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4-Nitro-*N'*-[(*E*)-3-pyridylmethylidene]-benzohydrazide

Tanveer Ahmad,<sup>a</sup> Muhammad Zia-ur-Rehman,<sup>b\*</sup> Hamid Latif Siddiqui,<sup>a</sup> Shahid Mahmud<sup>b</sup> and Masood Parvez<sup>c</sup>

<sup>a</sup>Institute of Chemistry, University of the Punjab, Lahore 54590, Pakistan, <sup>b</sup>Applied Chemistry Research Centre, PCSIR Laboratories Complex, Lahore 54600, Pakistan, and <sup>c</sup>Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4  
Correspondence e-mail: rehman\_pcsir@hotmail.com

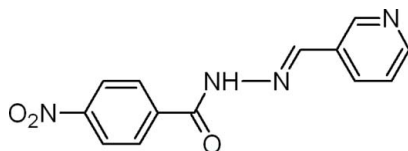
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Key indicators: single-crystal X-ray study; *T* = 123 K; mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$ ; disorder in main residue; *R* factor = 0.036; *wR* factor = 0.094; data-to-parameter ratio = 11.5.

In the title molecule,  $\text{C}_{13}\text{H}_{10}\text{N}_4\text{O}_3$ , the methyldene-hydrazide [ $-\text{C}(=\text{O})-\text{N}-\text{N}=\text{C}-$ ] fragment is essentially planar, with a maximum deviation of  $0.0228 (7) \text{ \AA}$ . The mean planes of the benzene and pyridine rings make dihedral angles of  $25.44 (6)$  and  $5.47 (7)^\circ$ , respectively, with the mean plane of the methyldene-hydrazide fragment. In the crystal structure, intermolecular  $\text{N}-\text{H} \cdots \text{N}$  hydrogen bonds link molecules into chains along the *b* axis. Additional stabilization is provided by weak intermolecular  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds. The O atoms of the nitro group are disordered over two sets of sites of equal occupancy.

Related literature

For the synthesis of related compounds, see: Zia-ur-Rehman *et al.* (2009). For the biological activity of benzohydrazides, see: Chakraborty & Patel (1996). For closely related structures, see: Raj *et al.* (2008); Fun *et al.* (2008); Wang *et al.* (2008); Qiu *et al.* (2009).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{10}\text{N}_4\text{O}_3$

$M_r = 270.25$

Monoclinic,  $P2_1/c$   
 $a = 14.6158 (3) \text{ \AA}$   
 $b = 8.1969 (2) \text{ \AA}$   
 $c = 10.3645 (2) \text{ \AA}$   
 $\beta = 100.609 (1)^\circ$   
 $V = 1220.49 (5) \text{ \AA}^3$

$Z = 4$   
Cu  $K\alpha$  radiation  
 $\mu = 0.91 \text{ mm}^{-1}$   
 $T = 123 \text{ K}$   
 $0.20 \times 0.16 \times 0.05 \text{ mm}$

Data collection

Bruker APEXII diffractometer  
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)  
 $T_{\text{min}} = 0.839$ ,  $T_{\text{max}} = 0.956$

10114 measured reflections  
2192 independent reflections  
2098 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.094$   
 $S = 1.06$   
2192 reflections  
190 parameters

66 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry ( $\text{ \AA}$ ,  $^\circ$ ).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
$\text{N2}-\text{H2N}\cdots\text{N4}^i$	0.88	2.09	2.9108 (14)	154
$\text{C8}-\text{H8}\cdots\text{O3}^{ii}$	0.95	2.50	3.1423 (14)	125
$\text{C11}-\text{H11}\cdots\text{O2}^{iii}$	0.95	2.49	3.364 (8)	152
$\text{C11}-\text{H11}\cdots\text{O2}^{iii}$	0.95	2.52	3.371 (8)	150
$\text{C13}-\text{H13}\cdots\text{O3}^{iv}$	0.95	2.57	3.1279 (14)	118

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5020).

