

In spite of the relatively large sample, the diffraction data were not extensive, yielding only 805 reflections with  $I > 3\sigma(I)$  out of 8063 measured. The large thermal motion of the molecules required the refinement of anisotropic displacement parameters. The least-squares refinement was completed without restrictions on the positional parameters, but with the displacement parameters restrained between adjacent atoms (Rollett, 1970). The good agreement between equivalent bonds within each molecule and between molecules indicated that there was no need to add geometric restraints.

The structure was solved by *SHELXS86* (Sheldrick, 1985) and refined by *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Molecular graphics: *CAMERON* (Pearce, Watkin & Prout, 1992).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1254). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## (1*S*,2*S*)-(+)-1,2-Bis[(dicyclohexyl)phosphinomethyl]cyclopentane.2CS<sub>2</sub> and (±)-1,2-Bis[(dicyclohexyl)phosphinomethyl]cyclopentane

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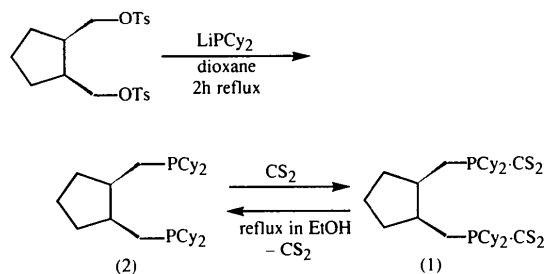
(Received 13 January 1995; accepted 17 March 1995)

## Abstract

The molecular structures of C<sub>33</sub>H<sub>56</sub>P<sub>2</sub>S<sub>4</sub>, (1), and C<sub>31</sub>H<sub>56</sub>P<sub>2</sub>, (2), are reported. The structure analysis of (1) is the first of a CS<sub>2</sub> adduct of an optically active diphosphine. The optically active compound (1) is not a simple CS<sub>2</sub> adduct but contains two zwitterionic structures, *i.e.* 1,2-cyclopentylenedimethylenebis[(dicyclohexyl)phosphoniocarbodithioate], C<sub>5</sub>H<sub>8</sub>[CH<sub>2</sub>P<sup>+</sup>(C<sub>6</sub>H<sub>11</sub>)<sub>2</sub>(CS<sub>2</sub>)<sup>-</sup>]<sub>2</sub>.

## Comment

Optically active peralkyl diphosphines are electron-rich phosphorus compounds and are expected to be effective ligands in asymmetric catalyses, such as asymmetric hydrogenation of various ketones (Tani, Suwa, Yamagata & Otsuka, 1982). The title compounds, (1) and (2), were prepared according to the scheme shown below (see *Experimental* also).



The molecular structure of (1) is shown in Fig. 1. The configurations of the two chiral centres, C(2) and C(6), are *S*. The bond angles around the two P atoms [105.7(3)–112.4(3)°] show that they have almost tetrahedral geometry. The four P—C distances at each P atom are almost equal [1.821(6)–1.835(6) Å]. The sums of the three angles around the CS<sub>2</sub> C atoms are 359.9 for C(32) and 360.0° for C(33). Thus, the environments of the C atoms of the CS<sub>2</sub> moieties are planar. The four C—S distances are also similar [1.639(6)–1.667(6) Å] and have values between those of a C—S single bond [1.80 Å for (CH<sub>3</sub>)<sub>2</sub>S; Pieger & Hayashi, 1961] and a C=S double bond [1.560(3) Å for CS<sub>2</sub>; Baenziger & Duax, 1968]. The average value of the P—C bond lengths is significantly shorter for the CS<sub>2</sub> adduct (1) than for the free diphosphine (2). These results suggest that the CS<sub>2</sub> complex is not a loose adduct but contains two zwitterionic structures with a negative charge spread over the CS<sub>2</sub> group and a positive charge on the P atom. A similar structure has been found in (C<sub>2</sub>H<sub>5</sub>)<sub>3</sub>P·CS<sub>2</sub> (Margulis & Templeton, 1962). The cyclopentane ring has a half-chair conformation with two dicyclohexylphosphinomethyl groups in axial positions.

