

## 2-Amino-4-methoxy-6-methylpyrimidin-1-ium picrate

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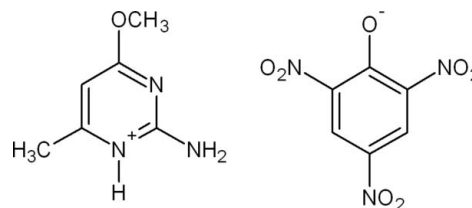
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Key indicators: single-crystal X-ray study;  $T = 200$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; disorder in main residue;  $R$  factor = 0.054;  $wR$  factor = 0.160; data-to-parameter ratio = 24.0.

In the title salt,  $\text{C}_6\text{H}_{10}\text{N}_3\text{O}^+\cdot\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$ , the dihedral angle between the mean planes of the benzene and pyridine rings is  $3.1(1)^\circ$ . In the cation, the methoxy group is almost coplanar with the pyridine ring [ $\text{C}-\text{O}-\text{C}-\text{N} = -0.6(2)^\circ$ ]. The *p*-nitro [ $\text{C}-\text{C}-\text{N}-\text{O} = -1.17(19)^\circ$ ] and one *o*-nitro [ $\text{C}-\text{C}-\text{N}-\text{O} = 1.83(19)^\circ$ ] group in the anion are essentially coplanar with the benzene ring. The other disordered *o*-nitro group containing the major occupancy [0.868(6)] O atom is twisted  $-29.0(2)^\circ$  from the mean plane of the benzene ring. A bifurcated  $\text{N}-\text{H}\cdots(\text{O},\text{O})$  hydrogen bond and weak  $\text{C}-\text{H}\cdots\text{O}$  intermolecular interaction between the cation and anion produce a network of infinite  $\text{O}-\text{H}\cdots\text{O}-\text{H}\cdots\text{O}-\text{H}$  chains along the *c* axis in the [101] plane which helps to establish crystal packing. Comparison to a DFT computational calculation indicates that significant conformational changes occur in the free state.

### Related literature

For the synthesis of imidazo[1,2-*a*]pyrimidines, see: Katritzky *et al.* (2003). For related structures, see: Ferguson *et al.* (1984); Glidewell *et al.* (2003); Narayana *et al.* (2008); Scheinbeim & Schempp, (1976); Schlueter *et al.* (2006); Subashini *et al.* (2006). For density functional theory, see: Hehre *et al.* (1986); Schmidt & Polik (2007).



### Experimental

#### Crystal data

$\text{C}_6\text{H}_{10}\text{N}_3\text{O}^+\cdot\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$   
 $M_r = 368.28$

Monoclinic,  $P2_1/c$

$a = 8.9442(3)$  Å

$b = 6.2793(3)$  Å

$c = 27.0354(8)$  Å

$\beta = 94.471(3)^\circ$

$V = 1513.78(10)$  Å<sup>3</sup>

$Z = 4$

Mo  $K\alpha$  radiation

$\mu = 0.14$  mm<sup>-1</sup>

$T = 200$  K

$0.52 \times 0.46 \times 0.35$  mm

#### Data collection

Oxford Diffraction Gemini diffractometer

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford Diffraction, 2007)

$T_{\min} = 0.986$ ,  $T_{\max} = 1.000$

15870 measured reflections

6133 independent reflections

3107 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.160$

$S = 0.94$

6133 reflections

256 parameters

24 restraints

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.43$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.29$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1B}-\text{H1BA}\cdots\text{O61B}^1$	0.88	2.09	2.883 (10)	150
$\text{N1B}-\text{H1BA}\cdots\text{O61A}^1$	0.88	2.13	2.9309 (17)	151
$\text{N1B}-\text{H1BB}\cdots\text{O1A}$	0.88	1.95	2.7223 (13)	146
$\text{N1B}-\text{H1BB}\cdots\text{O21A}$	0.88	2.20	2.8855 (14)	134
$\text{N2B}-\text{H2BA}\cdots\text{O1A}$	0.88	1.97	2.7380 (12)	145
$\text{N2B}-\text{H2BA}\cdots\text{O62B}$	0.88	2.55	3.303 (10)	144
$\text{N2B}-\text{H2BA}\cdots\text{O62A}$	0.88	2.62	3.381 (2)	145

Symmetry code: (i)  $x - 1, y, z$ .

