Regiochemistry of Radical Cyclisations (6-*exo*/7-*endo* and 7-*exo*/8-*endo*) of *N*-(*o*-Alkenylphenyl)-2,2-dichloroacetamides

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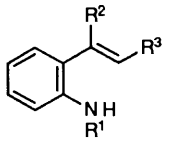
N-[*o*-(Alk-1-enyl)phenyl]-2,2-dichloroacetamides, when treated with 2.2 mol equiv. of Bu_3SnH in the presence of a catalytic amount of azoisobutyronitrile, gave quinolin-2(1*H*)-one (6-*exo* closure) and/or 2*H*-1-benzazepin-2-one (7-*endo* closure) systems. In general, the 6-*exo* cyclisation is favoured over the 7-*endo* closure, unless a large group such as phenyl is present at the 1-position of the alkene. *N*-[*o*-(1-Methylethenyl)phenyl]acetamide congeners underwent a 6-*exo* closure followed by rearrangement to give 1,5-dihydro-4-methyl-2*H*-1-benzazepin-2-ones. A similar treatment of *N*-[*o*-(prop-2-enyl)phenyl]acetamide derivatives gave 2*H*-1-benzazepin-2-ones (7-*exo*) and/or 1-benzazocin-2(1*H*)-ones (8-*endo*).

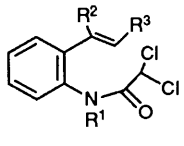
Recently we reported on the radical cyclisation of *N*-(prop-2-enyl)acetamide derivatives which proceeds in a highly regioselective manner to give five-membered lactams (5-*exo*-closure).[1,2] The 5-*exo* preference is also observed with the 2-methylprop-2-enyl congener. As an extension of this chemistry, we were then led to examine the regiochemistry of the α-carbamoylmethyl radical cyclisation in the formation of the larger ring. To this end, we chose *N*-[*o*-(alk-1-enyl)phenyl]-acetamide **2** (6-*exo*/7-*endo*) and *N*-[*o*-(alk-2-enyl)phenyl]-acetamide derivatives **17** (7-*exo*/8-*endo*) as suitable models. We report here the results of a study of the radical cyclisation of compounds **2** and **17**. In this paper a rearrangement that was encountered with *N*-[*o*-(1-methylethenyl)phenyl]acetamide† derivatives is also described.

The radical precursors **2a–h**, **3–6** and **17a**, **b** were prepared from the corresponding aniline derivatives **1a–h** and **16** by standard methods (see Experimental section).



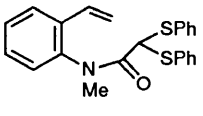
1a–h

a; $R^1 = Me$, $R^2 = R^3 = H$
b; $R^1 = R^3 = Me$, $R^2 = H$
c; $R^1 = R^2 = H$, $R^3 = Me$
d; $R^1 = R^2 = H$, $R^3 = CO_2Me$



2a–h

e; $R^1 = R^2 = Me$, $R^3 = H$
f; $R^1 = R^3 = H$, $R^2 = Me$
g; $R^1 = Me$, $R^2 = Ph$, $R^3 = H$
h; $R^1 = R^3 = H$, $R^2 = Ph$



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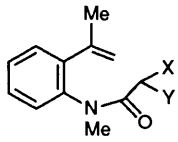


