suggested by the isomorphous nature of (II) to (I) (see *Abstract*). This suggests that the N could be in any of the four possible positions with equal probability. Furthermore, placing the N at either position 1 or 6 and refining isotropically resulted in a considerably larger thermal parameter for the N *versus* the C atoms at the other three equivalent positions.

We resolved to treat the model as thianthrene until after the H atoms were located in a difference map with the hope that the N could be identified by the lack of density at the ideal H position. The electron densities corresponding to H on positions 1, 4, 6, and 9 were, respectively, 0·48, 0·56, 0·79 and 0·65 e Å^{-3}. The other four positions had densities ranging from 0·71 to 0·80 e Å^{-3}. We, therefore, assigned the N to the position of lowest density realizing that positions 4 and 9 probably contain partial N occupancy. The final ΔF map produced a peak of 0·53 e Å^{-3} 1·01 Å from N(1), most probably a combination of the lone-pair density and partial H occupancy.

The final thermal parameters (Table 2) and geometrical results (Figs. 3 and 4) when compared to thianthrene substantiate a disordered structure. The S-containing six-membered ring is essentially identical to the same ring in thianthrene. However, neither aromatic ring of (II) parallels those of (I). The C—N distances were expected to be shorter [C—N range: 1·315–1·346 Å in 1,4,9-triazaphenoxathiin and 1·333–1·340 Å in 1,3-diazaphenoxathiin (Larson & Simonsen, 1983)]. All other bond lengths except those of the S-containing six-membered ring are shorter than suggested by the thianthrene results, the effect of the unresolved disorder. The thermal parameters can be explained by approximately equal partial occupancy for N at positions 4 and 9 with very little partial occupancy for N at position 6. The decrease in the thermal parameters of C(4) and C(9) due to less electron density placed there is offset by the mislocation due to the differences in pyridine and benzene geometries.

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Structure of Diphenylthiophosphinic Acid, $(C_6H_5)_2P(=S)OH$, at 140 K

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Abstract. $M_r = 234·3$, orthorhombic, space group *Pbca*, $a = 22·62$ (1), $b = 6·105$ (2), $c = 16·562$ (5) Å, $V = 2287·1$ Å^3, $Z = 8$, $D_m = 1·34$, $D_x = 1·36$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 3·9$ cm^{-1}, $F(000) = 976$, $T = 140$ K, $R = 0·049$ for 2578 independent reflections. The acid hydrogen atom is attached to the oxygen atom of the POS group. The molecules form endless chains along the glide plane in the [010] direction through strong O—H···S hydrogen bonds of 3·133 (8) Å length.

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Introduction. Crystals of phosphinic acids (Giordano & Ripamonti, 1967; Fenske, Mattes, Löns & Tebbe, 1973) and of dithiophosphinic acids (Henkel, 1976; Krebs, 1983) contain rather short intermolecular O—H···O or S—H···S hydrogen bonds. According to our recent study, $(CH_3)_2PSOH$ (HMTP) and (cyclo-$C_6H_{11})_2PSOH$ (HCTP) form dimers through strong intermolecular O—H···S hydrogen bonds with O—S distances 3·121 (1) and 3·143 (4) Å, respectively (Mattes, Mühlsiepen & Rühl, 1983). The present investigation was undertaken to determine the position of the acid hydrogen atom in diphenylthiophosphinic acid (HPTP), *i.e.* whether there is an O—H or S–H group present in the molecule, and to investigate the hydrogen-bond system and the degree of aggregation in the solid state. These structural details may depend on the nature of the substituents *R* at the phosphorus atom (Kabachnik, Mastrukova, Shipov & Mentyeva, 1960). HPTP has been used by us as a ligand (Mattes & Rühl, 1983). This study may also allow the structures of the ligand in the complexed and the free state to be compared.

