Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Hydrazino-1,3-dimethyl-4-nitro-1*H*-pyrazole

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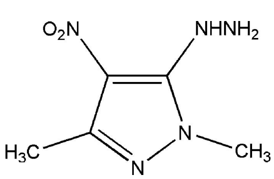
Received 2 July 2007; accepted 9 August 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.127; data-to-parameter ratio = 12.6.

Three distinct N—H···O,N interactions connect molecules of the title compound, $C_5H_9N_5O_2$, into a supramolecular assembly. The molecules are stacked in columns along the b axis. In this arrangement, the molecules are linked to each other by four two-center hydrogen bonds in an $R_2^4(12)$ ring motif. The N—H···O,N interactions have $D\cdots A$ distances ranging from 3.044 (2) to 3.182 (3) Å and D—H···A angles ranging from 138 (2) to 164 (2)° (where D and A are donor and acceptor, respectively).

Related literature

For related literature, see: Abu-Safieh *et al.* (2007); Allen *et al.* (1987); Bernstein *et al.* (1995); Burke-Laing & Laing (1976); Bustos *et al.* (2007); Guzei *et al.* (2007); Manfredini *et al.* (1992); Mavel *et al.* (1993); Sun *et al.* (2007); Tewari *et al.* (2002); Wang *et al.* (2007); Xia *et al.* (2007*a*,*b*); Yan (2007); Yu *et al.* (2007).



Experimental

Crystal data

$C_5H_9N_5O_2$
M_r = 171.17
Orthorhombic, *Pbca*
a = 8.3129 (3) Å
b = 14.340 (3) Å
c = 12.869 (4) Å
V = 1534.1 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.55 × 0.35 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: numerical (*Shape Tracing Software*; Rigaku, 2003)
T_{min} = 0.945, T_{max} = 0.967
4386 measured reflections
1846 independent reflections
1233 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
3 standard reflections every 400 reflections intensity decay: 3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.127
S = 1.04
1846 reflections
146 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	$D\cdots A$	D—H···A
N4—H4*A*···O1^{i}	0.84 (3)	2.23 (3)	3.044 (2)	164 (2)
N4—H4*B*···O2^{ii}	0.87 (3)	2.48 (3)	3.182 (3)	138 (2)
N3—H3···N2^{iii}	0.851 (18)	2.396 (17)	3.088 (2)	138.8 (16)

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *HELENA* (Spek, 1996); program(s) used to solve structure: *XS* in *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *XL* in *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *XCIF* in *SHELXTL*.

The Deanship of Scientific Research at The Hashemite University, Al al-Bayt University and Al-Balqa a Applied University (Jordan) are thanked for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG3110).

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supplementary materials

Acta Cryst. (2007). E**63**, o3814-o3815   [**doi:10.1107/S1600536807039517**]

5-Hydrazino-1,3-dimethyl-4-nitro-1*H*-pyrazole

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Comment

Substituted pyrazolo-1,2,4-triazines constitute an important family of heterocyclic compounds due to their possible biological activity. including antifungal, antiviral, anti-inflammatory, anticonvulsant, antidepressant, antihypertensive properties (Tewari *et al.*, 2002; Manfredini *et al.*, 1992; Mavel *et al.*, 1993). A novel method for the synthesis of many substituted pyrazolo[4,3 e][1,2,4]triazines has been reported very recently (Abu-Safieh *et al.*, 2007). This synthesis based on the preparation of 5-hydrazinopyrazole from 5-chloro-1,3-dimethyl-4-nitropyrazole followed by acylation and nitro group reduction to form the corresponding 4-amino-3-(acylhydrazino)pyrazoles. Then by using polyphosphoric acid *via* intramolecular oxidative cyclization the target pyrazolotriazines is obtained. In this paper, we report the crystal structure of the title compound, (I), which represents the first step product, 5-hydrazinopyrazole, of these reactions to the formation of pyrazolotriazines.

