

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-(2-Nitrophenyl)acetohydrazide

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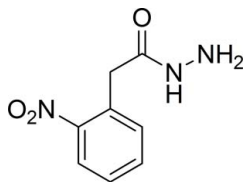
Received 12 November 2012; accepted 18 November 2012

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.038; wR factor = 0.096; data-to-parameter ratio = 14.5.

In the title compound, $\text{C}_8\text{H}_9\text{N}_3\text{O}_3$, the dihedral angle between the benzene ring and the acetohydrazide $\text{C}-\text{C}(=\text{O})-\text{N}-\text{N}$ plane [maximum deviation = 0.0471 (13) Å] is 87.62 (8)°. The nitro group is twisted by 19.3 (2)° with respect to the benzene ring. In the crystal, $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a double-column structure along the b axis.

Related literature

For the chemistry of hydrazides, see: Domiano *et al.* (1984). For the biological properties of hydrazides, see: Kalsi *et al.* (2006); Masunari & Tavares (2007); Singh *et al.* (1992). For related structures, see: Ahmad *et al.* (2012); Dutkiewicz *et al.* (2009); Liu & Gao (2012). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_8\text{H}_9\text{N}_3\text{O}_3$
 $M_r = 195.18$
 Monoclinic, $P2_1$
 $a = 6.6962$ (5) Å
 $b = 4.9388$ (4) Å
 $c = 13.3593$ (12) Å
 $\beta = 92.361$ (8)°

$V = 441.43$ (6) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 0.98$ mm⁻¹
 $T = 173$ K
 $0.36 \times 0.28 \times 0.08$ mm

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini) diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
 $T_{\min} = 0.667$, $T_{\max} = 0.925$

3829 measured reflections
 1967 independent reflections
 1824 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.096$
 $S = 1.05$
 1967 reflections
 136 parameters
 4 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³
 Absolute structure: Flack (1983), 836 Friedel pairs
 Flack parameter: 0.3 (3)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{i}}$	0.90 (1)	2.21 (2)	3.0752 (19)	163 (2)
$\text{N2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.85 (2)	2.03 (2)	2.8531 (18)	165 (2)

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + 1$; (ii) $x, y - 1, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

ASP thanks UOM for research facilities. JPJ acknowledges the NSF-MRI program (grant No. CHE1039027) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5219).

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supporting information

Acta Cryst. (2012). E68, o3436 [doi:10.1107/S1600536812047381]

2-(2-Nitrophenyl)acetohydrazide

A. S. Praveen, Jerry P. Jasinski, Amanda C. Keeley, H. S. Yathirajan and B. Narayana

S1. Comment

The chemistry of hydrazides has been intensely investigated in recent years due to their excellent coordinating capability (Domiano *et al.*, 1984). Hydrazides and their condensation products have displayed diverse range of biological properties such as anti-helminthic (Kalsi *et al.*, 2006), anti-leprotic (Masunari & Tavares, 2007) and anti-depressant (Singh *et al.*, 1992). The crystal structures of some hydrazides, viz., 2-(4-bromophenyl)acetohydrazide (Ahmad *et al.*, 2012), 2-(4-chlorophenoxy)acetohydrazide (Dutkiewicz *et al.*, 2009) and 2-(4-methoxyphenoxy)acetohydrazide (Liu & Gao, 2012) have been reported. In view of the importance of hydrazides, the crystal structure of title compound (I) is reported.

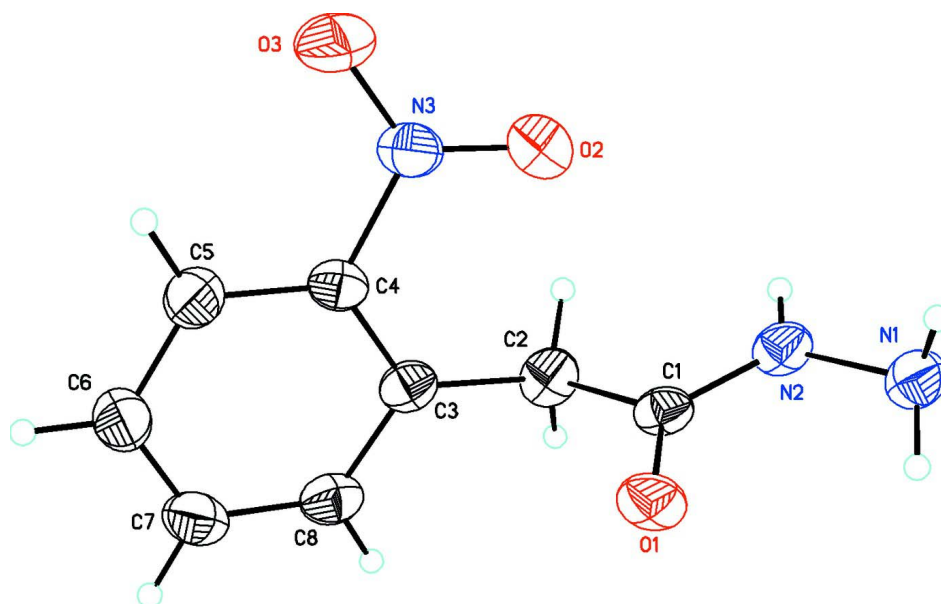
In the title compound, the dihedral angle between the benzene ring and acetohydrazide C2/C1/O1/N2/N1 plane is 87.62 (8)° (Fig. 1). The nitro group is twisted by 19.3 (2)° with the benzene ring. Bond lengths are in normal ranges (Allen *et al.*, 1987). In the crystal, N—H···O hydrogen bonds (Table 1) link the molecules into a double-column structure along the *b* axis (Fig. 2).

