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Structure of Two Modifications of 1,1-Diethyl-3-thiobenzoylthiourea

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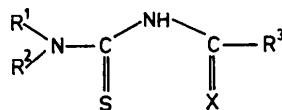
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Abstract. The title compound crystallizes in two polymorphic modifications (I and II). (I) $C_{12}H_{16}N_2S_2$, $M_r = 252.4$, monoclinic, $P2_1/n$, $a = 8.154(1)$, $b = 7.900(1)$, $c = 21.134(1)$ Å, $\beta = 98.92(1)^\circ$, $V = 1344.9(5)$ Å³, $Z = 4$, $D_x = 1.25$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.2$ cm⁻¹, $F(000) = 536$, room temperature. Final $R = 0.056$ for 1286 unique observed reflections. (II) $C_{12}H_{16}N_2S_2$, $M_r = 252.4$, monoclinic, $P2_1$, $a = 8.316(1)$, $b = 14.294(1)$, $c = 11.931(2)$ Å, $\beta = 103.67(1)^\circ$, $V = 1378.1(6)$ Å³, $Z = 4$, $D_x = 1.22$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.2$ cm⁻¹, $F(000) = 536$, room temperature. Final $R = 0.045$ for 1727 unique observed reflections. There are two independent molecules per asymmetric unit. The molecules in both modifications exist as NH tautomers and do not differ significantly in their bond distances and angles. The molecules of (I) as well as the two independent molecules of (II) adopt conformations approximately halfway between E, Z' and Z, Z' . The molecules in both modifications are connected by N–H...S interactions forming dimers, with H...S distances 2.41–2.52 Å.

Introduction. *N*-Chalcogenoacylthioureas are of great interest as chelating ligands in complex chemistry. Whereas the structures of *N*-chalcogenoacylthiourea complexes of transition-metal ions (Fitzl, Beyer, Sieler, Richter, Kaiser & Hoyer, 1977; Knuuttila, Knuuttila, Hennig & Beyer, 1982; Sieler, Richter, Braun, Beyer, Lindqvist & Andersen, 1985) have been described, the structures of the corresponding ligands are generally unknown. We now report the crystal structure of the title compound ($R^1 = R^2 = C_2H_5$, $R^3 = C_6H_5$, $X = S$).



$X = \text{Se, S, O, NR}$

There are three tautomeric forms possible in which the H atom is bonded to the *meso* N atom (dithio keto form), to the S atom of the thiourea moiety and to the S atom of the thiobenzoyl group (thio keto–thiol forms),

IR spectroscopy in the solid state showed the existence of the dithioketo form (ν_{NH} 3110, $\nu_{\text{C=S}}$ 1250, $\nu_{\text{N-C=S}}$ 1530 cm^{-1}).

If the molecule is planar, four limiting configurations Z, Z', Z, E', E, E' and E, Z' are possible (Walter & Krohn, 1973) which differ from each other by formal rotations around the C(S)-N(meso) and C(X)-N(meso) bonds. Because the molecules are often not planar the torsion angles $\omega_1[\text{C(X)-N(meso)-C(S)-S}]$ and $\omega_2[\text{C(S)-N(meso)-C(X)-X}]$ are introduced for a more accurate description of the conformation. The four limiting configurations have values of ω_1 and ω_2 of 0 or 180°: Z, Z' ($\omega_1 = 0, \omega_2 = 0^\circ$); Z, E' ($\omega_1 = 0, \omega_2 = 180^\circ$); E, E' ($\omega_1 = 180, \omega_2 = 180^\circ$); E, Z' ($\omega_1 = 180, \omega_2 = 0^\circ$). With these torsion angles it is possible to describe the conformation of the molecules by comparing the experimental angles with those defined for the limiting configuration. The angle ω_1 is always chosen positive. The sign of ω_2 also becomes positive when the atoms S and X lie on different sides of the plane defined by the atoms C(S), N(meso) and C(X). ω_2 becomes negative if S and X lie on the same side of the plane C(S), N(meso), C(X).

