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3-Nitro-1*H*-1,2,4-triazole

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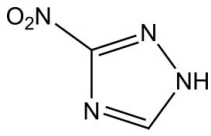
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(N–C) = 0.001 Å; R factor = 0.035; wR factor = 0.092; data-to-parameter ratio = 20.1.

The asymmetric unit of the title compound, $C_2H_2N_4O_2$, contains two crystallographically independent molecules in which the triazole rings are essentially planar, with maximum deviations of 0.003 (1) Å in both molecules. The dihedral angle between the two 1*H*-1,2,4-triazole rings is 56.58 (5)°. In the crystal, molecules are linked *via* intermolecular N—H···N and C—H···O hydrogen bonds, forming a supramolecular chain along the *b* axis.

Related literature

For details and applications of 1*H*-1,2,4-triazole derivatives, see: Desenko (1995); Vos *et al.* (1983); van Albada *et al.* (1984); Al-Kharafi *et al.* (1986); Gupta & Bhargava (1978); Jones *et al.* (1965); Bennur *et al.* (1976). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_2H_2N_4O_2$
M_r = 114.08
Monoclinic, $P2_1/c$
a = 8.7818 (1) Å
b = 10.0726 (2) Å
c = 9.9703 (1) Å
β = 107.081 (1)°
V = 843.03 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 100 K
0.48 × 0.33 × 0.30 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.928, T_{max} = 0.954
11450 measured reflections
3081 independent reflections
2768 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.05
3081 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2A—H1N1···N1A^{i}	0.885 (15)	1.995 (15)	2.8540 (9)	163.4 (15)
N2B—H1N2···N1B^{ii}	0.857 (16)	2.057 (16)	2.9128 (10)	176.0 (16)
C1A—H1AA···O2A^{iii}	0.93	2.50	3.1129 (10)	124
C1B—H1BA···O2B^{ii}	0.93	2.51	3.0451 (11)	117

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2634).

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3-Nitro-1*H*-1,2,4-triazole

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S1. Comment

1*H*-1,2,4-Triazole ring systems are typical planar six-π-electron partially aromatic systems, and are used, along with their derivatives, as starting materials for the synthesis of many heterocycles (Desenko, 1995). Substituted 1*H*-1,2,4-triazoles have also been actively studied as bridging ligands coordinating through their vicinal N atoms and some have special structures with interesting magnetic properties (Vos *et al.*, 1983; van Albada *et al.*, 1984). Studies also indicate that the 1*H*-1,2,4-triazole system is associated with anticorrosion (Al-Kharafi *et al.*, 1986) and anti-inflammatory action (Gupta & Bhargava, 1978) and other pharmacological activities by exhibiting antiviral, anti-asthmatic, diuretic, analgesic, antimicrobial, antidepressant and antifungal effects (Jones *et al.*, 1965; Bennur *et al.*, 1976).

The asymmetric unit of the title compound consists of two crystallographically independent 3-nitro-1*H*-1,2,4-triazole molecules (A & B) with very similar geometry (Fig. 1). The 1*H*-1,2,4-triazole units are essentially planar with maximum deviations of 0.003 (1) Å for atom N1A (molecule A) and 0.003 (1) Å for atom C2B (molecule B). The dihedral angle between the two 1*H*-1,2,4-triazole (N1A—N3A/ C1A–C2A) and (N1B—N3B/C1B–C2B) rings is 56.58 (5)°.

In the crystal structure (Fig. 2), molecules are connected *via* N2A—H1N1···N1A, N2B—H1N2···N1B, C1A—H1AA···O2A and C1B—H1BA···O2B (Table 1) hydrogen bonds to form a one-dimensional supramolecular chain along the *b*-axis.

S2. Experimental

Hot methanol solution (20 ml) of 3-nitro-1*H*-1,2,4-triazole (57 mg, Aldrich) was warmed over a heating magnetic stirrer for 5 minutes. The resulting solution was allowed to cool slowly at room temperature. Crystals of the title compound appeared from the mother liquor after a few days.

S3. Refinement

Atoms H1N1 and H1N2 were located from a difference Fourier map and refined freely [refined N—H distances 0.857 (16) and 0.885 (15) Å]. The remaining H atoms were positioned geometrically [C—H = 0.93 Å] and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C).

