Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(3,4-Dichlorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethyleneamino *N*-methylcarbamate

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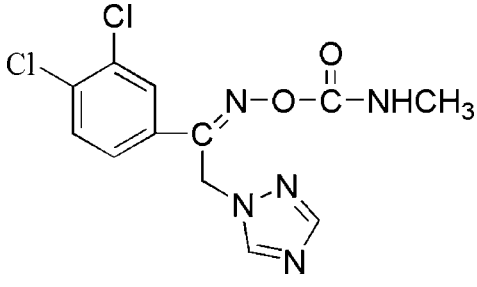
Received 6 June 2007; accepted 11 July 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.111; data-to-parameter ratio = 13.1.

In the title compound, $C_{12}H_{11}Cl_2N_5O_2$, the triazole and benzene rings make a dihedral angle of 89.79 (2)°. Intermolecular N—H···N and C—H···O hydrogen bonds link the molecules into zigzag chains running in the [101] direction.

Related literature

For the crystal structures of related compounds, see: Çoruh *et al.* (2003); Li *et al.* (2005); Puviarasan *et al.* (1999). For details of the biological activities of triazole compounds, see: Xu *et al.* (2002).



Experimental

Crystal data

$C_{12}H_{11}Cl_2N_5O_2$
M_r = 328.16
Triclinic, $P\overline{1}$
a = 6.016 (2) Å
b = 8.430 (2) Å
c = 14.721 (4) Å
α = 101.501 (4)°
β = 100.057 (4)°
γ = 93.977 (4)°
V = 716.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.873, T_{max} = 0.921
3665 measured reflections
2497 independent reflections
1863 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.111
S = 1.04
2497 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···N3^{i}	0.86	2.33	3.035 (3)	139
C9—H9···O2^{ii}	0.93	2.28	3.191 (3)	167

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2181).

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  doi:10.1107/S1600536807033880

supplementary materials

Acta Cryst. (2007). E**63**, o3512 [**doi:10.1107/S1600536807033880**]

1-(3,4-Dichlorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethyleneamino *N*-methylcarbamate

S.-Y. Niu, G.-S. Zhang, Y.-X. Jiang, P.-Y. Zhang and L.-Z. Xu

Comment

The derivatives of 1*H*-1,2,4-triazoles have been reported to possess various biological activities (Xu *et al.*, 2002). In a search for new triazole compounds with better biological activity, the title compound, (I), was synthesized. We report here the crystal structure of (I).

Bond lengths and angles of the triazole rings are in agreement with those in previous reports (Çoruh *et al.*, 2003; Li *et al.*, 2005). The N=C bond length [N4=C7 = 1.285 (4) Å] are close to the value reported in the literature (Puviarasan *et al.*, 1999). Atoms Cl1/Cl2/O1/N4/C7/C8/C11 lies in the benzene ring (C1/C2/C3/C4/C5/C6) plane, and the deviations from the least-squares plane through the ring atoms are all smaller than 0.046 (2) Å. The dihedral angle between the plane of benzene and triazole (N1/N2/N3/C9/C10) ring is 89.79 (2)°. Intermolecular hydrogen bonds N—H···N and C—H···O link the molecules into zigzag chains running in the [101] direction. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

1-(3,4-dichlorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanoxime (0.01 mol) was treated with triethylamine (0.0105 mol) and 40 ml trichloromethane, then the mixture was refluxed for 20 h. The resulting product was filtered off and dried *in vacuo*. The residue was purified by silica gel flash chromatography using a gradient from a 3:1 mixture of petroleum ether:ethyl acetate as eluent. (72% yield). mp 421–423 K. ^{1}H NMR ($CDCl_3$, 600 MHz) δ: 8.35 (s, 1H), 7.95 (s, 1H), 7.93–7.54 (m, 3H), 6.28 (s, 1H), 5.52 (s, 2H). IR (KBr) cm^{-1}: 3337, 3098, 2945, 2852, 1743, 1508. Anal. Calc. for $C_{12}H_{11}Cl_2N_5O_2$ (Mr=328.1): C 43.92, H 3.38, N 21.34; Found C 44.25, H 3.19, N 21.78.

