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2,5-Dimethoxybenzaldehyde thiosemicarbazone

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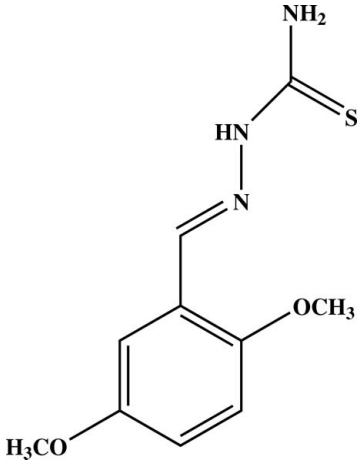
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 22.2.

In the title molecule, $C_{10}H_{13}N_3O_2S$, the dihedral angle between benzene and –N—C(=S)—N—N=C– planes is 9.20 (6)°. The two methoxy groups are coplanar with the benzene ring [C—O—C—C torsion angles of −2.31 (18) and −6.45 (17)°]. In the crystal structure, molecules are linked by intermolecular N—H···S, N—H···O and C—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For the biomedical properties of thiosemicarbazones, see: Beraldo & Gambino (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_{13}N_3O_2S$
M_r = 239.29
Orthorhombic, *Pbca*
a = 11.0713 (1) Å
b = 13.0603 (2) Å
c = 15.7808 (2) Å
V = 2281.82 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 100.0 (1) K
0.34 × 0.28 × 0.22 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.912, T_{max} = 0.943
18486 measured reflections
3486 independent reflections
2834 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.06
3486 reflections
157 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N2*···S1^{i}	0.84 (4)	2.718 (18)	3.5375 (12)	166 (2)
N3—H2*N3*···S1^{ii}	0.86 (1)	2.811 (13)	3.5047 (12)	139 (1)
N3—H1*N3*···O2^{iii}	0.86 (1)	2.145 (11)	2.9617 (15)	159 (2)
C3—H3*A*···O1^{iv}	0.93	2.51	3.3027 (16)	143

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2* and *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2701).

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supporting information

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2,5-Dimethoxybenzaldehyde thiosemicarbazone

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S1. Comment

Thiosemicarbazones are of great interest because of their profound biomedical properties (Beraldo *et al.*, 2004). Flexibility and bioactivity of these compounds arise due to the presence of amino group (–N═CH–) in addition to thio-amino moieties present in the skeleton of the molecule. We have synthesized the title compound and its crystal structure is reported here.

The bond lengths in the title molecule (Fig.1) are found to have normal values (Allen *et al.*, 1987). The two methoxy groups are coplanar with the benzene ring, with C9—O1—C1—C2 and C10—O2—C4—C3 torsion angles of -2.31 (18) and -6.45 (17)°, respectively. The dihedral angle between the C1—C6 and S1/N1—N3/C7/C8 planes is 9.20 (6)°.

In the crystal packing, the molecules are linked together by intermolecular N—H···S, N—H···O and C—H···O hydrogen bonds (Table 1) to form a three-dimensional network (Fig.2).

S2. Experimental

The title compound was synthesized by refluxing 2,5-dimethoxy benzaldehyde (0.075 mol) and thiosemicarbazone (0.05 mol) in methanol (100 ml) for 2 h. The solution was then allowed to cool, poured into a beaker containing water and stirred for 30 min. The product was separated by filtration and the crude sample obtained was recrystallized twice from hot methanol.

S3. Refinement

N-bound H atoms were located in a difference map and were refined with an N—H distance restraint of 0.86 (1) Å. C-bound H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and refined using a riding model with U_{iso}(H) = 1.2–1.5U_{eq}(C).

