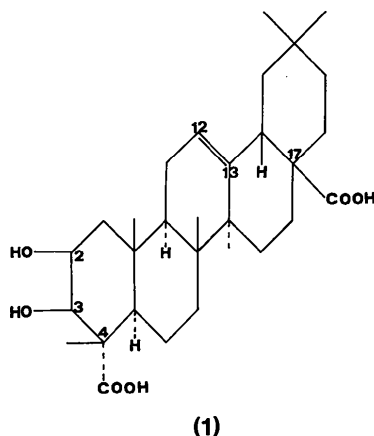


Table 2.* Figs. 1 and 2, prepared using *ORTEPII* (Johnson, 1976), show a perspective view of the molecule with atomic numbering and the molecular packing, respectively.

Related literature. Medicagenic acid (1) was first isolated from alfalfa (*Medicago sativa*) and was shown



* Lists of observed and calculated structure factors, anisotropic thermal parameters, H-atom positions and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51422 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

to be $2\beta,3\beta$ -dihydroxyolean-12-ene-23,28-dioic acid by various degradations and by a direct correlation with arjunolic acid which has a $2\alpha,3\beta$ -orientation (Djerassi, Thomas, Livingston & Thompson, 1957). The molecular geometry of (1) is very similar to that found in the natural compound maytenfolic acid (Nozaki, Suzuki, Lee & McPhail, 1982) and in a synthetic triterpene reported recently (Gzella, Zaprutko, Wrzeciono & Gdaniec, 1986).

The author is grateful to Professor K. Hostettmann (Lausanne) and his collaborators for supplying the crystals of medicagenic acid, and to the Swiss National Science Foundation for an equipment grant (No. 2.372-0.84).

References

- DJERASSI, C., THOMAS, D. B., LIVINGSTON, A. L. & THOMPSON, C. R. (1957). *J. Am. Chem. Soc.* **79**, 5292-5297.
- GZELLA, A., ZAPRUTKO, L., WRZECIONO, U. & GDANIEC, M. (1986). *Acta Cryst.* **C42**, 1859-1862.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- NOZAKI, H., SUZUKI, H., LEE, K.-H. & MCPHAIL, A. T. (1982). *J. Chem. Soc. Chem. Commun.* pp. 1048-1051.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1989), **C45**, 343-345

Structure of (4*S*)-4-(Methylthio)dibenzothiophene

BY L. V. DUNKERTON AND A. PANDEY

Department of Chemistry and Biochemistry, Southern Illinois University, Carbondale, IL 62901, USA

AND P. D. ROBINSON*

Department of Geology, Southern Illinois University, Carbondale, IL 62901, USA

(Received 25 July 1988; accepted 26 September 1988)

Abstract. $C_{13}H_{10}S_2$, $M_r = 230.34$, orthorhombic, $P2_12_12_1$, $a = 10.732$ (3), $b = 14.838$ (2), $c = 7.077$ (1) Å, $V = 1126.8$ (6) Å³, $Z = 4$, $D_x = 1.36$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 4.21$ cm⁻¹, $F(000) = 480$, $T = 296$ K, $R = 0.037$, 688 unique observed reflections. The compound was prepared by treating 4-lithiodibenzothiophene with dimethyl disulfide. The molecule is a planar dibenzothiophene having a methyl sulfide substituent at the IUPAC C4 position.

Experimental. Preparation by addition of *n*-butyllithium (one equivalent) to dibenzothiophene in THF, solution stirred for 5 h at 273 K. Dimethyl disulfide (one equivalent) added slowly, reaction mixture refluxed for 1.5 h, then stirred at 298 K for 12 h, quenched with water, neutralized with 10% HCl and extracted with dichloromethane. Extract concentrated to give colorless crystals in 70% yield. Crystals suitable for X-ray diffraction grown from dichloromethane with diffusion of a hexane layer. Crystal used for data collection $0.22 \times 0.20 \times 0.13$ mm, clear fragment cut from larger

* To whom all correspondence should be addressed.

