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1-[2-(4-Chlorophenyl)acetyl]-4-(2-methoxyphenyl)thiosemicarbazide

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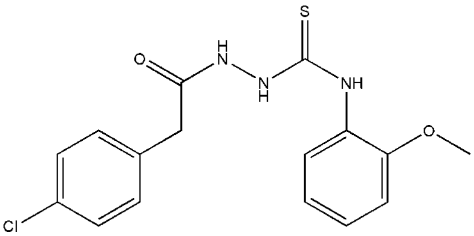
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.111; data-to-parameter ratio = 18.5.

The title compound, $C_{16}H_{16}ClN_3O_2S$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. In the molecule, the thiosemicarbazide group is approximately planar and forms dihedral angles of 33.03 (6) and 45.48 (5)° with the benzene rings. The structure is stabilized by intramolecular N—H⋯O, N—H⋯N and C—H⋯S hydrogen bonds and intermolecular N—H⋯O and N—H⋯S hydrogen bonds.

Related literature

For general background, see Shen *et al.* (1998); Mao *et al.* (1999); Antholine & Taketa (1982). For related literature, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{16}ClN_3O_2S$
M_r = 349.83
Monoclinic, $P2_1/n$
a = 5.935 (5) Å
b = 16.970 (5) Å
c = 17.038 (5) Å
β = 98.948 (5)°
V = 1695.1 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 293 (2) K
0.36 × 0.31 × 0.21 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.881, T_{max} = 0.928
10318 measured reflections
4083 independent reflections
3190 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
S = 1.04
4083 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯S1^{i}	0.862 (18)	2.435 (18)	3.269 (3)	163.1 (16)
N2—H2N⋯O1^{ii}	0.865 (19)	2.159 (18)	2.941 (3)	150.1 (16)
N3—H3N⋯O2	0.810 (18)	2.099 (17)	2.551 (3)	115.2 (15)
N3—H3N⋯N1	0.810 (18)	2.229 (17)	2.656 (3)	113.3 (14)
C11—H11⋯S1	0.93	2.61	3.261 (3)	128

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2274).

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supplementary materials

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1-[2-(4-Chlorophenyl)acetyl]-4-(2-methoxyphenyl)thiosemicarbazide

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Comment

Thiosemicarbazide is interesting because of the formation of complexes with biological activities (Shen *et al.*, 1998). Some substituted thiourea derivatives have shown interesting biological effects, including anti-HIV properties (Mao *et al.*, 1999), and thiourea derivatives have also been successfully screened for various biological actions (Antholine & Taketa, 1982). As a ligand with potential S– and N-atom donors, thiosemicarbazide is interesting because of the structural chemistry of its multifunctional coordination modes (monodentate, S-monodentate or N:*S*-bidentate). In order to investigate further this kind of ligand, we synthesized the title compound, and describe its structure here.

In the molecule of the titel compound (Fig. 1), the bond lengths and angles are in normal ranges (Allen *et al.*, 1987). The thiosemicarbazide is approximately planar, maximum displacement being 0.132 (2) Å for atom N2, and forms dihedral angles of 33.03 (6)° and 45.48 (5)° with the benzene rings. The dihedral angles between the benzene rings is 55.29 (3)°. The structure is stabilized by intra-molecular N—H···O, N—H···N and C—H···S hydrogen bonding and by inter-molecular N—H···O and N—H···S hydrogen bonding (Table 1).

Experimental

2-(4-Chlorophenyl)acetohydrazide (3.68 g, 20 mmol) was dissolved in absolute ethanol (200 ml). 2-Methoxyphenyl isothiocyanate (3.3 g, 20 mmol) was dissolved in ethanol (100 ml). The two solution were mixed with continuous stirring. The mixture was then refluxed and the completion of reaction was monitored by TLC. After the completion of the reaction (Fig. 2), the mixture was cooled to room temperature to get the crude solid. The crude crystalline solid was then filtered. Single crystals suitable for X-ray measurements were obtained by recrystallization from an aqueous-ethanol solution at room temperature (yield: 86%).

