

## References

- Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.
- Anisuzzaman, A. T. Md. (1995). PhD thesis, University of Strathclyde, Glasgow, Scotland.
- Antolini, L., Vezzosi, I. M., Battaglia, L. P. & Corradi, A. B. (1985). *J. Chem. Soc. Perkin Trans. 2*, pp. 237–239.
- Butcher, H., Hamor, T. A. & Martin, I. L. (1983). *Acta Cryst.* **C39**, 1469–1472.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Fleischer, E. B., Sung, N. & Hawkinson, S. (1968). *J. Phys. Chem.* **72**, 4311–4312.
- Glusker, J. P., Lewis, M. & Rossi, M. (1994). *Crystal Structure Analysis for Chemists and Biologists*, p. 423. Weinheim: VCH.
- Inui, S., Yamamoto, M., Nakae, H. & Asada, S. (1982). *Yakuzaigaku*, **42**, 71–78.
- Parkin, S., Moezzi, B. & Hope, H. (1995). *J. Appl. Cryst.* **28**, 53–56.
- Pertsin, A. J. & Kitaigorodsky, A. I. (1987). *The Atom–Atom Potential Method*, p. 112. Berlin: Springer-Verlag.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Silva, J. A. F. de, Bekersky, I., Brooks, M. A., Weinfeld, R. E., Glover, W. & Puglisi, C. V. (1974). *J. Pharm. Sci.* **63**, 1440–1445.
- Zsolnai, L. (1994). *ZORTEP. Interactive Graphics Program*. University of Heidelberg, Germany.

*Acta Cryst.* (1996). **C52**, 931–933

## Bis(diphenylphosphino)methane Disulfide

CLAIRE J. CARMALT,<sup>a</sup> ALAN H. COWLEY,<sup>b\*</sup> ANDREAS DECKEN,<sup>b</sup> YVONNE G. LAWSON<sup>a</sup> AND NICHOLAS C. NORMAN<sup>a</sup>

<sup>a</sup>Department of Chemistry, University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, England, and

<sup>b</sup>Department of Chemistry and Biochemistry, The University of Texas at Austin, Austin, TX 78712, USA. E-mail: cowley@mail.utexas.edu

(Received 21 June 1995; accepted 25 September 1995)

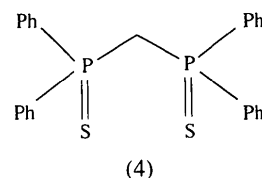
## Abstract

The title compound, methylenebis(diphenylphosphine sulfide),  $C_{25}H_{22}P_2S_2$ , has been structurally characterized and is found to be isostructural with its selenium analog.

## Comment

A number of compounds related to the title compound have been characterized previously by X-ray crystallography. Relevant structures include  $Ph_2PCH_2PPh_2$  (dppm) [(1); Schmidbaur, Reber, Schier, Wagner & Müller, 1988],  $Ph_2P(Se)CH_2P(Se)Ph_2$  (dppmSe<sub>2</sub>) [(2); Carroll & Titus, 1971] and the related compound  $Ph_2PCH_2P(Se)Ph_2$  (dppmSe) [(3); Colton, Hoskins &

Panagiotidou, 1987]. The title compound,  $Ph_2P(S)CH_2-P(S)Ph_2$  (dppmS<sub>2</sub>), (4), is isostructural with compound (2) and its crystal structure is reported herein.



The structure of (4) is comprised of discrete monomers with no short intermolecular interactions. A view of the molecular structure of (4) is shown in Fig. 1, with a packing view shown in Fig. 2. The P—S bond lengths [P(1)—S(1) 1.948 (1) and P(2)—S(2) 1.909 (1) Å] in (4) are slightly shorter than the corresponding P—Se bond distances found in compounds (2) [average P—Se 2.100 (4) Å] and (3) [P—Se 2.103 (1) Å]. All other bond lengths are similar to those observed in compounds (1)–(3) and deserve no special comment.

