Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Chloro-*N*-[3-methyl-1-(5-thioxo-4,5-dihydro-1,3,4-oxadiazol-2-yl)butyl]-benzamide

Yu-Gang Yan, Guo-Gang Tu, Ling-Dong Wang, Jian Liu and Shao-Hua Li*

Department of Medicinal Chemistry, NanChang University School of Pharmaceutical Science, 330006 NanChang, JiangXi, People's Republic of China
Correspondence e-mail: tugg199@yahoo.com

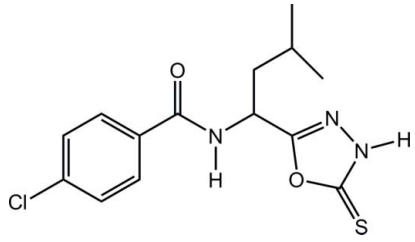
Received 9 May 2010; accepted 11 May 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.050; wR factor = 0.108; data-to-parameter ratio = 15.3.

In the title compound, $C_{14}H_{16}ClN_3O_2S$, the dihedral angle between the 4-chlorophenyl and 1,3,4-oxadiazole rings is 67.1 (1)° and the orientation of the amide N—H and C=O bonds is *anti*. In the crystal, molecules are linked by N—H···O and N—H···S hydrogen bonds.

Related literature

For the biological properties of thiadiazoles, see: Tu *et al.* (2008). For details of the synthesis, see: Ginzel *et al.* (1989); Boland *et al.* (2006); Havaldar & Patil (2009); Shriner & Furrow (1955). For related structures, see: Du *et al.* (2004); Ziyaev *et al.* (1992); Zareef *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{16}ClN_3O_2S$
M_r = 325.81
Orthorhombic, $P2_12_12_1$
a = 6.0171 (6) Å
b = 15.3120 (15) Å
c = 18.1493 (17) Å
V = 1672.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 298 K
0.42 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.864, T_{max} = 0.938
7892 measured reflections
2951 independent reflections
1447 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.108
S = 1.10
2951 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack (1983), 1219 Friedel pairs
Flack parameter: −0.09 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	1.87	2.720 (6)	171
N3—H3···S1^{ii}	0.86	2.78	3.495 (4)	142

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *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* and *publCIF* (Westrip, 2010).

The work was supported by the Key Technologies R & D Program of JiangXi (grant No. 20061B0100400), the Key Science & Technology Program of JiangXi (grant No. 2009BSA14100) and the Scientific Research Fund of NanChang University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5439).

References

Boland, Y., Hertsens, P., Marchand-Brynaert, J. & Garcia, Y. (2006). *Synthesis*, pp. 1504–1512.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Du, M., Zhao, X.-J. & Guo, J.-H. (2004). *Acta Cryst.* E**60**, o327–o328.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Ginzel, K. D., Brungs, P. & Steckhan, E. (1989). *Tetrahedron*, **45**, 1691–1701.
Havaldar, F. H. & Patil, A. R. (2009). *Asian J. Chem.* **21**, 5267–5272.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shriner, R. L. & Furrow, C. L. (1955). *Org. Synth.* **35**, 49–50.
Tu, G. G., Li, S. H., Huang, H. M., Li, G., Xiong, F., Mai, X., Zhu, H. W., Kuang, B. H. & Xu, W. F. (2008). *Bioorg. Med. Chem.* **16**, 6663–6668.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**. Submitted.
Zareef, M., Iqbal, R., Zaidi, J. H., Arfan, M. & Parvez, M. (2006). *Acta Cryst.* E**62**, o2481–o2483.
Ziyaev, A. A., Galust'yan, G. G., Sabirov, K., Nasirov, S., Tashkhodzhaev, B. & Yag'budaev, M. R. (1992). *Zh. Org. Khim.* **28**, 1538–1543.

supplementary materials

Acta Cryst. (2010). E**66**, o1381 [**doi:10.1107/S1600536810017368**]

4-Chloro-*N*-[3-methyl-1-(5-thioxo-4,5-dihydro-1,3,4-oxadiazol-2-yl)butyl]benzamide

Y.-G. Yan, G.-G. Tu, L.-D. Wang, J. Liu and S.-H. Li

Comment

The present oxadiazole derivate is in continuation to our previous work of the thiadiazole scaffold compounds and their biological activity (Tu *et al.*, 2008). The title compound (Figure 1) was synthesized according to literature procedures (Ginzel *et al.*, 1989; Boland *et al.*, 2006; Havaldar & Patil 2009). Here, we report the structure of the title compound.

