The molecular arrangement in the crystal viewed along **b** is illustrated in Fig. 2. The crystal is built up of individual molecules, with adjacent molecules held together by van der Waals forces.

References

AHMED, N. A. & KITAIGORODSKY, A. I. (1972). *Acta Cryst.* B**28**, 739–742.

AIKAWA, S., MARUYAMA, Y., OHASHI, Y. & SASADA, Y. (1978). *Acta Cryst.* B**34**, 2901–2904.

AKITA, Y. & OHTA, A. (1982). *Heterocycles,* **19**, 329–331 and references cited therein.

ASHIDA, T. (1973). *The Universal Crystallographic Computing System – Osaka*, pp. 55–61. The Computation Center, Osaka Univ., Japan.

BAUDOUR, J. L., DELUGEARD, Y. & CAILLEAU, H. (1976). *Acta Cryst.* B**32**, 150–154.

BRAAM, A. W. M., EIKELENBOOM, J. C., VAN DIJK, G. & VOS, A. (1981). *Acta Cryst.* B**37**, 259–261.

BRAAM, A. W. M., ESHUIS, A. & VOS, A. (1981). *Acta Cryst.* B**37**, 730–732.

CHEESEMAN, G. W. H. & WERSTIUK, E. S. G. (1972). *Adv. Heterocycl. Chem.* **14**, 99.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 72. Birmingham: Kynoch Press.

LAING, M. & SOMMERVILLE, P. (1976). *Acta Cryst.* B**32**, 2764–2767.

LIN, Y. C. & WILLIAMS, D. C. (1975). *Acta Cryst.* B**31**, 318–320.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data,* Univs. of York, England, and Louvain, Belgium.

MATSUO, M., MATSUMOTO S., KURIHARA, T., AKITA, Y., WATANABE, T. & OHTA, A. (1980). *Org. Magn. Reson.* **13**, 172–179.

OHTA, A., MASANO, S., IWAKURA, S., TAMURA, A., WATAHIKI, H., TSUTSUI, M., AKITA, Y. & WATANABE, T. (1982). *J. Heterocycl. Chem.* **19**. In the press.

SAMMES, P. G. (1975). *Fortschr. Chem. Org. Naturst.* **32**, 51.

WHEATLEY, P. J. (1957). *Acta Cryst.* **10**, 182–187.

Acta Cryst. (1983). C**39**, 139–141

4-Amino-1-phenyl-1,2,5,6-tetrahydrophosphorin-3-carbonitrile, $C_{12}H_{13}N_2P$

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Abstract. $M_r = 216\cdot2$, monoclinic, space group $P2_1/c$, $a = 11\cdot670$ (2), $b = 7\cdot794$ (2), $c = 12\cdot785$ (8) Å, $\beta = 100\cdot37$ (3)°, $U = 1143\cdot8$ Å^3, $Z = 4$, $D_x = 1\cdot26$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0\cdot205$ mm^{-1}, $T = 295$ K. Final $R = 0\cdot031$ for 1516 observed reflections. The molecules are hydrogen-bonded in the crystal, forming infinite chains extending along the crystallographic *c* axis.

Introduction. Phosphorinane, a heterocyclic analogue of cyclohexane, has been shown (Lambert & Oliver, 1971) to exist predominantly in the chair form in the liquid state, with the P—H bond disposed axially to the ring.

A number of X-ray structural determinations (McPhail, Breen & Quin, 1971; McPhail, Breen, Somers, Steele & Quin, 1971; McPhail, Luhan, Featherman & Quin, 1972) have demonstrated that this configurational preference persists in the solid state for l-phenyl- and l-methyl-substituted phosphorinanes. The considerable steric interactions between the heterocyclic ring and the P substituent are partially relieved, in the case of the phenyl group, by out-of-plane distortion at the phenyl *ipso* C atom. The present study is concerned with an X-ray structural study of a partially dehydrogenated phosphorinane in which, due to heterocyclic-ring flattening, the steric strain should be reduced.

