# Cyclisation studies of 5-arylalkyl-1,2,3,4,5,6,7,8-octahydroisoquinolines

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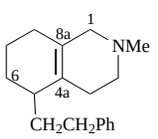
**Treatment of 2-methyl-5-(3-phenylpropyl)-1,2,3,4,5,6,7,8-octahydroisoquinoline with 48% HBr results in isomerisation of the double bond and cyclisation at position-5 of the octahydroisoquinoline ring system to give a novel spiro structure. This is in contrast to the corresponding reaction with 5-[1-(2-phenethyl)]-1,2,3,4,5,6,7,8-octahydroisoquinoline where cyclisation takes place at position-6 of the octahydroisoquinoline ring system. Similar treatment of 5-benzyl-2-methyl-1,2,3,4,5,6,7,8-octahydroisoquinoline results in cyclisation at position-7 of the octahydroisoquinoline ring system to give a novel tetracyclic structure.**

## Introduction

In a previous paper,[1] the cyclisation of 2-methyl-5-phenethyl-1,2,3,4,5,6,7,8-octahydroisoquinoline **1** under acidic conditions

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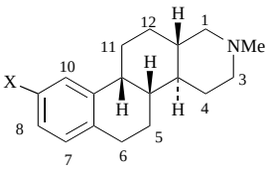


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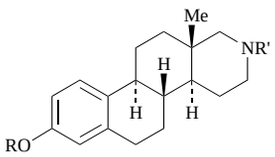


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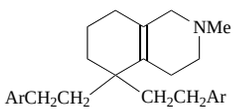



**4**

was described. Cyclisation was found to take place at position-6 of the octahydroisoquinoline ring system to give the tetracyclic structure **2**, which is a novel analogue of the 17-homo-D-aza-D-homoestra-1,3,5(10)triene ring system **3**.

It was proposed that the octahydroisoquinoline double bond isomerised under the acidic conditions as shown in Fig. 1 to give the required carbocation intermediate required for this cyclisation. One particularly interesting feature of this reaction is the absence of any cyclisation at position-4a of the octahydroisoquinoline ring. Even when isomerisation of the double bond to C-5 is blocked by the presence of two substituents at position-5, as in structure **4**, no cyclisation was observed at C-4a and starting material was recovered.

The isomerisation proposed in Fig. 1 proceeds through the favoured tetrasubstituted double bond isomer **5** and then to the less favoured trisubstituted double bond isomer **6** whereupon cyclisation takes place to form a new 6-membered ring.

By increasing the chain length of the alkyl chain between the aromatic ring and the octahydroisoquinoline ring by one C-unit, it seemed likely that the regioselectivity of the cyclisation could be altered since cyclisation at position-5 would give a more favoured 6-membered ring compared to the 7-membered ring which would be obtained from cyclisation at position-6 (Fig. 2).

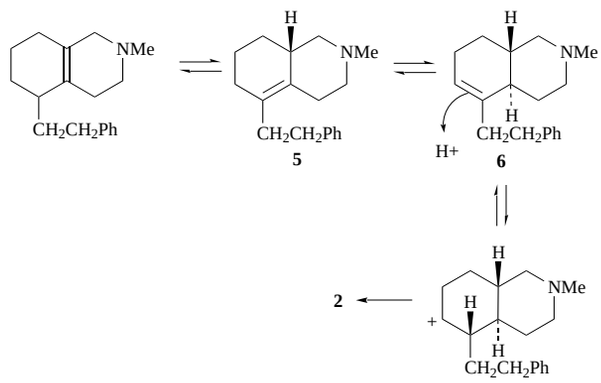



**Fig. 1**

## Results and discussion

The required octahydroisoquinoline intermediate **10** was synthesised by the same synthetic route described previously (Scheme 1). 5,6,7,8-Tetrahydroisoquinoline **7** was synthesised by a literature procedure[2] and alkylated in low yield with potassium amide and (3-iodopropyl)benzene to give the 5-substituted tetrahydroisoquinoline **8**. Treatment with iodomethane used dichloromethane as solvent rather than diethyl ether, since the starting material was insoluble in the latter solvent, to give the methiodide salt **9** which was reduced with sodium borohydride to give **10** in 81% yield over the two steps. The octahydroisoquinoline was heated with 48% HBr for 16 h and the resulting cyclised product **11** was purified by flash chromatography in 54% yield.

High resolution mass spectroscopy showed that the product had the same relative molecular mass as the octahydroisoquinoline starting material **10** while initial $^{1}$H and $^{13}$C NMR analysis clearly revealed that cyclisation had taken place on the aromatic ring at a position *ortho* to the alkyl substituent. The $^{13}$C spectrum (*J*-modulated/APT) showed the presence of two aliphatic CH signals and one aliphatic quaternary signal. The likely compounds which would fit these results are structures **11**, **12** and **13** resulting from cyclisation at positions 5, 8a and 4a respectively. The most likely product would be structure **11** resulting from the formation of a six-membered ring.

Various attempts were made to crystallise the resulting solid both as the free base and as the oxalate salt to allow an X-ray

**Table 1** $^{13}C$, $^{1}H$ and 2D NMR data for 2-methylspiro[(1,2,3,4,4a,5,6,7,8,8a-decahydroisoquinoline) -5, 1′-(1′,2′,3′,4′-tetrahydronaphthalene)] **11**[a]

| Position | $^{13}C$ data | JMOD | $^{1}H$ data[b] | HMBC |
|---|---|---|---|---|
| C-1 | 63.6 | $CH_2$ | 1.64m, 1-$H_{ax}$, 2.85m, 1-$H_{eq}$ | NMe, 3-$H_{eq}$, 8-$H_2$ |
| C-3 | 56.8 | $CH_2$ | 1.81m, 3-$H_{ax}$, 2.77m, 3-$H_{eq}$ | 1-$H_{ax}$, 1-$H_{eq}$, NMe, 4-$H_{ax}$ |
| C-4 | 26.0 | $CH_2$ | 1.05m, 4-$H_{eq}$, 1.43, 4-$H_{ax}$ | 4a-H |
| C-4a | 49.4 | CH | 1.50m, 4a-H | 8a-H, 1-$H_2$, 4-$H_2$ |
| C-5 | 39.9 | C | | 4a-H or 6-$H_{ax}$, 2′-$H_2$, 3′-$H_2$, 8-H |
| C-6 | 40.7 | $CH_2$ | 1.50m, 6-$H_{ax}$, 1.82m, 6-$H_{eq}$ | |
| C-7 | 21.2 | $CH_2$ | 1.52m, 7-$H_{ax}$, 1.63, 7-$H_{eq}$ | 6-$H_2$ |
| C-8 | 31.1 | $CH_2$ | 1.07m, 8-$H_{ax}$, 1.63m, 8-$H_{eq}$ | 8a-H, 7-$H_2$ |
| C-8a | 35.0 | CH | 1.75m, 8a-H | 1-$H_{ax}$, 1-$H_{eq}$, NMe, 8-$H_2$, 4a-H |
| C-2′ | 26.2 | $CH_2$ | 1.74m, 2′-H′, 1.88m, 2′-H″ | 4′-$H_2$, 3′-$H_2$ |
| C-3′ | 19.9 | $CH_2$ | 1.65–1.75m, 3′-$H_2$ | 2′-$H_2$, 4′-$H_2$ |
| C-4′ | 31.0 | $CH_2$ | 2.62–2.70m, 4′-$H_2$ | 2′-$H_2$, 3′-$H_2$, 5′-H, 6′-H |
| C-4a′ | 138.3 | C | | 4′-$H_2$ |
| C-5′ | 128.9 | CH | 6.99m, 5′-H | |
| C-6′ | 125.0 | CH | 7.02m, 6′-H | |
| C-7′ | 125.9 | CH | 7.13m, 7′-H | 8′-H |
| C-8′ | 125.8 | CH | 7.33br d (*J* 8.0), 8′-H | 7′-H, 5′-H |
| C-8a′ | 144.1 | C | | |
| NMe | 46.2 | $CH_3$ | 2.24s | 1-$H_2$, 3-$H_2$ |