Refinement

All H atoms were placed in calculated positions, with N—H = 0.86 and C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2(1.5 for methyl groups) times U_{eq}(C,*N*).

Figures

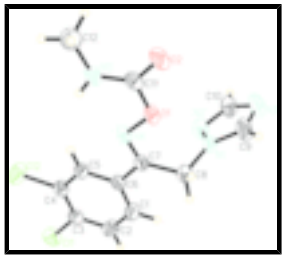

Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

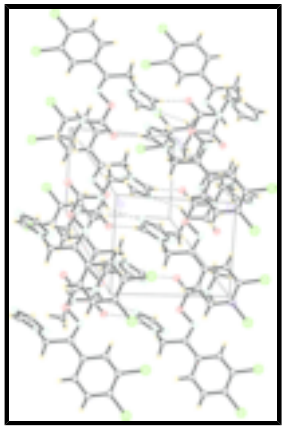

Fig. 2. A packing diagram of the molecule of the title compound. Hydrogen bonds are shown as dashed lines.

1-(3,4-Dichlorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethyleneamino *N*-methylcarbamate

Crystal data

$C_{12}H_{11}Cl_2N_5O_2$	$Z = 2$
$M_r = 328.16$	$F_{000} = 336$
Triclinic, $P\bar{1}$	$D_x = 1.522$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.016$ (2) Å	Cell parameters from 1527 reflections
$b = 8.430$ (2) Å	$\theta = 2.9$–26.3°
$c = 14.721$ (4) Å	$\mu = 0.47$ mm^{-1}
$\alpha = 101.501$ (4)°	$T = 294$ (2) K
$\beta = 100.057$ (4)°	Block, white
$\gamma = 93.977$ (4)°	0.30 × 0.20 × 0.18 mm
$V = 716.1$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2497 independent reflections
Radiation source: fine-focus sealed tube	1863 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 294$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 2.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 6$
$T_{min} = 0.873$, $T_{max} = 0.921$	$k = -10 \rightarrow 5$
3665 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.111$	$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.1094P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.004$
2497 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
191 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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	1.29314 (11)	−0.19171 (9)	0.01383 (5)	0.0566 (2)
Cl2	0.87282 (14)	−0.36122 (8)	0.07119 (6)	0.0773 (3)
O1	0.5256 (3)	0.19831 (19)	0.37808 (11)	0.0459 (4)
O2	0.2410 (3)	0.2024 (2)	0.45331 (12)	0.0561 (5)
N1	0.6867 (3)	0.4563 (2)	0.29428 (14)	0.0410 (5)
N2	0.5955 (4)	0.4215 (2)	0.20095 (15)	0.0516 (6)
N3	0.4610 (5)	0.6417 (3)	0.27541 (19)	0.0683 (7)
N4	0.6348 (3)	0.0963 (2)	0.31413 (13)	0.0396 (5)
N5	0.3195 (3)	−0.0440 (2)	0.37867 (14)	0.0458 (5)
H5	0.4090	−0.0934	0.3457	0.055*
C1	1.1004 (4)	0.1653 (3)	0.19469 (17)	0.0454 (6)
H1	1.1468	0.2754	0.2195	0.054*
C2	1.2155 (4)	0.0786 (3)	0.13105 (17)	0.0467 (6)
H2	1.3387	0.1304	0.1141	0.056*
C3	1.1487 (4)	−0.0836 (3)	0.09290 (16)	0.0404 (6)
C4	0.9628 (4)	−0.1587 (3)	0.11822 (17)	0.0427 (6)
C5	0.8491 (4)	−0.0720 (3)	0.18221 (17)	0.0417 (6)
H5A	0.7253	−0.1238	0.1988	0.050*
C6	0.9175 (4)	0.0918 (3)	0.22214 (15)	0.0354 (5)
C7	0.7949 (4)	0.1808 (3)	0.29180 (15)	0.0369 (5)
C8	0.8565 (4)	0.3608 (3)	0.33421 (19)	0.0482 (6)
H8A	0.8673	0.3800	0.4021	0.058*
H8B	1.0037	0.3952	0.3220	0.058*
C9	0.6048 (5)	0.5866 (3)	0.3364 (2)	0.0624 (8)

