acid groups about a center of symmetry. These dimers are then joined laterally to other dimers of the same type. Crystallographically different molecules are joined only by the hydrogen bonds $O(2)-H(2)\cdots O(9)$ and $O(7)-H(7)\cdots O(3)$.

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# Structure of Bis(phenylsulphonyl) Trisulphide, C12H10O4S5

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**Abstract.** $M_r = 378.52$, tetragonal, $P4_12_12$, $a = 7.758$ (1), $c = 26.204$ (4) Å, $V = 1577.1$ (1) Å$^3$, $Z = 4$, $D_m = 1.56$, $D_x = 1.594$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.7093 Å, $\mu$(Mo $K\alpha$) = 0.715 mm$^{-1}$, $T = 298$ K, $F(000) = 776$, $R = 0.042$ for 1146 observed reflections. The molecule contains a twofold axis. There are two kinds of S–S bond lengths: 2.026 (1) and 2.102 (1) Å, the longer bonds involving the S atoms with the dioxide substituents.

**Introduction.** The nature of the S–S bond has been of great interest for many decades. The structures of certain polysulphides and sulphonylsulphides were investigated by Dawson, Mathieson & Robertson (1948) and Mathieson & Robertson (1949). The S chain skeleton was confirmed later for various polysulphides of disulphonate $(O_3S-S_n-SO_3)^{2-}$ compounds (Stewart & Szymański, 1979a,b) and $\varphi-SO_x-S_n-SO_y-\varphi$ (Kiers & Vos, 1978). In order to compare the conformation of the polysulphide chain and to understand the effect of O substituents on the S–S bond, the title compound was synthesized by the method of

Troeger & Hornung (1899) and characterized by structure determination.

**Experimental.** Crystal obtained by slow evaporation of acetic acid solution. 390μm diameter spherical crystal. CAD-4 diffractometer. 16 reflections in $2\theta$ range 26°–31° used in the refinement of the cell parameters. $D_m$ by flotation method (CCl$_3$H/CBr$_3$H). Spherical absorption correction applied. $2\theta_{max} = 70°$, $-12 \le h \le 12$, $-42 \le l \le 42$, and $h > k$. Three standard reflections monitored every hour, variation less than 3%, 3468 unique reflections, 1146 of which observed (including Friedel pairs) with $I \ge 2\sigma(I)$. $R = 0.042$, $R_w = 0.020$, $w = 4F_o^2/\sigma^2(I)$ where $\sigma^2(I) = (P + t^2B)$. Structure solved by Patterson method. H atoms calculated, and then refined in the least-squares process. All non-hydrogen and H atoms refined anisotropically and isotropically, respectively. In final cycle $(\Delta/\sigma)_{max} = 0.71$. Peaks on final difference Fourier synthesis between 0.29 and $-0.30$ e Å$^{-3}$. Secondary-extinction-coefficient value 0.24 (length in μm). Atomic scattering factors calculated by the analytical form using the coefficients given in International Tables for X-ray Crytallography (1974); anomalous-dispersion

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calculation included. Computing programs from the NRCC SDP PDP-11 package (Gabe & Lee, 1981), and ORTEP (Johnson, 1965) from the Nonius SDP package.

**Discussion.** The title compound has a S—S chain structure as expected. The molecule has twofold symmetry passing through S(1); only half of the molecule is unique. The fractional coordinates and the $B_{eq}$ values of the non-hydrogen atoms are given in Table 1.* Bond distances and angles are in Table 2. The

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39602 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecular structure is shown in Fig. 1; the packing is given in Fig. 2. Because of the polar space group, a determination of the absolute configuration was tried by changing the sign of $f''$ (0.12 for S). There is no significant difference between the two configurations. However, the reported one is slightly better than the other ($R_w = 0.0198$ vs 0.0201).

