Crystal Structure of *N*–[(1-Hydroxy-3-phenylpropan-*(R,S)*-2-yl)carbamothioyl]-3-nitrobenzamide

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N–[(1-Hydroxy-3-phenylpropan-*(R,S)*-2-yl)carbamothioyl]-3-nitrobenzamide was synthesized and the crystal structure was determined by single-crystal X-ray diffractometry. The carbonyl and thiocarbonyl moieties point in approximately opposite directions. The unit-cell dimensions are $a = 7.226(2)\text{Å}$, $b = 7.408(2)\text{Å}$, $c = 16.557(5)\text{Å}$ with $\alpha = 84.990(4)^\circ$, $\beta = 83.809(9)^\circ$, $\gamma = 89.478(4)^\circ$, $V = 877.8(4)\text{Å}^3$.

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The chemistry of thiourea derivatives has attracted much attention in recent years due to their broad spectrum of biological activities, and as important intermediates in the preparations of metal complexes and heterocyclic compounds.^{1–3} Their potential coordination behavior with transition metals was reported.⁴ The biological activities of complexes with thiourea derivatives have been successfully screened for various biological activities.⁵ In addition, thioureas have been shown to possess antibacterial, antifungal, antitubercular, antithyroid and insecticidal properties.⁶ Also, these compounds have been widely used in enantioselective synthesis,^{7–9} such as in nitro-Mannich reactions, the aza-Henry reaction, and Michael addition. In this paper, we show the crystal structure of *N*–[(1-hydroxy-3-phenylpropan-*(R,S)*-2-yl)carbamothioyl]-3-nitrobenzamide. A chemical diagram of the complex is shown in Fig. 1.

The title compound was synthesized the following procedure. To a solution of 3-nitrobenzoyl chloride (1 mmol) in dichloromethane (5 mL) was added ammonium thiocyanate (1.3 mmol) and PEG-400 (0.1 mmol). The mixture was then stirred at room temperature for 60 min, and solution of (*S*)-2-amino-3-phenylpropan-1-ol (0.9 mmol) in dichloromethane (2 mL) was added. The mixture continuously stirred for 30 min. After the reaction completed, water (10 mL) was added the organic phase was dried with sodium sulfate. The solvent was removed in vacuum and the residue was purified by flash chromatography to give a white solid. Yield: 90%. Found: C, 56.94; H, 4.78; N, 8.01, calculated for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_4\text{S}$: C, 56.81; H, 4.77; N, 11.69; A single crystal suitable for X-ray characterization was obtained

by the slow evaporation of ethanol for about one week. Single crystals were collected *via* filtration, washed with a cold ethanol and dried at room temperature.

A single crystal of the complex with approximate dimensions of $0.24 \times 0.26 \times 0.30\text{ mm}$ was mounted in air. Reflection data were collected on a Bruker Smart Apex CCD area detector using ϕ - ω scans. The structure was solved by direct method using SHELXS and refined by the full-matrix least-squares method by SHELXL. To our surprise, racemization took place in the reaction processes although the chiral reagent ((*S*)-2-amino-3-phenylpropan-1-ol) is really optically active.

Table 1 Crystallographic data and experimental details

Chemical formula: $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_4\text{S}$	
Formula weight = 359.40	
$T = 291(2)\text{K}$	
Crystal system: Triclinic	Space group: $P\bar{1}$
$a = 7.226(2)\text{Å}$	$\alpha = 84.990(4)^\circ$
$b = 7.408(2)\text{Å}$	$\beta = 83.809(3)^\circ$
$c = 16.557(5)\text{Å}$	$\gamma = 89.478(4)^\circ$
$V = 877.8(4)\text{Å}^3$	$Z = 2$
$D_x = 1.360\text{ g/cm}^3$	
Radiation: Mo K_α ($\lambda = 0.71073\text{ Å}$)	
$\mu(\text{Mo } K_\alpha) = 0.211\text{ mm}^{-1}$	$F(0\ 0\ 0) = 376$
Crystal size = $0.24 \times 0.26 \times 0.30\text{ mm}$	
No. of reflections collected = 2536	
No. of independent reflections = 3370	
θ range for data collection: $2.48 \leq \theta \leq 26.00^\circ$	
Data/Restrains/Parameters = 3370/0/235	
Goodness-of-fit on $F^2 = 1.043$	
R indices [$I > 2\sigma(I)$]: $R1 = 0.0685$, $wR2 = 0.1350$	
R indices (all data): $R1 = 0.1106$, $wR2 = 0.1437$	
$(\Delta\rho)_{\text{max}} = 0.000$	
$(\Delta\rho)_{\text{max}} = 0.233\text{ eÅ}^{-3}$	
$(\Delta\rho)_{\text{min}} = -0.207\text{ eÅ}^{-3}$	
Measurements:	Bruker Smart Apex CCD
Program system:	SHELXTL
Structure determination:	direct methods
Refinement method:	full-matrix least-squares
CCDC	729967

