References

BENEDETTI, E. (1977). *Proceedings of the Fifth American Peptide Symposium*, edited by M. L. GOODMAN & J. MEIENHOFER, pp. 257–273, and references therein. New York: John Wiley.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040–1044.

HRUBY, V. J. (1974). *Chemistry and Biochemistry of Amino Acids, Peptides, and Proteins*, edited by B. WEINSTEIN, Vol. 3, pp. 1–188. New York: Marcel Dekker.

JOLAD, S. D., HOFFMANN, J. J., TORRANCE, S. J., WIEDHOPF, R. M., COLE, J. R., ARORA, S. K., BATES, R. B., GARGIULO, R. L. & KRIEK, G. R. (1977). *J. Am. Chem. Soc.* **99**, 8040–8044.

Acta Cryst. (1979). B**35**, 191–192

N-(1-Phenyl-1-phospholanio)-4-nitroanilide

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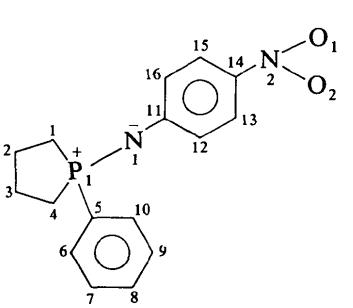
Abstract. $C_{16}H_{17}N_2O_2P$, $M_r = 300{\cdot}31$, monoclinic, $P2_1/c$, $a = 9{\cdot}69\ (3)$, $b = 16{\cdot}34\ (4)$, $c = 10{\cdot}82\ (4)$ Å, $\beta = 117{\cdot}90\ (3)°$ from diffractometer measurements (Mo $K\bar{\alpha}$ radiation), $V = 1513{\cdot}7$ Å^3, $Z = 4$, $D_c = 1{\cdot}317$ Mg m^{-3}, $F(000) = 632$, $\mu = 0{\cdot}145$ mm^{-1}. The heterocyclic ring has an irregular conformation.

Introduction. The title compound (I) was recrystallized from a benzene–ether mixture. It was hygroscopic, and a crystal 0·6 mm long, sealed in a Lindemann-glass capillary, was used for all measurements. Systematic absences (from precession photographs) $h0l$: $l = 2n + 1$, $0k0$: $k = 2n + 1$ indicated space group $P2_1/c$. Data were collected for $hk0$–10 with $\theta_{max} = 25°$ on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\bar{\alpha}$ radiation). This gave 2136 data of which 1357 unique reflexions with $I > 3\sigma(I)$ were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were applied, and the data scaled by a Wilson plot. The structure was solved by direct methods with *SHELX*76 (Sheldrick, 1976), which was used for all calculations. Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Full-matrix least-squares refinement (including isotropic H atoms) converged at $R = 0{\cdot}047$ for 1357 observed reflexions ($R = \sum||F_o| - |F_c||/\sum|F_o|$). In the final cycle all shifts in parameters were less than their e.s.d.'s. Positional parameters are given in Table 1, bond distances and angles in Table 2.*

Discussion. The structure determination (Fig. 1) was undertaken to investigate ring strain (Cadogan, Gosney

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33854 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.



(I)

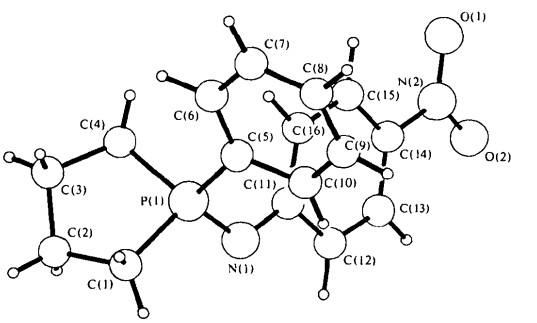


Fig. 1. General view of the molecule.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	*y*	*z*
P(1)	5081 (1)	6812 (1)	2877 (1)
N(1)	3871 (4)	7395 (2)	1688 (3)
N(2)	−162 (5)	9587 (2)	2380 (5)
O(1)	−95 (5)	9673 (2)	3528 (5)
O(2)	−1134 (5)	9944 (2)	1336 (5)
C(1)	5875 (7)	6098 (3)	2107 (7)
C(2)	5028 (9)	5302 (3)	1999 (8)
C(3)	4764 (10)	5232 (4)	3254 (8)
C(4)	4239 (7)	6042 (3)	3549 (7)
C(5)	6624 (4)	7363 (3)	4297 (4)
C(6)	7393 (6)	7078 (3)	5651 (6)
C(7)	8576 (6)	7522 (4)	6707 (6)
C(8)	9037 (6)	8246 (4)	6390 (7)
C(9)	8294 (8)	8548 (4)	5067 (8)
C(10)	7058 (7)	8120 (3)	4013 (6)
C(11)	2938 (5)	7929 (2)	1935 (4)
C(12)	1862 (5)	8398 (3)	793 (5)
C(13)	867 (6)	8940 (3)	943 (5)
C(14)	924 (5)	9041 (3)	2229 (5)
C(15)	1959 (5)	8593 (3)	3378 (5)
C(16)	2940 (5)	8050 (3)	3221 (5)
H(11)	6889 (64)	6045 (31)	2681 (56)
H(12)	5767 (45)	6313 (25)	1264 (46)
H(21)	5514 (59)	4850 (32)	1801 (58)
H(22)	3805 (81)	5311 (37)	1253 (74)
H(31)	3966 (61)	4857 (31)	3142 (57)
H(32)	5927 (68)	5135 (31)	4043 (60)
H(41)	3118 (74)	6119 (34)	3002 (62)
H(42)	4563 (60)	6140 (31)	4411 (59)
H(61)	7085 (49)	6543 (29)	5816 (47)
H(71)	9200 (63)	7280 (32)	7676 (61)
H(81)	9991 (60)	8603 (29)	7124 (55)
H(91)	8402 (59)	9027 (33)	4775 (55)
H(101)	6474 (51)	8308 (28)	3124 (51)
H(121)	1783 (44)	8338 (26)	−82 (45)
H(131)	174 (48)	9203 (26)	152 (45)
H(151)	1981 (44)	8636 (24)	4248 (44)
H(161)	3636 (42)	7740 (21)	4011 (41)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