H9	0.6438	0.6330	0.4006	0.075*
C10	0.4608 (5)	0.5358 (3)	0.1939 (2)	0.0580 (7)
H10	0.3718	0.5433	0.1368	0.070*
C11	0.3497 (4)	0.1160 (3)	0.40575 (16)	0.0417 (6)
C12	0.1374 (5)	−0.1376 (3)	0.4038 (2)	0.0616 (8)
H12A	0.1642	−0.1271	0.4710	0.092*
H12B	0.1316	−0.2501	0.3734	0.092*
H12C	−0.0044	−0.0975	0.3835	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0555 (4)	0.0657 (5)	0.0554 (4)	0.0165 (3)	0.0292 (3)	0.0097 (3)
Cl2	0.0810 (6)	0.0403 (4)	0.1080 (7)	−0.0039 (3)	0.0551 (5)	−0.0184 (4)
O1	0.0534 (10)	0.0356 (9)	0.0479 (10)	0.0124 (7)	0.0190 (8)	−0.0032 (7)
O2	0.0617 (11)	0.0552 (11)	0.0528 (10)	0.0217 (9)	0.0260 (9)	−0.0035 (8)
N1	0.0464 (12)	0.0286 (10)	0.0475 (12)	0.0063 (8)	0.0165 (9)	−0.0001 (8)
N2	0.0702 (15)	0.0400 (12)	0.0470 (13)	0.0127 (11)	0.0180 (11)	0.0063 (10)
N3	0.0821 (18)	0.0472 (14)	0.0761 (17)	0.0288 (13)	0.0179 (14)	0.0045 (13)
N4	0.0458 (12)	0.0331 (11)	0.0412 (11)	0.0143 (9)	0.0155 (9)	0.0012 (8)
N5	0.0538 (13)	0.0392 (12)	0.0514 (12)	0.0166 (9)	0.0264 (10)	0.0080 (9)
C1	0.0487 (15)	0.0377 (13)	0.0485 (14)	0.0001 (11)	0.0099 (12)	0.0075 (11)
C2	0.0441 (14)	0.0474 (15)	0.0504 (15)	−0.0014 (11)	0.0162 (12)	0.0106 (12)
C3	0.0394 (13)	0.0477 (15)	0.0368 (12)	0.0117 (11)	0.0118 (10)	0.0094 (11)
C4	0.0449 (14)	0.0342 (13)	0.0490 (14)	0.0074 (10)	0.0149 (11)	0.0029 (11)
C5	0.0392 (13)	0.0343 (13)	0.0520 (14)	0.0039 (10)	0.0182 (11)	0.0022 (11)
C6	0.0353 (13)	0.0332 (12)	0.0373 (12)	0.0083 (10)	0.0048 (10)	0.0070 (10)
C7	0.0361 (13)	0.0312 (12)	0.0401 (13)	0.0080 (10)	0.0031 (10)	0.0023 (10)
C8	0.0440 (15)	0.0357 (14)	0.0576 (16)	0.0055 (11)	0.0076 (12)	−0.0054 (11)
C9	0.077 (2)	0.0445 (16)	0.0593 (17)	0.0199 (14)	0.0163 (15)	−0.0105 (13)
C10	0.0747 (19)	0.0424 (16)	0.0625 (18)	0.0124 (14)	0.0167 (15)	0.0188 (13)
C11	0.0429 (14)	0.0489 (15)	0.0342 (12)	0.0145 (11)	0.0086 (11)	0.0065 (11)
C12	0.0643 (18)	0.0607 (18)	0.0730 (19)	0.0128 (14)	0.0342 (15)	0.0255 (15)

Geometric parameters (Å, °)