The oxadiazole ring is essentially planar and is inclined at 67.1 (1)° with respect to the *p*-cholobenzene ring. The N2=C2 and S1=C1 double bonds agree with the corresponding distances in three structures containing similar systems (Du *et al.*, 2004; Ziyaev *et al.*, 1992; Zareef *et al.*, 2006). The conformations of the N—H and C=O bonds are *anti* with respect to each other. The structure is stabilized by a network of intermolecular hydrogen bonds of the type N—H···S (Table 1, Figure 2).

Experimental

To a stirred solution of DL-leucine methyl ester hydrochloride (0.03 mol) in CH_2Cl_2 (20 ml) was added triethylamine (0.06 mol) at 273 K. After 0.5 h, a solution of *p*-chlorobenzoic acid chloride (0.03 mol) in CH_2Cl_2 (10 ml) was added. The mixture was stirred for 2 h at 273 K, then allowed to warm to r.t. for 24 h. Washed with 10% HCl, 1 N NaOH and water. The organic layer was evaporated *in vacuo* and the residue was recrystallized from methanol to give corresponding amides as a white solid.

A mixture of the amides (0.02 mol) and 80% hydrazine monohydrate (0.04 mol) in absolute methanol (20 ml) was heated under reflux over night. After cooling, a white solid was separated and recrystallized from methanol to give corresponding hydrazide.

A mixture of the hydrazide (0.01 mol), KOH (0.01 mol), CS_2 (0.05 mol), and ethanol (70 ml) was heated under reflux with stirring for 12 h. Ethanol was distilled off under reduced pressure and the residue was dissolved in water and then acidified with 10% HCl. The resulting precipitate was filtered, washed with water, and recrystallized from ethanol. Colourless blocks of (I) precipitated after several days.

Refinement

H atoms were positioned geometrically and refined using a riding model using SHELXL97 default values (Uiso(H) = 1.2 Ueq(C) for CH and CH_2 groups and Uiso(H) = 1.5 Ueq(C) for CH_3).

Figures

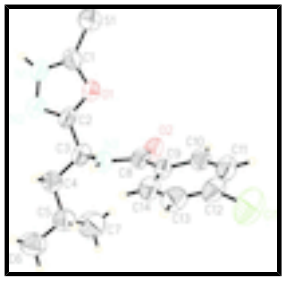


Fig. 1. Molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

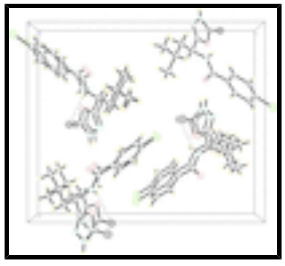


Fig. 2. The crystal packing of (I), viewed along the *a* axis with hydrogen bonds drawn as dashed lines.

4-Chloro-*N*-[3-methyl-1-(5-thioxo-4,5-dihydro-1,3,4-oxadiazol-2- yl)butyl]benzamide

Crystal data

$C_{14}H_{16}ClN_3O_2S$	$F(000) = 680$
$M_r = 325.81$	$D_x = 1.294$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 2117 reflections
$a = 6.0171$ (6) Å	$\theta = 2.6$–$21.7°$
$b = 15.3120$ (15) Å	$\mu = 0.36$ mm^{-1}
$c = 18.1493$ (17) Å	$T = 298$ K
$V = 1672.2$ (3) Å^3	Block, colourless
$Z = 4$	0.42 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer	2951 independent reflections
Radiation source: fine-focus sealed tube	1447 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.056$
ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 7$
$T_{min} = 0.864$, $T_{max} = 0.938$	$k = -18 \rightarrow 11$
7892 measured reflections	$l = -21 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0147P)^2 + 1.0529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.108$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.10$	$\Delta\rho_{max} = 0.31$ e Å^{-3}
2951 reflections	$\Delta\rho_{min} = -0.32$ e Å^{-3}
193 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.0034 (7)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1219 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.09 (14)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	1.2875 (3)	0.07537 (10)	−0.04447 (8)	0.1082 (7)
N1	0.4961 (8)	0.5638 (3)	0.2486 (2)	0.0653 (12)
H1	0.4553	0.6175	0.2515	0.078*
N2	0.6705 (8)	0.5299 (2)	0.2891 (2)	0.0679 (12)
N3	0.9177 (6)	0.3351 (2)	0.22952 (19)	0.0530 (10)
H3	1.0323	0.3577	0.2086	0.064*
O1	0.5141 (6)	0.4303 (2)	0.21792 (16)	0.0650 (10)
O2	0.6632 (6)	0.22820 (19)	0.23058 (17)	0.0672 (10)
S1	0.1860 (3)	0.51382 (10)	0.14928 (8)	0.0902 (5)
C1	0.3976 (8)	0.5066 (3)	0.2049 (2)	0.0601 (13)
C2	0.6745 (10)	0.4498 (3)	0.2683 (2)	0.0555 (13)
C3	0.8216 (9)	0.3780 (3)	0.2935 (2)	0.0572 (13)
H3A	0.7295	0.3352	0.3195	0.069*
C4	0.9999 (9)	0.4102 (3)	0.3469 (2)	0.0628 (14)
H4A	1.0827	0.4567	0.3233	0.075*

