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1-(3,5-Dimethoxybenzoyl)-4-(2-methoxyphenyl)thiosemicarbazide

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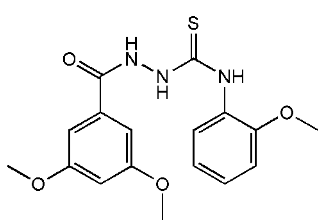
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 18.9.

The title compound, $C_{17}H_{19}N_3O_4S$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. 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, and intermolecular N—H···O, N—H···S, C—H···S and C—H···O hydrogen-bond interactions.

Related literature

For general background see: Allen *et al.* (1987); Shen *et al.* (1998); Mao *et al.* (1999); Antholine & Taketa (1982); for literature on a related structure see: Ji *et al.* (2002).



Experimental

Crystal data

$C_{17}H_{19}N_3O_4S$
M_r = 361.42
Monoclinic, $P2_1/c$
a = 15.371 (6) Å
b = 14.775 (6) Å
c = 7.904 (3) Å
β = 102.835 (6)°
V = 1750.2 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 293 (2) K
0.46 × 0.26 × 0.20 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.904, T_{max} = 0.960
13047 measured reflections
4293 independent reflections
3033 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.127
S = 1.10
4293 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1	0.86	2.28	2.595 (2)	102
N1—H1B···N3	0.86	2.20	2.645 (2)	112
C1—H1A···S1	0.93	2.78	3.292 (2)	116
N2—H2B···O3^{i}	0.86	2.29	3.088 (2)	155
N3—H3B···S1^{ii}	0.86	2.61	3.377 (2)	149
C4—H4A···S1^{iii}	0.93	2.77	3.618 (3)	152
C9—H9A···O4^{iv}	0.93	2.47	3.386 (2)	170

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{5}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{5}{2}$.

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*, *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: RZ2142).

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supplementary materials

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1-(3,5-Dimethoxybenzoyl)-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 (N-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 (Fig. 1), the bond lengths and angles are in normal ranges (Allen *et al.*, 1987; Ji *et al.*, 2002). Selected bond distances and angles within the thiosemicarbazide group are quoted in Table 1. The thiosemicarbazide group is approximately planar (maximum displacement 0.133 (2) Å for atom N2) and forms dihedral angles of 33.03 (6) and 45.48 (5)° with the benzene rings. The dihedral angle between the benzene rings is 56.29 (6)°. The molecular structure is stabilized by intramolecular N—H···O, N—H···N and C—H···S hydrogen bonds (Table 2). Intermolecular N—H···O, N—H···S, C—H···S, C—H···O hydrogen interactions link the molecules into an extended three-dimensional network.

Experimental

The title compound was prepared by the reaction of 3,5-dimethoxy bezohydrazide (3.92 g, 20 mmol) and 2-methoxyphenyl isothiocyanate (3.3 g, 20 mmol). Single crystals suitable for X-ray measurements were obtained by slow evaporation of an ethanol/water solution (60: 40 *v*/*v*) at room temperature (yield: 80%; m.p. 435–437 K).

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93–0.96 Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.2 U_{eq}(C) for methyl groups.

Figures

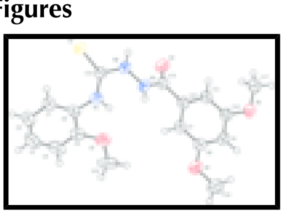

Fig. 1. The molecular structure of the title compound with 50% probability displacement ellipsoids.

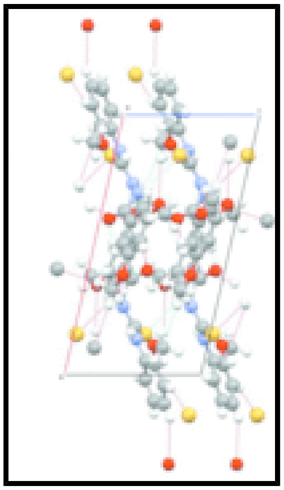

Fig. 2. A packing diagram of the title compound, viewed down the *b* axis. Intra- and inter-molecular hydrogen bonds are shown as dotted lines.

