Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Benzoyl-*N*′-(2-chloro-3-pyridyl)thiourea

Yu-Jie Ding,[a] Jian Yao,[b] Jian-Chao Wu,[b] Wen-Kui Dong[b]* and Jun-Feng Tong[b]

[a]Department of Biochemical Engineering, Anhui University of Technology and Science, Wuhu 241000, People's Republic of China, and [b]School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, People's Republic of China
Correspondence e-mail: dongwk@mail.lzjtu.cn

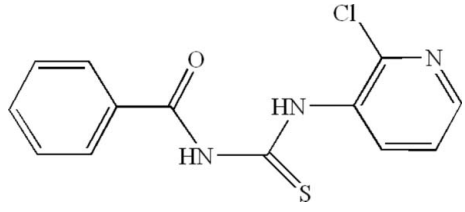
Received 9 July 2009; accepted 10 July 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.088; data-to-parameter ratio = 13.5.

The title compound, $C_{13}H_{10}ClN_3OS$, was prepared by the reaction of 3-amino-2-chloropyridine with benzoyl isothiocyanate at room temperature. The thiourea group makes dihedral angles of 47.17 (5) and 51.88 (4)°, respectively, with the benzene and pyridyl rings, while the angle between the benzene and pyridine rings is 8.91 (3)°. Intermolecular hydrogen-bond interactions link neighbouring molecules into an infinite supramolecular structure.

Related literature

For the biological activities of benzanilide and its N-substituted derivatives, see: Teoh *et al.* (1999); Campo *et al.* (2002). For the functions of related chlorophenyl compounds, see: Saeed *et al.* (2008); Gowda *et al.* (2008*a*,*b*,*c*). For an isomeric compound, see: Chai *et al.* (2008). For our previous work on thiourea and its derivatives, see: Dong *et al.* (2006, 2007, 2008*a*,*b*). For the synthetic procedure, see: Ding *et al.* (2008).



Experimental

Crystal data

$C_{13}H_{10}ClN_3OS$
M_r = 291.73
Monoclinic, $P2_1/c$
a = 3.9443 (4) Å
b = 14.9250 (15) Å
c = 22.268 (2) Å
β = 93.889 (1)°
V = 1307.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 298 K
0.41 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.839, T_{max} = 0.924
6459 measured reflections
2315 independent reflections
1661 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.088
S = 1.03
2315 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.94	2.633 (2)	137
N1—H1···S1^{i}	0.86	2.74	3.5982 (18)	178
C12—H12···O1^{ii}	0.93	2.70	3.324 (3)	125

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

The authors acknowledge financial support from the 'Jing Lan' Talent Engineering Funds of Lanzhou Jiaotong University and the Natural Science Foundation of the Department of Education, An-Hui Province (grant No. KJ2009B110).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2841).

References

Campo, R., Criado, J. J. & Garcia, E. (2002). *J. Inorg. Biochem.* **89**, 74–82.
Chai, L.-Q., Ding, Y.-J., Yang, X.-Q., Yan, H.-B. & Dong, W.-K. (2008). *Acta Cryst.* E**64**, o1407.
Ding, Y.-J., Chang, X.-B., Yang, X.-Q. & Dong, W.-K. (2008). *Acta Cryst.* E**64**, o658.
Dong, W.-K., Yan, H.-B., Chai, L.-Q., Lv, Z.-W. & Zhao, C.-Y. (2008*a*). *Acta Cryst.* E**64**, o1097.
Dong, W. K., Yang, X. Q., Chai, L. Q., Tian, Y. Q. & Feng, J. H. (2008*b*). *Phosphorus Sulfur Silicon Relat. Elem.* **183**, 1181–1187.
Dong, W.-K., Yang, X.-Q. & Feng, J.-H. (2006). *Acta Cryst.* E**62**, o3459–o3460.
Dong, W. K., Yang, X. Q., Xu, L., Wang, L., Liu, G. L. & Feng, J. H. (2007). *Z. Kristallogr. New Cryst. Struct.* **222**, 279–280.
Gowda, B. T., Foro, S., Sowmya, B. P. & Fuess, H. (2008*a*). *Acta Cryst.* E**64**, o1243.
Gowda, B. T., Foro, S., Sowmya, B. P. & Fuess, H. (2008*b*). *Acta Cryst.* E**64**, o1300.
Gowda, B. T., Foro, S., Sowmya, B. P. & Fuess, H. (2008*c*). *Acta Cryst.* E**64**, o861.
Saeed, A., Hussain, S. & Flörke, U. (2008). *Acta Cryst.* E**64**, o705.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Teoh, S. G., Ang, S. H. & Fun, H. K. (1999). *J. Organomet. Chem.* **580**, 17–21.

