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## Structure Reports

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## 3-Acetyl-1-(2,5-dimethylphenyl)thiourea

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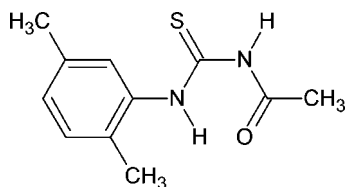
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.101; data-to-parameter ratio = 16.2.

In the title compound,  $\text{C}_{11}\text{H}_{14}\text{N}_2\text{OS}$ , the thioamide  $\text{C}=\text{S}$  and amide  $\text{C}=\text{O}$  bonds are *anti* to each other; the  $\text{N}-\text{H}$  bonds are also *anti* to each other. The molecular conformation is stabilized by an  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond. In the crystal, the molecules are linked into inversion dimers by pairs of  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds.

## Related literature

For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Gowda *et al.* (2001); Kumar *et al.* (2012); Shahwar *et al.* (2012), of *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007) and of *N*-chloroarylsulfonamides, see: Gowda & Ramachandra (1989); Shetty & Gowda (2004).



## Experimental

## Crystal data

 $\text{C}_{11}\text{H}_{14}\text{N}_2\text{OS}$  $M_r = 222.30$ Triclinic,  $P\bar{1}$  $a = 5.0312$  (2) Å $b = 10.9329$  (6) Å $c = 11.0568$  (7) Å $\alpha = 105.711$  (5)° $\beta = 100.020$  (5)° $\gamma = 93.037$  (4)° $V = 573.31$  (6) Å<sup>3</sup> $Z = 2$ Mo  $K\alpha$  radiation $\mu = 0.26$  mm<sup>-1</sup> $T = 293$  K $0.42 \times 0.38 \times 0.20$  mm

## Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector  
Absorption correction: multi-scan (*CrysAlis RED*; Oxford

Diffraction, 2009)  
 $T_{\min} = 0.899$ ,  $T_{\max} = 0.950$   
3641 measured reflections  
2344 independent reflections  
2068 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.007$

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$  $wR(F^2) = 0.101$  $S = 1.05$ 

2344 reflections

145 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.23$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.20$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}$	0.85 (2)	1.94 (2)	2.6382 (19)	139 (2)
$\text{N2}-\text{H2N}\cdots\text{S1}^i$	0.85 (2)	2.55 (2)	3.3904 (15)	169 (2)

Symmetry code: (i)  $-x + 1, -y + 2, -z$ .

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); 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: BT5960).

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## supporting information

*Acta Cryst.* (2012). E68, o2338 [https://doi.org/10.1107/S1600536812029601]

## 3-Acetyl-1-(2,5-dimethylphenyl)thiourea

**B. Thimme Gowda, Sabine Foro and Sharatha Kumar**

### S1. Comment

Thiourea and its derivatives are known to exhibit a wide variety of biological activities. As part of studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2001; Kumar *et al.*, 2012; Shahwar *et al.*, 2012); *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007) and *N*-chloroarylsulfonamides (Gowda & Ramachandra, 1989; Shetty & Gowda, 2004), in the present work, the crystal structure of 3-acetyl-1-(2,5-dimethylphenyl)thiourea has been determined (Fig. 1).

The conformation of the two N—H bonds are *anti* to each other. Furthermore, the conformations of the amide C=S and the C=O are also *anti* to each other and both the bonds are *anti* to the adjacent N—H bonds, similar to the *anti* conformation observed in 3-acetyl-1-(2,3-dimethylphenyl)thiourea (I) (Kumar *et al.*, 2012). The adjacent N—H bond is *syn* to the *ortho*-methyl group, compared to the *anti* conformation observed with respect to the *ortho*- and *meta*-methyl groups in the benzene ring of (I).

The side chain is oriented itself with respect to the phenyl ring with the C2—C1—N1—C7 and C6—C1—N1—C7 torsion angles of 83.44 (22)° and -100.65 (1/5)°, compared to the corresponding values of 83.59 (47)° and -99.89 (44)° in (I). The dihedral angle between the phenyl ring and the side chain is 79.0 (4)°, compared to the value of 81.33 (10)° in (I).

