Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[1-(4-Methoxyphenyl)ethylidene]-4-phenylthiosemicarbazide ethanol hemisolvate

Fangfang Jian* and Ying Li

New Materials & Function Coordination Chemistry Laboratory, Qingdao University of Science & Technology, Qingdao 266042, People's Republic of China

Correspondence e-mail: ffj2003@163169.net

Received 9 May 2006
Accepted 8 September 2006

The title compound, $C_{16}H_{17}N_3OS\cdot0.5C_2H_6O$, was prepared by the reaction of 4-methoxyacetophenone, hydrazine and phenyl isothiocyanate. The molecular structure and packing are stabilized by intramolecular N—H···N and intermolecular N—H···S and C—H···π interactions.

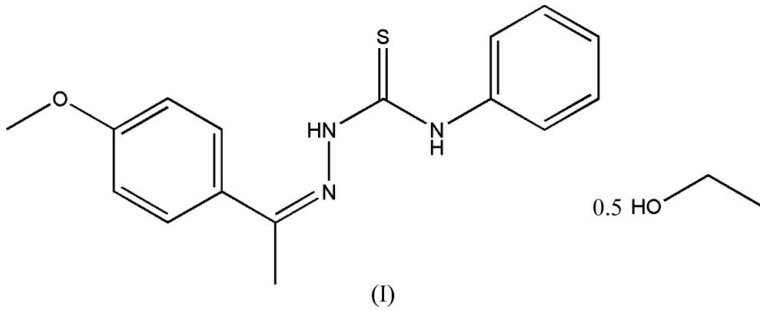
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
H-atom completeness 86%
Disorder in solvent or counterion
R factor = 0.092
wR factor = 0.314
Data-to-parameter ratio = 15.7

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

Comment

Thiourea (TU) is a very convenient nucleophile frequently used to study ligand substitution reactions in coordination chemistry due to its good solubility, neutral character and high nucleophilicity (Schiessl *et al.*, 2005). Thiourea derivatives have been successfully screened for various biological activities (Antholine & Taketa, 1982). Furthermore, TU and its derivatives have also been screened for allergenic and carcinogenic factors, and it has been shown that their presence inhibits nitrification in soil and water (Spataru *et al.*, 2005). As TU and its derivatives have unique characteristics, we synthesised the title compound, (I), and describe its structure here.



(I)

There are two symmetry-equivalent 1-[1-(4-methoxyphenyl)ethylidene]-4-phenylthiosemicarbazide molecules and one solvent molecule in the unit cell. Atoms S1, N1, N2, C6 and C7 are essentially coplanar ($p1$). The other five atoms, N2, N3, C8, C9 and C10, also define a plane ($p2$). The dihedral angles formed by the plane of the aryl ring with $p1$ and $p2$ are 53.46 (2) and 58.25 (1)° for phenyl ring C1–C6, and 22.95 (2) and 17.05 (3)° for benzene ring C10–C15, respectively. The dihedral angle between the two aryl rings is 61.54 (2)°. The dihedral angle formed by $p1$ and $p2$ is 7.02 (2)°.

In the crystal structure, molecules are paired by C—H···π interactions *via* H9 to the centroid ($Cg1$) of the C10–C15 ring of an adjacent molecule [C—H = 0.96 Å, H···$Cg1^{ii}$ = 2.88 Å, C···$Cg1^{ii}$= 3.775 (5) Å, C—H···$Cg1^{ii}$ angle = 155°] [symmetry code: (ii) 1 + x, y, z]. In addition, intramolecular N—H···N and intermolecular N—H···S and C—H···S interactions are observed (Table 1).

Experimental

The title compound was prepared by the reaction of hydrazine (1.0 g, 20 mmol), 4-methoxyacetophenone (3.0 g, 20 mmol) and phenyl isothiocyanate (2.7 g, 20 mmol). Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from ethanol solution at room temperature. (yield 11.2 g, 87.5%; m.p 436–438 K).