Table 1. Positional and equivalent isotropic thermal parameters for the non-H atoms and their e.s.d.'s

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
S(1)	0.1256 (2)	0.3509 (1)	0.4313 (2)	4.64 (8)
S(2)	-0.0978 (2)	0.4854 (1)	0.5351 (3)	6.0 (1)
C(1)	0.2359 (6)	0.3154 (4)	0.267 (1)	4.3 (3)
C(2)	0.3248 (7)	0.2491 (5)	0.294 (1)	5.3 (4)
C(3)	0.4045 (7)	0.2294 (5)	0.149 (1)	6.1 (4)
C(4)	0.3975 (8)	0.2740 (5)	-0.025 (1)	6.3 (4)
C(5)	0.3100 (7)	0.3408 (5)	-0.050 (1)	5.3 (3)
C(6)	0.2282 (6)	0.3628 (4)	0.094 (1)	4.3 (3)
C(7)	0.1326 (6)	0.4319 (4)	0.1027 (8)	3.8 (3)
C(8)	0.1012 (7)	0.4956 (5)	-0.0348 (9)	5.0 (3)
C(9)	0.0084 (7)	0.5567 (5)	0.004 (1)	5.4 (4)
C(10)	-0.0556 (6)	0.5570 (4)	0.173 (1)	4.8 (3)
C(11)	-0.0243 (6)	0.4947 (4)	0.3147 (9)	4.1 (3)
C(12)	0.0696 (6)	0.4331 (4)	0.2737 (8)	3.8 (3)
C(13)	-0.1902 (7)	0.5839 (5)	0.552 (1)	7.0 (4)

Table 2. Selected bond distances (\AA), bond angles ($^\circ$), and their e.s.d.'s

S(1)-C(1)	1.741 (7)	C(5)-C(6)	1.39 (1)
S(1)-C(12)	1.758 (6)	C(6)-C(7)	1.451 (9)
S(2)-C(11)	1.754 (6)	C(7)-C(12)	1.387 (8)
S(2)-C(13)	1.769 (8)	C(7)-C(8)	1.398 (9)
C(1)-C(2)	1.384 (9)	C(8)-C(9)	1.37 (1)
C(1)-C(6)	1.41 (1)	C(9)-C(10)	1.38 (1)
C(2)-C(3)	1.37 (1)	C(10)-C(11)	1.404 (9)
C(3)-C(4)	1.40 (1)	C(11)-C(12)	1.391 (8)
C(4)-C(5)	1.38 (1)		
C(1)-S(1)-C(12)	91.1 (3)	C(12)-C(7)-C(8)	118.8 (6)
C(11)-S(2)-C(13)	104.3 (4)	C(12)-C(7)-C(6)	112.9 (5)
C(2)-C(1)-C(6)	120.8 (7)	C(8)-C(7)-C(6)	128.3 (6)
C(2)-C(1)-S(1)	126.4 (6)	C(9)-C(8)-C(7)	118.7 (6)
C(6)-C(1)-S(1)	112.8 (5)	C(8)-C(9)-C(10)	122.5 (6)
C(3)-C(2)-C(1)	118.7 (7)	C(9)-C(10)-C(11)	119.8 (6)
C(2)-C(3)-C(4)	121.6 (7)	C(12)-C(11)-C(10)	117.2 (6)
C(5)-C(4)-C(3)	119.5 (8)	C(12)-C(11)-S(2)	117.4 (5)
C(4)-C(5)-C(6)	120.4 (7)	C(10)-C(11)-S(2)	125.3 (5)
C(5)-C(6)-C(1)	118.9 (6)	C(7)-C(12)-C(11)	123.0 (6)
C(5)-C(6)-C(7)	130.1 (6)	C(7)-C(12)-S(1)	112.2 (5)
C(1)-C(6)-C(7)	111.0 (6)	C(11)-C(12)-S(1)	124.8 (5)

