A route to dihydro[2]benzooxepino[4,5-*c*]pyridines and dihydrothieno[*d*][2]benzooxepines *via* the 1.7-electrocyclisation of carbonyl ylides

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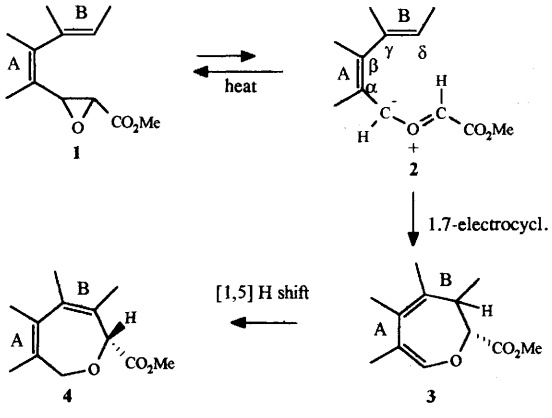
The cyclisation of diene-conjugated carbonyl ylides of the general type 2, in which the α,β; γ,δ diene function is formed by a benzene ring and either a thiophene or pyridine ring provides a new route to some hetero-fused dihydrobenzooxepines. The oxirane precursors for the carbonyl ylides were synthesised in a two-step scheme from readily available reactants.

Introduction

For many years 1,3-dipoles have been used extensively in the construction of five-membered heterocyclic rings *via* their cycloadditions with suitable dipolarophiles,[1] and by the 1.5-electrocyclisation reactions of α,β-unsaturated 1,3-dipoles.[2] More recently the 1.7-electrocyclisation of α,β; γ,δ-unsaturated 1,3-dipoles has been developed as a versatile route to seven membered heterocycles.[3] Such reactions have now been studied for a number of 1,3-dipoles and have been shown to provide effective routes to biologically important systems such as azepines, diazepines, oxepines and their benzo- and heterocyclo-fused derivatives.[3] In earlier work we have studied the 1.7-cyclisations of diazo-compounds[4] and nitrile ylides[5] and more recently have investigated, *via* the use of intramolecular competition reactions, the factors controlling the reactivity of nitrile ylides in these reactions.[6] The work described in this paper is concerned with diene-conjugated carbonyl ylides **2** (Scheme 1) and was carried out in preparation for a similar reactivity study[7] on 1,3-dipoles of this type.