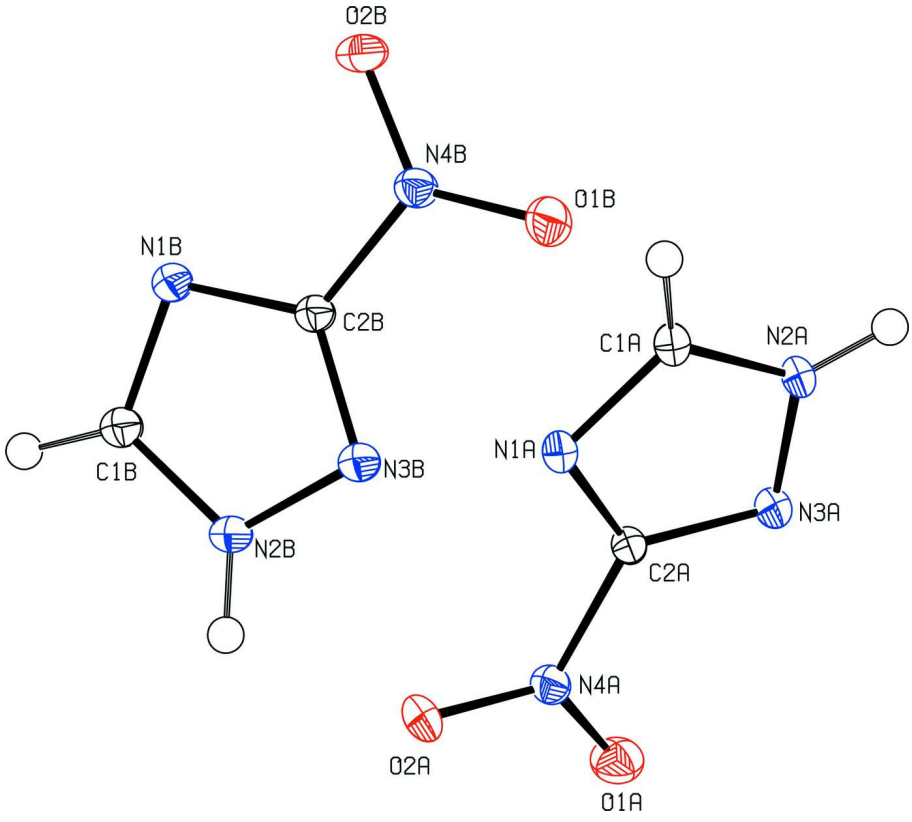

Figure 1

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

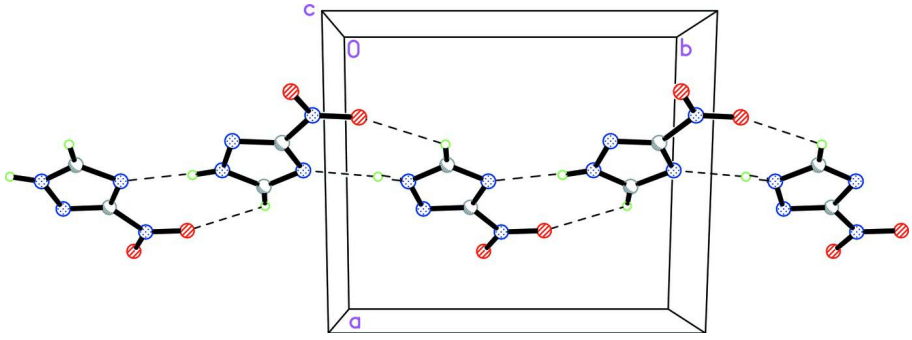

Figure 2

The crystal packing of the title compound, showing a hydrogen-bonded (dashed lines) molecular chain.