The title compound, (I), formed from the hydrazinolysis of 5-chloro-1,3-dimethyl-4-nitropyrazole in an S_N Ar addition-elimination reaction. A view of the structure of (I) and its atom-numbering scheme is shown in Fig. 1. Selected geometrical parameters are given in Table 1. The asymmetric unit of (I) is made up of one organic moiety composed of a central N-containing ring, with a methyl group connected to the 1-position of the ring, a methyl group in the 3-position of the ring, a nitro group (—NO_2) in the 4-position of the ring and a hydrazine group NH_2NH— group connected to the 5-position of the ring. The pyrazolo ring is planar, which can be attributed to a wide range of electron delocalization [maximum deviations of −0.0015 (9), −0.0014 (9) and 0.0018 (9) for N1, C4 and C5, respectively]. Compound (I) is comparable to other similar ones in Cambridge Structural Database (CSD; see for example: Yu *et al.*, 2007; Wang *et al.*, 2007; Xia *et al*, 2007*a,b*; Bustos *et al.*, 2007), except that the substituents at positions 3- (—NO_2) and 4- (NH_2NH—) are of the kind that are good candidates to participate in hydrogen bonding leading to a supramolecular architecture (see hydrogen bonding discussion below).

The bond distances of similar types within the ring are not equivalent [1.309 (2) and 1.3443 (17) Å for N2—C3 and N1—C5, respectively, and 1.410 (2) and 1.419 (2) Å for C4—C5 and C3—C4, respectively]. Furthermore, the N1—C6 bond distance is different (1.453 (2) Å) and significantly longer than the N1—C5 and N2—C3 bonds, which is indicative of some multiple bond character in both N1—C5 and N2—C3 (Guzei *et al.*, 2007; Yan, 2007; Wang *et al.*, 2007). The sum of the angles at N1 is 360°, indicating sp^2 hybridization. The N1—C6 bond length is closer to the average C_{ar}—Nsp^3 (pyramidal) value of 1.419 (17) Å than to the C_{ar}—Nsp^2 1.353 (7) Å (Allen *et al.*, 1987). The N1—N2 bond length is 1.3907 (18) Å, (smaller than a single bond) (1.41 Å; Burke-Laing & Laing, 1976; Guzei *et al.*, 2007; Yan, 2007; Wang *et al*, 2007). The remaining bond lengths in (I) show no unusual values (Sun *et al.*, 2007; Guzei *et al.*, 2007; Yan, 2007; Wang *et al.*, 2007).

There are three strong interactions (Table 2) of two types (N—H···O and N—H···N). These hydrogen bonds link molecules into two-dimensional corrugated sheets (Fig. 2) stacked along the *b* axis (Fig. 3). The significance of the hydrogen bonds is represented by relatively short D···A distances and D—H···A angles spanning 138 (2) to 164 (2)° (Table 2). These hydrogen bonds link the molecules to each other by four two-center N—H···O hydrogen bonds, in which the N—H and H_2N species of the hydrazino group lead to a number of intra- and intermolecular hydrogen bonds (Table 2). The NH_2 group of the H_2NH— species at (x, y, z) participitate in two N—H···O—NO hydrogen bonds [N4—H4A···O1 ($x - 1/2$, $-y + 3/2$, $-z +$

1) and N4—H4B···O2 (−*x* + 1/2, *y* − 1/2, *z*)], forming a centrosymmetric $R^4_2(12)$ ring (Bernstein *et al.*, 1995), Fig. 3. In addition to these hydrogen bonds occurring between the molecules in the inversion related columns, there are also N—H···N hydrogen bonds [N3—H3···N2 (*x*, −*y* + 3/2, *z* − 1/2)].

