

Table 3. *Effects of anomalous dispersion of Mo K α radiation measured on selected Friedel pairs*

$\Delta F^o = |F_h^o| - |F_{-h}^o|$ is the difference between the observed structure factors for the Friedel pair; $\Delta F^c = |F_h^c| - |F_{-h}^c|$ is the corresponding difference between the structure factors calculated from the coordinates of Table 1. The terms of anomalous dispersion used for the C atom and Mo radiation are $f' = 0.002$, $f'' = 0.002$.

<i>h</i>	<i>k</i>	<i>l</i>	θ	ΔF^o	ΔF^c
4	-4	9	14.2	10.6 (7)	9.2
-6	5	-6	16.3	-7.4 (8)	-6.8
2	-1	6	8.9	9.7 (4)	9.9
-1	4	6	13.9	4.3 (8)	3.9
-2	-1	-5	9.6	-9.7 (4)	-12.9
2	0	5	8.5	6.4 (4)	6.2
-3	0	-5	10.2	9.0 (8)	8.5
-2	-3	4	10.3	-7.2 (6)	-7.1
2	2	-4	9.2	11.1 (4)	9.3
2	0	-4	8.4	4.8 (6)	6.9
1	-1	-4	7.3	-13.8 (4)	-14.7
2	-3	-4	10.7	-4.3 (7)	-7.2
0	4	3	10.6	-9.4 (8)	-12.2
-1	-3	-3	9.5	-14.9 (6)	-13.1
2	-6	3	11.2	4.7 (6)	9.9
3	3	-2	11.8	6.3 (4)	5.2
-2	0	2	6.4	-5.7 (3)	-4.7
2	-4	-2	10.1	10.7 (6)	10.9
1	-3	1	6.8	18.5 (4)	17.0
4	5	-1	17.2	5.1 (11)	4.8

assumed *R* chirality at C(3). This result, moreover, agrees with that obtained by the chiral exciton coupling method (Adinolfi *et al.*, 1988).

Finally, it is worth noting that, apart from the formal difference in the chirality symbol (*R* instead of *S*), the title compound is stereochemically congruent, in conformation and configuration of the

dihydro- γ -pyrone ring, with Eucomol, a natural compound with the same heterocycle system, in which a hydroxyl group replaces the H atom at C(3) (Weber, Heller & Tamm, 1977).

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Structure of *N*,2-Dibenzoylthioacetamide

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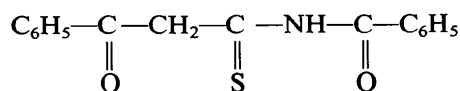
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Abstract. C₁₆H₁₃NO₂S, *M_r* = 283.35, monoclinic, *C*2/*c*, *a* = 28.40 (2), *b* = 3.927 (3), *c* = 24.25 (2) Å, β = 90.12 (6)°, *V* = 2705 (3) Å³, *Z* = 8, *D_x* = 1.39 g cm⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71073 Å, μ = 2.28 cm⁻¹,

F(000) = 1184, *T* = 143 K, *R* = 0.054 for 2407 observed reflections. The molecule exists as a keto-thio keto-enol tautomer with enolization of the O atom in the 2-acylthioacetamide fragment. The con-

formation of the molecule is best described as *Z, Z', E', Z'''*. This conformation is stabilized by intramolecular O—H...S and C—H...O hydrogen bonds. Hydrogen bonds of the type N—H...S link the molecules into centrosymmetric pairs. The compound combines the structural properties of the 2-chalcogenoacyl-chalcogenoacetamides and the *N*-chalcogenoacyl-chalcogenocarboxamides.