Fig. 1 Chemical diagram of the title compound.

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Fig. 2 ORTEP structure of *N*-[(1-hydroxy-3-phenylpropan-(*R,S*)-2-yl)carbamoithioly]-3-nitrobenzamide, showing 50% probability ellipsoids.

Table 2 Selected bond lengths (Å) and bond angles (°)

C5-N1	1.432(5)	C10-H10A	0.9700
C7-O1	1.223(4)	C10-H10B	0.9700
C7-N2	1.377(5)	C17-O3	1.404(4)
C8-N3	1.285(5)	C17-H17A	0.9700
C8-N2	1.387(5)	C17-H17B	0.9700
C8-S1	1.699(4)	N1-O4	1.163(4)
C9-N3	1.470(5)	N1-O2	1.229(4)
C9-C10	1.502(5)	N2-H2A	0.87(4)
C9-C17	1.530(5)	N3-H3A	0.85(4)
C9-H9	0.9800	O3-H3D	0.85(4)
C6-C5-N1	118.4(3)	O3-C17C9	117.2(3)
C4-C5-N1	121.6(3)	H17A-C17-H17B	107.3
O1-C7-N2	123.2(3)	O4-N1-O2	119.4(3)
O1-C7-C1	119.8(4)	O4-N1-C5	121.6(3)
N2-C7-C1	116.9(3)	O2-N1-C5	118.0(3)
N3-C8-N2	119.5(4)	C7-N2-C8	126.2(3)
N3-C8-S1	123.2(3)	C7-N2-H2A	106(3)
N2-C8-S1	116.9(3)	C8-N2-H2A	106(2)
N3-C9-C10	107.7(3)	C8-N3-C9	121.6(3)
N3-C9-C17	114.5(3)	C8-N3-H3A	107(3)
N3-C9-H9	108.7	C9-N3-H3A	107(3)
C10-C9-H9	108.7	C17-O3-H3D	106(3)

H atoms bonded to *N* and *O* atoms were located in a difference map and refined with distance restraints of O-H = 0.85(4) Å and N-H = 0.85(4)-0.87(4) Å, and with $U_{\text{iso}}(\text{H}) = 1.2\text{eq}(\text{N}, \text{O})$. Other H atoms were positioned geometrically and refined using a riding model with C-H = 0.93-0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2\text{eq}$. Crystallographic data and experimental details for structural analyses are summarized in Table 1. The atomic coordinates and the equivalent isotropic displacement parameters for the nonhydrogen atoms are listed in Table 2.

Table 3 Hydrogen-bonding geometry (Å, °)

D-H...A	D-H	H...A	D...A	D-H...A
O3-H3D...O1	0.85(4)	2.32(4)	3.031(4)	141(4)

In the crystal structure, the direction of the carbonyl is opposite to that of thiocarbonyl. The existence of hydrogen bonds in a benzoylthiourea molecular provides evidence for the coordination properties, suggesting the possibility of intermolecular hydrogen-bond-controlled coordination behavior of these ligands. For example,¹⁰ in the compound *cis*bis(*N*-benzoyl-*N'*-propylthiourea)dichloroplatinum, the S atoms of two ligands are coordinated to Pt(II), while the carbonyl O atom is still bound to N2-H, which is similar to a free ligand. The molecules also feature intra & intermolecular N-H...O hydrogen bonds (See hydrogen-bond geometry Table 3).

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