

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

BY NEIL GOODHAND AND THOMAS A. HAMOR

Department of Chemistry, University of Birmingham, Birmingham B15 2TT, England

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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).

Experimental. Both compounds were crystallized from ethanol. Approximate cell dimensions were obtained from oscillation, Weissenberg and precession photographs. Refined values and intensity data were measured on a Stoe STADI-2 diffractometer operating in the ω -scan mode. Crystal size: $0.7 \times 0.3 \times 0.5$ (I), $0.8 \times 0.8 \times 0.2$ mm (II). For the upper layers of reflections, $h21-h51$ for (I) and $2kl-8kl$ for (II), a variable-scan-range technique was used, ω being calculated from $A + B \sin \mu / \tan \theta'$ where μ is the equi-inclination angle and $2\theta'$ the azimuth angle. Values of A and B were 1.2 and 0.5 (I) and 1.2 and 0.9° (II). For the lower layers the scan ranges were 1.6 and 1.4° . Backgrounds were measured for 30 s at each end of the scan; scan speed $0.01^\circ \text{ s}^{-1}$; data collection range $4-25$ (I), $4-30^\circ$ (II); 2807 (I), 3382 (II) reflections were scanned of which 1060 (I), 1981 (II) were considered observed [$I > 2.5\sigma(I)$] and used in the analysis. Layer scale factors, which were all close to unity, were calculated from four zero-layer reflections that were remeasured after each layer of data collection. Both structures were solved by direct methods with *SHELX76* (Sheldrick, 1976) and refined by least squares on F values with anisotropic thermal parameters for all atoms. For the CF_3 groups in compound (I) two sites could be found for each F atom corresponding to two orientations of these groups. The coordinates of the disordered F atoms were not refined. Refinement of site occupation factors showed that in one case the two orientations occurred in the ratio 2:1, whereas in the other, the ratio was approximately 1:1 (see Table 1). For both structures least-squares refinement was terminated when all shift/e.s.d. ratios were < 0.1 . Final R and wR are 0.042 and 0.041 (I), and 0.042 and 0.045 (II), $w = 1/\sigma^2(F)$; residual electron density ± 0.2 (I) and $\pm 0.3 \text{ e } \text{\AA}^{-3}$ (II); atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Computations were carried out on the University of Birmingham ICL 1906A and on the CDC 7600 at the University of Manchester Regional Computer Centre.

Discussion. Atomic coordinates for compounds (I) and (II) are listed in Tables 1 and 2,* and bond lengths and angles are in Tables 3 and 4. The conformation of the molecules and the atom numbering are illustrated in Figs. 1 and 2.

The angle between the mean planes of the phenyl rings is 76.8 (5) [compound (I)] and 83.6 (2) $^\circ$ [compound (II)]. In both structures the phenyl rings

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52026 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic temperature factors (\AA^2) for compound (I)