3-Nitro-1*H*-1,2,4-triazole

Crystal data

$C_2H_2N_4O_2$
M_r = 114.08
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.7818 (1) Å
b = 10.0726 (2) Å
c = 9.9703 (1) Å
β = 107.081 (1)°
V = 843.03 (2) Å^3
Z = 8
$F(000)$ = 464
D_x = 1.798 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7180 reflections
θ = 2.9–32.6°
μ = 0.16 mm^{-1}

T = 100 K
Block, colourless

0.48 × 0.33 × 0.30 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.928, T_{max} = 0.954

11450 measured reflections
3081 independent reflections
2768 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 32.7°, θ_{min} = 2.9°
h = −13→11
k = −15→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.05
3081 reflections
153 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.2412P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s 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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1A	0.73082 (8)	−0.04055 (7)	0.51717 (7)	0.01914 (13)
O2A	0.85716 (9)	0.01680 (7)	0.73151 (6)	0.01888 (14)
N1A	1.01266 (8)	0.21389 (7)	0.64087 (7)	0.01307 (13)
N2A	0.99857 (9)	0.24376 (7)	0.42001 (7)	0.01273 (13)
N3A	0.89747 (8)	0.14183 (7)	0.41773 (7)	0.01286 (13)
N4A	0.82618 (9)	0.02813 (7)	0.60358 (7)	0.01348 (13)
C1A	1.06543 (10)	0.28585 (8)	0.55150 (8)	0.01356 (14)
H1AA	1.1381	0.3552	0.5768	0.016*
C2A	0.91199 (9)	0.13008 (8)	0.55271 (8)	0.01167 (14)
O1B	0.75840 (8)	0.41600 (7)	0.50676 (7)	0.02046 (14)
O2B	0.68377 (9)	0.58439 (6)	0.60867 (7)	0.01985 (14)

N1B	0.51771 (8)	0.42579 (7)	0.73353 (7)	0.01312 (13)
N2B	0.51813 (9)	0.20833 (7)	0.72132 (7)	0.01419 (13)
N3B	0.60998 (9)	0.24714 (7)	0.64058 (7)	0.01375 (13)
N4B	0.68913 (8)	0.46461 (7)	0.58504 (7)	0.01361 (13)
C1B	0.46484 (10)	0.31423 (8)	0.77581 (8)	0.01436 (15)
H1BA	0.4002	0.3102	0.8347	0.017*
C2B	0.60451 (9)	0.37710 (8)	0.65365 (8)	0.01189 (14)
H1N1	1.0120 (19)	0.2722 (16)	0.3403 (16)	0.034 (4)*
H1N2	0.5028 (18)	0.1259 (16)	0.7343 (15)	0.030 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0195 (3)	0.0170 (3)	0.0203 (3)	−0.0044 (2)	0.0049 (2)	−0.0008 (2)
O2A	0.0278 (3)	0.0179 (3)	0.0136 (3)	0.0007 (2)	0.0102 (2)	0.0037 (2)
N1A	0.0164 (3)	0.0135 (3)	0.0103 (3)	−0.0005 (2)	0.0055 (2)	−0.0003 (2)
N2A	0.0164 (3)	0.0133 (3)	0.0099 (3)	0.0001 (2)	0.0060 (2)	0.0011 (2)
N3A	0.0156 (3)	0.0128 (3)	0.0107 (3)	0.0004 (2)	0.0048 (2)	0.0009 (2)
N4A	0.0161 (3)	0.0118 (3)	0.0144 (3)	0.0020 (2)	0.0072 (2)	0.0018 (2)
C1A	0.0163 (3)	0.0142 (3)	0.0115 (3)	−0.0007 (3)	0.0060 (3)	−0.0005 (2)
C2A	0.0146 (3)	0.0109 (3)	0.0107 (3)	0.0013 (2)	0.0056 (2)	0.0010 (2)
O1B	0.0240 (3)	0.0185 (3)	0.0245 (3)	0.0046 (2)	0.0158 (3)	0.0025 (2)
O2B	0.0264 (3)	0.0106 (3)	0.0260 (3)	−0.0006 (2)	0.0131 (3)	0.0004 (2)
N1B	0.0151 (3)	0.0111 (3)	0.0143 (3)	0.0010 (2)	0.0062 (2)	0.0004 (2)
N2B	0.0178 (3)	0.0101 (3)	0.0161 (3)	−0.0002 (2)	0.0072 (2)	0.0006 (2)
N3B	0.0163 (3)	0.0111 (3)	0.0149 (3)	0.0009 (2)	0.0062 (2)	0.0007 (2)
N4B	0.0144 (3)	0.0119 (3)	0.0153 (3)	0.0015 (2)	0.0055 (2)	0.0018 (2)
C1B	0.0166 (3)	0.0119 (3)	0.0160 (3)	0.0006 (3)	0.0070 (3)	0.0005 (2)
C2B	0.0128 (3)	0.0103 (3)	0.0127 (3)	0.0007 (2)	0.0041 (2)	0.0007 (2)

