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N,N-Diethyl-*N'*-phenylacetylthiourea

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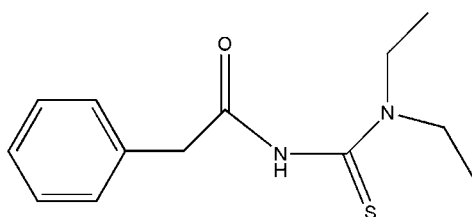
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.042; wR factor = 0.124; data-to-parameter ratio = 19.7.

The title thiourea molecule, $\text{C}_{13}\text{H}_{18}\text{N}_2\text{OS}$, adopts a folded conformation due to the steric hindrance of the two ethyl groups and the acetyl group. In the crystal structure, the acetyl O atom is not involved in hydrogen bonding, but intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds link the molecules into centrosymmetric dimers.

Related literature

For general background on the chemistry of thiourea derivatives, see: Choi *et al.* (2008); Jones *et al.* (2008); Kushwaha *et al.* (2008); Su *et al.* (2006). For related structures, see: Su (2005, 2007); Xian *et al.* (2004); Xian (2008).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{18}\text{N}_2\text{OS}$
 $M_r = 250.35$
 Monoclinic, $P2_1/c$
 $a = 11.578$ (7) Å
 $b = 12.804$ (8) Å

$c = 9.176$ (6) Å
 $\beta = 103.842$ (10)°
 $V = 1320.8$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.23$ mm⁻¹
 $T = 296$ (2) K

0.30 × 0.29 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2000)
 $T_{\min} = 0.933$, $T_{\max} = 0.944$

7619 measured reflections
 3080 independent reflections
 2484 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.124$
 $S = 1.05$
 3080 reflections

156 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.48$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}'\cdots\text{S1}^i$	0.86	2.69	3.404 (3)	141

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

Data collection: APEX2 (Bruker, 2001); cell refinement: APEX2 and SAINT (Bruker, 2001); data reduction: SAINT; 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2467).

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supporting information

Acta Cryst. (2008). E64, o2256 [doi:10.1107/S1600536808035290]

N,N*-Diethyl-*N'*-phenylacetylthiourea*Liang Xian****S1. Comment**

Thiourea and its derivatives attract special attention in recent years because of their broad applications, such as anion recognition, nonlinear optical material, catalysis *etc.*, and also due to high bioactivity and good coordination ability (Choi *et al.*, 2008; Kushwaha *et al.*, 2008; Jones *et al.*, 2008; Su *et al.*, 2006). For a long time, we have been interested in the influence of non-covalent interactions related to the substituted groups on the conformations of thiourea derivatives as well as their coordination abilities. Thiourea derivatives with different substituted groups coordinate different transition metal ions providing various structures. One of the key influence factors in coordination reactions is non-covalent interaction. However, the central ion also plays an important role. Triangle conformation is commonly observed in the coordination compound of benzoylthiourea with Cu(I) (Xian *et al.*, 2004). However, Cu₆ cluster structure was also obtained (Su *et al.*, 2005). Herewith we present the crystal structure of the title compound, (I).

The conformation and the packing diagram of (I) are shown in Figures 1 and 2, respectively. It can be seen that the title compound has a folded conformation which is similar to the structure we obtained before (CCDC No. 699688). The dihedral angle between the benzene ring and the plane O1/N1/C7/C8 is 69.12 (6)°, and the dihedral angle between the benzene ring and the plane S1/C9/N1/N2 is 67.19 (6)°. Apparently, stereo-hindrance effect of two ethyl groups and acetyl group is the main influence factor to the folded conformation. Because of the absence of hydrogen atom on N2, the acetyl oxygen atom does not take part in hydrogen bonding interactions. This is different from the other carbonylthiourea derivatives (Su *et al.*, 2006; Xian, 2008), in which the carbonyl oxygen atom often forms a six-membered hydrogen bonding ring. However, thiocarbonyl sulfur atom is involved in an intermolecular N—H···S hydrogen bond (Table 1), linking two molecules into centrosymmetric dimer, that was earlier observed in related structures (Su, 2007; Xian, 2008).