The hydrogen atom of the NH attached to the phenyl ring and the amide oxygen are involved in intramolecular hydrogen bonding. In the crystal, the molecules form inversion type dimers through N—H...S intermolecular hydrogen bonds (Table 1, Fig.2).

### S2. Experimental

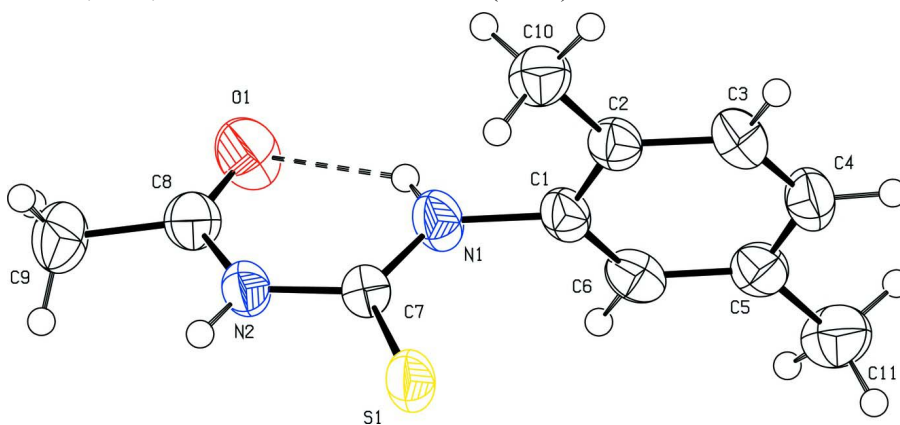
3-Acetyl-1-(2,5-dimethylphenyl)thiourea was synthesized by adding a solution of acetyl chloride (0.10 mol) in acetone (30 ml) dropwise to a suspension of ammonium thiocyanate (0.10 mol) in acetone (30 ml). The reaction mixture was refluxed for 30 min. After cooling to room temperature, a solution of 2,5-dimethylaniline (0.10 mol) in acetone (10 ml) was added and refluxed for 3 h. The reaction mixture was poured into acidified cold water. The precipitated title compound was recrystallized to constant melting point from acetonitrile. The purity of the compound was checked and characterized by its infrared spectrum.

Prism like colourless single crystals used in X-ray diffraction studies were grown in acetonitrile solution by slow evaporation of the solvent at room temperature.

### S3. Refinement

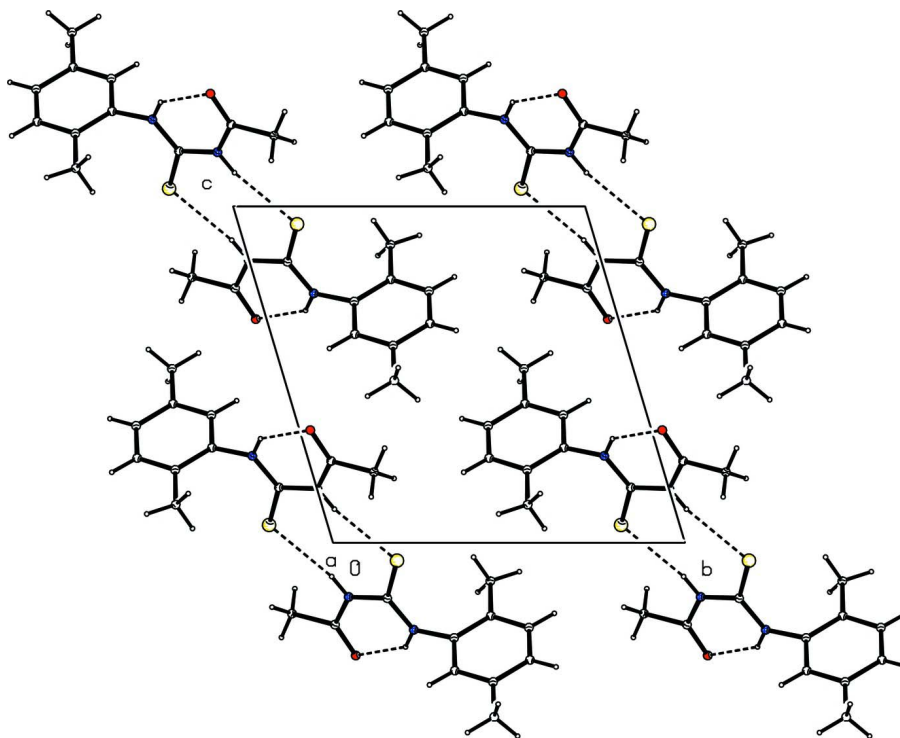
All H atoms bonded to C were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined using a riding model with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  (1.5 for methyl H atoms) and with aromatic C—H = 0.93 Å and methyl C—H = 0.96 Å. The amino H atoms were refined isotropically with the N—H distances restrained to 0.86 (2)Å.

