

proposed aryne mechanism for its formation from 2-lithiononafluorobiphenyl.

The phenyl rings are planar to within ± 0.01 Å, although packing effects are possibly indicated by the small displacement (up to 0.05 Å) of substituents from their respective ring planes. The dihedral angle between the central and terminal rings is $78.3(7)^\circ$ which minimizes non-bonding interactions. Evidence for repulsion between Br and the terminal rings is afforded by the fact that C(1)–C(2)–C(5) is 5° greater than C(3)–C(2)–C(5), and the displacement of Br from the plane of the central ring is about 0.03 Å greater than that of C(5).

C–F lengths lie within the range 1.333 to 1.360 Å with a mean of 1.347 Å. Intra-ring C–C lengths range from 1.353 to 1.385 Å with a mean of 1.370 Å and the inter-ring C–C length is 1.473 (14) Å. All bond lengths are uncorrected for thermal libration.

Molecular dimensions of *m*-perfluoroterphenyl and its other 2-substituted derivatives are not available for comparison. The inter-ring bond length and dihedral

angle are both less (by 0.02 Å and 7° respectively) than in 2-nitrononafluorobiphenyl (Hamor & Hamor, 1978).

There are no significantly short intermolecular contacts (Table 3).

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3-Chloro-1-ethyl-2-methyl-1-phenylindene

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Abstract. C₁₈H₁₇Cl, tetragonal, *P4*₁ or *P4*₃, *a* = *b* = 11.493 (2), *c* = 11.013 (2) Å, *Z* = 4, *D*_m = 1.22, *D*_x = 1.23 Mg m⁻³; m.p. 348 K. The structure was solved by *MULTAN*. A full-matrix least-squares refinement using 1530 symmetrically independent reflections with $\sin \theta / \lambda \leq 0.619$ Å⁻¹ gave an *R* value of 0.029 (*R*_w = 0.026, *w* = 1). Bond distances and angles are normal. The planes through the phenyl and indene rings make an angle of $80.3(8)^\circ$.

Introduction. The title compound was obtained by the reaction of 1-phenylpropyne with hydrogen chloride (Vittinghoff, 1980). This reaction was of interest in the investigation of the electrophilic addition of hydrogen halides to phenyl-substituted acetylenes, involving the initial formation of vinyl cation intermediates. The structure determination was undertaken to find out the correct substitution pattern of the indene ring.

Colourless prismatic crystals were obtained by recrystallization from an acetone–pentane solution. The space group and preliminary cell constants were determined by precession photographs which showed the tetragonal symmetry *4/m* and systematic absences *00l* with *l* ≠ *4n*. Characteristic space groups are *P4*₁ or *P4*₃. The cell parameters given above were obtained from the least-squares refinement of the angular positions for 17 reflections carefully centred on a Picker four-circle diffractometer, which was also used to collect the diffraction data.

A prismatic crystal of approximate dimensions 0.2 × 0.2 × 0.3 mm was chosen for data collection with the θ – 2θ scan technique (Mo *K*α radiation, λ = 0.71073 Å, graphite monochromator). The background was measured for 20 s at each limit of the scan range, which was varied to account for the α_1 – α_2 separation. Three reference reflections were measured at intervals of 100 reflections and no significant

decrease of intensity was found. Averaging over symmetrically equivalent reflections yielded 1530 reflections, of which 1324 were classified as observed [$I_{\text{obs}} > 3\sigma(I)$]. Lorentz and polarization corrections were applied but no absorption correction was made [$\mu(\text{Mo } K\alpha) = 0.25 \text{ mm}^{-1}$].

The structure was solved in space group $P4_1$ by straightforward application of *MULTAN* (Germain, Main & Woolfson, 1971) and difference Fourier syntheses. An E map based on the set of phases with the highest combined figure of merit revealed the positions of the Cl atom and of 14 C atoms. The missing C atoms and all H atoms could be located in difference Fourier maps.

The structure model was refined by the conventional full-matrix least-squares technique including isotropic temperature factors for H atoms and anisotropic

temperature factors for the other atoms (Stewart, 1976). During the last cycles of refinement an isotropic extinction correction was applied (Larson, 1967). The final R value was 0.029 for 1530 reflections with 206 'less-than' reflections, of which 126 had a calculated intensity greater than the less-than threshold of 3σ . 240 variables were refined. The weighted R value with unit weights was 0.026. The scattering factors were those of Cromer & Mann (1968) for nonhydrogen atoms and those of Stewart, Davidson & Simpson (1965) for the H atoms. The anomalous-dispersion correction for the Cl atom was taken from *International Tables for X-ray Crystallography* (1974). The enantiomorphous structure in space group $P4_3$ refined to a final R value of 0.030 ($R_w = 0.026$, $w = 1$). As expected, the difference is insignificant. It should be possible to determine the absolute configuration with Cu $K\alpha$ radiation. The atomic coordinates are listed in Table 1.*