As can be seen from the packing diagram (Fig. 3), the intermolecular hydrogen bonds (Table 2) and other less significant hydrogen bond interactions (longer interactions) cause to the formation of a three-dimensional supramolecular architecture, in which they may be effective in the stabilization of the crystal structure. Dipole-dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

The title compound (I), prepared according to the following method (Abu-Safieh *et al.*, 2007). $H_2NNH_2 \cdot H_2O$ (85%, 8 ml, 160 mmol) was added dropwise to a stirred solution of 5-chloro-1,3-dimethyl-4-nitropyrazole (88 mg, 5 mmol) dissolved in 30 ml absolute ethanol. After stirring for *ca* 15 min at ambient temperature, the mixture was refluxed (water bath) for *ca* 2 h. The solvent was removed *in vacuo* to dryness. The resulting residual solid, recrystallized from ethanol yielded X-ray quality yellow crystals (m.p. 452–453 K). Yield = 0.59 g (69%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.39 (s, 3H, CH_3—C(3)), 3.92 (br s,, 2H, N(4)H_2), 3.95 (s, 3H, N(1)—CH_3), 8.07 (br s, 1H, N(3)*H*); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.4 (C(7)), 39.3 (C(6)), 118.1 (C(3)), 145.0 (C(4)), 147.6 (C(5)); IR (cm^{-1}): 3339 (υ(N—H)), 2970 (υ(C—H)), 1664 (υ(C═N)), 1582, 1367 (υ(NO_2)).

Refinement

All H atoms were located in the difference map and refined independently with isotropic thermal displacement coefficients.

Figures

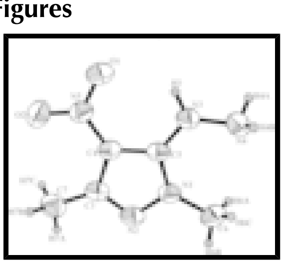


Fig. 1. A molecular drawing of (I) shown with 50% probability ellipsoids.

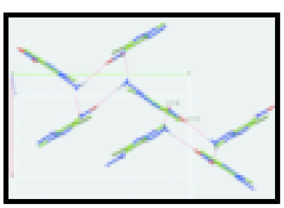


Fig. 2. Packing diagram of (I), shows the corrugated sheets of molecules. All hydrogen atoms not involved in hydrogen bonding were omitted for clarity. Hydrogen bonds are shown with dashed lines.

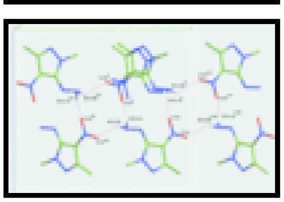


Fig. 3. Packing diagram of (I) shown along the *a* axis. All hydrogen atoms not involved in hydrogen bonding were omitted for clarity. Hydrogen bonds are shown with dashed lines. [Symmetry codes: (i) *x* − 1/2, −*y* + 3/2, −*z* + 1; (ii) −*x* + 1/2, *y* − 1/2, *z*; (iv) −*x*, −*y* + 1, −*z* + 1; (v) *x* + 1/2, −*y* + 3/2, −*z* + 1; (vi) −*x* + 1, −*y* + 2, −*z* + 1; (vii) −*x* + 1/2, *y* + 1/2, *z*].

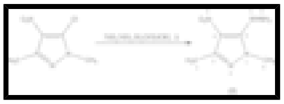


Fig. 4. The formation of the title compound.

5-Hydrazino-1,3-dimethyl-4-nitro-1*H*-pyrazole

Crystal data

$C_5H_9N_5O_2$	$F_{000} = 720$
$M_r = 171.17$	$D_x = 1.482$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1846 reflections
$a = 8.3129$ (3) Å	θ = 2.1–28.0º
$b = 14.340$ (3) Å	$\mu = 0.12$ mm^{-1}
$c = 12.869$ (4) Å	$T = 298$ (2) K
$V = 1534.1$ (6) Å^3	Block, yellow
$Z = 8$	0.55 × 0.35 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.027$
Radiation source: fine-focus sealed tube	$\theta_{max} = 28.0$º
Monochromator: graphite	$\theta_{min} = 3.2$º
$T = 298(2)$ K	$h = -1\rightarrow11$
ω scans	$k = -1\rightarrow18$
Absorption correction: numerical (Shape Tracing Software; Rigaku, 2003)	$l = -16\rightarrow16$
$T_{min} = 0.945$, $T_{max} = 0.967$	3 standard reflections
4386 measured reflections	every 400 reflections
1846 independent reflections	intensity decay: 3%
1233 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.0821P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.127$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
1846 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
146 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0061 (18)