Experimental. Crystal *ca* 0·22 × 0·25 × 0·44 mm, Enraf–Nonius CAD-4 diffractometer controlled by a PDP8/a computer, take-off angle 5°, Mo $K\alpha$ radiation filtered by a square-graphite-crystal monochromator, cell dimensions by least-squares refinement of the setting angles of 25 accurately centered reflections; 2005 independent reflections (to $\theta \leq 25°$), ω–2θ scans, *ZIGZAG* routine (Enraf–Nonius, 1977), background counts taken on each side of the peak, intensities of three standard reflections measured every 8000s, no significant change in intensities, 1516 observed [$I \geq 3\sigma(I)$], Lp correction; direct methods, automatic multisolution routine of *SHELX* 76 (Sheldrick, 1976) used with all unique reflections to generate a series of E maps, one of which correctly located all non-H atomic positions, full-matrix least-squares refinement with isotropic temperature factors gave $R = 0\cdot122$ for the observed reflections, anisotropic temperature factors

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reduced R to 0·072, all H atoms located from a difference synthesis and further refinement of non-H atoms anisotropically and H atoms isotropically reduced R to 0·031, a weighting scheme based on counting statistics was used with $w = [\sigma^2|F_o| + 0{\cdot}0040F_o^2]^{-1}$ minimizing $\sum w\Delta^2$, $R_w = 0{\cdot}037$, a final difference map showed a highest peak of 0·26e Å^{-3}; atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974), $F(000) = 456$.

Discussion. Fig. 1 is an *ORTEP* diagram (Johnson, 1976) of the molecule illustrating 30% probability ellipsoids for the non-H atoms and arbitrary spheres of radius 0·1 Å for the H atoms, together with the numbering scheme, whilst Fig. 2 shows the packing arrangement.

Atomic positions are listed in Table 1.* Bond lengths, angles and torsion angles are listed in Table 2.

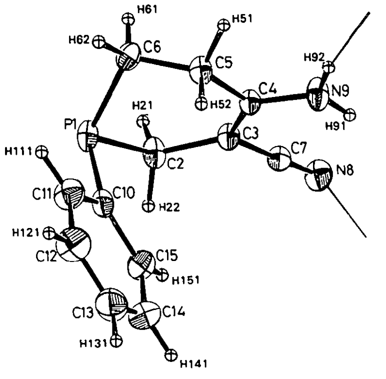

Fig. 1. The phosphorin molecule with the atom-numbering scheme and hydrogen bonds.

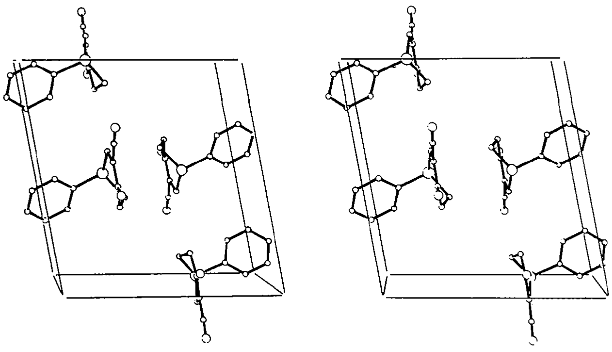

Fig. 2. Stereopair of the unit-cell contents viewed down the crystallographic b axis.

The heterocyclic-ring torsion angles (Table 2) indicate the considerable flattening imposed by the constraint of the ring olefinic bond. Atoms C(2), C(3), C(4) and C(5) lie on a plane to within ± 0·011 Å, with C(6)