Refinement

Imino H atoms were located in a difference Fourier map and refined isotropically. Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angle was refined to fit the electron density, $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined in riding mode with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Figures

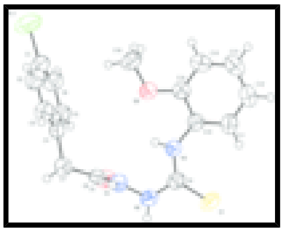


Fig. 1. The molecular structure of (I) with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Fig. 2. The reaction scheme.

1-[2-(4-Chlorophenyl)acetyl]-4-(2-methoxyphenyl)thiosemicarbazide

Crystal data

$C_{16}H_{16}ClN_3O_2S$

M_r = 349.83

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

a = 5.935 (5) Å

b = 16.970 (5) Å

c = 17.038 (5) Å

β = 98.948 (5)°

V = 1695.1 (16) Å^3

Z = 4

F_{000} = 728

D_x = 1.371 Mg m^{-3}

Melting point: 432(1) K

Mo $K\alpha$ radiation
λ = 0.71069 Å

Cell parameters from 1520 reflections

θ = 2.7–24.9°

μ = 0.36 mm^{-1}

T = 293 (2) K

Block, colourless

0.36 × 0.31 × 0.21 mm

Data collection

Bruker APEXII
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 293(2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min}$ = 0.881, $T_{\max}$ = 0.928

10318 measured reflections

4083 independent reflections

3190 reflections with $I > 2\sigma(I)$

R_{int} = 0.020

$\theta_{\max}$ = 28.3°

$\theta_{\min}$ = 1.7°

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 22$

$l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

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

$wR(F^2)$ = 0.111	$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.2584P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
4083 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
221 parameters	$\Delta\rho_{min}$ = −0.34 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 > 2sigma(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}
S1	0.46464 (7)	0.03318 (2)	0.20016 (3)	0.06242 (15)
Cl1	−0.51266 (12)	0.46846 (3)	−0.10995 (4)	0.1040 (2)
O1	−0.0087 (2)	0.09248 (7)	−0.02343 (7)	0.0602 (3)
O2	0.04254 (19)	0.30128 (6)	0.13643 (7)	0.0631 (3)
N3	0.2018 (2)	0.16318 (7)	0.16729 (8)	0.0513 (3)
C10	0.3444 (2)	0.22540 (9)	0.19978 (8)	0.0466 (3)
N2	0.0441 (2)	0.04438 (8)	0.13054 (9)	0.0533 (3)
N1	−0.1492 (2)	0.08226 (8)	0.09156 (8)	0.0520 (3)
C11	0.5588 (3)	0.21836 (11)	0.24479 (9)	0.0576 (4)
H11	0.6248	0.1689	0.2546	0.069*
C15	0.2504 (2)	0.30081 (9)	0.18458 (9)	0.0494 (3)
C8	−0.1695 (3)	0.09808 (8)	0.01264 (10)	0.0497 (3)
C9	0.2302 (2)	0.08540 (8)	0.16440 (9)	0.0466 (3)
C7	−0.4040 (3)	0.12490 (10)	−0.02463 (11)	0.0596 (4)
H7A	−0.5118	0.1114	0.0106	0.072*
H7B	−0.4481	0.0965	−0.0740	0.072*
C3	−0.5936 (3)	0.25642 (11)	−0.01775 (10)	0.0590 (4)
H3	−0.6926	0.2327	0.0124	0.071*
C4	−0.4195 (2)	0.21246 (9)	−0.04146 (9)	0.0481 (3)
C6	−0.2969 (3)	0.32856 (10)	−0.10469 (10)	0.0608 (4)
H6	−0.1955	0.3531	−0.1333	0.073*
C2	−0.6225 (3)	0.33490 (11)	−0.03821 (11)	0.0670 (5)
H2	−0.7416	0.3636	−0.0227	0.080*
C16	−0.0646 (3)	0.37530 (11)	0.11639 (12)	0.0712 (5)
H16A	−0.0862	0.4024	0.1641	0.107*