crystal. Rigaku AFC5S diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω - 2θ scans, scan speed 4° min^{-1} , maximum of three scan repetitions to obtain $\sigma F/F < 0.10$. Lattice parameters from least-squares fit of 21 strong reflections in 2θ range 26 - 35° . A total of 1190 reflections measured (h 0 to 12, k 0 to 17, l 0 to 8), 18 systematically extinct reflections deleted, data set comprised of 1172 unique reflections, 688 of which were considered observed [$I > 3\sigma(I)$], $(\sin\theta/\lambda)_{\text{max}} = 0.60 \text{ \AA}^{-1}$. Three standard reflections (111, 002, $\bar{2}31$) varied by -0.2 , -0.5 and -0.4% , respectively; no decay correction applied. Data corrected for Lorentz, polarization, and absorption (empirical ψ scan correction, five reflections, transmission range 0.96 - 1.00). Direct methods provided the atomic positions of all non-H atoms, full-matrix least-squares refinement of F magnitudes performed. Ring H atoms placed in geometrically correct positions ($C-H = 0.95 \text{ \AA}$) and fixed, methyl-H atomic sites geometrically optimized

from three difference Fourier peaks and fixed, all H atom B 's set at $1.2 \times B_{eq}$ of associated C atom. Regeneration of H positions and refinement to convergence repeated until shifts became insignificant. Final refinement performed with 136 variables including all non-H positional and anisotropic thermal parameters, and one scale factor. Convergence yielded $R = 0.037$, $wR = 0.045$ [$w = 1/\sigma^2(|F_o|)$], $S = 1.10$ and $(\Delta/\sigma)_{\text{max}} = 0.0004$. Identical refinement of the alternative enantiomorph provided a slightly inferior R factor and goodness of fit indicator. Final difference synthesis produced $(\Delta\rho)_{\text{max}} = 0.17$ and $(\Delta\rho)_{\text{min}} = -0.17 \text{ e \AA}^{-3}$. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). Positional and thermal parameters are listed in Table 1, selected interatomic distances and angles are tabulated in Table 2.* Fig. 1 portrays the molecular configuration, thermal motion and atom-numbering scheme. All computer programs from the TEXSAN

* Lists of structure factors, H-atom coordinates, intermolecular distances, torsion angles, anisotropic thermal parameters and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51423 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

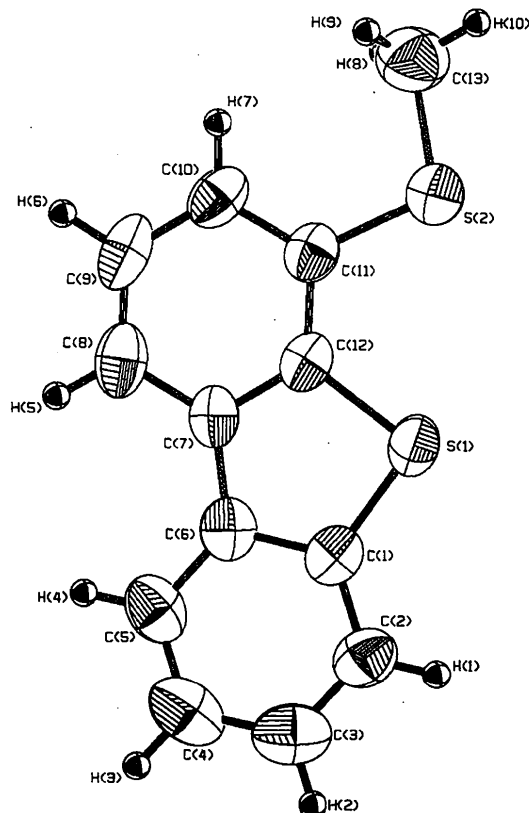


Fig. 1. Molecular structure and numbering scheme, thermal ellipsoids at the 50% probability level. H atoms shown with isotropic B 's of 1.0 \AA^2 .

crystal-structure-analysis package (Molecular Structure Corporation, 1985).

Related literature. The general procedure used in the synthesis of the title compound was originally employed by Tedjamulia, Tominaga, Castle & Lee (1983) in their preparation of 4-lithiodibenzothiophene. Utilizing their strategy various multisulfur-containing substituted dibenzothiophenes have been synthesized (Dunkerton, Barot & Nigam, 1987).

