Chemistry of Heterocyclic Compounds, Vol. 29, No. 11, 1993

REACTION BETWEEN PIPERIDINE-4-SPIRO-2′-OXIRANES AND NUCLEOPHILIC REAGENTS

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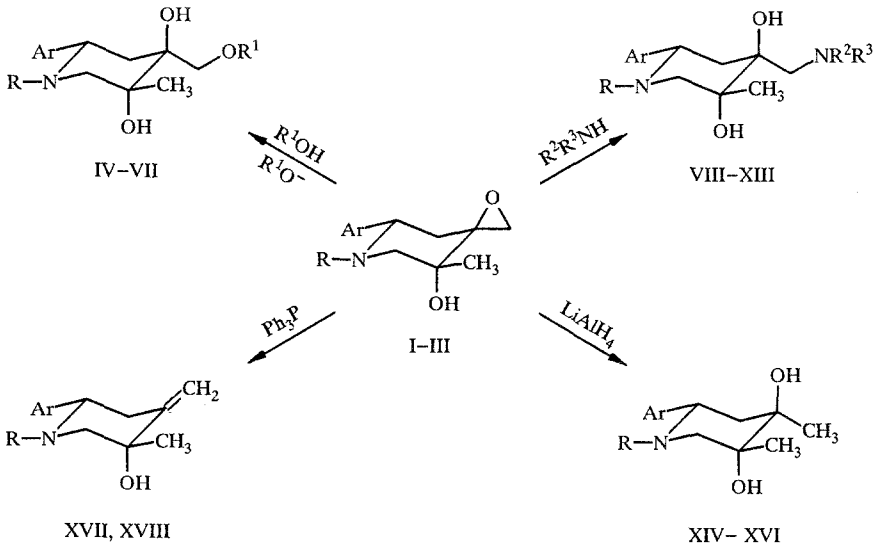
An investigation was made into the opening of the epoxide ring of piperidine-4-spiro-2′-oxiranes by N-, H-, C-, and P-nucleophiles, which yielded the corresponding 3,4-dihydroxy- and 3-hydroxypiperidine derivatives. When these epoxides were reacted with organomagnesium compounds, regrouping occurred, the piperidine ring reducing in size to a pyrrolidine ring and the latter being functionalized by a hydroxymethyl group.

Compounds containing a spirocyclic oxirane moiety are frequently encountered among both natural and synthetic biologically active substances. Prominent among the naturally occurring members of this series are the plant glycosides *Phyllantostatine* and *Phyllantoside*, which possess antitumor activity [1], and the potentially important toxin Jodrellin B [2]. Synthetic spirooxiranes of the steroid series exhibit antiandrogenic and anabolic activity [3], and exert an inhibiting effect on certain enzymes, in particular 3-oxo-Δ^5-steroidisomerase [4]. Adamantyl spirooxirane and some of its epoxide ring derivatives display antiviral activity [5]. All of this has evoked interest in the synthesis of both spirooxiranes and certain of their epoxy ring derivatives.

We have reported the synthesis of 3-hydroxypiperidine-4-spiro-2′-oxiranes (I-III) in a previous communication [6]. In the current work we have set out to study their reaction with nucleophilic reagents.



I, IV, VI, VIII, X, XII, XIV, XVII Ar = Ph, R = $PhCH_2$; II, V, VII, XI, XIII, XV, XVIII Ar = 4-$CH_3OC_6H_4$, R = $PhCH_2$; III, IX, XVI Ar = 4-$CH_3OC_6H_4$, R = CH_3; IV, V R^1 = CH_3; VI, VII R^1 = $(CH_3)_2CH$; VIII R^2 = R^3 = CH_3; IX R^2 = $PhCH_2$, R^3 = H; X, XI R^2, R^3 = $CH_2CH_2OCH_2CH_2$; XII, XIII R^2 = $CH_3(CH_2)_{11}$, R^3 = H