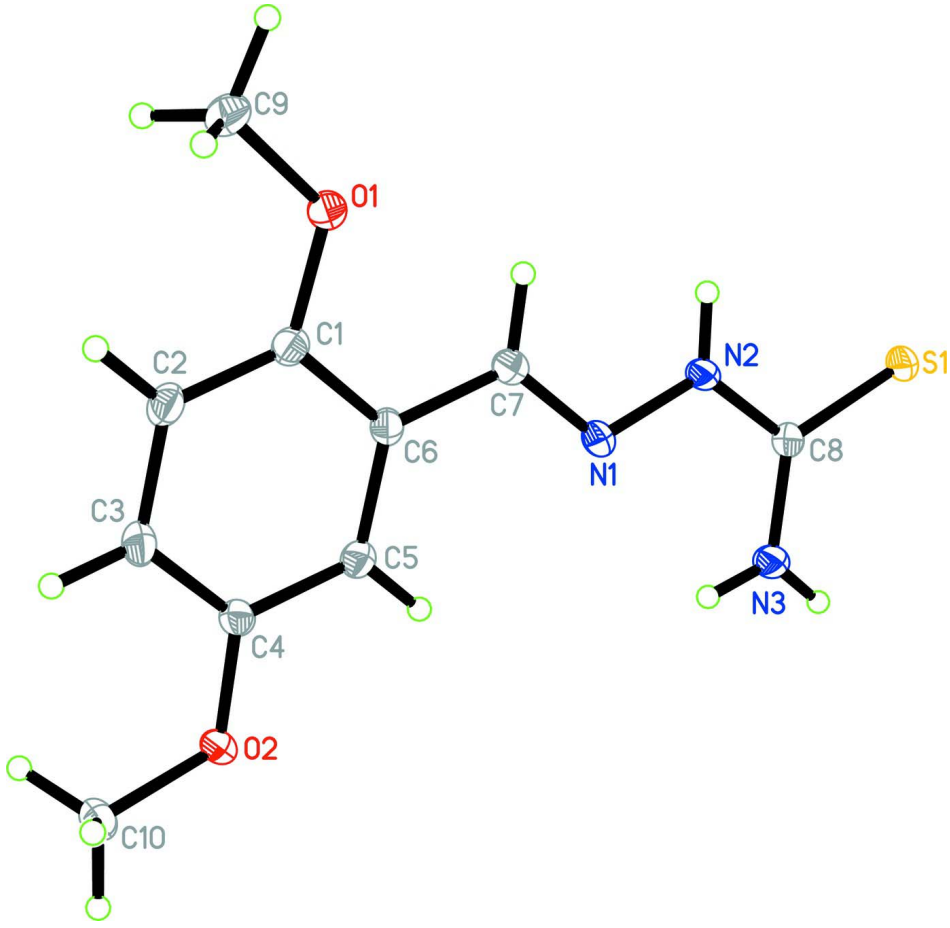

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

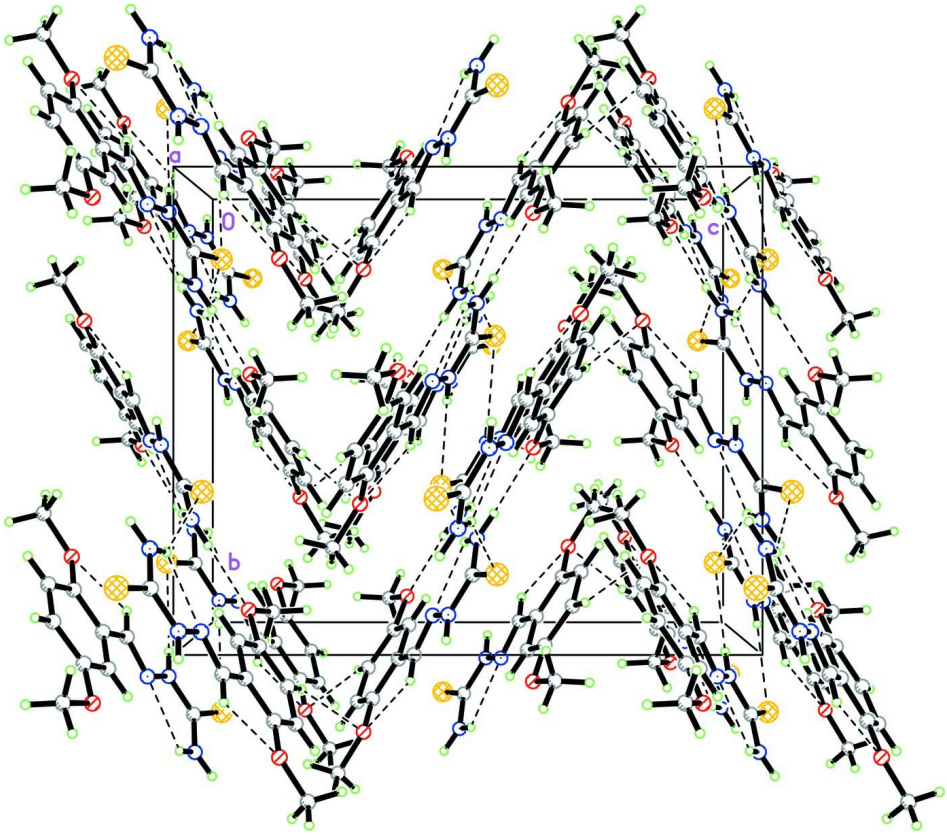

Figure 2

The crystal packing of the title compound, viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

2,5-Dimethoxybenzaldehyde thiosemicarbazone

Crystal data

$C_{10}H_{13}N_3O_2S$
$M_r = 239.29$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 11.0713$ (1) Å
$b = 13.0603$ (2) Å
$c = 15.7808$ (2) Å
$V = 2281.82$ (5) Å^3
$Z = 8$
$F(000) = 1008$
$D_x = 1.393$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4233 reflections
$\theta = 2.6$–$30.0°$
$\mu = 0.27$ mm^{-1}
$T = 100$ K
Block, colourless
0.34 × 0.28 × 0.22 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.912$, $T_{max} = 0.943$
18486 measured reflections
3486 independent reflections
2834 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 30.5°$, $\theta_{min} = 2.6°$
$h = -15 \rightarrow 11$
$k = -14 \rightarrow 18$
$l = -22 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.097$
$S = 1.06$
3486 reflections
157 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
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
