Yamada & Kawazura, 1978). Among adjacent columns, short contacts are found between S(2) and C(7) of the original unit and N(1′), shifted by (**a** + **b**)/2, at distances of 3·477 (4) and 3·305 (4) Å respectively.

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Structure of 5,5,5′,5′-Tetramethyl-2,2′-bi-1,3,2λ⁵-dioxaphosphorinane 2,2′-Disulphide

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Abstract. $C_{10}H_{20}O_4P_2S_2$, monoclinic, *C*2/*m*, *a* = 11·861 (12), *b* = 8·765 (8), *c* = 7·685 (5) Å, β = 81·05 (10)°, *Z* = 2, *F*(000) = 348. The dioxaphosphorinane rings adopt a chair conformation. The P=S bond of 1·903 (1) Å is equatorial. The P—P bond is 2·182 (2) Å. The molecule has a centre of symmetry at the middle of this bond and a mirror plane which is perpendicular to **b**.

Introduction. The present determination continues investigations on the crystal and molecular structure of derivatives with a 1,3,2-dioxaphosphorinane ring (Cameron, Gałdecki & Karolak-Wojciechowska, 1976; Cameron & Karolak-Wojciechowska, 1977). The title compound was prepared by Stec & Zwierzak (1967); it crystallizes in two different crystal forms (Gałdecki & Karolak-Wojciechowska, 1971). In this paper we report the crystal and molecular structure of one of these crystalline forms. The crystal system and approximate cell dimensions were determined from Weissenberg photographs. 663 unique reflections were measured on a Picker FACS-1 four-circle diffractometer. 617 reflections with $I > 2\sigma(I)$ were employed in the analysis. The data were corrected for Lorentz–polarization effects, but not for absorption.

Systematic absences *hkl* with *h* + *k* = 2*n* indicate space groups *C*2/*m*, *Cm* or *C*2. A centrosymmetric structure was suggested by normalized structure factor statistics. The molecules occupy special positions and must be on a mirror plane and twofold axis. The structure was solved with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) which was applied to 196 reflections with $|E| > 1{\cdot}20$. The P and S atoms were located from the *E* map based on the phases obtained. All atoms other than H were located from the difference synthesis with phases based on these two atoms.

The atomic positions were refined by a full-matrix least-squares method, with anisotropic temperature factors for non-hydrogen atoms, until *R* dropped to 0·10. A difference synthesis then revealed all the H atoms. Refinement was continued with H atoms included with isotropic temperature factors. For the 617 observed reflections the final *R* = 0·074 and $R_w = (\sum w^{1/2}\Delta/\sum w^{1/2}|F_o|) = 0{\cdot}089$. The weighting function $w = 1/[\sigma^2(F) + 0{\cdot}0366F^2]$ was applied. The final parameters are listed in Tables 1 and 2.*

Discussion. Bond lengths and angles are shown in Table 3. Fig. 1 displays the atom labelling. The packing of the molecules is shown in Fig. 2.

The 1,3,2-dioxaphosphorinane rings adopt the chair conformation with torsion angles O(12)—P—O(11)—C(11) − 24·8 (2), P—O(11)—C(11)—C(12) −

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35062 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/80/071683-02$01.00

−44·7 (3) and O(11)–C(11)–C(12)–C(15) = 58·9 (4)°. The P–S bond vector is in an equatorial position with respect to the ring. The dihedral angles between the plane formed by O(11), C(11), O(12) and C(15) and the planes through (i) P, O(11) and O(12) and (ii) C(11), C(12) and C(15) are 20·8 (3) and 52·4 (3)° respectively. The marked flattening of the ring at P is consistent with an interaction between the equatorial S atom of one dioxaphosphorinane ring and the axial H atoms at C(11) and C(15) of the second ring. This intramolecular distance is 3·04 Å; the sum of the van der Waals radii (S···H) is 3·05 Å.

