A stereoscopic view of the contents of one unit cell is shown in Fig. 3. The molecules touch almost exclusively *via* H···H contacts, with no distances substantially shorter than van der Waals diameters. There is only one short separation of 2·47 Å between O(16) in (x,y,z) and H(2) of the neighbouring molecule related by the centre of symmetry at $(\frac{1}{2},0,\frac{1}{2})$.

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2,2-Diphenylpent-4-ynenitrile (PHIPN)

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Abstract. $C_{17}H_{13}N$, orthorhombic, $Pn2_1a$, $Z = 4$, FW 231·31; $a = 9·8857\,(7)$, $b = 7·6927\,(5)$, $c = 17·1299\,(36)$ Å, $V = 1302·7\,(5)$ Å^3; $D_x = 1·177$, $D_m = 1·18$ g cm^{-3} (KI solution); $\lambda(\text{Mo } K\bar{\alpha}) = 0·7107$ Å, $\mu = 0·74$ cm^{-1}; dimensions: 0·70 × 0·5 × 0·4 mm; final $R = 0·05$. H atoms were located and refined isotropically. The C≡N and C≡C distances are 1·126 (4) and 1·180 (5) Å respectively, and the angles C—C≡N and C—C≡C are 178·8 (4) and 177·9 (3)°.

Introduction. A Philips 1100 automated diffractometer with monochromated Mo $K\bar{\alpha}$ radiation was used for collecting the 2025 unique reflexions up to 30° (2θ) in the $\omega/2\theta$ scan mode. 1519 intensities which were greater than twice the e.s.d. from counting statistics were considered as observed. Lorentz and polarization effects, but not absorption, were corrected. The noncentrosymmetric space group was decided from the distribution of the E values.

The structure was solved by direct (*MULTAN* 74, Main, Woolfson, Lessinger, Germain & Declercq, 1974) and Fourier methods. After least-squares refinement to an R of 0·14 a difference map revealed all the H atoms (which were refined with the other atoms) in the mixed mode (XRAY 70, Stewart, Kundell & Baldwin, 1970). The weighting scheme was derived from the ΔF *vs* F_o) and ΔF *vs* $\sin\theta/\lambda$ plots. It showed no dependence on $\sin\theta/\lambda$ and was: $w = 0·75/\sigma_F^2$ with $\sigma_F = 0·53 - 0·08|F_o|$ if $|F_o| < 3·6$, and $\sigma_F = 0·12 + 0·04|F_o|$ if $|F_o| > 3·6$. This scheme yielded $R = 0·05$ and $R_2 = 0·06$* with no trends in $\langle w\Delta^2F\rangle$. A final ΔF synthesis revealed no peaks outside the $\pm 0·15$ e Å^{-3} range.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1962) were used for all the atoms.

Discussion. The stereochemistry of PHIPN and its derivatives with Cu$^{\rm I}$, Ag$^{\rm I}$ and Au$^{\rm I}$ is being studied at the Institute of Inorganic Chemistry 'Elhuyar' (CSIC, Madrid) (Barral, Moreno & Santos, 1975). As a starting point a crystallographic study of the title compound has been undertaken.

Tables 1, 2 and 3 list the coordinates, the H thermal parameters and the derived bond lengths and angles. The numbering of the atoms is presented in Fig. 1. The angles involving phenyl H atoms range from 116 to 124°.

The geometry of the C(2)—C(1)≡N(6) group [1·486 (4), 1·126 (4) Å and 178·8 (4)°] agrees with the data in the literature (Kokkou & Rentzeperis, 1975; Ikemoto, Chikaishi, Yakushi & Kuroda, 1972). The acetyl group, C(3)—C(4)≡C(5), values of 1·450 (4), 1·180 (5) Å and 177·9 (9)°, also agree with reported

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32539 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final positional parameters* ($\times 10^4$) *and their standard deviations for non-hydrogen atoms*

	x	y	z
C(1)	3291 (3)	2573 (5)	2208 (1)
C(2)	3735 (2)	3308 (5)	1447 (1)
C(3)	4471 (3)	1808 (5)	1009 (2)
C(4)	3606 (3)	302	909 (2)
C(5)	2887 (5)	−917 (6)	852 (2)
N(6)	2976 (3)	2000 (6)	2784 (1)
C(7)	4671 (2)	4856 (5)	1619 (1)
C(8)	5849 (3)	5164 (6)	1195 (2)
C(9)	6611 (3)	6654 (6)	1349 (2)
C(10)	6236 (4)	7793 (6)	1915 (3)
C(11)	5075 (4)	7489 (6)	2343 (2)
C(12)	4294 (3)	6032 (5)	2192 (2)
C(13)	2513 (2)	3935 (5)	973 (1)
C(14)	1202 (3)	3862 (6)	1260 (2)
C(15)	138 (3)	4485 (7)	812 (2)
C(16)	359 (3)	5172 (6)	92 (2)
C(17)	1651 (3)	5221 (5)	−209 (2)
C(18)	2724 (3)	4608 (5)	232 (1)