supporting information

Acta Cryst. (2009). E**65**, o1903 [doi:10.1107/S1600536809027081]

N-Benzoyl-*N*′-(2-chloro-3-pyridyl)thiourea

Yu-Jie Ding, Jian Yao, Jian-Chao Wu, Wen-Kui Dong and Jun-Feng Tong

S1. Comment

Benzanilide and its N-substituted derivatives have been considered to be a class of privileged structural compounds, which usually have excellent biological activities (Teoh *et al.*, 1999; Campo *et al.*, 2002). However, the literatures are full of the function of the 2-chloro-4-nitrophenyl (Saeed *et al.*, 2008), 3,5-dichlorophenyl (Gowda *et al.*, 2008*a*) and 3-chlorophenyl (Gowda *et al.*, 2008*b*; Gowda *et al.*, 2008*c*) and also structures of benzamide and related compounds. As an extension of our work (Dong *et al.*, 2006; Dong *et al.*, 2007; Dong *et al.*, 2008*a*; Dong *et al.*, 2008*b*) on synthesis and structural characterization of thiourea and its derivatives, here report the synthesis and structure of the title compound.

In the molecule of the title compound, *N*-benzoyl-*N*′-(2-chloro-3-pyridyl)thiourea (Fig. 1), which is isomeric compound to its observed in the structures of *N*-(2-chlorobenzoyl)-*N*′-(3-pyridyl)thiourea (Chai *et al.*, 2008). The thiourea group makes dihedral angles of 47.17 (5)° and 51.88 (4)° with the benzene and pyridyl rings respectively, while the angle between the benzene and pyridine rings is 8.91 (3)°. The carbonyl group forms an intramolecular hydrogen bond with the N2—H2 group, which forms a six-membered ring (C2/N1/C1/N2/H2/O1) structure, the H2···O1 bond length is 1.94 Å. The C═O bond length with 1.218 (3) Å is longer than the average C═O bond length [1.200 Å], which is due to intramolecular hydrogen bonding. This is similar to the situation found in the structure of *N*-benzoyl-*N*′-(3-pyridyl)thiourea (Dong *et al.*, 2006). The crystal structure is further stabilized by intermolecular N1—H1···S1 and C12—H12···O1 hydrogen bonds interactions (Table 1, Fig. 2), which link neighbouring molecules into an infinite supramolecular structure.

S2. Experimental

N-Benzoyl-*N*′-(2-chloro-3-pyridyl)thiourea was synthesized according to an analogous method reported earlier (Ding *et al.*, 2008). Benzoyl chloride (702.8 mg, 5.00 mmol) was reacted with ammonium thiocyanate (380.6 mg, 5.00 mmol) in acetonitrile solution (25 ml) continuring stirring for 3 h at room temperature, to give the corresponding benzoyl isothiocyanate, which was added 3-amino-2-chloropyrldine (642.8 mg, 5.00 mmol). After stirring for 20 h at room temperature, the precipitate was reduced pressure filtered, washed successively with acetonitrile and diethyl ether. The product was dried *in vacuo*, and obtained 599.2 mg of needle-like crystalline solid. Yield, 41.07%. m.p. 424–426 K. Colorless single crystals suitable for X-ray diffraction studies were obtained after two weeks by slow evaporation from a mixture of ethyl acetate/acetone (1:1) of *N*-benzoyl-*N*′-(2-chloro-3-pyridyl)thiourea at room temperture. Analysis calculated for $C_{13}H_{10}ClN_3OS$ (%): C 53.52, H 3.45, N 14.40. Found: C 53.61, H 3.51, N 14.3.

S3. Refinement

H atoms were treated as riding atoms with distances C—H = 0.93 Å (CH), N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(C,N).