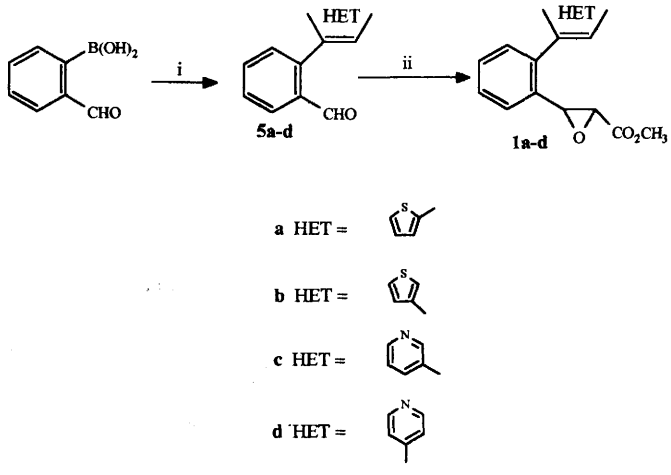
Scheme 1

Eberbach and his group have carried out extensive and elegant work on the cyclisation of diene-conjugated carbonyl ylides **2** with various types of unsaturated systems at A and B (Scheme 1).[3] In the system containing two benzene rings at A and B, the product isolated was the stable dihydrodibenzo-[*c*,*e*]oxepine, **4** (A,B = benzo).[8] In terms of our intended reactivity study we were interested in investigating the effects of substituents in the benzene ring under attack and also in determining the relative reactivity of heterocyclic rings at the B position. However systems of type **2** with one benzene ring and one heterocyclic ring at A and B had not previously been studied from a synthetic viewpoint and this work was therefore carried out in order to develop an effective route to the required oxiranes **1** and to study their decomposition *via* flash vacuum pyrolysis. In addition to preparing the way for the subsequent work on intramolecular competition reactions of carbonyl ylides it was hoped that this would result in a useful general route to heterobenzooxepines, some of which have been shown to have activity as non-steroidal anti-inflammatory agents,[9a–e] anti-ulcer agents[9f] and as thromboxane A_2 antagonists.[9g] A similar investigation into analogous nitrile ylides has recently shown that they cyclise readily onto both electron-rich and electron-poor heterocyclic rings to provide an easy and efficient route to furo-, pyrido- and thieno-[*d*][2]benzoazepines.[5]

Results and discussion

Synthesis of the oxiranes 1a–e

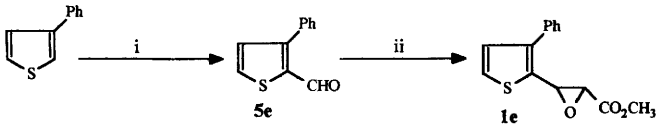
These were prepared in two steps *via* the route shown in Scheme 2. The first step utilised a Suzuki[10] type cross-coupling



Scheme 2 *Reagents:* i, HETBr, Pd^0; ii, $ClCH_2CO_2Me$, NaOMe, THF

reaction, using the improved Gronowitz[11] conditions, of *o*-formylphenylboronic acid with the appropriate bromo-heteroaromatic compound to yield the aldehydes **5a–d** in high yields. This is a convenient reaction as *o*-formylphenylboronic acid is commercially available or can be synthesised readily from protected *o*-bromobenzaldehyde.[12] 2-Formyl-3-phenylthiophene **5e** (Scheme 3) was synthesised by the Vilsmeier formylation of 3-phenylthiophene with *N*,*N*-dimethylformamide and phosphoryl chloride.[13] The oxiranes **1a–e** were then

obtained by the Darzens condensation of the aldehydes **5a–e** with methyl chloroacetate, using sodium methoxide as base in THF. Compounds **1a**, **b** and **d** were purified by Kugelrohr distillation but **1c** and **1e** polymerised on attempted distillation and, instead, were purified by column chromatography on activated alumina. Their ^{1}H and ^{13}C NMR spectra were consistent with the structures expected, the former showing a characteristic doublet of doublets for the methine protons of the oxirane ring with a typical J value of 1.9 ± 0.1 Hz indicating *trans* stereochemistry. In no case was any of the *cis* isomer detectable by 200 MHz NMR spectroscopy.



Scheme 3 *Reagents:* i, DMF, $POCl_3$; ii, $ClCH_2CO_2CH_3$, NaOMe, THF

Pyrolysis of the oxiranes 1a–e

Previous work by Eberbach has shown that diene-conjugated carbonyl ylides **2** can be generated by the thermal conrotatory ring opening of oxiranes[3,8] (Scheme 1). These species then undergo a formally conrotatory 1.7-electrocyclisation to give the intermediates **3** which then rearrange by an *in situ* [1,5] sigmatropic hydrogen shift to give the isolated products **4**. In cases where A and B are aromatic rings this final step restores their aromatic character. In this work we have subjected the oxiranes **1a–e** to flash vacuum pyrolysis[14] (FVP) at 625 °C at a pressure of 8 × 10^{-4} mmHg. This technique resulted in the successful pyrolysis of **1a**, **b** and **d** but not of the oxiranes **1c** and **1e** which polymerised to give a black tar on attempted distillation into the reaction furnace of the FVP apparatus. The three successful pyrolyses followed the expected reaction paths (Scheme 4) to give the new heterocyclic systems **4a**, **b** and **d** in moderate yields. Attempts to carry out liquid-phase pyrolysis of **1c** and **1e** in solutions of decalin and diphenylether, at reflux temperature for 24 h, gave rise only to the recovery of most of the starting material and no identifiable products.