[a] 400 MHz $^{1}H$ and 100 MHz $^{13}C$ relative to $CDCl_3$ = 7.27 and 77.23 ppm respectively. [b] Assignments based on $^{1}H$–$^{1}H$ and $^{1}H$–$^{13}C$ correlation experiments (HCCOBIDEC and HMBC).

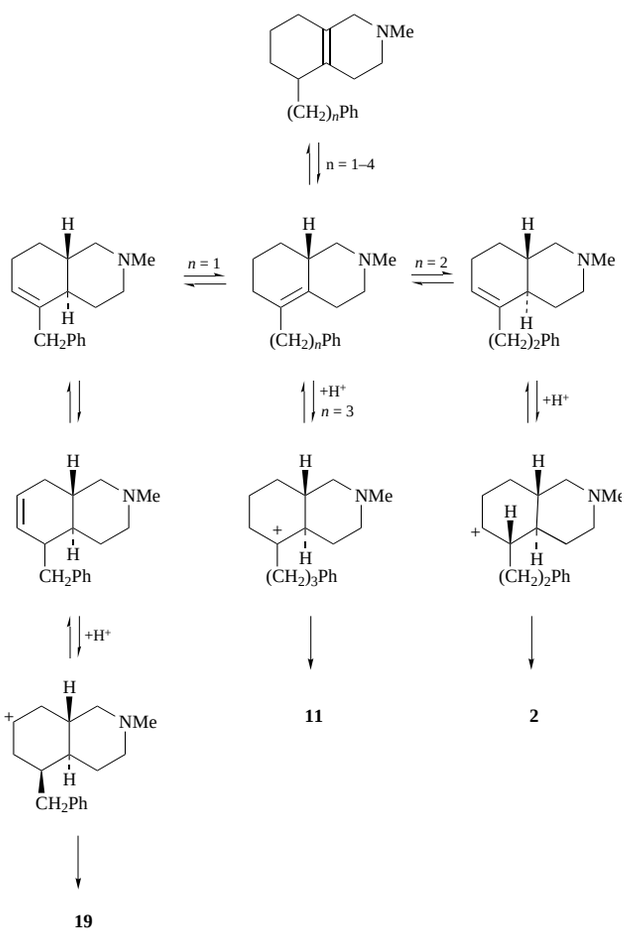
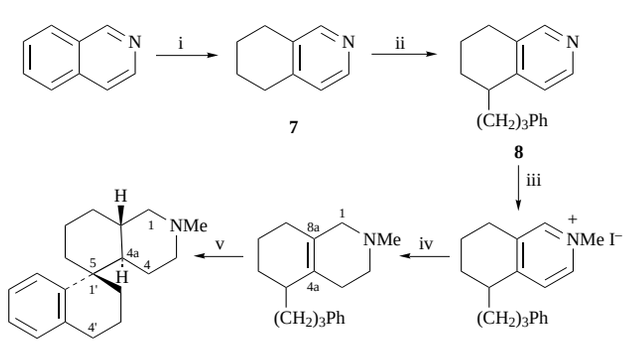



**Fig. 2**

**Table 2** $^{1}H$–$^{1}H$ COSY and NOESY correlations for 2-methylspiro[(1,2,3,4,4a,5,6,7,8,8a-decahydroisoquinoline)-5,1′-(1′,2′,3′,4′-tetrahydronaphthalene)] **11**

| Position | $^{1}H$–$^{1}H$ COSY | NOESY[a] |
|---|---|---|
| 1-$H_{ax}$ | 1-$H_{eq}$, 8a-H | NMe |
| 1-$H_{eq}$ | 1-$H_{ax}$, 8a-H | NMe |
| 3-$H_{ax}$ | 3-$H_{eq}$, 4-$H_2$ | 4-$H_2$, NMe |
| 3-$H_{eq}$ | 3-$H_{ax}$, 4-$H_2$ | 4-$H_2$, NMe |
| 4-$H_{eq}$ | 4a-H, 3-$H_2$ | 3-$H_2$, 4a-H |
| 4-$H_{ax}$ | 3-$H_2$ | 3-$H_{eq}$ |
| 4a-H | 8a-H, 4-$H_{eq}$ | 8′-H, 4-$H_{eq}$ |
| 6-$H_{ax}$ | | 8′-H, 7$H_2$ |
| 7-$H_2$ | 8-$H_2$ | 6-$H_2$, 8-$H_{ax}$ |
| 8-$H_{eq}$ | 7-$H_2$ | 8a-H′ |
| 8-$H_{ax}$ | 7-$H_2$ | 7-$H_2$ |
| 8a-H | 1-$H_2$, 4a-H | 8-$H_{eq}$ |
| 2′-$H_2$ | 3′-$H_2$ | 3′-$H_2$ |
| 3′-$H_2$ | 4′-$H_2$, 2′-$H_2$ | 4′-$H_2$, 2′-$H_2$ |
| 4′-$H_2$ | 3′-$H_2$ | 3′-$H_2$, 5′-H |
| 5′-H | | 4′-$H_2$ |
| 6′-H | 7′-H | 7′-H |
| 7′-H | 6′-H, 8′-H | 6′-H, 8′-H |
| 8′-H | 7′-H | 7′-H, 4a-H, 6-$H_{ax}$ |
| NMe | | 1-$H_2$, 3-$H_2$ |

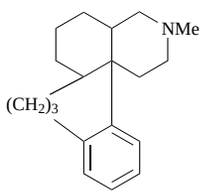
[a] Mixing times 0.4, 0.8 and 1.2 s in NOESY experiments.




**Scheme 1** *Reagents:* i, Pd/C, $H_2$, $CF_3CO_2H$; ii, $KNH_2$, $NH_3$, $Ph(CH_2)_3I$; iii, MeI, $CH_2Cl_2$; iv, $NaBH_4$, EtOH; v, HBr

analysis but these crystallisations were unsuccessful. Consequently, the structure of the cyclised product was determined by subjecting the product to 2D homonuclear chemical-shift correlation COSY, NOESY and heteronuclear correlated (HMBC)[3] spectral techniques (Tables 1 and 2). These experiments established structure **11** as the product of cyclisation (Table 1).