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5240).

## References

- Ferguson, G., Kaitner, B., Lloyd, D. & McNab, H. (1984). *J. Chem. Res (S)*, pp. 182-183.
- Glidewell, C., Low, J. N., Melguizo, M. & Quesada, A. (2003). *Acta Cryst. C* **59**, o9–o13.
- Hehre, W. J., Random, L., Schleyer, P. R. & Pople, J. A. (1986). In *Ab Initio Molecular Orbital Theory*. New York: Wiley.
- Katritzky, A. R., Xu, Y.-J. & Tu, H. (2003). *J. Org. Chem.* **68**, 4935–3937.
- Narayana, B., Sarojini, B. K., Prakash Kamath, K., Yathirajan, H. S. & Bolte, M. (2008). *Acta Cryst. E* **64**, o117–o118.
- Oxford Diffraction (2007). *CrysAlis PRO* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
- Scheinbeim, J. & Schempp, E. (1976). *Acta Cryst.* **B32**, 607–609.
- Schlueter, J. A., Funk, R. J. & Geiser, U. (2006). *Acta Cryst. E* **62**, o339–o341.
- Schmidt, J. R. & Polik, W. F. (2007). *WebMO Pro*. WebMO, LLC, Holland, MI, USA; available from <http://www.webmo.net>.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.
- Subashini, A., Muthiah, P. T., Bocelli, G. & Cantoni, A. (2006). *Acta Cryst. E* **62**, o3847–o3849.

## supporting information

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## 2-Amino-4-methoxy-6-methylpyrimidin-1-ium picrate

Jerry P. Jasinski, Ray J. Butcher, H. S. Yathirajan, B. Narayana and K. Prakash Kamath

### S1. Comment

The synthesis of imidazo[1,2-a]pyrimidines has been widely investigated and is one of the most common strategies in the use of 2-aminopyrimidine as the starting material (Katritzky *et al.*, 2003). Recently, the hydrogen-bonding patterns in 2-amino-4,6-dimethylpyrimidinium picrate has been reported (Subashini *et al.*, 2006). In continuation of our work on picrates of biologically important molecules, we have prepared a new picrate of 2-amino-4-methoxy-6-methylpyrimidine,  $[\text{C}_6\text{H}_{10}\text{N}_3\text{O}]^+$ ,  $[\text{C}_6\text{H}_2\text{N}_3\text{O}_7]^-$  and its crystal structure is reported.

The title compound, (I),  $\text{C}_{12}\text{H}_{12}\text{N}_6\text{O}_8$ , crystallizes as a salt with one  $\text{C}_6\text{H}_{10}\text{N}_3\text{O}^+$ ,  $\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$ , cation-anion pair in the asymmetric unit (Fig. 1). The dihedral angle between the mean planes of the benzene and pyridine rings is  $3.10^\circ$ . In the cation the methoxy group is almost coplanar with the pyridine ring [C6B–O1B–C5B–N3B torsion angle =  $-0.63$  ( $19^\circ$ )]. Bond distances and angles in both the cation and anion are in normal ranges (Allen, 2002). The *p* [torsion angle C3A–C4A–N4A–O41A =  $1.17$  ( $18^\circ$ )] and one *o* [torsion angle C1A–C2A–N2A–O21A =  $1.83$  ( $19^\circ$ )] nitro group in the anion are nearly coplanar with the benzene ring. The other disordered *o* nitro group containing the predominate oxygen atom (occupancy = 0.868 (6)) is twisted  $-29.0$  ( $2$ ) $^\circ$  from the mean plane of the benzene ring. Bifurcated intramolecular donor, N1B, [O1A [N1B–H1BB $\cdots$ O21A. & N1B–H1BB $\cdots$ O1A] and acceptor, O1A, [N1B–H1BB $\cdots$ O1A & N2B–H2BA $\cdots$ O1A] hydrogen bonds and a weak C3B–H3BB $\cdots$ O62A(0.868 (6)) hydrogen bond interaction (Table 2) between the cation and anion produces a network of infinite O–H $\cdots$ O–H $\cdots$ O–H chains along the *c* axis in the [101] plane which helps to establish crystal packing (Fig. 2).