1-(3,5-Dimethoxybenzoyl)-4-(2-methoxyphenyl)thiosemicarbazide

Crystal data

$C_{17}H_{19}N_3O_4S$	$F_{000} = 760$
$M_r = 361.42$	$D_x = 1.371$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 435(2) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 15.371$ (6) Å	Cell parameters from 1520 reflections
$b = 14.775$ (6) Å	$\theta = 2.7–24.9$°
$c = 7.904$ (3) Å	$\mu = 0.21$ mm^{-1}
$\beta = 102.835$ (6)°	$T = 293$ (2) K
$V = 1750.2$ (12) Å^3	Block, colourless
$Z = 4$	0.46 × 0.26 × 0.20 mm

Data collection

Bruker APEXII diffractometer	4293 independent reflections
Radiation source: rotating-anode generator	3033 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 293(2)$ K	$\theta_{max} = 28.7$°
φ and ω scans	$\theta_{min} = 1.9$°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -20 \rightarrow 20$
$T_{min} = 0.904$, $T_{max} = 0.960$	$k = -19 \rightarrow 19$
13047 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.127$

$S = 1.10$

4293 reflections

227 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0179P)^2 + 0.3525P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.26$ e Å^{-3}

$\Delta\rho_{min} = -0.32$ e Å^{-3}

Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0132 (17)

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	0.15553 (3)	0.37817 (3)	0.97813 (7)	0.04962 (16)
O1	0.11298 (9)	0.03948 (9)	0.8952 (2)	0.0637 (4)
O2	0.34541 (9)	−0.14431 (9)	1.4113 (2)	0.0639 (4)
O3	0.61762 (8)	−0.06156 (8)	1.2608 (2)	0.0558 (4)
O4	0.40068 (9)	0.20133 (9)	1.0677 (2)	0.0607 (4)
N1	0.13745 (9)	0.19931 (9)	1.0337 (2)	0.0487 (4)
H1B	0.1633	0.1526	1.0874	0.058*
N2	0.26333 (9)	0.26783 (9)	1.1842 (2)	0.0487 (4)
H2B	0.2910	0.3156	1.2297	0.058*
N3	0.29958 (9)	0.18262 (9)	1.2322 (2)	0.0470 (4)
H3B	0.2777	0.1484	1.3004	0.056*
C1	−0.01826 (12)	0.24193 (14)	0.9010 (3)	0.0602 (5)
H1A	−0.0109	0.2990	0.9519	0.072*
C2	−0.10062 (14)	0.21636 (17)	0.7993 (4)	0.0757 (7)
H2A	−0.1484	0.2565	0.7820	0.091*
C3	−0.11147 (16)	0.13246 (18)	0.7247 (4)	0.0800 (7)
H3A	−0.1665	0.1163	0.6556	0.096*
C4	−0.04169 (15)	0.07148 (16)	0.7508 (3)	0.0713 (6)
H4A	−0.0498	0.0145	0.6997	0.086*
C5	0.04030 (12)	0.09527 (13)	0.8531 (3)	0.0527 (5)
C6	0.05241 (11)	0.18213 (12)	0.9261 (2)	0.0468 (4)

