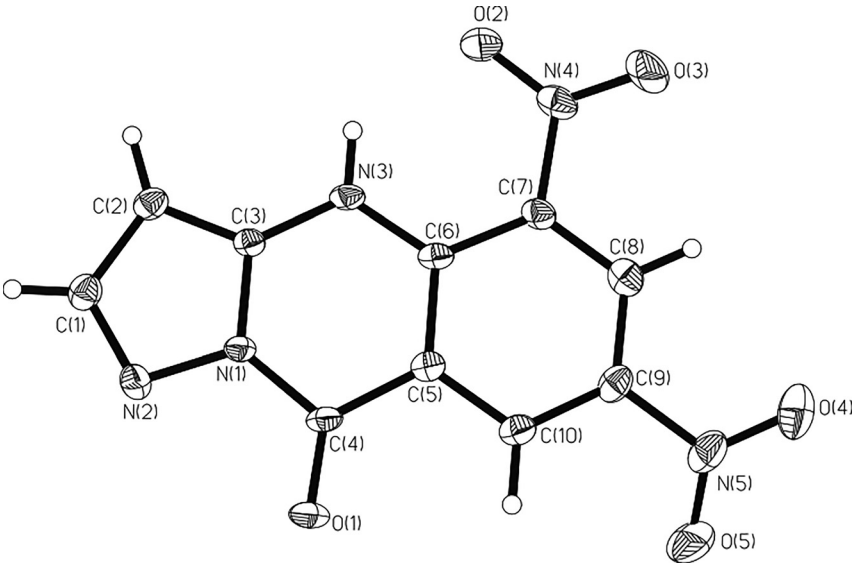
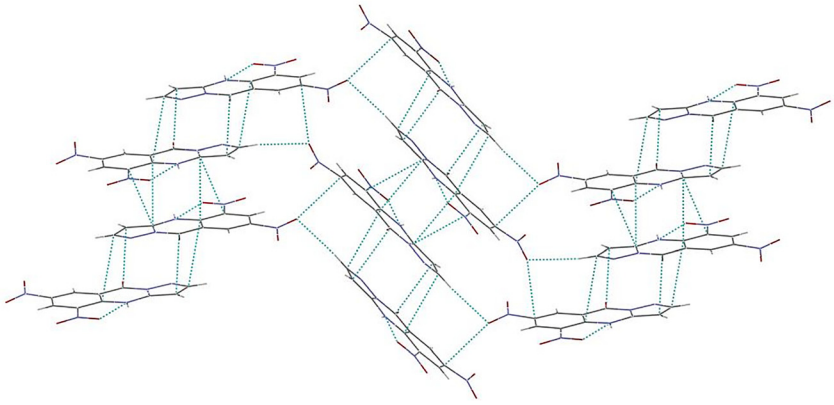
Shouwen Jin*, Qiong Zhang, Yaoqi Zhen, Chengzhe Shi and Daqi Wang

The crystal structure of 5,7-dinitropyrazolo[5,1-*b*]quinazolin-9(4*H*)-one, $C_{10}H_5N_5O_5$





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Abstract

$C_{10}H_5N_5O_5$, monoclinic, $P2_1/c$ (no. 14), a = 10.9546(11) Å, b = 7.3433(8) Å, c = 13.2567(15) Å, β = 102.972(5)°, V = 1039.19(19) Å^3, Z = 4, $R_{gt}(F)$ = 0.0630, $wR_{ref}(F^2)$ = 0.1622, T = 298 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Shouwen Jin**, Jiyang College, ZheJiang A & F University, Zhu'Ji 311800, P.R. China, E-mail: Jinsw@zafu.edu.cn. https://orcid.org/0009-0000-4846-7943

Qiong Zhang, Yaoqi Zhen and Chengzhe Shi, Jiyang College, ZheJiang A & F University, Zhu'Ji 311800, P.R. China

Daqi Wang, Department of Chemistry, Liaocheng University, Liaocheng, Shandong 252059, P.R. China

1 Source of materials

In a representative experiment 5,7-dinitropyrazolo[5,1-*b*]quinazolin-9(4*H*)-one (35.7 mg, 0.050 mmol) was dissolved in 8 mL of ethanol by heating the solution. The solution was then

