

## 4-[(2-Benzoyl-4-chlorophenyl)diazenyl]-3-methyl-1-phenyl-1H-pyrazol-5(4H)-one

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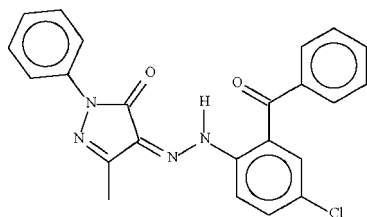
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.105; data-to-parameter ratio = 15.7.

In the title compound,  $\text{C}_{23}\text{H}_{17}\text{ClN}_4\text{O}_2$ , the amino H atom forms an intramolecular hydrogen bond to the exocyclic carbonyl O atom as well as to the O atom of the benzoyl group.

### Related literature

For the crystal structure of 1-phenyl-3-methyl-4-(4'-chlorophenyl)-pyrazol-5-one, whose amino H atom is intramolecularly hydrogen-bonded to the carbonyl O atom, see: Golinski *et al.* (1983).



### Experimental

#### Crystal data

$\text{C}_{23}\text{H}_{17}\text{ClN}_4\text{O}_2$   
 $M_r = 416.86$

Monoclinic,  $C2/c$   
 $a = 25.800$  (3) Å

$b = 12.124$  (1) Å  
 $c = 13.966$  (1) Å  
 $\beta = 119.179$  (1)°  
 $V = 3813.9$  (7) Å<sup>3</sup>  
 $Z = 8$

Mo  $K\alpha$  radiation  
 $\mu = 0.23$  mm<sup>-1</sup>  
 $T = 120$  (2) K  
 $0.35 \times 0.25 \times 0.10$  mm

#### Data collection

Bruker SMART APEX diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.791$ ,  $T_{\max} = 0.862$   
(expected range = 0.897–0.977)

10753 measured reflections  
4346 independent reflections  
3309 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.032$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.105$   
 $S = 1.00$   
4346 reflections  
276 parameters  
1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.30$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N4}-\text{H4}\cdots\text{O1}$	0.89 (1)	2.06 (2)	2.755 (2)	135 (2)
$\text{N4}-\text{H4}\cdots\text{O2}$	0.89 (1)	2.05 (2)	2.698 (2)	130 (2)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

We thank the Iranian Research Organization for Science and Technology and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2371).

### References

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**supplementary materials**

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## 4-[(2-Benzoyl-4-chlorophenyl)diazenyl]-3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one

Z. Bahreni, H. Rahmani and S. W. Ng

### Comment

(type here to add)

### Experimental

2-Amino-5-chlorobenzophenone (2.32 g, 0.01 mol) was suspended in strong hydrochloric acid (20 ml, pH ca. 5) at 273 K. A solution of sodium nitrite (0.69 g in 15 ml water) was added. Following the diazotization, an aqueous solution of 3-methyl-1-phenyl-2-pyrazoline-5-one (1.75 g, 0.01 mol) was added. The compound that separated was collected and recrystallized from ethanol.

### Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to 1.2 to 1.5 $U(\text{C})$ .

The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N—H 0.85±0.01 Å; its temperature factors was freely refined.

### Figures

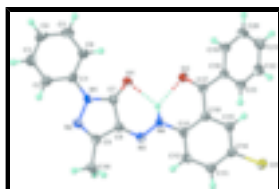


Fig. 1. Thermal displacement plot (Barbour, 2001) of  $\text{C}_{23}\text{H}_{17}\text{ClN}_4\text{O}_2$ ; probability levels are set at 70% and H-atoms are drawn as spheres of arbitrary radius.

## 4-[(2-Benzoyl-4-chlorophenyl)diazenyl]-3-methyl-1-phenyl-1*H*-pyrazol- 5(4*H*)-one

### Crystal data

$\text{C}_{23}\text{H}_{17}\text{ClN}_4\text{O}_2$

$M_r = 416.86$

Monoclinic,  $C2/c$

Hall symbol:  $-C 2yc$

$a = 25.800(3) \text{ \AA}$

$b = 12.124(1) \text{ \AA}$

$c = 13.966(1) \text{ \AA}$

$F_{000} = 1728$

$D_x = 1.452 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2643 reflections