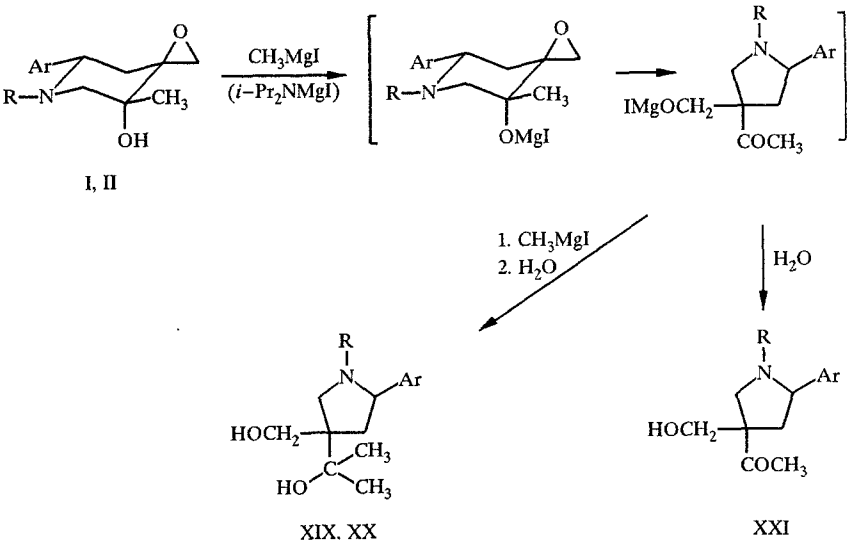
Belorussian Technological Institute, Minsk. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 11, pp. 1499-1503, November, 1993. Original article submitted August 19, 1993.

0009-3122/93/2911-1291$12.50

When epoxides I-III were reacted with base-catalyzed methyl or isopropyl alcohol, the products (IV-VII) obtained arose from the nucleophilic opening of the epoxide ring by an alkoxide ion. In every case the nucleophilic attack was directed towards the least spatially hindered carbon atom of the oxirane ring. The same regiospecificity was observed in the case of other nucleophiles. Thus, the reaction with primary and secondary amines afforded 3a,4a-dihydroxy-4e-alkyl(dialkyl)aminomethylpiperidines (VIII-XIII), while 3a,4a-dihydroxy-3e,4e-dimethylpiperidines (XIV-XVI) resulted from reduction with lithium aluminohydride. Members of the latter class of compound had previously been obtained by reacting the corresponding 3a-hydroxypiperidin-4-ones with organomagnesium compounds [7], but they only formed as the subsidiary products under those conditions. So, for instance, diol XVI was afforded in 19% yield, while its isomer with the opposite configuration at $C_{(4)}$ had a yield of 79%. By using oxiranes I-III, however, diols XIV-XVI can be obtained in high yields and as the sole products.

Piperidine-4-spiro-2′-oxiranes reacted with triphenylphosphine to produce 4-methylenepiperidines (XVII, XVIII). In essence the two-stage process comprising the methylenation of piperidin-4-ones by diazomethane and the deoxidation of epoxides by triphenylphosphine is equivalent to the Wittig reaction. However, the direct replacement of the carbonyl oxygen by a methylene group in the presence of triphenylmethylenephosphorane cannot be employed because of the need to use strong bases for generating the latter. At the same time, under strong base conditions 3-hydroxypiperidin-4-ones are known to undergo regrouping involving a reduction in size of the piperidine ring [8].

The structure of the synthesized compounds was corroborated by spectral data. For example, the PMR spectrum of compounds IV-XVIII exhibited signals for the protons of the methyl group at $C_{(3)}$, the aromatic ring, the hydroxyl groups, the R substituent at the nitrogen atom and the piperidine ring, as well as the $C_{(4)}$ methylene group protons. The protons at $C_{(5)}$ and $C_{(6)}$ formed an ABX spin system with coupling constants of $J_{H_5^e H_5^a} = 13.4\text{-}15.0$, $J_{H_5^e H_6^a} = 3.4\text{-}4.0$, and $J_{H_5^a H_6^a} = 10.9\text{-}12.3$ Hz. Among the absorption bands observed in the IR spectra of compounds IV-XVIII were those corresponding to hydroxyl groups at 3420-3460 cm^{-1}, C–H bonds at 2800-3060 cm^{-1}, and an aromatic ring at 1490 cm^{-1}. The exocyclic double bond absorption band in compounds XIX and XX appeared at 1650 cm^{-1}.



