Tetrahedron Vol. 44, No. 5, pp. 1567 to 1572, 1988
Printed in Great Britain.

0040-4020/88 $3.00+.00

A NEW APPROACH TO THE SYNTHESIS OF MORPHINANS AND 4a-ARYLDECAHYDROISOQUINOLINES

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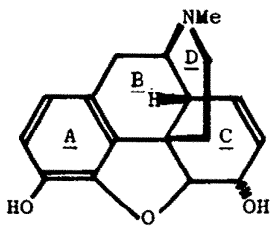
(*Received in UK 7 December* 1987)

Abstract-The Friedel-Crafts alkylation of benzene with 7a-carbethoxymethyloctahydrobenzofuran-2-one provided 1,2-di(carboxymethyl)-1-phenylcyclohexane. A two stage cyclisation of this diacid via intramolecular acylation gave 9,10-benzobicyclo[5.3.2.$0^{1,6}$]dodecan-8,12-dione as an attractive precursor of the morphinan ring system. Alternatively, the foregoing diacid could be cyclised to phenylbicyclo[4.3.0]nonan-8-one which was converted into 2-methyl-4a-phenyl-*trans*-decahydroisoquinoline.

A continuing point of departure in the quest for the perfect analgesic is the synthesis of molecules retaining a significant portion of the ring system of morphine (1). Typical examples are provided by the morphinans (2)[1], which lack only the furan ring, and the 4a-aryldecahydroisoquinolines (3)[2] which additionally lack ring B. Prior synthetic endeavours have been largely directed to the production of derivatives of (2) and (3) having similar functionality in the residual ring C to that found in morphine or its simple derivatives. The production of compounds in which one or more of the ring C carbon atoms has been replaced by a heteroatom appears to have received little or no attention, and indeed the existing syntheses seemed to be ill-suited for this purpose. The present paper reports the results of model studies aimed at establishing a more suitable synthetic route to such compounds.



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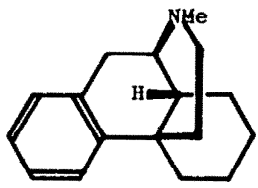

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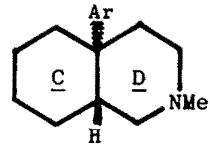
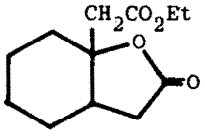


(3)

For reasons of synthetic economy a route which would enable the obtention of derivatives of both (2) and (3) from a common intermediate was especially attractive. Analogy with a route previously used for the synthesis of benzomorphan[3] suggested commencing with a preformed ring C ketone. For our model studies we employed cyclohexanone itself, although we anticipated that other cyclohexanones, tetrahydropyrones or piperidinones might be used instead. Following a literature[4]

procedure cyclohexanone was converted, through the pyrrolidine enamine and subsequent reaction with ethyl bromoacetate, into ethyl 2-oxocyclohexylacetate. The latter compound was then subjected to a Reformatsky with ethyl bromoacetate to give the lactone (4). An earlier paper[5] had shown that the addition of methylmagnesium iodide to ethyl 2-oxocyclohexylacetate generated a 2:1 mixture of the cis and trans lactones corresponding to (4), and the ^{1}H nmr spectrum of (4) itself showed, inter alia, two sets of absorptions for the methyl and methylene protons of the ethyl group in roughly 2:1 proportion suggesting that this also was a mixture of cis and trans isomers. Fredel-Crafts alkylation of benzene with the lactone (4) provided, after hydrolysis, the diacid (5). The ^{13}C nmr spectrum of (5) showed the presence of only fourteen distinct carbon atoms, indicating that only one of the possible diastereoisomers had been obtained.

In contrast to the behaviour of 3-methyl-3-phenyladipic acid[6], cyclisation of the diacid (5) with hot polyphosphoric acid only proceeded as far as the ketoacid (6). Further cyclisation to the diketone (7) could only be effected in miniscule yield on prolonged acid treatment with the remainder of the reactant being destroyed. A more satisfactory procedure entailed the conversion of (6) to the acid chloride with oxalyl chloride and subsequent reaction of the acid chloride with silver perchlorate.



