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Tetra-*n*-propyl-1,2-dithioxodi-λ^5-phosphane

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Abstract. $C_{12}H_{28}P_2S_2$, $M_r = 298.42$, monoclinic, $P2_1/n$, $a = 6.491\ (2)$, $b = 10.152\ (1)$, $c = 13.358\ (1)$ Å, $\beta = 100.74\ (1)°$, $V = 864.8\ (4)$ Å^3, $Z = 2$, $D_m = 1.135\ (4)$, $D_x = 1.146$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 4.58$ cm^{-1}, $F(000) = 324$, $T = 296$ K, $R = 0.033$ for 1137 unique reflections. The molecule lies on a crystallographic centre of symmetry. The geometry around the P atom is slightly distorted tetrahedral with the three angles involving the S atom larger than the other three angles. This distortion can be attributed to the bulk of the S atom and P═S being a double bond. Both the P—C(1)—C(2)—C(3) chain and P—C(4)—C(5)—C(6) chain are essentially planar.

Experimental. The molecule was obtained by an anomalous Grignard reaction (Nienbergall & Langenfeld, 1962). The colourless plate crystals were recrystallized from ethanol and the density was measured by flotation in an HgO/HCl mixture. A crystal of size 0.10 × 0.20 × 0.20 mm was used for data collection on a Rigaku AFC-5*S* diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters were determined from a least-squares fit of 25 centred reflections in the 2θ range 30.1–37.7°. Intensity data were measured by ω–2θ scans of rate 8° min^{-1} in ω, to maximum $\sin\theta/\lambda = 0.5947$ Å^{-1}, and for h,k,l range 0,0,−14 to 7,11,13. Of a total of 1766 reflections, 1617 were unique ($R_{\rm int} = 0.027$), and 1137 with $I > 3.00\sigma(I)$ were used for structure solution and refinement. Three standard reflections, monitored at intervals of 150, showed no significant intensity change. Lorentz, polarization and secondary-extinction (coefficient = 0.43107 × 10^{-6}) corrections were applied, but no absorption correction. The structure was solved by direct methods (Gilmore, 1984; Beurskens, 1984). H atoms were found from difference maps. Refinement, based on $|F|$ values, was carried out with full-matrix least-

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta).$$

	x	y	z	$B_{\rm eq}$
S	0.2175 (1)	0.16397 (7)	−0.08690 (6)	4.2 (1)
P	0.4315 (1)	0.09331 (6)	0.02220 (5)	2.9 (1)
C(1)	0.3362 (6)	0.0464 (3)	0.1373 (2)	4.3 (1)
C(2)	0.2608 (6)	0.1567 (3)	0.1952 (3)	5.2 (2)
C(3)	0.1596 (7)	0.1107 (4)	0.2813 (3)	6.2 (2)
C(4)	0.6597 (5)	0.1970 (3)	0.0599 (3)	3.8 (1)
C(5)	0.6173 (6)	0.3421 (3)	0.0702 (3)	4.7 (2)
C(6)	0.8127 (6)	0.4226 (4)	0.0984 (4)	5.9 (2)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

S—P	1.953 (1)	C(1)—C(2)	1.494 (4)
P—P$^{\rm i}$	2.220 (1)	C(2)—C(3)	1.502 (5)
P—C(1)	1.824 (3)	C(4)—C(5)	1.509 (4)
P—C(4)	1.811 (3)	C(5)—C(6)	1.497 (5)
S—P—P$^{\rm i}$	112.25 (5)	C(1)—P—C(4)	107.8 (2)
S—P—C(1)	114.8 (1)	P—C(1)—C(2)	115.8 (2)
S—P—C(4)	115.2 (1)	C(1)—C(2)—C(3)	113.3 (3)
P$^{\rm i}$—P—C(1)	102.6 (1)	P—C(4)—C(5)	116.0 (2)
P$^{\rm i}$—P—C(4)	102.9 (1)	C(4)—C(5)—C(6)	113.3 (3)
P$^{\rm i}$—P—C(1)—C(2)	−171.6 (2)	P—C(1)—C(2)—C(3)	172.7 (3)
P$^{\rm i}$—P—C(4)—C(5)	−165.6 (2)	P—C(4)—C(5)—C(6)	−178.2 (3)

Symmetry code: (i) $-x, -y, -z$.

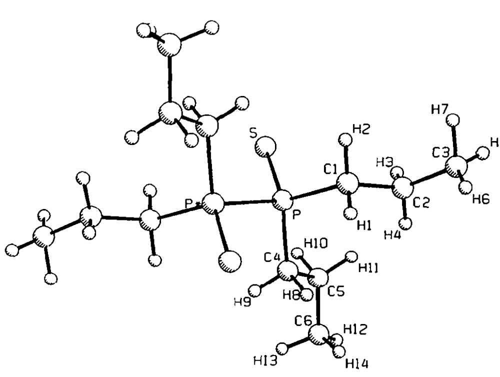

Fig. 1. Molecular structure with the numbering scheme.

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squares techniques, for 116 parameters including non-H atoms anisotropic and H atoms isotropic with fixed temperature factors. Final $R = 0.033$, $wR = 0.040$ with the weighting scheme $w = 1/\sigma^2(F_o)$, $S = 1.44$. Maximum final shift to e.s.d. ratio was 0.06, maximum and minimum peak heights in the final difference Fourier map were 0.26 and -0.15 e Å^{-3}. Neutral atomic scattering factors and f' and f'' values for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computer programs: *TEXSAN* (Molecular Structure Corporation, 1985) and *PLUTO* (Motherwell & Clegg, 1978). The molecule has a centre of symmetry. The atomic positions of non-H atoms are listed in Table 1.* Bond lengths, bond angles and selected torsion angles for the non-H atoms are given in Table 2. The molecular structure with the numbering scheme is shown in Fig. 1.

Related literature. Intramolecular symmetry is confirmed by previously reported IR and Raman spectroscopy studies (McQuillan & Oxton, 1978). The distortion of the tetrahedral configuration around the P atom, and the P—P^{i}, P—S, P—C bond lengths and bond angles are in good agreement with the structure of tetraethyldiphosphine disulfide (Dutta & Woolfson, 1961), bis(cyclotetramethylene)diphosphine disulfide (Lee & Goodacre, 1969) and bis(cyclopentamethylene)diphosphine disulfide (Lee & Goodacre, 1970).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55591 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1010]

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Structure of Ethyl 2-(3-Methyl-1,2,4-oxadiazol-5-yl)-2-phenylhydrazonoethanoate

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Abstract. $C_{13}H_{14}N_4O_3$, $M_r = 274.3$, orthorhombic, $Pna2_1$, $a = 19.048$ (6), $b = 10.507$ (4), $c = 6.691$ (4) Å, $V = 1339.1$ Å^3, $Z = 4$, $D_m = 1.36$ (flotation), $D_x = 1.360$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0.7107$ Å, $\mu = 0.933$ cm^{-1}, $F(000) = 576$, $T = 298$ K, $R = 0.0839$, $wR = 0.0643$ for 773 observed reflections. The whole molecule is approximately planar, which can be seen from the values of the torsion angles about the non-rigid bonds; the molecular dimensions are in agreement with those found in analogous compounds. An important feature of the molecule is an intramolecular N—H···N hydrogen bond, which is characterized by an N···N distance of 2.66 (1) Å.

Experimental. Single crystals were obtained by slow evaporation of a solution in absolute ethanol, under reduced constant pressure in a dry atmosphere, Approximate unit-cell parameters were determined from preliminary Weissenberg and precession photographs. A crystal of ~0.4 × 0.2 × 0.07 mm was used

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