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Dimethyl 5,6-dihydro-2*H*-1,3-dithiolo[4,5-*b*][1,4]dithiin-2-ylphosphonate

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Key indicators

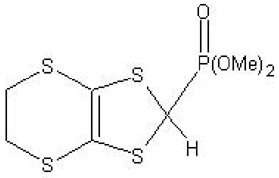
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.083
wR factor = 0.222
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_7H_{11}O_3PS_4$, the five-membered ring is in an envelope conformation. The average values of the S—Csp^3 and S—Csp^2 bond lengths are 1.813 (6) and 1.755 (8) Å, respectively. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds.

Comment

Phosphoranes of 1,3-dithiole derivatives are known as useful synthetic intermediates in the synthesis of tetrathiafulvalene (TTF) derivatives (Moore & Bryce, 1991). In continuation of our study of phosphoranes of 1,3-dithiole derivatives, the title compound, (I), has been synthesized. The crystal structure of the similar compound diethyl 5,6-dihydro-2*H*-1,3-dithiolo[4,5-*b*][1,4]dithiin-2-ylphosphonate (Shripad *et al.*, 1994), (II), has been reported previously. We present here the crystal structure of (I) (Fig. 1).



(I)

In (I), the five-membered ring is in an envelope conformation. The average values of the S—Csp^3 and S—Csp^2 bond lengths (Table 1) in (I) [1.813 (6) and 1.755 (8) Å, respectively] are similar to those in (II) [1.819 (7) and 1.76 (1) Å, respectively]. Atoms S1, S2, S3, S4, C3 and C4 are essentially coplanar, as expected, and atom C1 is almost coplanar with them. Atoms C2 and C5 lie on opposite sides of this plane. The length of the P1—O2 bond [1.459 (4) Å] proves its double-bond character, in contrast to the single bonds P1—O1 and P1—O3 (Table 1). The P—C bond in (I) [1.788 (5) Å] is slightly shorter than that in (II) [1.809 (2) Å]. The crystal packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 2).

Experimental

The title compound was prepared as a by-product in the preparation of BEDT-TTF [bis(ethylenedithio)tetrathiafulvalene; Varma *et al.*, (1987)]. 4,5-Ethylenedithio-1,3-dithiole-2-thione (1.0 g) was added to 30 ml trimethyl phosphite at 383 K for 2 h and the resulting solid BEDT-TTF was filtered off. The filtrate was concentrated under reduced pressure and purified by column chromatography (silica gel; chloroform/petroleum, 1:1) to obtain a viscous oil. The subsequent trituration with petroleum ether gave yellow crystals in the course of one day.

Crystal data

$C_7H_{11}O_3PS_4$
$M_r = 302.37$
Monoclinic, $P2_1/n$
$a = 9.8963$ (16) Å
$b = 9.3319$ (12) Å
$c = 13.4112$ (18) Å
$\beta = 95.234$ (12)°
$V = 1233.4$ (3) Å^3
$Z = 4$
$D_x = 1.628$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 69 reflections
$\theta = 5.0$–12.7°
$\mu = 0.88$ mm^{-1}
$T = 293$ (2) K
Plate, yellow
0.50 × 0.46 × 0.16 mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: ψ scan (*XSCANS*; Bruker, 1996)
$T_{\min} = 0.490$, $T_{\max} = 0.868$
3146 measured reflections
2395 independent reflections
1793 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.123$
$\theta_{\max} = 26.0°$
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 11$
$l = -16 \rightarrow 16$
3 standard reflections every 97 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.083$
$wR(F^2) = 0.222$
$S = 1.04$
2395 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1424P)^2 + 0.3726P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.81$ e Å^{-3}
$\Delta\rho_{\min} = -0.85$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.072 (9)