A density functional theory (DFT) geometry optimization molecular orbital calculation (Schmidt & Polik, 2007) was performed on the independent cation-anion pair ( $\text{C}_6\text{H}_{10}\text{N}_3\text{O}^+$ ,  $\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$ ) within the asymmetric unit with the B3LYP 6-31-G(d) basis set (Hehre *et al.*, 1986). Starting geometries were taken from X-ray refinement data. The dihedral angle between the mean planes of the benzene and pyridine rings increases to  $28.10^\circ$ . In the anion, the mean planes of the two *o*-nitro groups become twisted by  $23.14^\circ$  and  $24.20^\circ$ , respectively, from the mean plane of the benzene ring. The mean plane of the *p*-nitro group remains planar to the benzene ring. The mean plane of the methoxy group in the cation also remains planar to the pyridine ring. These observations suggest that the bifurcated N–H $\cdots$ (O,O) donor and acceptor hydrogen bonds and weak C–H $\cdots$ O intermolecular interactions play a significant role in crystal stability.

### S2. Experimental

4-Methoxy-6-methylpyrimidin-2-amine (1.39 g, 0.01 mol) was dissolved in 25 ml of ethanol. Picric acid (2.29 g, 0.01 mol) was dissolved in 15 ml of water. Both the solutions were mixed and to this, 5 ml of 5M HCl was added and stirred for few minutes. The formed complex was filtered and dried. Good quality crystals were grown from ethanol solution by slow evaporation (m. p.: 399 K). Composition: Found (Calculated): C: 39.09 (39.14); H: 3.24 (3.28); N: 22.77% (22.82%).

## S3. Refinement

All of the H atoms were placed in their calculated positions and then refined using the riding model with C—H = 0.95–0.98 Å, N—H = 0.88 Å, and with  $U_{\text{iso}}(\text{H}) = 1.18\text{--}1.52U_{\text{eq}}(\text{C},\text{N})$ .