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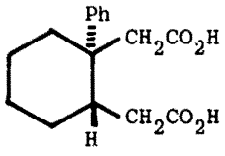

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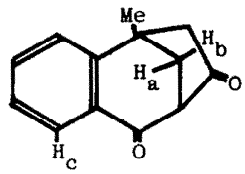

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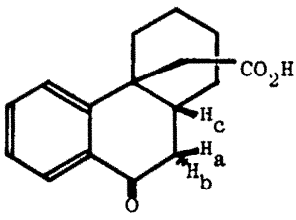

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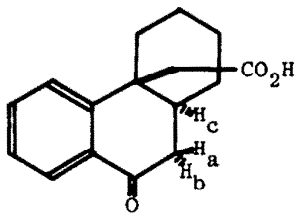

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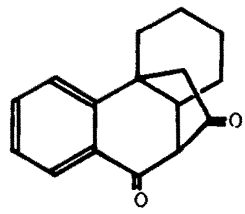
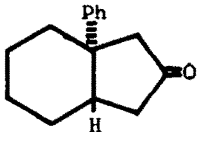


(7)

The stereochemistry of these compounds was established from their ^{1}H nmr spectra. In the spectrum of the ketoacid (6) H_a and H_b were observed as a doublet, J=5Hz, which is consistent with the stereochemistry shown in (6a) where there are identical dihedral angles between H_a and H_c, and H_b and H_c. In contrast, dihedral angles of ca. 180° and 60° between H_c and H_a, and H_c and H_b in (6b) would be expected to result in different coupling constants between these pairs of protons. These conclusions are further supported by the spectral properties of the diketone (7) where H-7 is observed as a doublet, J=12Hz, at 3.87ppm. as a result of its coupling with H-6 whereas a coupling constant of about zero would be anticipated for its C-6 epimer as the two protons would be virtually orthogonal. Additionally, no long range coupling could be detected between H-6 and H-4', but would again have been expected in the C-6 epimer, by analogy with observations on the diketone (8)[6]. Thus the diketone (7) has the same relative stereochemistry as morphinan and should be readily converted into this compound employing one or other of the routes previously reported[3] for the conversion of (8) into benzomorphan.

Our second objective, namely the synthesis of 4a-phenyl-trans-decahydroisoquinolines, was accomplished in the following way. Cyclisation of the diacid (5) with sodium acetate and acetic anhydride provided the ketone (9) in good yield. The trans ring fusion follows from the stereochemistry deduced above for compounds (6a) and (7). The initial intention was to convert (9) into the oximino-ketone (10), and rearrange this into the imide (11). Although analogy with the behaviour of similarly trans-fused cyclopentanones suggested[7] that ketone (9) would preferentially undergo reaction at the C-9 methylene group this did not prove to be the case. Nitrosation of (9) with butyl nitrite in the presence of hydrogen chloride gave a poor yield of the oximino-ketone (10), the orientation of which follows from the observation, inter alia, of a singlet for the two C-9 protons in the ^{1}H nmr spectrum. However, attempts to effect the conversion of (10) into (11) either by heating with polyphosphoric acid or by refluxing with acetic anhydride[8] were unsuccessful. As the Beckmann rearrangement of the oximes of related ketones is reported[9] to yield a mixture of both possible amides it was decided to convert (9) into the unsaturated ketone (12) and then rearrange the derived N-methylnitrone which is known to proceed unidirectionally[10]. Bromination of (9) with bromine in acetic acid provided the 7-bromo derivative as the geminal proton was observed at 4.58 ppm. as a triplet, rather than the singlet anticipated for the 9-bromo isomer.



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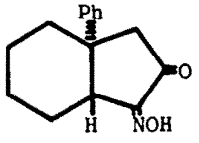

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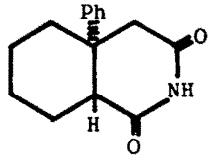

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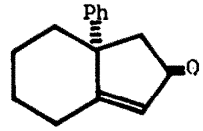

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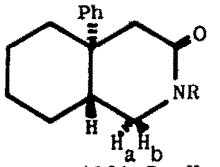
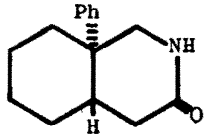


(13) R = H
(14) R = Me



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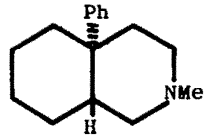

(16)

Presumably the two couplings observed for H-7 arise from interaction with H-6 and one of the methylene protons as established for compound (13), vide infra. Subsequent dehydrobromination with collidine gave the unsaturated ketone (12).

