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Synthesis and crystal structure of 2-azido-*N*-phenylacetamide, $C_8H_8N_4O$

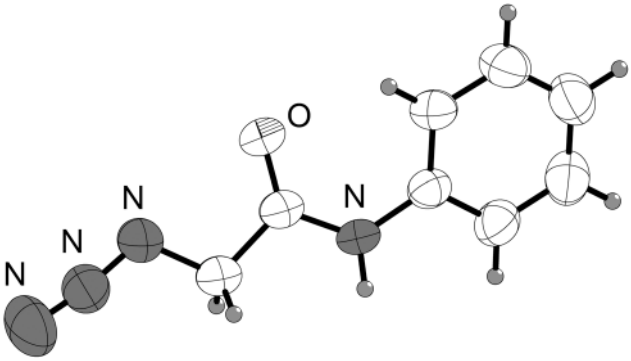

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.40 × 0.32 × 0.30 mm
Wavelength:	Cu *K*α radiation (1.54178 Å)
μ:	0.79 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE PHOTON 100, *ω*
θ_{max}, completeness:	72.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8365, 3383, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3204
$N(param)_{refined}$:	236
Programs:	Bruker [1], SHELX [2–4, 6], Diamond [5]

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Abstract

$C_8H_8N_4O$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.8085(13) Å, b = 16.533(4) Å, c = 18.171(4) Å, V = 1745.0(7) Å^3, Z = 8, $R_{gt}(F)$ = 0.0399, $wR_{ref}(F^2)$ = 0.1078, T = 296(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.

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Source of material

2–Chloro-*N*-(4-nitrophenyl)acetamide (0.006 mol) and sodium azide (0.0085 mol) were dissolved in a mixture of ethanol/water (70:30, *v:v*) then refluxed for 24 h at 80 °C. After cooling the title compound precipitated and was filtered off, dried and recrystallized from ethanol, yield: 77%. A portion of the product was dissolved in hot ethanol, the solution was filtered and the filtrate was left undisturbed for 5 days to form clear, colorless block crystals.

Experimental details

Hydrogen atoms were included as riding contributions in idealized positions. The Flack-Parsons parameter was calculated as -0.04(10) based on 1251 quotients [6].

Comment

Azides have found valuable applications in medicinal chemistry [7], molecular biology [8], and an increasing interest in the field of organic synthesis as intermediates for the preparation of heterocycles such as tetrazoles, triazolines, triazoles, etc. [9–11]. As part of our ongoing research in this area, we report the synthesis and molecular structure of the title azide.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2355 (3)	0.29894 (8)	0.23380 (8)	0.0657 (4)
N1	0.3120 (3)	0.19233 (9)	0.30918 (9)	0.0535 (4)
H1	0.272547	0.143385	0.319042	0.064*
N2	−0.1241 (5)	0.22663 (13)	0.17114 (14)	0.0871 (7)
N3	−0.2933 (4)	0.19339 (12)	0.14523 (12)	0.0707 (5)
N4	−0.4536 (5)	0.17043 (18)	0.11761 (17)	0.0993 (9)
C1	0.4922 (3)	0.22590 (11)	0.35168 (10)	0.0505 (4)
C2	0.5078 (4)	0.30827 (12)	0.36577 (12)	0.0625 (5)
H2	0.397649	0.343636	0.347233	0.075*
C3	0.6891 (5)	0.33725 (16)	0.40772 (14)	0.0754 (7)
H3	0.700620	0.392420	0.417011	0.090*
C4	0.8527 (5)	0.28540 (18)	0.43585 (12)	0.0741 (7)
H4	0.975150	0.305343	0.463358	0.089*
C5	0.8324 (5)	0.20377 (17)	0.42272 (13)	0.0729 (6)
H5	0.941466	0.168440	0.441990	0.087*
C6	0.6533 (5)	0.17367 (14)	0.38152 (11)	0.0638 (5)
H6	0.640454	0.118259	0.373706	0.077*
C7	0.1968 (3)	0.22964 (11)	0.25490 (10)	0.0510 (4)
C8	0.0165 (4)	0.17728 (12)	0.21914 (12)	0.0617 (5)
H8A	0.089937	0.134667	0.190952	0.074*
H8B	−0.079095	0.152361	0.256584	0.074*
O2	0.6474 (3)	0.46627 (9)	0.63830 (10)	0.0737 (5)
N5	0.4595 (3)	0.58698 (10)	0.62794 (10)	0.0587 (4)
H5A	0.457556	0.635003	0.646123	0.070*
N6	0.8264 (4)	0.50883 (14)	0.77325 (14)	0.0826 (7)
N7	1.0343 (4)	0.50238 (11)	0.76906 (11)	0.0679 (5)
N8	1.2245 (6)	0.4922 (2)	0.7732 (2)	0.1183 (12)
C9	0.3063 (4)	0.57312 (12)	0.56913 (11)	0.0561 (5)
C10	0.3275 (6)	0.51010 (14)	0.52004 (13)	0.0753 (7)
H10	0.450885	0.474455	0.523344	0.090*
C11	0.1625 (7)	0.50050 (18)	0.46569 (16)	0.0985 (11)
H11	0.174874	0.457034	0.433444	0.118*
C12	−0.0139 (7)	0.5518 (2)	0.45813 (16)	0.0967 (10)
H12	−0.122525	0.543667	0.421252	0.116*
C13	−0.0341 (6)	0.6168 (2)	0.50528 (16)	0.0950 (9)
H13	−0.154569	0.653305	0.499807	0.114*
C14	0.1267 (5)	0.62718 (18)	0.56094 (13)	0.0778 (7)
H14	0.113588	0.670847	0.592930	0.093*
C15	0.6087 (4)	0.53509 (12)	0.65948 (12)	0.0565 (5)
C16	0.7281 (5)	0.57151 (14)	0.72602 (15)	0.0742 (7)
H16A	0.849637	0.607510	0.709639	0.089*
H16B	0.618243	0.603172	0.754047	0.089*

The asymmetric unit consists of two independent molecules differing primarily in the orientation of the azide moiety. Thus in the molecule containing O1, the dihedral angle between the mean planes defined by N1/C7/C8/O1 and C8/N2/N3/N4 is 10.9(2)° (see the Figure), while the corresponding angle in the molecule containing O2 is 80.9(2)°. Also, in the molecule containing O1, the dihedral angle between the mean planes defined by C1···C6 and N1/C7/C8/O1 is 28.4(1)°, while the corresponding angle in the other molecule is 16.3(2)°. In the crystal, the two independent molecules are linked in alternating fashion by N1–H1···O2 and N5–H5A···O1 hydrogen bonds into helical chains extending along the *b*-axis direction (Table 2). The chains are joined into layers parallel to the *bc* plane by π interactions between the N3/N4 moiety of the azide substituent and the C1···C6 ring at $-x, y-1/2, -z+1/2$ (N4···centroid = 3.462(3) Å, N3···centroid = 3.695(3) Å, N3/N4···centroid = 93.0(2)°.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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