References

- CROMER, D. T. & WABER, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV, p. 71 (scattering factors), p. 148 (anomalous dispersion). Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- DUNKERTON, L. V., BAROT, B. C. & NIGAM, A. (1987). *J. Heterocycl. Chem.* **24**, 749–755.
- Molecular Structure Corporation (1985). *TEXSAN Structure Analysis Package*. MSC, 3304 Longmire Drive, College Station, Texas 77840, USA.
- TEDJAMULIA, M. L., TOMINAGA, Y., CASTLE, R. N. & LEE, M. L. (1983). *J. Heterocycl. Chem.* **20**, 861–866.

Acta Cryst. (1989). **C45**, 345–346

Structure of Phenacyl Phenyl Sulfone

BY M. KRAWIEC AND T. M. KRYGOWSKI

Department of Chemistry, University of Warsaw, Pasteura 1, 02-093 Warsaw, Poland

AND A. ZAKRZEWSKI

Institute of Technology and Chemical Engineering, Technical Academy, Seminaryjna 3, 85-326 Bydgoszcz, Poland

(Received 12 February 1988; accepted 26 September 1988)

Abstract. $C_{14}H_{12}O_3S$, $M_r = 260.31$, monoclinic, $P2_1/c$, $a = 9.231$ (3), $b = 5.363$ (1), $c = 25.742$ (10) Å, $\beta = 98.49$ (3)°, $V = 1260.5$ (7) Å³, $Z = 4$, $D_x = 1.371$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.07$ cm⁻¹, $F(000) = 544$, room temperature; final $R = 0.039$ for 1086 observed reflections, $F(hkl) > 3.92\sigma(F)$. Both benzene rings are planar within 0.03 Å. Five C–H bonds per molecule exhibit short contacts to O atoms: $d = 2.41$ (4), 2.51 (5), 2.77 (4), 2.67 (4) and 2.55 (5) Å.

Experimental. Phenacyl phenyl sulfone (hereafter abbreviated to PPS) was obtained following Borchardt, Janota & Zakrzewski (1987). It was crystallized from ethanol yielding colourless needles. Monoclinic symmetry was estimated from oscillation and Weissenberg photographs. Density not measured. A crystal ca 0.6 × 0.2 × 0.15 mm was mounted on a Syntex $P2_1$ single-crystal diffractometer. Systematic absences: $h0l$, l odd, $0k0$, k odd, space group $P2_1/c$. Cell constants determined by the least-squares treatment of 15 reflections with 2θ values between 4.5 and 13.2°. Intensities collected at room temperature using graphite-monochromated Mo $K\alpha$ radiation up to $2\theta = 50^\circ$, $-10 \leq h \leq 10$, $0 \leq k \leq 7$ and $0 \leq l \leq 31$, ω - 2θ scan mode. 2135 independent intensities were col-

lected, of which 165 were systematically absent. 1112 reflections classified as observed, $F_o > 3.92\sigma(F_o)$. Correction for Lorentz and polarization effects but not for absorption. Two standard reflections, 2% intensity variation. The structure was solved using *MULTAN80* (Main *et al.*, 1980) and refined by use of *SHELX76* (Sheldrick, 1976). $\sum w(|F_o| - |F_c|)^2$ was minimized with $w = k/[\sigma^2(F_o) + GF_o^2]$ ($k = 1.2266$, $G = 2.609 \times 10^{-3}$). Atomic scattering factors supplied by *SHELX76*. E map gave positions of all non-H atoms ($R = 0.45$). Location of H atoms and refinement of their positions led to final $R = 0.039$, $wR = 0.042$, $S = 1.0138$; the final difference density map gave extreme values -0.31 and $+0.17$ e Å⁻³; $(\Delta/\sigma)_{\max}$ in the last cycle 0.004 (mean 0.0009). Final positional parameters and equivalent isotropic temperature coefficients are given in Table 1.* Bond distances and bond angles are given in Table 2, the molecular structure and numbering of the atoms are shown in Fig. 1 [drawn with *PLUTO* (Motherwell, 1972)].

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51425 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.