$$U_{\text{eq}} = \frac{1}{3} \sum_{i=1}^3 \sum_{j=1}^3 U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
S1	-0.3346 (1)	0.1173 (4)	-0.3609 (1)	0.085
C1	-0.3383 (4)	0.1629 (13)	-0.2724 (3)	0.057
C2	-0.3028 (4)	0.0077 (12)	-0.2216 (3)	0.054
C3	-0.3140 (4)	0.0445 (12)	-0.1540 (3)	0.058
C4	-0.3621 (4)	0.2340 (13)	-0.1326 (3)	0.054
C5	-0.3978 (4)	0.3924 (13)	-0.1832 (3)	0.057
C6	-0.3864 (4)	0.3558 (13)	-0.2514 (3)	0.058
C7	-0.3756 (3)	0.2643 (11)	-0.0576 (2)	0.085
C1'	-0.2154 (4)	0.0456 (12)	-0.3646 (3)	0.057
C2'	-0.1423 (5)	0.1830 (13)	-0.3361 (3)	0.067
C3'	-0.0514 (5)	0.1223 (14)	-0.3428 (3)	0.069
C4'	-0.0273 (5)	-0.0643 (14)	-0.3804 (3)	0.069
C5'	-0.0998 (6)	-0.2036 (14)	-0.4091 (3)	0.077
C6'	-0.1916 (5)	-0.1503 (14)	-0.4013 (3)	0.073
C7'	0.0719 (2)	-0.1293 (14)	-0.3903 (3)	0.104
F2	-0.2560 (3)	-0.1873 (7)	-0.2389 (2)	0.083
F3	-0.2762 (2)	-0.1181 (6)	-0.1081 (2)	0.085
F5	-0.4462 (2)	0.5867 (7)	-0.1676 (2)	0.085
F6	-0.4241 (2)	0.5161 (7)	-0.2969 (2)	0.085
F2'	-0.1597 (2)	0.3792 (7)	-0.3006 (2)	0.098
F3'	0.0155 (3)	0.2659 (8)	-0.3121 (2)	0.117
F5'	-0.0856 (3)	-0.3988 (9)	-0.4463 (2)	0.121
F6'	-0.2600 (3)	-0.2924 (8)	-0.4303 (2)	0.111
F7A*	-0.2955	0.2546	-0.0208	0.139
F7B*	-0.4102	0.4832	-0.0459	0.124
F7C*	-0.4237	0.1052	-0.0369	0.190
F7D†	-0.3010	0.3635	-0.0246	0.225
F7E†	-0.4496	0.3550	-0.0462	0.156
F7F†	-0.3910	0.0651	-0.0261	0.128
F7A‡	0.0789	-0.1494	-0.4557	0.130
F7B‡	0.1388	0.0250	-0.3609	0.139
F7C‡	0.0990	-0.3257	-0.3615	0.143
F7D§	0.0815	-0.3227	-0.4297	0.201
F7E§	0.1193	-0.1551	-0.3994	0.214
F7F§	0.1022	0.0470	-0.4292	0.158

* Site occupation 0.66 (2).

† Site occupation 0.34 (2).

‡ Site occupation 0.51 (3).

§ Site occupation 0.49 (3).

Table 2. Fractional atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic temperature factors (\AA^2) for compound (II)

$$U_{\text{eq}} = \frac{1}{3} \sum_{i=1}^3 \sum_{j=1}^3 U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
S1	0.2005 (1)	0.7954 (1)	0.0586 (1)	0.062
C1	0.1723 (3)	0.6713 (3)	0.1354 (2)	0.046
C2	0.3200 (3)	0.5968 (3)	0.1626 (2)	0.046
C3	0.2977 (3)	0.4985 (3)	0.2188 (3)	0.047
C4	0.1244 (4)	0.4674 (3)	0.2452 (3)	0.048
C5	-0.0256 (3)	0.5384 (3)	0.2172 (3)	0.049
C6	-0.0009 (3)	0.6392 (3)	0.1644 (3)	0.048
C1'	0.3162 (4)	0.9155 (3)	0.1770 (2)	0.047
C2'	0.2334 (4)	0.9981 (3)	0.3150 (3)	0.054
C3'	0.3242 (5)	1.0917 (3)	0.4048 (3)	0.058
C4'	0.4970 (4)	1.1106 (3)	0.3588 (3)	0.055
C5'	0.5805 (5)	1.0325 (3)	0.2223 (3)	0.052
C6'	0.4910 (4)	0.9340 (3)	0.1339 (2)	0.048
F2	0.4913 (2)	0.6211 (2)	0.1330 (2)	0.060
F3	0.4435 (2)	0.4288 (2)	0.2430 (2)	0.066
F5	-0.1946 (2)	0.5109 (2)	0.2445 (2)	0.070
F6	-0.1487 (2)	0.7083 (2)	0.1402 (2)	0.068
F2'	0.0630 (3)	0.9850 (2)	0.3601 (2)	0.077
F3'	0.2398 (3)	1.1664 (2)	0.5372 (2)	0.092
F5'	0.7492 (2)	1.0470 (2)	0.1743 (2)	0.075
F6'	0.5794 (2)	0.8555 (2)	0.0023 (2)	0.067
N4	0.1015 (3)	0.3573 (3)	0.2992 (3)	0.062
N4'	0.5932 (4)	1.2139 (3)	0.4541 (3)	0.075
O1	0.1511 (4)	0.3754 (3)	0.4070 (3)	0.090
O2	0.0373 (4)	0.2538 (3)	0.2309 (3)	0.098
O1'	0.6180 (6)	1.1957 (3)	0.5540 (3)	0.131
O2'	0.6401 (5)	1.3084 (3)	0.4250 (4)	0.115