H16B	−0.2099	0.3668	0.0839	0.107*
H16C	0.0301	0.4065	0.0876	0.107*
C12	0.6742 (3)	0.28520 (13)	0.27499 (11)	0.0678 (5)
H12	0.8193	0.2804	0.3044	0.081*
C5	−0.2709 (3)	0.24995 (10)	−0.08487 (10)	0.0548 (4)
H5	−0.1519	0.2215	−0.1008	0.066*
C14	0.3653 (3)	0.36647 (10)	0.21688 (10)	0.0620 (4)
H14	0.3003	0.4162	0.2082	0.074*
C1	−0.4738 (3)	0.37023 (10)	−0.08173 (10)	0.0612 (4)
C13	0.5786 (3)	0.35796 (12)	0.26236 (11)	0.0702 (5)
H13	0.6569	0.4021	0.2844	0.084*
H2N	0.058 (3)	−0.0038 (11)	0.1154 (10)	0.060 (5)*
H1N	−0.267 (3)	0.0779 (10)	0.1151 (11)	0.065 (5)*
H3N	0.077 (3)	0.1785 (10)	0.1470 (10)	0.057 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0499 (2)	0.0534 (2)	0.0833 (3)	0.01080 (17)	0.0084 (2)	0.0213 (2)
Cl1	0.1198 (5)	0.0613 (3)	0.1301 (5)	0.0233 (3)	0.0165 (4)	0.0244 (3)
O1	0.0582 (7)	0.0519 (6)	0.0728 (7)	0.0018 (5)	0.0174 (6)	0.0054 (5)
O2	0.0571 (7)	0.0471 (6)	0.0800 (8)	0.0093 (5)	−0.0057 (6)	−0.0036 (5)
N3	0.0436 (7)	0.0437 (7)	0.0629 (8)	0.0046 (5)	−0.0029 (6)	0.0008 (6)
C10	0.0468 (7)	0.0492 (8)	0.0436 (7)	−0.0013 (6)	0.0067 (6)	−0.0002 (6)
N2	0.0489 (7)	0.0399 (7)	0.0698 (8)	0.0037 (5)	0.0048 (6)	0.0019 (6)
N1	0.0412 (7)	0.0505 (7)	0.0642 (8)	0.0012 (5)	0.0078 (6)	−0.0001 (6)
C11	0.0525 (9)	0.0649 (10)	0.0526 (8)	0.0017 (7)	−0.0011 (7)	0.0027 (7)
C15	0.0492 (8)	0.0502 (8)	0.0494 (8)	0.0003 (6)	0.0094 (6)	−0.0015 (6)
C8	0.0482 (8)	0.0338 (7)	0.0658 (9)	−0.0054 (6)	0.0051 (7)	−0.0012 (6)
C9	0.0454 (7)	0.0457 (8)	0.0501 (8)	0.0033 (6)	0.0115 (6)	0.0084 (6)
C7	0.0483 (8)	0.0532 (9)	0.0735 (10)	−0.0073 (7)	−0.0024 (7)	0.0029 (8)
C3	0.0510 (9)	0.0697 (11)	0.0583 (9)	0.0021 (7)	0.0147 (7)	0.0045 (8)
C4	0.0410 (7)	0.0540 (8)	0.0467 (7)	−0.0004 (6)	−0.0017 (6)	0.0005 (6)
C6	0.0588 (9)	0.0623 (10)	0.0625 (10)	0.0014 (8)	0.0137 (8)	0.0098 (8)
C2	0.0593 (10)	0.0709 (11)	0.0726 (11)	0.0182 (9)	0.0159 (8)	−0.0026 (9)
C16	0.0758 (12)	0.0563 (10)	0.0791 (12)	0.0208 (9)	0.0047 (9)	0.0049 (9)
C12	0.0558 (9)	0.0864 (14)	0.0574 (9)	−0.0123 (9)	−0.0032 (7)	−0.0049 (9)
C5	0.0469 (8)	0.0582 (9)	0.0607 (9)	0.0055 (7)	0.0130 (7)	0.0011 (7)
C14	0.0698 (10)	0.0495 (9)	0.0685 (10)	−0.0057 (8)	0.0164 (8)	−0.0070 (7)
C1	0.0648 (10)	0.0535 (9)	0.0629 (10)	0.0099 (8)	0.0025 (8)	0.0056 (7)
C13	0.0704 (11)	0.0718 (12)	0.0676 (11)	−0.0228 (9)	0.0082 (9)	−0.0141 (9)