Introduction. *N*-Chalcogenoacyl-chalcogenocarboxamides and *N*-chalcogenoacyl-chalcogenoureas are aza analogues of the well known dichalcogeno- β -diketones and the 2-chalcogenoacyl-chalcogenoacetamides, respectively. These groups of compounds show a different tautomeric behaviour. Whereas dichalcogeno- β -diketones such as dibenzoylmethane (Williams, 1966; Hollander, Templeton & Zalkin, 1973; Jones, 1976) and benzoyl(thiobenzoyl)methane (Power, Turner & Moore, 1974, 1976; Richter, Sieler, Kaiser & Uhlemann, 1976) and 2-chalcogenoacyl-chalcogenoacetamides (Kornis, Marks & Chidester, 1980; Iliopoulos, Fallon & Murray, 1986) exist in the solid state in the enol form, *N*-chalcogenoacyl-chalcogenocarboxamides such as dibenzamide (Mizrahi & Niven, 1983; Richter, 1984) and *N*-thiobenzoylbenzamide (Richter, Beyer, Kaiser, Lindqvist, Ljungström & Golič, 1983) and *N*-chalcogenoacyl-chalcogenoureas such as *N*-benzoyl-*N',N'*-diethylurea (Richter, 1985), *N*-benzoyl-*N',N'*-dimethylthiourea (Richter, 1985) and *N,N*-diethyl-*N'*-thiobenzoylthiourea (Braun, Richter, Sieler, Beyer, Lindqvist, Yanovsky & Struchkov, 1987) show the keto form in the solid state. An interesting test of this different tautomeric behaviour is offered by the structure determination of *N*,2-dibenzoylthioacetamide



which contains the two respective structural fragments side-by-side in the same molecule.

Further structural features of interest are the bond lengths and angles, the conformation of the molecule, which can be described by the torsion angles ω and the *Z,E* notation (Braun *et al.*, 1987), and the intra- and intermolecular hydrogen bonds.

Experimental. The compound was synthesized as described by Hünig, Hübner & Benzing (1962). Recrystallization from acetonitrile yielded yellow needles; m.p. 411 K. Crystal dimensions $0.1 \times 0.1 \times 0.3$ mm. Syntex *P2*₁ diffractometer with modified LT-1 low-temperature attachment, 143 K, graphite-monochromated Mo *K* α , ω scan, lattice parameters from measurement of 15 reflections, $9 \leq \theta \leq 27^\circ$, systematic absences *hkl*, $h + k = 2n + 1$, *h0l*, $l =$

Table 1. Atomic coordinates ($\times 10^5$, for H $\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$, for H $\text{\AA}^2 \times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
S(1)	52605 (2)	21775 (18)	43301 (2)	212 (2)
O(1)	65419 (6)	33947 (55)	54394 (7)	300 (6)
O(2)	59126 (6)	-10683 (55)	35778 (7)	266 (6)
N(1)	57562 (6)	29487 (58)	52280 (7)	185 (6)
C(1)	57631 (8)	16870 (65)	46908 (9)	166 (7)
C(2)	61336 (8)	36298 (67)	55813 (9)	194 (7)
C(3)	62367 (8)	-12390 (65)	39744 (9)	177 (7)
C(4)	61725 (8)	306 (68)	44971 (9)	179 (7)
C(5)	66786 (8)	-28876 (68)	38003 (10)	196 (7)
C(6)	70319 (9)	-37590 (75)	41755 (10)	261 (8)
C(7)	74444 (10)	-53095 (83)	39961 (13)	307 (9)
C(8)	75092 (10)	-59780 (78)	34412 (12)	299 (9)
C(9)	71608 (9)	-51115 (78)	30645 (11)	279 (8)
C(10)	67475 (9)	-36073 (70)	32399 (10)	236 (8)
C(11)	60000 (8)	47414 (67)	61495 (9)	186 (7)
C(12)	55681 (8)	39722 (72)	63841 (10)	222 (7)
C(13)	54783 (10)	49664 (90)	69245 (11)	335 (9)
C(14)	58174 (11)	66973 (87)	72257 (10)	357 (10)
C(15)	62454 (10)	74516 (80)	69929 (10)	315 (8)
C(16)	63399 (9)	64871 (70)	64551 (10)	241 (8)
H(1)	5501 (10)	3805 (75)	5320 (11)	32 (8)†
H(2)	5654 (11)	-228 (84)	3731 (12)	35 (9)†
H(3)	6446 (9)	35 (76)	4734 (11)	29 (8)†

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

$$\dagger U_{\text{iso}}.$$

$2n + 1$, L_p corrections, no absorption corrections, $0 < \theta \leq 29^\circ$, $h - 38 \rightarrow 38$, $k 0 \rightarrow 5$, $l 0 \rightarrow 32$, three standard reflections, 2% intensity variation, 3613 unique reflections, 2407 with $F_o \geq 3.92\sigma(F_o)$; structure solved by direct methods, block-matrix least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$, weights $w = 1/[\sigma_F^2 + (c|F_o|)^2]$ with refined $c = 0.023$, anisotropic for all non-H atoms, all H atoms obtained from a difference map, isotropic for all H atoms, 234 parameters. Final $R = 0.054$, $wR = 0.048$ for 2407 observed reflections, final difference Fourier map showed $\Delta\rho_{\text{max}} 0.35$ and $\Delta\rho_{\text{min}} -0.38 \text{ e}\text{\AA}^{-3}$, maximum Δ/σ in final cycle 0.031, average $\Delta/\sigma 0.007$, atomic scattering factors from *SHELXTL* (Sheldrick, 1981). View of the molecule and molecular packing diagram drawn with *ORTEP* (Johnson, 1976) and *PLUTO* (Motherwell, 1972), respectively.