C7	0.35912 (11)	−0.00066 (11)	1.2957 (2)	0.0458 (4)
H7A	0.3014	0.0118	1.3069	0.055*
C8	0.39811 (11)	−0.08474 (11)	1.3456 (2)	0.0458 (4)
C9	0.48417 (11)	−0.10345 (11)	1.3312 (2)	0.0452 (4)
H9A	0.5097	−0.1595	1.3649	0.054*
C10	0.53229 (10)	−0.03741 (11)	1.2657 (2)	0.0423 (4)
C11	0.49472 (10)	0.04567 (11)	1.2121 (2)	0.0425 (4)
H11A	0.5267	0.0888	1.1654	0.051*
C12	0.40784 (10)	0.06380 (10)	1.2293 (2)	0.0401 (4)
C13	0.18429 (10)	0.27610 (10)	1.0656 (2)	0.0398 (4)
C14	0.37060 (10)	0.15494 (11)	1.1684 (2)	0.0417 (4)
C15	0.10394 (18)	−0.05097 (14)	0.8277 (4)	0.0806 (7)
H15A	0.1593	−0.0827	0.8656	0.121*
H15B	0.0887	−0.0489	0.7032	0.121*
H15C	0.0577	−0.0818	0.8690	0.121*
C16	0.38136 (16)	−0.23039 (14)	1.4676 (3)	0.0704 (6)
H16A	0.3376	−0.2652	1.5088	0.106*
H16B	0.4333	−0.2228	1.5597	0.106*
H16C	0.3975	−0.2614	1.3724	0.106*
C17	0.67315 (12)	0.00578 (14)	1.2084 (3)	0.0586 (5)
H17A	0.7308	−0.0196	1.2098	0.088*
H17B	0.6796	0.0561	1.2869	0.088*
H17C	0.6463	0.0260	1.0932	0.088*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0436 (3)	0.0354 (2)	0.0669 (3)	0.00322 (17)	0.0061 (2)	0.00655 (19)
O1	0.0637 (9)	0.0389 (7)	0.0914 (12)	−0.0079 (6)	0.0234 (8)	−0.0095 (7)
O2	0.0528 (7)	0.0463 (7)	0.0977 (12)	0.0047 (6)	0.0273 (8)	0.0188 (7)
O3	0.0380 (6)	0.0455 (7)	0.0859 (10)	0.0085 (5)	0.0177 (6)	0.0024 (6)
O4	0.0609 (8)	0.0430 (7)	0.0833 (11)	0.0051 (6)	0.0272 (8)	0.0127 (7)
N1	0.0411 (8)	0.0340 (7)	0.0659 (11)	−0.0021 (6)	0.0011 (7)	0.0039 (6)
N2	0.0387 (7)	0.0315 (7)	0.0704 (11)	0.0051 (5)	0.0000 (7)	−0.0020 (6)
N3	0.0408 (7)	0.0357 (7)	0.0642 (10)	0.0085 (6)	0.0108 (7)	0.0078 (6)
C1	0.0420 (9)	0.0507 (11)	0.0848 (16)	−0.0022 (8)	0.0077 (10)	0.0057 (10)
C2	0.0420 (11)	0.0751 (15)	0.103 (2)	−0.0069 (10)	0.0004 (12)	0.0210 (13)
C3	0.0519 (12)	0.0922 (18)	0.0861 (18)	−0.0259 (12)	−0.0058 (12)	0.0148 (14)
C4	0.0697 (14)	0.0672 (14)	0.0735 (16)	−0.0310 (12)	0.0083 (12)	−0.0093 (11)
C5	0.0516 (10)	0.0476 (10)	0.0602 (13)	−0.0125 (8)	0.0154 (9)	−0.0007 (8)
C6	0.0393 (8)	0.0426 (9)	0.0568 (11)	−0.0077 (7)	0.0070 (8)	0.0033 (8)
C7	0.0355 (8)	0.0433 (9)	0.0578 (12)	0.0042 (7)	0.0086 (8)	0.0012 (8)
C8	0.0425 (9)	0.0393 (9)	0.0546 (11)	0.0003 (7)	0.0089 (8)	0.0023 (7)
C9	0.0425 (9)	0.0375 (8)	0.0540 (11)	0.0072 (7)	0.0074 (8)	0.0005 (7)
C10	0.0353 (8)	0.0403 (9)	0.0500 (10)	0.0048 (6)	0.0068 (7)	−0.0047 (7)
C11	0.0378 (8)	0.0382 (8)	0.0509 (11)	0.0002 (6)	0.0085 (7)	−0.0016 (7)
C12	0.0360 (8)	0.0365 (8)	0.0453 (10)	0.0030 (6)	0.0038 (7)	−0.0018 (7)
C13	0.0349 (8)	0.0339 (8)	0.0510 (10)	0.0031 (6)	0.0105 (7)	−0.0031 (7)

