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Bis[(*S*)-(–)-α-methylbenzyl]thiourea

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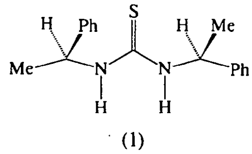
Abstract

The title compound, 1,3-bis[(*S*)-(−)-1-phenylethyl]thiourea, $C_{17}H_{20}N_2S$, exhibits bond lengths and angles consistent with the build-up of negative charge on the S atom. There are weak hydrogen bonds between molecules leading to helices disposed about crystallographic 3_1 axes.

Comment

Our recent interest in the application of guanidines as ligands for transition metals has lead us to isolate a number of different thiourea compounds as intermediates in their synthesis (Bailey, Blake, Kryszczuk, Parsons & Reed, 1995). We report here the crystal structure of one of these, namely, enantiomerically pure bis[(*S*)-(−)-α-methylbenzyl]thiourea, (1), which was synthesized *via* standard procedures from (*S*)-(−)-α-methylbenzylamine and carbon disulfide (Yamazaki, Higashi & Iguchi, 1974).



(1)

The crystal structure is composed of discrete molecules of (1) (Fig. 1), the bond lengths and angles (Table 1) being quite typical for acyclic thiourea compounds in the Cambridge Structural Database (Allen & Kennard, 1993; Fletcher, McMeeking & Parkin, 1996). The C—S bond length [1.710 (7) Å] lies at the low end of the range associated with C—S single bonds (1.70–1.80 Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1992), while the average C—N bond length [1.321 (8) Å] is similar to 'standard' C—N bonds in aromatic rings [1.336 (14) Å; Allen *et al.*, 1992]. Compound (1) should thus be properly regarded as a resonance hybrid of C=N-containing canonical forms, with only a relatively small contribution from the C=S-bonded form frequently used to represent thiourea compounds (see scheme below). The phenyl groups adopt a pseudo-antiperiplanar arrangement in order to minimize steric crowding of the α-methylbenzyl functionality.

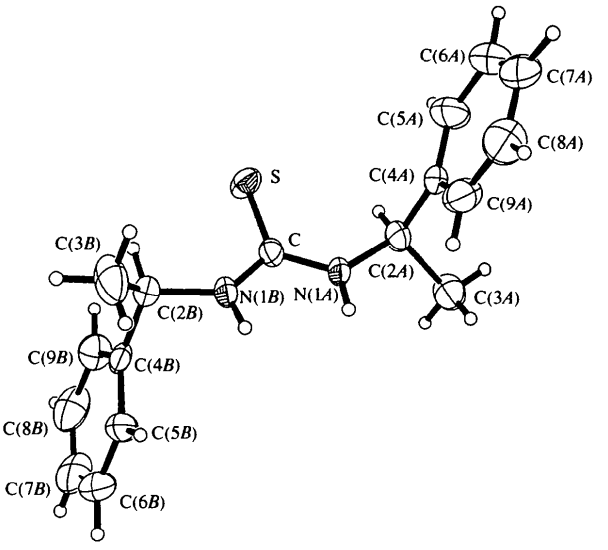
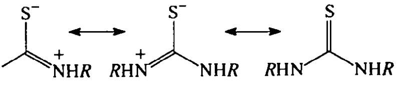


Fig. 1. A view of the title molecule with the atom-numbering scheme. Displacement ellipsoids enclose 30% probability surfaces. The dihedral angle between the planes of the phenyl rings is 11.6 (4)°.



R = —CH(Me)Ph

Hydrogen bonds link molecules to form a helix around the crystallographic threefold screw axis. Each molecule is linked to its neighbour in the helix by two N—H···S interactions [$N1A\cdots S^{i}$ 3.411 (6) and $N1B\cdots S^{i}$ 3.545 (7) Å; symmetry code: (i) $2-y$, $1+x-y$, $\frac{1}{3}+z$]. There appear to be no significant contacts between the helices.

Experimental

Crystals of (1) were grown from dichloromethane layered with diethyl ether. At 1.08 mm, the crystal selected for study is long by normal standards, but the crystals shattered when attempts were made to cut them. A 1.2 mm diameter collimator was used during data collection.

Crystal data

$C_{17}H_{20}N_2S$
$M_r = 284.41$
Trigonal
$P3_1$
$a = 10.3144\,(12)$ Å
$c = 13.177\,(2)$ Å
$V = 1214.1\,(3)$ Å^3
$Z = 3$
$D_x = 1.167$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 56 reflections
$\theta = 12.5–14.0°$
$\mu = 0.193$ mm^{-1}
$T = 296\,(2)$ K
Needle
$1.08 \times 0.16 \times 0.16$ mm
Colourless

Data collection

Stoe Stadi-4 four-circle diffractometer
ω–θ scans with width $(1.32 + 0.35\tan\theta)°$
Absorption correction: none
1890 measured reflections
1053 independent reflections
719 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.0451$
$\theta_{\rm max} = 22.51°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F > 4\sigma(F)] = 0.0456$
$wR(F^2) = 0.0788$
$S = 1.018$
1053 reflections
192 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.044$
$\Delta\rho_{\rm max} = 0.136$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.154$ e Å^{-3}

Extinction correction: *SHELXTL* (Sheldrick, 1995)
Extinction coefficient: 0.008 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.18 (17)

Table 1. *Selected geometric parameters (Å, °)*

S—C	1.710 (7)	N1*A*—C2*A*	1.444 (8)
C—N1*A*	1.318 (8)	N1*B*—C2*B*	1.446 (8)
C—N1*B*	1.324 (7)		
N1*A*—C—N1*B*	115.5 (6)	C—N1*A*—C2*A*	127.3 (6)
N1*A*—C—S	121.4 (5)	C—N1*B*—C2*B*	128.7 (7)
N1*B*—C—S	123.1 (6)		

The crystal diffracted only weakly at high angle, so data were not collected beyond $\theta = 22.5°$. H atoms attached to C atoms were placed in calculated positions and subsequently allowed to ride on their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Methyl groups were treated as rotating rigid groups with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. H atoms attached to N atoms were located in a difference synthesis; the positional parameters were refined subject to similarity restraints on the N—H distances and the isotropic displacement parameters refined freely.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXTL* (Sheldrick, 1995). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: HA1175). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2′,3′-Diphenyl-Substituted *p*-Terphenyls

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Abstract

The crystal structure analyses of the title substituted *p*-terphenyls, 2′-(*p*-methoxyphenyl)-3′-phenyl-*p*-terphenyl, $C_{31}H_{24}O$, and 2′,3′-bis(*p*-methoxyphenyl)-*p*-