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Synthesis and crystal structure of 2-azido-*N*-(4-nitrophenyl)acetamide, C₈H₇N₅O₃

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.44 × 0.35 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker Smart APEX, φ and ω
$\theta_{\max}$, completeness:	29.2°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18819, 2649, 0.034
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1983
$N(\text{param})_{\text{refined}}$:	145
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

C₈H₇N₅O₃, monoclinic, $P2_1/n$ (no. 14), $a = 5.0340(2)$ Å, $b = 21.1023(9)$ Å, $c = 9.2880(4)$ Å, $\beta = 94.909(1)^\circ$, $V = 983.04(7)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0470$, $wR_{\text{ref}}(F^2) = 0.1496$, $T = 298(2)$ 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.

Source of material

2-Chloro-*N*-(4-nitrophenyl)acetamide (0.011 moles) and sodium azide (0.015 moles) were dissolved in a mixture of ethanol/water (70:30) and refluxed for 24 h at 80 °C. After

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.1437(2)	0.54487(5)	0.40669(12)	0.0653(3)
O2	−0.1688(3)	0.53494(6)	0.23586(16)	0.0889(5)
O3	0.7374(3)	0.25286(5)	0.32470(11)	0.0712(4)
N1	0.0325(2)	0.51581(5)	0.30461(13)	0.0533(3)
N2	0.4732(2)	0.28701(5)	0.12922(11)	0.0444(3)
H2A	0.410723	0.276201	0.040075	0.053*
N3	0.9953(3)	0.17236(7)	0.13885(14)	0.0638(4)
N4	1.0544(2)	0.14690(6)	0.25608(13)	0.0571(3)
N5	1.1386(3)	0.12232(9)	0.35748(17)	0.0869(5)
C1	0.3655(2)	0.34342(5)	0.18049(12)	0.0395(3)
C2	0.4709(3)	0.37451(6)	0.30479(13)	0.0452(3)
H2	0.616100	0.357436	0.360242	0.054*
C3	0.3590(3)	0.43074(6)	0.34531(13)	0.0468(3)
H3	0.427305	0.451634	0.428558	0.056*
C4	0.1459(3)	0.45563(6)	0.26177(13)	0.0433(3)
C5	0.0379(3)	0.42568(7)	0.13825(15)	0.0540(3)
H5	−0.107007	0.443149	0.083261	0.065*
C6	0.1484(3)	0.36958(7)	0.09812(15)	0.0517(3)
H6	0.077740	0.348843	0.015162	0.062*
C7	0.6453(3)	0.24659(6)	0.20002(14)	0.0469(3)
C8	0.7163(3)	0.19014(7)	0.10932(16)	0.0573(4)
H8A	0.604170	0.154452	0.129897	0.069*
H8B	0.681799	0.200543	0.007717	0.069*

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cooling the title compound precipitated. The crude product was filtered off and dried. Recrystallization from ethanol yielded yellow crystals, yield: 69%.

Experimental details

H-atoms attached to carbon were placed in calculated positions while that attached to nitrogen was placed in a location derived from a difference map and its coordinates adjusted to give N—H = 0.91 Å. All were included as riding contributions.

Comment

Azides have found valuable applications in medicinal chemistry [5], molecular biology [6], and an increasing interest in the field of organic synthesis as intermediates for the preparation of heterocycles such as tetrazoles, triazolines, triazoles, etc. [7–9].

In the title molecule (see the figure), the plane of the nitro group (C4/N1/O1/O2) is inclined to that of the C1··C6 phenyl ring by 5.9(1)° while the N2/C7/O3/C8 plane is inclined by 14.8(1)°. This latter feature is likely due in part to the intramolecular C2—H2··O3 hydrogen bond and more likely by the π -system constructed by the phenyl-(NH)-(C=O) which is also seen in a directly related structure [10]. The azido group is rotated well out of this aforementioned plane as indicated by the C7—C8—N3—N4 torsion angle of 72.07(17)°. In the crystal, N2—H2A··O3 hydrogen bonds form chains extending parallel to (10-1). Weak C3—H3··O1 hydrogen bonds link the chains into zig zag layers which are associated *via* weak C6—H6··N5 hydrogen bonds.

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