Table 2. *Fractional coordinates* ($\times 10^4$), *thermal parameters and bond distances* (Å) *for the hydrogen atoms*

	x	y	z	B_{iso}	C–H
H(3*A*)	4748 (29)	2237 (42)	488 (17)	1·7 (5)	0·99 (3)
H(3*B*)	5351 (30)	1461 (46)	1327 (17)	1·7 (6)	1·06 (3)
H(5)	2385 (51)	−1863 (94)	769 (29)	6·2 (13)	0·89 (7)
H(8)	6101 (34)	4408 (58)	780 (22)	3·1 (7)	0·95 (4)
H(9)	7408 (49)	6869 (77)	995 (28)	5·9 (12)	1·01 (5)
H(10)	6824 (40)	8726 (63)	2022 (23)	3·9 (8)	0·94 (5)
H(11)	4810 (36)	8186 (59)	2744 (20)	3·1 (7)	0·91 (4)
H(12)	3404 (43)	5820 (63)	2488 (25)	4·4 (9)	1·03 (4)
H(14)	1035 (39)	3461 (57)	1800 (22)	3·8 (8)	0·99 (4)
H(15)	−783 (48)	4415 (78)	1061 (27)	6·0 (12)	1·05 (5)
H(16)	−399 (44)	5660 (62)	−202 (24)	4·0 (9)	0·98 (4)
H(17)	1864 (43)	5585 (69)	−739 (28)	4·7 (9)	0·97 (5)
H(18)	3625 (30)	4594 (48)	−2 (18)	2·3 (6)	0·98 (3)

values (Chiaroni, Riche & Pascard-Billy, 1975; Goldberg, 1975).

Thermal motion could account for the two short distances found in the phenyl rings, involving C(10) and C(15) (Gopalakrishna & Cartz, 1972). The phenyl rings are otherwise normal.

The angles around the two tetrahedral atoms C(2) and C(3) range respectively from 106·2 to 112·7° and from 108 to 112°. The molecular conformation about C(2) can be seen from the following angles between planes: [C(7)–C(8)–C(9)–C(10)–C(11)–C(12)] ∧ [C(3)–C(2)–C(7)] = 20·8°; [C(13)–C(14)–C(15)–C(16)–C(17)–C(18)] ∧ [C(1)–C(2)–C(13)] = 2·7°; and [C(3)–C(2)–C(7)] ∧ [C(4)–C(3)–C(2)] = 4·8°.

No hydrogen bonds are present, the packing in the molecule being due to van der Waals forces only. Nevertheless it is of interest to point out some intermolecular contacts which can be ascribed to interactions between the aromatic electron systems and the H atoms.

The H atoms are numbered according to the C atoms to which they are bonded and are involved in the following contacts (with molecules labelled as in Fig. 1): H(12,I)···C(10,II) = 2·819 Å, H(12,I)···C(9,II) = 2·742 Å (the distance to the centre of the ring is 3·204

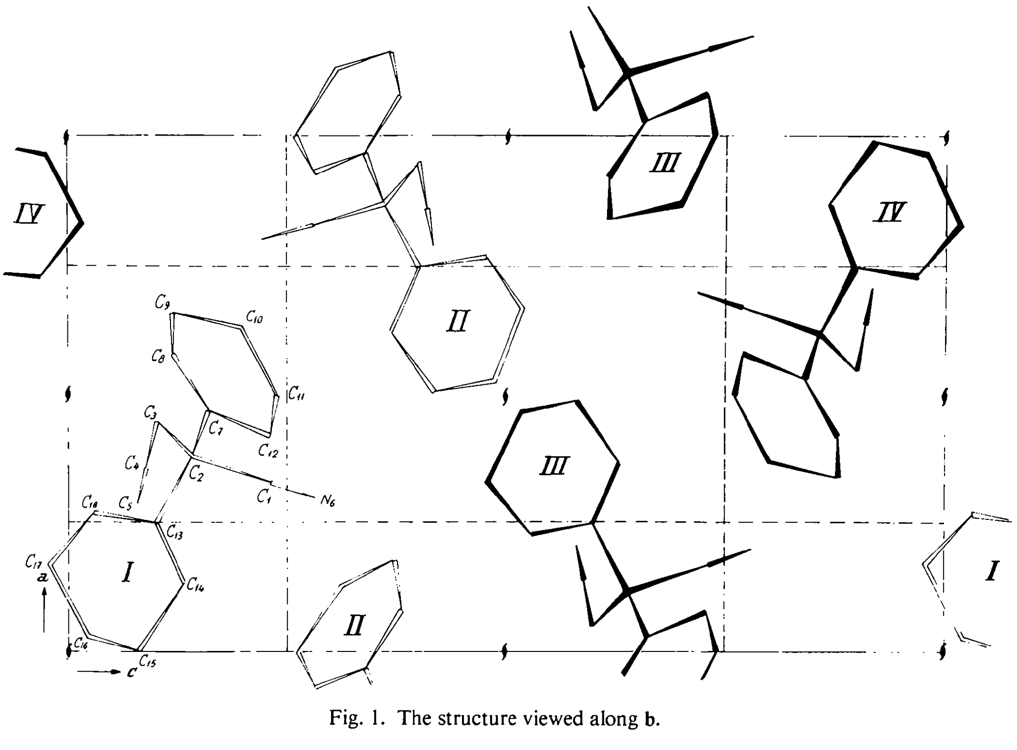

Fig. 1. The structure viewed along **b**.

