Ai-Zhen Weng, Hai-Yuan Quan, Liu-Yang Wang, Dong-Mei She and Xiang-Dong Mei*

Crystal structure of (2Z,2'Z)-1,1'-(pyridine-2,6-diyl) bis(3-hydroxy-3-phenylprop-2-en-1-one), $C_{23}H_{17}NO_4$

Table 1: Data collection and handling.

Crystal:	Rod-like, colorless
Size:	0.5 × 0.15 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	XtaLAB AFC12 (RINC), φ and ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	38528, 8079, 0.042
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6852
$N(\text{param})_{\text{refined}}$:	509
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SUPERFLIP [3], SHELX [4]

contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title compound was prepared according to the literature method [10–12]. Dimethyl pyridine-2,6-dicarboxylate (10 mmol), 60% sodium hydride (40 mmol) and xylene (50 mL) were stirred in a round-bottomed flask, and the mixing solution of acetophenone and xylene (40 mmol) was added dropwise at 383 K. The reaction mixture was heated to 433 K and refluxed 3 h. Then filtered and washed 3 times by petroleum ether. The filter cake was dissolved in the mixture of glacial acetic acid (25 mL) and anhydrous ethanol (25 mL), Cooling crystallization, filtering, filter cake was washed two times by a small amount of ethanol and ethyl ether without water. The product was a brown yellow solid, which was recrystallized with glycol dimethyl ether after drying. The title compound was dissolved in acetone and cyclohexane with the optimal ratio 2 : 3 at room temperature. Colourless block crystals were obtained through slow evaporation after one weeks.

Experimental details

All H atoms were placed at calculated positions. Data reduction and empirical absorption correction were performed using Olex2 [2], the crystal structure was solved by SUPERFLIP

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Abstract

$C_{23}H_{17}NO_4$, orthorhombic, $P2_12_12_1$ (no. 29), $a = 26.3525(8)$ Å, $b = 3.88640(10)$ Å, $c = 34.8871(10)$ Å, $V = 3573.01(18)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0419$, $wR_{\text{ref}}(F^2) = 0.1086$, $T = 180$ K.

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One of the two crystallographic independent molecules of the title crystal structure is shown in the figure. Tables 1 and 2

*Corresponding author: Xiang-Dong Mei, State Key Laboratory for Biology of Plant Diseases and Insect Pests, Institute of Plant Protection, Chinese Academy of Agricultural Sciences, Beijing 100193, P.R. China, e-mail: xhmei@ippcaas.cn

