

N*-Benzoyl-*N'*-(2-hydroxyethyl)thiourea*You-Ming Zhang,^{a*} Liang Xian,^a
Tai-Bao Wei^a and Li-Xiang Cai^b**^aDepartment of Chemistry, Northwest Normal University, Lanzhou, Gansu 730070, People's Republic of China, and ^bAnalysis and Measurement Center, Lanzhou University, Lanzhou, Gansu 730000, People's Republic of China

Correspondence e-mail: keji chu@nwnu.edu.cn

Key indicators

Single-crystal X-ray study

T = 293 KMean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$ *R* factor = 0.045*wR* factor = 0.138

Data-to-parameter ratio = 14.0

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

In the title compound, $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$, the carbonyl and thiocarbonyl moieties are pointing in approximately opposite directions, and the six atoms in the hydrogen-bonded ring structure are almost coplanar.

Comment

Thiourea compounds are excellent agents of bioactive substances. A number of biological activities are associated with substituted thiourea derivatives (Schroeder, 1955). A survey of the literature reveals that some work has been reported on benzoylthiourea, which has found plenty of applications as a facile and simple ligand in the determination of traces of transition metals, and as an available starting material in the preparation of a wide variety of metal complexes (Koch, 2001). As part of our work studying the coordination behaviour of benzoylthiourea and its bioactivity, and in continuation of previous work on benzoylthiourea (Wei & Zhang, 1998), the crystal structure of the title compound, (I), is reported. To date, the coordination compounds synthesized from *N*-benzoyl-*N'*-(2-hydroxyethyl)thiourea with Os(VIII) (Bhowal, 1975), and Pt(II) (Koch *et al.*, 1995) have been reported, and its cyclization reaction with H_2SO_4 has also been investigated (Klayman & Woods, 1975). In a ^{13}C NMR study (Imrich *et al.*, 1994), the differences between the benzoyl (CO) chemical shift values of *N*-monosubstituted and *N,N*-disubstituted thioureas indicated the existence of an intramolecular hydrogen bond, namely between the benzoyl CO and the NH groups. This indication also was supported by the ^1H NMR spectrum (Koch *et al.*, 1995).

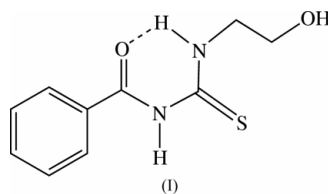


Fig. 1 shows the molecular structure of (I), indicating that the carbonyl and thiocarbonyl moieties point in approximately opposite directions. The six atoms in the hydrogen-bonded ring structure are almost coplanar. The N2—H pendant arm extends towards the carbonyl O atom and forms an intramolecular hydrogen bond between them; other intra- and intermolecular hydrogen bonds are also formed (Table 1). The structure is analogous to that observed in the crystal structures of *N*-propyl-*N'*-benzoylthiourea (Dago *et al.*, 1989), *N*-benzoyl-*N'*-(2,6-dimethylphenyl)thiourea (Usman *et al.*, 2002), *N*-benzoyl-*N'*-phenylthiourea (Yamin & Yusof, 2003a) and *N*-benzoyl-*N'*-*p*-bromophenylthiourea (Yamin & Yusof,

Received 11 April 2003

Accepted 9 May 2003

Online 16 May 2003

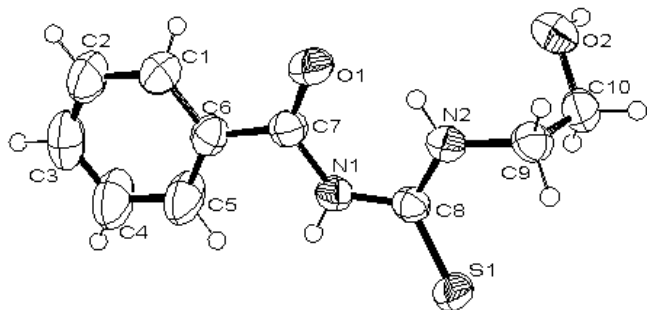


Figure 1
View of the molecule, showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 50% probability level.

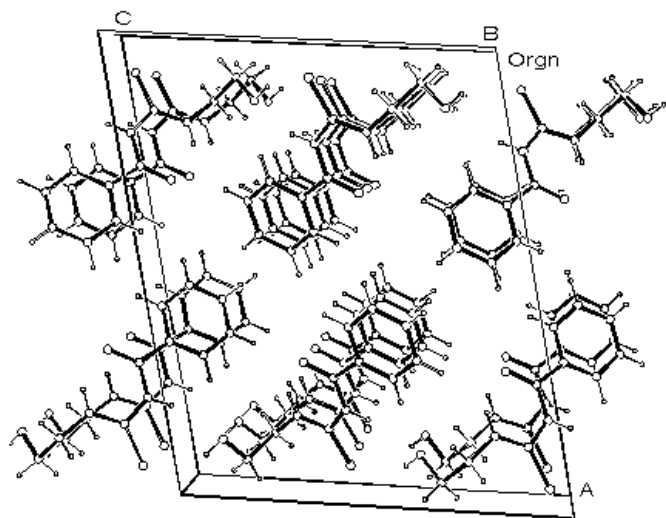


Figure 2
Crystal packing diagram.