Table 1. Final positional parameters and isotropic thermal parameters

The fractional atomic coordinates of the nonhydrogen atoms are multiplied by 10^4 , those of the H atoms by 10^3 ; the thermal parameters are multiplied by 10^3 . U is the refined isotropic temperature factor of the H atoms. $U_{\text{eq}} = \frac{1}{3}\text{trace}(U_{ij})$ refers to the nonhydrogen atoms.

	x	y	z	U_{eq}/U (\AA^2)
Cl	4924 (1)	8565 (1)	4957 (0)	70 (1)
C(1)	7837 (2)	7084 (2)	6288 (3)	47 (1)
C(2)	6721 (3)	7114 (2)	5516 (3)	49 (2)
C(3)	6188 (2)	8129 (3)	5678 (3)	48 (2)
C(4)	6787 (2)	8877 (2)	6559 (3)	46 (2)
C(5)	6517 (3)	9968 (2)	7033 (3)	54 (2)
C(6)	7281 (3)	10435 (3)	7872 (4)	63 (2)
C(7)	8271 (3)	9849 (3)	8221 (4)	64 (2)
C(8)	8532 (3)	8755 (3)	7751 (3)	54 (2)
C(9)	7784 (2)	8268 (2)	6924 (3)	48 (1)
C(10)	8934 (3)	7007 (3)	5470 (4)	64 (2)
C(11)	9053 (5)	7984 (5)	4552 (6)	98 (4)
C(12)	7761 (2)	6077 (2)	7199 (3)	44 (1)
C(13)	7063 (3)	6174 (3)	8216 (3)	56 (2)
C(14)	6946 (3)	5287 (3)	9047 (4)	68 (2)
C(15)	7546 (3)	4252 (3)	8871 (4)	69 (2)
C(16)	8236 (3)	4137 (3)	7872 (4)	64 (2)
C(17)	8344 (3)	5027 (3)	7035 (3)	54 (2)
C(18)	6348 (4)	6134 (4)	4737 (4)	69 (2)
H(5)	581 (3)	1037 (3)	670 (3)	60 (9)
H(6)	717 (3)	1119 (3)	825 (4)	85 (12)
H(7)	878 (3)	1016 (3)	885 (3)	68 (10)
H(8)	921 (3)	831 (3)	810 (4)	75 (11)
H(101)	890 (3)	633 (3)	510 (3)	57 (9)
H(102)	967 (3)	697 (3)	604 (3)	60 (10)
H(111)	970 (4)	791 (4)	410 (5)	106 (16)
H(112)	911 (4)	871 (4)	498 (4)	103 (15)
H(113)	831 (5)	816 (5)	403 (6)	161 (27)
H(13)	667 (3)	694 (3)	837 (3)	69 (10)
H(14)	638 (3)	541 (4)	974 (4)	93 (13)
H(15)	746 (3)	364 (4)	958 (4)	105 (14)
H(16)	859 (3)	342 (3)	774 (3)	63 (9)
H(17)	884 (2)	490 (2)	634 (3)	50 (8)
H(181)	556 (4)	623 (4)	430 (4)	107 (15)
H(182)	682 (5)	603 (5)	390 (6)	157 (22)
H(183)	655 (5)	545 (5)	497 (6)	148 (22)

Discussion. Bond distances and bond angles are given in Table 2. A perspective view of the molecule is in Fig. 1. All bond lengths and angles are normal (Lustenberger, Joss, Engel, Desch, Rutsch & Neuenschwander, 1979). The C(2)–C(3) bond length is 1.330 (4) \AA ,

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35602 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (\AA) and bond angles ($^\circ$) involving the nonhydrogen atoms, with standard deviations in parentheses