I, II

The reaction between spiro[3-hydroxypiperidine-4,2′-oxirane] compounds I and II and methyl magnesium iodide produced an unexpected result. Instead of 3,4-dihydroxy-3-methyl-4-ethylpiperidines the only reaction products proved to be 3-hydroxymethyl-3-(2-hydroxy-2-propyl)-pyrrolidines XIX and XX. It would appear that the mechanism by which they form involves deprotonation of the hydroxyl group, migration of the σ-bond from $C_{(3)}$ to $C_{(4)}$ (which causes the ring to reduce in size) and addition of a second methyl magnesium iodide molecule to the intermediate hydroxymethylacetylpyrrolidine.

This mechanism was corroborated by the isolation of small amounts of ketone XXI (in 13% yield) from the complex mixture that formed when diisopropylamino magnesium iodide acted on compound I. The base-catalyzed regroupings of tertiary alcohols containing a nucleophilic group in the α-position have been referred to in a previous communication [9], but there was no mention in the literature of the fact that the epoxide ring oxygen atom may play the part of the nucleophilic group in this process.

In the PMR spectra of pyrrolidine derivatives XIX-XXI singlet signals for the hydroxymethyl group protons appeared at 3.72 (XIX, XX) and 3.80 ppm (XXI). The acetyl group proton signal in compound XXI was observed at 2.16 ppm and the

isopropyl group proton signal in compounds XIX and XX at 1.19-1.21 ppm. Pyrrolidine ring proton signals formed an AMX spin system with coupling constants of $J_{H4^eH4^a}$ = 13.2-13.6, $J_{H4^eH5^a}$ = 8.8-9.4, and $J_{H4^aH5^a}$ = 7.4-7.8 Hz, which was in good agreement with literature data for compounds of similar structure [8].

EXPERIMENTAL

The PMR spectra of compounds IV-XXI were recorded in $CDCl_3$ on a Bruker WM-360 (360 MHz) or Tesla BS-567A (100 MHz) instrument. IR spectra were taken in KBr tablets using a Specord IR-75. Reactions were monitored using TLC on Silufol plates.

Elemental analysis data on C, H, and N for compounds IV-XXI was in line with calculated values.

The 1-alkyl-6-aryl-3a-hydroxy-3e-methylpiperidine-4-spiro-2′-oxiranes I-III used initially were obtained using the technique described in a previous work [6].

4e-Alkoxymethyl-6-aryl-1-benzyl-3a,4a-dihydroxy-3e-methyl-piperidines (IV-VII). After 0.0025 moles of oxirane I or II had been dissolved in 15 ml of methanol or propanol-2, 0.0025 moles of the corresponding alcoholate was added to it. The mixture was boiled using a reflux condenser for 3-4 h, the reaction course being monitored by means of TLC. After cooling, the mixture was evaporated, then the residue was treated with water (10 ml) and extracted with methylene chloride (2 × 20 ml). The extract was dried with sodium sulfate and, after evaporation, the residue was crystallized from hexane to yield compounds IV-VII.

IV:. mp 139-141°C. PMR spectrum, δ, ppm, coupling constant (J), Hz: 1.09 (3H, s, −); 1.80 (1H, d.d, J = 14.4 and 3.6); 1.92 (1H, d.d, J = 14.4 and 11.8); 2.45 (1H, d, J = 12.0); 2.60 (1H, d, J = 12.0); 2.94 (1H, d, J = 13.7); 3.24 (1H, d, J = 9.1); 3.32 (3H, s, −); 3.48 (1H, s, −); 3.59 (1H, d.d, J = 11.8 and 3.6); 3.64 (1H, d, J = 9.1); 3.75 (1H, d, J = 13.7); 7.23 (10H, m, −). Yield 85%.