$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 0.8407P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.01083 (3)	1.16558 (3)	0.45083 (2)	0.01697 (9)
O1	0.35907 (8)	0.80559 (7)	0.68704 (6)	0.0191 (2)
O2	0.72695 (8)	1.07776 (7)	0.61508 (6)	0.0165 (2)
N1	0.28066 (10)	1.06262 (9)	0.56220 (6)	0.0150 (2)
N2	0.16335 (10)	1.07175 (9)	0.53426 (7)	0.0157 (2)
N3	0.21269 (11)	1.23005 (9)	0.48405 (7)	0.0178 (2)
C1	0.45590 (12)	0.86889 (10)	0.67348 (7)	0.0150 (2)
C2	0.57116 (12)	0.85165 (10)	0.70424 (8)	0.0174 (3)
H2A	0.5868	0.7939	0.7370	0.021*
C3	0.66395 (12)	0.92047 (10)	0.68638 (8)	0.0175 (3)
H3A	0.7414	0.9085	0.7070	0.021*
C4	0.64053 (11)	1.00689 (10)	0.63780 (7)	0.0144 (2)
C5	0.52450 (12)	1.02595 (10)	0.60844 (7)	0.0145 (2)
H5A	0.5091	1.0846	0.5768	0.017*
C6	0.43114 (11)	0.95785 (10)	0.62609 (7)	0.0138 (2)
C7	0.30819 (12)	0.97659 (10)	0.59612 (8)	0.0153 (2)
H7A	0.2497	0.9259	0.6017	0.018*
C8	0.13017 (11)	1.15736 (10)	0.49192 (7)	0.0142 (2)
C9	0.37938 (13)	0.71375 (11)	0.73357 (9)	0.0231 (3)
H9A	0.3049	0.6766	0.7387	0.035*
H9B	0.4092	0.7304	0.7890	0.035*
H9C	0.4377	0.6724	0.7044	0.035*
C10	0.84490 (12)	1.06643 (11)	0.65093 (8)	0.0184 (3)