To improve values of R1, wR2, and GOOF one bad reflection (-1 3 3) was omitted from the refinement.



**Figure 1**

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.



**Figure 2**

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

### 3-Acetyl-1-(2,5-dimethylphenyl)thiourea

#### Crystal data

$C_{11}H_{14}N_2OS$

$M_r = 222.30$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 5.0312 (2) \text{ \AA}$

$b = 10.9329 (6) \text{ \AA}$

$c = 11.0568 (7) \text{ \AA}$

$\alpha = 105.711 (5)^\circ$

$\beta = 100.020 (5)^\circ$   
 $\gamma = 93.037 (4)^\circ$   
 $V = 573.31 (6) \text{ \AA}^3$   
 $Z = 2$   
 $F(000) = 236$   
 $D_x = 1.288 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2369 reflections  
 $\theta = 3.1\text{--}27.7^\circ$   
 $\mu = 0.26 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Prism, colourless  
 $0.42 \times 0.38 \times 0.20 \text{ mm}$

*Data collection*

Oxford Diffraction Xcalibur  
 diffractometer with a Sapphire CCD detector  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 Rotation method data acquisition using  $\omega$  and  
 phi scans  
 Absorption correction: multi-scan  
 (*CrysAlis RED*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.899$ ,  $T_{\max} = 0.950$

3641 measured reflections  
 2344 independent reflections  
 2068 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.007$   
 $\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 3.1^\circ$   
 $h = -5 \rightarrow 6$   
 $k = -11 \rightarrow 13$   
 $l = -13 \rightarrow 13$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.101$   
 $S = 1.05$   
 2344 reflections  
 145 parameters  
 2 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.2328P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$

*Special details*

**Experimental.** *CrysAlis RED* (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

**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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6145 (3)	0.73651 (15)	0.28289 (15)	0.0372 (4)
C2	0.6492 (3)	0.61308 (15)	0.21687 (15)	0.0377 (4)
C3	0.5232 (4)	0.51510 (16)	0.25210 (17)	0.0439 (4)
H3	0.5400	0.4308	0.2096	0.053*
C4	0.3747 (4)	0.53925 (17)	0.34786 (17)	0.0458 (4)
H4	0.2947	0.4711	0.3688	0.055*