V: mp 134-135°C. PMR spectrum: 1.10 (3H, s, −); 1.74 (1H, d.d, J = 14.5 and 4.2); 1.96 (1H, d.d, J = 14.5 and 10.6); 2.44 (1H, d, J = 11.5); 2.61 (1H, d, J = 11.5); 2.64 (1H, s, −); 2.92 (1H, d, J = 13.8); 3.24 (1H, d, J = 9.4); 3.34 (3H, s, −); 3.47 (1H, s, −); 3.55 (1H, d.d, J = 11.8 and 3.6); 3.64 (1H, d, J = 9.1); 3.87 (1H, d, J = 13.8 Hz); 3.89 (3H, s, −); 6.89 (2H, d, J = 8.5); 7.23 (5H, s, −); 7.34 (2H, d, J = 8.5). Yield 92%.

VI: mp 91-92°C. PMR spectrum: 1.10 (3H, s, −); 1.10 (3H, d, J = 6.0); 1.13 (3H, d, J = 6.0); 1.82 (1H, d.d, J = 14.4 and 3.9); 1.90 (1H, d.d, J = 14.4 and 11.6); 2.48 (1H, d, J = 11.6); 2.64 (1H, d, J = 11.6); 2.81 (1H, s, −); 2.97 (1H, d, J = 14.3); 3.26 (1H, d, J = 8.7); 3.56 (1H, septet, J = 6.0); 3.63 (1H, d.d, J = 11.6 and 3.9); 3.67 (1H, d, J = 9.1); 3.78 (1H, d, J = 13.7); 7.26 (10H, m, −). Yield 90%.

VII: mp 87-88°C. PMR spectrum: 1.03 (3H, s, −); 1.03 (3H, d, J = 6.0); 1.08 (3H, d, J = 6.0); 1.71 (1H, d.d, J = 14.1 and 3.2); 1.82 (1H, d.d, J = 14.1 and 11.7); 2.41 (1H, d, J = 11.2); 2.58 (1H, d, J = 11.2); 2.73 (1H, s, −); 2.88 (1H, d, J = 13.4); 3.22 (1H, d, J = 9.2); 3.44 (1H, septet, J = 6.0); 3.58 (1H, d.d, J = 11.6 and 3.9); 3.68 (1H, d, J = 8.7); 3.71 (1H, d, J = 13.4); 6.90 (2H, d, J = 8.8); 7.22 (5H, s, −); 7.36 (2H, d, J = 8.8). Yield 70%.

1-Alkyl-4e-alkyl(dialkyl)aminomethyl-6-aryl-3a,4a-dihydroxy-3e-methylpiperidines (VIII-XIII). A. A 0.0025 mole sample of oxirane I was dissolved in 10 ml of 1,4-dioxane, then 1 ml of a 33% aqueous dimethylamine solution was added and the mixture was kept at room temperature for 24 h. After the dioxane had been evaporated off, the residue was dissolved in a 2:1 ether–hexane mixture, and filtered through an aluminum oxide layer (1 cm), washing with 50 ml of the same mixture. The filtrate was evaporated and compound VIII was crystallized from the residue. mp 79-80°C. PMR spectrum: 1.04 (3H, s, −); 1.77 (1H, d.d, J = 14.4 and 3.6); 1.92 (1H, d.d, J = 14.4 and 11.9); 2.22 (1H, d, J = 14.2); 2.33 (6H, s, −); 2.60 (1H, d, J = 11.6); 2.78 (1H, d, J = 14.2); 2.87 (1H, d, J = 13.8); 3.35 (1H, s, −); 3.62 (1H, d.d, J = 11.9 and 3.6); 3.78 (1H, d, J = 13.8); 7.25 (10H, m, −). Yield 94%.