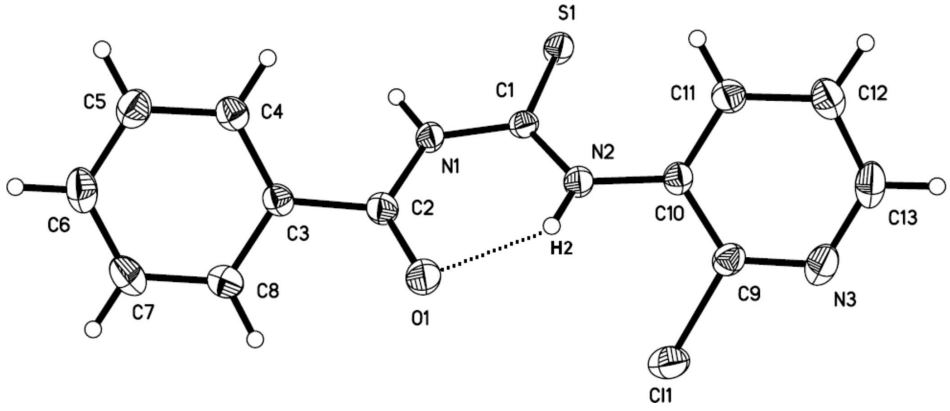


Figure 1

The molecular structure of the title compound with atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

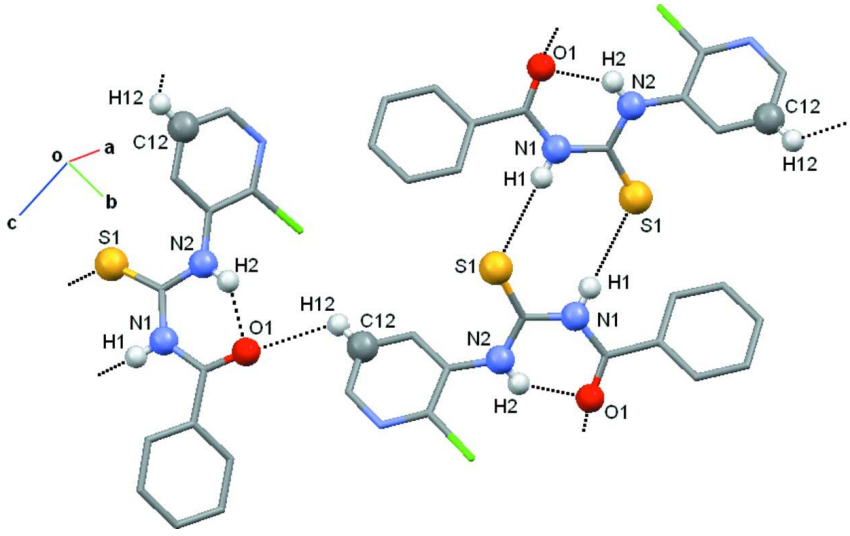


Figure 2

Part of the supramolecular structure of the title compound. Intramolecular and intermolecular hydrogen bonds of the title compound are shown as dashed lines.

N-Benzoyl-*N*′-(2-chloro-3-pyridyl)thiourea

Crystal data

$C_{13}H_{10}ClN_3OS$
$M_r = 291.73$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 3.9443\ (4)$ Å
$b = 14.9250\ (15)$ Å
$c = 22.268\ (2)$ Å
$\beta = 93.889\ (1)°$
$V = 1307.9\ (2)$ Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.482$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2087 reflections
$\theta = 2.3–25.0°$
$\mu = 0.45$ mm^{-1}
$T = 298$ K
Needle-like, colourless
0.41 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.839, T_{max} = 0.924
6459 measured reflections
2315 independent reflections
1661 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 25.0°, θ_{min} = 1.6°
h = −4→4
k = −17→14
l = −26→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.088
S = 1.03
2315 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2 + 0.2536P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.45798 (17)	0.45341 (4)	0.58736 (2)	0.0389 (2)
Cl1	0.4376 (2)	0.65386 (5)	0.77102 (3)	0.0581 (2)
N1	0.6989 (5)	0.61464 (12)	0.56704 (8)	0.0351 (5)
H1	0.6662	0.5986	0.5300	0.042*
N2	0.7371 (5)	0.57099 (12)	0.66633 (7)	0.0380 (5)
H2	0.8131	0.6244	0.6727	0.046*
N3	0.5774 (6)	0.49783 (16)	0.81887 (9)	0.0523 (6)
O1	0.8401 (6)	0.73435 (11)	0.62683 (7)	0.0629 (6)
C1	0.6406 (6)	0.54897 (14)	0.60938 (9)	0.0313 (5)
C2	0.8025 (7)	0.70230 (15)	0.57650 (10)	0.0387 (6)
C3	0.8633 (6)	0.75438 (14)	0.52169 (10)	0.0337 (6)
C4	1.0013 (6)	0.71616 (16)	0.47233 (10)	0.0374 (6)
H4	1.0520	0.6553	0.4724	0.045*
C5	1.0642 (7)	0.76769 (17)	0.42297 (11)	0.0475 (7)
H5	1.1643	0.7420	0.3905	0.057*
C6	0.9790 (7)	0.85723 (18)	0.42174 (12)	0.0541 (8)