The $^{13}C$ signals at 63.6 and 56.8 ppm were assigned to positions 1 and 3 respectively by comparison with *trans*-*N*-methyldecahydroisoquinoline.[4] These assignments were confirmed by HMBC correlations between the *N*-methyl protons

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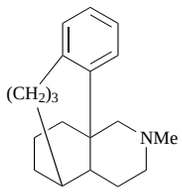


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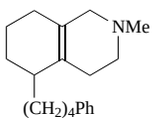


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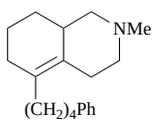


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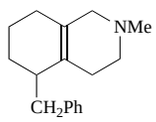


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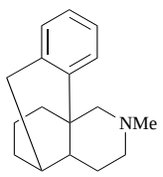


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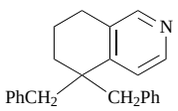


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and C-1/C-3. The protons at positions 1 and 3 were assigned as axial or equatorial based on their coupling patterns and coupling constants. The methyl group was established as being in the equatorial position due to observed NOESY interactions with both the equatorial and axial protons at positions 1 and 3. Further protons were assigned as being axial or equatorial based on $^{1}$H–$^{1}$H COSY and NOESY interactions.

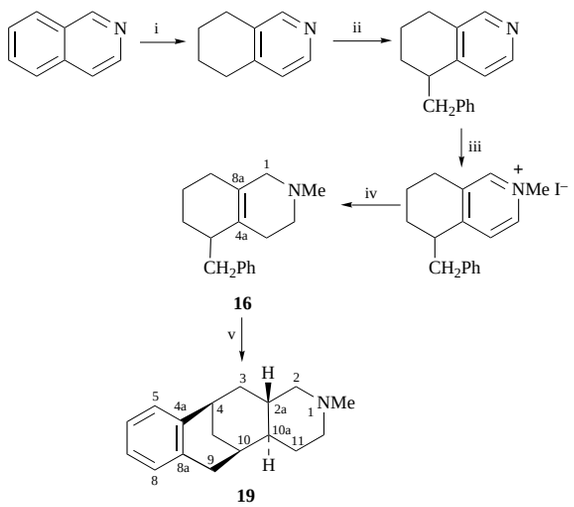
The stereochemistry of the three ring junctions was assigned as follows: The bond between C-4a and C-8a was assigned as *trans*, both on the basis of the diagnostic $^{13}$C chemical shifts, as well as by comparison with *trans*-*N*-methyldecahydroisoquinoline.[4] Further evidence for the *trans* arrangement comes from the fact that there is a COSY relationship between the 4a and 8a protons but no NOESY interaction.

The stereochemistry at the spiro centre was determined from $^{1}$H–$^{1}$H NOESY interactions. NOESY interactions involving the protons at positions 4a and 6(axial) with the aromatic proton at position-8′ established that the aromatic ring is directly attached to the decahydroisoquinoline ring at the equatorial position.

As anticipated, cyclisation at C-5 of the octahydroisoquinoline ring system was preferred over cyclisation at position-6 since the former produced a more stable six-membered ring. As with the previous study,[1] there was no evidence of a product arising from cyclisation at either C-4a or C-8a.

Further studies were carried out to investigate the effect of increasing the chain length between the decahydroisoquinoline ring system and the aromatic ring. Thus, the octahydroisoquinoline intermediate **14** was prepared where the methylene chain length of the phenylalkyl chain was increased to four carbon units. Treatment with 48% hydrobromic acid gave a mixture of products. NMR analysis of the crude mixture suggested that the major product was the isomerised octahydroisoquinoline **15**. Unlike for the octahydroisoquinolines **1** and **10**, the isomerisation of **14** involves the double bond moving out from the decahydroisoquinoline ring system to the side chain in order to obtain a six-membered cyclisation. The experimental evidence suggests that this is not favoured.

As a result of these experiments, it was decided to reinvestigate a previous report[5] which described the cyclisation of 5-benzyl-2-methyl-1,2,3,4,5,6,7,8-octahydroisoquinoline **16** as taking place at position-8a of the octahydroisoquinoline ring system to give structure **17**. The structure of **17** was determined from IR analyses and did not consider the possibility of other isomers, formed from cyclisation at other sites of the octahydroisoquinoline ring system. The required octahydroisoquinoline intermediate **16** was synthesised by the route shown in Scheme 2. 5,6,7,8-Tetrahydroisoquinoline was alkylated in 50% yield




**Scheme 2** *Reagents:* i, Pd/C, $H_2$, $CF_3CO_2H$; ii, $KNH_2$, $NH_3$, $PhCH_2Cl$; iii, MeI, $CH_2Cl_2$; iv, $NaBH_4$, EtOH; v, HBr

with potassium amide and benzyl chloride. A small quantity of the dialkylated product **18** was obtained in 8% yield. Treatment with iodomethane in diethyl ether gave the methiodide salt in 75% yield which was reduced with sodium borohydride to give **16** in 84% yield. The octahydroisoquinoline was heated with 48% HBr overnight to give one major product as identified by TLC along with a mixture of minor components. Purification by the crystallisation procedure described by Sugimoto *et al.*[5] was unsuccessful, since impurities were found to crystallise out along with the major product. Purification was carried out instead by distillation under reduced pressure followed by column chromatography.

The product was subjected to a variety of NMR techniques in order to determine the structure. The $^{13}$C spectrum (*J*-modulated/APT) showed the presence of four aliphatic CH signals and no aliphatic quaternary signal thus eliminating structure **17**. 2D Homonuclear chemical-shift correlation (COSY), NOESY and heteronuclear correlated (HMBC)[3] spectral techniques established that the structure was the methano bridged benzocyclooctenopyridine **19** (Tables 3 and 4).

The $^{13}$C signals at 62.5 and 56.3 ppm were assigned to positions 2 and 12, respectively, by comparison with *trans*-*N*-methyldecahydroisoquinoline.[4] The protons at positions 2 and 12 were assigned as axial or equatorial based on their coupling patterns and coupling constants. The methyl group was established as being in the equatorial position due to observed NOESY interactions with both the equatorial and axial protons at position-2. Further protons were assigned as being axial or equatorial based on COSY and NOESY interactions based on the above assignments.