C14	0.0361 (8)	0.0359 (8)	0.0499 (10)	0.0015 (6)	0.0029 (7)	−0.0013 (7)
C15	0.0966 (17)	0.0411 (11)	0.116 (2)	−0.0178 (11)	0.0493 (16)	−0.0168 (12)
C16	0.0807 (15)	0.0460 (11)	0.0895 (18)	0.0042 (10)	0.0297 (13)	0.0168 (11)
C17	0.0419 (9)	0.0575 (11)	0.0784 (15)	0.0035 (8)	0.0177 (10)	0.0062 (10)

Geometric parameters (Å, °)

S1—C13	1.6769 (17)	C4—C5	1.383 (3)
O1—C5	1.369 (2)	C4—H4A	0.9300
O1—C15	1.434 (2)	C5—C6	1.403 (3)
O2—C8	1.374 (2)	C7—C12	1.385 (2)
O2—C16	1.418 (2)	C7—C8	1.398 (2)
O3—C10	1.3681 (19)	C7—H7A	0.9300
O3—C17	1.431 (2)	C8—C9	1.381 (2)
O4—C14	1.217 (2)	C9—C10	1.392 (2)
N1—C13	1.337 (2)	C9—H9A	0.9300
N1—C6	1.415 (2)	C10—C11	1.382 (2)
N1—H1B	0.8600	C11—C12	1.398 (2)
N2—C13	1.365 (2)	C11—H11A	0.9300
N2—N3	1.3949 (19)	C12—C14	1.500 (2)
N2—H2B	0.8600	C15—H15A	0.9600
N3—C14	1.363 (2)	C15—H15B	0.9600
N3—H3B	0.8600	C15—H15C	0.9600
C1—C6	1.380 (3)	C16—H16A	0.9600
C1—C2	1.393 (3)	C16—H16B	0.9600
C1—H1A	0.9300	C16—H16C	0.9600
C2—C3	1.367 (4)	C17—H17A	0.9600
C2—H2A	0.9300	C17—H17B	0.9600
C3—C4	1.381 (3)	C17—H17C	0.9600
C3—H3A	0.9300		
C5—O1—C15	117.66 (17)	C8—C9—C10	119.24 (15)
C8—O2—C16	118.15 (15)	C8—C9—H9A	120.4
C10—O3—C17	117.66 (14)	C10—C9—H9A	120.4
C13—N1—C6	130.71 (15)	O3—C10—C11	124.11 (16)
C13—N1—H1B	114.6	O3—C10—C9	114.81 (14)
C6—N1—H1B	114.6	C11—C10—C9	121.07 (15)
C13—N2—N3	120.56 (14)	C10—C11—C12	118.91 (16)
C13—N2—H2B	119.7	C10—C11—H11A	120.5
N3—N2—H2B	119.7	C12—C11—H11A	120.5
C14—N3—N2	118.32 (15)	C7—C12—C11	120.92 (15)
C14—N3—H3B	120.8	C7—C12—C14	122.62 (15)
N2—N3—H3B	120.8	C11—C12—C14	116.45 (15)
C6—C1—C2	119.7 (2)	N1—C13—N2	114.41 (14)
C6—C1—H1A	120.2	N1—C13—S1	127.13 (13)
C2—C1—H1A	120.2	N2—C13—S1	118.45 (12)
C3—C2—C1	120.2 (2)	O4—C14—N3	121.67 (15)
C3—C2—H2A	119.9	O4—C14—C12	122.85 (16)
C1—C2—H2A	119.9	N3—C14—C12	115.48 (15)
C2—C3—C4	120.7 (2)	O1—C15—H15A	109.5