References

- Bruker (2004). *APEX2, SAINT, XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Chakraborty, J. & Patel, R. N. (1996). *J. Indian Chem. Soc.* **73**, 191–195.  
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
Fun, H.-K., Jebas, S. R., Sujith, K. V. & Kalluraya, B. (2008). *Acta Cryst.* **E64**, o2377.  
Qiu, F., He, X.-J., Sun, Y.-X. & Zhu, X. (2009). *Acta Cryst.* **E65**, o2050.  
Raj, B. N. B., Kurup, M. R. P. & Suresh, E. (2008). *Spectrochim. Acta Part A*, **71**, 1253–1260.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Wang, Y.-Z., Wang, M.-D., Diao, Y.-P. & Cai, Q. (2008). *Acta Cryst.* **E64**, o668.  
Zia-ur-Rehman, M., Choudary, J. A., Elsegood, M. R. J., Siddiqui, H. L. & Khan, K. M. (2009). *Eur. J. Med. Chem.* **44**, 1311–1316.

**supplementary materials**

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## 4-Nitro-*N'*-[(*E*)-3-pyridylmethylidene]benzohydrazide

T. Ahmad, M. Zia-ur-Rehman, H. L. Siddiqui, S. Mahmud and M. Parvez

### Comment

Schiff bases are well known for their anti-bacterial, anti-oxidant, and anti-tumor activities (Zia-ur-Rehman *et al.*, 2009). These are also considered as popular ligands in coordination chemistry due to their ease of synthesis and their ability to be readily modified both electronically and sterically (Chakraborty & Patel, 1996). We have synthesized a novel Schiff base, by the condensation of pyridine-3-carbaldehyde with *p*-nitrobenzohydrazide, and determined its crystal structure which is presented in this paper.

The structure of the title compound is presented in Fig. 1. The bond distances and angles agree with the corresponding bond distances and angles reported in closely related compounds (Raj *et al.*, 2008; Fun *et al.*, 2008; Wang *et al.*, 2008; Qiu *et al.*, 2009). The methylidenehydrazide fragment C7/C8/N2/N3/O3 in the title compound is essentially planar with maximum deviation being 0.0228 (7) Å for both C7 and N2 atoms. The mean-planes of the benzene ring (C1–C6) and pyridine ring (C9–C13/N4) make dihedral angles of 25.44 (6) and 5.47 (7)°, respectively, with the mean-plane of the methylidene hydrazide fragment. The structure is stabilized by extensive hydrogen bonding; details have been provided in Table 1.

### Experimental

A mixture of para-nitrobenzohydrazide (0.5 g, 2.76 mmoles), pyridine-3-carbaldehyde (0.26 ml, 2.76 mmoles), orthophosphoric acid (0.2 ml) and methanol (50.0 ml) was heated to reflux for a period of 3.5 hours followed by removal of the solvent under vacuum. The contents were allowed to cool and washed with cold methanol to yield the title compound. Crystals suitable for X-ray crystallographic studies were grown from a methanol solution of the title compound at room temperature by slow evaporation. Yield: 92%. M.p. 547 K.

### Refinement

Though all the H atoms could be distinguished in the difference Fourier map the H-atoms bonded to C-atoms were included at geometrically idealized positions and refined in riding-model approximation with N—H = 0.88 Å and C—H = 0.95 Å; the  $U_{\text{iso}}(\text{H})$  were allowed at  $1.2U_{\text{eq}}(\text{C/N})$ . The final difference map was essentially featureless. The nitro group was disordered over two sites with N and O atoms occupying equal site occupancy factors, commands SIMU and EADP in SHELXL-97 (Sheldrick, 2008) were used to model the disorder.

### Figures

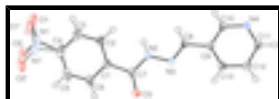


Fig. 1. The asymmetric unit of the title compound with the displacement ellipsoids plotted at 50% probability level (Farrugia, 1997); nitro group was disordered over two sites.

## 4-Nitro-*N*'-[(*E*)-3-pyridylmethylidene]benzohydrazide

### Crystal data

$C_{13}H_{10}N_4O_3$	$F(000) = 560$
$M_r = 270.25$	$D_x = 1.471 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Melting point: 547 K
Hall symbol: -P 2ybc	Cu $K\alpha$ radiation, $\lambda = 1.54178 \text{ \AA}$
$a = 14.6158 (3) \text{ \AA}$	Cell parameters from 10114 reflections
$b = 8.1969 (2) \text{ \AA}$	$\theta = 3.0\text{--}68.0^\circ$
$c = 10.3645 (2) \text{ \AA}$	$\mu = 0.91 \text{ mm}^{-1}$
$\beta = 100.609 (1)^\circ$	$T = 123 \text{ K}$
$V = 1220.49 (5) \text{ \AA}^3$	Plate, yellow
$Z = 4$	$0.20 \times 0.16 \times 0.05 \text{ mm}$