Experimental. The compound was synthesized as described by Beyer, Hartung & Widera (1984). Recrystallization from 2-propanol yielded yellow crystals; m.p. 366–369 K. Two polymorphic modifications were obtained. (I) Crystal dimensions 0.4 × 0.4 × 0.2 mm. Syntex $P2_1$ diffractometer, graphite-monochromated $\text{Mo K}\alpha$, $\omega/2\theta$ scans; lattice parameters from measurement of 15 reflections, systematic absences $h0l, h+l=2n+1, 0k0, k=2n+1$, Lp corrections, no absorption corrections, $0 < \theta \leq 28.1^\circ$, $h-10 \rightarrow 9, k0 \rightarrow 10, l0 \rightarrow 25$, two standard reflections, <1% intensity variation, 3235 unique reflections, 1286 with $F_o > 3\sigma(F_o)$. (II) Crystal dimensions 0.3 × 0.2 × 0.2 mm. Hilger & Watts diffractometer, graphite-monochromated $\text{Mo K}\alpha$, $\theta/2\theta$ scans, lattice parameters from 12 reflections, systematic absences $0k0, k=2n+1$, Lp corrections, no absorption corrections, $0 < \theta \leq 29^\circ$, $h-10 \rightarrow 9, k0 \rightarrow 15, l0 \rightarrow 14$, three standard reflections, no intensity variation, 2055 unique reflections, 1727 with $F_o > 3\sigma(F_o)$; structures solved by direct methods, full-matrix least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$, unit weights, anisotropic for all non-H atoms, all H atoms obtained from a difference map, but not refined, except for H(1) (I) and H(11), H(12) (II) calculated H-atom positions were used, thermal parameters set at U_{iso} of the attached atom. Final $R=0.056$ (I) and $R=0.045$ (II), final difference Fourier maps showed $\Delta\rho_{\text{max}} 0.26 \text{ e } \text{\AA}^{-3}$ (I) and $0.28 \text{ e } \text{\AA}^{-3}$ (II) and $\Delta\rho_{\text{min}} -0.29 \text{ e } \text{\AA}^{-3}$ (I and II); maximum Δ/σ in final cycle 0.37 (I) and 1.83 [for U_{12} of C(51)] (II); all other Δ/σ ratios < 1.0; average Δ/σ 0.08 (I) and 0.16 (II); atomic scattering factors from *SHELX76* (Sheldrick, 1976).

Views of the molecules and molecular packing diagrams drawn with *ORTEP* (Johnson, 1965) and *PLUTO* (Motherwell & Clegg, 1978), respectively.

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* The atom numbering is shown in Fig. 1. The molecular structures of the modifications (I) and (II) are illustrated in Fig. 2, bond lengths are given in Table 2.

* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, bond angles, hydrogen-bonded distances and stereoscopic views of the molecules have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43267 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