$\theta = 2.2\text{--}26.9^\circ$

$\mu = 0.23 \text{ mm}^{-1}$

$T = 120 \text{ K}$

# supplementary materials

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$\beta = 119.179 (1)^\circ$  Block, orange  
 $V = 3813.9 (7) \text{ \AA}^3$   $0.35 \times 0.25 \times 0.10 \text{ mm}$   
 $Z = 8$

## Data collection

Bruker SMART APEX diffractometer	4346 independent reflections
Radiation source: fine-focus sealed tube	3309 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.032$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
$\omega$ scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -33 \rightarrow 33$
$T_{\text{min}} = 0.791$ , $T_{\text{max}} = 0.862$	$k = -15 \rightarrow 12$
10753 measured reflections	$l = -18 \rightarrow 17$

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 2.1067P]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
4346 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
276 parameters	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{Å}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.176742 (18)	0.72052 (4)	0.10069 (4)	0.03104 (13)
O1	0.51920 (5)	0.65437 (10)	0.64529 (9)	0.0270 (3)
O2	0.42743 (6)	0.81878 (10)	0.49190 (10)	0.0301 (3)
N1	0.56804 (6)	0.48796 (11)	0.72365 (11)	0.0209 (3)
N2	0.55349 (6)	0.37433 (11)	0.70777 (11)	0.0208 (3)
N3	0.42128 (6)	0.49431 (11)	0.50462 (11)	0.0200 (3)
N4	0.40777 (6)	0.59936 (12)	0.47918 (11)	0.0204 (3)
H4	0.4336 (7)	0.6518 (13)	0.5167 (15)	0.037 (6)*
C1	0.62606 (7)	0.51899 (15)	0.80413 (13)	0.0214 (4)
C2	0.66295 (7)	0.44153 (15)	0.88027 (13)	0.0239 (4)
H2	0.6489	0.3691	0.8799	0.029*
C3	0.72042 (8)	0.47068 (16)	0.95664 (14)	0.0279 (4)

H3	0.7458	0.4175	1.0082	0.034*
C4	0.74136 (8)	0.57598 (16)	0.95878 (15)	0.0307 (4)
H4A	0.7810	0.5951	1.0108	0.037*
C5	0.70394 (8)	0.65311 (16)	0.88431 (15)	0.0310 (4)
H5	0.7179	0.7261	0.8866	0.037*
C6	0.64650 (8)	0.62588 (15)	0.80647 (14)	0.0270 (4)
H6	0.6213	0.6794	0.7552	0.032*
C7	0.52131 (7)	0.55349 (14)	0.65290 (13)	0.0204 (3)
C8	0.47428 (7)	0.47380 (14)	0.58702 (13)	0.0195 (3)
C9	0.49904 (7)	0.36666 (14)	0.62833 (13)	0.0201 (3)
C10	0.46871 (7)	0.25862 (14)	0.59029 (15)	0.0246 (4)
H10A	0.4962	0.1992	0.6323	0.037*
H10B	0.4559	0.2492	0.5122	0.037*
H10C	0.4340	0.2562	0.6012	0.037*
C11	0.35288 (7)	0.62787 (14)	0.38933 (13)	0.0198 (3)
C12	0.30754 (7)	0.55002 (14)	0.34178 (13)	0.0226 (4)
H12	0.3140	0.4770	0.3700	0.027*
C13	0.25322 (7)	0.57835 (15)	0.25382 (14)	0.0241 (4)
H13	0.2221	0.5255	0.2224	0.029*
C14	0.24456 (7)	0.68414 (15)	0.21197 (13)	0.0221 (4)
C15	0.28903 (7)	0.76156 (14)	0.25581 (13)	0.0213 (3)
H15	0.2822	0.8336	0.2252	0.026*
C16	0.34421 (7)	0.73499 (14)	0.34516 (13)	0.0200 (3)
C17	0.39169 (7)	0.82058 (14)	0.39359 (13)	0.0210 (3)
C18	0.39651 (7)	0.90830 (14)	0.32340 (13)	0.0205 (3)
C19	0.42522 (7)	1.00593 (14)	0.37423 (14)	0.0251 (4)
H19	0.4377	1.0172	0.4497	0.030*
C20	0.43574 (8)	1.08644 (15)	0.31600 (15)	0.0298 (4)
H20	0.4549	1.1531	0.3513	0.036*
C21	0.41840 (8)	1.07047 (15)	0.20623 (15)	0.0299 (4)
H21	0.4257	1.1259	0.1662	0.036*
C22	0.39037 (8)	0.97341 (15)	0.15512 (14)	0.0273 (4)
H22	0.3787	0.9623	0.0799	0.033*
C23	0.37924 (7)	0.89228 (14)	0.21274 (13)	0.0230 (4)
H23	0.3599	0.8259	0.1771	0.028*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0196 (2)	0.0284 (3)	0.0321 (2)	0.00060 (17)	0.00242 (17)	0.00243 (19)
O1	0.0295 (7)	0.0183 (7)	0.0261 (6)	-0.0014 (5)	0.0080 (5)	0.0012 (5)
O2	0.0339 (7)	0.0233 (7)	0.0217 (6)	-0.0045 (5)	0.0045 (5)	-0.0012 (5)
N1	0.0219 (7)	0.0173 (7)	0.0210 (7)	-0.0018 (6)	0.0086 (6)	0.0006 (5)
N2	0.0209 (7)	0.0177 (7)	0.0238 (7)	-0.0010 (6)	0.0109 (6)	0.0014 (6)
N3	0.0219 (7)	0.0200 (8)	0.0204 (7)	0.0008 (6)	0.0121 (6)	0.0028 (6)
N4	0.0210 (7)	0.0181 (8)	0.0195 (7)	-0.0015 (6)	0.0079 (6)	0.0013 (6)
C1	0.0192 (8)	0.0252 (9)	0.0193 (8)	-0.0022 (7)	0.0089 (7)	-0.0021 (7)
C2	0.0247 (9)	0.0248 (10)	0.0229 (8)	-0.0011 (7)	0.0121 (7)	-0.0003 (7)