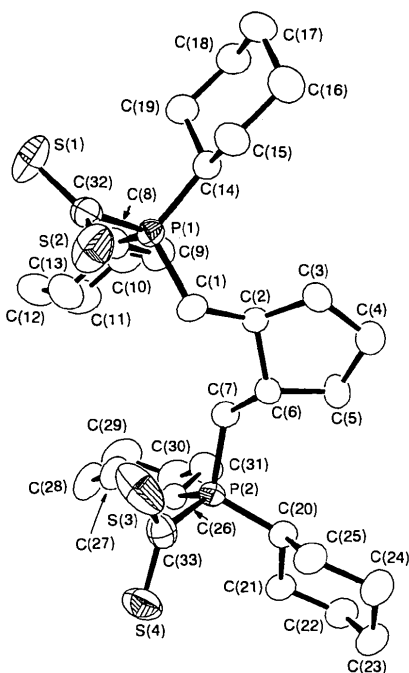


Fig. 1. Molecular structure of (1) (ORTEP; Johnson, 1976). Displacement ellipsoids are plotted at the 50% probability level.

The molecular structure of (2) is shown in Fig. 2. The molecular symmetry is *C*<sub>2</sub> with the C(1) atom lying on a crystallographic twofold axis. The cyclopentane ring has a half-chair conformation with two dicyclohexylphosphinomethyl groups in equatorial positions.

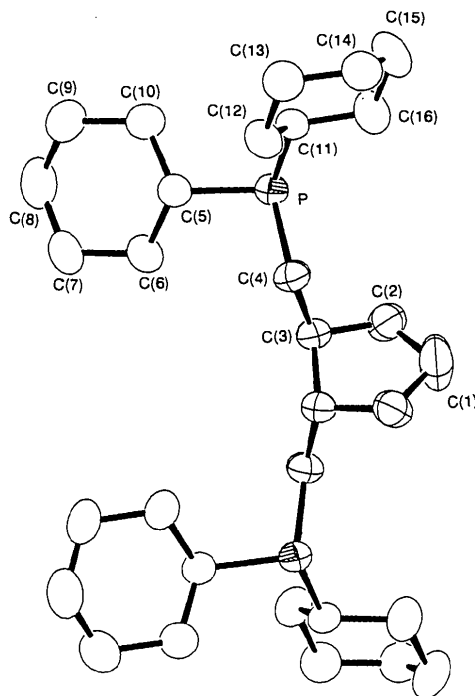


Fig. 2. Molecular structure of (2) (ORTEP; Johnson, 1976). Displacement ellipsoids are plotted at the 50% probability level.

## Experimental

The optically active title compound (1) was obtained as air-stable red crystals by treating the parent optically active phosphine, (1*S*,2*S*)-(+)-1,2-bis(dicyclohexylphosphinomethyl)cyclopentane, with CS<sub>2</sub> in absolute ethanol (48%, m.p. 393.6–396 K). The free phosphine { $[\alpha]_D^{25} +69.1^\circ$  (*c* 0.54, CHCl<sub>3</sub>); <sup>31</sup>P NMR (109.25 MHz, CDCl<sub>3</sub>, 308 K, 85% H<sub>3</sub>PO<sub>4</sub> as an external standard)  $\delta -7.20$  p.p.m.} can be easily recovered as an air-sensitive colourless solid by refluxing the CS<sub>2</sub> adduct (1) in ethanol and removing the solvent *in vacuo* (*cf.* Tani *et al.*, 1989). The racemic compound (2) was prepared from LiPPh<sub>2</sub> and the ditosylates of (±)-1,2-bis(hydroxymethyl)cyclopentane, and was isolated in a 25% yield after recrystallization from absolute hexanes as colourless crystals (m.p. 390–392 K). The density *D<sub>m</sub>* of compound (1) was measured by flotation in aqueous CsCl.

## Compound (1)

### Crystal data

C<sub>33</sub>H<sub>56</sub>P<sub>2</sub>S<sub>4</sub>

*M<sub>r</sub>* = 643.02

Monoclinic

*P*2<sub>1</sub>

*a* = 10.000(3) Å

*b* = 25.705(6) Å

*c* = 7.323(2) Å

$\beta$  = 107.84(3)°

*V* = 1791.9(8) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.192 Mg m<sup>-3</sup>

*D<sub>m</sub>* = 1.205(1) Mg m<sup>-3</sup>

Cu *K*α radiation

$\lambda$  = 1.54178 Å

Cell parameters from 41

reflections

$\theta$  = 20.0–28.7°

$\mu$  = 3.40 mm<sup>-1</sup>

*T* = 299(2) K

Prism

0.43 × 0.07 × 0.05 mm

Red

## Data collection

Rigaku AFC-5R diffractometer  
 $\omega$ - $2\theta$  scans  
 Absorption correction:  
 $\psi$  scan (North, Phillips & Mathews, 1968)  
 $T_{\min} = 0.886$ ,  $T_{\max} = 1.000$   
 6268 measured reflections  
 5789 independent reflections