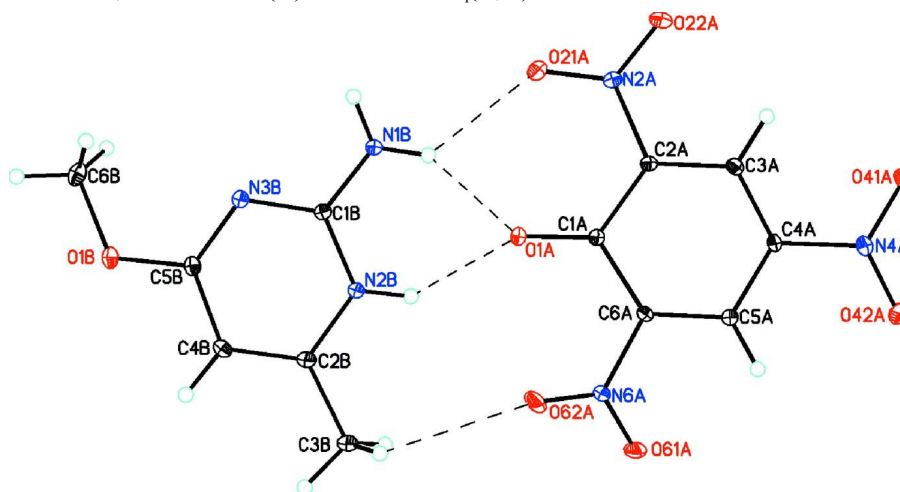


Figure 1

Molecular structure of the  $\text{C}_6\text{H}_{10}\text{N}_3\text{O}^+$ ,  $\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$  cation-anion pair showing the atom labeling scheme and 50% probability displacement ellipsoids. Only the predominate component of the disordered nitro group is displayed. Dashed lines indicate hydrogen bond interactions.

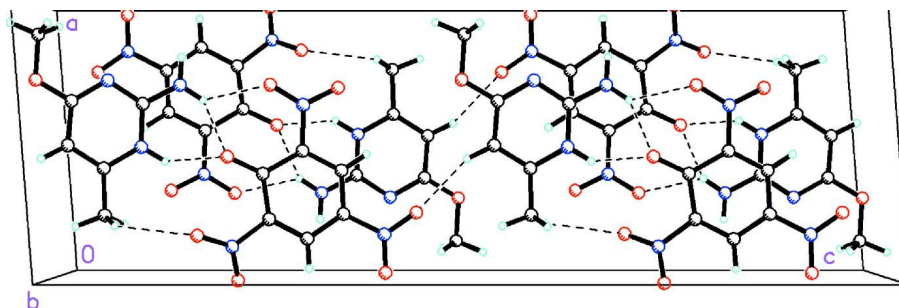


Figure 2

Packing diagram of the title compound, viewed down the  $b$  axis. Dashed lines indicate hydrogen bonds.

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## Crystal data

$\text{C}_6\text{H}_{10}\text{N}_3\text{O}^+ \cdot \text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$

$M_r = 368.28$

Monoclinic,  $P2_1/c$

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

$a = 8.9442(3)\ \text{\AA}$

$b = 6.2793(3)\ \text{\AA}$

$c = 27.0354(8)\ \text{\AA}$

$\beta = 94.471(3)^\circ$

$V = 1513.78(10)\ \text{\AA}^3$

$Z = 4$

$F(000) = 760$

$D_x = 1.616\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3951 reflections

$\theta = 4.6\text{--}34.8^\circ$

$\mu = 0.14\ \text{mm}^{-1}$

$T = 200\ \text{K}$

Prism, yellow

$0.52 \times 0.46 \times 0.35\ \text{mm}$

*Data collection*

Oxford Diffraction Gemini  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
Detector resolution: 10.5081 pixels mm<sup>-1</sup>  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan  
(*CrysAlis RED*; Oxford Diffraction, 2007)  