Geometric parameters (Å, °)

S1—C9	1.6818 (17)	C7—H7A	0.9700
Cl1—C1	1.7402 (18)	C7—H7B	0.9700
O1—C8	1.216 (2)	C3—C2	1.381 (3)
O2—C15	1.371 (2)	C3—C4	1.385 (2)
O2—C16	1.425 (2)	C3—H3	0.9300

N3—C9	1.3325 (19)	C4—C5	1.390 (2)
N3—C10	1.4112 (19)	C6—C1	1.372 (2)
N3—H3N	0.813 (18)	C6—C5	1.379 (2)
C10—C11	1.385 (2)	C6—H6	0.9300
C10—C15	1.404 (2)	C2—C1	1.376 (3)
N2—C9	1.356 (2)	C2—H2	0.9300
N2—N1	1.3904 (19)	C16—H16A	0.9600
N2—H2N	0.865 (19)	C16—H16B	0.9600
N1—C8	1.358 (2)	C16—H16C	0.9600
N1—H1N	0.864 (18)	C12—C13	1.362 (3)
C11—C12	1.382 (3)	C12—H12	0.9300
C11—H11	0.9300	C5—H5	0.9300
C15—C14	1.376 (2)	C14—C13	1.385 (3)
C8—C7	1.507 (2)	C14—H14	0.9300
C7—C4	1.514 (2)	C13—H13	0.9300

C15—O2—C16	118.35 (13)	C2—C3—H3	119.5
C9—N3—C10	133.17 (14)	C4—C3—H3	119.5
C9—N3—H3N	114.5 (12)	C3—C4—C5	118.21 (15)
C10—N3—H3N	112.4 (12)	C3—C4—C7	119.99 (14)
C11—C10—C15	119.03 (15)	C5—C4—C7	121.68 (14)
C11—C10—N3	126.56 (15)	C1—C6—C5	119.34 (16)
C15—C10—N3	114.39 (13)	C1—C6—H6	120.3
C9—N2—N1	121.53 (13)	C5—C6—H6	120.3
C9—N2—H2N	120.1 (12)	C1—C2—C3	119.44 (16)
N1—N2—H2N	113.9 (12)	C1—C2—H2	120.3
C8—N1—N2	120.24 (13)	C3—C2—H2	120.3
C8—N1—H1N	121.5 (12)	O2—C16—H16A	109.5
N2—N1—H1N	114.1 (12)	O2—C16—H16B	109.5
C12—C11—C10	119.65 (17)	H16A—C16—H16B	109.5
C12—C11—H11	120.2	O2—C16—H16C	109.5
C10—C11—H11	120.2	H16A—C16—H16C	109.5
O2—C15—C14	125.36 (15)	H16B—C16—H16C	109.5
O2—C15—C10	114.23 (13)	C13—C12—C11	121.00 (17)
C14—C15—C10	120.41 (15)	C13—C12—H12	119.5
O1—C8—N1	121.95 (15)	C11—C12—H12	119.5
O1—C8—C7	123.79 (15)	C6—C5—C4	121.14 (15)
N1—C8—C7	114.24 (14)	C6—C5—H5	119.4
N3—C9—N2	115.13 (13)	C4—C5—H5	119.4
N3—C9—S1	127.65 (12)	C15—C14—C13	119.51 (17)
N2—C9—S1	117.18 (12)	C15—C14—H14	120.2
C8—C7—C4	113.66 (12)	C13—C14—H14	120.2
C8—C7—H7A	108.8	C6—C1—C2	120.84 (17)
C4—C7—H7A	108.8	C6—C1—Cl1	119.29 (14)
C8—C7—H7B	108.8	C2—C1—Cl1	119.85 (14)
C4—C7—H7B	108.8	C12—C13—C14	120.32 (17)
H7A—C7—H7B	107.7	C12—C13—H13	119.8
C2—C3—C4	121.00 (16)	C14—C13—H13	119.8

