The C(methyl)—N bonds are shorter in the anion than in the cation. The differences are between 3σ and 6σ.

I wish to express my gratitude to Professor Ivar Olovsson for the facilities placed at my disposal. I am indebted to Dr Rolf Hesse and Dr Per Jennische for stimulating discussions. The English text was checked by Mr Rajesh Kumar.

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3-Methyl-α,α-diphenyl-1-isoquinolineethanol

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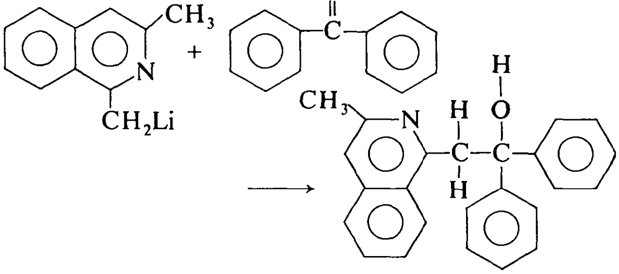
Abstract. $C_{24}H_{21}NO$, monoclinic, $P2_1/c$, a = 13·990 (1), b = 9·703 (1), c = 15·053 (1) Å, β = 117·32 (1)°, Z = 4, ρ_m = 1·241, ρ_c = 1·242 (1) g cm^{-3}, μ = 5·94 cm^{-1}, final $R(F^2)$ = 0·061. The condensation of benzophenone with the monolithiated derivative of 1,3-dimethylisoquinoline is shown to produce the title compound. The geometrical parameters of the aromatic rings are normal. The alcohol group is involved in an intramolecular hydrogen bond with the isoquinoline nitrogen [O···N, 2·702 (2) Å]. The average aromatic C—C distance (1·386 Å) and C—N distance (1·340 Å) are not unusual. The isoquinoline distances are compared with those found in another 1,3 derivative.

Introduction. The compound was prepared by condensation of benzophenone with the monolithiated derivative of 1,3-dimethylisoquinoline (McClure & Kaiser, 1977).



The prismatic crystals were obtained by slow evaporation of an ether solution. Precession photographs gave the systematic absences $h0l$, $l = 2n + 1$ and $0k0$, $k = 2n + 1$, establishing the space group as $P2_1/c$. A crystal (approximate dimensions 0·25 × 0·3 × 0·5 mm) was mounted on a programmed Picker four-circle diffractometer. Accurate cell dimensions and an orientation matrix were obtained by a least-squares fit* to the setting angles of 24 manually centered reflections, and a complete intensity data set was collected with Cu $K\alpha$ radiation (λ = 1·5418 Å) for $2\theta \leq 97$°. The θ–2θ scan technique (1° min^{-1}) was used with a variable width to account for α_1–α_2 splitting. Stationary counter–stationary crystal background measurements were made for 20 s at both ends of each scan. Intensities of 2348 reflections were measured, of which 1862 were independent. Three standards were measured after every fifty reflections and indicated no significant crystal decomposition. The agreement factor between equivalent reflections was 1·1%. The 1840

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* All calculations were performed on the IBM 370/168 computer system of the University of Missouri. The following programs from other scientists were used: W. C. Hamilton & J. A. Ibers, *NUPIK*, Picker input program; R. Doedens & J. A. Ibers, *NUCLS*, least-squares program, a modification of the Busing, Martin & Levy (1962) *ORFLS* program; Zalkin (1962), *FORDAP*, Fourier syntheses program; Busing, Martin & Levy (1964), *ORFFE*, function and error program; Johnson (1965), *ORTEP*, thermal ellipsoid plot program; W. C. Hamilton, *HORSE*, general absorption program; W. C. Hamilton, *SORTH*, sorting program; Germain, Main & Woolfson (1971), *MULTAN*, multiple tangent phasing program.