	x	y	z	U_{eq}^*
(I)				
S(1)	2721 (2)	5806 (2)	5339 (1)	62 (1)
S(2)	3611 (2)	1505 (3)	6518 (1)	78 (1)
N(1)	5148 (6)	3885 (6)	5944 (2)	47 (3)
N(2)	3805 (6)	5668 (6)	6589 (2)	54 (3)
C(1)	5041 (7)	2235 (7)	6124 (2)	46 (3)
C(2)	3916 (7)	5099 (7)	6002 (3)	48 (4)
C(3)	2450 (8)	6793 (9)	6697 (3)	63 (4)
C(4)	879 (9)	5838 (11)	6728 (3)	86 (5)
C(5)	5051 (9)	5258 (9)	7157 (3)	66 (4)
C(6)	6377 (9)	6620 (12)	7261 (3)	98 (6)
C(7)	6373 (7)	1138 (8)	5945 (2)	50 (4)
C(8)	7981 (8)	1747 (9)	5970 (3)	64 (4)
C(9)	9202 (9)	678 (14)	5804 (4)	85 (6)
C(10)	8803 (13)	-950 (13)	5604 (4)	96 (7)
C(11)	7220 (12)	-1535 (10)	5578 (3)	84 (5)
C(12)	5997 (8)	-526 (8)	5752 (3)	62 (4)
(II)				
S(11)	7065 (2)	4744	3606 (2)	61 (1)
S(21)	4858 (3)	3458 (3)	612 (2)	84 (2)
N(11)	4064 (7)	4450 (5)	2277 (5)	53 (4)
N(21)	5549 (9)	5659 (6)	1699 (6)	74 (5)
C(11)	3728 (9)	3722 (6)	1523 (6)	52 (5)
C(21)	5545 (9)	4992 (6)	2447 (6)	54 (5)
C(31)	4081 (12)	5920 (8)	782 (7)	79 (7)
C(41)	3012 (13)	6579 (10)	1267 (10)	107 (9)
C(51)	7001 (14)	6329 (11)	1845 (10)	113 (10)
C(61)	8109 (16)	5903 (11)	1263 (13)	154 (13)
C(71)	2199 (9)	3199 (6)	1571 (6)	50 (5)
C(81)	854 (10)	3656 (6)	1854 (7)	60 (5)
C(91)	-544 (11)	3133 (9)	1920 (8)	78 (7)
C(101)	-628 (12)	2210 (8)	1686 (8)	76 (7)
C(111)	682 (12)	1766 (7)	1408 (8)	67 (6)
C(121)	2062 (10)	2246 (6)	1338 (7)	58 (6)
S(12)	7917 (3)	30 (2)	5656 (2)	72 (1)
S(22)	5714 (3)	514 (3)	2366 (2)	97 (2)
N(12)	4912 (7)	398 (5)	4355 (5)	52 (4)
N(22)	6576 (9)	1692 (6)	4972 (7)	72 (5)
C(12)	4514 (9)	229 (6)	3212 (6)	58 (5)
C(22)	6466 (10)	775 (7)	4966 (7)	62 (6)
C(32)	5229 (13)	2332 (7)	4415 (11)	89 (8)
C(42)	4156 (15)	2565 (10)	5219 (16)	142 (12)
C(52)	8133 (12)	2172 (8)	5630 (10)	89 (8)
C(62)	9298 (13)	2300 (9)	4903 (12)	115 (10)
C(72)	2855 (9)	-222 (7)	2783 (6)	56 (5)
C(82)	1585 (9)	-78 (7)	3341 (6)	61 (5)
C(92)	19 (11)	-452 (8)	2903 (9)	80 (7)
C(102)	-307 (13)	-964 (8)	1893 (11)	87 (8)
C(112)	963 (17)	-1115 (8)	1329 (9)	91 (8)
C(122)	2513 (13)	-738 (8)	1760 (8)	83 (7)

* $U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

(I) The S(1), C(2), N(1), C(1), S(2) moiety deviates extensively from planarity. The conformation of the molecule is halfway between E, Z' and Z, Z' [$\omega_1 = 108.5(2)$, $\omega_2 = 8.7(2)^\circ$]. A similar conformation has also been found in 1-(4-chlorophenyl)-3-(2,6-difluorobenzoyl)urea (Cruse, 1978) and in *N*-acylselenoureas (Hope, 1965; Perez-Rodriguez & Lopez-Castro, 1969).

The H atom H(1) is bonded to the *meso* N atom N(1). Thus the NH-tautomeric dithioketo form which was proposed from the IR-spectroscopic investigations is confirmed.

The thiourea moiety S(1), C(2), N(1), N(2) and the C(2), N(2), C(3), C(5) moiety are planar.

The two C—S bond lengths differ significantly. Both have values in the range between single (1.81 Å) and double bonds (1.61 Å; Pauling, 1968), but the C(1)—S(2) bond is 0.04 Å shorter than the S(1)—C(2) bond, due to the +*M* effect of the diethylamide group and the -*I* effect of the phenyl ring.

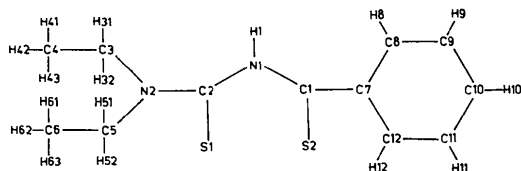


Fig. 1. The atom-numbering scheme used for (I). For modification (II), the atom numbering is derived by adding the digit '1' in the case of molecule 1, and the digit '2' in the case of molecule 2.

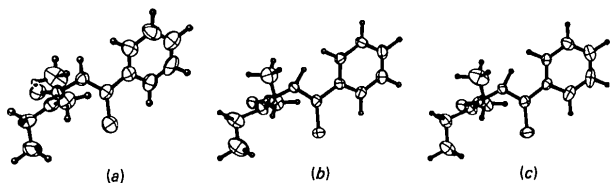


Fig. 2. Views of the molecules; (a) (I), (b) (II) molecule 1 and (c) (II) molecule 2.