C9—N3—C10—C11	−5.4 (3)	N1—C8—C7—C4	104.59 (16)

C9—N3—C10—C15	175.82 (15)	C2—C3—C4—C5	1.5 (2)
C9—N2—N1—C8	89.78 (18)	C2—C3—C4—C7	−174.54 (16)
C15—C10—C11—C12	1.1 (2)	C8—C7—C4—C3	−131.06 (16)
N3—C10—C11—C12	−177.59 (15)	C8—C7—C4—C5	53.0 (2)
C16—O2—C15—C14	1.1 (2)	C4—C3—C2—C1	−1.1 (3)
C16—O2—C15—C10	−178.85 (14)	C10—C11—C12—C13	1.2 (3)
C11—C10—C15—O2	177.07 (13)	C1—C6—C5—C4	−0.6 (3)
N3—C10—C15—O2	−4.04 (19)	C3—C4—C5—C6	−0.7 (2)
C11—C10—C15—C14	−2.8 (2)	C7—C4—C5—C6	175.30 (15)
N3—C10—C15—C14	176.04 (14)	O2—C15—C14—C13	−177.68 (15)
N2—N1—C8—O1	−12.7 (2)	C10—C15—C14—C13	2.2 (2)
N2—N1—C8—C7	168.94 (13)	C5—C6—C1—C2	1.1 (3)
C10—N3—C9—N2	176.74 (15)	C5—C6—C1—Cl1	−177.63 (14)
C10—N3—C9—S1	−1.1 (3)	C3—C2—C1—C6	−0.3 (3)
N1—N2—C9—N3	9.1 (2)	C3—C2—C1—Cl1	178.43 (14)
N1—N2—C9—S1	−172.85 (11)	C11—C12—C13—C14	−1.8 (3)
O1—C8—C7—C4	−73.8 (2)	C15—C14—C13—C12	0.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···S1[i]	0.862 (18)	2.435 (18)	3.269 (3)	163.1 (16)
N2—H2N···O1[ii]	0.865 (19)	2.159 (18)	2.941 (3)	150.1 (16)
N3—H3N···O2	0.810 (18)	2.099 (17)	2.551 (3)	115.2 (15)
N3—H3N···N1	0.810 (18)	2.229 (17)	2.656 (3)	113.3 (14)
C11—H11···S1	0.93	2.61	3.261 (3)	128