S2. Experimental

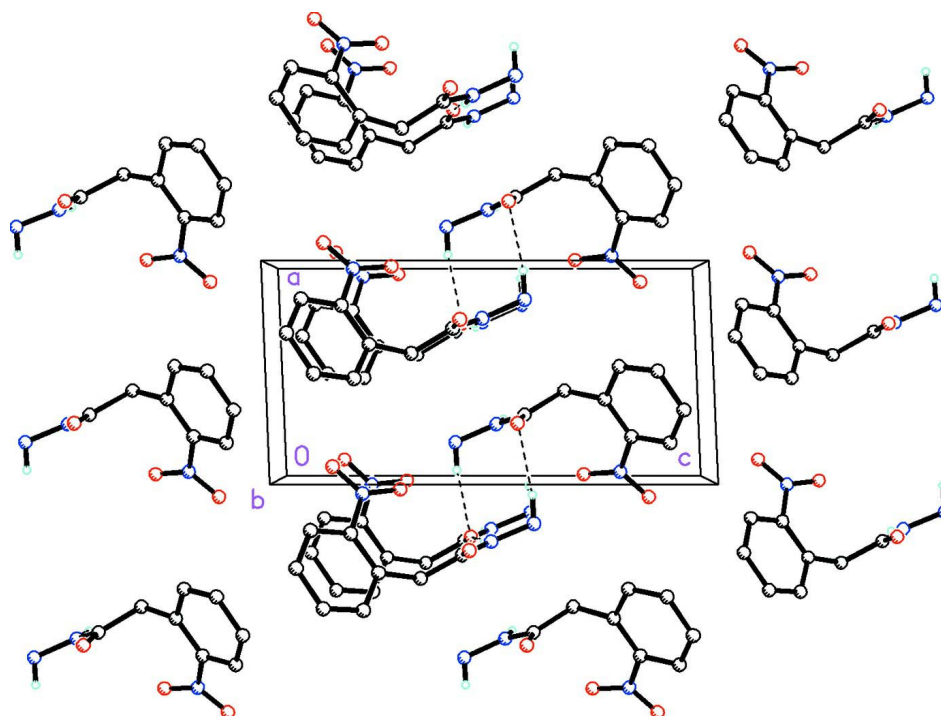
To a solution of methyl 2-(2-nitrophenyl)acetate (2 g, 10.14 mmol) in methanol (20 mL), hydrazine hydrate (2 mL) was added and the reaction mixture was stirred at room temperature for 8 hours (Fig. 3). After the completion of the reaction, methanol was removed under vacuum, water was added, precipitated solid was filtered and dried. The single crystal was grown from mixture methanol: water (2:1) by slow evaporation method and yield of the compound was 95%. (m.p.: 422-424 K).

S3. Refinement

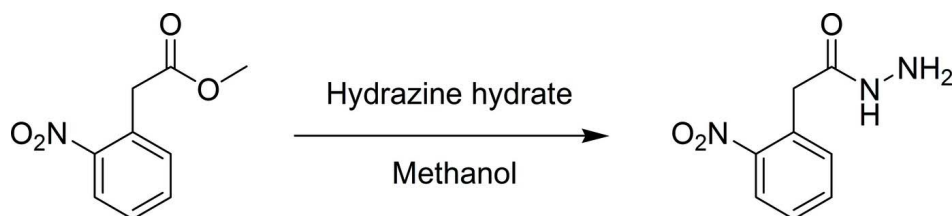
Atoms H1A, H1B and H2 were refined with a bond-length restraint N—H = 0.86 (2) Å. All remaining H atoms were placed in their calculated positions and then refined using the riding model with C—H lengths of 0.93 Å (CH) and 0.97 Å (CH₂). Isotropic displacement parameters were set to 1.2 times U_{eq} of the parent atom. The Flack parameter 0.3 (3) and the Hooft γ parameter of 0.45 (18) imply that the crystal used was an inversion twin.