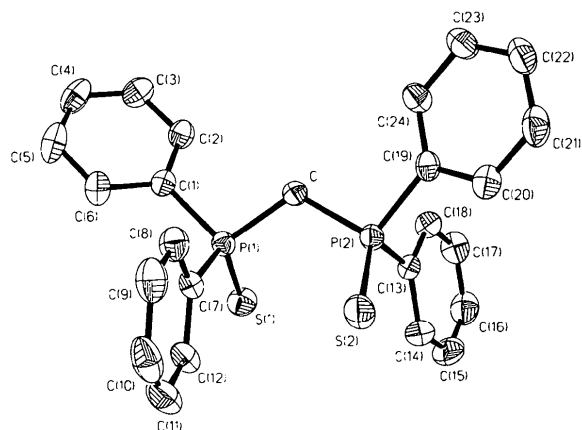


Fig. 1. The structure of compound (4) showing the atom-numbering scheme and 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

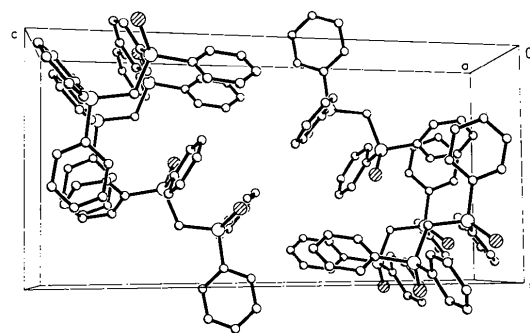


Fig. 2. A view of the packing in compound (4).

The coordination geometry around each P atom in (4) is distorted tetrahedral, with all bond angles similar to the corresponding angles in compound (2). The only significant difference between the dpmm moiety of (4) and dpmm, (1), itself is the P—C—P bond angle. In (1), the P—C—P angle is 106.2 (3)°, whereas in (4), the angle is several degrees larger with a value of 118.4 (2)° [P(1)—C—P(2)]. In compound (2), the P—C—P angle has a value of 117.9 (6)°, similar to (4), whereas in compound (3), the angle has an intermediate value of 110.6 (2)°. The structures of compounds (2) and (4) are also similar with regard to the Se—P···P—Se and S—P···P—S torsion angles, which are 95.4 and 95.64 (5)°, respectively.

## Experimental

A sample of sulfur (0.143 g, 4.46 mmol) was added to a solution of dpmm (0.858 g, 2.23 mmol) in toluene (10 ml) at room temperature resulting in a color change from colorless to pale yellow. The mixture was then heated and stirred for about 30 min. On cooling to 243 K, a crystalline pale-cream solid was obtained after a few days (0.790 g, 80% yield). X-ray quality crystals of (4) were subsequently obtained in near quantitative yield from the reaction between bis(biphenylene)Br (Carmalt, Cowley, Decken, Lawson & Norman, 1996) and dpmmS<sub>2</sub> in CH<sub>2</sub>Cl<sub>2</sub>, after solvent diffusion of hexane. Elemental analysis: found C 66.55, H 4.67%; calculated for C<sub>25</sub>H<sub>22</sub>P<sub>2</sub>S<sub>2</sub> C 66.95, H 4.95%.

### Crystal data

C<sub>25</sub>H<sub>22</sub>P<sub>2</sub>S<sub>2</sub>M<sub>r</sub> = 448.49

Monoclinic

P2<sub>1</sub>/n

a = 9.527 (1) Å

b = 10.731 (1) Å

c = 22.525 (2) Å

β = 99.63 (1)°

V = 2270.4 (4) Å<sup>3</sup>

Z = 4

D<sub>x</sub> = 1.312 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 9–12°

