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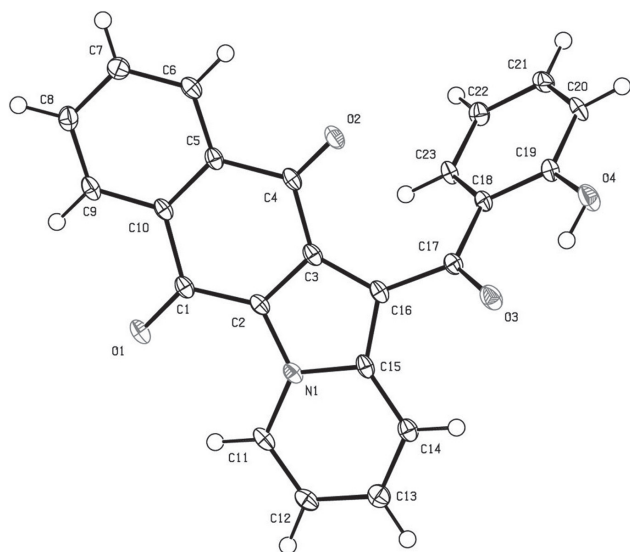


Table 1: Data collection and handling.

Crystal:	Orange lath
Size:	0.31 × 0.17 × 0.03 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	Bruker Kappa APEX-II, φ and ω
θ_{\max} , completeness:	30.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14398, 4810, 0.049
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3317
$N(\text{param})_{\text{refined}}$:	256
Programs:	Bruker [1], SHELX [2, 3]

Source of material

To a solution of 2,3-dichloronaphthalene-1,4-dione (0.5 g, 2.2 mmol) in anhydrous pyridine (50 mL), (*E*)-1-(2-hydroxyphenyl)-3-phenylprop-2-en-1-one (0.49 g, 2.2 mmol) was added and heated to 85 °C. After 24 h, the mixture was cooled to room temperature and diluted with water (250 mL) and the resulting precipitate was collected *via* filtration, dried and purified by preparative TLC (silica gel thickness = 1000 microns, 20 cm × 20 cm) using chloroform and methanol as mobile phase (20:1 *v/v*) to yield an orange solid. The solid was crystallized with a few drops of methanol in chloroform to furnish orange colored crystals. The melting point was determined as 537–538 K using a XT-4 melting point instrument (Beijing Taikē Instrument Co., Ltd, China). NMR spectra were performed on a DRX-500 Bruker NMR spectrometer (Bruker, Germany). ¹H-NMR (500 MHz, CDCl₃) δ : [ppm] 12.22 (1H, s), 9.82 (1H, d, $J = 7.0$ Hz), 8.30 (1H, d, $J = 7.5$ Hz), 8.13 (1H, d, $J = 7.6$ Hz), 7.79~7.76 (2H, m), 7.70 (1H, t, $J = 7.5$ Hz), 7.55~7.50 (2H, m), 7.39 (1H, t, $J = 7.5$ Hz), 7.21 (1H, t, $J = 6.7$ Hz), 7.13 (1H, d, $J = 8.3$ Hz), 6.79 (1H, t, $J = 7.6$ Hz); ¹³C-NMR (125 MHz, CDCl₃) δ : [ppm] 196.3, 180.9, 174.9, 162.8, 138.1, 136.7, 134.4, 133.9, 133.5, 133.2, 133.2, 128.2, 128.0, 127.4, 127.2, 126.4, 121.4, 120.8, 119.9, 118.8, 118.4, 117.6, 112.5. IR (ν_{\max} , cm⁻¹): 2953, 2920, 2851; 1662, 1638, 1619; 1578, 1496, 1466, 1430; 1388, 1366, 1312, 1289, 1254, 1220, 1189, 1146, 1108, 1062, 877, 844, 747. ESI-MS: 368.0 [M + 1]⁺; ACPI-MS: 368.0 [M + 1]⁺, 366.8 [M - 1]⁻.

Experimental details

H atoms were positioned geometrically with $d(\text{O-H}) = 0.90$ Å, $d(\text{C-H}) = 0.95$ Å (aromatic CH), and treated

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Abstract

C₂₃H₁₃NO₄, monoclinic, $P2_1/n$ (no. 14), $a = 11.6537(6)$ Å, $b = 5.1315(2)$ Å, $c = 26.8047(13)$ Å, $\beta = 96.266(3)^\circ$, $V = 1593.4(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0531$, $wR_{\text{ref}}(F^2) = 0.1432$, $T = 90.0(5)$ K.