4941 observed reflections  
 $[F_o > 2\sigma(F_o)]$   
 $R_{\text{int}} = 0.021$   
 $\theta_{\max} = 63.0^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -29 \rightarrow 29$   
 $l = 0 \rightarrow 8$   
 3 standard reflections  
 monitored every 200 reflections  
 intensity decay: none

## Refinement

Refinement on  $F$   
 $R = 0.047$   
 $wR = 0.048$   
 $S = 0.771$   
 4941 reflections  
 519 parameters  
 Only coordinates of H atoms refined  
 $w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.0019$   
 $\Delta\rho_{\max} = 0.84 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.62 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ ) for (1)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
P(1)	0.8032 (1)	0.2500	0.6434 (2)	0.035 (1)
P(2)	0.9125 (1)	0.4791 (1)	0.5149 (2)	0.034 (1)
C(1)	0.7349 (6)	0.3161 (2)	0.6001 (8)	0.040 (3)
C(2)	0.6902 (4)	0.3367 (2)	0.3937 (6)	0.033 (2)
C(3)	0.5340 (5)	0.3280 (2)	0.2829 (8)	0.044 (2)
C(4)	0.4998 (6)	0.3693 (2)	0.1266 (9)	0.055 (3)
C(5)	0.6155 (6)	0.4094 (2)	0.1812 (9)	0.054 (3)
C(6)	0.7044 (4)	0.3963 (2)	0.3859 (7)	0.035 (2)
C(7)	0.8599 (4)	0.4130 (2)	0.4355 (8)	0.038 (2)
C(8)	0.9863 (4)	0.2492 (2)	0.6444 (7)	0.042 (2)
C(9)	1.0076 (5)	0.2643 (2)	0.4534 (9)	0.053 (3)
C(10)	1.1642 (6)	0.2561 (3)	0.4682 (12)	0.072 (4)
C(11)	1.2612 (7)	0.2865 (3)	0.6347 (14)	0.082 (5)
C(12)	1.2346 (6)	0.2729 (3)	0.8218 (14)	0.080 (4)
C(13)	1.0806 (6)	0.2813 (3)	0.8104 (10)	0.064 (3)
C(14)	0.6990 (4)	0.2064 (2)	0.4578 (7)	0.036 (2)
C(15)	0.5534 (5)	0.1966 (2)	0.4869 (8)	0.044 (3)
C(16)	0.4633 (6)	0.1642 (2)	0.3233 (9)	0.052 (3)
C(17)	0.5341 (6)	0.1123 (2)	0.3077 (9)	0.053 (3)
C(18)	0.6760 (6)	0.1226 (2)	0.2781 (9)	0.050 (3)
C(19)	0.7716 (6)	0.1546 (2)	0.4421 (9)	0.048 (3)
C(20)	0.8053 (5)	0.5259 (2)	0.3420 (7)	0.036 (2)
C(21)	0.8793 (5)	0.5774 (2)	0.3238 (9)	0.044 (3)
C(22)	0.7816 (6)	0.6108 (2)	0.1689 (9)	0.050 (3)
C(23)	0.6428 (6)	0.6217 (2)	0.2113 (9)	0.051 (3)
C(24)	0.5701 (6)	0.5714 (2)	0.2307 (9)	0.052 (3)
C(25)	0.6676 (5)	0.5373 (2)	0.3866 (8)	0.044 (3)
C(26)	1.0967 (5)	0.4844 (2)	0.5249 (7)	0.038 (2)
C(27)	1.1928 (5)	0.4519 (2)	0.6901 (9)	0.054 (3)
C(28)	1.3463 (5)	0.4619 (3)	0.7025 (11)	0.071 (4)
C(29)	1.3737 (7)	0.4508 (3)	0.5165 (13)	0.083 (4)
C(30)	1.2770 (6)	0.4818 (3)	0.3533 (11)	0.071 (4)
C(31)	1.1220 (6)	0.4712 (2)	0.3334 (9)	0.055 (3)
C(32)	0.7929 (5)	0.2275 (2)	0.8764 (7)	0.045 (2)
S(1)	0.8953 (2)	0.1766 (1)	0.9647 (2)	0.072 (1)
S(2)	0.6806 (2)	0.2583 (1)	0.9637 (2)	0.068 (1)
C(33)	0.9022 (5)	0.4921 (2)	0.7567 (8)	0.050 (3)
S(3)	0.8335 (2)	0.4471 (1)	0.8591 (2)	0.097 (1)
S(4)	0.9706 (2)	0.5498 (1)	0.8385 (2)	0.071 (1)