While these transformations were in progress it was noted that the ^{13}C nmr spectrum of the oxime of (9) indicated that it was only one of the possible geometrical isomers. Treatment of this oxime with hot polyphosphoric acid yielded the amide (13). The ^{1}H nmr spectrum of (13) displayed signals for the C-1 protons at 2.99 ppm (t, J=11Hz) and at 3.29 ppm. The initial multiplet observed in the latter case simplified to a doublet of triplets (J= 11 and 4.9Hz) on exchange of the amide N-H with D_2O, and can be assigned to H_{1b}, which is approximately coplanar with the N-H proton. The 11 Hz coupling observed for this proton is clearly a geminal one with H_b and one of the 4.9 Hz couplings arises from interaction with H-8a, while a COSY spectrum indicates that the remaining one arises from a longer range coupling with a methylene proton at 1.95ppm. The 11 Hz couplings observed for H_{1b} are attributable to a geminal interaction with H_{1a} and a vicinal one with H-8a. These spectral data clearly establish the structure of the amide as (13) rather than the isomeric (15). The conversion of (13) to (16) was established by N-methylation with sodium hydride and methyl iodide, followed by a lithium aluminium hydride reduction of the intermediary (14).

EXPERIMENTAL

7a-Carbethoxymethyloctahydrobenzofuran-2-one (4). Ethyl bromoacetate (17 ml, 0.15 mol) in dry benzene (25 ml) was added dropwise to a stirred suspension of activated zinc dust (10g, 0.15 mol) in refluxing dry ether (100 ml). To this was added dropwise and with stirring ethyl 2-oxocyclohexylacetate (14.5 ml, 0.08 mol) in dry benzene over a period of 30 minutes. The resulting mixture was refluxed for a further 3 hours, cooled, poured onto ice and acidified with hydrochloric acid. The organic layer was separated off and the aqueous phase extracted with ether. The combined organic extracts were washed with saturated brine, dried ($MgSO_4$) and evaporated to give (4) (9g, 53%) bp 112-115° at 0.5 mm Hg. (Found: C, 63.93; H, 8.20. $C_{12}H_{18}O_4$ requires: C,63.72; H, 7.97%); IR 1780, 1735 cm^{-1}; NMR (90MHz) 1.18(t, 3H, J=7Hz, $CH_2\underline{CH_3}$), 1.3-1.65(m, 4H), 1.65-1.95(m, 3H), 2.2-2.6(m, 4H), 2.65 (s, 2H, $\underline{CH_2}CO_2Et$), 4.05 and 4.1(two quartets of roughly 2:1 intensity, J=7Hz, $\underline{CH_2}CH_3$); MS m/z 226(M^+,23), 209(52), 208(36), 198(37), 180(22), 139(87), 83(100).

1,2-Di(carboxymethyl)-1-phenylcyclohexane (5). The lactone (4) (40g,0.177 mol) in dry benzene (200 ml) was added dropwise to a stirred suspension of anhydrous aluminium chloride (80g, 0.6 mol) in dry benzene (150 ml) maintained below 5°. On completion of the addition the resulting mixture was stirred overnight at room temperature, and subsequently poured onto a mixture of ice and hydrochloric acid. The resulting two layers were separated, the aqueous layer extracted with ether and the combined organic extracts then extracted with 15% potassium hydroxide solution. The basic extracts together with potassium hydroxide (13g) and ethanol (25 ml) were heated under reflux for 4 hours, cooled and then poured onto ice and hydrochloric acid. The preipitated diacid (5) was recrystallised from methanol (23.5g, 48%) mp 164-166° (Found: C, 69.84; H, 7.26. $C_{16}H_{20}O_4$ requires: C, 69.56; H, 7.25%); IR(Nujol) 3400-2600, 1700 cm^{-1}; ^1H NMR (90MHz, pyridine-d_5) 1.3-1.7 (m,3H), 1.7-1.95(bd, 1H),1.95-2.4(m, 4H); 2.49(dd, 1H, J=8.4 and 3.7Hz,CH_2CO_2H), 2.50(dd, 1H, J=8.5 and 3.8Hz,CH_2CO_2H), 2.63(bt, 1H, J=12Hz), 3.0-3.06(2H,dd, outer lines obscured, CH_2CO_2H), 7.25-7.4(m, 5H, C_6H_5), 13.70(bs,2H, exchangeable with D_2O, CO_2H); ^{13}C NMR 128.8, 127.2, 126.4 and 44.9(CH); 40.9, 39.7, 39.6, 39.0, 34.1 and 33.2(CH_2); 147.3 and 43.65(q C's); 175.8 and 175.6(C=O); MS m/z 276(M^+,8), 258 (40), 240(10), 217(80), 203(62), 180(18), 170(27), 157(46), 154(59), 143(27), 129 (34), 117(50), 104(92), 91(100).

