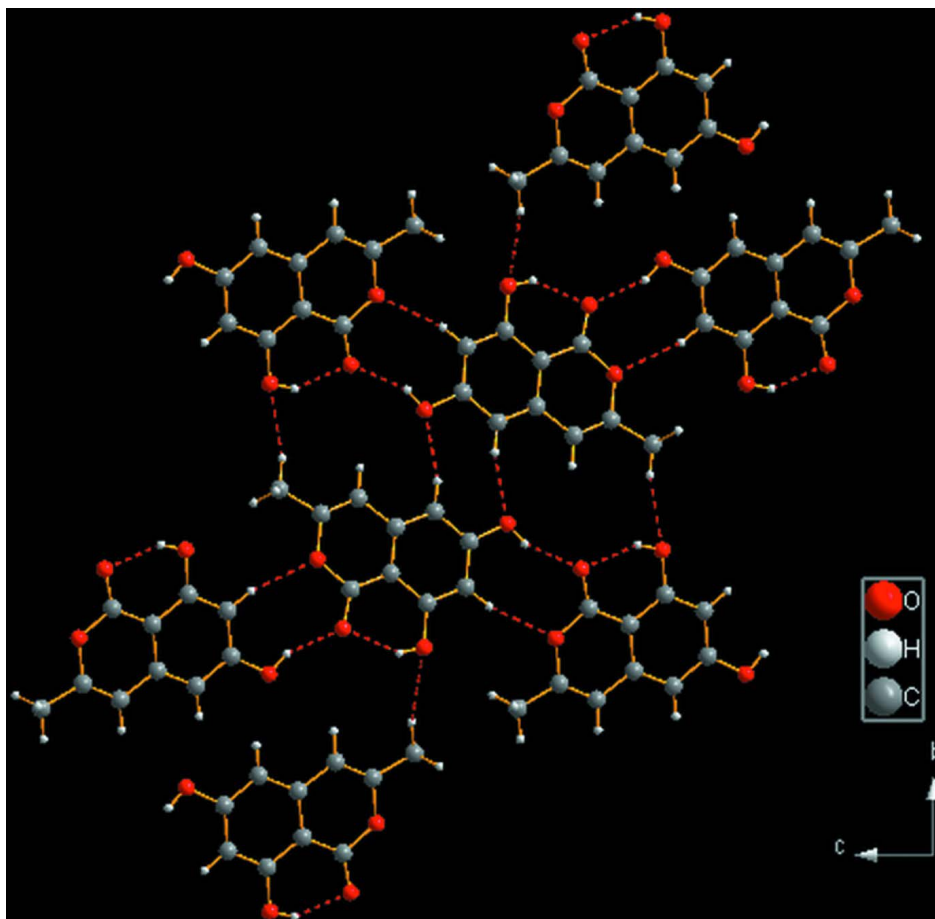


Figure 2

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

$C_{10}H_8O_4$

$M_r = 192.16$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 3.8201\ (7)\ \text{\AA}$

$b = 15.710\ (3)\ \text{\AA}$

$c = 14.196\ (2)\ \text{\AA}$

$\beta = 92.668\ (2)^\circ$

$V = 851.1\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 400$

$D_x = 1.500\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1738 reflections

$\theta = 2.6\text{--}25.5^\circ$

$\mu = 0.12\ \text{mm}^{-1}$

$T = 291\ \text{K}$

Block, yellow

$0.27 \times 0.20 \times 0.19\ \text{mm}$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.969$, $T_{\max} = 0.978$

4781 measured reflections

1586 independent reflections

1272 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 2.6^\circ$

$h = -4 \rightarrow 4$
 $k = -19 \rightarrow 13$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.094$
 $S = 1.04$
 1586 reflections
 131 parameters
 0 restraints
 0 constraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.1957P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.14 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.015 (3)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3 \cdots O2	0.82	1.92	2.6426 (16)	146
O4—H4 \cdots O2 ⁱ	0.82	1.96	2.7225 (15)	155
C5—H5 \cdots O4 ⁱⁱ	0.93	2.60	3.4659 (19)	155

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