2003b). The existence of hydrogen bonding in a benzoylthiourea molecular six-membered ring structure has significant implications for the coordination properties (Bourne & Koch, 1993), suggesting the possibility of intramolecular hydrogen-bond-controlled coordination behaviour of these ligands. In the coordination compound reported by Bourne & Kock, *viz.* *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 a hydrogen bond similar to that in the free ligands.

Experimental

Reagents and organic solvents were of analytical reagent grade and commercially available. Benzoyl chloride was treated with ammonium thiocyanate in CH₂Cl₂ under solid-liquid phase transfer catalysis conditions, using 3% polyethylene glycol-600 as the catalyst, to give the corresponding benzoyl isothiocyanate, which was reacted with ethanolamine to give the title compound. The solid was separated from the liquid phase by filtration, washed with CH₂Cl₂ and then dried in air. Single crystals were obtained by the slow evaporation of an ethanol solution after 2 weeks; one of them was selected optically for the diffraction study and glued to a glass fibre.

Crystal data

C₁₀H₁₂N₂O₂S
M_r = 224.28
 Monoclinic, *P*2₁/*c*
a = 17.083 (3) Å
b = 4.5490 (10) Å
c = 14.279 (3) Å
 β = 102.44 (3)° **precision OK?**
V = 1083.6 (4) Å³
Z = 4

D_x = 1.375 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 10–20°
 μ = 0.28 mm⁻¹
T = 293 (2) K
 Block, colorless
 0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 3712 measured reflections
 1908 independent reflections
 1564 reflections with *I* > 2σ(*I*)
R_{int} = 0.091

θ_{\max} = 25.0°
h = –20 → 19
k = –5 → 5
l = 0 → 16
 3 standard reflections
 frequency: 60 min
 intensity decay: 0.2%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.045
wR (*F*²) = 0.138
S = 1.09
 1908 reflections
 136 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.3405P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N2–H2A...O1	0.90	1.92	2.631 (2)	134
C1–H1B...O1	0.96	2.44	2.762 (3)	99
N1–H1A...O2 ⁱ	0.82	2.28	3.081 (3)	166
C5–H5A...O2 ⁱⁱ	0.96	2.41	3.361 (4)	171
O2–H2B...S1 ⁱⁱⁱ	0.85	2.44	3.206 (2)	150
C9–H9A...S1	0.96	2.68	3.091 (2)	106

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

The positions of all H atoms were fixed geometrically and refined as riding.

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1977); cell refinement: *CAD-4 SDP/VAX* (Enraf–Nonius, 1989); data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL/PC* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Natural Science Foundation (No. 29971026) of China and the Foundation (No. 02–18) of Northwest Normal University, which are gratefully acknowledged.

References

- Bhowal, S. K. (1975). *Indian J. Chem.* **13**, 92–94.
 Bourne, S. & Koch, K. R. (1993). *J. Chem. Soc. Dalton Trans.* pp. 2071–2072.
 Dago, A., Shepelev, Y., Fajardo, F., Alvarez, F. & Pomes, R. (1989). *Acta Cryst.* **C45**, 1192–1194.
 Enraf–Nonius (1989). *CAD-4 SDP/VAX*. Delft Instruments X-ray Diffraction, PO Box 811, 2600 AV Delft, The Netherlands.
 Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.
 Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.

- Imrich, J., Busova, T., Kristian, P. & Dzara, J. (1994). *Chem. Pap.* **48**, 42–46.
- Klayman, D. L. & Woods, T. S. (1975). *J. Org. Chem.* **40**, 2000–2002.
- Koch, K. R. (2001). *Coord. Chem. Rev.* **216–217**, 473–488.
- Koch, K. R., Sacht, C. & Bourne, S. (1995). *Inorg. Chim. Acta*, **232**, 109–115.
- Schroeder, D. C. (1955). *Chem. Rev.* pp. 181–228.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997a). *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL/PC*. Version 5.101. Bruker AXS Inc., Madison, Wisconsin, USA.
- Usman, A., Razak, I. A., Satar, S., Kadir, M. A., Yamin, B. M. & Fun, H.-K. (2002). *Acta Cryst.* **E58**, o656–o658.
- Wei, T. B. & Zhang, Y. M. (1998). *Synth. Commun.* **28**, 2851–2859.
- Yamin, B. M. & Yusof, M. S. M. (2003a). *Acta Cryst.* **E59**, o151–o152.
- Yamin, B. M. & Yusof, M. S. M. (2003b). *Acta Cryst.* **E59**, o340–o341.