4a-Carboxymethyl-1,2,3,4,4a,9,10,10a-octahydro-9-oxophenanthrene (6a).

Polyphosphoric acid (25 ml) was added to the diacid (5) (5.0g) and with efficient stiring the temperature was raised slowly to an external oil bath temperature of 120°, and heating then continued for 2 hours. The cooled reaction mixture was poured onto ice and the resulting solid filtered off. This was purefied by column chromatography on silica gel using petroleum ether: ethyl acetate: acetic acid (2:6:1) to give (6a) (4.1g, 87%), mp. 105-106° from pet. ether/ethyl acetate. (Found: C, 74.76; H,6.56. $C_{16}H_{18}O_3$ requires C, 74.41; H, 6.97%); IR(Nujol) 3400-2500, 1700, 1660, 1600, 770 cm^{-1}; NMR (90MHz) 1.45-2.45(m, 8H, 1-,2-,3-,4-H), 2.57 (d, 1H, J=12Hz, H-11), 2.61(d, 1H, J=12Hz, H-11),2.94(d,2H, J=7.5Hz, H-10), 3.2-3.4(m, 1H, H-10a), 7.28-7.60(m, 3H, H-5, H-6, H-7), 7.95(dd, 1H, J=9 and 2Hz), 10.4(bs, 1H, exchangeable with D_2O, -COOH); MS m/z 258(M^+, 45), 240(20), 222(25), 198(27), 157(57), 118(100), 114(60).

9,10-Benzobicyclo[5.3.2.$0^{1,6}$]dodecan-8,12-dione (7). The ketoacid (6a) (2g,0.008 mol) in dry benzene was treated with oxalyl chloride (1.0 ml,0.011 mol) and refluxed for 1 hour. The reaction mixture was then evaporated in vacuo to give the acid chloride as an oil. IR 1800, 1665 cm^{-1}. This was then dissolved in nitromethane (10 ml) and silver perchlorate (1.3g, 0.0063 mol) added tothe stirred solution. After stirring for 24 hours at room temperature the reaction mixture was filtered and the filtrate poured into water. Extraction with chloroform provided the crude product which was chromatographed on silica gel in toluene-ethyl acetate (4:1) to give the diketone (7) (0.4g, 28%) as an oil. (Found: C,79.8; H,6.34. $C_{16}H_{16}O_2$ requires: C, 80.0; H, 6.67%); IR(neat) 1740, 1680, 1610, 770 cm^{-1}; NMR (250MHz) 1.45-2.3(m, 8H, H-2, H-3, H-4, H-5), 2.4(m, 2H, H-11), 2.75-2.95(m, 1H, H-6), 3.5(d, 1H, J=12Hz, H-7), 7.1-7.3(m, 3H, H-1', H-2', H-3'), 7.85(dd, 1H, J=8 and 2Hz, H-4'); MS m/z 240(M^+, 5), 91(100).

1-Phenyl-trans-bicyclo[4.3.0]nonan-8-one (9).The diacid (5) (1g) in acetic anhydride (10ml) was refluxed for 2.5 hours when sodium acetate (1g) was added and refluxing continued for a further 2.5 hours. The reaction mixture was then cocentrated and the residual solid treated with methanol (10ml). The remaining solid was filtered off and the filtrate diluted with water. After basification with potassium bicarbonate the organic product was isolated by ether extraction and chromatographed on silica gel. Elution with petroeum ether/ethyl acetate (7:3) yielded the ketone (9) (0.57g, 74%) as a colourless oil. (Found: C, 84.25; H, 8.39. $C_{15}H_{18}O$ requires: C, 84.11; H, 8.41%); IR(neat) 1720 cm^{-1}; NMR (250MHz) 1.15-2.1 (m, 10H), 2.31(t, 1H, J=5.4Hz), 2.40(t, 1H, J=5.7Hz), 2.68(dt, 1H, J=12 and 3.5Hz), 7.1-7.4(m, 5H, C_6H_5); MS m/z 214(M^+, 66), 130(37), 110(22), 104(100), 95(35),91(82).