 $T_{\min} = 0.986$ ,  $T_{\max} = 1.000$

15870 measured reflections  
6133 independent reflections  
3107 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$   
 $\theta_{\max} = 34.9^\circ$ ,  $\theta_{\min} = 4.6^\circ$   
 $h = -11 \rightarrow 14$   
 $k = -9 \rightarrow 9$   
 $l = -42 \rightarrow 43$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.160$   
 $S = 0.94$   
6133 reflections  
256 parameters  
24 restraints  
Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map  
Hydrogen site location: inferred from  
neighbouring sites  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0847P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1A	0.55766 (10)	0.25342 (17)	0.27447 (3)	0.0282 (2)	
O21A	0.30234 (11)	0.2803 (2)	0.21683 (4)	0.0407 (3)	
O22A	0.31232 (12)	0.2676 (2)	0.13799 (4)	0.0545 (4)	
O41A	0.75641 (11)	0.24825 (17)	0.05802 (3)	0.0317 (2)	
O42A	0.97162 (12)	0.2438 (2)	0.09966 (4)	0.0505 (4)	
O61A	1.00309 (15)	0.1699 (4)	0.27556 (5)	0.0322 (5)	0.868 (6)
O62A	0.83577 (17)	0.3370 (5)	0.31386 (5)	0.0433 (6)	0.868 (6)
O61B	1.0068 (10)	0.311 (3)	0.2751 (3)	0.040 (3)	0.132 (6)
O62B	0.8309 (11)	0.195 (3)	0.3157 (3)	0.038 (3)	0.132 (6)
O1B	0.28372 (11)	0.24739 (17)	0.49248 (3)	0.0326 (2)	
N2A	0.37356 (11)	0.26925 (19)	0.18027 (4)	0.0241 (2)	
N4A	0.83482 (12)	0.24677 (19)	0.09742 (4)	0.0251 (2)	
N6A	0.87761 (11)	0.25092 (18)	0.27698 (4)	0.0222 (2)	
N1B	0.30287 (11)	0.25263 (17)	0.32338 (3)	0.0193 (2)	
H1BA	0.2044	0.2490	0.3191	0.023*	
H1BB	0.3568	0.2555	0.2975	0.023*	