C5	0.3422 (3)	0.66295 (17)	0.41356 (16)	0.0416 (4)
C6	0.4659 (3)	0.76177 (16)	0.37962 (16)	0.0412 (4)
H6	0.4489	0.8460	0.4223	0.049*
C7	0.6584 (3)	0.89287 (15)	0.16443 (16)	0.0366 (3)
C8	1.0446 (4)	1.06364 (16)	0.24317 (18)	0.0442 (4)
C9	1.1609 (4)	1.17630 (19)	0.2102 (2)	0.0571 (5)
H9A	1.0190	1.2277	0.1915	0.069*
H9B	1.2423	1.1473	0.1365	0.069*
H9C	1.2962	1.2263	0.2813	0.069*
C10	0.8130 (4)	0.58529 (19)	0.11363 (18)	0.0491 (4)
H10A	0.9914	0.6302	0.1463	0.059*
H10B	0.7259	0.6127	0.0425	0.059*
H10C	0.8268	0.4951	0.0859	0.059*
C11	0.1781 (4)	0.6891 (2)	0.5173 (2)	0.0587 (5)
H11A	0.2316	0.6389	0.5748	0.070*
H11B	-0.0111	0.6668	0.4797	0.070*
H11C	0.2089	0.7781	0.5638	0.070*
N1	0.7489 (3)	0.84398 (14)	0.25857 (15)	0.0438 (4)
H1N	0.895 (3)	0.8817 (18)	0.3093 (18)	0.053*
N2	0.8099 (3)	1.00118 (13)	0.16102 (15)	0.0401 (3)
H2N	0.751 (4)	1.0323 (18)	0.1002 (17)	0.048*
O1	1.1487 (3)	1.03056 (13)	0.33502 (14)	0.0603 (4)
S1	0.37879 (9)	0.83331 (4)	0.05301 (4)	0.04690 (16)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0373 (8)	0.0377 (8)	0.0365 (8)	0.0001 (6)	-0.0019 (6)	0.0171 (7)
C2	0.0372 (8)	0.0410 (9)	0.0350 (8)	0.0040 (7)	0.0017 (6)	0.0144 (7)
C3	0.0538 (10)	0.0346 (8)	0.0432 (9)	0.0047 (7)	0.0056 (8)	0.0130 (7)
C4	0.0507 (10)	0.0440 (9)	0.0473 (10)	-0.0005 (8)	0.0075 (8)	0.0229 (8)
C5	0.0399 (9)	0.0522 (10)	0.0353 (8)	0.0072 (7)	0.0031 (7)	0.0189 (7)
C6	0.0457 (9)	0.0378 (8)	0.0376 (9)	0.0091 (7)	0.0003 (7)	0.0105 (7)
C7	0.0362 (8)	0.0345 (8)	0.0414 (9)	0.0029 (6)	0.0082 (7)	0.0143 (7)
C8	0.0419 (9)	0.0344 (8)	0.0515 (10)	-0.0002 (7)	0.0051 (8)	0.0077 (7)
C9	0.0523 (11)	0.0441 (10)	0.0693 (13)	-0.0127 (8)	0.0051 (9)	0.0144 (9)
C10	0.0482 (10)	0.0552 (11)	0.0459 (10)	0.0076 (8)	0.0112 (8)	0.0161 (8)
C11	0.0547 (11)	0.0795 (14)	0.0489 (11)	0.0162 (10)	0.0157 (9)	0.0248 (10)
N1	0.0429 (8)	0.0393 (8)	0.0466 (8)	-0.0065 (6)	-0.0055 (6)	0.0191 (6)
N2	0.0376 (7)	0.0360 (7)	0.0477 (8)	-0.0014 (6)	0.0024 (6)	0.0183 (6)
O1	0.0610 (8)	0.0489 (8)	0.0598 (9)	-0.0096 (6)	-0.0144 (7)	0.0165 (6)
S1	0.0406 (2)	0.0483 (3)	0.0527 (3)	-0.00880 (18)	-0.00441 (19)	0.0269 (2)

*Geometric parameters (Å, °)*

C1—C6	1.385 (2)	C8—O1	1.213 (2)
C1—C2	1.386 (2)	C8—N2	1.375 (2)
C1—N1	1.436 (2)	C8—C9	1.495 (2)