The ketone was converted into the oxime by treatment with hydroxylamine hydrochloride and sodium acetate in aqueous ethanol. The oxime was obtained as a colourless oil after chromatography over silica gel in toluene/ethyl acetate (3:1) in 88% yield. (Found:C, 78.58; H, 7.85; N, 6.02. $C_{15}H_{19}NO$ requires: C, 78.60; H, 8.30; N, 6.11%); IR(neat) 3300, 1610 cm^{-1}.; ^1H NMR(90MHz) 1.25-2.16(m, 8H, H-2, H-3, H-4, H-5), 2.25-2.7(m, 4H, H-7,H-9), 3.35(m, 1H, H-6), 7.05-7.35(m, 5H, C_6H_5), 8.2(bs, 1H, exchangeable with D_2O, OH); ^{13}C NMR 31.00, 33.59, 34.15,34.31, 36.86, 38.63 (all CH_2's), 44.49(CH), 65.84(qC), 126.02, 126.83, 128.35(all CH's), 146.77(qC), 165(C=N)ppm.; MS m/z 229(M^+, 2), 85(65), 83(100).

7-Oximino-1-phenyl-trans-bicyclo[4.3.0]nonan-8-one (10). Hydrogen chloride gas was bubbled through a solution of the ketone (9) (2g, 0.0093mol) in dry ether (15ml). n-Butyl nitrite (2g, 0.019mol) was then added dropwise. After addition of the first portion of butyl nitrite the mixture became dark brown in colour, changing to yellow after a few minutes. At this point the reaction was warmed so that the ether began to reflux gently. The rest of the nitrite was slowly added, and subsequently stirring and bubbling of hydrogen chloride was continued for a further 15 minutes. The reaction mixture was allowed to stand overnight and then poured into water. Extraction with chloroform provided a brown gum which was chromatographed on silica gel in petroleum ether/ethyl acetate (1:1) to give (10) as an oil (0.5g, 22%). (Found: C,73.86; H, 6.73; N, 5.66. $C_{15}H_{17}NO_2$ requires: C, 74.07; H, 6.99; N,5.76%). IR(neat) 3300, 1690 cm^{-1}; NMR(90MHz) 1.25-2.6(m,8H, H-2,H-3,H-4,H-5), 3.1(m,1H, H-6), 3.5(s, 2H, H-9), 7.2-7.3(m, 5H, C_6H_5), 7.9(bs, 1H, exchangeable with D_2O,OH); MS m/z 243(M^+,6), 242(3), 170(5), 157(7), 143(9), 130(36), 129(21), 110(26), 104 (100), 95(25), 91(77).

4a-Phenyl-3-oxo-transdecahydroisoquinoline (13). The oxime of ketone (9) (1g, 0.0044 mol) was stirred for two hours with polyphosphoric acid (15g) at 120°C. After cooling the reaction mixture was poured onto ice and the organic products isolated by chloroform extraction. The crude product thus obtained was purified by chromatography over silica gel in toluene/ethyl acetate/methanol (6:3:1) to give (13) (0.85g, 85%), mp. 82-83°C from toluene. (Found: C, 78.35; H, 8.18; N, 5.72. $C_{15}H_{19}NO$ requires: C, 78.60; H, 8.29; N, 6.11%); IR(nujol) 3200, 1660 cm^{-1}.; ^1H NMR(250MHz) 1.14-1.85(m,4H), 1.85-2.15(m, 4H),2.40-2.75(m,3H),2.99(t, 1H, J= 11Hz), 3.29(m, 1H, appeared as a dt after D_2O exchange, J= 11 and 4.9Hz), 6.29 (bs, 1H, exchangeable with D_2O, NH), 7.17-7.34(m, 5H, C_6H_5); ^{13}C NMR 172.66(CO), 146.43(qC), 128.46(CHx2), 126.72(CHx2), 126.24(CH), 47.89(qC), 43.41(CH_2), 40.53 (CH_2), 38.30(CH), 37.88(CH_2), 37.27(CH_2), 36.35(CH_2), 33.11(CH_2); MS m/z 229(M^+, 87), 104(100).