S2. Experimental

All reagents and organic solvents were of analytical reagent grade and commercially available. Phenylacetyl chloride (1.55 g) was treated with ammonium thiocyanate (1.20 g) in CH₂Cl₂ under solid-liquid phase transfer catalysis conditions, using 3% polyethylene glycol-400 as catalyst, to give the corresponding phenylacetyl isothiocyanate, which was reacted with diethylamine (0.73 g) to give the title compound. The solid was separated from the liquid phase by filtration, washed with CH₂Cl₂ and then dried in air. Colorless single crystals suitable for X-ray analysis were obtained after one week by slow evaporation of a chloroform solution. The infrared spectrum was recorded in the range of 4000–400 cm⁻¹ on a Nicolet NEXUS 670 FT-IR spectrometer, using KBr pellets. ¹H NMR spectrum was obtained on an INOVA-400 MHz superconduction spectrometer, acetone-d₆ was used as solvent and TMS as internal standard, and the chemical shifts are expressed as delta. Elemental analyses were carried out on a PE-2400 elemental analysis instrument. Melting point determination was performed in YRT-3 melting point instrument (Tianjin) and was uncorrected. Melting Point: 92–94 °C. Elemental analysis (%) found (calcd.): C, 62.3(60.5); H, 7.2(6.9); N, 11.2(13.6); S, 12.8(10.9). IR (KBr, cm⁻¹): 3190 (N—H), 3079, 1711 (C=O), 1548(C=C), 1233(C=S), 1121. ¹H NMR(delta, p.p.m.): 2.06 (m, 3H, CH₃); 2.85

(m, 3H, CH₃); 3.70 (m, 6H, 3CH₂); 7.22–7.38 (m, 5H, C₆H₅); 9.25 (s, 1H, NH).

S3. Refinement

All H atoms were placed in calculated positions (C–H = 0.93–0.97 Å, N–H = 0.86 Å) and refined using the riding model approximation, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}$ of the parent atom.

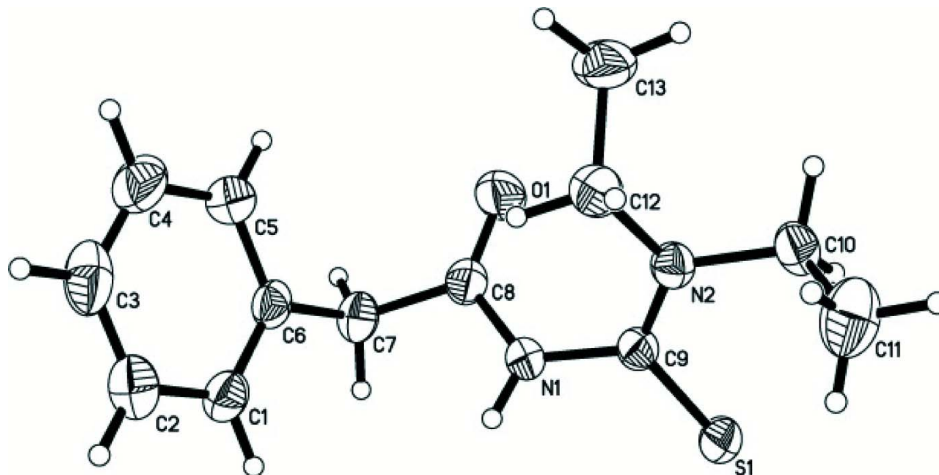
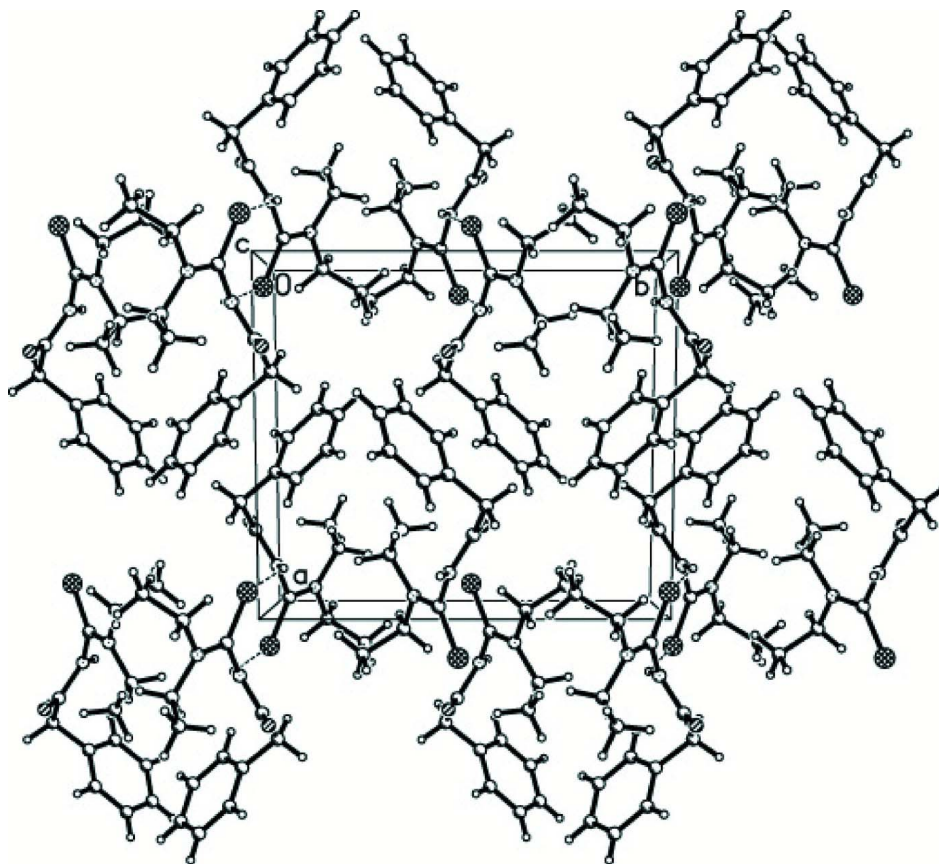