B. A 0.0025 mole sample of oxirane I-III was dissolved in 10 ml of propanol-2, to which was added 0.003 moles of the appropriate amine. The reaction mixture was then boiled for 3-4 h using a reflux condenser fitted with NaOH tube attachment. The course of the reaction was monitored using TLC. After cooling and evaporation, the residue was crystallized from hexane to yield compounds IX-XIII.

IX: mp 96-97°C. PMR spectrum: 1.09 (3H, s, −); 1.50 (1H, d.d, J = 13.3 and 3.6); 1.83 (1H, d.d, J = 13.3 and 11.3); 1.98 (3H, s, −); 2.46 (1H, d, J = 10.9); 2.70 (1H, d, J = 10.9); 2.88 (1H, d, J = 13.8); 3.20 (1H, d.d, J = 11.3 and 3.6); 3.75 (3H, s, −); 6.81 (2H, d, J = 8.1); 7.22 (5H, s, −); 7.25 (2H, d, J = 8.1). Yield 85%.

X: mp 121-122°C. PMR spectrum: 1.03 (3H, s, −); 1.68 (1H, d.d, J = 13.4 and 4.0); 1.98 (1H, d.d, J = 13.4 and 10.9); 2.30 (1H, d, J = 14.3); 2.53 (8H, m, −); 2.72 (1H, d, J = 14.3); 2.92 (1H, d, J = 13.4); 3.32 (3H, s, −); 3.57 (1H, d.d, J = 10.9 and 4.0); 3.60 (1H, d, J = 4.3); 3.65 (1H, d, J = 4.3); 3.78 (1H, d, J = 13.4); 7.25 (10H, m, −). Yield 90%.

XI: mp 170-171°C. PMR spectrum: 1.02 (3H, s, −); 1.67 (1H, d.d, J = 14.3 and 4.0); 1.97 (1H, d.d, J = 14.3 and 11.8); 2.31 (1H, d, J = 14.0); 2.55 (8H, m, −); 2.74 (1H, d, J = 14.0); 2.92 (1H, d, J = 13.4); 3.31 (1H, s, −); 3.53 (1H, d.d, J = 11.8 and 4.0); 3.61 (1H, d, J = 4.6); 3.66 (1H, d, J = 4.6); 3.78 (3H, s, −); 3.78 (1H, d, J = 13.4); 6.87 (2H, d, J = 9.3); 7.23 (5H, s, −); 7.33 (2H, d, J = 8.5). Yield 95%.

XII: Oil. PMR spectrum: 0.87 (3H, t, J = 6.5); 1.09 (3H, s, −); 1.23 (22H, m, −); 1.60 (1H, d.d, J = 14.4 and 3.8); 1.97 (1H, d.d, J = 14.4 and 11.8); 2.50 (1H, d, J = 13.2); 2.55 (1H, d, J = 12.9); 2.60 (1H, d, J = 12.9); 2.91 (1H, d, J = 13.2); 2.98 (1H, d, J = 13.6); 3.63 (1H, d.d, J = 12.0 and 3.8); 3.78 (1H, d, J = 13.6); 7.27 (10H, m, −). Yield 94%.

XIII: Oil. PMR spectrum: 1.10 (3H, s, −); 0.87 (3H, t, J = 6.5); 1.24 (22H, m, −); 1.58 (1H, d.d, J = 14.6 and 3.9); 1.92 (1H, d.d, J = 14.6 and 12.0); 2.46 (1H, d, J = 12.6); 2.48 (1H, d, J = 12.9); 2.57 (1H, d, J = 12.6); 2.85 (1H, d, J = 12.9); 2.92 (1H, d, J = 13.3); 3.55 (1H, d.d, J = 12.0 and 3.9); 3.74 (1H, d, J = 13.3); 3.77 (3H, s, −); 6.87 (2H, d, J = 6.5); 7.20 (5H, s, −); 7.35 (2H, d, J = 6.5). Yield 97%.