Experimental. HPTP synthetized according to literature (Sasse, 1963); single crystals by crystallization from toluene/benzene 1:1 mixture, m.p. 413 K; D_m by flotation; crystal 2 × 0·5 × 0·5 mm in Lindemann tube for data collection; Syntex $P2_1$ four-circle diffractometer, graphite monochromator; cell dimensions by least squares from 12 reflections ($11 < \theta < 17°$); no absorption correction; intensities of 2910 independent reflections with $2\theta < 54°$ determined, *h* 0–28, *k* 0–7, *l* 0–21; one standard reflection every 100 reflections, variation 2% about its mean, 2578 reflections with $I > 1{\cdot}96\sigma(I)$ considered observed and used in structure determination; structure solved by direct methods, full-matrix refinement on *F* with nonhydrogen atoms, anisotropic temperature factors, $w^{-1} = \sigma(F_o) + (0{\cdot}02\,F_o)^2$; H atoms from difference synthesis, refined isotropically; $R = 0{\cdot}049$, $R_w = 0{\cdot}055$ for 180 parameters;* max. $\Delta\rho$ excursion in final difference synthesis 0·82 e Å^{-3}, $\Delta_{max} < 0{\cdot}2\sigma$ in last cycle; Syntex (1976) *XTL/XTLE* programs used; atomic scattering factors of *XTL/XTLE* used.

Discussion. Final positional parameters are given in Table 1, calculated bond lengths and bond angles in Table 2. Fig. 1 shows details of the hydrogen bonding. The structure consists of individual molecules, which are linked into infinite chains by strong intermolecular O—H···S bonds with 3·138 (2) Å length. Molecular-

* Lists of structure factors, anisotropic thermal parameters and some bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38864 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (×10^4) *and isotropic thermal parameters* (Å^2)

	x	*y*	*z*	B_{eq}/B_{iso}*
P	1560·1 (2)	1701·3 (9)	6148·5 (3)	1·30 (3)
S	1831·1 (2)	−565 (1)	6896·5 (3)	1·73 (3)
O	1872·5 (7)	4006 (3)	6238 (1)	1·89 (8)
C(11)	794 (1)	2428 (4)	6273 (1)	1·69 (9)
C(12)	628 (1)	4414 (4)	6614 (2)	2·2 (1)
C(13)	26 (1)	4884 (5)	6722 (2)	1·6 (1)
C(14)	−393 (1)	3379 (5)	6486 (2)	2·7 (1)
C(15)	−230 (1)	1421 (5)	6139 (2)	2·7 (1)
C(16)	365 (1)	921 (4)	6036 (2)	2·3 (1)
C(21)	1646 (1)	927 (4)	5104 (1)	1·51 (9)
C(22)	1842 (1)	−1142 (4)	4881 (1)	1·81 (9)
C(23)	1916 (1)	−1637 (4)	4060 (1)	2·1 (1)
C(24)	1791 (1)	−78 (5)	3481 (2)	2·2 (1)
C(25)	1587 (1)	1972 (5)	3702 (2)	2·3 (1)
C(26)	1513 (1)	2485 (4)	4514 (1)	1·88 (9)
H(1)	951 (12)	5522 (51)	6821 (18)	3·1 (6)
H(2)	−57 (13)	6216 (53)	6985 (19)	2·5 (6)
H(3)	−783 (16)	3662 (69)	6543 (23)	4·0 (9)
H(4)	−502 (20)	145 (66)	5937 (23)	4·9 (6)
H(5)	465 (15)	−500 (52)	5788 (21)	2·9 (7)
H(6)	1911 (15)	−2279 (72)	5291 (23)	3·7 (6)
H(7)	2043 (13)	−2926 (55)	3904 (18)	2·1 (6)
H(8)	1801 (10)	−347 (40)	2981 (16)	1·0 (4)
H(9)	1479 (15)	3072 (53)	3303 (22)	3·2 (7)
H(10)	1430 (13)	3860 (53)	4646 (19)	2·1 (6)
H(11)	2240 (21)	4139 (70)	6423 (28)	6 (1)

* $B_{eq} = (B_{11}B_{22}B_{33})^{1/3}$ for nonhydrogen atoms.

Table 2. *Selected bond lengths* (Å) *and angles* (°) *in* HPTP

P–O	1·582 (2)	C(14)–C(15)	1·376 (5)
P–S	1·956 (1)	C(15)–C(16)	1·396 (4)
P–C(11)	1·799 (2)	C(21)–C(22)	1·387 (4)
P–C(21)	1·805 (3)	C(21)–C(26)	1·396 (4)
C(11)–C(12)	1·388 (4)	C(22)–C(23)	1·406 (4)
C(11)–C(16)	1·393 (4)	C(23)–C(24)	1·381 (4)
C(12)–C(13)	1·401 (4)	C(24)–C(25)	1·385 (5)
C(13)–C(14)	1·378 (4)	C(25)–C(26)	1·391 (4)
O–P–S	115·4 (1)	S–P–C(11)	113·8 (1)
O–P–C(11)	101·6 (2)	S–P–C(21)	112·8 (1)
O–P–C(21)	105·9 (2)	C(11)–P–C(21)	106·2 (2)