H10A	0.8965	1.1200	0.6303	0.028*
H10B	0.8777	1.0012	0.6350	0.028*
H10C	0.8397	1.0705	0.7116	0.028*
H1N2	0.1156 (16)	1.0222 (14)	0.5376 (11)	0.024 (4)*
H2N3	0.2833 (10)	1.2200 (13)	0.5055 (10)	0.025 (4)*
H1N3	0.1967 (18)	1.2835 (10)	0.4545 (10)	0.036 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01117 (16)	0.01533 (17)	0.02441 (16)	0.00118 (11)	−0.00228 (12)	0.00302 (11)
O1	0.0150 (5)	0.0161 (5)	0.0261 (5)	0.0003 (4)	0.0023 (4)	0.0075 (4)
O2	0.0115 (4)	0.0155 (5)	0.0224 (4)	−0.0017 (3)	−0.0022 (3)	0.0021 (3)
N1	0.0109 (5)	0.0158 (5)	0.0184 (5)	0.0003 (4)	−0.0023 (4)	0.0008 (4)
N2	0.0107 (5)	0.0134 (5)	0.0231 (5)	−0.0015 (4)	−0.0029 (4)	0.0036 (4)
N3	0.0139 (5)	0.0143 (5)	0.0251 (5)	−0.0012 (4)	−0.0037 (4)	0.0035 (4)
C1	0.0154 (6)	0.0134 (6)	0.0161 (5)	0.0008 (5)	0.0028 (5)	0.0010 (4)
C2	0.0187 (7)	0.0140 (6)	0.0196 (5)	0.0031 (5)	−0.0001 (5)	0.0035 (4)
C3	0.0143 (6)	0.0176 (6)	0.0205 (6)	0.0035 (5)	−0.0029 (5)	0.0008 (5)
C4	0.0129 (6)	0.0141 (6)	0.0161 (5)	0.0002 (5)	0.0007 (4)	−0.0015 (4)
C5	0.0152 (6)	0.0131 (6)	0.0154 (5)	0.0010 (5)	−0.0009 (4)	0.0013 (4)
C6	0.0125 (6)	0.0137 (6)	0.0151 (5)	0.0016 (5)	0.0000 (4)	0.0004 (4)
C7	0.0130 (6)	0.0152 (6)	0.0176 (5)	−0.0009 (5)	0.0001 (4)	0.0013 (4)
C8	0.0132 (6)	0.0137 (6)	0.0156 (5)	0.0010 (4)	0.0009 (4)	−0.0008 (4)
C9	0.0223 (7)	0.0168 (7)	0.0301 (7)	0.0002 (5)	0.0039 (6)	0.0085 (5)
C10	0.0127 (6)	0.0206 (7)	0.0218 (6)	0.0005 (5)	−0.0025 (5)	−0.0017 (5)

Geometric parameters (Å, º)