### Data collection

Bruker APEXII diffractometer	2192 independent reflections
Radiation source: fine-focus sealed tube graphite	2098 reflections with $I > 2\sigma(I)$
$\omega$ and $\varphi$ scans	$R_{\text{int}} = 0.016$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$\theta_{\text{max}} = 68.0^\circ$ , $\theta_{\text{min}} = 3.0^\circ$
$T_{\text{min}} = 0.839$ , $T_{\text{max}} = 0.956$	$h = -17 \rightarrow 17$
10114 measured reflections	$k = -9 \rightarrow 9$
	$l = -12 \rightarrow 12$

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.094$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.519P]$
2192 reflections	where $P = (F_o^2 + 2F_c^2)/3$
190 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
66 restraints	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	-0.0353 (11)	0.277 (2)	0.3850 (9)	0.0354 (16)	0.50
O1	-0.086 (3)	0.336 (5)	0.295 (3)	0.059 (3)	0.50
O2	-0.0552 (6)	0.1587 (11)	0.4450 (5)	0.0550 (12)	0.50
N1'	-0.0292 (11)	0.259 (2)	0.4237 (9)	0.0354 (16)	0.50
O1'	-0.082 (3)	0.311 (5)	0.321 (3)	0.059 (3)	0.50
O2'	-0.0525 (6)	0.1572 (11)	0.4997 (5)	0.0550 (12)	0.50
O3	0.36776 (6)	0.53420 (11)	0.67517 (8)	0.0267 (2)	
N2	0.37145 (6)	0.60961 (12)	0.46461 (9)	0.0179 (2)	
H2N	0.3442	0.6031	0.3817	0.022*	
N3	0.45517 (6)	0.69107 (12)	0.49965 (9)	0.0183 (2)	
N4	0.66815 (7)	1.02086 (12)	0.31200 (10)	0.0203 (2)	
C1	0.23641 (8)	0.46958 (15)	0.51171 (12)	0.0203 (3)	
C2	0.17661 (9)	0.52595 (17)	0.40038 (13)	0.0261 (3)	
H2	0.1965	0.6096	0.3484	0.031*	
C3	0.08809 (9)	0.46038 (19)	0.36515 (14)	0.0329 (3)	
H3	0.0466	0.4988	0.2898	0.040*	
C4	0.06168 (9)	0.33806 (18)	0.44215 (16)	0.0352 (4)	
C5	0.11925 (10)	0.27997 (17)	0.55309 (16)	0.0361 (4)	
H5	0.0992	0.1953	0.6040	0.043*	
C6	0.20687 (9)	0.34797 (16)	0.58836 (14)	0.0280 (3)	
H6	0.2472	0.3114	0.6655	0.034*	
C7	0.33183 (8)	0.54003 (14)	0.55923 (11)	0.0189 (3)	
C8	0.48376 (8)	0.75796 (14)	0.40288 (11)	0.0182 (3)	
H8	0.4490	0.7430	0.3168	0.022*	
C9	0.56840 (8)	0.85680 (14)	0.42110 (11)	0.0179 (3)	
C10	0.59341 (8)	0.92553 (15)	0.30967 (11)	0.0191 (3)	
H10	0.5552	0.9035	0.2270	0.023*	
C11	0.72083 (8)	1.05187 (15)	0.42965 (12)	0.0212 (3)	
H11	0.7744	1.1186	0.4334	0.025*	
C12	0.70067 (8)	0.99095 (15)	0.54626 (12)	0.0219 (3)	
H12	0.7395	1.0168	0.6277	0.026*	
C13	0.62364 (8)	0.89241 (15)	0.54267 (11)	0.0201 (3)	
H13	0.6085	0.8497	0.6213	0.024*	

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0219 (18)	0.035 (3)	0.051 (5)	-0.0047 (17)	0.010 (4)	-0.013 (4)
O1	0.027 (3)	0.086 (9)	0.059 (8)	-0.015 (4)	-0.004 (5)	-0.022 (6)

