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2-Aminoanilinium picrate

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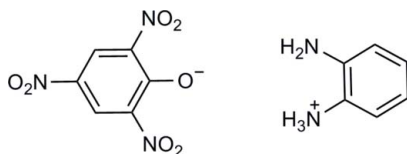
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.120; data-to-parameter ratio = 15.1.

In the title compound, $\text{C}_6\text{H}_9\text{N}_2^+\cdot\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$, the three nitro groups of the anion are twisted from the central benzene ring at dihedral angles of 5.4 (1), 27.1 (1) and 32.9 (1)°. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots(\text{O},\text{O})$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds link the cations and anions into layers parallel to the bc plane.

Related literature

For the crystal structures of picric acid complexes, see: Harrison *et al.* (2007); Li (2009); Saminathan *et al.* (2007); Sivaramkumar *et al.* (2010). For their conformational features and charge-transfer processes, see: Nagata *et al.* (1995); Smith *et al.* (2004).



Experimental

Crystal data

 $\text{C}_6\text{H}_9\text{N}_2^+\cdot\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$
 $M_r = 337.26$

 Monoclinic, $P2_1/c$
 $a = 13.2938$ (11) Å

 $b = 6.9959$ (6) Å

 $c = 15.2967$ (13) Å

 $\beta = 92.629$ (1)°

 $V = 1421.1$ (2) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.13$ mm⁻¹
 $T = 298$ K

 $0.16 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1997)

 $T_{\min} = 0.979$, $T_{\max} = 0.987$

16897 measured reflections

3514 independent reflections

 2394 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.120$
 $S = 0.93$

3514 reflections

232 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}$	0.922 (18)	2.048 (19)	2.9607 (18)	170.0 (14)
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.946 (16)	1.852 (17)	2.7731 (16)	163.9 (14)
$\text{N2}-\text{H2A}\cdots\text{O7}$	0.946 (16)	2.514 (15)	2.8558 (17)	101.4 (11)
$\text{N1}-\text{H1B}\cdots\text{O7}^{\text{i}}$	0.813 (18)	2.424 (19)	3.2264 (17)	169.6 (16)
$\text{N2}-\text{H2A}\cdots\text{O1}^{\text{ii}}$	0.946 (16)	2.581 (16)	2.9872 (18)	106.2 (11)
$\text{N2}-\text{H2B}\cdots\text{O2}^{\text{ii}}$	0.858 (17)	2.448 (16)	3.122 (2)	135.9 (14)
$\text{N2}-\text{H2B}\cdots\text{O6}^{\text{iii}}$	0.858 (17)	2.556 (16)	2.9956 (17)	112.9 (12)
$\text{N2}-\text{H2C}\cdots\text{N1}^{\text{iv}}$	0.858 (18)	2.063 (18)	2.904 (2)	166.1 (16)

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; 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.

The authors are grateful to Xiangfan University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2788).

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supporting information

Acta Cryst. (2010). E66, o3235 [https://doi.org/10.1107/S1600536810047057]

2-Aminoanilinium picrate

Rong Peng and Yanping Zhao

S1. Comment

Picric acid is widely used in munitions and explosives. In microscopy, it also serves as a reagent for staining samples, *e.g.*, Gram staining. The crystal structures of a large number of picrate salts and picric acid complexes have been studied (Harrison *et al.*, 2007; Li, 2009; Saminathan *et al.*, 2007; Sivaramkumar *et al.*, 2010) to understand the conformational features and charge transfer processes (Nagata *et al.*, 1995; Smith *et al.*, 2004). We herein report the crystal structure of the title compound (I) (Fig. 1).

In (I), three nitro groups of the anion are twisted from the central benzene ring at 5.4 (1), 27.1 (1) and 32.9 (1)°, respectively. In the crystal structure, intermolecular N—H \cdots O and N—H \cdots N hydrogen bonds (Table 1) link cations and anions into layers parallel to *bc* plane.