Table 3. *Molecular geometry for compound (I)*

(a) Bond lengths (Å) with e.s.d.'s in parentheses			
S1—C1	1.757 (5)	S1—C1'	1.768 (6)
C1—C2	1.374 (8)	C1'—C2'	1.369 (8)
C1—C6	1.368 (8)	C1'—C6'	1.374 (8)
C2—C3	1.366 (7)	C2'—C3'	1.370 (8)
C2—F2	1.344 (6)	C2'—F2'	1.337 (7)
C3—C4	1.356 (8)	C3'—C4'	1.344 (8)
C3—F3	1.349 (6)	C3'—F3'	1.342 (7)
C4—C5	1.384 (8)	C4'—C5'	1.370 (9)
C4—C7	1.511 (6)	C4'—C7'	1.504 (7)
C5—C6	1.378 (7)	C5'—C6'	1.377 (8)
C5—F5	1.343 (6)	C5'—F5'	1.340 (7)
C6—F6	1.335 (6)	C6'—F6'	1.340 (7)
C7—F7A	1.289	C7'—F7'A	1.300
C7—F7B	1.350	C7'—F7'B	1.370
C7—F7C	1.223	C7'—F7'C	1.275
C7—F7D	1.311	C7'—F7'D	1.344
C7—F7E	1.221	C7'—F7'E	1.152
C7—F7F	1.304	C7'—F7'F	1.347

(b) Bond angles (°) with e.s.d.'s in parentheses			
C1—S1—C1'	102.0 (3)	S1—C1'—C2'	124.0 (5)
S1—C1—C2	124.9 (6)	S1—C1'—C6'	120.0 (5)
S1—C1—C6	118.8 (5)	C2'—C1'—C6'	115.8 (6)
C2—C1—C6	116.1 (5)	C1'—C2'—C3'	121.2 (6)
C1—C2—C3	122.1 (6)	C1'—C2'—F2'	119.5 (6)
C1—C2—F2	119.1 (5)	C3'—C2'—F2'	119.2 (6)
C3—C2—F2	118.8 (6)	C2'—C3'—C4'	123.4 (7)
C2—C3—C4	122.4 (6)	C3'—C4'—C5'	115.8 (6)
C3—C4—C5	116.1 (6)	C4'—C5'—C6'	121.7 (7)
C4—C5—C6	121.6 (6)	C1'—C6'—C5'	121.8 (6)
C1—C6—C5	121.8 (6)	C1'—C6'—F6'	118.7 (7)
C1—C6—F6	120.6 (6)	C5'—C6'—F6'	119.4 (7)
C5—C6—F6	117.6 (6)		

(c) Torsion angles (°); e.s.d.'s 0.9°			
C1'—S1—C1—C2	-49.9	C1—S1—C1'—C2'	-52.5
C1'—S1—C1—C6	135.8	C1—S1—C1'—C6'	131.7

Table 4. *Molecular geometry for compound (II)*

(a) Bond lengths (Å) with e.s.d.'s in parentheses			
S1—C1	1.768 (3)	S1—C1'	1.773 (3)
C1—C2	1.382 (3)	C1'—C2'	1.386 (3)
C1—C6	1.389 (3)	C1'—C6'	1.378 (4)
C2—C3	1.365 (4)	C2'—C3'	1.372 (4)
C2—F2	1.344 (3)	C2'—F2'	1.334 (3)
C3—C4	1.380 (3)	C3'—C4'	1.375 (4)
C3—F3	1.330 (3)	C3'—F3'	1.334 (3)
C4—C5	1.382 (4)	C4'—C5'	1.369 (4)
C4—N4	1.460 (4)	C4'—N4'	1.474 (4)
C5—C6	1.366 (4)	C5'—C6'	1.382 (4)
C5—F5	1.337 (3)	C5'—F5'	1.337 (3)
C6—F6	1.338 (3)	C6'—F6'	1.339 (3)
N4—O1	1.210 (3)	N4'—O1'	1.188 (3)
N4—O2	1.211 (3)	N4'—O2'	1.191 (3)