C–H bonds: 0·88 (4)–1·05 (4); mean: 0·95 (4)
C–C–C angles: 119·3 (3)–121·1 (3); mean: 120·2 (3)

Hydrogen bonds

O···H(11)···S	3·138 (2)	O···H(11)···S	179 (5)
H(11)···S	2·24 (6)	P–O–H(11)	122 (4)
O–H(11)	0·90 (6)	P–S···H(11)	97 (2)
		P–S···O	97·57 (4)

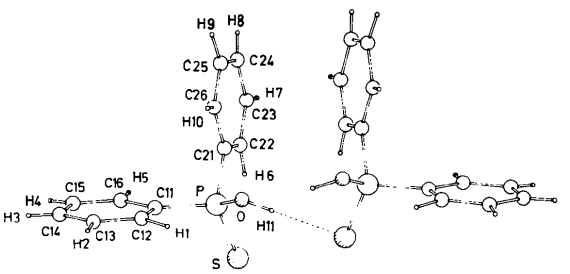

Fig. 1. Structure of the hydrogen bond viewed in the [010] direction and atom numbering scheme of HPTP.

weight measurements indicate that HPTP is also highly associated in solutions. As in HMTP and HCTP (Mattes, Mühlsiepen & Rühl, 1983) the acid hydrogen atom is located in HPTP at the oxygen atom of the POS group. The O···S distance [3·138 (2) Å] is shorter than the sum of the van der Waals radii of oxygen and sulfur (~ 3·2 Å). This is not the case for the S—H···S hydrogen bonds in dithiophosphinic acids, with an S···S distance of 3·75–3·83 Å (Henkel, 1976; Krebs, 1983). Therefore, the O—H···S hydrogen bond has to be considered as a rather strong hydrogen bond. Its strength is mainly due to the high polarity of the O—H bond. The bond is linear within the experimental error. The P=S···H (or the more accurately determined P=S···O angle) is considerably smaller than 120°; this value can be expected if an acceptor atom is doubly bonded and this atom sp^2 hybridized. In thiophosphinic and dithiophosphinic acids the size of this angle varies between 90 and 110°. In the present work it is close to 97°, indicating a slightly higher *s* character of the P=S bond (and a higher *p* character of the acceptor orbital) compared to HMTP and HCTP. Here the values 103·3 (5) and 107 (1)° have been found. This might be the reason for the slight shortening of the P=S bond from 1·966 (1) and 1·973 (1) Å in the latter compounds to 1·956 (1) Å in HPTP. The overall hydrogen-bond pattern is also different in these three compounds, because HMTP and HCTP form dimers in the solid state and HPTP is polymeric. But the local structure of the hydrogen bond is not affected. The question whether (di)thiophosphinic acids form dimers or polymers in the solid state is probably determined by crystal packing effects. The P—O bond length of 1·582 (2) Å is typical for a single bond. Whereas the distances *d*(PO) and *d*(PS) remain essentially unchanged after deprotonation and complex formation, *e.g.* with Sb^{III}, the OPS angle slightly decreases in HPTP from 115·4 to approximately 111° for chelating ligands, but remains unchanged for bridging ligands (Mattes & Rühl, 1983). The remaining bond lengths of HPTP and distances are normal.

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Structure of 5-Amino-4-phenyl-3,4-dihydro-2*H*-pyrrol-2-one, $C_{10}H_{10}N_2O$

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Abstract. $M_r = 174·19$, monoclinic, $P2_1/a$, $a = 12·770$ (2), $b = 5·761$ (1), $c = 12·001$ (2) Å, $\beta = 97·94$ (2)°, $V = 874·5$ (3) Å^3, $Z = 4$, $D_x = 1·323$ (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0·627$ mm^{-1}, $F(000) = 368$, $T = 293$ K. Final $R = 0·043$ for 1085 observed reflections. The C—N—C—N fragment of the molecule seems to reveal the diffused electron system, the exocyclic N atom being of amino character. Both amino H atoms form hydrogen bridges to the carbonyl O and hetero N atoms of neighbouring molecules, thus creating planar systems parallel to the *ab* plane of the crystal.

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