S2. Experimental

Benzene-1,2-diamine (0.32 g, 3.0 mmol) and picric acid (0.69 g, 3.0 mmol) were mixed in 15 ml ethanol. The mixture was kept at room temperature for two weeks, after which time needle like yellow crystals (0.16 x 0.12 x 0.10 mm) suitable for single-crystal X-ray diffraction were obtained.

S3. Refinement

The O- and N-bound H atoms were located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically (C—H = 0.95 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

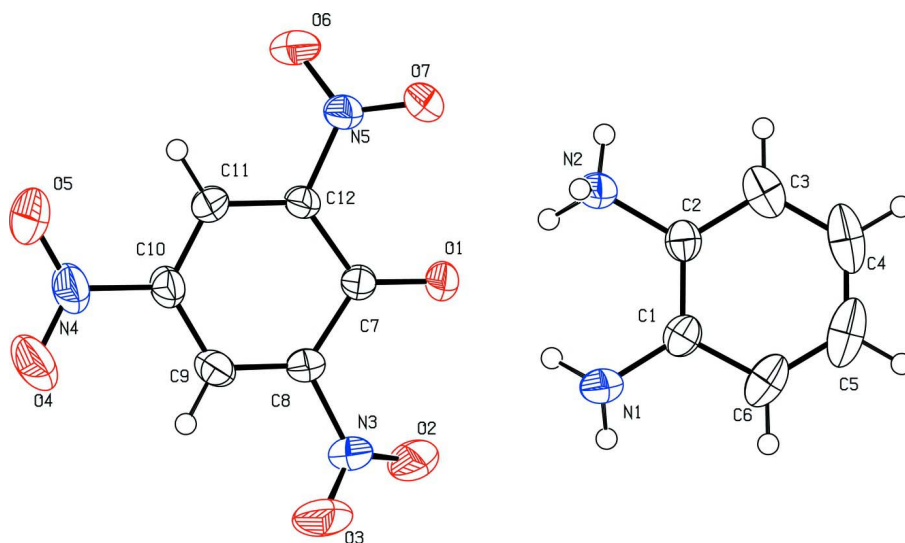


Figure 1

The molecular structure of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

2-Aminoanilinium 2,4,6-trinitrophenolate

Crystal data

$C_6H_9N_2^+ \cdot C_6H_2N_3O_7^-$

$M_r = 337.26$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 13.2938$ (11) Å

$b = 6.9959$ (6) Å

$c = 15.2967$ (13) Å

$\beta = 92.629$ (1)°

$V = 1421.1$ (2) Å³

$Z = 4$

$F(000) = 696$

$D_x = 1.576$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3836 reflections

$\theta = 2.7$ – 24.9 °

$\mu = 0.13$ mm⁻¹

$T = 298$ K

Block, yellow

$0.16 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ϕ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1997)