2-Methyl-4a-phenyl-3-oxo-transdecahydroisoquinoline (14). The amide (13) (0.6g, 0.0026 mol) and sodium hydride (0.25g, 0.010 mol) in a mixture of toluene (10ml) and N,N-dimethylformamide (15ml) were stirred together for 1 hour. Methyl iodide (0.5ml, 0.008 mol) was added and the mixture allowed to stir overnight. After careful addition of water the resulting mixture was extracted with chloroform. The combined extracts were washed with saturated brine, dried ($MgSO_4$) and evaporated to afford (14) as a pale yellow syrup (0.21g, 33%). IR(neat) 1650 cm^{-1}; NMR(90MHz) 1.1-1.9(m, 8H, H-5,H-6,H-7,H-8), 2.4-2.5(m, 1H,H-8a), 2.8(s, 2H,H-4), 3.1(s, 3H, NCH_3), 3.9(m,1H, H-1), 4.15(t, 1H, J=9Hz, H-1), 7.3-7.5(m, 5H, C_6H_5); MS m/z 243(M^+,47), 115(28), 110(100), 91(26).

2-Methyl-4a-phenyl-trans-decahydroisoquinoline (16). Lithium aluminium hydride (0.1g) was added in portions over 10 minutes to a stirred solution of the amide (14) (0.2g) in dry ether (25ml) and the mixture heated under reflux overnight. After the careful addition of just sufficient water to destroy the excess hydride the precipitated alumina was filtered off. The ether filtrate was then extracted with 2M hydrochloric acid. The acid extracts were neutralised with aqueous ammonia and the amine (16) isolated by ether extraction. Although previously obtained[2] as a low melting solid our small sample (0.12g, 65%)failed to crystallise. NMR(90MHz) 1.2-1.9(m, 8H, H-5,H-6,H-7,H-8), 2.2-2.3(m,1H), 2.5(s,3H,NCH_3), 2.95(m,1H), 3.05-3.15(m,1H), 7.2-7.5(m,5H, C_6H_5); MS m/z 229(M^+, 77), 228(65), 151(18), 104(36), 96(28), 91(47), 58(100).

1-Phenylbicyclo[4.3.0]non-6-en-8-one (12). To a solution of the ketone (9) (1g, 0.0047 mol) in glacial acetic acid (5ml) was added over about 10 minutes bromine (0.8g, 0.005mol) with shaking and the temperature kept below 20°C. When the addition was complete the mixture was poured into ice-water and the product extracted with ether. The material thus obtained was chromatographed over silica gel in hexane/ethyl acetate (4:1) to give the bromoketone (1.1g, 79%) as a yellow oil of limited stability. IR(neat) 1760cm^{-1}; NMR(90MHz) 0.95-2.2(m, 8H, H-2,H-3, H-4,H-5), 2.27(s,2H, H-9), 2.6-2.85(m,1H, H-6), 4.58(t,1H,J=5.4Hz,H-7), 7.0-7.7 (m,5H,C_6H_5); MS m/z 294(M^+,13), 292(M^+, 16), 213(10), 198(14), 196(14), 183(19), 172(27), 170(30), 150(14), 129(17), 118(30), 117(55), 116(34), 91(46), 77(46). The bromoketone (0.6g) in collidine (10ml) was refluxed for 24 hours. After cooling the mixture was poured into water and extracted with chloroform. The

combined organic extracts were washed with dilute sulphuric acid and saturated brine, dried ($MgSO_4$) and evaporated in vacuo. The resulting crude product was chromatographed over silica gel in toluene/ethyl acetate (7:1) to give the unsaturated ketone (12) (0.29g, 65%) as a colourless oil. (Found: M^+ 212. $C_{15}H_{16}O$ requires: M^+ 212); NMR(90MHz) 0.95-1.9(m,6H, H-2,H-3,H-4), 2.1(s,2H,H-9), 2.3-2.6(m,2H,H-5), 5.92(bs,1H,H-7), 6.95-7.5(m,5H,C_6H_5).

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