A comparison of the bond lengths and bond angles in similar S-chain compounds is listed in Table 3. It is obvious that the S—S bond is similar to that in sulphur (2.050 Å; Coppens, Yang, Blessing, Cooper & Larsen, 1977), but the S—S bond with the dioxide substituents on one of the S atoms is lengthened somewhat.
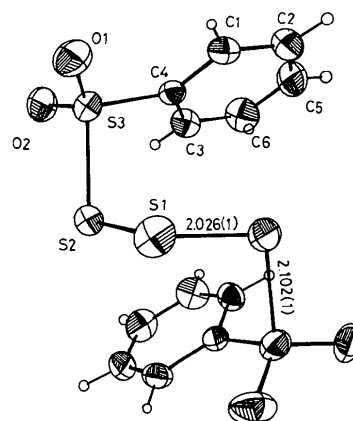
### Table 1. *Fractional atomic coordinates and $B_{eq}$ values*

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

|  | x | y | z | $B_{eq}(\text{Å}^2)$ |
|---|---|---|---|---|
| S(1) | 0.2699 (1) | 0.2699 | 0 | 5.78 (7) |
| S(2) | 0.2132 (1) | 0.5333 (1) | 0.28234 (3) | 5.17 (5) |
| S(3) | 0.1366 (1) | 0.3782 (1) | 0.22060 (3) | 5.02 (6) |
| O(1) | 0.0051 (3) | 0.4603 (3) | 0.1917 (1) | 6.8 (2) |
| O(2) | 0.1041 (3) | 0.2169 (3) | 0.2450 (1) | 7.1 (2) |
| C(1) | 0.3324 (4) | 0.4501 (4) | 0.1373 (1) | 4.6 (2) |
| C(2) | 0.4858 (5) | 0.4408 (4) | 0.1104 (1) | 5.7 (2) |
| C(3) | 0.4629 (4) | 0.2764 (4) | 0.2032 (1) | 4.9 (2) |
| C(4) | 0.3238 (3) | 0.3655 (3) | 0.1837 (1) | 3.8 (2) |
| C(5) | 0.6223 (4) | 0.3538 (5) | 0.1297 (1) | 6.6 (3) |
| C(6) | 0.6123 (4) | 0.2692 (4) | 0.1756 (1) | 6.4 (2) |



Fig. 1. The molecular structure with 40% probability thermal ellipsoids. (Distances in Å.)

### Table 2. *Bond lengths (Å) and bond angles (°)*

| | | | |
|---|---|---|---|
| S(1)—S(2) | 2.026 (1) | C(1)—C(2) | 1.385 (4) |
| S(2)—S(3) | 2.102 (1) | C(1)—C(4) | 1.385 (4) |
| S(3)—O(1) | 1.422 (2) | C(2)—C(5) | 1.353 (5) |
| S(3)—O(2) | 1.428 (2) | C(3)—C(4) | 1.379 (4) |
| S(3)—C(4) | 1.747 (3) | C(3)—C(6) | 1.367 (4) |
| | | C(5)—C(6) | 1.372 (4) |
| S(2')—S(1)—S(2) | 106.98 (7) | C(1)—C(2)—C(5) | 120.7 (2) |
| S(1)—S(2)—S(3) | 102.41 (4) | C(2)—C(5)—C(6) | 121.4 (3) |
| S(2)—S(3)—C(4) | 102.89 (9) | C(5)—C(6)—C(3) | 119.5 (3) |
| O(1)—S(3)—C(4) | 109.1 (1) | C(6)—C(3)—C(4) | 119.3 (2) |
| O(2)—S(3)—C(4) | 110.2 (1) | C(3)—C(4)—C(1) | 121.7 (3) |
| O(1)—S(3)—O(2) | 120.3 (1) | C(4)—C(1)—C(2) | 117.6 (3) |



Fig. 2. Packing diagram.

### Table 3. *Comparison of bond lengths (Å) and bond angles (°) in similar S-chain compounds*

| | $\varphi SO_2S_3SO_2\varphi$ | $\varphi SO_2SSO_2\varphi$ | $\varphi SS\varphi$ | $CH_3\varphi SSO\varphi CH_3$ | $\varphi SSOS\varphi$ | $K_2S_3O_6$ | $K_2S_4O_6$ |
|---|---|---|---|---|---|---|---|
| S—S | 2.026 (1) | | 2.023 (1) | | | 2.097 (1) | 2.013 (9) 2.020 (4) |
| S—S($O_n$) | 2.102 (1) | 2.07 (2) | | 2.108 (2) | 2.132 (2) | 2.064 (1) | |
| S—O | 1.422 (2) 1.428 (2) | 1.41 (4) | | 1.457 (5) | 1.476 (4) | 1.443 (2) | 1.44 (1) |
| S—C | 1.747 (3) | 1.76 (2) | 1.789 (1) 1.788 (1) | 1.776 (5) | 1.775 (4) | | |
| S—S—S | 107.0 (1) | 106.5 (1) | | | 88.3 (2) | 106.4 | 100.5 106.2 |
| S—S—C | 102.9 (1) | 101.7 (1) | 106.5 105.2 | 93.1 (2) 99.0 (4) | 98.0 (2) 97.5 (1) | | |
| Reference | (a) | (b) | (c) | (d) | (d) | (e) | (f) |