(b) Bond angles (°) with e.s.d.'s in parentheses			
C1—S1—C1'	99.7 (1)	S1—C1'—C2'	121.8 (2)
S1—C1—C2	122.3 (2)	S1—C1'—C6'	121.0 (2)
S1—C1—C6	120.5 (2)	C2'—C1'—C6'	117.2 (2)
C2—C1—C6	117.1 (2)	C1'—C2'—C3'	121.0 (3)
C1—C2—C3	122.0 (2)	C1'—C2'—F2'	118.9 (2)
C1—C2—F2	119.4 (2)	C3'—C2'—F2'	120.0 (2)
C3—C2—F2	118.6 (2)	C2'—C3'—C4'	120.7 (2)
C2—C3—C4	119.7 (2)	C3'—C4'—C5'	119.4 (2)
C3—C4—C5	119.7 (2)	C4'—C5'—C6'	119.4 (2)
C4—C5—C6	119.6 (2)	C1'—C6'—C5'	122.2 (2)
C1—C6—C5	121.9 (2)	C1'—C6'—F6'	119.9 (2)
C1—C6—F6	119.6 (2)	C5'—C6'—F6'	117.9 (2)
C5—C6—F6	118.6 (2)	C4'—N4'—O1'	117.1 (3)
C4—N4—O1	117.5 (3)	C4'—N4'—O2'	117.7 (3)
C4—N4—O2	117.1 (3)	O1'—N4'—O2'	125.2 (3)
O1—N4—O2	125.3 (3)		

(c) Torsion angles (°); e.s.d.'s 0.3°			
C1'—S1—C1—C2	-55.6	C1—S1—C1'—C2'	-62.8
C1'—S1—C1—C6	127.8	C1—S1—C1'—C6'	119.4

are inclined at approximately the same angle to the central C(1)—S—C(1') plane, 46.9 (6) and 49.8 (6)° in (I), and 53.4 (2) and 61.0 (2)° in (II), corresponding to the 'twist' conformation in the notation of Van Der Heijden, Griffith, Chandler & Robertson (1975). The nitro groups of (II) are oriented at angles of 57.8 (3) and 57.4 (3)° to their respective phenyl planes, compared with 12.6° in DTFNPS (Goodhand & Hamor, 1979), 6.3° in 4-nitrodiphenyl sulfide (Krajewski, Riva di Sanseverino, Dondoni & Mangini, 1975) and 1.2° in 4-dimethylamino-4'-nitrodiphenyl sulfide (Krajewski *et al.*, 1975). The relatively large angle in (II) may be attributed to the need to avoid close contacts between the O atoms of the nitro group and the two adjacent fluorine substituents, F...O distances being in the range 2.85–2.88 Å. In the DTFNPS structure (Goodhand & Hamor, 1979) a much closer F...O contact of 2.58 Å occurs, the unfavourable energetics of which are compensated by an attractive interaction between the other O atom and the adjacent H atom. The O atoms of the nitro groups in the title compound (II) have relatively large thermal parameters, and this could be an indication of some freedom of rotation about the C—NO₂ bonds, or of some degree of disorder in the orientations of the nitro groups.

The aromatic C—C bond lengths are in the range 1.344–1.384, mean 1.369 Å in (I) and 1.365–1.389, mean 1.377 Å in (II). They compare with a mean of 1.374 Å in DTFNPS. These values indicate a slight shortening of the aromatic bonds relative to hydrocarbon analogues. The C—S bond lengths average 1.763 and 1.770 Å in the two compounds in good agreement with the corresponding bonds in

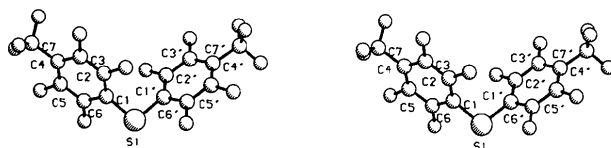


Fig. 1. Stereoscopic view of molecule (I) in a direction perpendicular to the plane of atoms C(1), S, C(1') drawn with PLUTO (Motherwell & Clegg, 1978).