4 X = Cl, Y = SMe
5 X = Y = SPh
6 X = Cl, Y = H

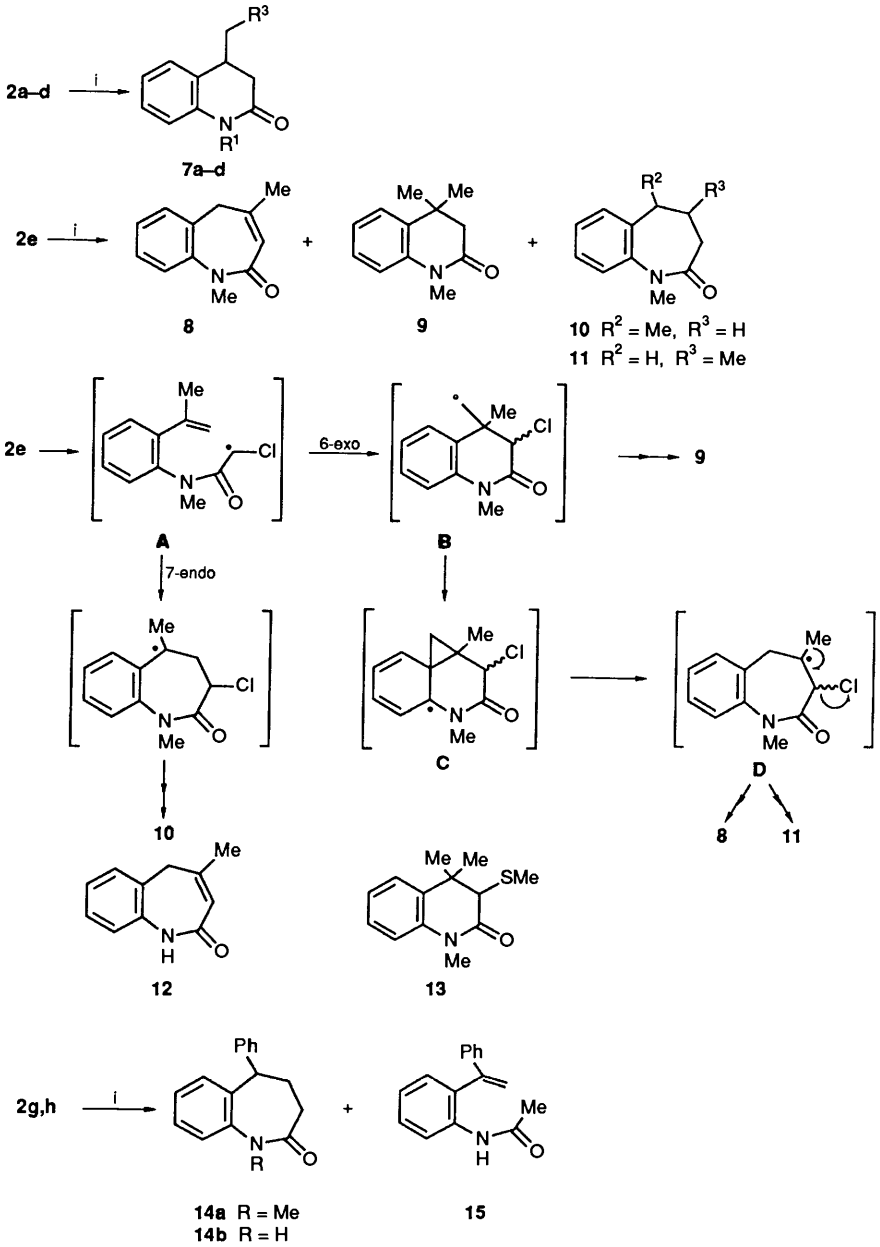
A mixture of tributyltin hydride (Bu_3SnH) (1.1 mol equiv.) and a catalytic amount of azoisobutyronitrile (AIBN) in toluene was added to a boiling solution of the dichloroacetamide **2a** in toluene (6×10^{-3} mol dm^{-3}) during 40 min, and the resulting mixture was refluxed for an additional hour. To eliminate the second chlorine atom from the initially formed cyclised product (this procedure is not necessary for the cyclisation of the monochloroacetamide derivatives), a solution of Bu_3SnH (1.1 mol equiv.) and AIBN in toluene was added, and the mixture was again heated under reflux for 2 h. Evaporation of the solvent followed by chromatography on silica gel afforded 3,4-dihydro-1,4-dimethylquinolin-2(1*H*)-one **7a**[3] in 49% yield. The same product **7a** was also obtained from the bis(phenylthio)acetamide **3** in 50% yield upon treatment with Bu_3SnH (2.2 mol equiv.) (when 1.1 mol equiv. of Bu_3SnH was used, the reaction was very slow). Similarly the dichloroacetamides **2b–d** with Bu_3SnH (2.2 mol equiv.) gave the corresponding 3,4-dihydroquinolin-2(1*H*)-ones **7b** (79% yield), **7c** (100%) and **7d** (100%). In view of the fact that cyclisation of the *N*-unsubstituted *N*-(prop-2-enyl)acetamides is an unfavourable process,[1,4] it is somewhat surprising that the *N*-unsubstituted acetamides **2c** and **2d** gave the cyclised products **7c**, **d** in excellent yields.‡ The closeness of the radical centre and acceptor may be responsible for this anomaly.

In contrast, similar treatment of 2,2-dichloro-*N*-[*o*-(1-methylethenyl)phenyl]acetamide **2e** gave a rearranged product, 1,5-dihydro-1,4-dimethyl-2*H*-1-benzazepin-2-one **8** (68% yield), the 3,4-dihydroquinolin-2(1*H*)-one **9**[3] (24%), and an inseparable mixture of 1,3,4,5-tetrahydro-1,5-dimethyl- **10** and 1,3,4,5-tetrahydro-1,4-dimethyl-2*H*-1-benzazepin-2-ones **11** (10%; 2:1). The structure **8** was deduced from the spectroscopic and chemical evidence. The ^1H NMR spectrum of compound **8** revealed a methyl signal at δ 1.97 (d, *J* 1.5 Hz), a methylene singlet at δ 3.23, and an olefinic proton signal centred at δ 5.60 (1 H, m). Catalytic hydrogenation of compound **8** over 10% Pd-carbon in methanol gave compound **11**. The structures **10**[6] and **11** were deduced from a comparison of the NMR spectrum of the mixture and the gas–liquid chromatography (GLC) retention time with those of each authentic sample.