Table 1. *Fractional coordinates* (×10^4 *for non*-H *atoms;* ×10^3 *for* H), *equivalent isotropic U values for non*-H *atoms and isotropic U for* H (Å^2 × 10^3)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}
P(1)	3032 (1)	1047 (1)	9991 (1)	47
C(2)	3348 (1)	2695 (2)	11023 (1)	46
C(3)	3392 (1)	4539 (1)	10648 (1)	39
C(4)	3370 (1)	5077 (1)	9628 (1)	37
C(5)	3361 (1)	3878 (2)	8704 (1)	44
C(6)	3784 (1)	2077 (2)	9007 (1)	43
C(7)	3510 (1)	5780 (1)	11475 (1)	45
N(8)	3629 (1)	6751 (1)	12165 (1)	55
N(9)	3342 (1)	6748 (1)	9355 (1)	60
C(10)	1513 (1)	1414 (2)	9379 (1)	52
C(11)	1104 (1)	582 (4)	8414 (1)	56
C(12)	−43 (2)	673 (4)	7922 (2)	63
C(13)	−825 (1)	1600 (4)	8384 (2)	74
C(14)	−450 (2)	2421 (4)	9333 (2)	102
C(15)	710 (1)	2344 (2)	9824 (1)	67
H(21)	412 (2)	240 (3)	1143 (2)	59 (7)
H(22)	271 (2)	252 (3)	1150 (2)	59 (7)
H(51)	387 (2)	445 (3)	825 (2)	50 (6)
H(52)	256 (2)	387 (2)	831 (2)	31 (5)
H(61)	462 (2)	208 (3)	934 (2)	45 (6)
H(62)	369 (2)	134 (3)	842 (2)	52 (6)
H(91)	326 (2)	752 (3)	980 (2)	56 (7)
H(92)	344 (2)	702 (3)	875 (3)	63 (8)
H(111)	165 (3)	−9 (3)	806 (2)	71 (8)
H(121)	−27 (3)	6 (4)	721 (3)	89 (10)
H(131)	−162 (3)	172 (3)	805 (2)	79 (8)
H(141)	−93 (3)	307 (4)	966 (3)	89 (9)
H(151)	94 (2)	286 (4)	1046 (2)	68 (8)

Table 2. *Bond lengths, angles and selected torsion angles*

(*a*) Distances (Å)			
P(1)—C(2)	1·831 (2)	C(5)—C(6)	1·516 (3)
P(1)—C(6)	1·842 (2)	C(7)—N(8)	1·152 (3)
P(1)—C(10)	1·827 (2)	C(10)—C(11)	1·398 (3)
C(2)—C(3)	1·519 (3)	C(10)—C(15)	1·385 (3)
C(3)—C(4)	1·364 (3)	C(11)—C(12)	1·376 (4)
C(3)—C(7)	1·420 (2)	C(12)—C(13)	1·378 (4)
C(4)—C(5)	1·505 (3)	C(13)—C(14)	1·371 (4)
C(4)—N(9)	1·348 (3)	C(14)—C(15)	1·388 (4)
(*b*) Angles (°)			
C(6)—P(1)—C(2)	97·3 (1)	C(5)—C(6)—P(1)	113·9 (1)
C(10)—P(1)—C(2)	104·7 (1)	N(8)—C(7)—C(3)	177·8 (2)
C(10)—P(1)—C(6)	100·6 (1)	C(11)—C(10)—P(1)	117·1 (2)
C(3)—C(2)—P(1)	116·8 (1)	C(15)—C(10)—P(1)	125·5 (2)
C(4)—C(3)—C(2)	126·6 (2)	C(15)—C(10)—C(11)	117·2 (2)
C(7)—C(3)—C(2)	114·4 (1)	C(12)—C(11)—C(10)	122·0 (2)
C(7)—C(3)—C(4)	119·0 (2)	C(13)—C(12)—C(11)	119·7 (3)
C(5)—C(4)—C(3)	123·8 (2)	C(14)—C(13)—C(12)	119·5 (3)
N(9)—C(4)—C(3)	122·6 (2)	C(15)—C(14)—C(13)	120·7 (3)
N(9)—C(4)—C(5)	113·6 (2)	C(14)—C(15)—C(10)	120·9 (2)
C(6)—C(5)—C(4)	114·7 (2)		
(*c*) Torsion angles (°)			
P(1)—C(2)—C(3)—C(4)	−9·3 (2)	C(4)—C(5)—C(6)—P(1)	53·1 (2)
C(2)—C(3)—C(4)—C(5)	−3·6 (4)	C(5)—C(6)—P(1)—C(2)	−56·3 (2)
C(3)—C(4)—C(5)—C(6)	−19·7 (4)	C(6)—P(1)—C(2)—C(3)	34·3 (2)

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* Lists of structure factors, anisotropic thermal parameters bond lengths and angles involving H atoms and a diagram showing intermolecular hydrogen bonding have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38190 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU. England.

0·521 (1) Å above and P(1) 0·331 (1) Å below the plane. The short C(3)–C(7) and C(4)–N(9) single bonds, together with the long C(3)–C(4) double bond indicate considerable π-electron delocalization over the C(7)–C(3)–C(4)–N(9) fragment.