H4B	0.9271	0.4348	0.3898	0.075*
C5	1.1631 (10)	0.3406 (3)	0.3725 (3)	0.0793 (17)
H5	1.2452	0.3206	0.3290	0.095*
C6	1.3329 (10)	0.3803 (4)	0.4260 (3)	0.097 (2)
H6A	1.2604	0.3948	0.4715	0.145*
H6B	1.3955	0.4321	0.4046	0.145*
H6C	1.4490	0.3387	0.4353	0.145*
C7	1.0511 (12)	0.2623 (4)	0.4053 (3)	0.129 (3)
H7A	0.9645	0.2799	0.4471	0.193*
H7B	1.1615	0.2208	0.4205	0.193*
H7C	0.9557	0.2360	0.3692	0.193*
C8	0.8305 (9)	0.2601 (3)	0.2021 (2)	0.0520 (12)
C9	0.9491 (9)	0.2185 (3)	0.1398 (3)	0.0518 (13)
C10	0.8393 (9)	0.1520 (3)	0.1025 (2)	0.0575 (13)
H10	0.6966	0.1359	0.1168	0.069*
C11	0.9413 (10)	0.1093 (3)	0.0440 (3)	0.0673 (15)
H11	0.8661	0.0660	0.0181	0.081*
C12	1.1541 (11)	0.1319 (3)	0.0247 (3)	0.0663 (15)
C13	1.2613 (9)	0.1986 (3)	0.0598 (3)	0.0681 (15)
H13	1.4027	0.2153	0.0446	0.082*
C14	1.1604 (9)	0.2411 (3)	0.1174 (3)	0.0639 (14)
H14	1.2355	0.2858	0.1416	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1383 (16)	0.0987 (12)	0.0876 (10)	0.0156 (12)	0.0383 (11)	−0.0126 (9)
N1	0.069 (3)	0.046 (3)	0.081 (3)	0.002 (3)	−0.002 (3)	−0.005 (2)
N2	0.078 (3)	0.047 (3)	0.079 (3)	0.005 (3)	−0.012 (3)	−0.004 (2)
N3	0.050 (3)	0.044 (2)	0.066 (3)	−0.006 (2)	0.007 (2)	−0.0108 (19)
O1	0.079 (3)	0.045 (2)	0.071 (2)	0.002 (2)	−0.019 (2)	−0.0068 (17)
O2	0.069 (3)	0.046 (2)	0.086 (2)	−0.004 (2)	0.016 (2)	−0.0039 (17)
S1	0.0914 (12)	0.0756 (10)	0.1037 (11)	−0.0050 (10)	−0.0315 (10)	0.0091 (9)
C1	0.064 (4)	0.058 (3)	0.058 (3)	−0.009 (3)	−0.004 (3)	−0.002 (3)
C2	0.074 (4)	0.037 (3)	0.056 (3)	0.004 (3)	0.001 (3)	−0.005 (2)
C3	0.072 (4)	0.046 (3)	0.053 (3)	0.001 (3)	0.009 (3)	−0.002 (2)
C4	0.074 (4)	0.056 (3)	0.058 (3)	0.001 (3)	−0.007 (3)	−0.004 (3)
C5	0.088 (5)	0.075 (4)	0.074 (4)	0.001 (4)	−0.015 (4)	0.007 (3)
C6	0.094 (5)	0.109 (5)	0.088 (4)	0.010 (4)	−0.017 (4)	0.003 (3)
C7	0.140 (7)	0.092 (5)	0.154 (6)	−0.018 (5)	−0.040 (5)	0.056 (5)
C8	0.056 (3)	0.043 (3)	0.057 (3)	0.000 (3)	0.000 (3)	0.001 (2)
C9	0.057 (3)	0.037 (3)	0.062 (3)	−0.002 (3)	−0.006 (3)	−0.003 (2)
C10	0.062 (4)	0.044 (3)	0.067 (3)	−0.004 (3)	0.001 (3)	0.002 (2)
C11	0.097 (5)	0.048 (3)	0.056 (3)	0.001 (3)	−0.001 (3)	−0.001 (3)
C12	0.084 (5)	0.055 (3)	0.059 (3)	0.011 (4)	0.007 (3)	0.005 (3)
C13	0.063 (4)	0.068 (4)	0.074 (3)	0.005 (3)	0.014 (3)	0.006 (3)
C14	0.067 (4)	0.051 (3)	0.074 (3)	−0.003 (3)	−0.002 (3)	0.000 (3)

