

***N,N'*-Bis[(4-chloroanilino)thiocarbonyl]-
isophthalamide dimethylformamide solvate****You-Ming Zhang, Cheng Cao, Qi
Lin and Tai-Bao Wei***College of Chemistry and Chemical Engineering,
Gansu Key Laboratory of Polymer Materials,
Northwest Normal University, Lanzhou, Gansu
730070, People's Republic of China

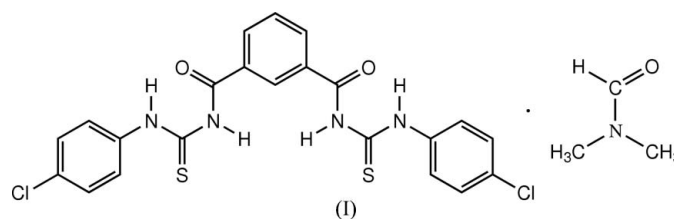
Correspondence e-mail: weitaobao@126.com

Key indicatorsSingle-crystal X-ray study
 $T = 289\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.036
 wR factor = 0.088
Data-to-parameter ratio = 14.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The principal molecule of the title compound, $\text{C}_{22}\text{H}_{16}\text{Cl}_2\text{N}_4\text{O}_2\text{S}_2\cdot\text{C}_3\text{H}_7\text{NO}$, is not planar; the three benzene rings are tilted with respect to each other, with dihedral angles of 5.51 (11), 60.06 (12) and 64.33 (11)°. Intermolecular hydrogen bonding and π - π stacking help to stabilize the crystal structure.

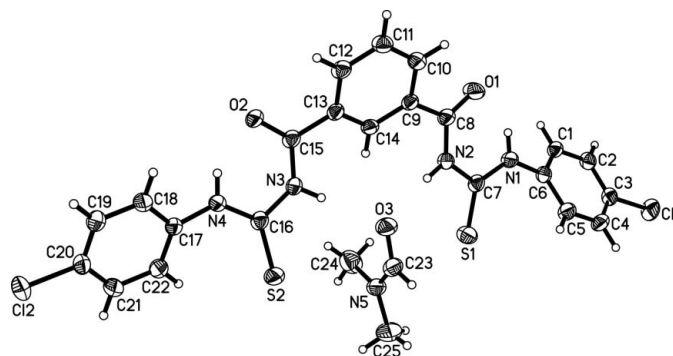
Received 1 March 2006
Accepted 30 March 2006**Comment**

Acythiureas and their coordination compounds have been widely studied because of their potential use in extraction, separation, medicine, agriculture and analytical chemistry (Schroeder, 1955; Antholine & Taketa, 1982). As part of our work on acyothiurea derivatives (Zhang *et al.*, 2003), we report here the structure of the title compound, (I).



The molecular structure of (I) is shown in Fig. 1. The molecule is not planar; the three benzene rings are tilted with respect to each other; the dihedral angles are 64.33 (11) (between the C1- and C9-containing rings), 60.07 (12) (between the C1- and C17-containing ring) and 5.51 (11)° (between the C9- and C17-containing rings).

Intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds are observed in the crystal structure (Table 1). The centroid-to-centroid separation of 3.8057 (15) Å indicates the existence of π - π stacking between the parallel C1- and C1ⁱ-containing benzene rings [symmetry code: (i) $2 - x, 2 - y, 1 - z$].

**Figure 1**

The asymmetric unit of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Experimental

An acetone solution (5 ml) of isophthaloyl dichloride (3 mmol) was added to an acetone solution (5 ml) of NH_4SCN (7 mmol), and the mixture was subjected to ultrasonic irradiation for 10 min. Chloroaniline (6 mmol) was added and the mixture was irradiated for a further 10 min. The reaction mixture was poured into a water-ice mixture and stirred for 15 min. The resulting white precipitate was filtered off and washed with ethanol three times. Single crystals of (I) were obtained by recrystallization from a DMF solution.

Crystal data

$\text{C}_{22}\text{H}_{16}\text{Cl}_2\text{N}_4\text{O}_2\text{S}_2\cdot\text{C}_3\text{H}_7\text{NO}$	$Z = 4$
$M_r = 576.50$	$D_x = 1.466 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 14.703 (3) \text{ \AA}$	$\mu = 0.45 \text{ mm}^{-1}$
$b = 8.479 (1) \text{ \AA}$	$T = 289 (2) \text{ K}$
$c = 21.223 (4) \text{ \AA}$	Block, colorless
$\beta = 99.25 (2)^\circ$	$0.48 \times 0.44 \times 0.34 \text{ mm}$
$V = 2611.4 (8) \text{ \AA}^3$	

Data collection

Siemens P4 diffractometer	$R_{\text{int}} = 0.015$
ω scans	$\theta_{\text{max}} = 26.0^\circ$
Absorption correction: none	3 standard reflections
5911 measured reflections	every 97 reflections
5133 independent reflections	intensity decay: 6.4%
3480 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 0.97$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
5133 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
353 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0060 (5)

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}$	0.86 (1)	1.92 (2)	2.640 (2)	141 (2)
$\text{N2}-\text{H2N}\cdots\text{O3}$	0.85 (1)	2.19 (1)	3.039 (2)	171 (2)
$\text{N3}-\text{H3N}\cdots\text{O3}$	0.85 (1)	2.19 (1)	3.035 (2)	172 (2)
$\text{N4}-\text{H4N}\cdots\text{O2}$	0.85 (1)	1.88 (1)	2.612 (2)	143 (2)

H atoms on N atoms were located in a difference Fourier map and refined isotropically. Methyl H atoms were placed in calculated positions, with $\text{C}-\text{H} = 0.96 \text{ \AA}$ and their torsion angles refined to fit the electron density, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Other H atoms were placed in calculated positions, with $\text{C}-\text{H} = 0.93 \text{ \AA}$, and refined in riding mode, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The work was supported by the Natural Science Foundation of China (No. 20371040), the Key Scientific and Technical Research Project of the Ministry of Education of China (No. 205161) and the Youth Foundation of Gansu province (No. 3YS051-A25-010).

References

- Antholine, W. & Taketa, F. (1982). *J. Inorg. Biochem.* **16**, 145–154.
 Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
 Schroeder, D. C. (1955). *Chem. Rev.* **55**, 181–228.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Zhang, Y.-M., Xian, L. & Wei, T.-B. (2003). *Acta Cryst.* **C59**, m473–m474.