H6	1.0176	0.8917	0.3881	0.065*
C7	0.8371 (7)	0.89566 (17)	0.47016 (12)	0.0534 (8)
H7	0.7760	0.9558	0.4688	0.064*
C8	0.7846 (7)	0.84549 (16)	0.52092 (11)	0.0459 (7)
H8	0.6974	0.8723	0.5543	0.055*
C9	0.5921 (6)	0.54468 (16)	0.76902 (10)	0.0386 (6)
C10	0.7247 (6)	0.51388 (15)	0.71689 (9)	0.0331 (6)
C11	0.8605 (7)	0.42887 (16)	0.71795 (11)	0.0415 (6)
H11	0.9587	0.4060	0.6844	0.050*
C12	0.8487 (7)	0.37809 (17)	0.76953 (11)	0.0488 (7)
H12	0.9360	0.3202	0.7713	0.059*
C13	0.7047 (8)	0.41517 (19)	0.81819 (12)	0.0547 (8)
H13	0.6955	0.3805	0.8527	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0473 (4)	0.0371 (4)	0.0322 (3)	−0.0108 (3)	0.0018 (3)	−0.0030 (3)
Cl1	0.0701 (5)	0.0542 (4)	0.0505 (4)	0.0118 (4)	0.0069 (4)	−0.0115 (3)
N1	0.0486 (13)	0.0312 (11)	0.0250 (10)	−0.0073 (10)	−0.0004 (9)	−0.0021 (8)
N2	0.0557 (14)	0.0300 (11)	0.0279 (10)	−0.0078 (10)	−0.0009 (9)	−0.0024 (8)
N3	0.0615 (17)	0.0609 (16)	0.0348 (12)	−0.0110 (13)	0.0049 (11)	0.0027 (11)
O1	0.1150 (19)	0.0399 (10)	0.0337 (10)	−0.0175 (11)	0.0031 (10)	−0.0068 (8)
C1	0.0322 (14)	0.0317 (12)	0.0301 (12)	0.0023 (11)	0.0040 (10)	−0.0022 (10)
C2	0.0478 (17)	0.0328 (14)	0.0352 (14)	−0.0048 (12)	0.0005 (12)	−0.0013 (11)
C3	0.0360 (15)	0.0304 (13)	0.0339 (13)	−0.0053 (11)	−0.0040 (11)	−0.0005 (10)
C4	0.0382 (15)	0.0346 (13)	0.0387 (14)	−0.0050 (11)	−0.0031 (12)	−0.0006 (11)
C5	0.0510 (18)	0.0526 (17)	0.0391 (14)	−0.0094 (14)	0.0046 (13)	−0.0014 (12)
C6	0.065 (2)	0.0529 (18)	0.0436 (16)	−0.0102 (15)	−0.0023 (14)	0.0158 (13)
C7	0.062 (2)	0.0355 (15)	0.0610 (19)	−0.0001 (14)	−0.0046 (16)	0.0089 (13)
C8	0.0536 (18)	0.0362 (15)	0.0479 (15)	0.0007 (13)	0.0037 (13)	−0.0032 (12)
C9	0.0424 (16)	0.0402 (14)	0.0331 (13)	−0.0059 (12)	0.0014 (11)	−0.0041 (11)
C10	0.0360 (15)	0.0332 (13)	0.0294 (12)	−0.0059 (11)	−0.0029 (10)	0.0006 (10)
C11	0.0479 (17)	0.0374 (14)	0.0386 (14)	−0.0008 (12)	−0.0017 (12)	−0.0028 (11)
C12	0.0556 (19)	0.0400 (15)	0.0492 (16)	−0.0053 (14)	−0.0078 (14)	0.0067 (13)
C13	0.067 (2)	0.0562 (18)	0.0398 (16)	−0.0159 (16)	−0.0051 (14)	0.0147 (14)

Geometric parameters (Å, º)

