

(E)-O-Ethyl N-(3-methoxyphenyl)thio-carbamate

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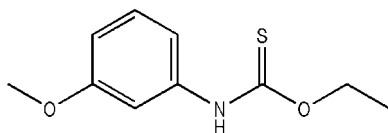
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.057; wR factor = 0.152; data-to-parameter ratio = 15.7.

The molecule of the title compound, $\text{C}_{10}\text{H}_{13}\text{NO}_2\text{S}$, is not planar and displays an *E* conformation about the (S)C—N bond. It is in the thione form. Intramolecular C—H \cdots O and intermolecular N—H \cdots S hydrogen bonds are present.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related literature, see: Ho *et al.* (2005, 2006).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{13}\text{NO}_2\text{S}$

$M_r = 211.27$

Monoclinic, $P2_1/n$

$a = 14.931$ (3) Å

$b = 4.866$ (1) Å

$c = 15.538$ (3) Å

$\beta = 107.75$ (3)°

$V = 1075.2$ (4) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.28$ mm⁻¹

$T = 294$ (2) K

$0.46 \times 0.26 \times 0.20$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.879$, $T_{\max} = 0.946$

2170 measured reflections
2089 independent reflections
1596 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.152$

$S = 1.14$

2089 reflections

133 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.38$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots S1 ⁱ	0.87 (3)	2.60 (3)	3.464 (2)	170.0 (3)
C5—H5 \cdots O1	0.93	2.18	2.772 (3)	120

Symmetry code: (i) $-x + 1, -y + 2, -z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; 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: HK2259).

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supplementary materials

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(*E*)-*O*-Ethyl *N*-(3-methoxyphenyl)thiocarbamate

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Comment

The title compound, (I), was investigated as a part of a study of related *O*-alkyl thiocarbamate molecules (Ho *et al.*, 2005) and their phosphine gold(I) complexes (Ho *et al.*, 2006).

In the molecule of the title compound, (I), (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angles between the planar A (O2/C6/C10) and B (S1/O1/N1/C1—C4) moieties and benzene ring C (C1—C6) are A/B = 8.72 (3)°, A/C = 3.12 (3)° and B/C = 11.47 (2)°. The C3—S1 [1.663 (2) Å] bond distance indicates that the molecule is in the thione form; the conformation about the central C—N bond is *E*.

The intramolecular C—H···O and intermolecular N—H···S hydrogen bonds (Table 1) may be effective in the stabilization of the crystal structure.

Experimental

Compound (I) was prepared by refluxing 3-methoxyphenyl isothiocyanate (1.65 g, 10 mmol) with absolute ethanol (50 ml) using a literature procedure (Ho *et al.*, 2005). The white precipitate, which was obtained upon concentration of the reaction solution, was dissolved in hot dichloromethane and layered with ethanol, which resulted in the formation of colorless crystals (yield: 1.32 g, 80%, m.p. 425–427 K).

Refinement

H atom of NH group was located in difference syntheses and refined isotropically [N—H = 0.87 (3) Å and $U_{\text{iso}}(\text{H}) = 0.059 (8) \text{ \AA}^2$]. The remaining H atoms were positioned geometrically, with C—H = 0.93, 0.96 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

Figures

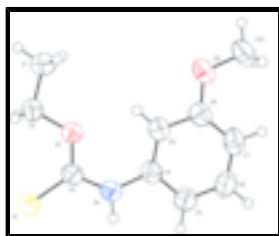


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.



Fig. 2. The synthetic route for the formation of the title compound.

(E)—O-Ethyl N-(3-methoxyphenyl)thiocarbamate

Crystal data

$C_{10}H_{13}NO_2S$	$F_{000} = 448$
$M_r = 211.27$	$D_x = 1.305 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Melting point: 425(2) K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation
$a = 14.931(3) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 4.866(1) \text{ \AA}$	Cell parameters from 1520 reflections
$c = 15.538(3) \text{ \AA}$	$\theta = 2.7\text{--}24.9^\circ$
$\beta = 107.75(3)^\circ$	$\mu = 0.28 \text{ mm}^{-1}$
$V = 1075.2(4) \text{ \AA}^3$	$T = 294(2) \text{ K}$
$Z = 4$	Block, colorless
	$0.46 \times 0.26 \times 0.20 \text{ mm}$