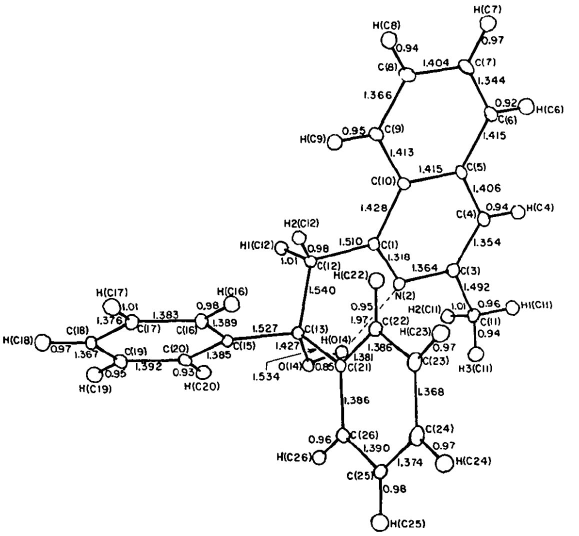

Fig. 1. Molecular geometry, bond distances and thermal ellipsoids for 3-methyl-α,α-diphenyl-1-isoquinolineethanol. Standard deviations are C—C (±0·003 Å); C—O and C—N (±0·002 Å); O—H and C—H (±0·02–0·03 Å).

reflections for which $F_o^2 \geq 2{\cdot}0\sigma(F_o^2)$ were used for the structure solution and refinement. The usual Lorentz, polarization, and absorption corrections were applied. The transmission factor ranged from 0·85 to 0·88.

The nonhydrogen portion of the structure was solved by direct methods (Germain, Main & Woolfson, 1971) and refined by a full-matrix least-squares method [minimizing $\sum w(F_o^2 - F_c^2)^2$] with anisotropic temperature factors to $R(F^2) = 0{\cdot}179$. A subsequent difference Fourier synthesis revealed all hydrogen atom positions. Inclusion of these in the refinement with fixed isotropic temperature factors about 1·0 Å^2 larger than the atom to which they are attached gave a final $R(F^2) = 0{\cdot}061$ and $wR(F^2) = 0{\cdot}098$ where $w = 1/\sigma^2(F_o^2)$ and $\sigma = -0{\cdot}189F_o^4 + 0{\cdot}906F_o^2$ for $F_o^2 < 3{\cdot}58$; $\sigma = -0{\cdot}0278F_o^4 + 0{\cdot}410F_o^2$ for $3{\cdot}58 \leq F_o^2 < 9{\cdot}49$; $\sigma = -0{\cdot}00126F_o^4 + 0{\cdot}147F_o^2$ for $9{\cdot}49 \leq F_o^2 < 43{\cdot}78$; $\sigma = -0{\cdot}000196F_o^4 + 0{\cdot}0956F_o^2$ for $43{\cdot}78 \leq F_o^2 < 128{\cdot}1$; and $\sigma = 0{\cdot}0543F_o^2$ for $F_o^2 \geq 128{\cdot}1$. This weighting scheme was chosen to make $\Delta F^2/\sigma$ nearly independent of F_o^2. Atomic scattering factors for nonhydrogen atoms were taken from Ibers (1962) and those of H from Stewart, Davidson & Simpson (1965). The standard deviation of an observation of unit weight was 1·41.

The final positional parameters are given in Table 1 and important bond angles in Table 2.* Fig. 1 shows the molecular geometry and bond distances.

Table 1. *Positional parameters for 3-methyl-α,α-diphenyl-1-isoquinolineethanol*

Standard deviations are given in parentheses in this table and other portions of the manuscript.