6-Aryl-1-benzyl-3a,4a-dihydroxy-3e,4e-dimethylpiperidines (XIV-XVI). A 20 ml sample of absolute ether and 0.002 moles of lithium aluminum hydride were placed in a round bottomed flask fitted with a reflux condenser equipped with calcium chloride tube and dropping funnel. Then a solution of 0.0025 moles of oxirane I-III in 30 ml of absolute ether was added dropwise with stirring. When all the solution had been added, the mixture was stirred for a further 20 min, then 30 ml of water was added dropwise with great care. The ethereal layer was separated off and the aqueous phase was extracted with ether (3 × 20 ml). Then the extract was dried with sodium sulfate and evaporated, and the residue was crystallized from hexane to give products XIV-XVI.

XIV: mp 105-106°C. PMR spectrum: 1.00 (3H, s, −); 1.15 (3H, s, −); 1.55 (1H, d.d, J = 14.2 and 3.6); 2.04 (1H, d.d, J = 14.2 and 11.6); 2.45 (1H, d, J = 11.6); 2.56 (1H, d, J = 11.6); 2.92 (1H, d, J = 14.0); 3.55 (1H, d.d, J = 11.6 and 3.6); 3.73 (1H, d, J = 14.0); 7.26 (10H, m, −). Yield 83%.

XV: mp 109-110°C. PMR spectrum: 1.18 (3H, s, −); 1.24 (3H, s, −); 1.53 (1H, d.d, J = 13.8 and 3.2); 2.00 (1H, d.d, J = 13.8 and 11.8); 2.00 (3H, s, −); 2.47 (1H, d, J = 10.9); 2.64 (1H, d, J = 10.9); 3.19 (1H, d.d, J = 11.8 and 3.2); 3.65 (1H, s, −); 3.79 (3H, s, −); 6.85 (2H, d, 8.3); 7.34 (2H, d, J = 8.5). Yield 76%.

XVI: mp 101-102°C. PMR spectrum: 1.08 (3H, s, −); 1.23 (3H, s, −); 1.29 (1H, s, −); 1.62 (1H, d.d, J = 14.0 and 3.1); 2.10 (1H, d.d, J = 14.0 and 11.7); 2.50 (1H, d, J = 11.4); 2.59 (1H, d, J = 11.4); 2.94 (1H, d, J = 13.1); 3.46 (1H, s, −); 3.54 (1H, d.d, J = 11.7 and 3.1); 3.76 (1H, d, J = 13.1); 3.80 (3H, s, −); 6.91 (2H, d, J = 8.6); 7.22 (5H, s, −); 7.34 (2H, d, J = 8.5). Yield 91%.

6-Aryl-1-benzyl-3a-hydroxy-3e-methyl-4-methylenepiperidines (XVII, XVIII). A 0.0025 mole sample of oxirane I or II was dissolved in 10 ml of o-xylene, to which was added 0.005 moles of triphenylphosphine and 0.001 mole of hydroquinone. After the mixture had been boiled for 5 h using a reflux condenser, it was applied to a column packed with silica gel L40/100 (2 × 25 cm) and eluted with a 1:1 methylene chloride−hexane mixture to yield compounds XVII, XVIII.

XVII: mp 88-89°C. PMR spectrum: 1.32 (3H, s, −); 2.09 (1H, d, J = 11.6); 2.39 (1H, d.d, J = 15.0 and 3.4); 2.84 (1H, d.d, J = 15.0 and 11.4); 2.95 (1H, d, J = 11.6); 3.01 (1H, d, J = 13.8); 3.29 (1H, d.d, J = 11.4 and 3.4); 3.85 (1H, s, −); 3.88 (1H, d, J = 13.8); 4.76 (1H, s, −); 4.90 (1H, s, −); 7.26 (10H, m, −). Yield 71%.