Geometric parameters (Å, °)

Cl1—C12	1.723 (5)	C5—C6	1.535 (7)
N1—C1	1.321 (5)	C5—H5	0.9800
N1—N2	1.382 (5)	C6—H6A	0.9600
N1—H1	0.8600	C6—H6B	0.9600
N2—C2	1.283 (5)	C6—H6C	0.9600
N3—C8	1.357 (5)	C7—H7A	0.9600
N3—C3	1.454 (5)	C7—H7B	0.9600
N3—H3	0.8600	C7—H7C	0.9600
O1—C2	1.363 (5)	C8—C9	1.481 (6)
O1—C1	1.382 (5)	C9—C14	1.379 (6)
O2—C8	1.232 (5)	C9—C10	1.390 (6)
S1—C1	1.629 (5)	C10—C11	1.390 (6)
C2—C3	1.483 (6)	C10—H10	0.9300
C3—C4	1.527 (6)	C11—C12	1.372 (7)
C3—H3A	0.9800	C11—H11	0.9300
C4—C5	1.522 (6)	C12—C13	1.366 (6)
C4—H4A	0.9700	C13—C14	1.373 (6)
C4—H4B	0.9700	C13—H13	0.9300
C5—C7	1.498 (7)	C14—H14	0.9300
C1—N1—N2	114.3 (4)	C5—C6—H6B	109.5
C1—N1—H1	122.9	H6A—C6—H6B	109.5
N2—N1—H1	122.9	C5—C6—H6C	109.5
C2—N2—N1	102.5 (4)	H6A—C6—H6C	109.5
C8—N3—C3	121.5 (4)	H6B—C6—H6C	109.5
C8—N3—H3	119.3	C5—C7—H7A	109.5
C3—N3—H3	119.3	C5—C7—H7B	109.5
C2—O1—C1	106.8 (3)	H7A—C7—H7B	109.5
N1—C1—O1	103.3 (4)	C5—C7—H7C	109.5
N1—C1—S1	132.6 (4)	H7A—C7—H7C	109.5
O1—C1—S1	124.0 (4)	H7B—C7—H7C	109.5
N2—C2—O1	113.1 (5)	O2—C8—N3	119.8 (4)
N2—C2—C3	128.9 (5)	O2—C8—C9	122.9 (4)
O1—C2—C3	117.9 (4)	N3—C8—C9	117.2 (4)
N3—C3—C2	109.0 (3)	C14—C9—C10	118.6 (5)
N3—C3—C4	111.9 (4)	C14—C9—C8	124.2 (4)
C2—C3—C4	112.1 (4)	C10—C9—C8	117.3 (5)
N3—C3—H3A	107.9	C11—C10—C9	120.4 (5)
C2—C3—H3A	107.9	C11—C10—H10	119.8
C4—C3—H3A	107.9	C9—C10—H10	119.8
C5—C4—C3	114.9 (4)	C12—C11—C10	119.3 (5)
C5—C4—H4A	108.5	C12—C11—H11	120.4
C3—C4—H4A	108.5	C10—C11—H11	120.4
C5—C4—H4B	108.5	C13—C12—C11	120.7 (5)
C3—C4—H4B	108.5	C13—C12—Cl1	119.7 (5)
H4A—C4—H4B	107.5	C11—C12—Cl1	119.6 (5)
C7—C5—C4	113.0 (5)	C12—C13—C14	120.1 (5)