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

Fig. 1

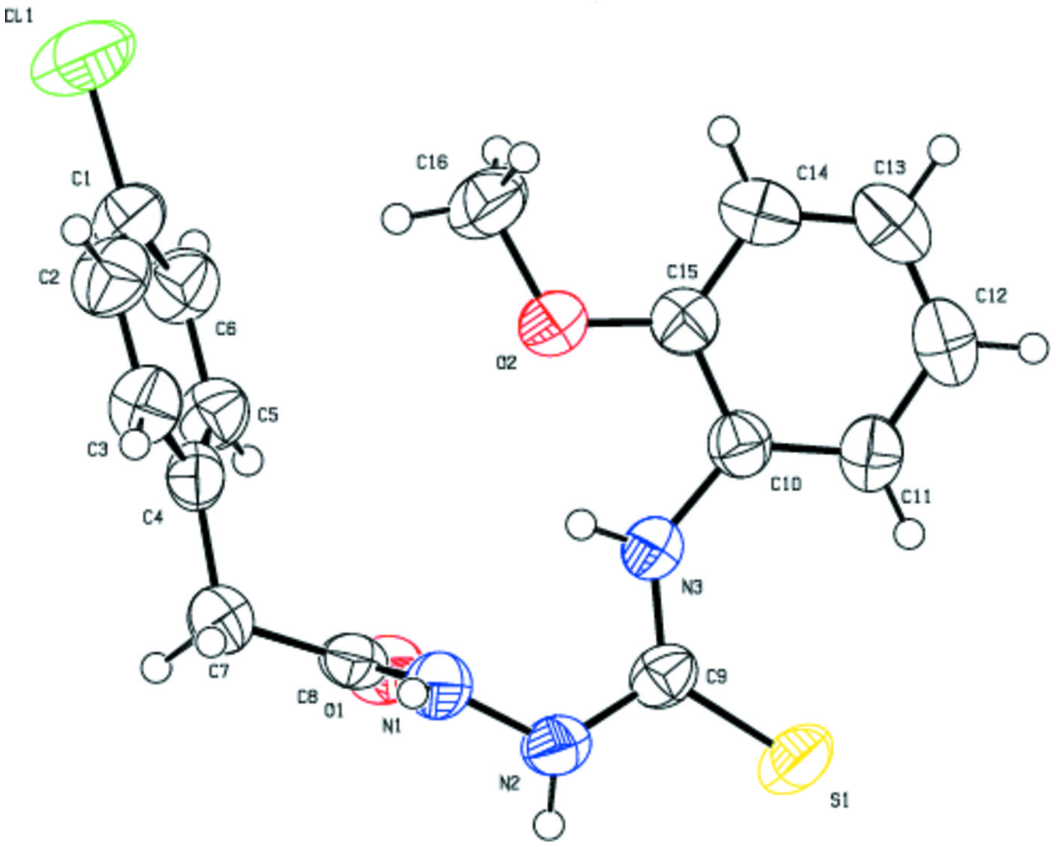
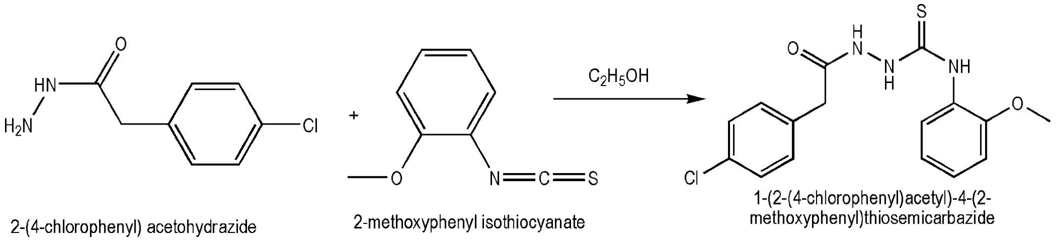

CL1
C1
C2
C6
C5
C3
C4
C7
C8
O1
N1
N2
C9
S1
N3
C10
C11
C12
C13
C14
C15
O2
C16

Fig. 2


O
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H_2N
Cl
+
O
N=C=S
C_2H_5OH
S
H
O
N
N
H
NH
O
Cl
2-(4-chlorophenyl) acetohydrazide
2-methoxyphenyl isothiocyanate
1-(2-(4-chlorophenyl)acetyl)-4-(2-methoxyphenyl)thiosemicarbazide