P(1)–N(1)	1·589 (3)	C(1)–H(11)	0·887 (53)
P(1)–C(1)	1·801 (5)	C(1)–H(12)	0·937 (43)
P(1)–C(4)	1·825 (5)	C(2)–H(21)	0·952 (53)
P(1)–C(5)	1·805 (4)	C(2)–H(22)	1·077 (67)
N(1)–C(11)	1·370 (5)	C(3)–H(31)	0·948 (52)
N(2)–O(1)	1·221 (5)	C(3)–H(32)	1·059 (55)
N(2)–O(2)	1·228 (5)	C(4)–H(41)	0·972 (60)
N(2)–C(14)	1·446 (5)	C(4)–H(42)	0·848 (53)
C(1)–C(2)	1·513 (7)	C(6)–H(61)	0·966 (46)
C(2)–C(3)	1·500 (8)	C(7)–H(71)	1·014 (56)
C(3)–C(4)	1·504 (8)	C(8)–H(81)	1·067 (52)
C(5)–C(6)	1·377 (6)	C(9)–H(91)	0·869 (52)
C(5)–C(10)	1·386 (6)	C(10)–H(101)	0·911 (45)
C(6)–C(7)	1·386 (7)	C(12)–H(121)	0·918 (43)
C(7)–C(8)	1·364 (7)	C(13)–H(131)	0·911 (42)
C(8)–C(9)	1·360 (8)	C(15)–H(151)	0·935 (42)
C(9)–C(10)	1·395 (8)	C(16)–H(161)	0·950 (37)
C(11)–C(12)	1·412 (6)		
C(11)–C(16)	1·405 (6)		
C(12)–C(13)	1·375 (6)		
C(13)–C(14)	1·376 (6)		
C(14)–C(15)	1·386 (6)		
C(15)–C(16)	1·367 (6)		

C(1)–P(1)–N(1)	109·1 (2)	C(10)–C(5)–C(6)	117·9 (5)
C(4)–P(1)–N(1)	116·0 (2)	C(7)–C(6)–C(5)	121·8 (5)
C(4)–P(1)–C(1)	96·0 (2)	C(8)–C(7)–C(6)	119·3 (6)
C(5)–P(1)–N(1)	113·1 (2)	C(9)–C(8)–C(7)	120·4 (6)
C(5)–P(1)–C(1)	110·7 (2)	C(10)–C(9)–C(8)	120·5 (6)
C(5)–P(1)–C(4)	110·6 (3)	C(9)–C(10)–C(5)	120·0 (6)
C(11)–N(1)–P(1)	121·9 (3)	C(12)–C(11)–N(1)	117·3 (4)
O(2)–N(2)–O(1)	122·3 (4)	C(16)–C(11)–N(1)	126·0 (4)
C(14)–N(2)–O(1)	119·4 (4)	C(16)–C(11)–C(12)	116·7 (4)
C(14)–N(2)–O(2)	118·3 (4)	C(13)–C(12)–C(11)	121·1 (5)
C(2)–C(1)–P(1)	105·0 (4)	C(14)–C(13)–C(12)	119·9 (4)
C(3)–C(2)–C(1)	108·2 (5)	C(13)–C(14)–N(2)	120·1 (4)
C(4)–C(3)–C(2)	110·0 (5)	C(15)–C(14)–N(2)	118·9 (4)
C(3)–C(4)–P(1)	105·3 (4)	C(15)–C(14)–C(13)	120·9 (4)
C(6)–C(5)–P(1)	123·8 (4)	C(16)–C(15)–C(14)	119·0 (4)
C(10)–C(5)–P(1)	118·2 (4)	C(15)–C(16)–C(11)	122·3 (4)

& Wall, 1977). The heterocyclic ring has an irregular conformation with torsion angles P(1)–C(4) = 5, C(4)–C(3) = −27, C(3)–C(2) = 42, C(2)–C(1) = −36 and C(1)–P(1) = 18°.

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References

CADOGAN, J. I. G., GOSNEY, I. & WALL, K. (1977). Unpublished work.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 99. Birmingham: Kynoch Press.

SHELDRICK, G. M. (1976). *SHELX* 76 program system for crystal structure determination. Univ. of Cambridge, England.