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C3	0.0263 (9)	0.0311 (10)	0.0233 (9)	0.0028 (8)	0.0097 (7)	0.0006 (8)
C4	0.0226 (9)	0.0373 (11)	0.0264 (9)	-0.0057 (8)	0.0073 (7)	-0.0078 (8)
C5	0.0309 (10)	0.0271 (10)	0.0325 (10)	-0.0085 (8)	0.0133 (8)	-0.0061 (8)
C6	0.0272 (9)	0.0239 (10)	0.0270 (9)	-0.0015 (7)	0.0109 (7)	0.0012 (7)
C7	0.0229 (8)	0.0209 (9)	0.0180 (8)	0.0010 (7)	0.0105 (7)	0.0024 (6)
C8	0.0209 (8)	0.0204 (9)	0.0184 (8)	-0.0010 (6)	0.0106 (7)	0.0017 (6)
C9	0.0193 (8)	0.0224 (9)	0.0205 (8)	0.0006 (7)	0.0111 (7)	0.0029 (7)
C10	0.0216 (8)	0.0198 (9)	0.0295 (9)	-0.0019 (7)	0.0102 (7)	-0.0006 (7)
C11	0.0184 (8)	0.0223 (9)	0.0185 (8)	0.0008 (6)	0.0090 (7)	-0.0007 (6)
C12	0.0245 (8)	0.0191 (9)	0.0251 (9)	-0.0001 (7)	0.0129 (7)	0.0036 (7)
C13	0.0211 (8)	0.0218 (9)	0.0276 (9)	-0.0047 (7)	0.0106 (7)	-0.0006 (7)
C14	0.0157 (8)	0.0261 (9)	0.0208 (8)	0.0021 (7)	0.0062 (6)	0.0000 (7)
C15	0.0214 (8)	0.0180 (9)	0.0233 (8)	0.0026 (6)	0.0100 (7)	0.0007 (7)
C16	0.0211 (8)	0.0193 (9)	0.0205 (8)	0.0003 (7)	0.0107 (7)	-0.0011 (6)
C17	0.0204 (8)	0.0178 (8)	0.0212 (8)	0.0024 (6)	0.0074 (7)	-0.0016 (7)
C18	0.0168 (7)	0.0180 (9)	0.0229 (8)	0.0016 (6)	0.0067 (6)	0.0008 (7)
C19	0.0231 (8)	0.0214 (9)	0.0248 (9)	-0.0009 (7)	0.0070 (7)	-0.0036 (7)
C20	0.0294 (9)	0.0188 (9)	0.0353 (10)	-0.0049 (7)	0.0110 (8)	-0.0018 (8)
C21	0.0300 (10)	0.0241 (10)	0.0348 (10)	-0.0013 (8)	0.0151 (8)	0.0053 (8)
C22	0.0266 (9)	0.0275 (10)	0.0247 (9)	0.0021 (7)	0.0099 (7)	0.0021 (7)
C23	0.0207 (8)	0.0192 (9)	0.0247 (9)	-0.0007 (7)	0.0076 (7)	-0.0019 (7)