## supplementary materials

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O2	0.0311 (8)	0.0435 (8)	0.093 (4)	-0.0147 (6)	0.018 (3)	0.007 (3)
N1'	0.0219 (18)	0.035 (3)	0.051 (5)	-0.0047 (17)	0.010 (4)	-0.013 (4)
O1'	0.027 (3)	0.086 (9)	0.059 (8)	-0.015 (4)	-0.004 (5)	-0.022 (6)
O2'	0.0311 (8)	0.0435 (8)	0.093 (4)	-0.0147 (6)	0.018 (3)	0.007 (3)
O3	0.0277 (5)	0.0329 (5)	0.0186 (4)	-0.0062 (4)	0.0022 (3)	0.0030 (4)
N2	0.0158 (5)	0.0208 (5)	0.0168 (5)	-0.0024 (4)	0.0021 (4)	-0.0006 (4)
N3	0.0156 (5)	0.0187 (5)	0.0205 (5)	-0.0013 (4)	0.0030 (4)	-0.0014 (4)
N4	0.0184 (5)	0.0215 (5)	0.0215 (5)	0.0005 (4)	0.0053 (4)	0.0010 (4)
C1	0.0192 (6)	0.0194 (6)	0.0240 (6)	-0.0001 (4)	0.0080 (5)	-0.0052 (5)
C2	0.0207 (6)	0.0318 (7)	0.0263 (6)	-0.0023 (5)	0.0058 (5)	-0.0034 (5)
C3	0.0207 (6)	0.0429 (8)	0.0345 (7)	-0.0011 (6)	0.0032 (5)	-0.0121 (6)
C4	0.0184 (6)	0.0315 (7)	0.0579 (9)	-0.0057 (5)	0.0130 (6)	-0.0211 (7)
C5	0.0293 (7)	0.0217 (7)	0.0631 (10)	-0.0037 (5)	0.0234 (7)	-0.0006 (7)
C6	0.0252 (6)	0.0227 (6)	0.0387 (7)	0.0003 (5)	0.0126 (5)	0.0015 (6)
C7	0.0197 (6)	0.0167 (6)	0.0206 (6)	0.0005 (4)	0.0045 (4)	-0.0013 (5)
C8	0.0178 (5)	0.0181 (6)	0.0182 (5)	0.0010 (4)	0.0021 (4)	-0.0005 (4)
C9	0.0170 (6)	0.0163 (6)	0.0206 (6)	0.0023 (4)	0.0040 (4)	-0.0012 (4)
C10	0.0180 (6)	0.0196 (6)	0.0193 (6)	0.0012 (4)	0.0024 (4)	-0.0013 (5)
C11	0.0161 (5)	0.0207 (6)	0.0271 (6)	-0.0016 (4)	0.0045 (5)	-0.0013 (5)
C12	0.0198 (6)	0.0235 (6)	0.0212 (6)	-0.0003 (5)	0.0008 (5)	-0.0029 (5)
C13	0.0208 (6)	0.0210 (6)	0.0191 (6)	0.0016 (5)	0.0050 (5)	0.0004 (5)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—O1	1.19 (3)	C2—H2	0.9500
N1—O2	1.213 (16)	C3—C4	1.380 (2)
N1—C4	1.518 (16)	C3—H3	0.9500
N1'—O2'	1.236 (15)	C4—C5	1.379 (2)
N1'—O1'	1.27 (2)	C5—C6	1.383 (2)
N1'—C4	1.460 (17)	C5—H5	0.9500
O3—C7	1.2205 (15)	C6—H6	0.9500
N2—C7	1.3532 (15)	C8—C9	1.4617 (16)
N2—N3	1.3826 (13)	C8—H8	0.9500
N2—H2N	0.8800	C9—C10	1.3929 (16)
N3—C8	1.2788 (15)	C9—C13	1.3964 (16)
N4—C10	1.3397 (16)	C10—H10	0.9500
N4—C11	1.3411 (16)	C11—C12	1.3886 (17)
C1—C6	1.3919 (18)	C11—H11	0.9500
C1—C2	1.3920 (18)	C12—C13	1.3806 (17)
C1—C7	1.5061 (16)	C12—H12	0.9500
C2—C3	1.3865 (18)	C13—H13	0.9500
O1—N1—O2	124 (2)	C4—C5—H5	120.8
O1—N1—C4	125 (2)	C6—C5—H5	120.8
O2—N1—C4	111.0 (8)	C5—C6—C1	120.48 (13)
O2'—N1'—O1'	125 (2)	C5—C6—H6	119.8
O2'—N1'—C4	124.5 (9)	C1—C6—H6	119.8
O1'—N1'—C4	111 (2)	O3—C7—N2	124.48 (11)
C7—N2—N3	119.29 (9)	O3—C7—C1	120.77 (11)
C7—N2—H2N	120.4	N2—C7—C1	114.75 (10)