References: (a) this work; (b) Mathieson & Robertson (1949); (c) Sacerdoti & Gilli (1975); (d) Kiers & Vos (1978); (e) Stewart & Szymański (1979a); (f) Stewart & Szymański (1979b).

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# Structure of Fluorene, C₁₃H₁₀, at 159 K

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**Abstract.** $M_r = 166.22$, orthorhombic, *Pnma*, $a = 8.365$ (3), $b = 18.745$ (4), $c = 5.654$ (2) Å, $V = 886.4$ (5) Å³, $Z = 4$, $F(000) = 352$, $D_x = 1.25$ g cm⁻³, $\lambda$(Mo $K\bar{\alpha}$) = 0.71069 Å, $T = 159$ K, $\mu = 0.654$ cm⁻¹, 1250 unique reflections, $R = 0.043$. The fluorene molecule has mirror symmetry, but is slightly non-planar. The asymmetric unit [C(1) through C(7)], however, is planar, giving the molecule a 'V' shape (dihedral angle 1.3°). The crystal is well ordered.

**Introduction.** The crystal structure of dibenzofuran has been recently shown to exhibit a disorder such that 9% of the molecules adopt an alternative orientation (Reppart, Gallucci, Lundstedt & Gerkin, 1984). In view of that result, it was of interest to determine whether fluorene, a molecule of quite similar geometry, might also manifest a disordered crystal structure.

The only previously reported quantitative study of the fluorene structure was by Burns & Iball (1955), who used Fourier projections and least-squares refinement for the 0*kl*, *h*0*l* and *hk*0 zones to determine the atomic coordinates. On the basis of this limited analysis, these authors reported no disorder.

We report here the first complete three-dimensional analysis of the fluorene crystal structure.

**Experimental.** Commercial fluorene (Eastman, #598) purified by vertical zone refining, 250 passes; clear, colorless crystal taken from center of ingot, cut into parallelepiped, 0.53 × 0.59 × 0.60 mm. Syntex *P*1̄ diffractometer, LT-1 low-temperature attachment. Set-

ting angles for 25 reflections (19° < 2θ < 31°) used to determine unit-cell constants and crystal orientation. Temperature measured at cold-stream exit nozzle during data collection and at crystal site after data collection with a Fluke 2100A digital thermometer (type *K* thermocouple). The temperature measured at the site of the crystal was 159 K, estimated uncertainty ±2 K. Data collected for 3278 reflections with +*h*, +*k*, ±*l*. ω–2θ scan, 4° < 2θ < 60°, scan range 2θ Mo $K\alpha_1$ − 1.0° to 2θ Mo $K\alpha_2$ + 1.1°, background/scan-time ratio 0.5. 1250 unique reflections, $R_{int} = 0.02$, 1017 reflections with $I > 3\sigma_I$ used in refinement. Uncertainties assigned using $\sigma_I^2 = R^2(C + 4B) + (0.02I)^2$; *C* total counts, *R* scan rate, *B* total background counts, *I* intensity. Six standard reflections (022, 051, 131, 191, 231, 270), maximum intensity variation 6%. No absorption correction. Systematic absences as well as the results from the zero-moment test of Howells, Phillips & Rogers (1950) suggested that the space group had not changed from that of the room-temperature structure, *Pnma*. Least-squares refinement using *SHELX*76 (Sheldrick, 1976); $\sum w(|F_o| - |F_c|)^2$ minimized, $w = \sigma_F^{-2}$. Initial C positions taken from Burns & Iball (1955), H-atom positions from $F_o - F_c$ map. Final refinement cycle: $R = 0.043$, $wR = 0.045$, $S = 2.9$, max. $\Delta/\sigma = 0.004$, max. and min. peak heights on final $F_o - F_c$ map 0.24 and −0.21 e Å⁻³, with peaks located near bond centers and troughs located near the centers of the rings of the molecule. Scattering factor for C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965).