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## Key indicators

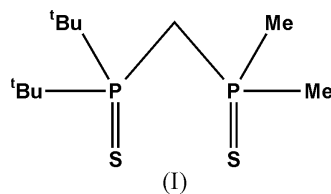
Single-crystal X-ray study  
 $T = 143\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.027  
 $wR$  factor = 0.072  
Data-to-parameter ratio = 23.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.(Di-*tert*-butylphosphino)(dimethylphosphino)methane  
disulfide

The title compound,  $\text{C}_{11}\text{H}_{26}\text{P}_2\text{S}_2$ , displays crystallographic mirror symmetry. Key bond lengths ( $\text{\AA}$ ) are  $\text{P1}-\text{C}(\text{methylene})$  1.8464 (18),  $\text{P1}-\text{C}(\text{butyl})$  1.8711 (13),  $\text{P2}-\text{C}(\text{methylene})$  1.8266 (18),  $\text{P2}-\text{C}(\text{methyl})$  1.7948 (15),  $\text{P1}=\text{S}$  1.9631 (8) and  $\text{P2}=\text{S}$  1.9552 (8), where P1 is the di-*tert*-butylphosphino P atom and P2 is the dimethylphosphino P atom. The angle  $\text{P}-\text{C}-\text{P}$  [ $124.31(10)^\circ$ ] is wide and  $\text{S}=\text{P}-\text{C}(\text{methylene})$  [ $108.91(6)^\circ$ ] narrow. Steric pressure from the *tert*-butyl groups may cause some of the molecular dimensions to depart from normal values.

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## Comment

The title compound, (I), arose as an unexpected product during studies of triphosphines (Krill *et al.*, 1993; see Experimental). The molecule, which is shown in Fig. 1, displays crystallographic mirror symmetry, with both P and S atoms and the central atom C1 lying in the mirror plane at  $y = 0.25$ . The configuration about the bond  $\text{P1}-\text{C1}$  is that atoms S1 and P2 are exactly synperiplanar; in contrast, atoms S2 and P1 are exactly antiperiplanar about  $\text{C1}-\text{P2}$ . A search of the Cambridge Structural Database (Allen & Kennard, 1993; Version of April 2002) revealed only one other bis(dialkyl)-phosphinomethane disulfide, namely the tetramethyl analogue, henceforth 'Me4' (Karsch *et al.*, 1992); this has no imposed symmetry (but twofold symmetry to a close approximation) and has  $\text{S}-\text{P}-\text{C}-\text{P}$  torsion angles of  $48.4$  and  $48.7(5)^\circ$ .



Selected molecular dimensions for (I) are presented in Table 1. The  $\text{P}=\text{S}$  and  $\text{P}-\text{C}(\text{methylene})$  bond lengths in (I) are comparable with those of Me4 [ $\text{P}=\text{S}$  1.958 (1) and 1.960 (1)  $\text{\AA}$ , and  $\text{P}-\text{C}$  1.822 (2) and 1.818 (2)  $\text{\AA}$ ], although  $\text{P}-\text{C2}$  is slightly longer. This may be attributable to the steric pressure of the bulky *tert*-butyl group, as may the appreciably different bond lengths  $\text{P1}-\text{C2}$  and  $\text{P2}-\text{C6}$ . The bond angle  $\text{S1}=\text{P1}-\text{C1}$  appears normal [*cf.* Me4;  $115.5(1)$  and  $116.6(1)^\circ$ ], but  $\text{S2}=\text{P2}-\text{C1}$  is unusually narrow at  $108.91(6)^\circ$ . The angle at the methylene C atom is wide [*cf.* Me4;  $119.0(1)^\circ$ ], which may also be caused by steric effects; comparable structures for a more detailed analysis are not available.

## Experimental

Attempts to recrystallize bis[(di-*tert*-butylphosphanyl)methyl]-methylphosphane trisulfide (Krill *et al.*, 1993) from diethyl ether-toluene led unexpectedly to crystals of the title compound.

### Crystal data

$C_{11}H_{26}P_2S_2$	Mo $K\alpha$ radiation
$M_r = 284.38$	Cell parameters from 52 reflections
Orthorhombic, $Pnma$	$\theta = 10\text{--}11.5^\circ$
$a = 18.298$ (5) Å	$\mu = 0.53$ mm $^{-1}$
$b = 12.792$ (3) Å	$T = 143$ (2) K
$c = 6.586$ (2) Å	Tablet, colourless
$V = 1541.6$ (7) Å $^3$	$0.7 \times 0.6 \times 0.5$ mm
$Z = 4$	
$D_x = 1.225$ Mg m $^{-3}$	

### Data collection

Stoe Stadi-4 diffractometer	$h = -2 \rightarrow 23$
$\omega/\theta$ scans	$k = -9 \rightarrow 16$
2346 measured reflections	$l = -8 \rightarrow 8$
1854 independent reflections	3 standard reflections
1712 reflections with $I > 2\sigma(I)$	frequency: 60 min
$R_{int} = 0.020$	intensity decay: none
$\theta_{max} = 27.5^\circ$	

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 0.6264P]$
$R[F^2 > 2\sigma(F^2)] = 0.027$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.072$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.37$ e Å $^{-3}$
1854 reflections	$\Delta\rho_{min} = -0.35$ e Å $^{-3}$
80 parameters	
H-atom parameters constrained	

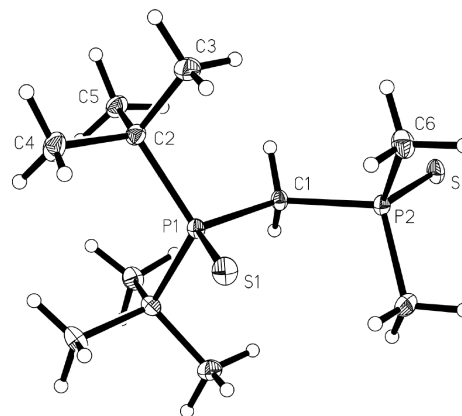
**Table 1**

Selected geometric parameters (Å, °).

P1—C1	1.8464 (18)	P2—C6	1.7948 (15)
P1—C2	1.8711 (13)	P2—C1	1.8266 (18)
P1—S1	1.9631 (8)	P2—S2	1.9552 (8)
C1—P1—C2	103.61 (5)	C6—P2—C1	108.37 (6)
C2—P1—C2 <sup>i</sup>	114.34 (8)	C6—P2—S2	112.87 (5)
C1—P1—S1	114.18 (6)	C1—P2—S2	108.91 (6)
C2—P1—S1	110.43 (5)	P2—C1—P1	124.31 (10)
C6 <sup>i</sup> —P2—C6	105.27 (11)		
S2—P2—C1—P1	180.0	S1—P1—C1—P2	0.0
C2—P1—C1—P2	120.17 (5)	C1—P1—C2—C4	−179.55 (10)

Symmetry code: (i)  $x, \frac{1}{2} - y, z$ .

The methylene H atoms were included using a riding model, while methyl H atoms were identified in difference syntheses and refined



**Figure 1**

The molecule of the title compound in the crystal. Ellipsoids represent 30% probability levels. H-atom radii are arbitrary. Only the asymmetric unit is numbered.

using idealized rigid methyl groups allowed to rotate but not tip. C—H bond lengths were fixed at 0.98 and 0.99 Å for methyl and methylene H atoms, respectively;  $U_{iso}(H)$  values were fixed at 1.2 times the  $U_{eq}$  values of the parent atom.

Data collection: *DIF4* (Stoe & Cie, 1992); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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