	x	*y*	*z*
C(1)	0·3858 (1)	−0·2501 (2)	−0·1369 (1)
N(2)	0·4501 (1)	−0·1466 (2)	−0·1297 (1)
C(3)	0·5498 (1)	−0·1717 (2)	−0·1213 (1)
C(4)	0·5847 (2)	−0·3022 (2)	−0·1200 (1)
C(5)	0·5201 (2)	−0·4165 (2)	−0·1262 (1)
C(6)	0·5535 (2)	−0·5545 (3)	−0·1243 (2)
C(7)	0·4887 (2)	−0·6599 (3)	−0·1295 (2)
C(8)	0·3864 (2)	−0·6362 (2)	−0·1368 (2)
C(9)	0·3512 (2)	−0·5042 (2)	−0·1393 (1)
C(10)	0·4166 (1)	−0·3908 (2)	−0·1347 (1)
C(11)	0·6161 (2)	−0·0471 (3)	−0·1126 (2)
C(12)	0·2786 (2)	−0·2115 (2)	−0·1425 (2)
C(13)	0·2253 (1)	−0·0786 (2)	−0·1997 (1)
O(14)	0·2942 (1)	0·0363 (1)	−0·1541 (1)
C(15)	0·1236 (1)	−0·0566 (2)	−0·1880 (1)
C(16)	0·0348 (2)	−0·1412 (2)	−0·2389 (2)
C(17)	−0·0555 (2)	−0·1316 (2)	−0·2243 (2)
C(18)	−0·0583 (2)	−0·0360 (2)	−0·1579 (2)
C(19)	0·0278 (2)	0·0492 (2)	−0·1080 (2)
C(20)	0·1189 (2)	0·0403 (2)	−0·1226 (2)
C(21)	0·1961 (1)	−0·0837 (2)	−0·3111 (1)
C(22)	0·1918 (2)	−0·2043 (2)	−0·3615 (1)
C(23)	0·1581 (2)	−0·2046 (3)	−0·4636 (2)
C(24)	0·1295 (2)	−0·0844 (3)	−0·5170 (2)
C(25)	0·1350 (2)	0·0371 (3)	−0·4681 (2)
C(26)	0·1681 (2)	0·0376 (2)	−0·3658 (2)
H(C4)	0·6536 (19)	−0·3169 (23)	−0·1138 (15)
H(C6)	0·6218 (21)	−0·5704 (23)	−0·1173 (17)
H(C7)	0·5148 (18)	−0·7534 (26)	−0·1247 (15)
H(C8)	0·3417 (18)	−0·7104 (25)	−0·1397 (16)
H(C9)	0·2832 (18)	−0·4866 (22)	−0·1425 (15)
H1(C11)	0·6801 (21)	−0·0702 (23)	−0·1180 (17)
H2(C11)	0·6343 (17)	0·0010 (23)	−0·0477 (19)
H3(C11)	0·5797 (19)	0·0161 (24)	−0·1649 (19)
H1(C12)	0·2907 (15)	−0·1976 (19)	−0·0717 (15)
H2(C12)	0·2259 (16)	−0·2862 (21)	−0·1713 (14)
H(O14)	0·3555 (18)	0·0119 (22)	−0·1463 (15)
H(C15)	0·0367 (16)	−0·2110 (21)	−0·2854 (15)
H(C17)	−0·1185 (18)	−0·1948 (24)	−0·2646 (17)
H(C18)	−0·1215 (20)	−0·0317 (22)	−0·1474 (16)
H(C19)	0·0278 (18)	0·1162 (24)	−0·0617 (17)
H(C20)	0·1778 (18)	0·0989 (21)	−0·0903 (16)
H(C22)	0·2120 (16)	−0·2893 (23)	−0·3266 (15)
H(C23)	0·1551 (18)	−0·2925 (26)	−0·4952 (18)
H(C24)	0·1022 (18)	−0·0850 (22)	−0·5894 (20)
H(C25)	0·1154 (18)	0·1252 (25)	−0·5035 (18)
H(C25)	0·1714 (16)	0·1210 (22)	−0·3307 (16)

Discussion. The primary purpose of this study was to establish the site of metallation on interaction of 1,3-dimethylisoquinoline with several organoalkalis. Metallation of both 1-methyl- and 3-methylisoquinoline has been observed (Cannon & Webster, 1957; Knutson & Kaiser, 1977). The benzophenone adduct was chosen for the structural study because of its stability and favorable crystal properties.

