Chen-Hong Hao, Jiao Meng, Jing Tian, Xiao-Fang Luo* and Zong-Cheng Wang*

Synthesis and crystal structure of 4-(2,4-dinitrophenoxy)benzaldehyde, $C_{13}H_8N_2O_6$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.38 × 0.17 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.13 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13473, 2562, 0.048
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1876
$N(\text{param})_{\text{refined}}$:	190
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

The molecular structure is shown in the Figure (showing 40% probability displacement ellipsoids). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Abstract

$C_{13}H_8N_2O_6$, triclinic, $P\bar{1}$ (no. 2), $a = 6.9561(5)$ Å, $b = 7.3040(5)$ Å, $c = 13.2883(8)$ Å, $\alpha = 100.988(3)^\circ$, $\beta = 95.557(3)^\circ$, $\gamma = 107.424(3)^\circ$, $V = 623.76(7)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0376$, $wR_{\text{ref}}(F^2) = 0.1008$, $T = 150(2)$ K.

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***Corresponding authors:** Xiao-Fang Luo, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan, 425199, People's Republic of China; and Hunan Engineering Technology Research Center for Comprehensive Development and Utilization of Biomass Resources, Hunan University of Science and Engineering, Yongzhou, 425199, People's Republic of China, E-mail: luoxf@huse.edu.cn; and Zong-Cheng Wang, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan, 425199, People's Republic of China; Hunan Engineering Technology Research Center for Comprehensive Development and Utilization of Biomass Resources, Hunan University of Science and Engineering, Yongzhou, 425199, People's Republic of China; and College of Chemistry and Chemical Engineering, Central South University, Changsha, 410083, People's Republic of China, E-mail: wangzongch@huse.edu.cn. <https://orcid.org/0000-0002-0723-4083>

Chen-Hong Hao, Jiao Meng and Jing Tian, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan, 425199, People's Republic of China

Source of material

4-Hydroxybenzaldehyde (122 mg, 10 mmol), anhydrous potassium carbonate (276 mg, 2 mmol), 2,4-dinitrofluorobenzene (186 mg, 10 mmol) and anhydrous dichloromethane (15 mL) were added to a 50 mL round flask. The mixture was stirred at room temperature for 2–3 h. After the reaction was complete, the reaction mixture was concentrated under reduced pressure, and the residue was purified with ethyl acetate – petroleum ether (1:2, v/v) as eluent by silica gel chromatography column to obtain a light yellow solid (195 mg, yield 68%). The product was dissolved in 80% ethanol and recrystallized to get colourless transparent blocks.

Experimental details

H atoms were included in calculated positions and refined using a riding model, with C–H distances constrained to 0.98 Å, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Comment

Thioredoxin reductase (TrxR) is one of the important targets for inhibiting tumor growth [5]. TrxR inhibitors

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.2467 (2)	1.08268 (19)	−0.11791 (11)	0.0519 (3)
O2	0.84046 (19)	1.02839 (17)	0.59963 (10)	0.0469 (3)
O3	0.6287 (2)	0.81285 (18)	0.66314 (10)	0.0479 (3)
O4	0.02244 (19)	0.32438 (17)	0.42557 (11)	0.0481 (3)
O5	0.22268 (19)	0.51835 (17)	0.19702 (9)	0.0452 (3)
O6	−0.0833 (2)	0.4776 (2)	0.31894 (11)	0.0549 (4)
N1	0.0516 (2)	0.4485 (2)	0.37286 (12)	0.0421 (4)
N2	0.6880 (2)	0.8835 (2)	0.59039 (12)	0.0407 (3)
C1	0.5691 (3)	0.7892 (2)	0.48646 (13)	0.0371 (4)
C2	0.3766 (3)	0.6564 (2)	0.47831 (13)	0.0371 (4)
H2	0.324662	0.621622	0.538031	0.044*
C3	0.2521 (2)	0.8163 (2)	−0.04020 (13)	0.0395 (4)
C4	0.2627 (2)	0.5762 (2)	0.38073 (14)	0.0374 (4)
C5	0.2698 (3)	0.5324 (2)	0.02341 (14)	0.0408 (4)
H5	0.287257	0.406349	0.012922	0.049*
C6	0.3398 (3)	0.6182 (2)	0.29140 (13)	0.0398 (4)
C7	0.2349 (3)	0.6219 (2)	0.11781 (13)	0.0393 (4)
C8	0.2109 (3)	0.8994 (2)	0.05570 (14)	0.0410 (4)
H8	0.189513	1.023713	0.066190	0.049*
C9	0.6511 (3)	0.8368 (2)	0.40003 (14)	0.0409 (4)
H9	0.784099	0.929183	0.407888	0.049*
C10	0.2012 (3)	0.8019 (2)	0.13489 (14)	0.0420 (4)
H10	0.172044	0.856929	0.200038	0.050*
C11	0.2787 (3)	0.6324 (3)	−0.05553 (14)	0.0418 (4)
H11	0.303436	0.574478	−0.121222	0.050*
C12	0.5370 (3)	0.7480 (2)	0.30267 (14)	0.0426 (4)
H12	0.593546	0.775753	0.242817	0.051*
C13	0.2704 (3)	0.9237 (3)	−0.12384 (14)	0.0446 (4)
H13	0.303247	0.863968	−0.186923	0.054*

inhibit the activity of TrxR and reduce the reduced Trx that is involved in the regulation of DNA synthesis, reduction of peroxide and apoptosis [6], thus lead to the death of tumor cells [7]. 2,4-Dinitrochlorobenzene is a reliable TrxR inhibitor [8]. In addition, some 2,4-dinitrophenyl ethers and 2,4-dinitrobenzene sulfonamides also exhibit TrxR inhibitory activity [9–11]. Therefore a series of 2,4-dinitrobenzene compounds were synthesized as TrxR inhibitors.

In the molecular structure of the title compound, there are two benzene rings. Bond lengths and angles are in the expected ranges [12]. The aldehyde group and the connected benzene ring are coplanar. At the same time, N atoms on the two nitro groups and the connected benzene ring are also coplanar, but the four O atoms on the two nitro groups are out of plane. The dihedral angle of the two benzene rings is 76.16(5)°.

The TrxR inhibitory activity of the title compound was determined by the TrxR activity detection kit. The results showed that the inhibition rate of the title compound against TrxR was 75.3(2.7) at 100 μM. Reports of the crystal

structure are helpful to understand the mechanism of these 2,4-dinitrobenzene compounds and to develop more active 2,4-dinitrobenzene TrxR inhibitors.

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