C2—C3—H3A	119.6	O1—C15—H15B	109.5
C4—C3—H3A	119.6	H15A—C15—H15B	109.5
C3—C4—C5	119.9 (2)	O1—C15—H15C	109.5
C3—C4—H4A	120.1	H15A—C15—H15C	109.5
C5—C4—H4A	120.1	H15B—C15—H15C	109.5
O1—C5—C4	125.29 (19)	O2—C16—H16A	109.5
O1—C5—C6	115.12 (16)	O2—C16—H16B	109.5
C4—C5—C6	119.57 (19)	H16A—C16—H16B	109.5
C1—C6—C5	119.90 (17)	O2—C16—H16C	109.5
C1—C6—N1	124.34 (17)	H16A—C16—H16C	109.5
C5—C6—N1	115.58 (16)	H16B—C16—H16C	109.5
C12—C7—C8	118.96 (15)	O3—C17—H17A	109.5
C12—C7—H7A	120.5	O3—C17—H17B	109.5
C8—C7—H7A	120.5	H17A—C17—H17B	109.5
O2—C8—C9	123.99 (16)	O3—C17—H17C	109.5
O2—C8—C7	115.11 (15)	H17A—C17—H17C	109.5
C9—C8—C7	120.89 (16)	H17B—C17—H17C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···O1	0.86	2.28	2.595 (2)	102
N1—H1B···N3	0.86	2.20	2.645 (2)	112
C1—H1A···S1	0.93	2.78	3.292 (2)	116
N2—H2B···O3^{i}	0.86	2.29	3.088 (2)	155
N3—H3B···S1^{ii}	0.86	2.61	3.377 (2)	149
C4—H4A···S1^{iii}	0.93	2.77	3.618 (3)	152
C9—H9A···O4^{iv}	0.93	2.47	3.386 (2)	170

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+5/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*, *y*−1/2, −*z*+3/2; (iv) −*x*+1, *y*−1/2, −*z*+5/2.

Fig. 1

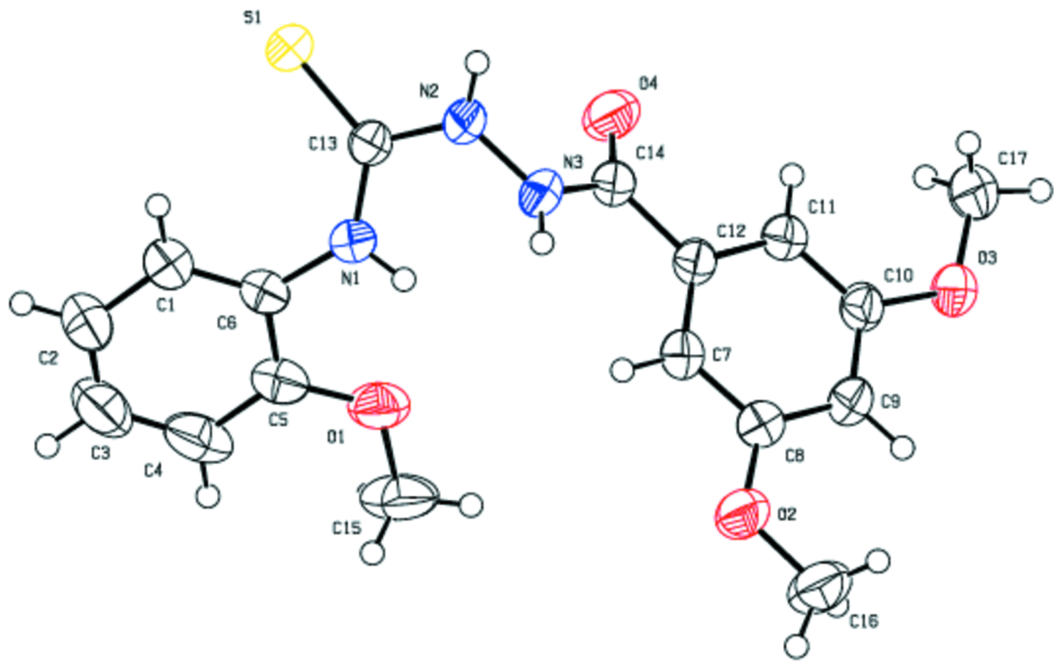
S1
N2
O4
C13
N3
C14
C17
C11
C12
N1
C1
C6
C10
O3
C2
C7
C5
C9
O1
C3
C4
C8
O2
C15
C16

Fig. 2