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## Structure Reports

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# 1,3-Dimethyl-4-phenylsulfanyl-1H-pyrazol-5-ol

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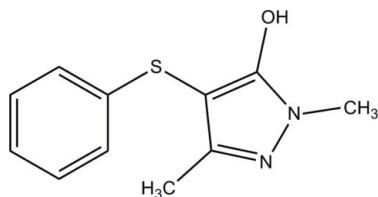
Received 8 February 2011; accepted 9 February 2011

 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.104; data-to-parameter ratio = 21.3.

In the title compound,  $\text{C}_{11}\text{H}_{12}\text{N}_2\text{OS}$ , the pyrazole ring makes a dihedral angle of  $85.40(8)^\circ$  with the phenyl ring. In the crystal, intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds link molecules into a two-dimensional network parallel to the  $bc$  plane.

## Related literature

For pyrazole derivatives and their microbial activity, see: Ragavan *et al.* (2009, 2010). For related structures, see: Shahani *et al.* (2009, 2010*a,b,c*). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



## Experimental

### Crystal data

 $\text{C}_{11}\text{H}_{12}\text{N}_2\text{OS}$ 
 $M_r = 220.30$ 

 Orthorhombic,  $Pbca$ 
 $a = 10.9479(2)$  Å

 $b = 11.3470(3)$  Å

 $c = 17.7392(4)$  Å

 $V = 2203.67(9)$  Å<sup>3</sup>
 $Z = 8$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.27$  mm<sup>-1</sup>
 $T = 100$  K

 $0.33 \times 0.13 \times 0.11$  mm

### Data collection

 Bruker SMART APEXII CCD  
 area-detector diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2009)  
 $T_{\min} = 0.917$ ,  $T_{\max} = 0.971$ 

 12209 measured reflections  
 3027 independent reflections  
 2406 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.041$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$ 
 $wR(F^2) = 0.104$ 
 $S = 1.04$ 

3027 reflections

142 parameters

 H atoms treated by a mixture of  
 independent and constrained  
 refinement

 $\Delta\rho_{\text{max}} = 0.33$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.31$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\text{N1}\cdots\text{O1}^{\text{i}}$	0.94 (2)	1.71 (2)	2.6446 (16)	173 (2)
$\text{C3}-\text{H3A}\cdots\text{O1}^{\text{ii}}$	0.93	2.53	3.2549 (19)	135

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: IS2676).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Ragavan, R. V., Vijayakumar, V. & Kumari, N. S. (2009). *Eur. J. Med. Chem.* **44**, 3852–3857.
- Ragavan, R. V., Vijayakumar, V. & Kumari, N. S. (2010). *Eur. J. Med. Chem.* **45**, 1173–1180.
- Shahani, T., Fun, H.-K., Ragavan, R. V., Vijayakumar, V. & Sarveswari, S. (2009). *Acta Cryst.* **E65**, o3249–o3250.
- Shahani, T., Fun, H.-K., Ragavan, R. V., Vijayakumar, V. & Sarveswari, S. (2010*a*). *Acta Cryst.* **E66**, o142–o143.
- Shahani, T., Fun, H.-K., Ragavan, R. V., Vijayakumar, V. & Sarveswari, S. (2010*b*). *Acta Cryst.* **E66**, o1357–o1358.
- Shahani, T., Fun, H.-K., Ragavan, R. V., Vijayakumar, V. & Sarveswari, S. (2010*c*). *Acta Cryst.* **E66**, o1482–o1483.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

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## supporting information

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**1,3-Dimethyl-4-phenylsulfanyl-1*H*-pyrazol-5-ol**