Table 2. Bond lengths (Å)

(I)		(II) (molecules 1 and 2)			
S(1)—C(2)	1.675 (6)	S(11)—C(21)	1.677 (7)	S(12)—C(22)	1.673 (8)
S(2)—C(1)	1.639 (6)	S(21)—C(11)	1.640 (8)	S(22)—C(12)	1.629 (8)
N(1)—C(1)	1.365 (7)	N(11)—C(11)	1.361 (9)	N(12)—C(12)	1.347 (9)
N(1)—C(2)	1.408 (7)	N(11)—C(21)	1.428 (9)	N(12)—C(22)	1.431 (10)
N(2)—C(2)	1.335 (7)	N(21)—C(21)	1.306 (10)	N(22)—C(22)	1.314 (10)
N(2)—C(3)	1.464 (7)	N(21)—C(31)	1.481 (10)	N(22)—C(32)	1.477 (12)
N(2)—C(5)	1.482 (7)	N(21)—C(51)	1.520 (14)	N(22)—C(52)	1.511 (12)
C(1)—C(7)	1.483 (8)	C(11)—C(71)	1.488 (10)	C(12)—C(72)	1.499 (11)
C(3)—C(4)	1.496 (9)	C(31)—C(41)	1.503 (14)	C(32)—C(42)	1.494 (16)
C(5)—C(6)	1.517 (10)	C(51)—C(61)	1.416 (17)	C(52)—C(62)	1.457 (15)
C(7)—C(8)	1.390 (8)	C(71)—C(81)	1.404 (10)	C(72)—C(82)	1.390 (10)
C(8)—C(9)	1.391 (10)	C(81)—C(91)	1.399 (12)	C(82)—C(92)	1.391 (11)
C(9)—C(10)	1.377 (12)	C(91)—C(101)	1.347 (13)	C(92)—C(102)	1.380 (14)
C(10)—C(11)	1.364 (11)	C(101)—C(111)	1.368 (13)	C(102)—C(112)	1.397 (15)
C(11)—C(12)	1.371 (9)	C(111)—C(121)	1.356 (12)	C(112)—C(122)	1.380 (14)
C(7)—C(12)	1.396 (8)	C(71)—C(121)	1.390 (11)	C(72)—C(122)	1.396 (12)

The C—N bonds involving C(1) and C(2) (Fig. 1) also have lengths which lie between single- (1.47 Å) and double-bond (1.29 Å) values.

The phenyl ring is tilted by $37.5(2)^\circ$ with respect to the plane defined by the atoms N(1), C(1), S(2).

Fig. 3(a) shows the crystal structure of (I). Pairs of molecules related by centres of symmetry are connected by N—H...S interactions forming dimers, with S(1)...H(1) 2.41 Å.

(II) The symmetry-independent molecules are identical with respect to their bond distances and angles and both deviate considerably from planarity.

The molecules exist in the NH-tautomeric dithioketo form.

As in modification (I) the conformation of the molecules is halfway between E, Z' and Z, Z' . The torsion angles ω_1 and ω_2 have values of $100.1(2)$ and $7.6(2)^\circ$ (molecule 1) and $97.6(2)$ and $5.0(2)^\circ$ (molecule 2).

The molecules in modification (II) do not show any major structural differences from those in (I). Table 3 gives a comparison of selected bond distances and angles of the free ligand in both modifications with those of the nickel chelate, which also exists in two polymorphic modifications (Sieler *et al.*, 1985).

The thiourea moieties in the two molecules S(11), C(21), N(11), N(21) and S(12), C(22), N(12), N(22) as well as the C(21), N(21), C(31), C(51) unit are planar, whereas in C(22), N(22), C(32), C(52) unit deviations from planarity of up to 6σ occur.

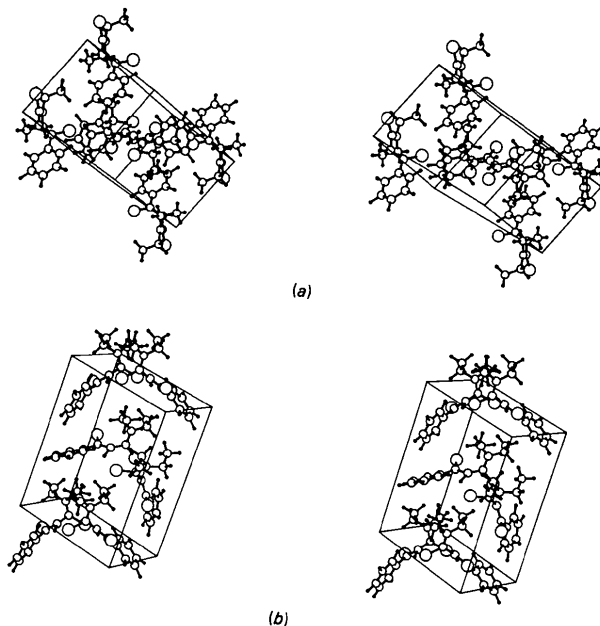


Fig. 3. Stereoscopic views of the molecular packing; (a) (I), (b) (II). (I) The *x* axis points towards the viewer, *y* down the page and *z* across. (II) The *z* axis points towards the viewer, *x* down the page and *y* across.