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level.

**Figure 2**

Packing diagram of the title compound viewed along the c axis. Intermolecular hydrogen bonds are shown as dashed lines.

N,N-Diethyl-*N'*-phenylacetylthiourea

Crystal data

$C_{13}H_{18}N_2OS$

$M_r = 250.35$

Monoclinic, $P2_1/c$

$a = 11.578$ (7) Å

$b = 12.804$ (8) Å

$c = 9.176$ (6) Å

$\beta = 103.842$ (10)°

$V = 1320.8$ (15) Å³

$Z = 4$

$F(000) = 536$

$D_x = 1.259$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3844 reflections

$\theta = 2.4$ – 29.9°

$\mu = 0.23$ mm⁻¹

$T = 296$ K

Block, colorless

$0.30 \times 0.29 \times 0.25$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2000)

$T_{\min} = 0.934$, $T_{\max} = 0.944$

7619 measured reflections

3080 independent reflections

2484 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 28.0^\circ$, $\theta_{\min} = 1.8^\circ$

$h = -15 \rightarrow 13$

$k = -16 \rightarrow 16$

$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.124$
 $S = 1.05$
 3080 reflections
 156 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.2848P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.48 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$

Special details

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.61035 (14)	0.09311 (14)	0.9820 (2)	0.0472 (4)
H1	0.6752	0.0776	1.0607	0.057*
C2	0.53176 (18)	0.17042 (15)	0.9993 (2)	0.0570 (5)
H2	0.5441	0.2071	1.0892	0.068*
C3	0.43555 (19)	0.19328 (17)	0.8842 (3)	0.0663 (6)
H3	0.3821	0.2449	0.8962	0.080*
C4	0.41832 (18)	0.1398 (2)	0.7515 (3)	0.0716 (6)
H4	0.3531	0.1552	0.6733	0.086*
C5	0.49718 (16)	0.06344 (17)	0.7335 (2)	0.0577 (5)
H5	0.4856	0.0280	0.6426	0.069*
C6	0.59380 (13)	0.03890 (13)	0.84970 (19)	0.0406 (4)
C7	0.67692 (14)	-0.04934 (13)	0.8329 (2)	0.0457 (4)
H7A	0.7249	-0.0684	0.9311	0.055*
H7B	0.6303	-0.1099	0.7909	0.055*
C8	0.75749 (14)	-0.01928 (13)	0.73300 (19)	0.0426 (4)
C9	0.95069 (13)	0.06202 (11)	0.73941 (17)	0.0359 (3)
C10	1.02328 (16)	0.14815 (14)	0.5447 (2)	0.0488 (4)
H10A	0.9893	0.1590	0.4384	0.059*
H10B	1.0782	0.0899	0.5550	0.059*
C11	1.0909 (2)	0.24531 (17)	0.6105 (3)	0.0755 (6)
H11A	1.0370	0.3033	0.5999	0.113*
H11B	1.1517	0.2601	0.5583	0.113*
H11C	1.1268	0.2340	0.7149	0.113*
C12	0.81587 (16)	0.18024 (14)	0.5634 (2)	0.0491 (4)
H12A	0.8331	0.2545	0.5695	0.059*