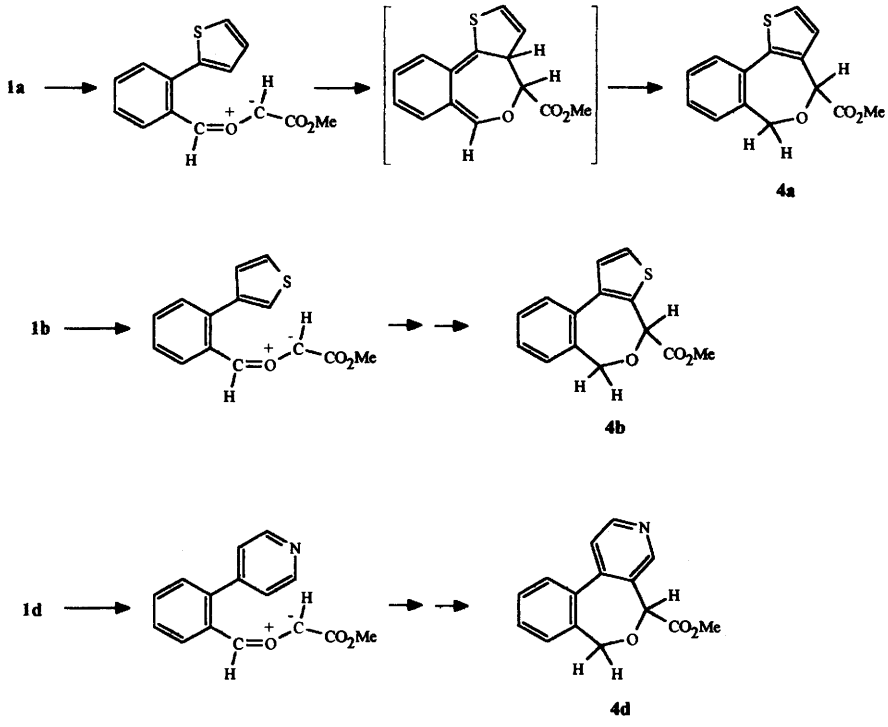
The hetero-fused dihydrobenzooxepines **4a**, **b** and **d** were identified by comparison of their ^{1}H and ^{13}C NMR spectra with those of the known dihydrodibenzo[*c*,*e*]oxepines **4** (A and B = benzo).[8] The ^{1}H NMR spectra showed a characteristic singlet for the methine proton and a doublet of doublets for the methylene protons of the oxepine ring, with corresponding signals in the ^{13}C spectra.

Conclusions

This work has shown that the 1.7-electrocyclisation of carbonyl ylides is a viable route to hetero-fused dihydrobenzooxepines irrespective of whether the heterocyclic ring under attack is electron rich or electron poor. This parallels earlier observations on the cyclisation of analogous nitrile ylides and again highlights the value of electrocyclic aromatic substitution in the formation of benzo- or hetero-fused heterocyclic systems. This contrasts with intramolecular electrophilic aromatic substitution which works well only for electron-rich rings. The utility of this route to heterobenzooxepines is enhanced by the easy synthesis of their oxiranes precursors *via* Pd^0 catalysed cross-coupling chemistry from readily available starting materials. However it is also clear that the generality of the method is limited by possible difficulties in the generation of these carbonyl ylide intermediates by FVP as some of their oxirane precursors polymerise in the inlet of the pyrolysis apparatus.