N3—N2—H2N	120.4	N3—C8—C9	121.82 (10)
C8—N3—N2	113.72 (9)	N3—C8—H8	119.1
C10—N4—C11	117.17 (10)	C9—C8—H8	119.1
C6—C1—C2	119.84 (12)	C10—C9—C13	117.86 (11)
C6—C1—C7	116.99 (11)	C10—C9—C8	117.52 (10)
C2—C1—C7	123.10 (11)	C13—C9—C8	124.57 (11)
C3—C2—C1	120.18 (13)	N4—C10—C9	123.95 (11)
C3—C2—H2	119.9	N4—C10—H10	118.0
C1—C2—H2	119.9	C9—C10—H10	118.0
C4—C3—C2	118.43 (14)	N4—C11—C12	123.02 (11)
C4—C3—H3	120.8	N4—C11—H11	118.5
C2—C3—H3	120.8	C12—C11—H11	118.5
C5—C4—C3	122.72 (12)	C13—C12—C11	119.31 (11)
C5—C4—N1'	110.9 (4)	C13—C12—H12	120.3
C3—C4—N1'	126.3 (4)	C11—C12—H12	120.3
C5—C4—N1	126.4 (4)	C12—C13—C9	118.67 (11)
C3—C4—N1	110.8 (4)	C12—C13—H13	120.7
C4—C5—C6	118.33 (13)	C9—C13—H13	120.7
C7—N2—N3—C8	-177.02 (10)	N1—C4—C5—C6	178.1 (7)
C6—C1—C2—C3	-0.52 (19)	C4—C5—C6—C1	-1.3 (2)
C7—C1—C2—C3	-177.23 (11)	C2—C1—C6—C5	1.50 (19)
C1—C2—C3—C4	-0.57 (19)	C7—C1—C6—C5	178.40 (11)
C2—C3—C4—C5	0.7 (2)	N3—N2—C7—O3	-5.01 (17)
C2—C3—C4—N1'	177.6 (8)	N3—N2—C7—C1	174.08 (9)
C2—C3—C4—N1	-177.4 (6)	C6—C1—C7—O3	-23.95 (17)
O2'—N1'—C4—C5	2.3 (16)	C2—C1—C7—O3	152.85 (12)
O1'—N1'—C4—C5	-180 (2)	C6—C1—C7—N2	156.92 (11)
O2'—N1'—C4—C3	-174.9 (10)	C2—C1—C7—N2	-26.28 (17)
O1'—N1'—C4—C3	3(3)	N2—N3—C8—C9	176.74 (10)
O2'—N1'—C4—N1	168 (6)	N3—C8—C9—C10	-179.54 (11)
O1'—N1'—C4—N1	-14 (4)	N3—C8—C9—C13	-2.28 (18)
O1—N1—C4—C5	174 (3)	C11—N4—C10—C9	-0.60 (17)
O2—N1—C4—C5	-4.7 (15)	C13—C9—C10—N4	1.38 (18)
O1—N1—C4—C3	-8(3)	C8—C9—C10—N4	178.82 (10)
O2—N1—C4—C3	173.3 (9)	C10—N4—C11—C12	-0.44 (17)
O1—N1—C4—N1'	158 (7)	N4—C11—C12—C13	0.65 (18)
O2—N1—C4—N1'	-21 (4)	C11—C12—C13—C9	0.17 (18)
C3—C4—C5—C6	0.2 (2)	C10—C9—C13—C12	-1.11 (17)
N1'—C4—C5—C6	-177.1 (7)	C8—C9—C13—C12	-178.35 (11)

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>
N2—H2N...N4 <sup>i</sup>	0.88	2.09	2.9108 (14)	154.
C3—H3...O1	0.95	2.37	2.72 (4)	101.
C5—H5...O2'	0.95	2.30	2.666 (8)	102.
C8—H8...O3 <sup>ii</sup>	0.95	2.50	3.1423 (14)	125.
C11—H11...O2 <sup>iii</sup>	0.95	2.49	3.364 (8)	152.

## supplementary materials

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C11—H11…O2 <sup>iii</sup>	0.95	2.52	3.371 (8)	150.
C13—H13…O3 <sup>iv</sup>	0.95	2.57	3.1279 (14)	118.

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



Fig. 1