Table 1. *Final atomic positional parameters* ($\times 10^4$) *and isotropic thermal parameters* ($\times 10^3$) *with e.s.d.'s in parentheses*

	x	y	z	$U_{\rm iso}$ (Å^2)
P	844 (1)	0	397 (1)	22
S	734 (1)	0	2893 (1)	40
O(11)	1466 (2)	1440 (2)	−506 (2)	29
C(11)	2102 (2)	1395 (3)	−2291 (4)	48
C(12)	2853 (3)	0	−2609 (5)	29
C(13)	3380 (4)	0	−4552 (6)	48
C(14)	3786 (4)	0	−1461 (6)	40

Table 2. *Final* H-*atom positional parameters* ($\times 10^4$) *and isotropic thermal parameters* ($\times 10^3$) *with e.s.d.'s in parentheses*

	x	y	z	$U_{\rm iso}$ (Å^2)
H(111)	253 (2)	232 (3)	−271 (3)	30
H(112)	153 (2)	167 (3)	−313 (3)	30
H(131)	299 (4)	0	−543 (7)	50
H(132)	381 (3)	89 (4)	−493 (4)	50
H(141)	362 (4)	0	−7 (5)	40
H(142)	431 (2)	102 (4)	−165 (3)	40

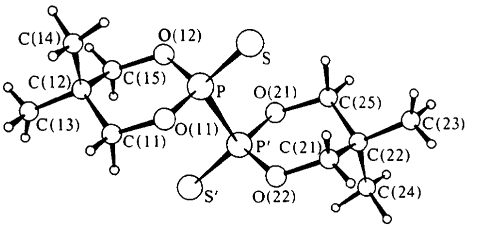

Fig. 1. The atom labelling for a molecule of $C_{10}H_{20}O_4P_2S_2$.

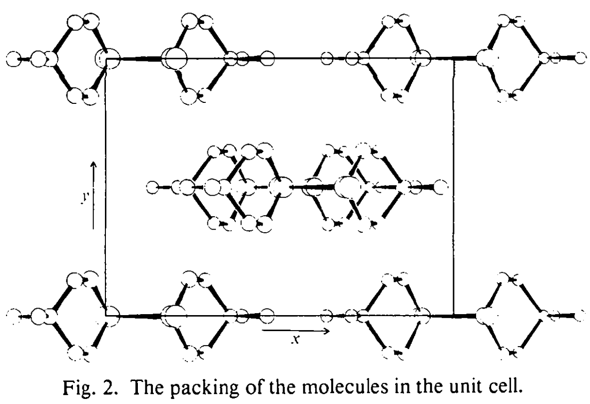

Fig. 2. The packing of the molecules in the unit cell.

Table 3. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

P–S	1·903 (1)	P'–P–S	111·1 (1)
O(11)–P	1·568 (2)	P'–P–O(11)	105·4 (1)
P–P	2·182 (2)	S–P–O(11)	113·6 (1)
O(11)–C(11)	1·460 (3)	O(12)–P–O(11)	107·2 (1)
C(11)–C(12)	1·509 (3)	C(11)–O(11)–P	122·2 (1)
C(12)–C(13)	1·526 (6)	C(12)–C(11)–O(11)	112·3 (2)
C(12)–C(14)	1·519 (6)	C(13)–C(12)–C(11)	107·4 (2)
C(11)–H(111)	0·98 (3)	C(14)–C(12)–C(11)	111·8 (2)
C(11)–H(112)	1·03 (3)	C(14)–C(12)–C(13)	110·1 (4)
C(13)–H(131)	0·88 (5)	C(11)–C(12)–C(15)	108·1 (2)
C(13)–H(132)	0·95 (3)		
C(14)–H(141)	1·06 (4)		
C(14)–H(142)	1·09 (3)		

The bond lengths and angles in the 1,3,2-dioxaphosphorinane ring are similar to those found in other compounds containing this ring (Grand, Martin, Robert & Tordjman, 1975; Cameron, Gałdecki & Karolak-Wojciechowska, 1976; Cameron & Karolak-Wojciechowska, 1977; Wieczorek, Sheldrick, Karolak-Wojciechowska, Mikołajczyk & Ziemnicka, 1979). The P–P bond of 2·182 (2) Å lies within the range observed in many compounds. The thiophosphoryl P=S bond of 1·903 (1) Å is similar to the value found for substituted phosphates.

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