The stereochemistry of the four ring junctions was assigned as follows: The bond between 2a and 10a was assigned as *trans*, both on the basis of the diagnostic $^{13}$C chemical shifts[6] as well as by comparison with *trans*-*N*-methyldecahydroisoquinoline[4] and the lack of a NOESY interaction between the protons at these positions. NOESY interactions between the hydrogen at position-4 and both hydrogens at position-3 establish the former as being in the equatorial position. The remaining ring junction at position-10 must have the proton in an equatorial position due to stereochemical constraints. This is confirmed by

**Table 3** $^{13}$C, $^{1}$H and 2D NMR data for (±)-(2aα,4β,10β,10aβ)-1-methyl-1,2,2a,3,4,9,10,10a,11,12-decahydro-4,10-methanobenzocycloocteno-[7,8-*c*]pyridine **19** [a]

| Position | $^{13}$C data | DEPT | $^{1}$H data [b] | HMBC |
|---|---|---|---|---|
| C-2 | 62.5 | $CH_2$ | 1.17t (*J* 11.0), 2-$H_{ax}$, 2.62m, 2-$H_{eq}$ | 10a-H, 12-$H_2$, 2a-H, 3-$H_2$ |
| C-2a | 33.3 | CH | 1 62m, 2a-$H_{ax}$ | 2-$H_2$, 10a-H, 3-$H_{eq}$ |
| C-3 | 36.2 | $CH_2$ | 0.95m, 3-$H_{ax}$, 2.05m, 3-$H_{eq}$ | 2a-H, 10a-H, methano-$H_2$, 5-H |
| C-4 | 31.7 | CH | 2.91m, 4-$H_{eq}$ | methano-$H_2$ |
| C-4a | 145.9 | C | | 4-H, 6-H |
| C-5 | 129.2 | CH | 6.96m, 5-H | 4-H |
| C-6 | 125.7 | CH | 7.01m, 6-H | |
| C-7 | 126.0 | CH | 7.03m, 7-H | |
| C-8 | 130.1 | CH | 6.94m, 8-H | |
| C-8a | 134.8 | C | | 7-H, 10-H |
| C-9 | 36.8 | $CH_2$ | 2.48d (*J* 16.0), 9-$H_{exo}$, 2.74m, 9-$H_{endo}$ | |
| C-10 | 32.3 | CH | 1.68m, 10-H | 9-$H_2$, 10a-H methano-$H_2$ |
| C-10a | 40.0 | CH | 0.64m, 10a-H | 10-H |
| C-11 | 33.8 | $CH_2$ | 1.38m, 11-$H_{eq}$, 1.65m, 11-$H_{ax}$ | 10a-H, 12-$H_2$ |
| C-12 | 56.3 | $CH_2$ | 1.59m, 12-$H_{ax}$, 2.70m, 12-$H_{eq}$ | 2-$H_2$, 11-$H_2$, NMe |
| NMe | 46.2 | $CH_3$ | 2.04s | 2-$H_2$, 12-$H_2$ |
| methano bridge | 25.9 | $CH_2$ | 1.36m, methano $H_{eq}$ 1.85m, methano $H_{ax}$ | 9-$H_2$, 10-H |

[a] 400 MHz $^{1}$H and 100 MHz $^{13}$C relative to $CHCl_3$ = 7.27 and 77.23 ppm. [b] Assignments based on $^{1}$H–$^{1}$H and $^{1}$H–$^{13}$C correlation experiments (HCCOBIDEC and HMBC).

**Table 4** $^{1}$H–$^{1}$H COSY and NOESY correlations for (±)-(2aα,4β,10β,10aβ)-1-methyl-1,2,2a,3,4,9,10,10a,11,12-decahydro-4,10-methanobenzocycloocteno[7,8-*c*]pyridine **19**

| Position | $^{1}$H–$^{1}$H COSY | NOESY |
|---|---|---|
| 2-$H_{eq}$ | 2a-H, 2-$H_{ax}$, NMe | 3-$H_{eq}$, NMe |
| 2-$H_{ax}$ | 2-$H_{eq}$, 2a-H | NMe |
| 2a-H | 10a-H, 3-$H_2$, 2-$H_{eq}$, 2-$H_{ax}$ | 3-$H_{eq}$ |
| 3-$H_{eq}$ | 2a-H | 4-H, 2a-H, 2-$H_{eq}$ |
| 3-$H_{ax}$ | 2a-H, 4-H | 4-H |
| 4-H | methano-H′, 3-$H_{ax}$ | 5-H, 3-$H_{eq}$, 3-$H_{ax}$ |
| 5-H | | 4-H |
| 6-H | | |
| 7-H | | |
| 8-H | | 9-$H_{exo}$ |
| 9-$H_{endo}$ | methano-$H_{ax}$ | 11-$H_{ax}$ |
| 9-$H_{exo}$ | | 8-H, methano-$H_{eq}$ |
| 10-H | methano-$H_{eq}$ | 10a-H |
| 10a-H | 2a-H, 11$H_2$ | 10-H |
| 11-$H_{eq}$ | 10a-H, 12-$H_{ax}$ | |
| 11-$H_{ax}$ | 10a-H, 12-$H_{ax}$ | 9-$H_{endo}$ |
| 12-$H_{ax}$ | 11-$H_2$ | |
| NMe | 2-$H_{eq}$ | 2-$H_2$ |
| methano-$H_{ax}$ | 9-$H_{endo}$ | |
| methano-$H_{eq}$ | 10-H | |

a $^{1}$H–$^{1}$H NOESY interaction between this proton and the axial proton at position-10a. Thus, the NMR analysis establishes that the major cyclisation product from the reaction of 5-benzyl-2-methyl-1,2,3,4,5,6,7,8-octahydroisoquinoline with HBr results from cyclisation at position-7 of the octahydroisoquinoline ring system. Other products were observed on TLC but these were in such small quantity that they were not identified.

It is therefore possible that the product obtained by Sugimoto *et al.*[5] was structure **19** and not structure **17** as they reported. If this is the case, it could well account for the poor analgesic results reported in their paper. The stereochemical relationship between the aromatic ring and the basic centre (both important groups for receptor binding) in structure **17** is quite similar to the relationship of these groups in *N*-methylmorphinan and comparable analgesic activity might have been expected. By comparison, the stereochemical relationship between the aromatic ring and the nitrogen atom in structure **19** is markedly different. The formation of structure **19** would arise from isomerisation of the octahydroisoquinoline double bond under acid conditions to give the carbocation at position-7 which would then cyclise to give the observed product (Fig. 2).

These studies demonstrate that acid-catalysed isomerisation of the double bond in 5-substituted arylalkyl octahydroisoquinolines is a facile process and leads to the formation of six-membered rings in preference to more strained five-membered or seven-membered rings. The specific position of cyclisation is thus controlled by the length of the arylalkyl chain. This isomerisation is apparently constrained to the ring without the heteroatom, since no cyclised products were observed either to the heterocyclic ring or to the side chain.