Tara Shahani, Hoong-Kun Fun, R. Venkat Ragavan, V. Vijayakumar and S. Sarveswari

**S1. Comment**

Antibacterial and antifungal activities of the azoles are most widely studied and some of them are in clinical practice as anti-microbial agents. However, the azole-resistant strain had led to the development of new antimicrobial compounds. In particular pyrazole derivatives are extensively studied and used as antimicrobial agents. Pyrazole is an important class of heterocyclic compounds and many pyrazole derivatives are reported to have the broad spectrum of biological properties, such as anti-inflammatory, antifungal, herbicidal, anti-tumour, cytotoxic, molecular modelling, and antiviral activities. Pyrazole derivatives also act as antiangiogenic agents, A3 adenosine receptor antagonists, neuropeptide YY5 receptor antagonists, kinase inhibitor for treatment of type 2 diabetes, hyperlipidemia, obesity, and thromboplatinmimetics. Recently urea derivatives of pyrazoles have been reported as potent inhibitors of p38 kinase. Since the high electronegativity of halogens (particularly chlorine and fluorine) in the aromatic part of the drug molecules play an important role in enhancing their biological activity, we are interested to have 4-fluoro or 4-chloro substitution in the aryls of 1,5-diaryl pyrazoles. As part of our on-going research aiming the synthesis of new antimicrobial compounds, we have reported the synthesis of novel pyrazole derivatives and their microbial activities (Ragavan *et al.*, 2009, 2010). The structure of the title compound is presented here.

In the title compound, (Fig. 1), the 1*H*-pyrazol ring (C7–C9/N1/N2) [maximum deviation of 0.00117 (14) Å] makes a dihedral angle of 85.40 (8)° with the phenyl ring (C1–C6). The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and comparable to those closely related structures (Shahani *et al.*, 2009, 2010*a,b,c*).

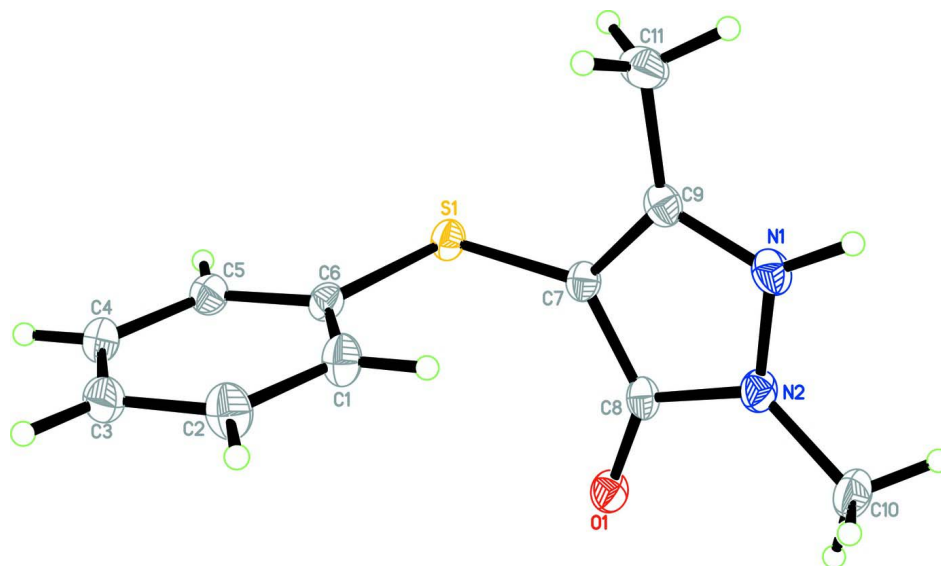
In the crystal packing (Fig. 2), pairs of intermolecular N1—H1N1···O1 and C3—H3A···O1 hydrogen bonds (Table 1) link the molecules into two-dimensional networks parallel to the *bc* plane.

**S2. Experimental**

The compound has been synthesized using the method available in the literature (Ragavan *et al.*, 2009) and recrystallized using the ethanol-chloroform 1:1 mixture (yield 60%, *m.p.* 444 K).