C7—C5—C6	111.4 (5)	C12—C13—H13	120.0
C4—C5—C6	110.2 (4)	C14—C13—H13	120.0
C7—C5—H5	107.3	C13—C14—C9	120.9 (5)
C4—C5—H5	107.3	C13—C14—H14	119.6
C6—C5—H5	107.3	C9—C14—H14	119.6
C5—C6—H6A	109.5		
C1—N1—N2—C2	0.1 (6)	C3—C4—C5—C6	−179.7 (4)
N2—N1—C1—O1	0.3 (5)	C3—N3—C8—O2	1.7 (7)
N2—N1—C1—S1	178.2 (4)	C3—N3—C8—C9	−176.2 (4)
C2—O1—C1—N1	−0.5 (5)	O2—C8—C9—C14	−165.1 (4)
C2—O1—C1—S1	−178.7 (3)	N3—C8—C9—C14	12.7 (7)
N1—N2—C2—O1	−0.5 (5)	O2—C8—C9—C10	14.3 (7)
N1—N2—C2—C3	−178.1 (5)	N3—C8—C9—C10	−167.9 (4)
C1—O1—C2—N2	0.7 (5)	C14—C9—C10—C11	0.1 (7)
C1—O1—C2—C3	178.6 (4)	C8—C9—C10—C11	−179.3 (4)
C8—N3—C3—C2	−98.1 (5)	C9—C10—C11—C12	1.8 (7)
C8—N3—C3—C4	137.4 (4)	C10—C11—C12—C13	−3.5 (7)
N2—C2—C3—N3	−129.7 (5)	C10—C11—C12—Cl1	176.5 (3)
O1—C2—C3—N3	52.8 (6)	C11—C12—C13—C14	3.2 (8)
N2—C2—C3—C4	−5.2 (8)	Cl1—C12—C13—C14	−176.7 (4)
O1—C2—C3—C4	177.2 (4)	C12—C13—C14—C9	−1.2 (7)
N3—C3—C4—C5	−54.7 (6)	C10—C9—C14—C13	−0.4 (7)
C2—C3—C4—C5	−177.5 (4)	C8—C9—C14—C13	178.9 (4)
C3—C4—C5—C7	−54.3 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	1.87	2.720 (6)	171
N3—H3···S1^{ii}	0.86	2.78	3.495 (4)	142

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

Fig. 1

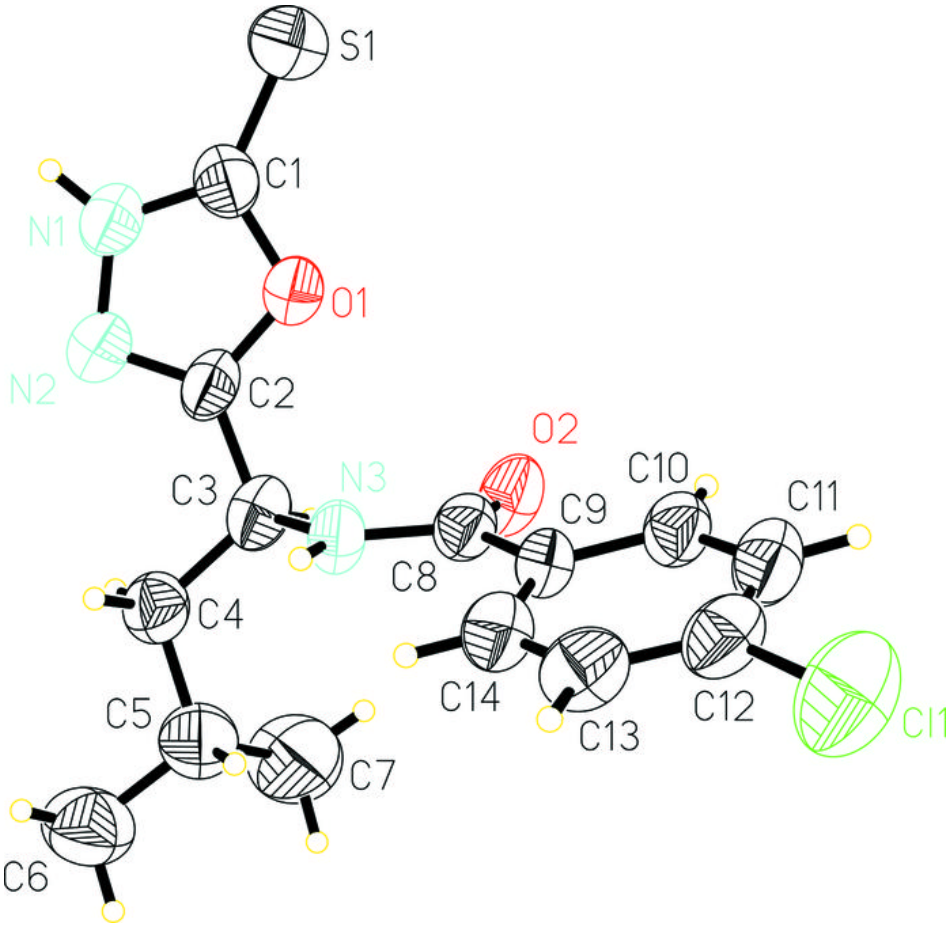


Fig. 2