Secondary atom site location: difference Fourier map

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > 2\mathrm{sigma}(F^2)$ 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}
O1	0.21521 (17)	0.87426 (10)	0.56075 (8)	0.0607 (4)
N1	0.05276 (17)	0.72636 (9)	0.80896 (9)	0.0422 (4)
O2	0.32124 (19)	0.96616 (9)	0.67586 (10)	0.0670 (4)
N2	0.11316 (18)	0.78454 (9)	0.88612 (9)	0.0464 (4)
N3	0.05167 (19)	0.71807 (10)	0.62276 (11)	0.0485 (4)
H3	0.087 (2)	0.7452 (13)	0.5686 (13)	0.050 (5)*
C3	0.1893 (2)	0.85253 (11)	0.83932 (11)	0.0420 (4)
N4	−0.0454 (2)	0.63832 (13)	0.61765 (12)	0.0563 (4)
H4A	−0.108 (3)	0.6468 (16)	0.5671 (19)	0.074 (7)*
H4B	0.010 (3)	0.5904 (19)	0.598 (2)	0.095 (10)*
C4	0.17954 (19)	0.83961 (10)	0.73019 (11)	0.0383 (4)
N5	0.24162 (16)	0.89595 (10)	0.65299 (11)	0.0446 (4)
C5	0.08993 (18)	0.75750 (11)	0.71340 (10)	0.0378 (4)
C6	−0.0289 (3)	0.64202 (16)	0.84255 (17)	0.0608 (6)
H6C	−0.137 (4)	0.647 (2)	0.830 (2)	0.130 (11)*
C7	0.2681 (3)	0.92806 (15)	0.89924 (15)	0.0574 (5)
H7C	0.239 (3)	0.9836 (17)	0.8771 (18)	0.077 (7)*
H7A	0.234 (3)	0.9278 (17)	0.972 (2)	0.084 (7)*
H7B	0.388 (3)	0.9269 (19)	0.888 (2)	0.101 (9)*
H6A	−0.003 (4)	0.631 (2)	0.905 (3)	0.109 (9)*
H6B	0.010 (4)	0.592 (3)	0.810 (3)	0.149 (15)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0709 (9)	0.0775 (9)	0.0338 (6)	−0.0148 (7)	0.0073 (6)	0.0072 (6)
N1	0.0459 (8)	0.0492 (7)	0.0315 (6)	0.0003 (6)	0.0011 (5)	0.0045 (5)
O2	0.0827 (10)	0.0577 (8)	0.0607 (8)	−0.0225 (8)	0.0039 (7)	0.0039 (6)
N2	0.0558 (9)	0.0531 (8)	0.0302 (6)	0.0059 (7)	−0.0001 (6)	0.0000 (5)
N3	0.0568 (9)	0.0576 (8)	0.0311 (6)	−0.0131 (7)	0.0003 (6)	0.0011 (6)
C3	0.0471 (9)	0.0447 (8)	0.0340 (7)	0.0098 (7)	−0.0015 (7)	−0.0002 (6)

N4	0.0603 (10)	0.0643 (10)	0.0443 (8)	−0.0149 (9)	−0.0077 (8)	−0.0001 (7)
C4	0.0412 (8)	0.0419 (7)	0.0318 (7)	0.0056 (7)	0.0018 (6)	0.0017 (6)
N5	0.0461 (8)	0.0486 (8)	0.0392 (7)	−0.0007 (7)	0.0045 (6)	0.0049 (6)
C5	0.0363 (8)	0.0462 (8)	0.0308 (7)	0.0066 (7)	0.0023 (6)	0.0038 (6)
C6	0.0703 (14)	0.0654 (12)	0.0469 (10)	−0.0140 (11)	0.0002 (10)	0.0173 (9)
C7	0.0729 (15)	0.0538 (11)	0.0456 (10)	0.0033 (10)	−0.0042 (9)	−0.0093 (8)