The phenyl group is axial to the hetero ring with the projection of its plane almost parallel to the P(1)–C(2) bond [the torsion angle C(2)–P(1)–C(10)–C(11) is *ca* 166·6 (3)°]. The steric crowding imposed by this configuration results in a slight displacement [0·135 (1) Å] of the P atom from the phenyl-group mean plane, and the phenyl group itself departs from D_{6h} symmetry, primarily because of the closure of the C(15)–C(10)–C(11) bond angle [117·2 (2)°]. This type of behavior has been discussed by Domenicano & Vaciago (1975) who concluded that the internal angles at the *ipso* C atoms are sensitive to the nature of the phenyl-ring substituents.

An examination of intermolecular contacts revealed only one distance which is significantly less than the sum of the relevant van der Waals radii.* This contact, of magnitude 2·31 (3) Å, is between N(8) of one molecule and H(92′) in an adjacent, glide-plane-related molecule, with an N(8)–N(9′) distance of 3·108 (2) Å and the N(8)···H(92′)–N(9′) angle equal to 170 (3)°. The structure is thus built up of $C_{12}H_{13}N_2P$ moieties hydrogen-bonded through N(8)···H(92′)–N(9′) links, forming infinite molecular chains extending along **c** (Fig 3).*

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* van der Waals radii were taken from Pauling (1967) except for H, which is taken from Bauer (1972).

* See deposition footnote.

References

Bauer, W. H. (1972). *Acta Cryst.* **B28**, 1456–1465.

Domenicano, A. & Vaciago, A. (1975). *Acta Cryst.* **B31**, 2533–2554.

Enraf–Nonius (1977). CAD-4 *Operating Manual.* Enraf–Nonius, Delft.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Johnson, C. K. (1976). *ORTEP.* Report ORNL–5138. Oak Ridge National Laboratory, Tennessee.

Lambert, J. B. & Oliver, W. L. (1971). *Tetrahedron,* **29**, 4245–4252.

McPhail, A. T., Breen, J. J. & Quin, L. D. (1971). *J. Am. Chem. Soc.* **93**, 2574–2575.

McPhail, A. T., Breen, J. J., Somers, J. H., Steele, J. C. H. & Quin, L. D. (1971). *Chem. Commun.* pp. 1020–1021.

McPhail, A. T., Luhan, P. A., Featherman, S. I. & Quin, L. D. (1972). *J. Am. Chem. Soc.* **94**, 2126–2127.

Pauling, L. (1967). *The Nature of the Chemical Bond.* New York: Cornell Univ. Press.

Sheldrick, G. M. (1976). *SHELX.* Program for crystal structure determination. Univ. of Cambridge, England.

International Union of Crystallography

Acta Cryst. (1983). C**39**, 141–142

Notes for Authors

Revised *Notes for Authors* for *Acta Crystallographica* and *Journal of Applied Crystallography* have been published in the January 1983 issue of *Acta Crystallographica,* Section A, pages 174–186. Reprints of these directions may be obtained on request from any of the Editors. These *Notes* give information on submission procedures, categories of contributions and details of format for all parts of a paper. However, for the use of contributors to *Acta Crystallographica,* Section C, Appendix I, which gives the criteria for papers to be published in Sections B and C and the format for papers to be published in Section C, is reprinted below.

APPENDIX I

Criteria for publication in *Acta Crystallographica,* Sections B and C, and format for papers to be published in Section C

Papers will only be considered for publication in Section B of *Acta Crystallographica* if they meet the following three criteria:

1. The paper must contain a major structural element. This component may be an original determination of one or more structures (a single structure should generally have been studied under more than one condition of temperature or pressure), a theoretical structural investigation including new methodology, or a study of structural relationships based on a search of the literature. The calibre of this component should be at least as high as was previously required for acceptance in Section B up to 1982.
2. The paper should also present an experimental and/or theoretical contribution to one of the natural sciences that is novel, original and of high quality.
3. The paper should combine these two types of contribution to provide new structural insight for that science or for crystallography.

Papers which present the results of a crystal structure determination or of several such determinations, but are concerned with the crystal and molecular structure alone, will only satisfy the first of these criteria and will therefore be considered for publication in Section C.

Subject to the Co-editor's discretion, papers submitted for consideration in *Acta Cryst.,* Section C should conform with the following arrangement [*Acta Cryst.* (1981), **B38**, 699–700]:

The *Title* will consist of the name of the substance and the