Geometric parameters (Å, º)

O1A—N4A	1.2241 (10)	O1B—N4B	1.2239 (9)
O2A—N4A	1.2289 (9)	O2B—N4B	1.2329 (9)
N1A—C1A	1.3329 (10)	N1B—C1B	1.3307 (10)
N1A—C2A	1.3455 (10)	N1B—C2B	1.3462 (10)
N2A—C1A	1.3383 (10)	N2B—C1B	1.3421 (10)
N2A—N3A	1.3531 (10)	N2B—N3B	1.3539 (9)
N2A—H1N1	0.885 (15)	N2B—H1N2	0.857 (16)
N3A—C2A	1.3194 (9)	N3B—C2B	1.3178 (10)
N4A—C2A	1.4506 (10)	N4B—C2B	1.4476 (10)
C1A—H1AA	0.9300	C1B—H1BA	0.9300
C1A—N1A—C2A	101.26 (6)	C1B—N1B—C2B	100.99 (7)
C1A—N2A—N3A	110.72 (6)	C1B—N2B—N3B	110.52 (7)
C1A—N2A—H1N1	129.9 (10)	C1B—N2B—H1N2	128.3 (10)
N3A—N2A—H1N1	119.4 (10)	N3B—N2B—H1N2	121.2 (10)
C2A—N3A—N2A	100.64 (6)	C2B—N3B—N2B	100.52 (6)

O1A—N4A—O2A	125.11 (7)	O1B—N4B—O2B	124.56 (7)
O1A—N4A—C2A	118.18 (6)	O1B—N4B—C2B	118.56 (7)
O2A—N4A—C2A	116.70 (7)	O2B—N4B—C2B	116.86 (6)
N1A—C1A—N2A	110.12 (7)	N1B—C1B—N2B	110.33 (7)
N1A—C1A—H1AA	124.9	N1B—C1B—H1BA	124.8
N2A—C1A—H1AA	124.9	N2B—C1B—H1BA	124.8
N3A—C2A—N1A	117.27 (7)	N3B—C2B—N1B	117.63 (7)
N3A—C2A—N4A	121.04 (7)	N3B—C2B—N4B	121.29 (7)
N1A—C2A—N4A	121.66 (6)	N1B—C2B—N4B	121.08 (7)
C1A—N2A—N3A—C2A	−0.05 (8)	C1B—N2B—N3B—C2B	0.10 (9)
C2A—N1A—C1A—N2A	−0.45 (9)	C2B—N1B—C1B—N2B	−0.54 (9)
N3A—N2A—C1A—N1A	0.33 (10)	N3B—N2B—C1B—N1B	0.30 (10)
N2A—N3A—C2A—N1A	−0.27 (9)	N2B—N3B—C2B—N1B	−0.49 (9)
N2A—N3A—C2A—N4A	−178.46 (7)	N2B—N3B—C2B—N4B	179.31 (7)
C1A—N1A—C2A—N3A	0.46 (9)	C1B—N1B—C2B—N3B	0.66 (9)
C1A—N1A—C2A—N4A	178.64 (7)	C1B—N1B—C2B—N4B	−179.14 (7)
O1A—N4A—C2A—N3A	−5.31 (11)	O1B—N4B—C2B—N3B	4.58 (12)
O2A—N4A—C2A—N3A	173.84 (7)	O2B—N4B—C2B—N3B	−176.50 (8)
O1A—N4A—C2A—N1A	176.57 (7)	O1B—N4B—C2B—N1B	−175.62 (8)
O2A—N4A—C2A—N1A	−4.27 (11)	O2B—N4B—C2B—N1B	3.29 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2*A*—H1*N*1···N1*A*^{i}	0.885 (15)	1.995 (15)	2.8540 (9)	163.4 (15)
N2*B*—H1*N*2···N1*B*^{ii}	0.857 (16)	2.057 (16)	2.9128 (10)	176.0 (16)
C1*A*—H1*AA*···O2*A*^{iii}	0.93	2.50	3.1129 (10)	124
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Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) −*x*+2, *y*+1/2, −*z*+3/2.