**Figure 1**

Molecular structure of the title compound showing the atom labeling scheme and 50% probability displacement ellipsoids.

**Figure 2**

Packing diagram of the title compound viewed along the *b* axis. Dashed lines indicate N—H...O hydrogen bonds. H atoms not involved in the hydrogen bonds have been removed for clarity.

**Figure 3**

Synthesis of the title compound.

2-(2-Nitrophenyl)acetohydrazide*Crystal data* $C_8H_9N_3O_3$ $M_r = 195.18$ Monoclinic, $P2_1$

Hall symbol: P 2yb

 $a = 6.6962$ (5) Å $b = 4.9388$ (4) Å $c = 13.3593$ (12) Å $\beta = 92.361$ (8)° $V = 441.43$ (6) Å³ $Z = 2$ $F(000) = 204$ $D_x = 1.468$ Mg m⁻³Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 1694 reflections

 $\theta = 3.3\text{--}32.5^\circ$ $\mu = 0.98$ mm⁻¹ $T = 173$ K

Chunk, colorless

 $0.36 \times 0.28 \times 0.08$ mm*Data collection*Oxford Diffraction Xcalibur (Eos, Gemini)
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.0416 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2010)

 $T_{\min} = 0.667$, $T_{\max} = 0.925$

3829 measured reflections

1967 independent reflections

1824 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$ $\theta_{\max} = 89.1^\circ$, $\theta_{\min} = 7.3^\circ$ $h = -8 \rightarrow 8$ $k = -6 \rightarrow 6$ $l = -17 \rightarrow 17$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.096$ $S = 1.05$

1967 reflections

136 parameters

4 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.016P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.19$ e Å⁻³ $\Delta\rho_{\min} = -0.17$ e Å⁻³Absolute structure: Flack (1983), 836 Friedel
pairs

Absolute structure parameter: 0.3 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.27090 (17)	0.8745 (2)	0.55928 (9)	0.0360 (3)
O2	0.0246 (2)	0.4300 (4)	0.71073 (10)	0.0632 (5)
O3	-0.0790 (2)	0.4790 (4)	0.85895 (11)	0.0576 (4)
N1	0.1751 (2)	0.4904 (3)	0.41437 (10)	0.0360 (3)
H1A	0.202 (3)	0.660 (4)	0.4013 (15)	0.043*
H1B	0.042 (2)	0.494 (5)	0.4186 (13)	0.043*
N2	0.2580 (2)	0.4374 (3)	0.51138 (10)	0.0318 (3)
H2	0.286 (3)	0.275 (4)	0.5269 (13)	0.038*
N3	0.0398 (2)	0.5265 (3)	0.79401 (10)	0.0358 (4)
C1	0.3052 (2)	0.6316 (3)	0.57676 (12)	0.0274 (3)
C2	0.4158 (2)	0.5384 (4)	0.67189 (12)	0.0338 (4)
H2A	0.3753	0.3547	0.6868	0.041*
H2B	0.5580	0.5363	0.6609	0.041*
C3	0.3777 (2)	0.7162 (3)	0.76100 (11)	0.0290 (3)
C4	0.2066 (2)	0.7141 (3)	0.81839 (11)	0.0293 (3)
C5	0.1834 (2)	0.8801 (4)	0.90089 (12)	0.0358 (4)
H5	0.0682	0.8696	0.9372	0.043*
C6	0.3327 (3)	1.0607 (4)	0.92857 (13)	0.0391 (4)
H6	0.3182	1.1750	0.9831	0.047*
C7	0.5050 (3)	1.0703 (4)	0.87408 (13)	0.0392 (4)
H7	0.6064	1.1916	0.8922	0.047*
C8	0.5260 (2)	0.9000 (4)	0.79295 (12)	0.0338 (4)
H8	0.6435	0.9080	0.7583	0.041*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0441 (6)	0.0188 (6)	0.0449 (7)	0.0021 (5)	-0.0010 (5)	0.0046 (5)
O2	0.0697 (9)	0.0736 (12)	0.0466 (8)	-0.0375 (9)	0.0085 (6)	-0.0133 (8)
O3	0.0440 (7)	0.0653 (11)	0.0651 (9)	-0.0193 (7)	0.0216 (6)	-0.0069 (8)
N1	0.0431 (7)	0.0306 (8)	0.0346 (7)	-0.0004 (7)	0.0062 (6)	0.0013 (6)
N2	0.0423 (7)	0.0197 (7)	0.0337 (7)	0.0046 (6)	0.0070 (5)	0.0036 (6)
N3	0.0339 (7)	0.0332 (9)	0.0405 (8)	-0.0061 (6)	0.0037 (6)	0.0013 (6)
C1	0.0296 (7)	0.0197 (8)	0.0336 (8)	0.0031 (6)	0.0087 (6)	0.0028 (6)
C2	0.0387 (8)	0.0261 (9)	0.0369 (9)	0.0096 (7)	0.0048 (6)	0.0034 (7)
C3	0.0314 (7)	0.0254 (8)	0.0303 (7)	0.0034 (6)	0.0006 (6)	0.0079 (7)
C4	0.0288 (7)	0.0242 (8)	0.0347 (8)	-0.0011 (6)	0.0007 (6)	0.0043 (7)
C5	0.0385 (8)	0.0353 (10)	0.0338 (8)	-0.0005 (8)	0.0048 (6)	0.0011 (7)
C6	0.0509 (10)	0.0333 (10)	0.0330 (9)	-0.0026 (8)	-0.0003 (7)	-0.0012 (7)
C7	0.0432 (9)	0.0332 (10)	0.0404 (9)	-0.0089 (8)	-0.0076 (7)	0.0077 (8)