Experimental

NMR spectra were run as solutions in deuteriochloroform. Chemical shifts are recorded as δ values and J values are given in Hz. In the ^{13}C spectra, carbon multiplicity was established by DEPT. Mass spectra were obtained using electron ionisation at 70 eV. Preparative chromatography was carried out by the 'dry-column flash' technique[15] using silica gel (15 μm, Fluka Kieselgel GF254) and eluting solvents based on hexane admixed with ether. Ether refers to diethyl ether. Evaporation of solvents indicates evaporation of a mixture under reduced pressure using a rotary evaporator. All drying of solutions was done with anhydrous magnesium sulfate.



Scheme 4

Tetrahydrofuran (THF) was distilled under nitrogen from sodium diphenylketyl immediately before use. 1,2-Dimethoxyethane (DME) was passed through a column of activated alumina immediately before use. *o*-Formylphenylboronic acid[12] was obtained from Aldrich Chemical Company. 3-Phenylthiophene-2-carbaldehyde **5e** was prepared by a known route.[13]

Synthesis of the 2-substituted benzaldehydes 5a–d

2-(2-Thienyl)benzaldehyde 5a. 2-Bromothiophene (0.6 g, 3.68 mmol) and tetrakis(triphenylphosphine)palladium(0) (0.11 g, 0.095 mmol) were added to DME (20 cm^3) with stirring under dry nitrogen. After 20 min a solution of sodium carbonate (0.35 g, 3.3 mmol) in water (5 cm^3) and *o*-formylphenylboronic acid (0.5 g, 3.3 mmol) were added to the mixture which was then heated under reflux for 12 h. The DME was evaporated and the mixture extracted with dichloromethane. The organic layer was dried, passed through a bed of activated alumina and then evaporated. Distillation of the residue gave **5a** as a yellow oil (0.56 g, 90%), bp 170 °C/0.5 mmHg (Found: M^+, 188.029 04. $C_{11}H_8OS$ requires *M*, 188.029 59); δ_H(200 MHz) 7.0–7.15 (2 H, m, Ar-H), 7.4–7.6 (4 H, m, Ar-H), 7.95–8.0 (1 H, m, Ar-H) and 10.1 (1 H, s, CHO); δ_C(50 MHz) 191.7 (CHO), 138.4 (C-1, phenyl), 137.9 (C-2, thiophene), 133.8 (C-2, phenyl), 133.2, 131.0, 129.3, 127.9, 127.5 and 127.1 (remaining aromatic CH signals, overlap of one CH signal); ν_{max}(Nujol)/cm^{-1} 1693 (C=O). The following compounds were prepared by the same method using the appropriate heterocyclic bromide.

2-(3-Thienyl)benzaldehyde 5b. (86%, 12 h) Yellow oil, bp 160 °C/0.5 mmHg (Found: M^+, 188.030 90. $C_{11}H_8OS$ requires *M*, 188.029 59); δ_H(200 MHz) 7.15–7.6 (6 H, m, Ar-H), 7.95–8.0 (1 H, m, Ar-H) and 10.1 (1 H, s, CHO); δ_C(50 MHz) 192.0 (CHO), 140.0 (C-1, phenyl), 137.9 (C-3, thiophene), 133.6 (C-2, phenyl), 133.3, 130.2, 129.0, 127.4, 127.2, 126.0 and 124.7 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1690 (C=O).

2-(3-Pyridyl)benzaldehyde 5c. (65%, 24 h) White solid, mp 67–68 °C from hexane–ether (95:5) (Found: C, 78.6; H, 5.0; N, 7.4%; M^+, 183.066 76. $C_{12}H_9NO$ requires C, 78.7; H, 4.9; N, 7.65%; *M*, 183.068 41); δ_H(200 MHz) 7.3–7.7 (5 H, m, Ar-H), 8.0 (1 H, dd, *J* 7.5 and 1.0, Ar-H), 8.6–8.7 (2 H, m, Ar-H) and 9.9 (1 H, s, CHO); δ_C(50 MHz) 191.0 (CHO), 149.8 (C-2, pyridyl), 149.1 (C-4, pyridyl), 141.5 (C-1, phenyl), 133.5 (C-2, phenyl), 133.4 (C-1, pyridyl), 137.0, 133.7, 130.8, 128.4, 128.3 and 122.9 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1695 (C=O).