Table 3. *Bond distances* (Å), *bond angles* (°) *and their standard deviations for the heavy atoms*

C(1)–N(6)	1·126 (4)	N(6)–C(1)–C(2)	178·8 (4)	C(8)–C(9)	1·397 (6)	C(7)–C(8)–C(9)	119·4 (3)
C(1)–C(2)	1·486 (4)			C(9)–C(10)	1·358 (6)	C(8)–C(9)–C(10)	121·2 (3)
C(2)–C(3)	1·557 (5)	C(1)–C(2)–C(3)	106·2 (3)	C(10)–C(11)	1·382 (6)	C(9)–C(10)–C(11)	119·8 (4)
C(2)–C(7)	1·537 (4)	C(7)–C(2)–C(13)	109·4 (3)	C(11)–C(12)	1·385 (6)	C(10)–C(11)–C(12)	120·0 (4)
C(2)–C(13)	1·533 (3)	C(1)–C(2)–C(7)	107·8 (2)			C(11)–C(12)–C(7)	120·8 (3)
		C(1)–C(2)–C(13)	110·5 (2)	C(13)–C(14)	1·387 (4)	C(2)–C(13)–C(14)	122·4 (2)
		C(3)–C(2)–C(7)	112·7 (2)	C(13)–C(18)	1·388 (4)	C(2)–C(13)–C(18)	118·9 (2)
		C(3)–C(2)–C(13)	110·3 (2)			C(14)–C(13)–C(18)	118·7 (3)
C(3)–C(4)	1·450 (4)	C(2)–C(3)–C(4)	111·9 (2)	C(14)–C(15)	1·387 (5)	C(13)–C(14)–C(15)	119·9 (3)
C(4)–C(5)	1·180 (5)	C(3)–C(4)–C(5)	117·9 (3)	C(15)–C(16)	1·360 (6)	C(14)–C(15)–C(16)	121·0 (3)
C(7)–C(8)	1·393 (4)	C(2)–C(7)–C(8)	122·4 (3)	C(16)–C(17)	1·378 (5)	C(15)–C(16)–C(17)	119·9 (3)
C(7)–C(12)	1·386 (5)	C(2)–C(7)–C(12)	118·7 (2)	C(17)–C(18)	1·385 (4)	C(16)–C(17)–C(18)	119·8 (3)
		C(8)–C(7)–C(12)	118·9 (3)			C(17)–C(18)–C(14)	120·7 (3)

Å); and H(5,I)···C(17,I′) = 2·892 Å, H(5,I)···C(18,I′) = 2·886 Å (the distance to the centre of the ring is 2·947 Å). Thus, H(5) is almost facing a ring in the (I′) molecule [which is a distance of *b* away from (I)]. H(9,I)···C(18,IV) = 2·979 Å, but this interaction has poorer geometry. The angle between the phenyl rings is 82·3°.

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cis-2-Phenyl-4-thiolanone 1-Oxide

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Abstract. $C_{10}H_{10}O_2S$, monoclinic, $P2_1/c$, a = 10·858 (7), b = 9·546 (7), c = 21·118 (14) Å, β = 122·66 (2)° at 25 °C, M_r = 194·3, Z = 8, D_m = 1·37, D_x = 1·400 g cm^{-1}. The structure was solved by direct methods and refined to a final R of 0·047 for 1762 observed intensities. The configuration of the phenyl group with respect to the sulfoxide oxygen is *cis*.

Introduction. White, prismatic crystals of the high-melting racemic modification of 2-phenyl-4-thiolanone 1-oxide [m.p. 149–152 °C (dec.)] were obtained by slow recrystallization from ethyl acetate. A single crystal, 0·2 × 0·04 × 0·08 mm, was used for data collection. Preliminary precession photographs showing systematic absences of $h0l$ for $l = 2n + 1$ and $0k0$ for $k = 2n + 1$ identified the space group as $P2_1/c$. Unit-cell parameters were refined by the least-squares method with the diffractometer angles of 12 reflections measured at $\pm 2\theta$ by the automatic centering routine (λ = 0·71069 Å). Intensity data were collected with Mo $K\alpha$ radiation on a Picker FACS-1 diffractometer equipped with a graphite monochromator. All reflec-

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* Contribution No. 2983.