H12B	0.7629	0.1657	0.6284	0.059*
C13	0.75285 (19)	0.15330 (19)	0.4033 (2)	0.0675 (6)
H13A	0.7990	0.1781	0.3363	0.101*
H13B	0.6759	0.1858	0.3788	0.101*
H13C	0.7438	0.0789	0.3935	0.101*
N1	0.85660 (11)	0.03650 (10)	0.80451 (15)	0.0398 (3)
H1'	0.8610	0.0570	0.8949	0.048*
N2	0.92772 (11)	0.12212 (10)	0.61873 (14)	0.0387 (3)
O1	0.73627 (12)	-0.04082 (12)	0.60141 (15)	0.0640 (4)
S1	1.08598 (3)	0.01581 (4)	0.82130 (5)	0.04902 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0353 (8)	0.0537 (9)	0.0528 (10)	-0.0047 (7)	0.0107 (7)	0.0001 (8)
C2	0.0569 (11)	0.0526 (10)	0.0681 (12)	-0.0026 (8)	0.0276 (9)	-0.0033 (9)
C3	0.0603 (12)	0.0608 (11)	0.0887 (16)	0.0197 (10)	0.0394 (11)	0.0248 (11)
C4	0.0507 (11)	0.0962 (16)	0.0667 (13)	0.0215 (11)	0.0120 (10)	0.0285 (12)
C5	0.0466 (10)	0.0793 (13)	0.0460 (10)	0.0026 (9)	0.0088 (8)	0.0047 (9)
C6	0.0312 (7)	0.0456 (8)	0.0473 (9)	-0.0053 (6)	0.0135 (6)	0.0047 (7)
C7	0.0391 (8)	0.0452 (8)	0.0553 (10)	-0.0041 (7)	0.0166 (7)	0.0006 (7)
C8	0.0372 (8)	0.0469 (8)	0.0445 (9)	0.0000 (7)	0.0114 (7)	-0.0029 (7)
C9	0.0347 (7)	0.0387 (7)	0.0351 (7)	-0.0001 (6)	0.0101 (6)	-0.0047 (6)
C10	0.0481 (9)	0.0569 (10)	0.0460 (9)	-0.0014 (8)	0.0203 (7)	0.0064 (8)
C11	0.0678 (13)	0.0557 (11)	0.1072 (19)	-0.0136 (10)	0.0293 (13)	0.0072 (12)
C12	0.0502 (9)	0.0500 (9)	0.0473 (9)	0.0154 (7)	0.0124 (7)	0.0034 (7)
C13	0.0625 (12)	0.0880 (15)	0.0467 (10)	0.0285 (11)	0.0026 (9)	0.0024 (10)
N1	0.0344 (6)	0.0527 (8)	0.0340 (6)	-0.0010 (5)	0.0115 (5)	-0.0037 (6)
N2	0.0375 (7)	0.0411 (7)	0.0386 (7)	0.0032 (5)	0.0115 (5)	-0.0004 (5)
O1	0.0590 (8)	0.0866 (10)	0.0472 (8)	-0.0209 (7)	0.0143 (6)	-0.0180 (7)
S1	0.0346 (2)	0.0687 (3)	0.0442 (3)	0.00839 (18)	0.01022 (17)	0.00658 (19)

Geometric parameters (Å, °)

C1—C6	1.371 (2)	C9—N1	1.401 (2)
C1—C2	1.379 (3)	C9—S1	1.6747 (17)
C1—H1	0.9300	C10—N2	1.469 (2)
C2—C3	1.371 (3)	C10—C11	1.516 (3)
C2—H2	0.9300	C10—H10A	0.9700
C3—C4	1.370 (4)	C10—H10B	0.9700
C3—H3	0.9300	C11—H11A	0.9600
C4—C5	1.374 (3)	C11—H11B	0.9600
C4—H4	0.9300	C11—H11C	0.9600
C5—C6	1.384 (2)	C12—N2	1.474 (2)
C5—H5	0.9300	C12—C13	1.515 (3)
C6—C7	1.516 (2)	C12—H12A	0.9700
C7—C8	1.506 (2)	C12—H12B	0.9700
C7—H7A	0.9700	C13—H13A	0.9600