The structural study clearly reveals that the condensation occurred at the 1-methyl carbon. The conformation of the molecule is apparently largely deter-

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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 33586 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond angles* (°) *in* 3-*methyl-α,α-diphenyl*-1-*isoquinolineethanol*

C(10)–C(1)–C(12)	121·3 (2)	C(12)–C(13)–C(21)	113·7 (1)
C(10)–C(1)–N(2)	122·6 (2)	C(15)–C(13)–C(21)	109·6 (1)
C(12)–C(1)–N(2)	116·0 (2)	C(15)–C(13)–C(14)	107·2 (1)
C(1)–N(2)–C(3)	120·2 (2)	O(14)–C(13)–C(21)	109·5 (1)
N(2)–C(3)–C(4)	120·9 (2)	C(13)–O(14)–N(2)	82·9 (1)
N(2)–C(3)–C(11)	115·6 (2)	C(16)–C(15)–C(20)	118·1 (1)
C(4)–C(3)–C(11)	123·5 (2)	C(13)–C(15)–C(20)	121·8 (2)
C(3)–C(4)–C(5)	121·3 (2)	C(13)–C(15)–C(16)	119·9 (2)
C(4)–C(5)–C(10)	117·8 (2)	C(15)–C(16)–C(17)	121·5 (2)
C(4)–C(5)–C(6)	123·3 (2)	C(16)–C(17)–C(18)	119·6 (2)
C(6)–C(5)–C(10)	118·9 (2)	C(17)–C(18)–C(19)	119·8 (2)
C(5)–C(6)–C(7)	120·7 (2)	C(18)–C(19)–C(20)	120·9 (2)
C(6)–C(7)–C(8)	121·1 (2)	C(19)–C(20)–C(15)	120·1 (2)
C(7)–C(8)–C(9)	119·8 (2)	C(13)–C(21)–C(22)	123·4 (2)
C(8)–C(9)–C(10)	120·7 (2)	C(13)–C(21)–C(26)	118·9 (2)
C(9)–C(10)–C(5)	118·7 (2)	C(22)–C(21)–C(26)	117·6 (2)
C(9)–C(10)–C(1)	124·2 (2)	C(21)–C(22)–C(23)	121·2 (2)
C(1)–C(10)–C(5)	117·1 (2)	C(22)–C(23)–C(24)	120·6 (2)
C(1)–C(12)–C(13)	116·9 (2)	C(23)–C(24)–C(25)	119·2 (2)
C(12)–C(13)–O(14)	109·8 (1)	C(24)–C(25)–C(26)	120·4 (2)
C(12)–C(13)–C(15)	106·9 (1)	C(25)–C(26)–C(21)	121·0 (2)

mined by the intramolecular hydrogen bond between the OH group and the isoquinoline nitrogen atom [O···N, 2·702 (2); O–H, 0·85 (2); N···H, 1·97 (2) Å; and O–H···N, 145 (2)°]. There are no unusual intermolecular contacts.

The geometry of the isoquinoline system can be compared with that observed for 1-chloro-3-hydroxyisoquinoline (Ammon & Wheeler, 1974). In that compound the two C–N distances are 1·304 (4) and 1·366 (4) Å as compared with 1·318 (2) and 1·364 (2) Å in the present study. The general pattern of variation in aromatic C–C distances in the earlier study was also similar to that observed here with a range of 1·350–1·436 Å compared with 1·343–1·427 Å in the present study.

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Structure de la Galosémide

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Abstract. $C_{15}H_{14}F_3N_3O_3S$, M_r = 373·35, m.p. 168·4 °C, triclinic, space group $P\bar{1}$, Z = 4, a = 14·128 (3), b = 15·176 (2), c = 8·116 (1) Å, α = 91·66 (1), β = 103·79 (2), γ = 105·12 (2)°, V = 1624 Å^3, d_x = 1·53 g cm^{-3}. The structure has been determined from 4653 independent intensities collected on a four-circle automatic diffractometer. It was solved by direct methods and refined by least-squares methods to a final R value of 0·092. The two independent molecules in the asymmetric unit have rather different conformations with disorder in the side chain for one of them. Molecular packing (N···N bonds of 2·765 and 2·885 Å) and some geometrical parameters seem to indicate a proton transfer from the SO_2N group to the pyridyl ring.

Introduction. Les cristaux de galosémide (fournie par le Laboratoire du Professeur Ch. Lapière) ont été obtenus par évaporation lente d'une solution (éthanol + éther de pétrole). Les intensités des rayons X diffractés ont été mesurées à l'aide d'un diffractomètre à quatre cercles Hilger & Watts (radiation Cu $K\alpha$). Nombre de