Geometric parameters (Å, °)

O1—N5	1.2467 (18)	N4—H4A	0.84 (3)
N1—C5	1.3443 (17)	N4—H4B	0.87 (3)
N1—N2	1.3907 (18)	C4—N5	1.3805 (19)
N1—C6	1.453 (2)	C4—C5	1.410 (2)
O2—N5	1.2404 (18)	C6—H6C	0.92 (3)
N2—C3	1.309 (2)	C6—H6A	0.84 (3)
N3—C5	1.3347 (19)	C6—H6B	0.90 (4)
N3—N4	1.401 (2)	C7—H7C	0.88 (3)
N3—H3	0.851 (18)	C7—H7A	0.98 (3)
C3—C4	1.419 (2)	C7—H7B	1.01 (3)
C3—C7	1.482 (3)		
C5—N1—N2	111.77 (13)	O2—N5—C4	120.25 (14)
C5—N1—C6	131.00 (15)	O1—N5—C4	118.26 (14)
N2—N1—C6	117.12 (13)	N3—C5—N1	127.16 (15)
C3—N2—N1	107.03 (12)	N3—C5—C4	127.85 (13)
C5—N3—N4	121.60 (14)	N1—C5—C4	104.99 (12)
C5—N3—H3	116.1 (12)	N1—C6—H6C	110 (2)
N4—N3—H3	122.3 (12)	N1—C6—H6A	108 (2)
N2—C3—C4	109.31 (14)	H6C—C6—H6A	116 (3)
N2—C3—C7	121.23 (15)	N1—C6—H6B	111 (2)
C4—C3—C7	129.46 (16)	H6C—C6—H6B	110 (3)
N3—N4—H4A	105.9 (16)	H6A—C6—H6B	102 (3)
N3—N4—H4B	110.6 (18)	C3—C7—H7C	112.0 (15)
H4A—N4—H4B	102 (2)	C3—C7—H7A	111.6 (15)
N5—C4—C5	125.16 (13)	H7C—C7—H7A	104 (2)
N5—C4—C3	127.92 (15)	C3—C7—H7B	110.7 (15)
C5—C4—C3	106.90 (13)	H7C—C7—H7B	104 (2)
O2—N5—O1	121.49 (13)	H7A—C7—H7B	115 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4A···O1^{i}	0.84 (3)	2.23 (3)	3.044 (2)	164 (2)
N4—H4B···O2^{ii}	0.87 (3)	2.48 (3)	3.182 (3)	138 (2)
N3—H3···N2^{iii}	0.851 (18)	2.396 (17)	3.088 (2)	138.8 (16)

Symmetry codes: (i) *x*−1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1/2, *y*−1/2, *z*; (iii) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