$T_{\min} = 0.979$, $T_{\max} = 0.987$

16897 measured reflections

3514 independent reflections

2394 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 2.7$ °

$h = -17 \rightarrow 17$

$k = -9 \rightarrow 9$

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.120$

$S = 0.93$

3514 reflections

232 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

$$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.38865 (11)	0.36646 (19)	0.40921 (9)	0.0336 (3)
C2	0.34942 (10)	0.3253 (2)	0.48958 (9)	0.0325 (3)
C3	0.24721 (11)	0.3359 (2)	0.50234 (12)	0.0467 (4)
H3	0.2225	0.3084	0.5568	0.056*
C4	0.18224 (13)	0.3879 (2)	0.43324 (16)	0.0612 (5)
H4	0.1133	0.3938	0.4407	0.073*
C5	0.22011 (15)	0.4306 (2)	0.35353 (15)	0.0617 (5)
H5	0.1764	0.4655	0.3071	0.074*
C6	0.32123 (14)	0.4227 (2)	0.34162 (11)	0.0484 (4)
H6	0.3455	0.4553	0.2875	0.058*
C7	0.68910 (10)	0.11086 (18)	0.55689 (8)	0.0280 (3)
C8	0.77757 (10)	0.1384 (2)	0.50680 (9)	0.0315 (3)
C9	0.87421 (11)	0.1403 (2)	0.54126 (9)	0.0361 (3)
H9	0.9277	0.1672	0.5061	0.043*
C10	0.89116 (10)	0.1016 (2)	0.62926 (9)	0.0370 (4)
C11	0.81281 (10)	0.0667 (2)	0.68247 (9)	0.0346 (3)
H11	0.8253	0.0365	0.7412	0.042*
C12	0.71596 (10)	0.07703 (19)	0.64812 (8)	0.0291 (3)
N1	0.49196 (10)	0.3622 (2)	0.39598 (9)	0.0406 (3)
H1A	0.5270 (13)	0.274 (2)	0.4304 (11)	0.049*
H1B	0.5052 (13)	0.351 (2)	0.3449 (12)	0.049*
N2	0.41690 (10)	0.2695 (2)	0.56298 (8)	0.0367 (3)
H2A	0.4726 (13)	0.195 (2)	0.5473 (10)	0.044*
H2B	0.3848 (12)	0.218 (2)	0.6043 (11)	0.044*
H2C	0.4430 (12)	0.373 (3)	0.5843 (11)	0.044*
N3	0.76527 (10)	0.1700 (2)	0.41270 (8)	0.0436 (3)
N4	0.99307 (10)	0.0980 (2)	0.66670 (10)	0.0588 (4)
N5	0.63727 (9)	0.04886 (18)	0.70943 (8)	0.0365 (3)
O1	0.60100 (7)	0.11173 (15)	0.52432 (6)	0.0377 (3)
O2	0.69569 (9)	0.0908 (2)	0.37119 (7)	0.0630 (4)
O3	0.82684 (10)	0.2717 (2)	0.37879 (8)	0.0740 (4)
O4	1.06230 (9)	0.1404 (2)	0.61973 (10)	0.0814 (5)