### *Geometric parameters (Å, °)*

C11—C14	1.738 (2)	C10—H10A	0.9800
O1—C7	1.227 (2)	C10—H10B	0.9800
O2—C17	1.225 (2)	C10—H10C	0.9800
N1—C7	1.378 (2)	C11—C12	1.394 (2)
N1—C1	1.416 (2)	C11—C16	1.408 (2)
N1—N2	1.417 (2)	C12—C13	1.383 (2)
N2—C9	1.301 (2)	C12—H12	0.9500
N3—C8	1.312 (2)	C13—C14	1.382 (2)
N3—N4	1.323 (2)	C13—H13	0.9500
N4—C11	1.402 (2)	C14—C15	1.374 (2)
N4—H4	0.885 (9)	C15—C16	1.398 (2)
C1—C2	1.390 (2)	C15—H15	0.9500
C1—C6	1.393 (2)	C16—C17	1.492 (2)
C2—C3	1.385 (2)	C17—C18	1.492 (2)
C2—H2	0.9500	C18—C19	1.393 (2)
C3—C4	1.381 (3)	C18—C23	1.397 (2)
C3—H3	0.9500	C19—C20	1.380 (3)
C4—C5	1.382 (3)	C19—H19	0.9500
C4—H4A	0.9500	C20—C21	1.385 (3)
C5—C6	1.384 (3)	C20—H20	0.9500
C5—H5	0.9500	C21—C22	1.383 (3)
C6—H6	0.9500	C21—H21	0.9500
C7—C8	1.471 (2)	C22—C23	1.387 (2)
C8—C9	1.438 (2)	C22—H22	0.9500
C9—C10	1.485 (2)	C23—H23	0.9500

C7—N1—C1	129.3 (2)	H10B—C10—H10C	109.5
C7—N1—N2	112.1 (1)	C12—C11—N4	120.4 (2)
C1—N1—N2	118.7 (1)	C12—C11—C16	119.9 (2)
C9—N2—N1	107.2 (1)	N4—C11—C16	119.7 (1)
C8—N3—N4	116.3 (1)	C13—C12—C11	120.4 (2)
N3—N4—C11	119.8 (1)	C13—C12—H12	119.8
N3—N4—H4	121 (1)	C11—C12—H12	119.8
C11—N4—H4	120 (1)	C12—C13—C14	119.4 (2)
C2—C1—C6	120.0 (2)	C12—C13—H13	120.3
C2—C1—N1	119.5 (2)	C14—C13—H13	120.3
C6—C1—N1	120.5 (2)	C15—C14—C13	121.2 (2)
C3—C2—C1	119.6 (2)	C15—C14—C11	118.9 (1)
C3—C2—H2	120.2	C13—C14—C11	119.9 (1)
C1—C2—H2	120.2	C14—C15—C16	120.4 (2)
C4—C3—C2	120.9 (2)	C14—C15—H15	119.8
C4—C3—H3	119.6	C16—C15—H15	119.8
C2—C3—H3	119.6	C15—C16—C11	118.6 (2)
C3—C4—C5	119.2 (2)	C15—C16—C17	119.9 (2)
C3—C4—H4A	120.4	C11—C16—C17	121.5 (2)
C5—C4—H4A	120.4	O2—C17—C18	119.4 (2)
C4—C5—C6	121.2 (2)	O2—C17—C16	119.9 (2)
C4—C5—H5	119.4	C18—C17—C16	120.7 (1)
C6—C5—H5	119.4	C19—C18—C23	119.0 (2)
C5—C6—C1	119.2 (2)	C19—C18—C17	117.7 (2)
C5—C6—H6	120.4	C23—C18—C17	122.9 (2)
C1—C6—H6	120.4	C20—C19—C18	120.6 (2)
O1—C7—N1	128.6 (2)	C20—C19—H19	119.7
O1—C7—C8	127.8 (2)	C18—C19—H19	119.7
N1—C7—C8	103.7 (1)	C19—C20—C21	120.2 (2)
N3—C8—C9	126.3 (2)	C19—C20—H20	119.9
N3—C8—C7	127.8 (2)	C21—C20—H20	119.9
C9—C8—C7	105.8 (1)	C20—C21—C22	119.7 (2)
N2—C9—C8	111.2 (2)	C20—C21—H21	120.1
N2—C9—C10	122.0 (2)	C22—C21—H21	120.1
C8—C9—C10	126.8 (2)	C21—C22—C23	120.5 (2)
C9—C10—H10A	109.5	C21—C22—H22	119.7
C9—C10—H10B	109.5	C23—C22—H22	119.7
H10A—C10—H10B	109.5	C22—C23—C18	119.9 (2)
C9—C10—H10C	109.5	C22—C23—H23	120.0
H10A—C10—H10C	109.5	C18—C23—H23	120.0
C7—N1—N2—C9	0.5 (2)	N3—N4—C11—C12	14.3 (2)
C1—N1—N2—C9	-178.1 (1)	N3—N4—C11—C16	-164.0 (1)
C8—N3—N4—C11	177.4 (1)	N4—C11—C12—C13	179.4 (2)
C7—N1—C1—C2	168.3 (2)	C16—C11—C12—C13	-2.4 (2)
N2—N1—C1—C2	-13.4 (2)	C11—C12—C13—C14	1.2 (3)
C7—N1—C1—C6	-12.6 (3)	C12—C13—C14—C15	0.3 (3)
N2—N1—C1—C6	165.8 (2)	C12—C13—C14—C11	179.4 (1)
C6—C1—C2—C3	-1.4 (3)	C13—C14—C15—C16	-0.7 (3)
N1—C1—C2—C3	177.8 (2)	C11—C14—C15—C16	-179.8 (1)