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.40 × 0.27 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.15 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4720, 1822, 0.101
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1198
$N(param)_{refined}$:	181
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	−0.0591 (2)	0.1999 (3)	0.56412 (16)	0.0263 (6)
N2	−0.1695 (2)	0.1107 (4)	0.56298 (19)	0.0342 (7)
N3	0.0647 (2)	0.3139 (3)	0.45404 (17)	0.0274 (6)
H3	0.0744	0.3353	0.3925	0.033*
N4	0.2916 (2)	0.5086 (4)	0.4287 (2)	0.0373 (7)
N5	0.4350 (3)	0.5120 (5)	0.8014 (2)	0.0489 (8)
O1	0.00120 (18)	0.2210 (4)	0.73773 (15)	0.0458 (7)
O2	0.2145 (2)	0.4646 (3)	0.35048 (16)	0.0498 (7)
O3	0.3864 (2)	0.5937 (4)	0.42782 (18)	0.0565 (8)
O4	0.5207 (3)	0.6098 (4)	0.7905 (2)	0.0747 (10)
O5	0.4281 (2)	0.4410 (5)	0.8821 (2)	0.0702 (9)
C1	−0.2157 (3)	0.0822 (4)	0.4633 (2)	0.0337 (8)
H1	−0.2912	0.0225	0.4381	0.040*
C2	−0.1408 (3)	0.1497 (4)	0.3993 (2)	0.0309 (8)
H2	−0.1558	0.1449	0.3275	0.037*
C3	−0.0407 (2)	0.2244 (4)	0.4668 (2)	0.0243 (7)
C4	0.0239 (3)	0.2515 (4)	0.6550 (2)	0.0280 (7)
C5	0.1371 (2)	0.3389 (4)	0.6372 (2)	0.0255 (7)
C6	0.1542 (2)	0.3694 (4)	0.5367 (2)	0.0247 (7)
C7	0.2660 (3)	0.4597 (4)	0.5284 (2)	0.0293 (8)
C8	0.3546 (3)	0.5083 (4)	0.6143 (2)	0.0340 (8)
H8	0.4278	0.5661	0.6072	0.041*
C9	0.3357 (3)	0.4722 (5)	0.7099 (2)	0.0338 (8)
C10	0.2265 (3)	0.3925 (4)	0.7230 (2)	0.0316 (8)
H10	0.2130	0.3749	0.7891	0.038*

filtered into a test tube and left standing at room temperature. After about 20 days colorless block crystals were obtained.

2 Experimental details

Hydrogen atoms attached to the C atoms were placed in calculated positions with d(C–H) = 0.93 Å.

3 Comment

Many heterocyclic compounds containing pyrazole residues showed potential biological activities and some of them had been employed in medicine [4]. 4*H*-pyrazolo[5,1-*b*]quinazolin-9-one and its derivatives are important N-containing tricyclic compounds with unique biological properties, which have been investigated in detail [4–8].

In the title crystal structure all of the bond distances and angles are in the normal range and the molecule is flat [9, 10] (upper part of the Figure) 5,7-dinitropyrazolo[5,1-*b*] quinazolin-9(4*H*)-one create a dimer by the pi···pi contacts from the aryl moieties with C–C_g = 3.354–3.384 Å, here both molecules were inversionally related. There exists the intramolecular N–H···O hydrogen bond of 2.610(3) Å from the 4-NH and the 5-NO_2. The dimers were linked into 1D chain by the pi···pi contact from the aryl moieties of 5,7-dinitropyrazolo [5,1-*b*]quinazolin-9(4*H*)-one with C–C_g = 3.228–3.398 Å to establish 1D chain in the *b*-axis (see lower part of the Figure). The chains were connected by the CH–O contact of 3.550 Å from the pyrazole CH and one O at the 7-NO_2, and O–pi contact from the same O of the CH–O contact and the aryl kernel with O–C_g = 3.092 Å to get 2D sheet (Figure 2). The O–C_g separation was shorter than the document [9].

Here the respective 5,7-dinitropyrazolo[5,1-*b*]quinazolin-9(4*H*)- one at the adjacent chains were rotated by ca. 60° with each other. The sheets were stacked in the third direction by the N–H···O hydrogen bond of the N–H···C=O type with N–O = 2.807(3) Å, CH–O contact of 3.502 Å from the pyrazole CH and one O at the 5-NO_2, CH–O contact of 3.067 Å from the same pyrazole CH and the C=O, CH–O contact of 3.138 Å from the phenyl CH and one O at the 5-NO_2, CH–O contact of 3.104 Å from the phenyl CH and one O at the 5-NO_2, and CH–N contact of 3.435 Å from the same phenyl CH and the pyrazole ring N to yield 3D ABAB layer net (see lower part of the Figure).

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