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* The atomic numbering and the molecular structure are shown in Fig. 1. Bond lengths and angles are given in Table 2.

The molecular structure confirms the tautomeric behaviour of the 2-chalcogenoacyl-chalcogenoacetamides and the *N*-chalcogenoacyl-chalcogeno-

* Lists of structure amplitudes, anisotropic thermal parameters and additional H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51949 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°)

S(1)—C(1)	1.684 (2)	C(5)—C(6)	1.396 (3)
O(1)—C(2)	1.214 (3)	C(5)—C(10)	1.402 (3)
O(2)—C(3)	1.331 (3)	C(6)—C(7)	1.391 (4)
O(2)—H(2)	0.89 (3)	C(7)—C(8)	1.384 (4)
N(1)—C(1)	1.394 (3)	C(8)—C(9)	1.388 (4)
N(1)—C(2)	1.397 (3)	C(9)—C(10)	1.382 (4)
N(1)—H(1)	0.83 (3)	C(11)—C(12)	1.386 (3)
C(1)—C(4)	1.414 (3)	C(11)—C(16)	1.396 (3)
C(2)—C(11)	1.495 (3)	C(12)—C(13)	1.392 (4)
C(3)—C(4)	1.375 (3)	C(13)—C(14)	1.386 (4)
C(3)—C(5)	1.475 (3)	C(14)—C(15)	1.374 (4)
C(4)—H(3)	0.97 (3)	C(15)—C(16)	1.385 (4)
C(3)—O(2)—H(2)	107 (2)	C(3)—C(5)—C(6)	122.1 (2)
C(1)—N(1)—C(2)	129.0 (2)	C(3)—C(5)—C(10)	119.1 (2)
C(1)—N(1)—H(1)	114 (2)	C(6)—C(5)—C(10)	118.7 (2)
C(2)—N(1)—H(1)	115 (2)	C(5)—C(6)—C(7)	120.5 (2)
S(1)—C(1)—N(1)	115.6 (2)	C(6)—C(7)—C(8)	120.1 (3)
S(1)—C(1)—C(4)	125.2 (2)	C(7)—C(8)—C(9)	119.8 (3)
N(1)—C(1)—C(4)	119.2 (2)	C(8)—C(9)—C(10)	120.5 (3)
O(1)—C(2)—N(1)	123.0 (2)	C(5)—C(10)—C(9)	120.3 (2)
O(1)—C(2)—C(11)	121.9 (2)	C(2)—C(11)—C(12)	122.7 (2)
N(1)—C(2)—C(11)	115.2 (2)	C(2)—C(11)—C(16)	117.1 (2)
O(2)—C(3)—C(4)	123.7 (2)	C(12)—C(11)—C(16)	120.0 (2)
O(2)—C(3)—C(5)	113.8 (2)	C(11)—C(12)—C(13)	119.3 (2)
C(4)—C(3)—C(5)	122.5 (2)	C(12)—C(13)—C(14)	120.4 (3)
C(1)—C(4)—C(3)	125.7 (2)	C(13)—C(14)—C(15)	120.2 (2)
C(1)—C(4)—H(3)	118 (2)	C(14)—C(15)—C(16)	120.1 (3)
C(3)—C(4)—H(3)	116 (2)	C(11)—C(16)—C(15)	119.9 (2)

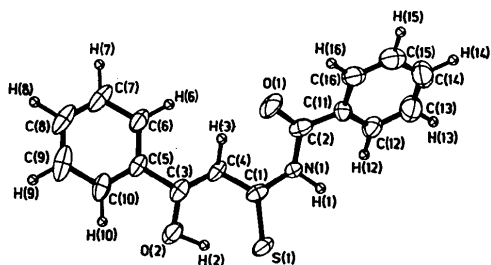


Fig. 1. View of the molecule with atomic numbering.