N2B	0.52320 (11)	0.25912 (18)	0.37421 (3)	0.0194 (2)
H2BA	0.5741	0.2630	0.3477	0.023*
N3B	0.28659 (11)	0.24940 (18)	0.40787 (3)	0.0209 (2)
C1A	0.61812 (12)	0.2561 (2)	0.23423 (4)	0.0173 (2)
C2A	0.53717 (12)	0.2594 (2)	0.18562 (4)	0.0175 (2)
C3A	0.60696 (13)	0.2561 (2)	0.14188 (4)	0.0183 (2)
H3AA	0.5490	0.2588	0.1109	0.022*
C4A	0.76183 (13)	0.2487 (2)	0.14336 (4)	0.0184 (2)
C5A	0.85022 (12)	0.2451 (2)	0.18818 (4)	0.0186 (2)
H5AA	0.9565	0.2389	0.1888	0.022*
C6A	0.77977 (12)	0.2506 (2)	0.23132 (4)	0.0173 (2)
C1B	0.36982 (12)	0.2538 (2)	0.36869 (4)	0.0169 (2)
C2B	0.59876 (14)	0.2586 (2)	0.42009 (4)	0.0243 (3)
C3B	0.76596 (15)	0.2631 (3)	0.42302 (5)	0.0375 (4)
H3BA	0.8052	0.2642	0.4579	0.056*
H3BB	0.7996	0.3914	0.4065	0.056*
H3BC	0.8030	0.1366	0.4066	0.056*
C4B	0.51754 (14)	0.2534 (3)	0.46052 (5)	0.0300 (3)
H4BA	0.5652	0.2521	0.4932	0.036*
C5B	0.35982 (14)	0.2501 (2)	0.45220 (4)	0.0235 (3)
C6B	0.12228 (16)	0.2460 (3)	0.48535 (5)	0.0344 (3)
H6BA	0.0799	0.2454	0.5177	0.052*
H6BB	0.0891	0.1184	0.4667	0.052*
H6BC	0.0880	0.3732	0.4668	0.052*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1A	0.0188 (4)	0.0497 (7)	0.0167 (4)	0.0001 (4)	0.0047 (3)	0.0008 (4)
O21A	0.0198 (4)	0.0763 (9)	0.0263 (5)	0.0020 (5)	0.0042 (4)	-0.0057 (5)
O22A	0.0213 (5)	0.1159 (13)	0.0248 (5)	-0.0006 (6)	-0.0071 (4)	0.0054 (6)
O41A	0.0354 (5)	0.0456 (6)	0.0138 (4)	-0.0003 (5)	-0.0010 (3)	0.0000 (4)
O42A	0.0219 (5)	0.1061 (12)	0.0246 (5)	0.0017 (6)	0.0091 (4)	0.0009 (6)
O61A	0.0179 (6)	0.0502 (13)	0.0273 (6)	0.0077 (7)	-0.0054 (4)	-0.0034 (7)
O62A	0.0343 (7)	0.0769 (18)	0.0180 (6)	0.0094 (9)	-0.0035 (5)	-0.0166 (8)
O61B	0.021 (4)	0.077 (9)	0.023 (4)	-0.006 (5)	-0.005 (3)	0.002 (5)
O62B	0.023 (4)	0.069 (8)	0.022 (4)	-0.009 (5)	0.001 (3)	0.008 (4)
O1B	0.0272 (5)	0.0534 (7)	0.0183 (4)	-0.0004 (5)	0.0076 (3)	0.0000 (4)
N2A	0.0153 (4)	0.0341 (7)	0.0226 (5)	-0.0017 (5)	-0.0001 (4)	0.0013 (5)
N4A	0.0252 (5)	0.0352 (6)	0.0153 (4)	-0.0001 (5)	0.0031 (4)	0.0001 (5)
N6A	0.0180 (4)	0.0324 (6)	0.0157 (4)	-0.0022 (5)	-0.0016 (3)	0.0014 (5)
N1B	0.0148 (4)	0.0265 (5)	0.0163 (4)	0.0003 (5)	-0.0002 (3)	-0.0010 (4)
N2B	0.0152 (4)	0.0278 (6)	0.0152 (4)	-0.0004 (5)	0.0001 (3)	0.0002 (4)
N3B	0.0180 (4)	0.0273 (6)	0.0177 (4)	0.0012 (5)	0.0025 (3)	0.0001 (4)
C1A	0.0162 (5)	0.0202 (6)	0.0155 (4)	0.0003 (5)	0.0019 (4)	-0.0006 (5)
C2A	0.0143 (4)	0.0208 (6)	0.0171 (5)	-0.0007 (5)	-0.0005 (4)	-0.0006 (5)
C3A	0.0196 (5)	0.0191 (6)	0.0158 (4)	-0.0012 (5)	-0.0011 (4)	0.0003 (5)
C4A	0.0196 (5)	0.0229 (6)	0.0129 (4)	0.0003 (5)	0.0031 (4)	0.0006 (5)