Ai-Zhen Weng, Hai-Yuan Quan, Liu-Yang Wang and Dong-Mei She: State Key Laboratory for Biology of Plant Diseases and Insect Pests, Institute of Plant Protection, Chinese Academy of Agricultural Sciences, Beijing 100193, P.R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.38358(7)	−0.0101(5)	0.70532(6)	0.0419(5)
H1	0.3615	0.0399	0.6898	0.063*
O2	0.34375(7)	0.2838(5)	0.64955(6)	0.0424(5)
O3	0.50446(8)	1.0387(5)	0.51324(5)	0.0446(5)
O4	0.59647(8)	1.0661(6)	0.52861(6)	0.0440(5)
H4A	0.5701	1.0805	0.5162	0.066*
N1	0.43982(8)	0.6215(5)	0.58924(6)	0.0270(4)
C1	0.47164(10)	−0.0544(6)	0.71379(7)	0.0286(5)
C2	0.52078(10)	−0.0441(7)	0.69951(8)	0.0354(6)
H2	0.5268	0.0436	0.6751	0.043*
C3	0.56103(12)	−0.1640(8)	0.72141(9)	0.0465(8)
H3	0.5938	−0.1582	0.7116	0.056*
C4	0.55239(13)	−0.2922(8)	0.75783(9)	0.0495(8)
H4	0.5794	−0.3706	0.7726	0.059*
C5	0.50392(13)	−0.3036(8)	0.77216(9)	0.0471(8)
H5	0.4982	−0.3888	0.7967	0.057*
C6	0.46361(12)	−0.1894(7)	0.75040(8)	0.0381(6)
H6	0.4308	−0.2023	0.7602	0.046*
C7	0.42796(10)	0.0657(6)	0.69116(7)	0.0281(5)
C8	0.43203(9)	0.2472(6)	0.65728(7)	0.0276(5)
H8	0.4639	0.3008	0.6475	0.033*
C9	0.38808(10)	0.3515(6)	0.63744(7)	0.0298(6)
C10	0.39283(10)	0.5412(6)	0.60045(7)	0.0283(5)
C11	0.34961(10)	0.6262(7)	0.57939(8)	0.0328(6)
H11	0.3174	0.5705	0.5883	0.039*
C12	0.35600(11)	0.7947(7)	0.54506(8)	0.0370(6)
H12	0.3280	0.8499	0.5301	0.044*
C13	0.40411(11)	0.8811(7)	0.53300(8)	0.0344(6)
H13	0.4091	0.9968	0.5100	0.041*
C14	0.44522(10)	0.7913(6)	0.55609(7)	0.0287(5)
C15	0.49784(11)	0.8883(6)	0.54561(7)	0.0311(6)
C16	0.53809(10)	0.8198(7)	0.57047(7)	0.0310(6)
H16	0.5319	0.7095	0.5937	0.037*
C17	0.58735(10)	0.9146(6)	0.56093(8)	0.0314(6)
C18	0.63208(10)	0.8511(6)	0.58547(8)	0.0316(6)
C19	0.68008(11)	0.9413(7)	0.57266(9)	0.0396(7)
H19	0.6838	1.0408	0.5486	0.047*
C20	0.72221(11)	0.8858(8)	0.59499(10)	0.0473(8)
H20	0.7541	0.9471	0.5859	0.057*
C21	0.71740(11)	0.7392(8)	0.63089(10)	0.0468(8)
H21	0.7459	0.7019	0.6460	0.056*
C22	0.66985(11)	0.6488(8)	0.64403(9)	0.0448(7)
H22	0.6664	0.5507	0.6682	0.054*
C23	0.62768(11)	0.7021(7)	0.62185(8)	0.0361(6)
H23	0.5959	0.6387	0.6310	0.043*
O5	0.84718(8)	1.6599(5)	0.46834(6)	0.0400(5)
H5A	0.8208	1.6980	0.4801	0.060*
O6	0.75474(8)	1.6774(5)	0.48313(5)	0.0416(5)
O7	0.58874(7)	0.9502(5)	0.34839(6)	0.0407(5)
O8	0.62747(7)	0.6359(6)	0.29285(6)	0.0419(5)
H8A	0.6055	0.7241	0.3063	0.063*
N2	0.68724(8)	1.2569(5)	0.40877(6)	0.0272(4)
C24	0.88082(10)	1.3758(6)	0.41426(8)	0.0301(5)
C25	0.87468(11)	1.2490(8)	0.37746(9)	0.0388(6)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H25	0.8424	1.2350	0.3669	0.047*
C26	0.91638(12)	1.1429(8)	0.35629(9)	0.0456(7)
H26	0.9121	1.0642	0.3313	0.055*
C27	0.96425(11)	1.1541(7)	0.37237(10)	0.0459(8)
H27	0.9922	1.0796	0.3584	0.055*
C28	0.97040(12)	1.2745(8)	0.40874(11)	0.0483(8)
H28	1.0027	1.2801	0.4195	0.058*
C29	0.92946(10)	1.3885(7)	0.42996(9)	0.0385(7)
H29	0.9343	1.4733	0.4546	0.046*
C30	0.83712(10)	1.4894(6)	0.43762(7)	0.0297(6)
C31	0.78690(10)	1.4121(6)	0.42775(7)	0.0292(5)
H31	0.7801	1.2939	0.4051	0.035*
C32	0.74708(10)	1.5112(6)	0.45156(7)	0.0295(5)
C33	0.69371(10)	1.4298(6)	0.44138(7)	0.0281(5)
C34	0.65336(11)	1.5372(7)	0.46450(7)	0.0321(6)
H34	0.6592	1.6510	0.4875	0.038*
C35	0.60465(10)	1.4695(7)	0.45219(8)	0.0340(6)
H35	0.5770	1.5395	0.4668	0.041*
C36	0.59734(10)	1.2989(6)	0.41838(8)	0.0325(6)
H36	0.5647	1.2548	0.4095	0.039*
C37	0.63958(9)	1.1919(6)	0.39739(7)	0.0275(5)
C38	0.63386(10)	1.0003(6)	0.36062(7)	0.0290(6)
C39	0.67671(10)	0.8793(6)	0.34132(7)	0.0279(5)
H39	0.7088	0.9227	0.3513	0.033*
C40	0.67211(10)	0.6964(6)	0.30763(7)	0.0291(5)
C41	0.71530(10)	0.5554(6)	0.28587(7)	0.0302(6)
C42	0.70754(12)	0.4356(7)	0.24875(8)	0.0389(7)
H42	0.6754	0.4466	0.2378	0.047*
C43	0.74763(14)	0.3000(8)	0.22804(10)	0.0501(8)
H43	0.7423	0.2228	0.2031	0.060*
C44	0.79499(14)	0.2788(8)	0.24396(9)	0.0500(8)
H44	0.8217	0.1854	0.2300	0.060*
C45	0.80311(12)	0.3958(8)	0.28077(9)	0.0463(7)
H45	0.8353	0.3810	0.2916	0.056*
C46	0.76365(11)	0.5350(7)	0.30168(9)	0.0378(6)
H46	0.7694	0.6154	0.3264	0.045*

program [3] and refined by full-matrix least-squares refinement with SHELX program [4].

Comment

Pyridine derivatives are known as important intermediates widely used in the synthesis of drugs [5, 6] and pesticides [7, 8]. Pyridine pesticides have the characteristics of high activity, low toxicity and high efficiency, and have good environmental compatibility with human beings and organisms, which opens up a new world for the development of chemical pesticides. Pyridine and benzene are bioisoster, which are similar in many aspects, but the significant difference between them is the hydrophobic parameters (benzene is 1.96, pyridine is 0.65). Therefore, the new compounds which are obtained by pyridine instead of benzene tend to show a

higher biological activity, low toxicity, high systemic or higher selectivity, etc.

(2*Z*,2'*Z*)-1,1'-(pyridine-2,6-diyl)-bis(3-hydroxy-3-phenyl-prop-2-en-1-one) and its derivatives are important pharmaceutical and pesticides intermediates [9–12]. For example, a new type of pyridine amide fungicides, has a unique mechanism, which was developed by Bayer company. It is mainly used for the prevention and control of oomycete diseases such as downy mildew, disease, etc., and interact with other compounds without resistance. The field test results show that foliar spray not only has a good control effect, it also has long-term effect [13].

The title molecule contains one pyridine ring and two symmetrical phenyl-ketone type moieties. In most cases, the enol structure of such compounds are unstable and transform into lower energy, more stable isomers, ketones or aldehydes. However, the addition of benzene ring to pyridine-based structures has found that benzene ring is conducive to stabilizing enol structures, while pyridine does not have this function. Bond lengths and angles in the title structure are in accord with a related one [14].

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