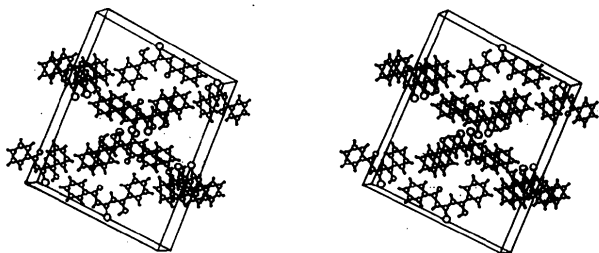


Fig. 2. Stereoscopic view of the molecular packing. The y axis points towards the viewer, x down the page and z across.

Table 3. Comparison of selected bond lengths (Å) and angles (°) of *N,2-dibenzoylthioacetamide* C₁₆H₁₃NO₂S, *benzoyl(thiobenzoyl)methane* C₁₅H₁₂OS, *N-benzoyl-N',N'-dimethylthiourea* C₁₀H₁₂N₂OS and *N-thiobenzoylbenzamide* C₁₄H₁₁NOS

	C ₁₆ H ₁₃ - NO ₂ S	C ₁₅ H ₁₂ - OS	C ₁₀ H ₁₂ - N ₂ OS*	C ₁₄ H ₁₁ - NOS*†
S(1)—C(1)	1.684 (2)	1.685 (2)	1.678 (3)	1.637 (4)
O(1)—C(2)	1.214 (3)		1.216 (4)	1.228 (5)
N(1)—C(1)	1.394 (3)		1.406 (4)	1.383 (5)
N(1)—C(2)	1.397 (3)		1.387 (4)	1.405 (5)
O(2)—C(3)	1.331 (3)	1.321 (3)		
C(1)—C(4)	1.414 (3)	1.406 (3)		
C(3)—C(4)	1.375 (3)	1.380 (3)		
S(1)—C(1)—N(1)	115.6 (2)		119.3 (2)	124.3 (3)
O(1)—C(2)—N(1)	123.0 (2)		122.8 (3)	123.3 (4)
C(1)—N(1)—C(2)	129.0 (2)		122.5 (2)	125.8 (4)
S(1)—C(1)—C(4)	125.2 (2)	124.5 (2)		
O(2)—C(3)—C(4)	123.7 (2)	122.4 (2)		
C(1)—C(4)—C(3)	125.7 (2)	126.3 (2)		
C(3)—O(2)—H(2)	107 (2)	107 (2)		
S(1)—H(2)—O(2)	156 (3)	157 (3)		

* Mean values of two independent molecules.

† Monoclinic modification.

164.0, $\omega[C(1)N(1)C(2)O(1)] = -5.3^\circ$. The O(2)C(3)C(4)C(1)S(1)N(1)C(2)O(1) moiety deviates clearly from planarity. The phenyl rings C(5)—C(10) and C(11)—C(16) are tilted by 10 and 21° against the planes through O(2)C(3)C(4) and O(1)C(2)N(1), respectively.

The observed conformation is stabilized by an intramolecular hydrogen bond between O(2) and S(1) with distances O(2)⋯S(1) 2.898 (2) Å and H(2)⋯S(1) 2.06 (3) Å. The angle O(2)—H(2)—S(1) is 156 (3)°. A further stabilizing intramolecular contact exists between H(3) and O(1) [2.18 (3) Å]. The distance C(4)⋯O(1) is 2.839 (3) Å and the angle C(4)—H(3)—O(1) 125 (2)°.

Both C—N bonds in the *N*-thioacylbenzamide fragment, C(1)—N(1) and C(2)—N(1), have the same lengths which lie between single- (1.47 Å) and double-bond (1.27 Å) values. In contrast to this equality the C—C bonds in the 2-acylthioacetamide fragment C(1)—C(4) and C(3)—C(4) have a small but significant difference which shows a partial localization of a double bond between C(3) and C(4) in accordance with the direction of enolization. The bond C(2)—O(1) is nearly a double bond, but the bonds C(1)—S(1) and C(3)—O(2) are significantly longer than double bonds.

The crystal structure is stabilized by intermolecular NH⋯S hydrogen bonds between centrosymmetrically arranged molecules with distances N(1)⋯S(1)' 3.628 (2) and H(1)⋯S(1)' 2.81 (3) Å and an angle N(1)—H(1)—S(1)' 169 (3)° (Fig. 2).