XVIII: 122-123°C. PMR spectrum: 1.28 (3H, s, −); 2.00 (1H, s, J = 11.0); 2.33 (1H, d.d, J = 13.2 and 3.1); 2.77 (1H, d.d, J = 13.2 and 11.0); 2.85 (1H, d, J = 11.0); 2.94 (1H, d, J = 13.5); 3.20 (1H, d.d, J = 11.0 and 3.1); 3.67 (1H, s, −); 3.77 (3H, s, −); 3.78 (1H, d, J = 13.5); 4.74 (1H, s, −); 4.87 (1H, s, −); 6.92 (2H, d, J = 8.2); 7.23 (5H, s, −); 7.37 (2H, d, J = 8.2). Yield 70%.

5-Aryl-1-benzyl-3-hydroxymethyl-3-(2-hydroxy-2-propyl)piperidines (XIX, XX). A 0.01 mole sample of methyl iodine in 20 ml of absolute ether was added dropwise over 1 h to 0.24 g of magnesium ribbon in 40 ml of absolute ether with gentle heating. After the magnesium had dissolved, the mixture was stirred for a further 5 min, then a solution of 0.0025 moles of epoxide I or II in 30 ml of absolute ether was added dropwise over 10 min. After a further 20 min stirring, the mixture was carefully treated with water and the ethereal layer was separated off. Then the aqueous phase was extracted with ether (2 × 20 ml), the extract was dried with sodium sulfate and evaporated to yield compounds XIX or XX.

XIX: Oil. PMR spectrum: 1.21 (6H, s, −); 1.73 (1H, d.d, J = 13.2 and 8.8); 2.14 (1H, s, −); 2.42 (1H, d.d, J = 13.2 and 7.4); 2.49 (1H, d, J = 9.1); 2.92 (1H, d, J = 12.9); 2.93 (1H, d, J = 9.1); 3.43 (1H, d.d, J = 8.8 and 7.4); 3.72 (2H, s, −); 3.83 (1H, d, J = 12.9); 7.27 (10H, m, −). Yield 76%.

XX: Oil. PMR spectrum: 1.19 (6H, s, −); 1.72 (1H, d.d, J = 13.6 and 9.4); 2.18 (1H, s, −); 2.39 (1H, d.d, J = 13.6 and 7.5); 2.46 (1H, d, J = 19.4); 2.91 (1H, d, J = 13.3); 2.91 (1H, d, J = 9.4); 3.37 (1H, d.d, J = 9.4 and 7.5); 3.72 (2H, s, −); 3.81 (3H, s, −); 3.82 (1H, d, J = 13.3); 6.91 (2H, d, J = 9.2); 7.24 (5H, s, −); 7.39 (2H, d, J = 9.2). Yield 72%.

3-Acetyl-1-benzyl-3-benzyl-3-hydroxymethyl-5-phenylpiperidine (XXI). A 0.8 ml sample of ethyl iodide in 20 ml of absolute ether was added dropwise to 0.24 g of magnesium ribbon. When the magnesium had dissolved, 0.01 moles of diisopropylamine was added, the mixture was stirred for 10 min, then a solution of 0.77 g (0.0025) of epoxide I was added dropwise to it. Thirty minutes later the mixture was carefully treated with water and after the organic phase had been separated off, the water layer was extracted with ether (2 × 30 ml). The combined extracts were dried with sodium sulfate and evaporated, the residue being chromatographed on silica gel L40/100 and eluted with a 1:2 ether−hexane mixture to give compound XXI. Oil. PMR spectrum: 1.78 (1H, d.d, J = 13.2 and 8.8); 2.16 (3H, s, −); 2.52 (1H, d.d, J = 13.6 and 7.8); 2.54 (1H, d, J = 9.7); 2.98 (1H, d, J = 12.9); 3.09 (1H, d, J = 9.7); 3.12 (1H, s, −); 3.49 (1H, d.d, J = 9.4 and 7.8); 3.80 (2H, s, −); 3.83 (1H, d, J = 12.9); 7.25 (10H, m, −). Yield 13%.

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