C8	0.0300 (7)	0.0351 (10)	0.0362 (8)	-0.0023 (7)	0.0003 (6)	0.0095 (7)
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Geometric parameters (Å, °)

O1—C1	1.242 (2)	C2—H2B	0.9700
O2—N3	1.2107 (19)	C3—C8	1.399 (2)
O3—N3	1.2236 (18)	C3—C4	1.405 (2)
N1—N2	1.413 (2)	C4—C5	1.387 (2)
N1—H1A	0.877 (16)	C5—C6	1.379 (3)
N1—H1B	0.898 (14)	C5—H5	0.9300
N2—C1	1.327 (2)	C6—C7	1.390 (3)
N2—H2	0.845 (16)	C6—H6	0.9300
N3—C4	1.477 (2)	C7—C8	1.384 (3)
C1—C2	1.516 (2)	C7—H7	0.9300
C2—C3	1.509 (2)	C8—H8	0.9300
C2—H2A	0.9700		
N2—N1—H1A	106.5 (14)	C8—C3—C4	115.04 (15)
N2—N1—H1B	107.5 (12)	C8—C3—C2	118.50 (14)
H1A—N1—H1B	102 (2)	C4—C3—C2	126.45 (15)
C1—N2—N1	122.93 (15)	C5—C4—C3	123.33 (15)
C1—N2—H2	118.6 (13)	C5—C4—N3	115.94 (13)
N1—N2—H2	118.3 (13)	C3—C4—N3	120.72 (14)
O2—N3—O3	122.93 (16)	C6—C5—C4	119.47 (15)
O2—N3—C4	118.92 (13)	C6—C5—H5	120.3
O3—N3—C4	118.13 (14)	C4—C5—H5	120.3
O1—C1—N2	122.50 (16)	C5—C6—C7	119.32 (16)
O1—C1—C2	122.07 (16)	C5—C6—H6	120.3
N2—C1—C2	115.32 (15)	C7—C6—H6	120.3
C3—C2—C1	113.10 (14)	C8—C7—C6	120.18 (17)
C3—C2—H2A	109.0	C8—C7—H7	119.9
C1—C2—H2A	109.0	C6—C7—H7	119.9
C3—C2—H2B	109.0	C7—C8—C3	122.65 (15)
C1—C2—H2B	109.0	C7—C8—H8	118.7
H2A—C2—H2B	107.8	C3—C8—H8	118.7
N1—N2—C1—O1	3.6 (2)	O3—N3—C4—C5	-18.1 (2)
N1—N2—C1—C2	-172.72 (13)	O2—N3—C4—C3	-20.3 (2)
O1—C1—C2—C3	32.4 (2)	O3—N3—C4—C3	160.87 (17)
N2—C1—C2—C3	-151.21 (14)	C3—C4—C5—C6	1.0 (2)
C1—C2—C3—C8	-103.44 (17)	N3—C4—C5—C6	179.95 (15)
C1—C2—C3—C4	77.9 (2)	C4—C5—C6—C7	-0.9 (3)
C8—C3—C4—C5	0.0 (2)	C5—C6—C7—C8	0.0 (3)
C2—C3—C4—C5	178.63 (16)	C6—C7—C8—C3	1.0 (3)
C8—C3—C4—N3	-178.96 (14)	C4—C3—C8—C7	-1.0 (2)
C2—C3—C4—N3	-0.3 (2)	C2—C3—C8—C7	-179.73 (16)
O2—N3—C4—C5	160.74 (17)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1B \cdots O1 ⁱ	0.90 (1)	2.21 (2)	3.0752 (19)	163 (2)
N2—H2 \cdots O1 ⁱⁱ	0.85 (2)	2.03 (2)	2.8531 (18)	165 (2)

Symmetry codes: (i) $-x, y-1/2, -z+1$; (ii) $x, y-1, z$.