Cl1—C3	1.727 (2)	C1—H1	0.9300
Cl2—C4	1.719 (2)	C2—C3	1.373 (3)
O1—C11	1.388 (3)	C2—H2	0.9300
O1—N4	1.424 (2)	C3—C4	1.390 (3)
O2—C11	1.213 (3)	C4—C5	1.382 (3)
N1—C9	1.320 (3)	C5—C6	1.391 (3)
N1—N2	1.352 (3)	C5—H5A	0.9300
N1—C8	1.460 (3)	C6—C7	1.481 (3)
N2—C10	1.309 (3)	C7—C8	1.514 (3)
N3—C9	1.312 (4)	C8—H8A	0.9700
N3—C10	1.345 (4)	C8—H8B	0.9700
N4—C7	1.285 (3)	C9—H9	0.9300
N5—C11	1.318 (3)	C10—H10	0.9300

N5—C12	1.448 (3)	C12—H12A	0.9600
N5—H5	0.8600	C12—H12B	0.9600
C1—C2	1.384 (3)	C12—H12C	0.9600
C1—C6	1.385 (3)		
C11—O1—N4	113.69 (16)	C1—C6—C7	122.5 (2)
C9—N1—N2	109.4 (2)	C5—C6—C7	119.6 (2)
C9—N1—C8	129.2 (2)	N4—C7—C6	116.11 (19)
N2—N1—C8	121.33 (18)	N4—C7—C8	122.3 (2)
C10—N2—N1	102.3 (2)	C6—C7—C8	121.6 (2)
C9—N3—C10	102.4 (2)	N1—C8—C7	110.93 (19)
C7—N4—O1	110.06 (18)	N1—C8—H8A	109.5
C11—N5—C12	120.7 (2)	C7—C8—H8A	109.5
C11—N5—H5	119.6	N1—C8—H8B	109.5
C12—N5—H5	119.6	C7—C8—H8B	109.5
C2—C1—C6	121.4 (2)	H8A—C8—H8B	108.0
C2—C1—H1	119.3	N3—C9—N1	110.9 (3)
C6—C1—H1	119.3	N3—C9—H9	124.6
C3—C2—C1	120.2 (2)	N1—C9—H9	124.6
C3—C2—H2	119.9	N2—C10—N3	114.9 (3)
C1—C2—H2	119.9	N2—C10—H10	122.5
C2—C3—C4	119.2 (2)	N3—C10—H10	122.5
C2—C3—Cl1	120.16 (19)	O2—C11—N5	127.4 (2)
C4—C3—Cl1	120.65 (19)	O2—C11—O1	114.8 (2)
C5—C4—C3	120.4 (2)	N5—C11—O1	117.73 (19)
C5—C4—Cl2	119.09 (19)	N5—C12—H12A	109.5
C3—C4—Cl2	120.48 (18)	N5—C12—H12B	109.5
C4—C5—C6	120.8 (2)	H12A—C12—H12B	109.5
C4—C5—H5A	119.6	N5—C12—H12C	109.5
C6—C5—H5A	119.6	H12A—C12—H12C	109.5
C1—C6—C5	117.9 (2)	H12B—C12—H12C	109.5
C9—N1—N2—C10	−0.4 (3)	C1—C6—C7—N4	−177.0 (2)
C8—N1—N2—C10	−177.9 (2)	C5—C6—C7—N4	2.8 (3)
C11—O1—N4—C7	179.96 (19)	C1—C6—C7—C8	2.7 (3)
C6—C1—C2—C3	0.5 (4)	C5—C6—C7—C8	−177.5 (2)
C1—C2—C3—C4	0.7 (4)	C9—N1—C8—C7	141.5 (3)
C1—C2—C3—Cl1	−179.79 (19)	N2—N1—C8—C7	−41.6 (3)
C2—C3—C4—C5	−1.1 (4)	N4—C7—C8—N1	−74.3 (3)
Cl1—C3—C4—C5	179.40 (18)	C6—C7—C8—N1	106.1 (2)
C2—C3—C4—Cl2	179.50 (19)	C10—N3—C9—N1	0.2 (3)
Cl1—C3—C4—Cl2	0.0 (3)	N2—N1—C9—N3	0.1 (3)
C3—C4—C5—C6	0.3 (4)	C8—N1—C9—N3	177.3 (2)
Cl2—C4—C5—C6	179.70 (19)	N1—N2—C10—N3	0.6 (3)
C2—C1—C6—C5	−1.3 (4)	C9—N3—C10—N2	−0.6 (4)
C2—C1—C6—C7	178.5 (2)	C12—N5—C11—O2	1.3 (4)
C4—C5—C6—C1	0.9 (3)	C12—N5—C11—O1	−179.1 (2)
C4—C5—C6—C7	−178.9 (2)	N4—O1—C11—O2	−173.63 (18)
O1—N4—C7—C6	−177.40 (17)	N4—O1—C11—N5	6.7 (3)
O1—N4—C7—C8	2.9 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N3^{i}	0.86	2.33	3.035 (3)	139
C9—H9···O2^{ii}	0.93	2.28	3.191 (3)	167