## Experimental

### General details

Chromatography was carried out on silica gel (Merck 9385) at normal pressures. Mps (uncorrected) were obtained on a Kofler block. IR spectra were recorded as thin films on a Perkin-Elmer 298 infrared spectrophotometer. $^{1}$H and $^{13}$C NMR spectra were recorded at 90, 200 and 400 MHz on JEOL EX90, JEOL FX200 and Bruker AMX400MHz spectrometers respectively using $CDCl_3$ a solvent; chemical shifts are given in ppm values relative to the solvent $CDCl_3$ as internal reference, $\delta$ 7.27 ppm for $^{1}$H NMR spectra, $\delta$ 77.23 ppm for $^{13}$C NMR spectra. The number of hydrogens attached to each carbon in the $^{13}$C NMR spectra were determined by DEPT and JMOD/APT techniques. *J*-Values are given in Hz to the nearest 0.5 Hz. Low resolution and high resolution mass spectra were recorded on a V.G. Masslab 7070F spectrometer and a JEOL 505HA spectrometer, with only molecular ions ($M^+$) and major peaks being reported. The progress of all reactions was followed by TLC analysis using Kieselgel 60 $F_{254}$ plates which were visualised by UV fluorescence ($\lambda_{max}$ = 254 nm), and by staining with iodine vapour. $R_F$ Values are quoted to the nearest 0.05. All solvents were distilled before use. Anhydrous diethyl ether was obtained by distillation from sodium/benzophenone ketal under nitrogen. Anhydrous dichloromethane was obtained by distillation from phosphorus pentaoxide and stored over molecular sieves (4 Å). Anhydrous acetone was obtained by storage of redistilled acetone over molecular sieves (4 Å). Triethylamine was distilled from potassium hydroxide pellets. Isoquinoline, benzyl chloride, 3-phenylpropan-1-ol, 4-phenylbutyric acid and methanesulfonyl chloride were obtained from the Aldrich Chemical Company and distilled before use. Organic solutions were dried over magnesium sulfate and concentrated under reduced pressure at not more than 40 °C.

### 5,6,7,8-Tetrahydroisoquinoline

Isoquinoline was reduced with hydrogen over a 5% palladium

charcoal catalyst using trifluoroacetic acid as solvent by the method of Vierhapper and Eliel.[2]

**5-(3-Phenylpropyl)-5,6,7,8-tetrahydroisoquinoline 8**

Potassium (2.93 g, 75.0 mmol) was added in portions to a solution of ammonia (350 $cm^3$) containing $Fe(NO_3)_3 \cdot 9H_2O$ (60 mg) to produce a blue solution. The solution was kept and occasionally swirled for 20 min, by which time the blue colour had dissipated. 5,6,7,8-Tetrahydroisoquinoline **7** (10.0 g, 75.0 mmol) was added dropwise over 10 min and the reaction mixture was kept for a further 20 min and occasionally swirled to give a orange–red solution. (3-Iodopropyl)benzene (18.45 g, 75.0 mmol) was added over a period of 10 min, with swirling of the mixture, to give an orange solution which was kept overnight to give a green–brown residue. The residue was taken up in ice–water and chloroform. The aqueous phase was extracted twice with chloroform then the organic extracts were combined, washed twice with water, then extracted twice with 4 mol $dm^{-3}$ hydrochloric acid. The acidic extracts were washed three times with chloroform, then neutralised (pH paper) with sodium hydrogen carbonate and extracted twice with chloroform. The organic extracts were dried, filtered and concentrated to give a yellow oil, which was distilled under reduced pressure to give the mono-substituted tetrahydroisoquinoline **8** (2.35 g, 12.5%); bp 150–180 °C/3 mmHg; $R_F$ 0.38 (EtOAc) (Found: N, 5.59%; $M^+$, 251.1714. $C_{18}H_{21}N$ requires N, 5.57%; *M*, 251.1674); *m*/*z* 251, 146, 132, 117 and 91; $\nu_{max}$(film)/$cm^{-1}$ 3035m, 2950s, 2870m, 1600m, 1505m, 1460m, 835m, 755m and 705s; $\delta_H$ 8.29 (1 H, s, 1-H), 8.28 (1 H, d, *J* 5.0, 3-H), 7.38–7.00 (5 H, m, Ar-H), 6.98 (1 H, d, *J* 5.0, 4-H), 3.08–2.24 (5 H, m) and 2.24–1.20 (8 H, m); $\delta_C$ 150.2 (CH; C-1), 150.1 (C; C-4a), 146.4 (CH; C-3), 142.2 (C-1′), 132.8 (C; C-8a), 128.3 (CH; C-2′ -3′, -5′ and -6′), 125.8 (CH; C-4′), 122.9 (CH; C-4), 36.8 (CH; C-5), 35.9 ($CH_2$), 35.3 ($CH_2$), 28.7 ($CH_2$), 26.9 ($CH_2$), 26.5 ($CH_2$) and 19.7 ($CH_2$; C-7).

**2-Methyl-5-(3-phenylpropyl)-1,2,3,4,5,6,7,8-octahydroisoquinoline 10**

The tetrahydroisoquinoline **8** (2.35 g, 9.4 mmol) was taken up in anhydrous dichloromethane (200 $cm^3$); the solution was treated with iodomethane (2.67 g, 1.17 $cm^3$, 18.8 mmol) and stirred for 24 h. The solvent was removed under reduced pressure to give the methiodide **9** (3.69 g, 100%); $R_F$ 0.30 (MeOH–AcOH; 10:1); $\nu_{max}$(film)/$cm^{-1}$ 3020, 2930, 2860, 1640, 1495, 1480, 1455, 1310 and 1240; $\delta_H$ 9.05 (1 H, s, 1-H), 8.84 (1 H, d, *J* 6.0, 3-H), 7.71 (1 H, d, *J* 6.0, 4-H), 7.35–7.21 (5 H, m, Ar-H), 4.50 (3 H, s, NMe), 3.35–2.66 (5 H, m) and 1.83–1.73 (8 H, m); $\delta_C$ 162.2 (C; C-4a), 144.9 (C; C-8a), 141.5 (CH; C-1), 141.3 (CH; C-3), 138.6 (C; C-1′), 128.8 (CH; C-2′ and -6′), 128.5 (CH; C-3′ and -5′), 127.1 (CH; C-4), 126.1 (CH; C-4), 48.4 ($CH_3$; NMe), 38.0 (CH; C-5), 35.6 ($CH_2$), 34.9 ($CH_2$), 28.6 ($CH_2$), 25.6 ($CH_2$) and 18.4 ($CH_2$; C-7).

The methiodide was used for the following step without further purification. The methiodide was taken up in ethanol (100 $cm^3$), and the mixture treated at room temperature with sodium boranuide (0.55, 14.6 mmol) in portions, while being stirred. The solution was stirred overnight. The solvent was removed under reduced pressure and the residue was taken up in a mixture of dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were dried, filtered and concentrated to give a residue which was distilled under reduced pressure to give the octahydroisoquinoline **10** as a yellow oil (2.04 g, 81%); bp 120 °C/3 mmHg; $R_F$ 0.42 (MeOH–AcOH; 10:1) (Found: $M^+$, 269.2115. $C_{19}H_{27}N$ requires *M*, 269.2143); *m*/*z* 269, 150, 122 and 91; $\nu_{max}$(film)/$cm^{-1}$ 3035m, 2940s, 2795s, 1606m, 1500m, 1460s, 810m, 750m and 703s; $\delta_H$ 7.24–7.09 (5 H, m, Ar-H), 2.80–2.30 (7 H, m), 2.33 (3 H, s, NMe) and 2.00–1.20 (12 H, m); $\delta_C$ 152.7 (C; C-1′), 129.5 (C; C-4a), 128.3 (CH; C-2′ and -6′), 128.2 (CH; C-3′ and -5′), 127.0 (C; C-8a), 125.5 (CH, C-4′), 59.1 ($CH_2$; C-1), 52.8 ($CH_2$; C-3), 45.8 ($CH_3$; NMe), 38.0 (CH; C-5), 36.2 ($CH_2$), 32.2 ($CH_2$), 29.1 ($CH_2$), 28.8 ($CH_2$), 28.0 ($CH_2$), 27.7 ($CH_2$) and 20.0 ($CH_2$; C-7).