S1—C1	1.657 (2)	C4—H4	0.9300
Cl1—C9	1.741 (2)	C5—C6	1.378 (3)
N1—C2	1.382 (3)	C5—H5	0.9300
N1—C1	1.390 (3)	C6—C7	1.374 (4)
N1—H1	0.8600	C6—H6	0.9300
N2—C1	1.340 (3)	C7—C8	1.383 (3)
N2—C10	1.416 (3)	C7—H7	0.9300
N2—H2	0.8600	C8—H8	0.9300
N3—C9	1.316 (3)	C9—C10	1.384 (3)

N3—C13	1.332 (3)	C10—C11	1.377 (3)
O1—C2	1.218 (3)	C11—C12	1.379 (3)
C2—C3	1.480 (3)	C11—H11	0.9300
C3—C4	1.382 (3)	C12—C13	1.373 (4)
C3—C8	1.395 (3)	C12—H12	0.9300
C4—C5	1.378 (3)	C13—H13	0.9300
C2—N1—C1	128.66 (18)	C7—C6—H6	120.0
C2—N1—H1	115.7	C5—C6—H6	120.0
C1—N1—H1	115.7	C6—C7—C8	120.5 (2)
C1—N2—C10	125.59 (19)	C6—C7—H7	119.8
C1—N2—H2	117.2	C8—C7—H7	119.8
C10—N2—H2	117.2	C7—C8—C3	119.5 (2)
C9—N3—C13	116.4 (2)	C7—C8—H8	120.3
N2—C1—N1	114.80 (19)	C3—C8—H8	120.3
N2—C1—S1	125.53 (17)	N3—C9—C10	124.9 (2)
N1—C1—S1	119.66 (15)	N3—C9—Cl1	116.11 (19)
O1—C2—N1	121.9 (2)	C10—C9—Cl1	119.02 (18)
O1—C2—C3	122.4 (2)	C11—C10—C9	117.4 (2)
N1—C2—C3	115.72 (19)	C11—C10—N2	122.3 (2)
C4—C3—C8	119.5 (2)	C9—C10—N2	120.2 (2)
C4—C3—C2	122.2 (2)	C10—C11—C12	119.0 (2)
C8—C3—C2	118.3 (2)	C10—C11—H11	120.5
C5—C4—C3	120.4 (2)	C12—C11—H11	120.5
C5—C4—H4	119.8	C13—C12—C11	118.3 (2)
C3—C4—H4	119.8	C13—C12—H12	120.8
C6—C5—C4	120.0 (2)	C11—C12—H12	120.8
C6—C5—H5	120.0	N3—C13—C12	123.9 (2)
C4—C5—H5	120.0	N3—C13—H13	118.0
C7—C6—C5	120.1 (2)	C12—C13—H13	118.0
C10—N2—C1—N1	−175.8 (2)	C4—C3—C8—C7	1.8 (4)
C10—N2—C1—S1	5.3 (3)	C2—C3—C8—C7	−179.7 (2)
C2—N1—C1—N2	−8.8 (4)	C13—N3—C9—C10	0.8 (4)
C2—N1—C1—S1	170.2 (2)	C13—N3—C9—Cl1	−178.04 (19)
C1—N1—C2—O1	−3.9 (4)	N3—C9—C10—C11	−2.1 (4)
C1—N1—C2—C3	176.3 (2)	Cl1—C9—C10—C11	176.72 (18)
O1—C2—C3—C4	144.0 (3)	N3—C9—C10—N2	−178.1 (2)
N1—C2—C3—C4	−36.2 (3)	Cl1—C9—C10—N2	0.7 (3)
O1—C2—C3—C8	−34.6 (4)	C1—N2—C10—C11	50.7 (3)
N1—C2—C3—C8	145.3 (2)	C1—N2—C10—C9	−133.5 (2)
C8—C3—C4—C5	0.7 (4)	C9—C10—C11—C12	2.0 (3)
C2—C3—C4—C5	−177.8 (2)	N2—C10—C11—C12	177.9 (2)
C3—C4—C5—C6	−2.2 (4)	C10—C11—C12—C13	−0.8 (4)
C4—C5—C6—C7	1.2 (4)	C9—N3—C13—C12	0.6 (4)
C5—C6—C7—C8	1.2 (4)	C11—C12—C13—N3	−0.6 (4)
C6—C7—C8—C3	−2.7 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	1.94	2.633 (2)	137
N1—H1···S1^{i}	0.86	2.74	3.5982 (18)	178
C12—H12···O1^{ii}	0.93	2.70	3.324 (3)	125

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, *y*−1/2, −*z*+3/2.