Table 2. Selected geometric parameters ( $\text{Å}$ ,  $^\circ$ ) for (1)

P(1)—C(1)	1.824 (6)	P(1)—C(8)	1.828 (6)
P(1)—C(14)	1.822 (6)	P(1)—C(32)	1.835 (5)
P(2)—C(7)	1.821 (6)	P(2)—C(20)	1.834 (6)
P(2)—C(26)	1.825 (5)	P(2)—C(33)	1.835 (6)
C(32)—S(1)	1.664 (6)	C(32)—S(2)	1.654 (6)
C(33)—S(3)	1.639 (6)	C(33)—S(4)	1.667 (6)
C(1)—P(1)—C(8)	109.4 (3)	C(1)—P(1)—C(14)	110.2 (3)
C(1)—P(1)—C(32)	109.3 (3)	C(8)—P(1)—C(14)	109.9 (3)
C(8)—P(1)—C(32)	109.5 (3)	C(14)—P(1)—C(32)	108.5 (2)
C(7)—P(2)—C(20)	109.9 (3)	C(7)—P(2)—C(26)	105.7 (3)
C(7)—P(2)—C(33)	112.4 (3)	C(20)—P(2)—C(26)	110.4 (3)
C(20)—P(2)—C(33)	110.7 (3)	C(26)—P(2)—C(33)	107.6 (2)
P(1)—C(1)—C(2)	118.2 (3)	P(2)—C(7)—C(6)	120.4 (3)
P(1)—C(8)—C(9)	114.5 (4)	P(1)—C(14)—C(15)	110.0 (4)
P(1)—C(8)—C(13)	111.7 (4)	P(1)—C(14)—C(19)	114.4 (3)
P(2)—C(20)—C(21)	115.3 (4)	P(2)—C(26)—C(27)	111.7 (3)
P(2)—C(20)—C(25)	110.5 (4)	P(2)—C(26)—C(31)	113.2 (3)
P(1)—C(32)—S(1)	113.9 (2)	P(1)—C(32)—S(2)	116.5 (4)
S(1)—C(32)—S(2)	129.5 (4)	P(2)—C(33)—S(3)	118.0 (3)
P(2)—C(33)—S(4)	111.7 (3)	S(3)—C(33)—S(4)	130.3 (3)

## Compound (2)

## Crystal data

$\text{C}_{31}\text{H}_{56}\text{P}_2$   
 $M_r = 490.73$   
 Monoclinic  
 $C2/c$   
 $a = 29.558 (2) \text{ Å}$   
 $b = 9.5439 (5) \text{ Å}$   
 $c = 11.039 (1) \text{ Å}$   
 $\beta = 101.157 (9)^\circ$   
 $V = 3055.2 (5) \text{ Å}^3$   
 $Z = 4$   
 $D_x = 1.067 \text{ Mg m}^{-3}$

Cu  $K\alpha$  radiation

$\lambda = 1.54178 \text{ Å}$   
 Cell parameters from 49 reflections  
 $\theta = 25.46\text{--}28.86^\circ$   
 $\mu = 1.387 \text{ mm}^{-1}$   
 $T = 294 (1) \text{ K}$   
 Prism  
 $0.53 \times 0.40 \times 0.23 \text{ mm}$   
 Colourless

## Data collection

Rigaku AFC-5R diffractometer  
 $\omega < 60^\circ (2\theta) < \omega$ - $2\theta$  scans  
 Absorption correction:  
 $\psi$  scans (North, Phillips & Mathews, 1968)  
 $T_{\min} = 0.700$ ,  $T_{\max} = 0.997$   
 4662 measured reflections  
 2279 independent reflections

2081 observed reflections  
 $[F_o > 2\sigma(F_o)]$   
 $R_{\text{int}} = 0.040$   
 $\theta_{\max} = 60.0^\circ$   
 $h = -33 \rightarrow 33$   
 $k = -10 \rightarrow 10$   
 $l = 0 \rightarrow 12$   
 3 standard reflections  
 monitored every 200 reflections  
 intensity decay: none