**(±)-2-Methylspiro[(1,2,3,4,4a,5,6,7,8,8a-decahydroisoquinoline)-5, 1′-(1′,2′,3′,4′-tetrahydronaphthalene)] 11**

The octahydroisoquinoline (0.96 g, 3.57 mmol) **10** was heated to reflux and stirred in 48% hydrobromic acid (10.0 $cm^3$) overnight. The solvent was removed under reduced pressure and the residue was taken up in water. The aqueous solution was basified with aqueous potassium hydrogen carbonate and extracted twice with chloroform. The combined extracts were dried, filtered and concentrated to give an oil which was purified by column chromatography (silica gel, 70–230 mesh, 60 Å) using $CHCl_3$–MeOH, 10:1 as eluent to give the spiro product **11** as a slightly yellow solid (0.52 g, 54%); $R_F$ 0.29 ($CHCl_3$–MeOH; 10:1) (Found: $M^+$, 269.2174. $C_{19}H_{27}N$ requires *M*, 269.2143); *m*/*z* 269, 268, 110, 96 and 58; $\nu_{max}$(film)/$cm^{-1}$ 2910, 2770, 1675, 1485, 1460, 1445, 1380, 1285 and 1145; $\delta_H$ see Table 1; $\delta_C$ see Table 1.

**1-Methylsulfonyloxy-4-phenylbutane**

4-Phenylbutanol (19.7 g, 130 mmol) was taken up in anhydrous dichloromethane (250 $cm^3$) containing triethylamine (25.0 $cm^3$) and cooled in an ice bath. Methanesulfonyl chloride (16.1 g, 140 mmol) was added with stirring at such a rate that the internal temperature did not rise above 5 °C,[7] and stirring was continued for an additional 16 h at room temperature. The solution was washed with ice cold water, ice cold 4 M hydrochloric acid, saturated aqueous sodium hydrogen carbonate and finally water. The organic solution was dried, filtered and concentrated to give the ester as a yellow oil (28.4 g, 96%); $R_F$ 0.60 (EtOAc–light petroleum 40–60 °C, 2:3) (Found: C, 57.84; H, 6.74%; $M^+$, 288.0811. $C_{11}H_{16}SO_3$ requires C, 57.87; H, 7.07%; *M*, 228.0820); $\nu_{max}$(film)/$cm^{-1}$ 3000, 2920, 2840, 1590, 1480, 1440, 1340 and 1160; *m*/*z* 288, 132, 117, 104 and 94; $\delta_H$ 7.18 (5 H, m, Ar-H), 4.16 (2 H, t, *J* 6.0, $CH_2O$), 2.89 (3 H, s, $CH_3$), 2.62 (2 H, m, $ArCH_2$) and 1.71 (4 H, m, $CH_2CH_2CH_2OH$); $\delta_C$ 141.9 (C; C-1′), 128.4 (CH; C-2′, -3′, 5′ and -6′), 125.9 (CH; C-4′), 69.9 ($CH_2$; C-1), 37.1 ($CH_3$), 35.1 ($CH_2$; C-4), 28.6 ($CH_2$; C-2) and 27.9 ($CH_2$; C-3).

**(4-Iodobutyl)benzene**

The above ester (28.4 g, 124 mmol) was taken up in anhydrous acetone (350 $cm^3$) and the stirred solution was treated[8] with sodium iodide (82.5 g, 0.55 mol) for 24 h at room temperature to give a precipitate. The mixture was concentrated and the resulting slurry was taken up in water and diethyl ether. The organic and aqueous phases were separated and the aqueous phase was extracted with diethyl ether. The organic extracts were combined, washed with water, then dried, filtered and concentrated to give the iodide as a yellow oil (30.8 g, 95%); $R_F$ 0.50 (light petroleum 60–80 °C) (Found: H, 4.90. $C_9H_{11}I$ requires H, 4.82%); *m*/*z* 133 and 91; $\delta_H$ 7.21 (5 H, m, Ar-H), 3.12 (2 H, t, *J* 6.0, $CH_2I$), 2.58 (2 H, t, *J* 6.0, $ArCH_2$) and 1.74 (4 H, m, $ArCH_2CH_2CH_2$); $\delta_C$ 142.0 (C; C-1′), 128.2 (CH; C-2′, -3′, -5′ and -6′), 125.7 (CH; C-4′), 34.6 ($CH_2$; $ArCH_2$), 32.8 ($CH_2$; $CH_2CH_2I$), 32.0 ($CH_2$; $ArCH_2CH_2$) and 6.7 ($CH_2$; $CH_2I$).

**5-(4-Phenylbutyl)-5,6,7,8-tetrahydroisoquinoline**

Potassium (5.86 g, 150 mmol) was added in portions to a solution of ammonia (600 $cm^3$) containing $Fe(NO_3)_3 \cdot 9H_2O$ (60 mg) to produce a blue solution. The solution was kept and occasionally swirled for 20 min, by which time the blue colour had dissipated. 5,6,7,8-Tetrahydroisoquinoline **7** (20.0 g, 150.0 mmol) was added dropwise over 10 min and the reaction mixture was kept for a further 20 min and occasionally swirled to give an orange–red solution. (4-Iodobutyl)benzene (39.0 g, 150.0 mmol) was added over a period of 10 min, with swirling of the mixture, to give an orange solution which was kept over-

night to give a green–brown residue. The residue was taken up in ice–water and chloroform. The aqueous phase was extracted twice with chloroform then the organic extracts were combined, washed twice with water, then extracted twice with 4 mol $dm^{-3}$ hydrochloric acid. The acidic extracts were washed three times with chloroform, then neutralised (pH paper) with sodium hydrogen carbonate and extracted twice with chloroform. The organic extracts were dried, filtered and concentrated to give the mono-substituted tetradroisoquinoline as a yellow oil (6.49 g, 16%); $R_F$ 0.45 ($CHCl_3$–MeOH, 10:1) (Found: H, 8.43; N, 5.07%; $M^+$, 265.1808. $C_{19}H_{23}N$ requires H, 8.74; N, 5.28%; *M*, 265.1830); *m*/*z* 265, 146, 132, 104, 91 and 77; $\nu_{max}$(film)/$cm^{-1}$ 3015, 2935, 2860, 1595, 1495, 1455 and 1415; $\delta_H$ 8.27 (2 H, m, 1- and 3-H), 7.19 (5 H, m, Ar-H), 6.97 (1 H, d, *J* 5.0, 4-H), 2.67–2.53 (5 H, m) and 1.79–1.40 (10 H, m); $\delta_C$ 150.4 (CH; C-1), 150.0 (C; C-4a), 146.6 (CH; C-3), 142.4 (C-1′), 132.7 (C; C-8a), 128.3 (CH; C-3′ and -5′), 128.2 (CH; C-2′ and C-6′), 125.7 (CH; C-4′), 122.9 (CH; C-4), 36.9 (CH; C-5), 35.8 ($CH_2$), 31.5 ($ArCH_2CH_2$), 26.9 ($CH_2$), 26.7 ($CH_2$), 26.6 ($CH_2$) and 19.7 ($CH_2$; C-7).