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.488 (10)	C5—P1	1.788 (5)
C1—S1	1.804 (6)	C5—S4	1.812 (5)
C2—S2	1.812 (6)	C5—S3	1.823 (5)
C3—C4	1.331 (8)	C6—O1	1.432 (8)
C3—S1	1.752 (5)	C7—O3	1.438 (7)
C3—S3	1.771 (5)	O1—P1	1.574 (4)
C4—S2	1.742 (5)	O2—P1	1.459 (4)
C4—S4	1.755 (5)	O3—P1	1.566 (4)
C2—C1—S1	115.8 (5)	C7—O3—P1	119.5 (4)
C1—C2—S2	112.9 (5)	O2—P1—O3	115.3 (2)
C4—C3—S1	129.3 (4)	O2—P1—O1	114.3 (2)
C4—C3—S3	116.9 (4)	O3—P1—O1	103.4 (2)
S1—C3—S3	113.7 (3)	O2—P1—C5	113.0 (2)
C3—C4—S2	126.7 (4)	O3—P1—C5	101.8 (2)
C3—C4—S4	117.5 (4)	O1—P1—C5	107.9 (2)
S2—C4—S4	115.6 (3)	C3—S1—C1	102.9 (3)
P1—C5—S4	117.3 (3)	C4—S2—C2	98.7 (3)
P1—C5—S3	109.0 (3)	C3—S3—C5	93.9 (2)
S4—C5—S3	108.3 (2)	C4—S4—C5	94.8 (2)
C6—O1—P1	121.0 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O2$^{\rm i}$	0.97	2.53	3.484 (8)	168
C5—H5A···O2$^{\rm ii}$	0.98	2.29	3.234 (5)	162

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, -y, -z$.

All H atoms were positioned geometrically and allowed to ride on their attached atoms, with C—H = 0.96–0.98 Å and $U_{\rm iso}$(H) = 1.2–1.5$U_{\rm eq}$(C). The quality of the crystal (and hence of the diffraction data) was poor, leading to a high *R* factor in the final refinement.

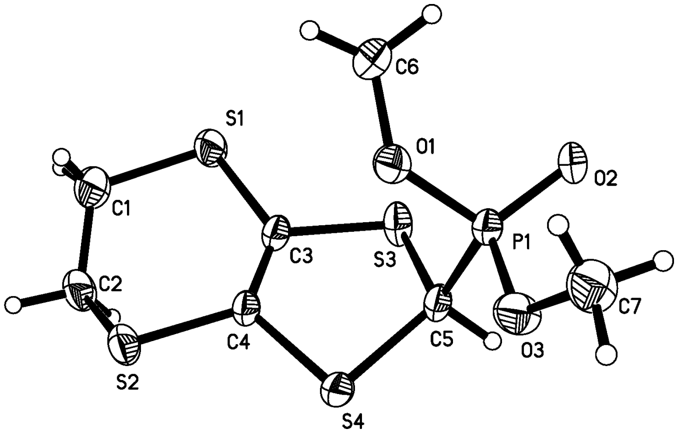


Figure 1
View of (I), with 30% probability displacement ellipsoids.

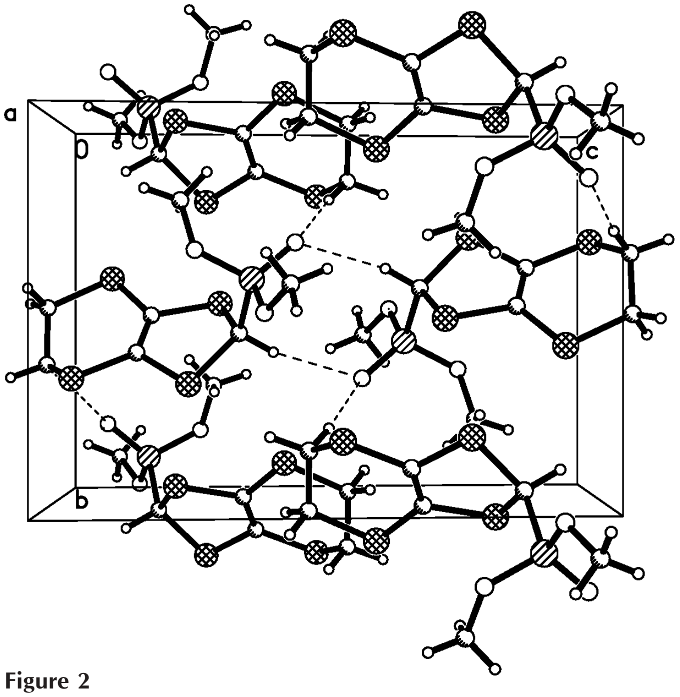


Figure 2
Packing diagram, with the weak C—H···O hydrogen bonds indicated by dashed lines.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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