N,2-Dibenzoylthioacetamide combines the characteristic structural properties of a dichalcogeno-β-diketone/2-chalcogenoacyl-chalcogenoacetamide

carbonamides, respectively. In the 2-acylthioacetamide fragment the O atom is enolized, whereas the *N*-thioacylbenzamide fragment shows the keto form.

The conformation of the molecule is best described as *Z,Z',E',Z''* { $\omega[O(2)C(3)C(4)C(1)] = 0.3$, $\omega[C(3)C(4)C(1)S(1)] = -4.4$, $\omega[S(1)C(1)N(1)C(2)] =$

and its aza analogue. Table 3 shows some of its bond lengths and angles in comparison with the appropriate parameters in benzoyl(thiobenzoyl)methane (Richter *et al.*, 1976), *N*-benzoyl-*N,N'*-dimethylthiourea (Richter, 1985) and *N*-thiobenzoylbenzamide (Richter *et al.*, 1983). Whereas the agreement between the 2-acylthioacetamide part of the molecule with benzoyl(thiobenzoyl)methane is excellent, there are some marked differences especially regarding the angles between the *N*-thioacylbenzamide part of the molecule and the structures of *N*-thiobenzoylbenzamide and *N*-benzoyl-*N,N'*-dimethylthiourea. The main reason should be the considerably distorted conformation of this part $\{\omega_1[\text{S}(1)\text{C}(1)\text{N}(1)\text{C}(2)] = 164.0$ and $\omega_2[\text{C}(1)\text{N}(1)\text{C}(2)\text{O}(1)] = -5.3^\circ\}$ in comparison with *N*-thiobenzoylbenzamide ($\omega_1 = 22.3$ and $\omega_2 = 18.2^\circ$) and *N*-benzoyl-*N,N'*-dimethylthiourea ($\omega_1 = 120.8$ and $\omega_2 = 2.4^\circ$).

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Structures of Polyfluoroaromatic Compounds. XI.* Structures of Two Fluorinated Diphenyl Sulfides

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Abstract. (I) Octafluoro-4,4'-bis(trifluoromethyl)diphenyl sulfide, $\text{C}_{14}\text{F}_{14}\text{S}$, $M_r = 466.2$, monoclinic, $P2_1/c$, $a = 14.37$ (2), $b = 5.59$ (1), $c = 19.57$ (2) Å, $\beta = 96.38$ (5)°, $V = 1562$ Å³, $Z = 4$, $D_x = 1.98$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.72$ cm⁻¹, $F(000) = 904$, $T = 293$ K, $R = 0.042$ for 1060 observed reflections. (II) Octafluoro-4,4'-dinitrodiphenyl sulfide, $\text{C}_{12}\text{F}_8\text{N}_2\text{O}_4\text{S}$, $M_r = 420.2$, triclinic, $P\bar{1}$, $a = 7.42$ (1), $b = 10.11$ (1), $c = 10.71$ (1) Å, $\alpha = 113.25$ (5), $\beta = 82.40$ (7), $\gamma = 83.05$ (6)°, $V = 718$ Å³, $Z = 2$, $D_x = 1.94$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.48$ cm⁻¹, $F(000) = 412$, $T = 293$ K, $R = 0.042$ for 1981 observed reflections. Both compounds adopt the twist conformation with the rings inclined at 46.9 (6) and 49.8 (6)° in (I), and 53.4 (2) and 61.0 (2)° in (II)

to the central C—S—C plane. The bond angles at S are 102.0 (3) and 99.7 (1)° in (I) and (II), respectively; the C—S bond lengths average 1.763 (6) and 1.770 (3) Å.

Introduction. In an earlier paper of this series (Goodhand & Hamor, 1979), the structure of hexafluoro-4,4'-dinitro-3*H*,3'*H*-diphenyl sulfide (henceforth DTFNPS) was described and compared with hydrocarbon analogues. It was noted that the C—S—C angle was some 4° smaller than in the unfluorinated analogues. We now report the structures of octafluoro-4,4'-bis(trifluoromethyl)diphenyl sulfide (I) and octafluoro-4,4'-dinitrodiphenyl sulfide (II) (Coe, Milner, Tatlow & Wragg, 1972) as part of a continuing study of highly fluorinated organic molecules.

* Part X: Goodhand & Hamor (1982).