O5	1.00595 (10)	0.0516 (3)	0.74296 (10)	0.1001 (6)
O6	0.65667 (9)	-0.04467 (18)	0.77549 (7)	0.0549 (3)
O7	0.55494 (8)	0.1226 (2)	0.69490 (7)	0.0632 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0363 (8)	0.0312 (8)	0.0327 (7)	-0.0020 (6)	-0.0049 (6)	-0.0030 (6)
C2	0.0295 (7)	0.0315 (7)	0.0361 (8)	-0.0009 (6)	-0.0016 (6)	-0.0024 (6)
C3	0.0334 (8)	0.0406 (9)	0.0666 (11)	0.0004 (7)	0.0061 (8)	-0.0058 (8)
C4	0.0295 (9)	0.0461 (10)	0.1064 (17)	0.0022 (7)	-0.0120 (10)	-0.0108 (11)
C5	0.0573 (12)	0.0437 (10)	0.0804 (14)	0.0053 (8)	-0.0370 (11)	-0.0043 (9)
C6	0.0590 (11)	0.0405 (9)	0.0436 (9)	-0.0007 (8)	-0.0204 (8)	0.0008 (7)
C7	0.0277 (7)	0.0304 (7)	0.0260 (7)	0.0019 (5)	0.0014 (5)	-0.0020 (5)
C8	0.0340 (7)	0.0363 (8)	0.0244 (7)	0.0001 (6)	0.0034 (6)	0.0000 (6)
C9	0.0303 (8)	0.0436 (9)	0.0352 (8)	-0.0035 (6)	0.0088 (6)	-0.0026 (6)
C10	0.0254 (7)	0.0499 (9)	0.0355 (8)	-0.0007 (6)	-0.0014 (6)	-0.0066 (7)
C11	0.0335 (8)	0.0458 (9)	0.0243 (7)	0.0019 (6)	-0.0010 (6)	-0.0029 (6)
C12	0.0262 (7)	0.0372 (8)	0.0242 (7)	-0.0009 (6)	0.0043 (5)	-0.0023 (5)
N1	0.0413 (8)	0.0535 (8)	0.0273 (6)	-0.0016 (6)	0.0048 (6)	0.0025 (6)
N2	0.0340 (7)	0.0485 (8)	0.0280 (6)	0.0019 (6)	0.0048 (5)	-0.0027 (6)
N3	0.0453 (8)	0.0586 (8)	0.0276 (6)	0.0047 (7)	0.0078 (6)	0.0068 (6)
N4	0.0280 (7)	0.0973 (12)	0.0506 (9)	0.0024 (7)	-0.0036 (7)	-0.0095 (8)
N5	0.0316 (7)	0.0518 (8)	0.0263 (6)	-0.0020 (6)	0.0043 (5)	-0.0022 (5)
O1	0.0271 (5)	0.0558 (7)	0.0299 (5)	0.0053 (4)	-0.0026 (4)	-0.0021 (5)
O2	0.0590 (8)	0.1025 (11)	0.0270 (6)	-0.0075 (7)	-0.0025 (6)	-0.0022 (6)
O3	0.0754 (9)	0.1017 (11)	0.0460 (7)	-0.0163 (8)	0.0145 (7)	0.0306 (7)
O4	0.0284 (7)	0.1396 (14)	0.0766 (10)	-0.0101 (7)	0.0079 (6)	-0.0095 (9)
O5	0.0424 (8)	0.199 (2)	0.0570 (9)	0.0039 (10)	-0.0191 (6)	0.0133 (11)
O6	0.0575 (8)	0.0729 (8)	0.0355 (6)	0.0063 (6)	0.0145 (5)	0.0171 (6)
O7	0.0332 (6)	0.1180 (11)	0.0390 (7)	0.0164 (7)	0.0102 (5)	0.0097 (7)