C5A	0.0159 (5)	0.0229 (6)	0.0168 (5)	-0.0005 (5)	0.0007 (4)	0.0007 (5)
C6A	0.0163 (5)	0.0218 (6)	0.0134 (4)	0.0010 (5)	-0.0014 (3)	0.0004 (5)
C1B	0.0154 (5)	0.0172 (5)	0.0181 (5)	0.0001 (5)	0.0008 (4)	-0.0002 (5)
C2B	0.0170 (5)	0.0354 (7)	0.0200 (5)	-0.0001 (6)	-0.0023 (4)	0.0009 (5)
C3B	0.0174 (5)	0.0706 (12)	0.0235 (6)	-0.0024 (7)	-0.0037 (4)	0.0003 (7)
C4B	0.0225 (6)	0.0505 (9)	0.0164 (5)	-0.0006 (7)	-0.0022 (4)	-0.0003 (6)
C5B	0.0227 (5)	0.0320 (7)	0.0162 (5)	-0.0005 (6)	0.0039 (4)	0.0009 (5)
C6B	0.0252 (6)	0.0494 (9)	0.0302 (6)	-0.0014 (7)	0.0124 (5)	-0.0013 (7)

*Geometric parameters (Å, °)*

O1A—C1A	1.2523 (13)	N3B—C5B	1.3204 (15)
O21A—N2A	1.2190 (13)	N3B—C1B	1.3416 (14)
O22A—N2A	1.2284 (14)	C1A—C2A	1.4503 (15)
O41A—N4A	1.2287 (13)	C1A—C6A	1.4545 (16)
O42A—N4A	1.2205 (14)	C2A—C3A	1.3797 (15)
O61A—N6A	1.2357 (17)	C3A—C4A	1.3834 (16)
O62A—N6A	1.2182 (17)	C3A—H3AA	0.9500
O61B—N6A	1.221 (10)	C4A—C5A	1.3944 (15)
O62B—N6A	1.210 (9)	C5A—C6A	1.3685 (15)
O1B—C5B	1.3283 (14)	C5A—H5AA	0.9500
O1B—C6B	1.4418 (17)	C2B—C4B	1.3593 (17)
N2A—C2A	1.4605 (14)	C2B—C3B	1.4916 (18)
N4A—C4A	1.4477 (14)	C3B—H3BA	0.9800
N6A—C6A	1.4565 (14)	C3B—H3BB	0.9800
N1B—C1B	1.3210 (14)	C3B—H3BC	0.9800
N1B—H1BA	0.8800	C4B—C5B	1.4112 (18)
N1B—H1BB	0.8800	C4B—H4BA	0.9500
N2B—C2B	1.3654 (15)	C6B—H6BA	0.9800
N2B—C1B	1.3687 (14)	C6B—H6BB	0.9800
N2B—H2BA	0.8800	C6B—H6BC	0.9800
C5B—O1B—C6B	117.54 (10)	C3A—C4A—C5A	121.63 (10)
O21A—N2A—O22A	122.12 (11)	C3A—C4A—N4A	119.53 (10)
O21A—N2A—C2A	120.33 (10)	C5A—C4A—N4A	118.84 (10)
O22A—N2A—C2A	117.55 (10)	C6A—C5A—C4A	118.20 (10)
O42A—N4A—O41A	123.06 (10)	C6A—C5A—H5AA	120.9
O42A—N4A—C4A	118.35 (10)	C4A—C5A—H5AA	120.9
O41A—N4A—C4A	118.59 (10)	C5A—C6A—C1A	124.91 (10)
O62B—N6A—O62A	43.1 (8)	C5A—C6A—N6A	115.86 (10)
O62B—N6A—O61B	121.1 (6)	C1A—C6A—N6A	119.22 (9)
O62A—N6A—O61B	104.4 (6)	N1B—C1B—N3B	119.52 (10)
O62B—N6A—O61A	106.5 (6)	N1B—C1B—N2B	118.64 (9)
O62A—N6A—O61A	123.08 (13)	N3B—C1B—N2B	121.84 (10)
O61B—N6A—O61A	42.4 (9)	C4B—C2B—N2B	118.22 (11)
O62B—N6A—C6A	120.6 (5)	C4B—C2B—C3B	123.65 (11)
O62A—N6A—C6A	119.45 (12)	N2B—C2B—C3B	118.13 (11)
O61B—N6A—C6A	118.3 (4)	C2B—C3B—H3BA	109.5