2-(4-Pyridyl)benzaldehyde 5d. (87%, 12 h) White solid, mp 66–67 °C from hexane–ether (95:5) (Found: C, 78.5; H, 4.7; N, 7.5%; M^+, 183.068 06. $C_{12}H_9NO$ requires C, 78.7; H, 4.9; N, 7.65%; *M*, 183.068 41); δ_H(200 MHz) 7.2–7.3 (2 H, m, Ar-H), 7.36 (1 H, dd, *J* 7.5 and 1.3, Ar-H), 7.4–7.7 (2 H, m, Ar-H), 8.0 (1 H, dd, Ar-H), 8.65–8.7 (2 H, m, Ar-H) and 9.9 (1 H, s, CHO); δ_C(50 MHz) 190.9 (CHO), 149.5 (C-3, 5, pyridyl), 145.6 (C-1, phenyl), 142.4 (C-1, pyridyl), 133.1 (C-2, phenyl), 133.7, 130.1, 128.8, 128.1 and 124.5 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1697 (C=O).

Synthesis of oxiranes 1a–e

Methyl 2,3-epoxy-3-(2-thienyl-2-phenyl)propanoate 1a. Sodium methoxide (0.3 g, 5.55 mmol) was added to a solution of 2-(2-thienyl)benzaldehyde **5a** (0.35 g, 1.86 mmol) and methyl chloroacetate (0.66 g, 6.0 mmol) in THF (20 cm^3) and heated under reflux under dry nitrogen for 5 h. After evaporation of the mixture, the residue was dissolved in dichloromethane, and the mixture was washed with water, dried and passed through a bed of activated alumina. Evaporation of the solvent and distillation of the residue gave **1a** (0.38 g, 79%) as a colourless oil, bp 210–215 °C/0.5 mmHg (Found: M^+, 260.052 33. $C_{14}H_{12}O_3S$ requires *M*, 260.050 72); δ_H(200 MHz) 3.5 (1 H, d, *J* 2.0, 2-H), 3.8 (3 H, s, OCH_3), 4.25 (1 H, d, 3-H), 7.0–7.1 (2 H, m, Ar-H) and 7.3–7.5 (5 H, m, Ar-H); δ_C(50 MHz) 168.2 (C=O), 140.4 (C-2, thiophene), 133.9 (C-1, phenyl), 132.6 (C-2, phenyl), 56.7 (C-3), 56.6 (C-2), 52.4 (OCH_3), 130.1, 128.4, 128.1, 127.3, 126.9, 126.1 and 124.5 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1747 (C=O). The following compounds were prepared by the same method.

Methyl 2,3-epoxy-3-(3-thienyl-2-phenyl)propanoate 1b. (76%, 5 h) Colourless oil, bp 220–225 °C/0.5 mmHg (Found: M^+, 260.049 57. $C_{14}H_{12}O_3S$ requires *M*, 260.050 72); δ_H(200 MHz) 3.52 (1 H, d, *J* 1.9, 2-H), 3.8 (3 H, s, OCH_3), 4.1 (1 H, d, 3-H), 7.1–7.25 (2 H, m, Ar-H) and 7.3–7.4 (5 H, m, Ar-H); δ_C(50 MHz) 168.2 (C=O), 139.6 (C-3, thiophene), 136.2 (C-1, phenyl), 132.3 (C-2, phenyl), 56.5 (C-3), 56.3 (C-2), 52.4 (OCH_3), 129.4, 129.0, 128.3, 127.6, 125.7, 124.2 and 123.2 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1744 (C=O).