Table 3. Comparison of selected bond lengths (Å) and angles (°) of the ligand and the corresponding nickel chelate, $[\text{Ni}(\text{C}_{12}\text{H}_{15}\text{N}_2\text{S}_2)_2]$ (Sieler *et al.*, 1985)

	$\text{C}_{12}\text{H}_{16}\text{N}_2\text{S}_2$		$[\text{Ni}(\text{C}_{12}\text{H}_{15}\text{N}_2\text{S}_2)_2]$	
	(I)	(II)*	(I)	(II)
S(1)—C(2)	1.675 (6)	1.675 (8)	1.75 (2)	1.73 (1)
S(2)—C(1)	1.639 (6)	1.635 (8)	1.71 (2)	1.71 (1)
N(1)—C(2)	1.408 (7)	1.430 (10)	1.29 (3)	1.34 (2)
N(1)—C(1)	1.365 (7)	1.359 (9)	1.27 (3)	1.29 (2)
N(2)—C(2)	1.335 (7)	1.310 (10)	1.41 (3)	1.35 (2)
S(1)—C(2)—N(1)	119.0 (4)	117.8 (7)	129 (2)	130 (1)
C(2)—N(1)—C(1)	123.3 (5)	123.5 (6)	127 (2)	127 (1)
S(2)—C(1)—N(1)	123.9 (4)	123.3 (6)	134 (2)	132 (1)

* Mean value of the two independent molecules.

As in (I), C(1)—S(2) is about 0.04 Å shorter than C(2)—S(1) which corresponds to distances in thioureas (Kunchur & Truter, 1958; Elcombe & Taylor, 1968; Dias & Truter, 1964) and thioamides (Truter, 1960; Walter, Harto & Voss, 1976).

The C(1)—N(1), C(2)—N(1) and C(2)—N(2) bonds are again intermediate between single and double bonds. C(5)—C(6) (mean value 1.44 Å) is slightly shorter and N(2)—C(5) (mean value 1.52 Å) is longer than the expected values of 1.54 and 1.47 Å (Pauling, 1968). Deviations of this kind are already known from *N*-benzoylthiourea chelates (Fitzl *et al.*, 1977; Knuutila *et al.*, 1982).

The phenyl rings in the two molecules C(71)—C(121) and C(72)—C(122) are tilted by 32.3(3) and 26.8(3)° to the N(11), C(11), S(21) and N(12), C(12), S(22) planes, respectively.

Fig. 3(b) shows the crystal structure of modification (II). Pairs of symmetrically independent molecules are

linked by N—H...S interactions forming dimers. The distances H(12)...S(11) ($1-x, \frac{1}{2}+y, 1-z$) and H(11)...S(12) ($1-x, y-\frac{1}{2}, 1-z$) are 2.42 and 2.52 Å respectively.

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Structure of 2,4,5,7-Tetramethyl-9,10-phenanthroquinone

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Abstract. $\text{C}_{18}\text{H}_{16}\text{O}_2$, $M_r = 264.32$, monoclinic, $I2/a$, $a = 19.389$ (3), $b = 8.708$ (1), $c = 17.554$ (3) Å, $\beta = 112.11$ (1)°, $V = 2745.8$ (8) Å³, $Z = 8$, $D_x = 1.279$ g cm⁻³, $\text{Mo } K\alpha$, $\lambda = 0.7093$ Å, $\mu = 0.766$ cm⁻¹,

$F(000) = 1120$, $T = 298$ K, $R = 0.046$ for 1889 reflections with $I > 2.5\sigma(I)$. The repulsion between the two methyl groups at C(4) and C(5) and the electrostatic repulsion from the orthoquinone functionality cause a severe deviation from planarity for the phenanthroquinone skeleton. The torsional angle C(4)—C(12)—

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