## Refinement

Refinement on  $F$   
 $R = 0.054$   
 $wR = 0.050$   
 $S = 2.578$   
 2081 reflections  
 262 parameters  
 All H-atom parameters refined  
 $w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{\max} = 0.0002$   
 $\Delta\rho_{\max} = 0.46 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.27 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>) for (2)

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

	x	y	z	U <sub>eq</sub>
C(1)	1/2	-0.1899 (5)	1/4	0.095 (4)
C(2)	0.4882 (1)	-0.0968 (3)	0.3497 (3)	0.060 (2)
C(3)	0.4838 (1)	0.0508 (3)	0.2968 (2)	0.042 (1)
C(4)	0.4339 (1)	0.0860 (3)	0.2348 (3)	0.046 (1)
P	0.39587 (2)	0.11679 (7)	0.34781 (7)	0.0428 (3)
C(5)	0.3922 (1)	0.3117 (3)	0.3470 (3)	0.046 (1)
C(6)	0.4394 (1)	0.3787 (3)	0.3894 (4)	0.064 (2)
C(7)	0.4362 (1)	0.5384 (3)	0.3957 (4)	0.074 (2)
C(8)	0.4038 (1)	0.5846 (4)	0.4788 (4)	0.086 (3)
C(9)	0.3566 (2)	0.5216 (4)	0.4356 (7)	0.105 (3)
C(10)	0.3594 (1)	0.3613 (4)	0.4292 (5)	0.085 (3)
C(11)	0.3383 (1)	0.0664 (3)	0.2571 (3)	0.044 (1)
C(12)	0.3247 (1)	0.1266 (3)	0.1265 (3)	0.056 (2)
C(13)	0.2764 (1)	0.0795 (3)	0.0643 (3)	0.061 (2)
C(14)	0.2723 (1)	-0.0782 (3)	0.0612 (3)	0.062 (2)
C(15)	0.2850 (1)	-0.1397 (4)	0.1903 (3)	0.070 (2)
C(16)	0.3334 (1)	-0.0931 (3)	0.2539 (3)	0.059 (2)

Table 4. Selected geometric parameters (Å, °) for (2)

C(1)—C(2)	1.507 (4)	C(2)—C(3)	1.521 (4)
C(3)—C(3 <sup>1</sup> )	1.540 (4)	C(3)—C(4)	1.537 (4)
P—C(4)	1.857 (3)	P—C(5)	1.863 (3)
P—C(11)	1.862 (3)		
C(2)—C(1)—C(2 <sup>1</sup> )	107.8 (2)	C(1)—C(2)—C(3)	106.3 (2)
C(2)—C(3)—C(4)	112.2 (3)	C(2)—C(3)—C(3 <sup>1</sup> )	103.8 (2)
C(4)—C(3)—C(3 <sup>1</sup> )	111.6 (2)	C(3)—C(4)—P	112.9 (1)
C(4)—P—C(5)	101.4 (1)	C(4)—P—C(11)	102.0 (1)
C(5)—P—C(11)	102.0 (1)	P—C(5)—C(6)	111.6 (2)
P—C(5)—C(10)	110.6 (2)	P—C(11)—C(12)	117.1 (2)
P—C(11)—C(16)	110.1 (2)		

Symmetry code: (i) 1 - x, y, 1/2 - z.

The positional parameters for all non-H atoms were determined by direct methods (Sheldrick, 1985) for both compounds. The refinements were carried out by full-matrix least-squares techniques (Imoto, 1990). The  $\Delta f'$  and  $\Delta f''$  components of anomalous dispersion were included in the calculation for P and S atoms of compound (1) and for all non-H atoms of compound (2) (Cromer, 1974).

The chosen model for compound (1) has residuals of  $R = 0.047$  and  $wR = 0.048$ , while the other enantiomeric structure gave  $R$  and  $wR$  values of 0.056 and 0.060, respectively. When the  $R$ -factor ratio test (Hamilton, 1965) is applied to  $wR$ , the latter enantiomeric model can be rejected.

$F_o$  data were collected at the Faculty of Pharmaceutical Sciences, Osaka University. All calculations were carried out on an NEC ACOS S3700 computer at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1173). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 4,5-Diazafluoren-9-one

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## Abstract

The 4,5-diazafluoren-9-one molecules, C<sub>11</sub>H<sub>6</sub>N<sub>2</sub>O, are planar and lie in parallel planes. The presence of a C—H...N intermolecular hydrogen bond is observed in this structure.

## Comment

Extensive studies have been carried out on the synthesis and uses of the title compound, (I) (Eckhard &

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