μ = 0.385 mm<sup>-1</sup>

T = 293 (2) K

Irregular

0.60 × 0.54 × 0.35 mm

Colorless

### Data collection

Enraf–Nonius CAD-4 diffractometer

2θ/ω scans

Absorption correction: none

5331 measured reflections

3976 independent reflections

3052 observed reflections

[I &gt; 2σ(I)]

R<sub>int</sub> = 0.024θ<sub>max</sub> = 24.97°

h = -1 → 11

k = -1 → 12

l = -26 → 26

3 standard reflections

frequency: 60 min

intensity decay: 5%

### Refinement

Refinement on F<sup>2</sup>R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.046wR(F<sup>2</sup>) = 0.108

S = 1.124

(Δ/σ)<sub>max</sub> = 0.001Δρ<sub>max</sub> = 0.409 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.168 e Å<sup>-3</sup>

Extinction correction: none

3974 reflections

329 parameters

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.04P)<sup>2</sup> + 1.00P]where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3

Atomic scattering factors

from *International Tables for Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_j$$

|       | x           | y          | z          | U <sub>eq</sub> |
|-------|-------------|------------|------------|-----------------|
| P(1)  | 0.0083 (1)  | 0.9115 (1) | 0.2358 (1) | 0.041 (1)       |
| P(2)  | -0.0180 (1) | 0.7375 (1) | 0.1222 (1) | 0.044 (1)       |
| S(1)  | 0.1454 (1)  | 1.0281 (1) | 0.2120 (1) | 0.060 (1)       |
| S(2)  | -0.1863 (1) | 0.8229 (1) | 0.0812 (1) | 0.071 (1)       |
| C     | 0.0171 (3)  | 0.7554 (3) | 0.2042 (1) | 0.043 (1)       |
| C(1)  | 0.0350 (3)  | 0.8840 (2) | 0.3164 (1) | 0.045 (1)       |
| C(2)  | 0.1452 (3)  | 0.8092 (3) | 0.3432 (1) | 0.056 (1)       |
| C(3)  | 0.1691 (4)  | 0.7908 (4) | 0.4048 (1) | 0.070 (1)       |
| C(4)  | 0.0857 (4)  | 0.8476 (4) | 0.4400 (2) | 0.076 (1)       |
| C(5)  | -0.0233 (4) | 0.9221 (4) | 0.4143 (2) | 0.079 (1)       |
| C(6)  | -0.0490 (4) | 0.9417 (3) | 0.3525 (2) | 0.064 (1)       |
| C(7)  | -0.1759 (3) | 0.9570 (2) | 0.2154 (1) | 0.045 (1)       |
| C(8)  | -0.2833 (3) | 0.8794 (3) | 0.2285 (1) | 0.058 (1)       |
| C(9)  | -0.4241 (3) | 0.9100 (4) | 0.2073 (2) | 0.070 (1)       |
| C(10) | -0.4567 (4) | 1.0162 (4) | 0.1745 (2) | 0.079 (1)       |
| C(11) | -0.3523 (4) | 1.0932 (4) | 0.1625 (2) | 0.076 (1)       |
| C(12) | -0.2110 (4) | 1.0643 (3) | 0.1833 (1) | 0.059 (1)       |
| C(13) | 0.1439 (3)  | 0.7815 (2) | 0.0956 (1) | 0.047 (1)       |
| C(14) | 0.1431 (4)  | 0.8785 (3) | 0.0553 (1) | 0.065 (1)       |
| C(15) | 0.2635 (5)  | 0.9086 (4) | 0.0323 (2) | 0.083 (1)       |
| C(16) | 0.3861 (5)  | 0.8432 (4) | 0.0497 (2) | 0.079 (1)       |
| C(17) | 0.3898 (4)  | 0.7484 (4) | 0.0897 (2) | 0.070 (1)       |
| C(18) | 0.2704 (3)  | 0.7160 (3) | 0.1129 (1) | 0.055 (1)       |
| C(19) | -0.0338 (3) | 0.5697 (3) | 0.1118 (1) | 0.044 (1)       |
| C(20) | -0.1043 (3) | 0.5269 (3) | 0.0567 (1) | 0.060 (1)       |
| C(21) | -0.1212 (4) | 0.4011 (4) | 0.0460 (2) | 0.072 (1)       |
| C(22) | -0.0712 (4) | 0.3173 (3) | 0.0897 (2) | 0.070 (1)       |
| C(23) | -0.0011 (4) | 0.3576 (3) | 0.1441 (2) | 0.067 (1)       |
| C(24) | 0.0188 (3)  | 0.4837 (3) | 0.1555 (1) | 0.056 (1)       |