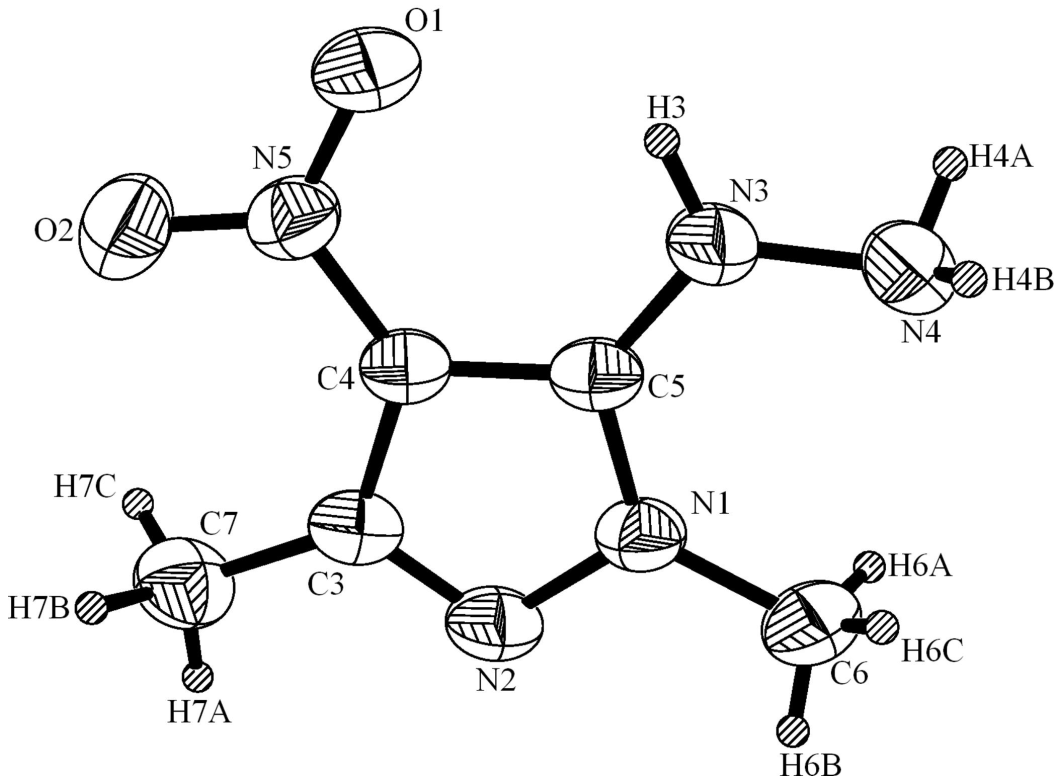

O1
H3
N5
H4A
N3
O2
H4B
N4
C4
C5
H7C
N1
C7
H6A
C3
H7B
H6C
N2
C6
H7A
H6B

Fig. 2

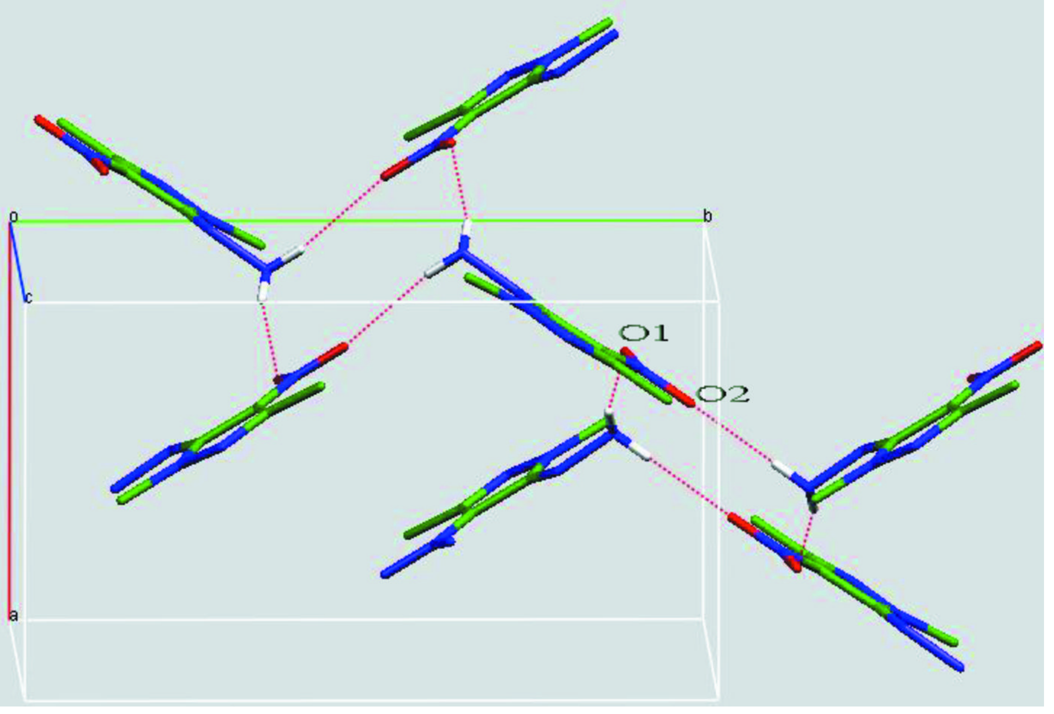


Fig. 3

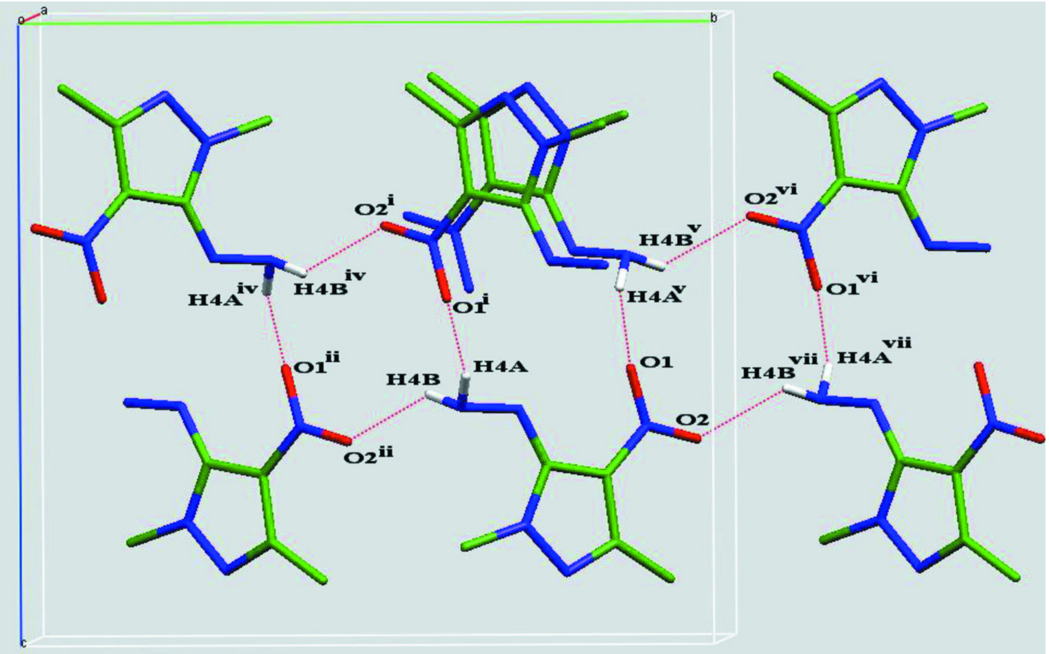