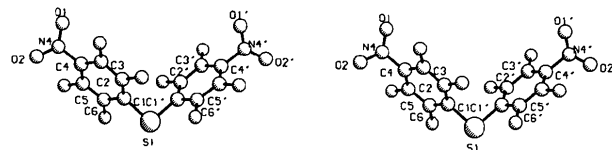


Fig. 2. Stereoscopic view of molecule (II). View direction as for Fig. 1.

DTFNPS (mean 1.771 Å), but slightly shorter than in a selection of unfluorinated diphenyl sulfides where the mean length is 1.780 Å. Such a shortening of bond lengths in fluorinated compounds has been noted previously (Goodhand & Hamor, 1979, and references therein).

With respect to the bond angle at S, Goodhand & Hamor (1979) noted that in the fluorinated compound DTFNPS this was 99.7°, significantly smaller than in unfluorinated analogues, where this angle ranged from 102.9 to 105.6, mean 103.7°. More recent studies have, however, revealed somewhat smaller C—S—C angles in a small number of diphenyl sulfide systems, the smallest value (101.2°) occurring in the crystal structure of 2-nitro-1,3-bis-(phenylthio)benzene (Lynch, Simonsen, Miller, Turley & Martin, 1985). The C—S—C angles in the title compounds, 102.0° in (I) and 99.7° in (II) are, nevertheless, still consistent with the overall trend noted previously.

There are no abnormal intermolecular contact distances in either structure. The shortest contact, 2.744 Å, occurs in structure (I), between F2' and F7'E of the disordered trifluoromethyl group of the molecule at $-x, \frac{1}{2} + y, -\frac{1}{2} - z$.

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Malotilate: Structure and Phase Transformation

BY D. R. VEGA AND R. F. BAGGIO*

Departamento de Física, Comisión Nacional de Energía Atómica, Av. del Libertador 8250, Buenos Aires 1429, Argentina

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Abstract. C₁₂H₁₆O₄S₂, *M_r* = 288.4. Two phases: phase *A*: monoclinic, *P*2₁/*a*, *a* = 22.425 (3), *b* = 8.273 (2), *c* = 15.713 (3) Å, β = 96.66 (1)°, *V* = 2895 (1) Å³, *Z* = 8, *D_x* = 1.323, *D_m* = 1.320 (2) g cm⁻³, Mo *K*α radiation, λ = 0.7107 Å, μ = 3.55 cm⁻¹, *F*(000) = 1216, final conventional *R* factor = 0.071, for 1404 observed [*I* > 2σ(*I*)] reflexions out of 4530 unique; phase *B*: triclinic, *P*1̄, *a* = 11.515 (2), *b* = 11.840 (2), *c* = 12.097 (2) Å, α = 94.57 (2), β = 102.91 (2), γ = 100.81 (2)°, *V* = 1566 (1) Å³, *Z* = 4, *D_x* = 1.212, *D_m* = 1.215 (2) g cm⁻³, Mo *K*α radiation, λ = 0.7107 Å, μ = 3.28 cm⁻¹, *F*(000) = 608, final conventional *R* = 0.059, for 2338 observed [*I* > 3σ(*I*)] reflexions out of 4585 unique. Data gathered at room temperature. In

both structures the asymmetric unit consists of two independent moieties, which in form *A* are found to pack more closely than in form *B*, as seen from the measured density values. This is achieved through an overall greater departure of the molecule from planarity giving rise to a geometry more amenable to dense stacking. Intermolecular interactions are mainly van der Waals, with only a few weak hydrogen bonds present (shortest O...H: 2.32 Å).

Introduction. Malotilate [C₁₂H₁₆O₄S₂, diisopropyl 1,3-dithiol-2-ylidenemalonate, (I)] is a very powerful hepatoprotector and activator, which in the last few years has merited the increasing attention of a number of research workers in the field of biology, to the point of having specific meetings organized on the subject (Symposium on Malotilate held at the 7th

* Author to whom correspondence should be addressed.