Table 2. Selected geometric parameters (Å, °)

|                                                             |            |                  |           |
|-------------------------------------------------------------|------------|------------------|-----------|
| P(1)—C(7)                                                   | 1.806 (3)  | P(2)—C(13)       | 1.808 (3) |
| P(1)—C(1)                                                   | 1.814 (3)  | P(2)—C(19)       | 1.819 (3) |
| P(1)—C                                                      | 1.827 (3)  | P(2)—C           | 1.831 (3) |
| P(1)—S(1)                                                   | 1.948 (1)  | P(2)—S(2)        | 1.941 (1) |
| C—C bond distances: 1.354 (4)–1.393 (6), average 1.374 (12) |            |                  |           |
| C(7)—P(1)—C(1)                                              | 105.4 (1)  | C—P(2)—S(2)      | 115.3 (1) |
| C(7)—P(1)—C                                                 | 104.8 (1)  | P(1)—C—P(2)      | 118.4 (2) |
| C(1)—P(1)—C                                                 | 103.5 (1)  | C(2)—C(1)—P(1)   | 120.1 (2) |
| C(7)—P(1)—S(1)                                              | 115.1 (1)  | C(6)—C(1)—P(1)   | 121.3 (2) |
| C(1)—P(1)—S(1)                                              | 113.09 (9) | C(12)—C(7)—P(1)  | 120.1 (2) |
| C—P(1)—S(1)                                                 | 113.9 (1)  | C(8)—C(7)—P(1)   | 120.2 (2) |
| C(13)—P(2)—C(19)                                            | 105.8 (1)  | C(14)—C(13)—P(2) | 120.2 (2) |
| C(13)—P(2)—C                                                | 106.5 (1)  | C(18)—C(13)—P(2) | 121.5 (2) |
| C(19)—P(2)—C                                                | 103.4 (1)  | C(24)—C(19)—P(2) | 123.9 (2) |
| C(13)—P(2)—S(2)                                             | 113.7 (1)  | C(20)—C(19)—P(2) | 117.4 (2) |
| C(19)—P(2)—S(2)                                             | 111.22 (9) |                  |           |

H atoms were located in the difference synthesis map and their coordinates refined with common isotropic displacement parameters. C—H bond distances range from 0.85 to 0.96 (3) Å, with an average value of 0.91 (3) Å.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *XCAD4 Software* (Siemens, 1993). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXL93*.

The authors wish to thank NSF and the Robert A. Welch Foundation for generous financial support, and SERC for a studentship (CJC). NCN also thanks the Royal Society for additional supporting funds.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1168). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Carmalt, C. J., Cowley, A. H., Decken, A., Lawson, Y. G. & Norman, N. C. (1996). Unpublished results.  
 Carroll, P. J. & Titus, D. D. (1971). *J. Chem. Soc. Dalton Trans.* pp. 824–829.  
 Colton, R., Hoskins, B. F. & Panagiotidou, P. (1987). *Aust. J. Chem.* **40**, 1909–1912.  
 Enraf–Nonius (1993). *CAD-4 EXPRESS*. Version 5.1. Enraf–Nonius, Delft, The Netherlands.  
 Schmidbaur, H., Reber, G., Schier, A., Wagner, F. E. & Müller, G. (1988). *Inorg. Chim. Acta*, **147**, 143–150.  
 Sheldrick, G. M. (1990). *Acta Cryst. A* **46**, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1995). *SHELXTL-Plus*. Release 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1993). *XCAD4 Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1996). **C52**, 933–935