Methyl 2,3-epoxy-3-(3-pyridyl-2-phenyl)propanoate 1c. (62%, 5 h) Yellow oil purified by chromatography on activated alumina, eluting with ether (Found: M^+, 255.089 02. $C_{15}H_{13}NO_3$ requires *M*, 255.089 54); δ_H(200 MHz) 3.5 (1 H, d, *J* 1.8, 2-H), 3.75 (3 H, s, OCH_3), 3.92 (1 H, d, 3-H), 7.3–7.5 (5 H, m, Ar-H), 7.65–7.7 (1 H, m, Ar-H) and 8.6–8.65 (2 H, m, Ar-H); δ_C(50 MHz) 167.9 (C=O), 149.4, 148.7 (C-2, C-4 pyridyl), 137.8 (C-1, pyridyl), 133.7 (C-1, phenyl), 132.5 (C-2, phenyl), 56.6 (C-3), 56.0 (C-2), 52.5 (OCH_3), 136.2, 131.2, 129.8, 128.6, 128.4 and 124.5 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1750 (C=O).

Methyl 2,3-epoxy-3-(4-pyridyl-2-phenyl)propanoate 1d. (61%, 4 h) White solid, mp 63–64 °C from hexane–ether (95:5) (Found: C, 70.3; H, 5.3; N, 5.45%; M^+, 255.089 12. $C_{15}H_{13}NO_3$ requires C, 70.5, H, 5.1; N, 5.5%; *M*, 255.089 54); δ_H(200 MHz) 3.47 (1 H, d, *J* 1.9, 2-H), 3.73 (3 H, s, OCH_3), 3.9 (1 H, d, 3-H), 7.25–7.4 (6 H, m, Ar-H) and 8.5–8.6 (2 H, m, Ar-H); δ_C(50 MHz) 167.9 (C=O), 149.7 (C-3, C-5, pyridyl), 147.2 (C-1, pyridyl), 138.7 (C-1, phenyl), 132.1 (C-2, phenyl), 56.6 (C-3), 56.0 (C-2), 52.5 (OCH_3), 129.2, 128.8, 128.7, 124.5 and 123.9 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1750 (C=O).

Methyl 2,3-epoxy-3-(3-phenyl-2-thienyl)propanoate 1e. (71%, 5 h) Yellow oil purified by chromatography on activated alumina, eluting with hexane–ether (75:25) (Found: M^+, 260.052 72. $C_{14}H_{12}O_3S$ requires *M*, 260.050 72); δ_H(200 MHz) 3.78 (1 H, d, *J* 1.8, 2-H), 3.8 (3 H, s, OCH_3), 4.32 (1 H, dd, *J* 1.9, 3-H) and 7.08–7.47 (7 H, m, Ar-H); δ_C(50 MHz) 168.0 (C=O), 143.7 (C-2, thiophene), 134.8 (C-3, thiophene), 132.5 (C-1, phenyl), 57.2 (C-3), 54.2 (C-2), 52.5 (OCH_3), 129.5, 128.6, 128.5, 127.5 and 125.0 (remaining aromatic CH signals); ν_{max}(Nujol)/cm^{-1} 1745 (C=O).

Flash vacuum pyrolysis of the oxiranes 1a,b and d to give heterobenzooxepines 4a,b and d

General method for flash vacuum pyrolysis. The reactant was distilled at 8 × 10^{-4} mmHg through a furnace tube (35 × 2.5 cm) maintained at 625 °C and the product was collected in a U-tube cooled by liquid nitrogen, which was situated at the exit of the furnace. This was successful for compounds **1a,b** and **d** but **1c** and **1e** decomposed in the inlet system to give black tars.