CCDC no.: 1941465

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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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	0.56928(11)	−0.2702(2)	0.06371(5)	0.0191(3)
O2	0.39483(10)	0.5568(2)	0.15726(5)	0.0177(3)
O3	0.09611(11)	0.5444(2)	0.06004(4)	0.0197(3)
O4	−0.00752(11)	0.7681(2)	0.12774(5)	0.0218(3)
H4O	0.010(2)	0.717(4)	0.0973(10)	0.033 [*]
N1	0.33258(12)	−0.0672(2)	0.03466(5)	0.0134(3)
C1	0.52921(14)	−0.0858(3)	0.08571(6)	0.0138(3)
C2	0.41347(14)	0.0127(3)	0.07365(6)	0.0135(3)
C3	0.36254(14)	0.2088(3)	0.09978(6)	0.0139(3)
C4	0.43211(14)	0.3608(3)	0.13882(6)	0.0133(3)
C5	0.55334(14)	0.2688(3)	0.15213(6)	0.0132(3)
C6	0.62359(14)	0.4053(3)	0.18879(6)	0.0154(3)
H6	0.5926	0.5458	0.2061	0.018 [*]
C7	0.73880(15)	0.3358(3)	0.19995(6)	0.0171(3)
H7	0.7865	0.4298	0.2248	0.021 [*]
C8	0.78447(15)	0.1301(3)	0.17505(6)	0.0174(3)
H8	0.8637	0.0856	0.1823	0.021 [*]
C9	0.71445(15)	−0.0109(3)	0.13952(6)	0.0161(3)
H9	0.7455	−0.1554	0.1234	0.019 [*]
C10	0.59931(14)	0.0572(3)	0.12727(6)	0.0136(3)
C11	0.34066(15)	−0.2625(3)	−0.00030(6)	0.0153(3)
H11	0.4077	−0.3687	0.0009	0.018 [*]
C12	0.25092(15)	−0.2995(3)	−0.03641(6)	0.0166(3)
H12	0.2547	−0.4353	−0.0603	0.020 [*]
C13	0.15200(15)	−0.1385(3)	−0.03890(6)	0.0171(3)
H13	0.0912	−0.1625	−0.0651	0.021 [*]
C14	0.14328(14)	0.0519(3)	−0.00369(6)	0.0153(3)
H14	0.0769	0.1602	−0.0054	0.018 [*]
C15	0.23385(14)	0.0853(3)	0.03506(6)	0.0138(3)
C16	0.24945(14)	0.2513(3)	0.07717(6)	0.0138(3)
C17	0.15555(14)	0.4160(3)	0.09268(6)	0.0133(3)
C18	0.12771(13)	0.4131(3)	0.14494(6)	0.0127(3)
C19	0.04765(14)	0.5944(3)	0.16008(6)	0.0141(3)
C20	0.02286(14)	0.6000(3)	0.21010(6)	0.0165(3)
H20	−0.0274	0.7294	0.2208	0.020 [*]
C21	0.07135(14)	0.4179(3)	0.24362(6)	0.0159(3)
H21	0.0536	0.4215	0.2774	0.019 [*]
C22	0.14633(14)	0.2279(3)	0.22882(6)	0.0156(3)
H22	0.1778	0.1001	0.2521	0.019 [*]
C23	0.17427(14)	0.2274(3)	0.18005(6)	0.0141(3)
H23	0.2259	0.0991	0.1700	0.017 [*]

as riding atoms. For all H atoms, isotropic displacement parameters were calculated as $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Comment

The naphthofuranoquinone core which is integral part of various anticancer natural products such as Balsaminone A [4] was achieved *via* simple base-catalyzed condensation reaction between 1,4-naphthoquinone and the corresponding phenol. Intermolecular *O*-alkylation followed by intramolecular C—C-bond formation are believed to be essential steps for the formation of naphthofuranoquinone scaffold [5]. In an attempt to synthesize the naphthofuranoquinones

derived from coupling of 1,4-naphthoquinone with *trans*-2'-hydroxychalcone resulted an unusual compound instead of naphthofuranoquinone. Crystal structure analysis confirmed the formation of benzpyrido[1,2-*a*]indole-dione as the product. This intermolecular cyclization mechanism was very interesting and formation of such product with 2'-hydroxy acetophenone is reported. In order to form such fused compounds, it appears that the building block *o*-hydroxy acetophenone was generated *in situ* from *trans*-2'-hydroxychalcone *via* some kind of *retro*-aldol-like decomposition [6]. A different method of synthesizing the compound directly using substituted acetophenone react with 1,4-naphthoquinone confirmed this mechanism [7]. The title crystal structure is only built up by C₂₃H₁₃NO₄ molecules, in which all geometric parameters are normal. There is an intramolecular hydrogen bond: O4—H4O···O3 ($d(\text{O4—H4O} \cdots \text{O3}) = 2.559 \text{ \AA}$; $\alpha(\text{O4—H4O} \cdots \text{O3}) = 151^\circ$). During a database search for the molecular core scaffold, three crystal structures were found *viz.* 12-Benzoyl-2-methylbenzo[1,2-*a*]indole-6,11-dione [8]; 12-(4-Chlorobenzoyl)-2-methylbenzo[*f*]pyrido[1,2-*a*]indole-6,11-dione [7] and 12-(4-Methoxybenzoyl)-2-methyl-benzof[*f*]pyrido[1,2-*a*]indole-6,11-dione [9]. With a hydroxy group instead of methyl, methyl/chloro or methyl/methoxy group, the title crystal shows a different hydrogen bonding scheme.

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