## supplementary materials

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C1—C2—C3—C4	0.6 (3)	C14—C15—C16—C11	-0.5 (2)
C2—C3—C4—C5	0.8 (3)	C14—C15—C16—C17	-178.6 (2)
C3—C4—C5—C6	-1.4 (3)	C12—C11—C16—C15	2.0 (2)
C4—C5—C6—C1	0.6 (3)	N4—C11—C16—C15	-179.8 (1)
C2—C1—C6—C5	0.8 (3)	C12—C11—C16—C17	-179.9 (2)
N1—C1—C6—C5	-178.4 (2)	N4—C11—C16—C17	-1.6 (2)
C1—N1—C7—O1	-1.6 (3)	C15—C16—C17—O2	148.1 (2)
N2—N1—C7—O1	180.0 (2)	C11—C16—C17—O2	-30.0 (2)
C1—N1—C7—C8	177.7 (2)	C15—C16—C17—C18	-32.3 (2)
N2—N1—C7—C8	-0.7 (2)	C11—C16—C17—C18	149.6 (2)
N4—N3—C8—C9	-179.0 (2)	O2—C17—C18—C19	-23.7 (2)
N4—N3—C8—C7	-1.8 (2)	C16—C17—C18—C19	156.7 (2)
O1—C7—C8—N3	2.3 (3)	O2—C17—C18—C23	149.5 (2)
N1—C7—C8—N3	-177.0 (2)	C16—C17—C18—C23	-30.1 (2)
O1—C7—C8—C9	180.0 (2)	C23—C18—C19—C20	0.9 (3)
N1—C7—C8—C9	0.6 (2)	C17—C18—C19—C20	174.4 (2)
N1—N2—C9—C8	-0.1 (2)	C18—C19—C20—C21	-0.7 (3)
N1—N2—C9—C10	-179.3 (1)	C19—C20—C21—C22	0.1 (3)
N3—C8—C9—N2	177.3 (2)	C20—C21—C22—C23	0.3 (3)
C7—C8—C9—N2	-0.4 (2)	C21—C22—C23—C18	-0.1 (3)
N3—C8—C9—C10	-3.5 (3)	C19—C18—C23—C22	-0.5 (2)
C7—C8—C9—C10	178.8 (2)	C17—C18—C23—C22	-173.6 (2)

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

<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>
N4—H4 $\cdots$ O1	0.89 (1)	2.06 (2)	2.755 (2)	135 (2)
N4—H4 $\cdots$ O2	0.89 (1)	2.05 (2)	2.698 (2)	130 (2)



Fig. 1