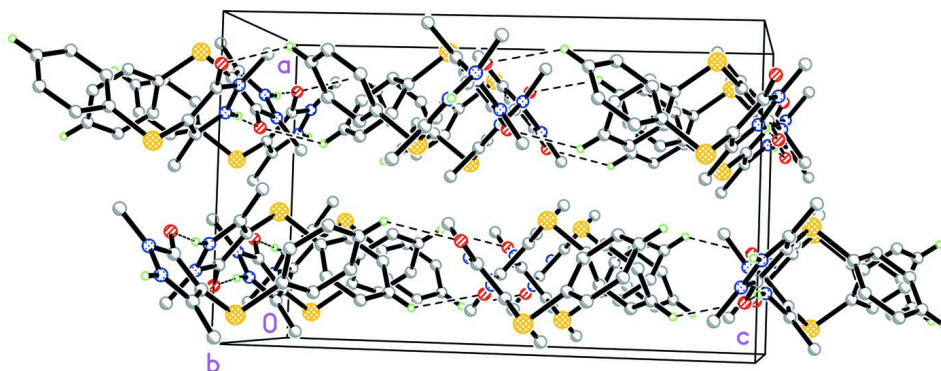
**S3. Refinement**

The H atoms bound to C atoms were positioned geometrically (C—H = 0.93–0.96 Å) with  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5U_{\text{eq}}(\text{C})$ . The H atoms attached to the N atom was located from the difference map and refined freely, [N—H = 0.94 (2) Å].



**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom numbering scheme.



**Figure 2**

The crystal structure of the title compound viewed approximately along the *b* axis. Intermolecular interactions are shown in dashed lines. Hydrogen bond not involved in intermolecular interactions are omitted for clarity.

### 1,3-Dimethyl-4-phenylsulfanyl-1*H*-pyrazol-5-ol

#### Crystal data

$C_{11}H_{12}N_2OS$

$M_r = 220.30$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 10.9479$  (2) Å

$b = 11.3470$  (3) Å

$c = 17.7392$  (4) Å

$V = 2203.67$  (9) Å<sup>3</sup>

$Z = 8$

$F(000) = 928$

$D_x = 1.328$  Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2873 reflections

$\theta = 2.8\text{--}29.1^\circ$

$\mu = 0.27$  mm<sup>-1</sup>

$T = 100$  K

Block, colourless

$0.33 \times 0.13 \times 0.11$  mm

*Data collection*

Bruker SMART APEXII CCD area-detector  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2009)  
 $T_{\min} = 0.917$ ,  $T_{\max} = 0.971$

12209 measured reflections  
3027 independent reflections  
2406 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.041$   
 $\theta_{\text{max}} = 29.4^\circ$ ,  $\theta_{\text{min}} = 2.3^\circ$   
 $h = -11 \rightarrow 15$   
 $k = -15 \rightarrow 15$   
 $l = -16 \rightarrow 24$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.104$   
 $S = 1.04$   
3027 reflections  
142 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.8061P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{\AA}^{-3}$

*Special details*

**Experimental.** The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.09449 (3)	0.31932 (3)	0.09298 (2)	0.01802 (11)
O1	0.32686 (10)	0.34450 (8)	-0.02532 (6)	0.0207 (2)
N1	0.20240 (12)	0.61926 (10)	0.00845 (8)	0.0202 (3)
N2	0.28173 (12)	0.54374 (10)	-0.02791 (7)	0.0192 (3)
C1	0.28747 (15)	0.36136 (13)	0.19278 (9)	0.0225 (3)
H1A	0.3057	0.4285	0.1649	0.027*
C2	0.35663 (15)	0.33304 (13)	0.25590 (9)	0.0254 (3)
H2A	0.4211	0.3816	0.2700	0.031*
C3	0.33066 (15)	0.23327 (14)	0.29808 (9)	0.0236 (3)
H3A	0.3766	0.2154	0.3407	0.028*
C4	0.23531 (15)	0.16043 (13)	0.27606 (9)	0.0243 (3)
H4A	0.2180	0.0929	0.3037	0.029*
C5	0.16551 (14)	0.18758 (13)	0.21304 (9)	0.0212 (3)
H5A	0.1019	0.1382	0.1986	0.025*

