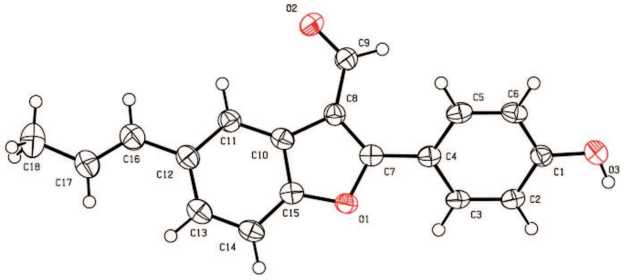
Guoyong Luo, Wude Yang, Tianqiong Lang*, Chenliang Zhao and Jingjie Zhang*

The crystal structure of (*E*)-2-(4-hydroxyphenyl)-5-(prop-1-en-1-yl)benzofuran-3-carbaldehyde, $C_{18}H_{14}O_3$



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Abstract

$C_{18}H_{14}O_3$, monoclinic, $P2_1/n$ (no. 14), $a = 11.115(4)$ Å, $b = 6.878(3)$ Å, $c = 18.024(7)$ Å, $\beta = 99.964(8)°$, $V = 1357.1(9)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0601$, $wR_{ref}(F^2) = 0.1892$, $T = 173$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The aerial parts of *Itea ilicifolia* were collected in October 2016 at Longli County, Guizhou province, China. The EtOAc fraction of its ethanol extract was subjected to a silica gel column eluted with petroleum ether and EtOAc in gradient from 1:0 to 0:1. The eluents of petroleum ether and EtOAc in 5:1 was then separated by column chromatography repeatedly to obtain the title compound, of which the structure was further determined by spectroscopic analysis. The data used in this study was collected from a block-shaped crystal grown from MeOH.

***Corresponding authors: Tianqiong Lang and Jingjie Zhang,** School of Pharmacy, Guiyang University of Chinese Medicine, Guiyang 550025, P.R. China, e-mail: 376519015@qq.com (T. Lang), zjj523@126.com (J. Zhang)
Guoyong Luo, Wude Yang and Chenliang Zhao: School of Pharmacy, Guiyang University of Chinese Medicine, Guiyang 550025, P.R. China

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10517, 3064, 0.069
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1861
$N(param)_{refined}$:	192
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.64057(15)	0.3006(2)	0.21303(9)	0.0303(4)
O2	0.57456(18)	0.2886(3)	0.45714(10)	0.0469(6)
O3	0.06620(17)	0.3219(3)	0.10210(11)	0.0460(6)
H3	0.0594	0.2713	0.0592	0.069*
C1	0.1855(2)	0.3193(4)	0.13591(14)	0.0309(6)
C2	0.2748(2)	0.2211(4)	0.10617(13)	0.0310(6)
H2	0.2541	0.1538	0.0597	0.037*
C3	0.3935(2)	0.2208(4)	0.14385(13)	0.0296(6)
H3A	0.4544	0.1553	0.1223	0.036*
C4	0.4265(2)	0.3147(3)	0.21296(13)	0.0292(6)
C5	0.3341(2)	0.4143(4)	0.24179(13)	0.0331(6)
H5	0.3539	0.4809	0.2885	0.040*
C6	0.2164(2)	0.4172(4)	0.20392(14)	0.0336(6)
H6	0.1556	0.4864	0.2242	0.040*
C7	0.5497(2)	0.3043(3)	0.25449(13)	0.0291(6)
C8	0.5961(2)	0.2961(4)	0.33051(14)	0.0306(6)
C9	0.5270(3)	0.2815(4)	0.39092(14)	0.0347(6)
H9	0.4409	0.2656	0.3787	0.042*
C10	0.7266(2)	0.2877(4)	0.33684(14)	0.0309(6)
C11	0.8253(2)	0.2732(4)	0.39613(15)	0.0346(6)
H11	0.8118	0.2697	0.4467	0.042*
C12	0.9431(2)	0.2641(4)	0.38069(15)	0.0349(6)
C13	0.9606(3)	0.2739(4)	0.30565(16)	0.0367(6)
H13	1.0417	0.2713	0.2957	0.044*
C14	0.8651(2)	0.2874(4)	0.24536(15)	0.0341(6)
H14	0.8782	0.2930	0.1947	0.041*
C15	0.7493(2)	0.2920(4)	0.26365(14)	0.0300(6)
C16	1.0449(3)	0.2451(4)	0.44437(17)	0.0439(7)
H16	1.0227	0.2420	0.4929	0.053*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C17	1.1626(3)	0.2317(4)	0.44294(18)	0.0448(7)
H17	1.1874	0.2348	0.3951	0.054*
C18	1.2598(3)	0.2122(5)	0.5104(2)	0.0580(9)
H18A	1.3044	0.0903	0.5073	0.087*
H18B	1.2227	0.2113	0.5559	0.087*
H18C	1.3165	0.3220	0.5125	0.087*

Experimental details

A 0.28 mm × 0.15 mm × 0.12 mm sized crystal was selected and the diffraction experiments were performed on a Bruker APEX-II CCD diffractometer. The structure was solved with the ShelXT [3] structure solution program using Intrinsic Phasing and refined with the ShelXL refinement package [4] using least squares minimisation. All hydrogen atoms were positioned geometrically without any constraints or restraints, with C—H distance of 0.95–0.98 Å. The U_{iso} values were fixed at 1.2 times of all the C(H) group, and 1.5 times of the C(H, H, H) and O(H) group.

Comment

3-Formyl-2-arylbenzofurans, a small kind of natural products with unique skeleton, has attracted much attention for their various biological effects including cytotoxic, anti-inflammatory, antioxidant, and antimicrobial activity [5–8]. The crystal structure of the analogue of title compound was reported before, which was obtained from the same plant source [8]. Since the spectral patterns are similar to those of allenic structures [9, 10], X-ray crystallography analysis is important for the unambiguous structure elucidation of 3-formyl-2-arylbenzofurans.

The bond distances and bond angles are all in normal ranges in the crystal structure of the title compound. In addition to basic skeleton of 2-arylbenzofuran, functional groups: one hydroxyl, one formyl, and one propenyl groups, were identified by the following characteristic distances: d(C1—O3) = 1.360(3) Å, d(C9—O2) = 1.220(3) Å, d(C16—C17) = 1.317(4) Å, d(C17—C18) = 1.486(4) Å. The intermolecular connection was achieved through hydrogen bonding.

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