C7—H7B	0.9700	C13—H13B	0.9600
C8—O1	1.205 (2)	C13—H13C	0.9600
C8—N1	1.377 (2)	N1—H1'	0.8600
C9—N2	1.322 (2)		
C6—C1—C2	120.61 (17)	N2—C10—C11	112.09 (16)
C6—C1—H1	119.7	N2—C10—H10A	109.2
C2—C1—H1	119.7	C11—C10—H10A	109.2
C3—C2—C1	120.1 (2)	N2—C10—H10B	109.2
C3—C2—H2	120.0	C11—C10—H10B	109.2
C1—C2—H2	120.0	H10A—C10—H10B	107.9
C4—C3—C2	119.81 (19)	C10—C11—H11A	109.5
C4—C3—H3	120.1	C10—C11—H11B	109.5
C2—C3—H3	120.1	H11A—C11—H11B	109.5
C3—C4—C5	120.2 (2)	C10—C11—H11C	109.5
C3—C4—H4	119.9	H11A—C11—H11C	109.5
C5—C4—H4	119.9	H11B—C11—H11C	109.5
C4—C5—C6	120.4 (2)	N2—C12—C13	113.49 (14)
C4—C5—H5	119.8	N2—C12—H12A	108.9
C6—C5—H5	119.8	C13—C12—H12A	108.9
C1—C6—C5	118.89 (17)	N2—C12—H12B	108.9
C1—C6—C7	120.65 (15)	C13—C12—H12B	108.9
C5—C6—C7	120.42 (17)	H12A—C12—H12B	107.7
C8—C7—C6	111.86 (14)	C12—C13—H13A	109.5
C8—C7—H7A	109.2	C12—C13—H13B	109.5
C6—C7—H7A	109.2	H13A—C13—H13B	109.5
C8—C7—H7B	109.2	C12—C13—H13C	109.5
C6—C7—H7B	109.2	H13A—C13—H13C	109.5
H7A—C7—H7B	107.9	H13B—C13—H13C	109.5
O1—C8—N1	122.88 (15)	C8—N1—C9	124.21 (14)
O1—C8—C7	122.95 (16)	C8—N1—H1'	117.9
N1—C8—C7	114.16 (15)	C9—N1—H1'	117.9
N2—C9—N1	118.15 (13)	C9—N2—C10	119.78 (13)
N2—C9—S1	124.09 (12)	C9—N2—C12	124.49 (13)
N1—C9—S1	117.75 (12)	C10—N2—C12	115.02 (14)
C6—C1—C2—C3	0.4 (3)	O1—C8—N1—C9	-8.9 (3)
C1—C2—C3—C4	-0.6 (3)	C7—C8—N1—C9	172.09 (14)
C2—C3—C4—C5	0.0 (3)	N2—C9—N1—C8	62.2 (2)
C3—C4—C5—C6	0.8 (3)	S1—C9—N1—C8	-118.74 (15)
C2—C1—C6—C5	0.4 (2)	N1—C9—N2—C10	-178.25 (14)
C2—C1—C6—C7	-177.23 (15)	S1—C9—N2—C10	2.7 (2)
C4—C5—C6—C1	-1.0 (3)	N1—C9—N2—C12	11.9 (2)
C4—C5—C6—C7	176.60 (17)	S1—C9—N2—C12	-167.16 (12)
C1—C6—C7—C8	-107.78 (18)	C11—C10—N2—C9	-88.3 (2)
C5—C6—C7—C8	74.7 (2)	C11—C10—N2—C12	82.5 (2)
C6—C7—C8—O1	-96.5 (2)	C13—C12—N2—C9	-122.62 (19)
C6—C7—C8—N1	82.50 (18)	C13—C12—N2—C10	67.1 (2)

Hydrogen-bond geometry (Å, °)

<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>
N1—H1' \cdots S1 ⁱ	0.86	2.69	3.404 (3)	141

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