C6	0.19092 (13)	0.28904 (12)	0.17148 (8)	0.0173 (3)
C7	0.16428 (13)	0.43840 (11)	0.04922 (8)	0.0166 (3)
C8	0.26321 (13)	0.43076 (11)	-0.00304 (8)	0.0164 (3)
C9	0.13144 (14)	0.55679 (12)	0.05474 (8)	0.0178 (3)
C10	0.38173 (15)	0.58669 (13)	-0.07387 (10)	0.0239 (3)
H10A	0.4171	0.5222	-0.1013	0.036*
H10B	0.4427	0.6217	-0.0420	0.036*
H10C	0.3517	0.6446	-0.1087	0.036*
C11	0.03683 (15)	0.61354 (14)	0.10271 (9)	0.0248 (3)
H11A	0.0198	0.6912	0.0839	0.037*
H11B	0.0661	0.6189	0.1536	0.037*
H11C	-0.0365	0.5672	0.1016	0.037*
H1N1	0.1983 (19)	0.700 (2)	-0.0036 (13)	0.047 (6)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.01876 (19)	0.01847 (18)	0.0168 (2)	-0.00372 (13)	-0.00109 (13)	0.00267 (13)
O1	0.0267 (6)	0.0139 (4)	0.0215 (6)	0.0008 (4)	0.0046 (4)	0.0000 (4)
N1	0.0264 (7)	0.0129 (5)	0.0212 (7)	0.0017 (5)	-0.0005 (5)	-0.0006 (5)
N2	0.0240 (6)	0.0131 (5)	0.0206 (7)	-0.0002 (5)	0.0034 (5)	0.0007 (5)
C1	0.0273 (8)	0.0198 (7)	0.0204 (8)	-0.0046 (6)	-0.0027 (6)	0.0034 (6)
C2	0.0285 (8)	0.0257 (7)	0.0221 (9)	-0.0037 (6)	-0.0065 (7)	-0.0002 (6)
C3	0.0255 (8)	0.0298 (8)	0.0155 (8)	0.0053 (6)	-0.0012 (6)	0.0006 (6)
C4	0.0247 (8)	0.0260 (7)	0.0222 (8)	0.0015 (6)	0.0032 (6)	0.0082 (6)
C5	0.0197 (7)	0.0213 (7)	0.0225 (8)	-0.0015 (6)	0.0013 (6)	0.0043 (6)
C6	0.0196 (7)	0.0188 (6)	0.0134 (7)	0.0007 (5)	0.0019 (5)	0.0003 (5)
C7	0.0193 (7)	0.0148 (6)	0.0156 (7)	-0.0014 (5)	-0.0003 (5)	0.0005 (5)
C8	0.0224 (7)	0.0127 (6)	0.0141 (7)	-0.0017 (5)	-0.0018 (6)	0.0001 (5)
C9	0.0207 (7)	0.0179 (6)	0.0149 (7)	0.0009 (5)	-0.0035 (6)	-0.0002 (5)
C10	0.0283 (8)	0.0184 (7)	0.0250 (8)	-0.0043 (6)	0.0050 (7)	0.0032 (6)
C11	0.0248 (8)	0.0250 (7)	0.0245 (9)	0.0071 (6)	-0.0008 (6)	-0.0021 (6)

*Geometric parameters (Å, °)*

S1—C7	1.7356 (14)	C3—H3A	0.9300
S1—C6	1.7809 (15)	C4—C5	1.389 (2)
O1—C8	1.2648 (17)	C4—H4A	0.9300
N1—C9	1.3343 (19)	C5—C6	1.395 (2)
N1—N2	1.3801 (17)	C5—H5A	0.9300
N1—H1N1	0.94 (2)	C7—C9	1.3942 (19)
N2—C8	1.3708 (17)	C7—C8	1.428 (2)
N2—C10	1.4494 (19)	C9—C11	1.487 (2)
C1—C2	1.389 (2)	C10—H10A	0.9600
C1—C6	1.391 (2)	C10—H10B	0.9600
C1—H1A	0.9300	C10—H10C	0.9600
C2—C3	1.387 (2)	C11—H11A	0.9600
C2—H2A	0.9300	C11—H11B	0.9600