C(1)–C(2)	1.540 (4)	C(6)–C(7)	1.377 (5)
C(1)–C(9)	1.531 (4)	C(7)–C(8)	1.393 (5)
C(1)–C(10)	1.553 (5)	C(8)–C(9)	1.372 (4)
C(1)–C(12)	1.534 (4)	C(10)–C(11)	1.517 (7)
C(2)–C(3)	1.330 (4)	C(12)–C(13)	1.382 (5)
C(2)–C(18)	1.480 (5)	C(12)–C(17)	1.392 (4)
C(3)–Cl	1.730 (3)	C(13)–C(14)	1.377 (5)
C(3)–C(4)	1.468 (4)	C(14)–C(15)	1.389 (5)
C(4)–C(5)	1.392 (4)	C(15)–C(16)	1.362 (6)
C(4)–C(9)	1.402 (4)	C(16)–C(17)	1.383 (5)
C(5)–C(6)	1.383 (5)		
C(2)–C(1)–C(9)	101.5 (2)	C(5)–C(6)–C(7)	121.4 (3)
C(2)–C(1)–C(10)	111.0 (3)	C(6)–C(7)–C(8)	121.1 (3)
C(2)–C(1)–C(12)	109.3 (2)	C(7)–C(8)–C(9)	118.7 (3)
C(9)–C(1)–C(10)	110.4 (2)	C(8)–C(9)–C(1)	129.9 (3)
C(9)–C(1)–C(12)	111.7 (3)	C(8)–C(9)–C(4)	119.9 (3)
C(10)–C(1)–C(12)	112.5 (2)	C(1)–C(9)–C(4)	110.2 (2)
C(1)–C(2)–C(3)	109.2 (3)	C(1)–C(10)–C(11)	114.7 (3)
C(1)–C(2)–C(18)	123.0 (3)	C(1)–C(12)–C(13)	120.1 (2)
C(3)–C(2)–C(18)	127.8 (3)	C(1)–C(12)–C(17)	122.8 (3)
C(2)–C(3)–C(4)	112.7 (3)	C(13)–C(12)–C(17)	117.1 (3)
C(2)–C(3)–Cl	125.4 (2)	C(12)–C(13)–C(14)	122.4 (3)
C(4)–C(3)–Cl	121.9 (2)	C(13)–C(14)–C(15)	119.6 (4)
C(3)–C(4)–C(5)	132.1 (3)	C(14)–C(15)–C(16)	119.0 (4)
C(3)–C(4)–C(9)	106.3 (2)	C(15)–C(16)–C(17)	121.3 (3)
C(5)–C(4)–C(9)	121.6 (3)	C(16)–C(17)–C(12)	120.7 (3)
C(4)–C(5)–C(6)	117.3 (3)		

which is typical for a double bond. The C—H bonds in the methyl groups range from 0.86 to 1.07 Å with a mean standard deviation of 0.06 Å. The other C—H bonds have distances between 0.88 and 1.06 Å with a mean standard deviation of 0.03 Å. The corresponding isotropic temperature factors lie between 0.10 and 0.16 Å² for the methyl H atoms and between 0.05 and 0.10 Å² for the other H atoms.

The phenyl ring is planar. For the indene ring system the deviations from planarity are small with a maximum value of 0.02 (1) Å. In the crystalline state the planes through the indene and phenyl rings form an angle of 80.3 (8)° with each other. There are no intermolecular contact distances shorter than the sums of the corresponding van der Waals radii. The packing of the molecules in the unit cell is shown in Fig. 2.

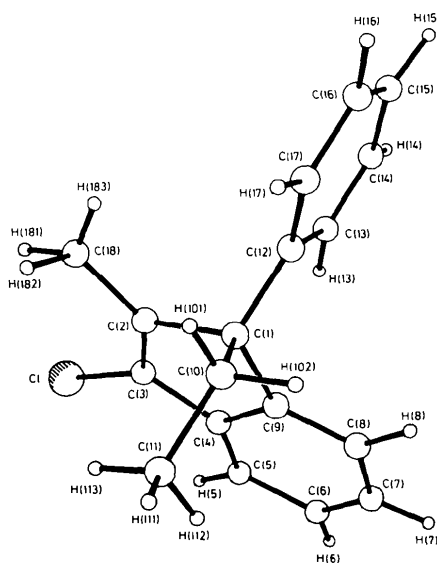


Fig. 1. Perspective view of the C₁₈H₁₇Cl molecule with numbering of the atoms.

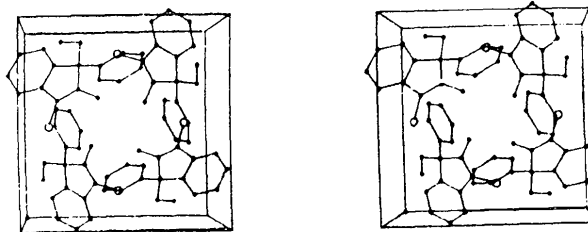


Fig. 2. Stereoscopic view of the molecular packing viewed along *c*.

All calculations were performed on a Univac 1108 computer at the Rechenzentrum der Universität Karlsruhe. The structure plots have been made with the *SHELXTL* system.

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(–)-2(*R*)-[2,3-Dihydro-2(*R*)-isopropyl-4-oxo-4*H*-1-benzopyran-6-yl]-*N*-[1(*R*)-phenylethyl]propionamide: a Chromanone Amide with Three Chiral Centres

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Abstract. C₂₃H₂₇NO₃, orthorhombic, *P*2₁2₁2₁, *Z* = 4, *a* = 34.314 (6), *b* = 11.866 (2), *c* = 5.059 (1) Å, *V* = 2059.8 Å³, *D*_c = 1.18 Mg m⁻³, λ(Cu Kα, Ni-filtered) = 1.5418 Å, *R* = 0.049 for 1207 reflexions. 0567-7408/80/123193-04\$01.00

All H atoms were located. The compound had been synthesized using an (*R*)- α -methylbenzylamine; the present study shows that all three asymmetric centres have the same chirality.

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