S1—C8	1.6938 (13)	C2—H2A	0.93
O1—C1	1.3706 (16)	C3—C4	1.3888 (18)
O1—C9	1.4242 (16)	C3—H3A	0.93
O2—C4	1.3788 (15)	C4—C5	1.3881 (17)
O2—C10	1.4308 (15)	C5—C6	1.3917 (18)
N1—C7	1.2813 (16)	C5—H5A	0.93
N1—N2	1.3768 (15)	C6—C7	1.4617 (18)
N2—C8	1.3533 (16)	C7—H7A	0.93
N2—H1N2	0.838 (18)	C9—H9A	0.96
N3—C8	1.3235 (17)	C9—H9B	0.96
N3—H2N3	0.861 (9)	C9—H9C	0.96
N3—H1N3	0.857 (9)	C10—H10A	0.96
C1—C2	1.3837 (19)	C10—H10B	0.96
C1—C6	1.4087 (17)	C10—H10C	0.96
C2—C3	1.3938 (19)		
C1—O1—C9	117.71 (10)	C6—C5—H5A	119.8
C4—O2—C10	117.46 (10)	C5—C6—C1	119.26 (12)
C7—N1—N2	115.73 (11)	C5—C6—C7	121.33 (11)

C8—N2—N1	119.06 (11)	C1—C6—C7	119.41 (12)
C8—N2—H1N2	119.9 (12)	N1—C7—C6	120.23 (12)
N1—N2—H1N2	120.5 (12)	N1—C7—H7A	119.9
C8—N3—H2N3	118.7 (11)	C6—C7—H7A	119.9
C8—N3—H1N3	119.5 (14)	N3—C8—N2	116.85 (12)
H2N3—N3—H1N3	121.6 (17)	N3—C8—S1	123.70 (10)
O1—C1—C2	124.64 (11)	N2—C8—S1	119.44 (10)
O1—C1—C6	115.35 (11)	O1—C9—H9A	109.5
C2—C1—C6	120.00 (12)	O1—C9—H9B	109.5
C1—C2—C3	120.25 (12)	H9A—C9—H9B	109.5
C1—C2—H2A	119.9	O1—C9—H9C	109.5
C3—C2—H2A	119.9	H9A—C9—H9C	109.5
C4—C3—C2	119.87 (12)	H9B—C9—H9C	109.5
C4—C3—H3A	120.1	O2—C10—H10A	109.5
C2—C3—H3A	120.1	O2—C10—H10B	109.5
O2—C4—C5	115.78 (11)	H10A—C10—H10B	109.5
O2—C4—C3	124.03 (11)	O2—C10—H10C	109.5
C5—C4—C3	120.19 (12)	H10A—C10—H10C	109.5
C4—C5—C6	120.39 (12)	H10B—C10—H10C	109.5
C4—C5—H5A	119.8		

C7—N1—N2—C8	−174.96 (11)	C4—C5—C6—C1	−0.54 (18)
C9—O1—C1—C2	−2.31 (18)	C4—C5—C6—C7	179.74 (11)
C9—O1—C1—C6	179.11 (11)	O1—C1—C6—C5	−179.27 (11)
O1—C1—C2—C3	179.55 (12)	C2—C1—C6—C5	2.07 (18)
C6—C1—C2—C3	−1.93 (19)	O1—C1—C6—C7	0.45 (17)
C1—C2—C3—C4	0.25 (19)	C2—C1—C6—C7	−178.20 (11)
C10—O2—C4—C5	173.96 (11)	N2—N1—C7—C6	178.52 (10)
C10—O2—C4—C3	−6.45 (17)	C5—C6—C7—N1	−8.95 (18)
C2—C3—C4—O2	−178.28 (11)	C1—C6—C7—N1	171.33 (11)
C2—C3—C4—C5	1.30 (19)	N1—N2—C8—N3	−3.50 (17)
O2—C4—C5—C6	178.47 (11)	N1—N2—C8—S1	175.42 (9)
C3—C4—C5—C6	−1.14 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···S1^{i}	0.84 (4)	2.718 (18)	3.5375 (12)	166 (2)
N3—H2*N*3···S1^{ii}	0.86 (1)	2.81 (1)	3.5047 (12)	139 (1)
N3—H1*N*3···O2^{iii}	0.86 (1)	2.15 (1)	2.9617 (15)	159 (2)
C3—H3*A*···O1^{iv}	0.93	2.51	3.3027 (16)	143

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