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N-Ethoxycarbonyl-*N*′-phenylthiourea

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.096
Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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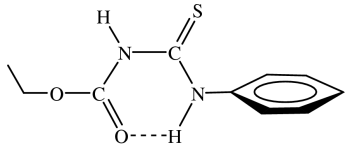
The title compound, $C_{10}H_{12}N_2O_2S$, adopts a *cis–trans* configuration, where the phenyl group and the ethoxycarbonyl moiety lie, respectively, *cis* and *trans* relative to the S atom across the thiourea C—N bonds. Both N—H atoms participate in intermolecular hydrogen bonds and one also forms an intramolecular hydrogen bond. The molecules in the crystal pack in alternating orientations to form ribbons.

Comment

Thiourea compounds are excellent bioactive agents. A number of biological activities are associated with substituted thiourea derivatives (Schroeder, 1955; Antholine & Taketa, 1982), and some *N*-substituted-*N*′-alkoxycarbonylthiourea compounds have been used as antifungal agents. *N*-Substituted-*N*′-alkoxycarbonylthiourea compounds have also attracted considerable attention in recent years because of their coordination ability with transition metal ions such as Cu^{I}, Zn^{II} and Cd^{II} (Shen *et al.*, 1997). As a part of our research into the coordination behaviour, synthesis and biological activities of *N*-substituted-*N*′-alkoxycarbonylthioureas (Zhang *et al.*, 2000, 2001), the crystal structure of the title compound, (I), has been determined.



(I)

In the molecular structure of (I), the carbonyl and thiocarbonyl moieties point in approximately opposite directions. The compound adopts a *cis–trans* configuration, where the phenyl group and the ethoxycarbonyl moiety lie, respectively, *cis* and *trans* relative to the S atom across the thiourea C—N bonds. Both N—H atoms participate in the formation of hydrogen bonds. An intramolecular hydrogen bond exists between atoms N1 and O1 (Table 1).

The molecules are connected *via* N—H···O and N—H···S hydrogen bonds (Fig. 1 and Table 1) and pack in alternating orientations in a ribbon-like fashion, approximately parallel to the **b** direction.

The molecular structure of (I) is analogous to that observed in the crystal structure of *N*-(*o*-nitrophenyl)-*N*′-methoxycarbonylthiourea (Shen, Shi, Kang, Liu *et al.*, 1998) and *N*-(*p*-nitrophenyl)-*N*′-ethoxycarbonylthiourea (Shen, Shi, Kang, Tong *et al.*, 1998). The existence of intramolecular hydrogen bonds in thiourea molecules has significant implications for their coordination properties (Bourne & Koch, 1993). In the

coordination compound reported by Bourne & Koch (1993), namely *cis*-bis(*N*-benzoyl-*N*′-propylthiourea)dichloroplatinum(II), the two ligand molecules bind to Pt^{II} *via* the S atoms only, the carbonyl O atom being locked into position by hydrogen bonds similar to that in the free ligands.

Experimental

Ethyl chloroformate was treated with potassium thiocyanate in ethyl acetate under the conditions of solid-liquid phase transfer catalysis, using 3% polyethylene glycol-400 as the catalyst, to give the corresponding ethoxycarbonyl isothiocyanate, which was reacted with aniline to give the title compound. The solid was separated from the liquid phase by filtration, washed with ethyl acetate and then dried in air. Single crystals were obtained, after two weeks, by slow evaporation of an ethanol solution.

Crystal data

$C_{10}H_{12}N_2O_2S$
$M_r = 224.28$
Triclinic, $P\overline{1}$
$a = 5.787$ (1) Å
$b = 10.218$ (2) Å
$c = 10.501$ (2) Å
$\alpha = 109.39$ (2)°
$\beta = 94.41$ (2)°
$\gamma = 100.04$ (1)°
$V = 570.6$ (2) Å^3
$Z = 2$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 31 reflections
$\theta = 3.5$–13.8°
$\mu = 0.27$ mm^{-1}
$T = 296$ (2) K
Block, colorless
0.58 × 0.44 × 0.30 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scan (*XSCANS*; Siemens, 1994)
$T_{min} = 0.858$, $T_{max} = 0.923$
2611 measured reflections
2277 independent reflections
1823 reflections with $I > 2\sigma(I)$
$R_{int} = 0.009$
$\theta_{max} = 26.5°$
$h = 0 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$
3 standard reflections
every 97 reflections
intensity decay: 2.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.096$
$S = 1.06$
2277 reflections
138 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.1635P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.099 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···S1^{i}	0.86	2.51	3.3556 (16)	167
N1—H1N···O1^{ii}	0.86	2.52	3.2082 (19)	138
N1—H1N···O1	0.86	2.02	2.697 (2)	135

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

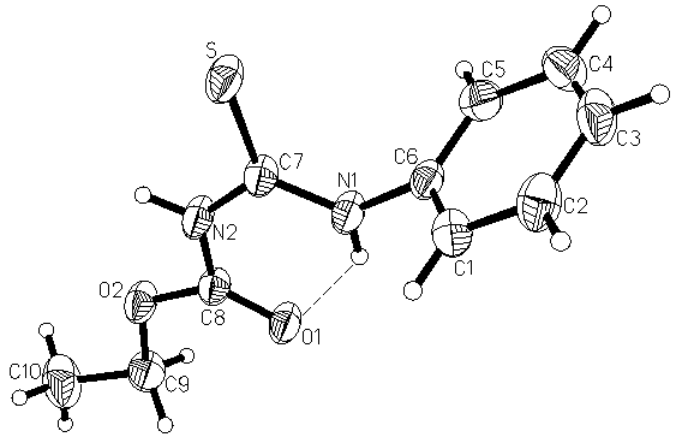


Figure 1
View of the title compound showing the atomic labeling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

The H atoms were included in the riding-model approximation.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Antholine, W. & Taketa, F. (1982). *J. Inorg. Biochem.* **16**, 145–154.
Bourne, S. & Koch, K. R. (1993). *J. Chem. Soc. Dalton Trans.* pp. 2071–2072.
Schroeder, D. C. (1955). *Chem. Rev.* pp. 181–228.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Shen, X., Shi, X., Kang, B., Liu, Y., Tong, Y., Jiang, H. & Chen, K. (1998). *Polyhedron*, **17**, 4049–4058.
Shen, X., Shi, X., Kang, B., Tong, Y., Liu, Y., Gu, L., Liou, Q. & Huang, Y. (1998). *Polyhedron*, **18**, 33–37.
Shen, X., Wen, T., Liu, Q., Huang, X., Kang, B., Wu, X., Huang, Z. & Gu, L. (1997). *Polyhedron*, **16**, 2605–2611.
Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1998). *SHELXTL*. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison Wisconsin, USA.
Zhang, Y. M., Wei, T. B. & Gao, L. M. (2000). *Indian J. Chem. Sect. B*, **39**, 700–702.
Zhang, Y. M., Wei, T. B. & Gao, L. M. (2001). *Synth. Commun.* **31**, 3099–3105.