**2-Methyl-5-(4-phenylbutyl)-1,2,3,4,5,6,7,8-octahydroisoquinoline 14**

5-(4-Phenylbutyl)-5,6,7,8-tetrahydroisoquinoline (6.49 g, 24.5 mmol) was taken up in anhydrous diethyl ether (75 $cm^3$); the solution was treated with iodomethane (3.50 g, 24.5 mmol) and stirred for 24 h. The resulting precipitate was filtered off to give 2-methyl-5-(4-phenylbutyl)-5,6,7,8-tetrahydroisoquinolium iodide as a hygroscopic yellow solid (7.93 g, 80%); $R_f$ 0.30 ($CHCl_3$–MeOH, 10:1) (Found: H, 6.40; N, 3.46. $C_{20}H_{26}NI$ requires H, 6.44; N, 3.44%); $\nu_{max}$(film)/$cm^{-1}$ 3025, 2940, 2860, 1645, 1510, 1455, 1315 and 1240; $\delta_H$ 9.19 (1 H, s, 1-H), 8.95 (1 H, d, *J* 6.0, 3-H), 7.72 (1 H, d, *J* 6.0, 4-H), 7.20 (5 H, m, Ar-H), 4.53 (3 H, s, NMe), 2.97 (3 H, m), 2.58 (2 H, m, $ArCH_2$) and 1.79–1.54 (10 H, m); $\delta_C$ 161.7 (C; C-4a), 144.6 (CH; C-1), 142.1 (C; C-1′), 141.4 (CH; C-3), 138.5 (C; C-8a), 128.3 (CH; C-2′, -3′, -5′ and -6′), 127.2 (CH; C-4), 125.7 (CH; C-4′), 48.1 ($CH_3$; NMe), 38.0 (CH; C-5), 35.6 ($CH_2$), 35.4 ($CH_2$), 31.1 ($CH_2$), 26.4 ($CH_2$), 25.5 ($CH_2$) and 19.3 (CH; C-7).

The methiodide was used for the next step without further purification. The methiodide (7.63 g, 19 mmol) was taken up in ethanol (100 $cm^3$), and the mixture treated at room temperature with sodium boranuide (1.46 g, 38 mmol) in portions while being stirred. The solution was stirred overnight. The solvent was removed under reduced pressure and the residue was taken up in a mixture of dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were dried, filtered and concentrated to give the octahydroisoquinoline **14** as a yellow oil (4.54 g, 84%); $R_F$ 0.50 ($CHCl_3$–MeOH; 10:1) (Found: $M^+$, 283.2319. $C_{20}H_{29}N$ requires *M*, 283.2300); *m*/*z* 283, 150, 122, 91 and 77; $\nu_{max}$(film)/$cm^{-1}$ 3020, 2930, 2850, 2770, 1660, 1600, 1500 and 1450; $\delta_H$ 7.25–7.18 (5 H, m, Ar-H), 2.68–2.41 (6 H, m), 2.30 (3 H, s, NMe) and 2.28–1.47 (15 H, m); $\delta_C$ 142.8 (C; C-1′), 129.6 (C; C-4a), 128.3 (CH; C-3′ and -5′), 128.2 (CH; C-2′ and -6′), 126.9 (C; C-8a), 125.6 (CH; C-4′), 59.0 ($CH_2$; C-1), 52.8 ($CH_2$, C-3), 45.8 ($CH_3$; NMe), 38.1 (CH; C-5), 36.0 ($CH_2$; $ArCH_2$), 32.3 ($CH_2$), 31.8 ($CH_2$), 28.8 ($CH_2$), 28.0 ($CH_2$), 27.6 ($CH_2$), 26.9 ($CH_2$) and 20.0 ($CH_2$; C-7).

**5-Benzyl-5,6,7,8-tetrahydroisoquinoline**

Potassium (3.52 g, 89.9 mmol) was added in portions to a solution of ammonia (500 $cm^3$) containing $Fe(NO_3)_3 \cdot 9H_2O$ (70 mg) to produce a blue solution. The solution was kept and occasionally swirled for 20 min, by which time the blue colour had dissipated. 5,6,7,8-Tetrahydroisoquinoline (9.2 g, 69.2 mmol) was added dropwise over a period of 10 min and the reaction mixture was kept for a further 20 min, and occasionally swirled to give an orange–red solution. Benzyl chloride (8.67 g, 68.5 mmol) was added over a period of 20 min, with swirling of the mixture, to give an orange solution which was kept overnight to give a green–brown residue. The residue was taken up in water and diethyl ether. The aqueous solution was extracted with diethyl ether then the organic extracts were combined, washed twice with water, then extracted three times with 4 mol $dm^{-3}$ hydrochloric acid. The acidic extracts were washed with diethyl ether, then neutralised (pH paper) with sodium hydrogen carbonate then were basified with 5 mol $dm^{-3}$ aqueous sodium hydroxide before extraction twice with diethyl ether. The organic extracts were washed with water, dried, filtered and concentrated to give an oil which was distilled under reduced pressure. The distillate was redistilled under reduced pressure to give the mono-substituted tetrahydroisoquinoline as a clear oil (7.67 g, 50%); bp 128–130 °C/0.05 mmHg; $R_F$ 0.50 (EtOAc), 0.35 ($CHCl_3$–MeOH, 10:1) (Found: $M^+$, 233.1360. $C_{16}H_{17}N$ requires *M*, 233.1361); $\nu_{max}$(film)/$cm^{-1}$ 2940s, 1593m, 1496m, 1455m, 1415m, 755m and 705s; *m*/*z* 223, 132, 117, 104, 91, 77, 65 and 51; $\delta_H$(90 MHz) 8.30 (1 H, s, 1-H), 8.25 (1 H, d, *J* 5.0, 3-H), 7.16–6.85 (5 H, m, Ar-H), 6.98 (1 H, d, *J* 5.0, 4-H), 3.20–2.80 (2 H, m), 2.80–2.50 (3 H, m) and 2.00–1.80 (4 H, m); $\delta_C$(90 MHz) 150.5 (CH; C-1), 148.9 (C; C-4a), 146.5 (CH; C-3), 139.3 (C; C-1′), 132.7 (C; C-8a), 129.1 (CH; C-2′), 128.4 (CH; C-3′), 126.2 (CH; C-4′), 123.1 (CH; C-4), 42.4 (CH; Ar $CH_2$), 38.8 (CH; C-5), 26.5 ($CH_2$), 26.4 ($CH_2$) and 19.2 ($CH_2$; C-7).