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

Fig. 1

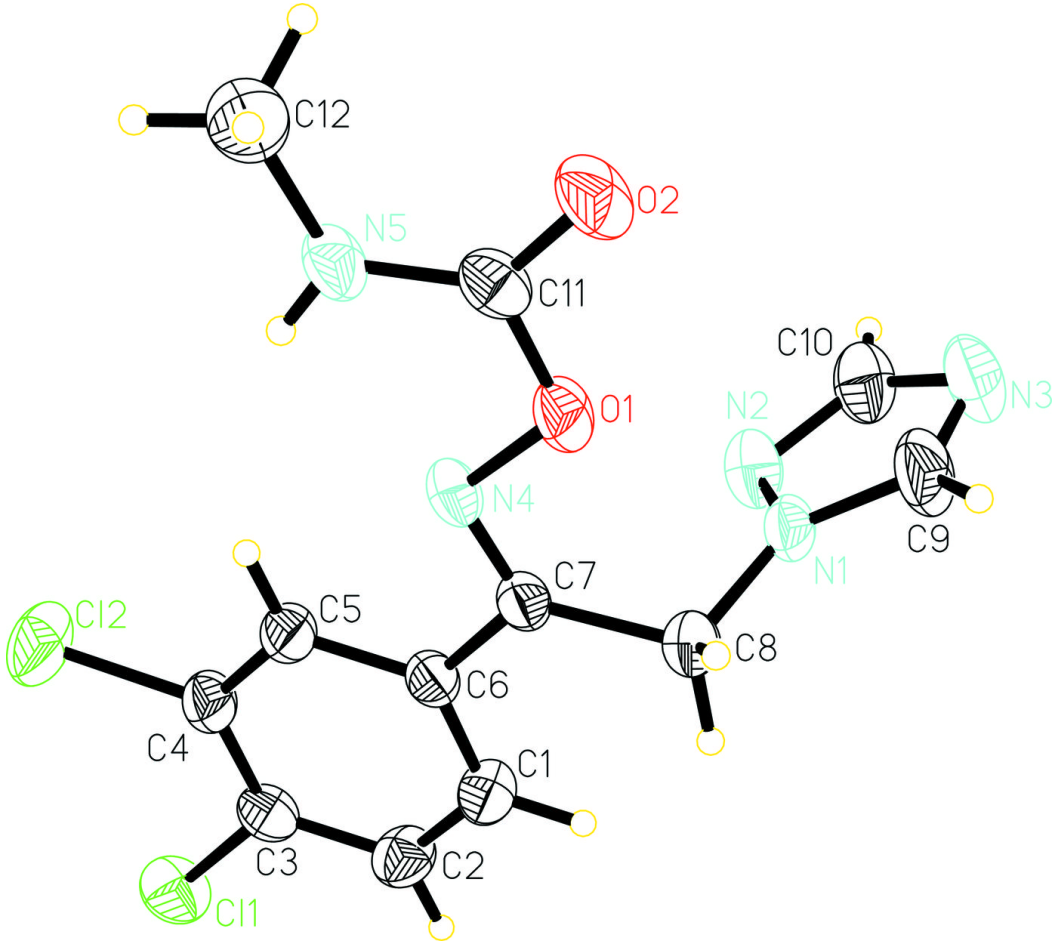

Fig. 2