Crystal data

$C_{16}H_{17}N_3OS\cdot0.5C_2H_6O$
M_r = 322.42
Triclinic, $P\overline{1}$
a = 5.936 (1) Å
b = 11.674 (2) Å
c = 13.962 (3) Å
α = 68.50 (3)°
β = 84.83 (3)°
γ = 87.53 (3)°
V = 896.5 (3) Å^3
Z = 2
D_x = 1.194 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 293 (2) K
Block, yellow
0.35 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
3488 measured reflections
3152 independent reflections
2151 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 25.0°
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.092
$wR(F^2)$ = 0.314
S = 1.25
3152 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.042 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N3	0.86	2.14	2.566 (5)	110
N2—H2A···S1^{i}	0.86	2.80	3.660 (4)	176
C9—H9A···S1^{i}	0.96	2.80	3.464 (5)	127

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

The ethanol solvent molecule is disordered and a site-occupancy factor of 0.5 was used. The H atoms of the disordered ethanol could not be located in a difference Fourier map. Atoms (O2, C17 and C18) were refined isotropically and the bond lengths were fixed [C—O = 1.42 (1) Å and C—C = 1.52 (1) Å]. The amine (N—H = 0.86 Å), aromatic (C—H = 0.93 Å) and aliphatic (C—H = 0.96 Å) H atoms were all placed in calculated positions and refined in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq} (parent atom) or 1.5U_{eq}(methyl C). The highest peak is located 1.58 Å from atom C17.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

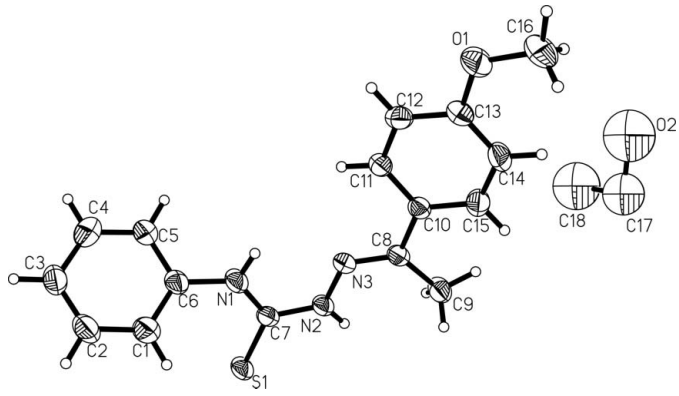


Figure 1
The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms were not located for the disordered ethanol molecule.

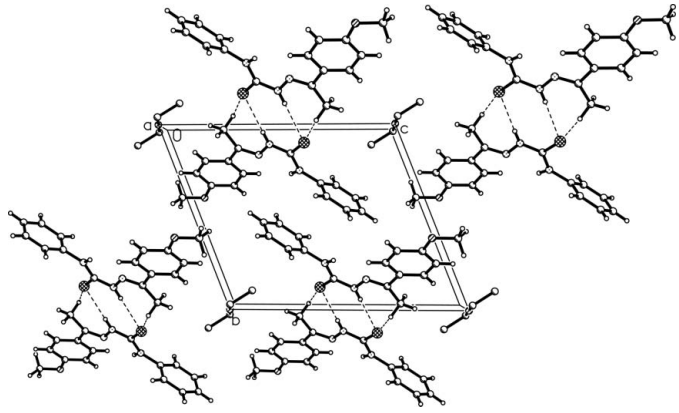


Figure 2
Packing diagram, projected down the *a* axis, of the title compound. Hydrogen bonds are shown as dashed lines.

The authors thank the Natural Science Foundation of Shandong Province (No. Y2005B04).

References

Antholine, W. & Taketa, F. (1982). *J. Inorg. Biochem.* **16**, 145–154.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Schiessl, W. C., Summa, N. K., Weber, C. F., Gubo, S., Ducker-Benfer, C., Puchta, R., Van Eikema Hommes, N. J. R. & Van Eldik, R. (2005). *Z. Anorg. Allg. Chem.* **631**, 2812–2819.
Sheldrick, G. M. (1990). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Spataru, N., Spataru, T. & Fujishima, A. (2005). *Electroanalysis*, **17**, 800–805.