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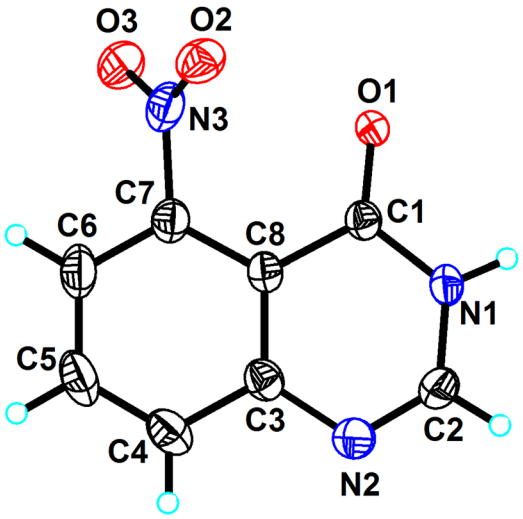
Crystal structure of 5-nitroquinazolin-4(3*H*)-one, $C_8H_5N_3O_3$



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Abstract

$C_8H_5N_3O_3$, monoclinic, $P2_1/c$ (no. 14), a = 9.1778(16) Å, b = 7.0270(10) Å, c = 12.518(2) Å, β = 92.930(6)°, V = 806.3(2) Å^3, Z = 4, $R_{gt}(F^2)$ = 0.0469, $wR_{ref}(F^2)$ = 0.1353, T = 298 K.

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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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size	0.30 × 0.20 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,349, 1649, 0.057
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1166
N(param)$_{refined}$:	127
Programs:	Bruker [1], SHELX [2], Diamond [3], OLEX2 [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.61612 (17)	0.87150 (19)	0.58046 (11)	0.0446 (4)
O2	0.9037 (2)	0.8163 (3)	0.66380 (14)	0.0623 (5)
O3	0.8060 (2)	0.6235 (3)	0.77371 (13)	0.0673 (6)
N1	0.54138 (19)	0.7828 (2)	0.41209 (13)	0.0353 (4)
H1	0.490277	0.884867	0.403954	0.042*
N2	0.6052 (2)	0.4941 (2)	0.33246 (14)	0.0417 (5)
N3	0.8388 (2)	0.6705 (3)	0.68402 (14)	0.0454 (5)
C1	0.6226 (2)	0.7593 (3)	0.50592 (14)	0.0311 (5)
C2	0.5370 (2)	0.6540 (3)	0.33114 (16)	0.0401 (5)
H2	0.480090	0.683791	0.269818	0.048*
C3	0.6944 (2)	0.4568 (3)	0.42335 (16)	0.0353 (5)
C4	0.7676 (3)	0.2811 (3)	0.4296 (2)	0.0487 (6)
H4	0.757618	0.195296	0.373151	0.058*
C5	0.8531 (3)	0.2363 (3)	0.5181 (2)	0.0566 (7)
H5	0.898377	0.117895	0.522276	0.068*
C6	0.8741 (3)	0.3638 (3)	0.60229 (19)	0.0525 (6)
H6	0.933974	0.332640	0.661903	0.063*
C7	0.8050 (2)	0.5356 (3)	0.59571 (16)	0.0380 (5)
C8	0.7109 (2)	0.5865 (3)	0.50855 (15)	0.0316 (4)

Source of material

All reagents were of analytical grade and used as purchased without further purification. A mixture of amino-6-nitrobenzoic acid (0.010 mol, 1.8210 g) and formamide (0.100 mol, 4.5020 g) in a microwave reaction vessel was heated in a microwave reactor at 140 °C for 1 h. The resulting mixture was cooled to room temperature, filtered, washed with water and dried at 50 °C for 10 h in vacuum oven to obtain the title compound of formula. Yield: 86.3%. *M*. pt: 523–527 K. ^{1}H NMR (DMSO-d6, 400 MHz): δ12.72 (*br.s*, 1H), 8.28 (*s*, 1H, H-2), 7.76 (*t*, *J* = 8.4 Hz, 1*H*), 7.41 (*d*, *J* = 7.8 Hz, 1H),

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7.32 (*d*, *J* = 8.4 Hz, 1H). The solid was recrystallized in EtOH and yellow block crystals of the title compound were obtained by slow evaporation over a period of 1 week. Elemental analysis Anal. Calcd. for $C_8H_5N_3O_3$: C, 50.27; H, 2.64; N, 21.98%. Found: C, 50.30; H, 2.63; N, 21.90%.

Experimental details

The structure was solved by Shelxt. Absorption corrections were applied by using multi-scan program [1]. All the hydrogen atoms were fixed geometrically and allowed to ride on their attached atoms with $U_{iso} = 1.2U_{eq}$ of the attached atoms.

Comment

Quinazoline is an important intermediate for drugs synthesis and its derivatives exhibit many pharmaceutical performances including antifungal [5, 6], antibacterial [7], anti-virus [8], and antimicrobial [9], antiprotozoal activities [10]. Furthermore, the compounds may possess antitumor properties that they showed potent anticancer activity at low concentrations [11–13].

The asymmetric unit of the title compound $C_8H_5N_3O_3$ is a fused aromatic molecule (see the figure). The nitro group is not co-planar with the core to which it is connected as seen in the value of the O2–N3–C7–C6 torsion angle of 62.8(3)°. The benzo moiety is not completely coplanar with the pyrimidine moiety due to the steric hindrance effect of the nitro group, showing a slight puckering conformation. The plane comprising C6, C7 and C8 makes a dihedral angle of 8.5(4)° with the plane formed by N1, C1 and C8. The C–N bond lengths fall in the range of 1.286(3) to 1.477(3) Å that are in agreement with that found in $[C_{16}H_{13}N_3O_3]$ [14]. The O2–N3–O3 bond angle is 124.3(2)°, and the C–N–C bond angles range from 116.1(2)° to 123.6(2)°, which are comparable with the values for similar compounds [15–17]. In the crystal structure, intermolecular hydrogen bonds (N1–H1···O1^1: 2.831(2) Å and 167.2°: 1 – *x*, 2 – *y*, 1 – *z*) link the adjacent molecules into centrosymmetric dimers.

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