Methyl 4,6-dihydrothieno[3,2-*d*][2]benzooxepin-4-carboxylate 4a. From the oxirane **1a** (0.25 g, 0.96 mmol); distillation temperature 130–150 °C; pyrolysis time 1 h. Dry-column flash chromatography, eluting with hexane–ether (75:25), gave compound **4a** as a yellow oil (0.2 g, 80%), bp 170–180 °C/0.5 mmHg (Found: M^+, 260.052 16. $C_{14}H_{12}O_3S$ requires *M*, 260.050 72); δ_H(200 MHz) 3.7 (3 H, s OMe), 4.67 (1 H, d, *J* 13.1, 6-H_{ax}), 4.81 (1 H, d, *J* 13.1, 6-H_{eq}), 5.55 (1 H, s, 4-H) and 7.1–7.65 (6 H, m, Ar-H); δ_C(50 MHz) 170.5 (C=O), 139.1 (C-10b), 137.0 (C-3a), 133.8, 133.2, (C-6a, C-10a), 128.5, 128.4, 128.1, 127.4, 127.3, 125.0 (aromatic C-H), 78.4 (C-4), 69.6 (C-6) and 52.3 (OMe); ν_{max}(Nujol)/cm^{-1} 1743 (C=O).

Methyl 4,6-dihydrothieno[2,3-*d*][2]benzooxepine-4-carboxylate 4b. From the oxirane **1b** (0.25 g, 0.96 mmol); distillation temperature 130–150 °C; pyrolysis time 1 h. Dry-column flash

chromatography, *eluting* with hexane–ether (75:25), gave compound **4b** as a yellow oil (0.19 g, 76%), bp 170–180 °C/0.5 mmHg) (Found: M^+, 260.049 66. $C_{14}H_{12}O_3S$ requires *M*, 260.050 72); δ_H(200 MHz) 3.7 (3 H, s, OMe), 4.6 (2 H, 6-$H_{ax/eq}$), 5.45 (1 H, s, 4-H) and 7.30–7.61 (6 H, Ar-H); δ_C(50 MHz) 170.5 (C=O), 140.2 (C-3a), 136.9 (C-10b), 135.5 (C-10a), 133.1 (C-6a), 129.3, 128.7, 127.6, 127.5, 127.1, 125.6 (aromatic C-H), 74.7 (C-4), 69.0 (C-6) and 52.5 (OMe); ν_{max}(Nujol)/cm^{-1} 1747 (C=O).

Methyl 5,7-dihydro[2]benooxepino[4,5-*c*]pyridine-5-carboxylate 4d. From the oxirane **1d** (0.25 g, 0.98 mmol); distillation temperature 140–160 °C; pyrolysis time 1 h. Column chromatography on activated alumina, eluting with hexane–dichloromethane (25:75), gave compound **4d** as a white solid (0.14g, 56%), mp 169–170 °C, from hexane–ether (50:50) (Found: C, 70.8; H, 4.95; N, 5.3%; M^+, 255.089 81. $C_{15}H_{13}NO_3$ requires C, 70.55; H, 5.1; N, 5.5%; *M*, 255.089 54); δ_H(200 MHz) 3.4 (3 H, s, OMe), 4.4 (1 H, d, *J* 11.9, 7-H_{ax}), 4.54 (1 H, d, *J* 11.9, 7-H_{eq}), 5.5 (1 H, s, 5-H), 7.42–7.53 (5 H, m, Ar-H), 8.57 (1 H, br s, Ar-H) and 8.73–8.76 (1 H, m, Ar-H); δ_C(50 MHz) 170.9 (C=O), 150.7 and 150.1 (C-2, C-4), 147.8 (C-4a), 137.5 (C-11b), 135.1 (C-11a), 129.0 (C-7a), 130.1, 130.0, 129.4, 127.3, 121.7 (aromatic C-H), 74.5 (C-5), 67.3 (C-7) and 52.1 (OMe); ν_{max}(Nujol)/cm^{-1} 1751 (C=O).

Acknowledgements

We thank the EPSRC for a postdoctoral fellowship (D. F. O'S.) and Dr H. McNab for advice on flash vacuum pyrolysis.

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Paper 5/06075C
Received 14*th September* 1995
Accepted 12*th October* 1995