

Bis(N-isopropyl-N-phenylthiocarbamoyl) disulfide

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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
Disorder in main residue
 R factor = 0.041
 wR factor = 0.125
Data-to-parameter ratio = 15.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis(*N*-isopropyl-*N*-phenylthiocarbamoyl) disulfide

The NCS₂ groups in the title compound, C₂₀H₂₃N₂S₄, are approximately perpendicular to each other, forming a dihedral angle of 80.10 (7)°. The S—S bond length is 2.0086 (14) Å and the distance between the two terminal S atoms is 3.940 (3) Å.

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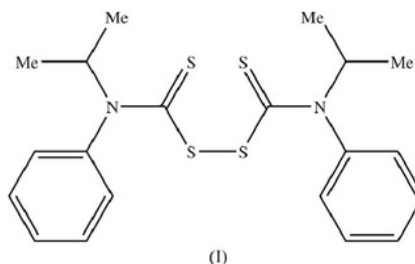
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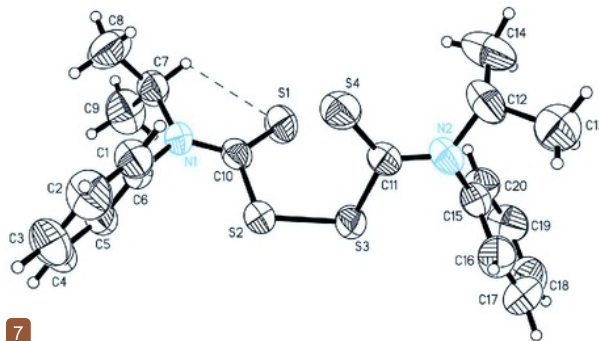
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Comment

Some metal–dithiocarbamate complexes are not stable in solution and, upon recrystallization, lead to the formation of decomposition products. One such example is dipyrrolidinylthiuram disulfide, which was obtained from the recrystallization of [Fe{S₂CN(CH₂)₄}]₃ from methanol solution (Yamin *et al.*, 1996). Similarly, the title compound, (I), was obtained after recrystallization from a chloroform solution of [Eu(S₂NC₁₀H₁₃)₃(1,10-phenanthroline)].



The molecular structure of (I), Fig. 1, is isostructural with other thiuram disulfides, such as tetraethylthiuram disulfide (Karle *et al.*, 1967) and bis(*N*-methyl-*N*-phenylthiocarbamoyl)disulfide (Fun *et al.*, 2001). The S2—S3, C10—S2, C10—S1, C11—S3 and C11—S4 bond distances in (I) are in agreement with these thiuram disulfides (Table 1). However,



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Figure 1

The molecular structure of (I), with 50% probability displacement ellipsoids. The dashed line denotes a C—H...S hydrogen bond. Only the major disordered component of C13 is shown.

the C10—S2—S3—C11 torsion angle of 78.70 (13)° is smaller than the value of approximately 96° observed in tetraethylthiuram disulfide (Karle *et al.*, 1967).

Each of the S3/S4/N2/C11/C15 and S1/S2/N1/C7/C10 fragments is planar, with the maximum deviation for either of these being 0.027 (2) Å for atom N1. The dihedral angle between the two least-squares planes through these groups is 80.10 (7)°. The two phenyl groups, C1—C6 and C15—C20, are inclined to each other by 67.70 (15)°.

The molecule of (I) is stabilized by an intramolecular interaction, with C7—H7···S1 = 2.59 Å [C7···S1 = 3.068 (4) Å], and the angle subtended at the H atom is 110°.

Experimental

The starting Eu(C₁₀H₁₂NS₂)₃(1,10-phenanthroline)] complex was prepared following the method described by Su *et al.* (1995). Recrystallization of this complex from chloroform solution yielded crystals of (I) (m.p. 416–418 K).

Crystal data

C₂₀H₂₃N₂S₄
M_r = 419.64
 Triclinic, *P*1̄
a = 8.911 (7) Å
b = 11.568 (9) Å
c = 12.595 (10) Å
 α = 113.744 (11)°
 β = 97.157 (14)°
 γ = 106.082 (13)°
V = 1100.1 (15) Å³

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 T_{\min} = 0.868, T_{\max} = 0.957
 9761 measured reflections

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.125$
 $S = 1.05$
 3795 reflections
 240 parameters
 H-atom parameters constrained

$Z = 2$
 $D_x = 1.267 \text{ Mg m}^{-3}$
 Mo *K* α radiation
 Cell parameters from 3456 reflections
 $\theta = 1.8\text{--}25.0^\circ$
 $\mu = 0.44 \text{ mm}^{-1}$
 $T = 298 \text{ (2) K}$
 Block, colourless
 0.33 × 0.32 × 0.10 mm

3795 independent reflections
 2703 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $2\theta = 25.0^\circ$
 $h = -10 \rightarrow 10$
 $k = -13 \rightarrow 13$
 $l = -14 \rightarrow 14$

$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.0622P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.40 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1—C10	1.642 (3)	N1—C6	1.446 (3)
S2—C10	1.817 (3)	N1—C7	1.484 (3)
S2—S3	2.0078 (15)	N2—C11	1.333 (3)
S3—C11	1.819 (3)	N2—C15	1.445 (3)
S4—C11	1.644 (3)	N2—C12	1.492 (3)
N1—C10	1.339 (3)		
N1—C10—S1	126.3 (2)	N2—C11—S4	127.04 (19)
N1—C10—S2	110.83 (18)	N2—C11—S3	110.37 (18)
S1—C10—S2	122.82 (15)	S4—C11—S3	122.58 (15)
C6—N1—C10—S1	174.94 (17)	C15—N2—C11—S4	179.36 (18)
C6—N1—C10—S2	−5.4 (3)	C15—N2—C11—S3	−1.7 (3)

All H atoms were placed geometrically in ideal positions and allowed to ride on their parent C atoms, with C—H = 0.93 Å (aromatic), 0.99 Å (methine) and 0.97 Å (methyl), and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic and methine-C})$ and $1.5U_{\text{eq}}(\text{methyl-C})$. Atom C13 was found to be disordered and was refined isotropically in two alternative positions, with refined occupancy factors of 0.71 (2) and 0.29 (2).

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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