C3—C4	1.388 (2)	C11—H11C	0.9600
C7—S1—C6	103.83 (7)	C1—C6—S1	123.32 (11)
C9—N1—N2	108.92 (11)	C5—C6—S1	117.02 (11)
C9—N1—H1N1	129.0 (13)	C9—C7—C8	107.44 (12)
N2—N1—H1N1	121.9 (13)	C9—C7—S1	127.24 (12)
C8—N2—N1	109.70 (12)	C8—C7—S1	125.24 (10)
C8—N2—C10	127.39 (13)	O1—C8—N2	122.79 (13)
N1—N2—C10	121.97 (11)	O1—C8—C7	131.88 (13)
C2—C1—C6	119.79 (14)	N2—C8—C7	105.33 (12)
C2—C1—H1A	120.1	N1—C9—C7	108.57 (13)
C6—C1—H1A	120.1	N1—C9—C11	121.85 (13)
C3—C2—C1	120.80 (15)	C7—C9—C11	129.57 (14)
C3—C2—H2A	119.6	N2—C10—H10A	109.5
C1—C2—H2A	119.6	N2—C10—H10B	109.5
C2—C3—C4	119.26 (15)	H10A—C10—H10B	109.5
C2—C3—H3A	120.4	N2—C10—H10C	109.5
C4—C3—H3A	120.4	H10A—C10—H10C	109.5
C3—C4—C5	120.56 (14)	H10B—C10—H10C	109.5
C3—C4—H4A	119.7	C9—C11—H11A	109.5
C5—C4—H4A	119.7	C9—C11—H11B	109.5
C4—C5—C6	119.92 (14)	H11A—C11—H11B	109.5
C4—C5—H5A	120.0	C9—C11—H11C	109.5
C6—C5—H5A	120.0	H11A—C11—H11C	109.5
C1—C6—C5	119.66 (14)	H11B—C11—H11C	109.5
C9—N1—N2—C8	-1.48 (17)	N1—N2—C8—O1	-177.46 (13)
C9—N1—N2—C10	-171.15 (14)	C10—N2—C8—O1	-8.5 (2)
C6—C1—C2—C3	0.0 (2)	N1—N2—C8—C7	2.10 (16)
C1—C2—C3—C4	0.9 (2)	C10—N2—C8—C7	171.06 (14)
C2—C3—C4—C5	-0.8 (2)	C9—C7—C8—O1	177.55 (16)
C3—C4—C5—C6	-0.2 (2)	S1—C7—C8—O1	-5.6 (2)
C2—C1—C6—C5	-1.0 (2)	C9—C7—C8—N2	-1.96 (16)
C2—C1—C6—S1	178.43 (12)	S1—C7—C8—N2	174.91 (11)
C4—C5—C6—C1	1.1 (2)	N2—N1—C9—C7	0.18 (17)
C4—C5—C6—S1	-178.38 (12)	N2—N1—C9—C11	179.36 (13)
C7—S1—C6—C1	7.87 (15)	C8—C7—C9—N1	1.12 (17)
C7—S1—C6—C5	-172.70 (12)	S1—C7—C9—N1	-175.67 (11)
C6—S1—C7—C9	-100.38 (14)	C8—C7—C9—C11	-177.97 (15)
C6—S1—C7—C8	83.36 (14)	S1—C7—C9—C11	5.2 (2)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N1 $\cdots$ O1 <sup>i</sup>	0.94 (2)	1.71 (2)	2.6446 (16)	173 (2)
C3—H3A $\cdots$ O1 <sup>ii</sup>	0.93	2.53	3.2549 (19)	135

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