## Absolute Configuration of 6-*O*-*p*-Bromobenzoylsalvileucolide Methyl Ester

ANTHONY LINDEN,\* MATHIAS JUCH AND PETER RÜEDI

*Organisch-chemisches Institut, Universität Zürich,  
 Winterthurerstrasse 190, CH-8057 Zürich, Switzerland.  
 E-mail: alinden@oci.unizh.ch*

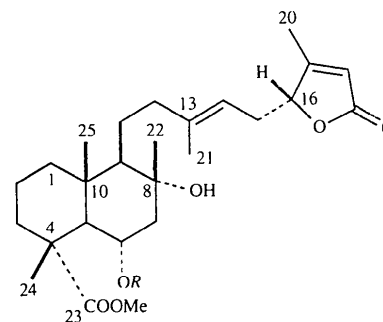
(Received 11 July 1995; accepted 10 October 1995)

## Abstract

The absolute configuration of the title compound, methyl 8-(4-bromobenzoyloxy)-5-[5-(2,5-dihydro-3-methyl-5-oxo-2-furanyl)-3-methyl-3-pentenyl]-6-hydroxy-1,4a,6-trimethyldecahydronaphthalene-1-carboxylate,  $C_{33}H_{43}BrO_7$ , a derivative of the sesterterpenoid salvileucolide methyl ester isolated from the aerial parts of two Iranian *Salvia* species, has been determined at 173 K. The compound belongs to the normal (10*R*) series and the configuration at C(16) is also *R*.

## Comment

Salvileucolide methyl ester, (I), has been isolated as the major constituent from the aerial parts of *Salvia hypoleuca* (Rustaiyan, Niknejad, Nazarians, Jakupovic & Bohlmann, 1982) and *S. sahendica* (Matloubi Moghaddam, Zaynizadeh & Rüedi, 1995), both species being endemic to Iran. In the previous studies, the conformation of (I) was assigned by spectroscopic methods, especially by extensive  $^1H$  and  $^{13}C$  NMR experiments. The relative configuration at C(16) and the absolute configuration of the molecule, however, remained undetermined. Chemical transformation of (I) to an established reference compound (e.g. a suitable lab-dane derivative) and comparison of the chiroptical data would quite easily provide the absolute configuration of the skeleton. The determination of the stereochemistry at C(16) [e.g. by degradation of (I) to a 3-hydroxy-pentanoic acid derivative], however, is not expected to be unambiguously feasible without difficulties, probably due to racemization. This fact prompted us to undertake the X-ray analysis of the 6-*O*-*p*-bromobenzoyl ester, (II), where the Br atom greatly facilitates the direct determination of the absolute configuration. We now report the crystal structure of (II), which thereby establishes the structure of (I).



(I) *R* = H  
 (II) *R* = *p*-Br-benzoyl

The correct absolute configuration of (II), determined by refinement of the structure and the enantiopole parameter according to the method of Flack (1983), is depicted in Fig. 1. The compound has the 4*R*,6*S*,8*R*,9*R*,10*R*,13*E*,16*R* configuration. Therefore, as previously assumed (Rustaiyan *et al.*, 1982; Matloubi Moghaddam *et al.*, 1995), the natural product (I) belongs to the normal cyclic sesterterpenoid (10*R*) series and has an *R* configuration at C(16).

The bond lengths and angles generally have normal values. The two most significant exceptions are the values of 101.2(4) and 130.3(6)° for C(3)—C(4)—C(23) and C(18)—C(17)—C(20), respectively. These angles probably result from steric interactions between the two neighbouring ester groups in the first case and between the coplanar methyl group, C(20), and