Data collection

Bruker APEX II diffractometer	2089 independent reflections
Radiation source: fine-focus sealed tube	1596 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.022$
$T = 294(2) \text{ K}$	$\theta_{\text{max}} = 26.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.7^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = 0 \rightarrow 18$
$T_{\text{min}} = 0.879$, $T_{\text{max}} = 0.946$	$k = 0 \rightarrow 5$
2170 measured reflections	$l = -19 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.057$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.152$	$w = 1/[\sigma^2(F_o^2) + (0.0832P)^2 + 0.1652P]$
$S = 1.14$	where $P = (F_o^2 + 2F_c^2)/3$
2089 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
133 parameters	$\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$
	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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6768 (2)	1.1857 (7)	0.39864 (18)	0.0726 (8)
H1A	0.6188	1.2469	0.4067	0.109*
H1B	0.7273	1.3002	0.4333	0.109*
H1C	0.6885	0.9988	0.4187	0.109*
C2	0.67037 (17)	1.2031 (6)	0.30106 (17)	0.0558 (6)
H2A	0.6543	1.3885	0.2787	0.067*
H2B	0.7299	1.1531	0.2923	0.067*
C3	0.56958 (16)	1.0016 (5)	0.16453 (15)	0.0472 (6)
C4	0.44867 (16)	0.6494 (5)	0.17244 (16)	0.0473 (6)
C5	0.46054 (18)	0.6384 (5)	0.26386 (17)	0.0557 (6)
H5	0.5033	0.7549	0.3032	0.067*
C6	0.40873 (18)	0.4539 (5)	0.29714 (18)	0.0548 (6)
C7	0.34422 (18)	0.2818 (6)	0.2405 (2)	0.0613 (7)
H7	0.3093	0.1585	0.2630	0.074*
C8	0.3328 (2)	0.2977 (6)	0.1490 (2)	0.0705 (8)
H8	0.2888	0.1843	0.1096	0.085*
C9	0.38430 (19)	0.4755 (6)	0.11432 (18)	0.0624 (7)
H9	0.3762	0.4795	0.0526	0.075*
C10	0.3885 (3)	0.2534 (7)	0.4304 (2)	0.0796 (9)
H10A	0.3212	0.2683	0.4096	0.119*
H10B	0.4106	0.2764	0.4948	0.119*
H10C	0.4068	0.0757	0.4149	0.119*
N1	0.49870 (15)	0.8261 (4)	0.13049 (15)	0.0520 (5)
O1	0.59723 (11)	1.0119 (4)	0.25404 (10)	0.0557 (5)
O2	0.42811 (17)	0.4596 (5)	0.38897 (14)	0.0848 (7)
S1	0.61763 (5)	1.18383 (16)	0.09943 (4)	0.0622 (3)
H1	0.4762 (19)	0.813 (6)	0.072 (2)	0.059 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0718 (17)	0.095 (2)	0.0482 (14)	-0.0156 (17)	0.0135 (13)	-0.0013 (15)

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C2	0.0533 (13)	0.0623 (16)	0.0503 (13)	-0.0124 (12)	0.0134 (11)	0.0039 (12)
C3	0.0488 (12)	0.0462 (13)	0.0456 (12)	0.0041 (11)	0.0126 (9)	0.0063 (10)
C4	0.0500 (12)	0.0399 (13)	0.0509 (12)	0.0040 (10)	0.0136 (10)	0.0069 (10)
C5	0.0599 (14)	0.0531 (15)	0.0545 (14)	-0.0074 (12)	0.0181 (11)	0.0019 (12)
C6	0.0584 (14)	0.0501 (14)	0.0593 (14)	0.0019 (12)	0.0230 (12)	0.0057 (12)
C7	0.0529 (14)	0.0526 (16)	0.0768 (18)	-0.0034 (12)	0.0175 (12)	0.0162 (14)
C8	0.0651 (17)	0.0644 (19)	0.0716 (18)	-0.0197 (14)	0.0052 (14)	0.0072 (15)
C9	0.0656 (15)	0.0607 (17)	0.0528 (14)	-0.0098 (13)	0.0059 (12)	0.0093 (13)
C10	0.100 (2)	0.078 (2)	0.0716 (19)	-0.0127 (18)	0.0423 (17)	0.0170 (16)
N1	0.0602 (12)	0.0513 (13)	0.0428 (11)	-0.0061 (10)	0.0134 (9)	0.0024 (9)
O1	0.0588 (10)	0.0622 (11)	0.0434 (9)	-0.0128 (8)	0.0116 (7)	0.0052 (8)
O2	0.1115 (16)	0.0883 (16)	0.0626 (12)	-0.0362 (14)	0.0383 (12)	0.0054 (11)
S1	0.0624 (4)	0.0757 (5)	0.0499 (4)	-0.0128 (3)	0.0190 (3)	0.0094 (3)

Geometric parameters (\AA , $^\circ$)