The residue from the distillation solidified and was triturated with diethyl ether. The residue (2.29 g) was crystallised from propan-2-ol to give 5,5-dibenzyl-5,6,7,8-tetrahydroisoquinoline (1.81 g, 5.78 mmol, 8%); mp 117.0–118.5 °C; $R_F$ 0.55 (EtOAc) (Found: C, 88.43; H, 7.40; N, 4.54. $C_{23}H_{23}N$ requires C, 88.14; H, 7.40; N, 4.47%); *m*/*z* 313, 222, 130, 118, 91, 77 and 65; $\nu_{max}$(KBr)/$cm^{-1}$ 3025m, 2935s, 2865m, 1602m, 1592m, 1496s, 1456s, 1412m, 833m, 752s and 700s; $\delta_H$(200 MHz) 8.41 (1 H, d, *J* 5.0, 3-H), 8.22 (1 H, s, 1-H), 7.34 (1 H, d, *J* 5.0, 4-H), 3.20 (2 H, d, *J* 13.5, C*H*′H″Ph), 2.81 (2 H, d, *J* 12.4, CH′*H*″Ph), 2.40 (2 H, t, *J* 6.0, 8-H), 1.65 (2 H, m) and 1.51 (2 H, m); $\delta_C$(200 MHz) 150.6 (CH; C-1), 150.6 (C; C-4a), 146.3 (CH; C-3), 137.6 (C; C-1′), 134.0 (C; C-8a), 130.6 (CH), 127.7 (CH), 126.2 (CH), 122.7 (CH), 47.8 ($CH_2$), 42.4 (C), 30.4 ($CH_2$), 27.2 ($CH_2$) and 18.7 ($CH_2$).

**5-Benzyl-2-methyl-1,2,3,4,5,6,7,8-octahydroisoquinoline 16**

The above tetrahydroisoquinoline (7.67 g, 34.4 mmol) was taken up in anhydrous diethyl ether (125 $cm^3$); the solution was treated with iodomethane (5.05 g, 2.20 $cm^3$, 35.6 mmol) and stirred for 7 days to give the methiodide as a precipitate (9.44 g, 75%); $R_F$ 0.20 ($CHCl_3$–MeOH, 10:1); $\nu_{max}$(KBr)/$cm^{-1}$ 3025, 2930, 1645, 1620, 1495 and 1450; $\delta_H$(90 MHz) 9.20 (1 H, s, 1-H), 8.82 (1 H, d, *J* 4.5, 3-H), 7.65 (1 H, d, *J* 4.5, 4-H), 7.60–6.80 (5 H, m, Ar-H), 4.58 (3 H, s, NMe), 3.40–2.60 (m), 2.20–1.30 (m); $\delta_C$(90 MHz) 160.5 (C; C-4a), 145.0 (CH; C-1), 141.1 (CH; C-3), 138.7 (C), 138.1 (C), 129.2 (CH; Ar), 128.8 (CH; Ar), 127.6 (CH), 127.0 (CH), 48.4 ($CH_3$; $NCH_3$), 41.9 ($CH_2$; C-8), 39.9 (CH; C-5), 26.6 ($CH_2$), 25.4 ($CH_2$) and 17.8 ($CH_2$; C-7).

The methiodide (5.0 g, 13.7 mmol) was taken up in ethanol (100 $cm^3$), and the mixture was cooled in an ice bath and treated with sodium boranuide (1.06 g, 28.0 mmol) in portions while being stirred. The solution was allowed to reach room temp. and was stirred overnight. The solvent was removed under reduced pressure and the residue was taken up in a mixture of diethyl ether and dilute aqueous sodium hydroxide. The phases were separated and the aqueous phase was extracted with diethyl ether. The organic extracts were combined and extracted twice with dilute hydrochloric acid. The acidic extracts were washed with diethyl ether, neutralised (pH paper) with sodium hydrogen carbonate, then basified with aqueous sodium hydroxide and extracted twice with diethyl ether. The organic extracts were washed with water, dried, filtered and concentrated to give an oil which was distilled under reduced pressure to give the octahydroisoquinoline **16** (2.77 g, 11.49 mmol, 84%); bp 124.0–126.0 °C/0.06 mmHg; $R_F$ 0.25 ($CHCl_3$–MeOH, 10:1) (Found:

C, 84.43; H, 9.72; N, 5.78%; $M^+$, 241.1667. $C_{17}H_{23}N$ requires C, 84.59; H, 9.60; N, 5.80%; *M*, 241.1830); *m*/*z* 241, 150, 122, 107, 91, 79, 77 and 65; $\nu_{max}$(film)/$cm^{-1}$ 2950, 2850, 2795, 1610, 1500, 1460 and 1395; $\delta_H$(200 MHz) 7.30–7.09 (5 H, m, Ar-H), 2.82 (1 H, br d, *J* 13.5, 1-H′), 2.66 (1 H, br d, *J* 13.5, 1-H″) and 2.34 (3 H, s, NMe); $\delta_C$(90 MHz) 141.7 (C; C-1′), 129.2 (C; C-4a), 129.1 (CH; C-2′ and -6′), 128.2 (CH; C-3′ and -5′), 127.7 (C; C-8a), 125.7 (CH; C-4′), 59.0 ($CH_2$; C-1), 52.8 ($CH_2$; C-3), 45.8 ($CH_3$; $NCH_3$), 40.3 (CH; C-5), 39.1 ($CH_2$; $PhCH_2$), 29.1 ($CH_2$), 28.1 ($CH_2$), 27.0 ($CH_2$) and 19.3 (CH; C-7).

**(±)-(2aα,4β,10β,10aβ)-1-methyl-1,2,2a,3,4,9,10a,11,12-decahydro-4,10-methanobenzocyclooctenо[7,8-*c*]pyridine 12/19**

The octahydroisoquinoline (2.94 g, 12 mmol) **16** was heated to reflux and stirred in 48% HBr (30.0 $cm^3$) overnight. The solvent was removed under reduced pressure and the residue was taken up in water. The aqueous solution was basified with aqueous potassium hydrogen carbonate and extracted twice with chloroform. The combined extracts were dried, filtered and concentrated to give a residue which was distilled under reduced pressure to give an oil (1.53 g, 53%); bp 170 °/C3 mmHg.

A sample of the distillate (1.00 g, 4.14 mmol) was further purified by column chromatography using as eluent ($CHCl_3$–$Et_3N$; 7:1) to give the cyclised structure **19** as a tan coloured oil (0.32 g); $R_F$ 0.35 ($CHCl_3$–MeOH; 10:1) (Found: $M^+$, 241.3762. $C_{17}H_{23}N$ requires *M*, 241.3777); $\nu_{max}$(film)/$cm^{-1}$ 2920, 2850, 1660 and 1450; $\delta_H$(400 MHz) and $\delta_C$(100 MHz) see Table 1.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