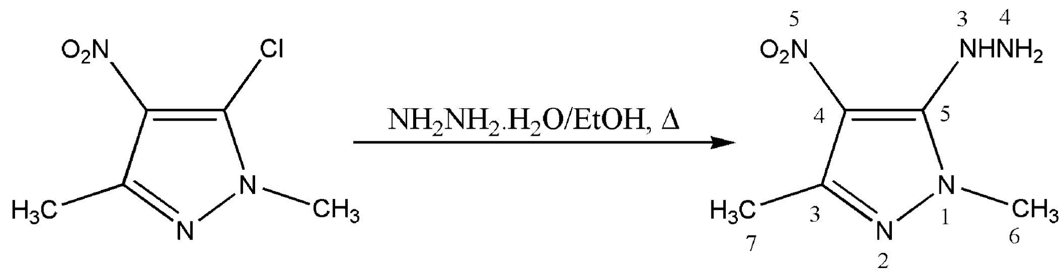

a
b
c
O2^i
H4B^v
O2^vi
H4A^iv
H4B^iv
O1^i
H4A^v
O1^vi
O1^ii
H4B
H4A
O1
H4B^vii
H4A^vii
O2
O2^ii

Fig. 4


O2N
Cl
H3C
N
N
CH3
NH2NH2.H2O/EtOH, Δ
5
3 4
O2N
NHNH2
4
5
H3C
3
N
1
N
CH3
7
2
6

(I)