O61A—N6A—C6A	117.40 (10)	C2B—C3B—H3BB	109.5
C1B—N1B—H1BA	120.0	H3BA—C3B—H3BB	109.5
C1B—N1B—H1BB	120.0	C2B—C3B—H3BC	109.5
H1BA—N1B—H1BB	120.0	H3BA—C3B—H3BC	109.5
C2B—N2B—C1B	121.33 (9)	H3BB—C3B—H3BC	109.5
C2B—N2B—H2BA	119.3	C2B—C4B—C5B	117.54 (11)
C1B—N2B—H2BA	119.3	C2B—C4B—H4BA	121.2
C5B—N3B—C1B	116.75 (10)	C5B—C4B—H4BA	121.2
O1A—C1A—C2A	124.65 (10)	N3B—C5B—O1B	119.64 (11)
O1A—C1A—C6A	123.03 (10)	N3B—C5B—C4B	124.31 (10)
C2A—C1A—C6A	112.30 (9)	O1B—C5B—C4B	116.05 (11)
C3A—C2A—C1A	123.31 (10)	O1B—C6B—H6BA	109.5
C3A—C2A—N2A	115.63 (10)	O1B—C6B—H6BB	109.5
C1A—C2A—N2A	121.06 (9)	H6BA—C6B—H6BB	109.5
C2A—C3A—C4A	119.64 (10)	O1B—C6B—H6BC	109.5
C2A—C3A—H3AA	120.2	H6BA—C6B—H6BC	109.5
C4A—C3A—H3AA	120.2	H6BB—C6B—H6BC	109.5
O1A—C1A—C2A—C3A	-178.06 (12)	C2A—C1A—C6A—N6A	178.84 (11)
C6A—C1A—C2A—C3A	0.43 (19)	O62B—N6A—C6A—C5A	-158.8 (10)
O1A—C1A—C2A—N2A	2.7 (2)	O62A—N6A—C6A—C5A	151.0 (2)
C6A—C1A—C2A—N2A	-178.78 (11)	O61B—N6A—C6A—C5A	22.2 (11)
O21A—N2A—C2A—C3A	-177.43 (13)	O61A—N6A—C6A—C5A	-26.1 (2)
O22A—N2A—C2A—C3A	2.02 (18)	O62B—N6A—C6A—C1A	21.3 (10)
O21A—N2A—C2A—C1A	1.83 (19)	O62A—N6A—C6A—C1A	-29.0 (2)
O22A—N2A—C2A—C1A	-178.72 (13)	O61B—N6A—C6A—C1A	-157.8 (11)
C1A—C2A—C3A—C4A	0.2 (2)	O61A—N6A—C6A—C1A	153.92 (16)
N2A—C2A—C3A—C4A	179.42 (12)	C5B—N3B—C1B—N1B	179.78 (12)
C2A—C3A—C4A—C5A	-0.2 (2)	C5B—N3B—C1B—N2B	-0.12 (19)
C2A—C3A—C4A—N4A	-179.57 (12)	C2B—N2B—C1B—N1B	-179.40 (12)
O42A—N4A—C4A—C3A	178.82 (13)	C2B—N2B—C1B—N3B	0.5 (2)
O41A—N4A—C4A—C3A	-1.17 (19)	C1B—N2B—C2B—C4B	-0.2 (2)
O42A—N4A—C4A—C5A	-0.59 (19)	C1B—N2B—C2B—C3B	179.57 (13)
O41A—N4A—C4A—C5A	179.42 (13)	N2B—C2B—C4B—C5B	-0.3 (2)
C3A—C4A—C5A—C6A	-0.5 (2)	C3B—C2B—C4B—C5B	179.86 (15)
N4A—C4A—C5A—C6A	178.91 (12)	C1B—N3B—C5B—O1B	179.54 (12)
C4A—C5A—C6A—C1A	1.2 (2)	C1B—N3B—C5B—C4B	-0.5 (2)
C4A—C5A—C6A—N6A	-178.78 (12)	C6B—O1B—C5B—N3B	-0.6 (2)
O1A—C1A—C6A—C5A	177.37 (13)	C6B—O1B—C5B—C4B	179.43 (14)
C2A—C1A—C6A—C5A	-1.14 (19)	C2B—C4B—C5B—N3B	0.8 (2)
O1A—C1A—C6A—N6A	-2.6 (2)	C2B—C4B—C5B—O1B	-179.30 (14)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1B—H1BA...O61B <sup>i</sup>	0.88	2.09	2.883 (10)	150
N1B—H1BA...O61A <sup>i</sup>	0.88	2.13	2.9309 (17)	151
N1B—H1BB...O1A	0.88	1.95	2.7223 (13)	146



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<i>N1B—H1BB···O21A</i>	0.88	2.20	2.8855 (14)	134
<i>N2B—H2BA···O1A</i>	0.88	1.97	2.7380 (12)	145
<i>N2B—H2BA···O62B</i>	0.88	2.55	3.303 (10)	144
<i>N2B—H2BA···O62A</i>	0.88	2.62	3.381 (2)	145

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Symmetry code: (i)  $x-1, y, z$ .