N1—H1	0.87 (3)	C5—C6	1.384 (3)
C1—C2	1.492 (4)	C5—H5	0.9300
C1—H1A	0.9600	C6—O2	1.367 (3)
C1—H1B	0.9600	C6—C7	1.374 (4)
C1—H1C	0.9600	C7—C8	1.381 (4)
C2—O1	1.451 (3)	C7—H7	0.9300
C2—H2A	0.9600	C8—C9	1.372 (4)
C2—H2B	0.9600	C8—H8	0.9300
C3—O1	1.326 (3)	C9—H9	0.9300
C3—N1	1.337 (3)	C10—O2	1.415 (3)
C3—S1	1.663 (2)	C10—H10A	0.9600
C4—C5	1.378 (3)	C10—H10B	0.9600
C4—C9	1.388 (4)	C10—H10C	0.9600
C4—N1	1.421 (3)		
O1—C2—C1	106.3 (2)	O2—C6—C5	114.3 (2)
O1—C3—N1	113.3 (2)	C7—C6—C5	121.2 (2)
O1—C3—S1	124.31 (18)	C6—C7—C8	117.9 (2)
N1—C3—S1	122.42 (18)	C9—C8—C7	122.1 (3)
C5—C4—C9	119.5 (2)	C8—C9—C4	119.3 (3)
C5—C4—N1	125.2 (2)	C3—N1—C4	131.9 (2)
C9—C4—N1	115.3 (2)	C6—N1—H1B	82.0
C4—C5—C6	120.0 (2)	C3—O1—C2	119.90 (18)
O2—C6—C7	124.4 (2)	C6—O2—C10	118.4 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots S1 ⁱ	0.87 (3)	2.60 (3)	3.464 (2)	170.0 (3)
C5—H5 \cdots O1	0.93	2.18	2.772 (3)	120

Symmetry codes: (i) $-x+1, -y+2, -z$.

Fig. 1

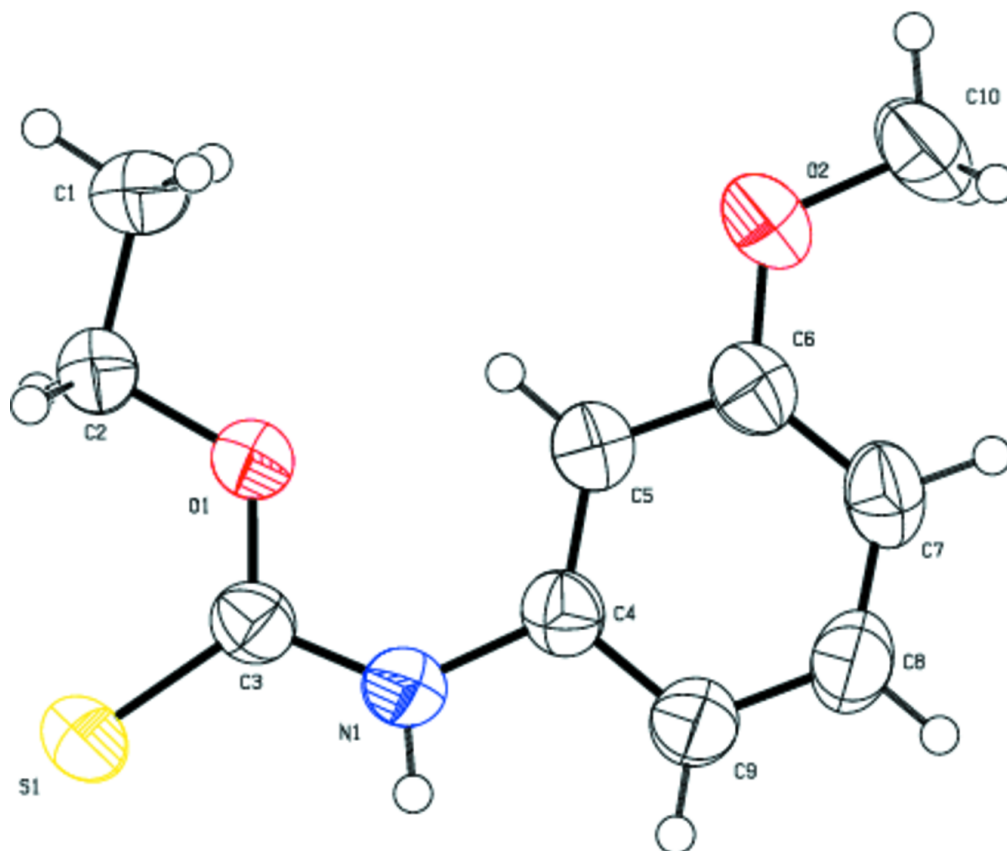


Fig. 2