Geometric parameters (Å, °)

C1—C2	1.387 (2)	C9—H9	0.9300
C1—C6	1.393 (2)	C10—C11	1.373 (2)
C1—N1	1.3977 (19)	C10—N4	1.447 (2)
C2—C3	1.383 (2)	C11—C12	1.3699 (19)
C2—N2	1.4579 (19)	C11—H11	0.9300
C3—C4	1.383 (3)	C12—N5	1.4501 (17)
C3—H3	0.9300	N1—H1A	0.922 (18)
C4—C5	1.373 (3)	N1—H1B	0.813 (18)
C4—H4	0.9300	N2—H2A	0.946 (16)
C5—C6	1.366 (3)	N2—H2B	0.858 (17)
C5—H5	0.9300	N2—H2C	0.858 (18)
C6—H6	0.9300	N3—O3	1.2182 (17)
C7—O1	1.2517 (15)	N3—O2	1.2295 (17)
C7—C12	1.4441 (18)	N4—O5	1.215 (2)

C7—C8	1.4457 (19)	N4—O4	1.2296 (19)
C8—C9	1.366 (2)	N5—O7	1.2211 (16)
C8—N3	1.4580 (18)	N5—O6	1.2214 (15)
C9—C10	1.381 (2)		
C2—C1—C6	117.44 (14)	C11—C10—C9	121.21 (13)
C2—C1—N1	122.40 (13)	C11—C10—N4	118.98 (13)
C6—C1—N1	120.08 (15)	C9—C10—N4	119.81 (13)
C3—C2—C1	121.70 (14)	C12—C11—C10	119.25 (13)
C3—C2—N2	118.64 (14)	C12—C11—H11	120.4
C1—C2—N2	119.66 (12)	C10—C11—H11	120.4
C4—C3—C2	119.30 (17)	C11—C12—C7	124.44 (12)
C4—C3—H3	120.4	C11—C12—N5	115.98 (12)
C2—C3—H3	120.4	C7—C12—N5	119.57 (12)
C5—C4—C3	119.63 (17)	C1—N1—H1A	113.9 (10)
C5—C4—H4	120.2	C1—N1—H1B	113.6 (12)
C3—C4—H4	120.2	H1A—N1—H1B	111.0 (16)
C6—C5—C4	120.84 (17)	C2—N2—H2A	114.5 (10)
C6—C5—H5	119.6	C2—N2—H2B	111.7 (11)
C4—C5—H5	119.6	H2A—N2—H2B	112.2 (15)
C5—C6—C1	121.06 (17)	C2—N2—H2C	107.0 (11)
C5—C6—H6	119.5	H2A—N2—H2C	104.6 (15)
C1—C6—H6	119.5	H2B—N2—H2C	106.2 (16)
O1—C7—C12	124.81 (12)	O3—N3—O2	123.18 (13)
O1—C7—C8	123.88 (12)	O3—N3—C8	117.51 (14)
C12—C7—C8	111.28 (12)	O2—N3—C8	119.29 (13)
C9—C8—C7	124.77 (13)	O5—N4—O4	123.30 (15)
C9—C8—N3	116.13 (13)	O5—N4—C10	118.21 (15)
C7—C8—N3	119.10 (12)	O4—N4—C10	118.49 (15)
C8—C9—C10	118.86 (13)	O7—N5—O6	122.04 (12)
C8—C9—H9	120.6	O7—N5—C12	119.47 (12)
C10—C9—H9	120.6	O6—N5—C12	118.46 (12)
C6—C1—C2—C3	1.1 (2)	N4—C10—C11—C12	177.54 (14)
N1—C1—C2—C3	177.94 (14)	C10—C11—C12—C7	4.1 (2)
C6—C1—C2—N2	-178.80 (13)	C10—C11—C12—N5	-176.71 (13)
N1—C1—C2—N2	-2.0 (2)	O1—C7—C12—C11	176.54 (14)
C1—C2—C3—C4	0.3 (2)	C8—C7—C12—C11	-1.78 (19)
N2—C2—C3—C4	-179.72 (14)	O1—C7—C12—N5	-2.6 (2)
C2—C3—C4—C5	-0.9 (2)	C8—C7—C12—N5	179.07 (12)
C3—C4—C5—C6	-0.1 (3)	C9—C8—N3—O3	-30.9 (2)
C4—C5—C6—C1	1.6 (3)	C7—C8—N3—O3	148.08 (14)
C2—C1—C6—C5	-2.1 (2)	C9—C8—N3—O2	147.45 (14)
N1—C1—C6—C5	-178.99 (14)	C7—C8—N3—O2	-33.5 (2)
O1—C7—C8—C9	179.16 (14)	C11—C10—N4—O5	4.8 (3)
C12—C7—C8—C9	-2.51 (19)	C9—C10—N4—O5	-175.38 (17)
O1—C7—C8—N3	0.2 (2)	C11—C10—N4—O4	-175.78 (16)
C12—C7—C8—N3	178.56 (12)	C9—C10—N4—O4	4.0 (2)

C7—C8—C9—C10	4.3 (2)	C11—C12—N5—O7	152.66 (14)
N3—C8—C9—C10	-176.74 (13)	C7—C12—N5—O7	-28.1 (2)
C8—C9—C10—C11	-1.8 (2)	C11—C12—N5—O6	-25.50 (19)
C8—C9—C10—N4	178.44 (14)	C7—C12—N5—O6	153.72 (13)
C9—C10—C11—C12	-2.2 (2)		

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—H1A \cdots O1	0.922 (18)	2.048 (19)	2.9607 (18)	170.0 (14)
N2—H2A \cdots O1	0.946 (16)	1.852 (17)	2.7731 (16)	163.9 (14)
N2—H2A \cdots O7	0.946 (16)	2.514 (15)	2.8558 (17)	101.4 (11)
N1—H1B \cdots O7 ⁱ	0.813 (18)	2.424 (19)	3.2264 (17)	169.6 (16)
N2—H2A \cdots O1 ⁱⁱ	0.946 (16)	2.581 (16)	2.9872 (18)	106.2 (11)
N2—H2B \cdots O2 ⁱⁱ	0.858 (17)	2.448 (16)	3.122 (2)	135.9 (14)
N2—H2B \cdots O6 ⁱⁱⁱ	0.858 (17)	2.556 (16)	2.9956 (17)	112.9 (12)
N2—H2C \cdots N1 ^{iv}	0.858 (18)	2.063 (18)	2.904 (2)	166.1 (16)

Symmetry codes: (i) $x, -y+1/2, z-1/2$; (ii) $-x+1, -y, -z+1$; (iii) $-x+1, y+1/2, -z+3/2$; (iv) $-x+1, -y+1, -z+1$.