C2—C3	1.393 (2)	C9—H9A	0.9600
C2—C10	1.497 (2)	C9—H9B	0.9600
C3—C4	1.376 (3)	C9—H9C	0.9600
C3—H3	0.9300	C10—H10A	0.9600
C4—C5	1.384 (3)	C10—H10B	0.9600
C4—H4	0.9300	C10—H10C	0.9600
C5—C6	1.388 (2)	C11—H11A	0.9600
C5—C11	1.502 (3)	C11—H11B	0.9600
C6—H6	0.9300	C11—H11C	0.9600
C7—N1	1.319 (2)	N1—H1N	0.852 (15)
C7—N2	1.387 (2)	N2—H2N	0.849 (15)
C7—S1	1.6692 (17)		
C6—C1—C2	122.16 (15)	C8—C9—H9A	109.5
C6—C1—N1	117.34 (15)	C8—C9—H9B	109.5
C2—C1—N1	120.37 (15)	H9A—C9—H9B	109.5
C1—C2—C3	116.24 (15)	C8—C9—H9C	109.5
C1—C2—C10	122.39 (15)	H9A—C9—H9C	109.5
C3—C2—C10	121.37 (16)	H9B—C9—H9C	109.5
C4—C3—C2	122.01 (16)	C2—C10—H10A	109.5
C4—C3—H3	119.0	C2—C10—H10B	109.5
C2—C3—H3	119.0	H10A—C10—H10B	109.5
C3—C4—C5	121.29 (16)	C2—C10—H10C	109.5
C3—C4—H4	119.4	H10A—C10—H10C	109.5
C5—C4—H4	119.4	H10B—C10—H10C	109.5
C4—C5—C6	117.50 (16)	C5—C11—H11A	109.5
C4—C5—C11	121.20 (17)	C5—C11—H11B	109.5
C6—C5—C11	121.31 (17)	H11A—C11—H11B	109.5
C1—C6—C5	120.80 (15)	C5—C11—H11C	109.5
C1—C6—H6	119.6	H11A—C11—H11C	109.5
C5—C6—H6	119.6	H11B—C11—H11C	109.5
N1—C7—N2	116.17 (15)	C7—N1—C1	124.80 (14)
N1—C7—S1	124.34 (12)	C7—N1—H1N	116.3 (14)
N2—C7—S1	119.49 (12)	C1—N1—H1N	118.9 (14)
O1—C8—N2	122.97 (16)	C8—N2—C7	128.05 (15)
O1—C8—C9	122.62 (17)	C8—N2—H2N	116.1 (14)
N2—C8—C9	114.41 (16)	C7—N2—H2N	115.9 (14)
C6—C1—C2—C3	0.7 (2)	C4—C5—C6—C1	0.4 (2)
N1—C1—C2—C3	176.46 (14)	C11—C5—C6—C1	-179.34 (16)
C6—C1—C2—C10	-179.04 (16)	N2—C7—N1—C1	176.57 (15)
N1—C1—C2—C10	-3.3 (2)	S1—C7—N1—C1	-3.1 (3)
C1—C2—C3—C4	-0.6 (3)	C6—C1—N1—C7	-100.6 (2)
C10—C2—C3—C4	179.20 (17)	C2—C1—N1—C7	83.4 (2)
C2—C3—C4—C5	0.4 (3)	O1—C8—N2—C7	-1.7 (3)
C3—C4—C5—C6	-0.3 (3)	C9—C8—N2—C7	178.47 (17)
C3—C4—C5—C11	179.49 (17)	N1—C7—N2—C8	0.6 (3)
C2—C1—C6—C5	-0.7 (2)	S1—C7—N2—C8	-179.70 (14)

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N1—C1—C6—C5                    -176.52 (14)

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*Hydrogen-bond geometry (Å, °)*

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<i>D—H···A</i>	<i>D—H</i>	<i>H···A</i>	<i>D···A</i>	<i>D—H···A</i>
N1—H1N···O1	0.85 (2)	1.94 (2)	2.6382 (19)	139 (2)
N2—H2N···S1 <sup>i</sup>	0.85 (2)	2.55 (2)	3.3904 (15)	169 (2)

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Symmetry code: (i)  $-x+1, -y+2, -z$ .