The same rearrangement was also observed with the dichloroacetamide **2f**, the 2-chloro-2-(methylthio)acetamide **4**, and the bis(phenylthio)acetamide **5**, giving the rearranged 2*H*-1-benzazepin-2-ones **12** (44%), **8** (48%) and **8** (45%), respectively, along with other, minor products (see Experimental section). Treatment of the chloroacetamide **6** with Bu_3SnH (1.1 mol equiv.) gave compound **9** (19%), and a mixture of benzazepinones **10** and **11** (24% total yield, 1:1).

One possible mechanism for the formation of the rearranged product **8** would involve a neophyl radical intermediate (B) formed by a 6-*exo* closure of the initially formed radical (A). The radical (B) could then attack the phenyl ring activated

† *N*-(*o*-Isopropenylphenyl)acetamide.
‡ In contrast, the *N*-(prop-2-ynyl)acetamide counterparts have recently been shown to undergo efficient radical cyclisation to give the five-membered lactams.[5]



Scheme 1 *Reagents:* i, Bu_3SnH (2 mol equiv.), AIBN

Table 1 Effect of the substituents at the radical centre on the α-carbamoylmethyl radical cyclisation

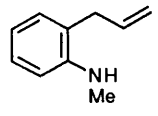
Entry	Starting material	Products (%)[a] The 7-*exo* product	The 8-*endo* product	Others
1	**17a**[b]	**11** (48)		*d*
2	**17b**[c]		**21** (47)	**17f** (10)
3	**17c**[c]	**11** (38)	**20** (44)	
4	**17d**[c]	**18** (58)	**22** (36)	
5	**17e**[c]	**19** (23)	**23** (63)	

[a] Isolated yield. [b] The reaction was carried out using Bu_3SnH (2.2 mol equiv.) in refluxing toluene. [c] The reaction was carried out using Bu_3SnH (1.1 mol equiv.) in refluxing toluene. [d] Some unidentified products were formed.

by the *o*-acylamino group to give the three-membered-ring intermediate (C), which undergoes ring opening followed by dechlorination to give product **8** (Scheme 1). The formation of compound **11** is of special interest, since it may result from trapping of the postulated radical intermediate (D) by hydrogen abstraction from Bu_3SnH. A similar process (a neophyl rearrangement) has been postulated in a radical transformation of 2-bromo-4-methoxy-3-(2-methylprop-2-enyloxy)-benzaldehyde to 5-formyl-8-methoxy-3-methylbenzopyran.[7] Compound **10** is a 7-*endo* cyclisation product of the radical (A).

Our results thus far indicate that the 6-*exo*-closure is much favoured over the 7-*endo*-closure in cyclisation of the α-carbamoylmethyl radicals derived from the *N*-[*o*-(alk-1-enyl)-phenyl]acetamides **2a–f** and **3–6**. However, cyclisation of the 1-phenyl congener **2g** proceeded exclusively in a 7-*endo* manner to give the 2*H*-1-benzazepin-2-one **14a**[8] in 83% yield. Similarly, compound **2h** gave **14b**[9] in 43% yield, along with the reduction product **15** (33%). Two explanations for these results are: (i) the presence of the phenyl group at the 1-position retards the rate of 6-*exo* cyclisation due to steric reasons and (ii) the transition states leading to the 7-*endo* products are stabilised by resonance with the 1-phenyl group. Probably both the steric and the electronic factors are operating to favour the 7-*endo* cyclisation.

We next investigated the behaviour of the *N*-[*o*-(prop-2-enyl)phenyl]acetamide derivatives **17** (7-*exo*/8-*endo*). The dichloroacetamide **17a**, upon treatment with Bu_3SnH (2.2 mol equiv.), gave only the 2*H*-1-benzazepin-2-one **11** (a 7-*exo* product) in 49% yield. Interestingly, when the bis(phenylthio)acetamide **17b** was treated with Bu_3SnH (1.1 mol equiv.), the 1-benzazocin-2(1*H*)-one **21** (an 8-*endo* product) was obtained in 47% yield, together with the reduction product **17f** (9%). The structure **21** was confirmed by desulphurisation with Raney nickel to the known 1-benzazocin-2(1*H*)-one **20**.[10] These observations suggest that regiochemistry of the cyclisation of compounds **17** is highly affected by the substituent on the radical centre.



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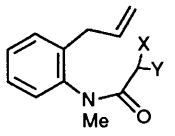
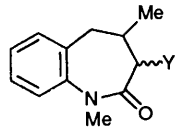
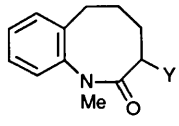


17a–f

a; X = Y = Cl
b; X = Y = SPh
c; X = Cl, Y = H
d; X = Cl, Y = Me
e; X = Cl, Y = Ph
f; X = H, Y = SPh



18 Y = Me
19 Y = Ph



20 Y = H
21 Y = SPh
22 Y = Me
23 Y = Ph

In order to study further the effect of the substituent on the regiochemistry, several radical precursors **17c–e** were synthesized and subjected to the cyclisation conditions. The results are summarised in Table 1. Unfortunately, no simple relationship between the structure and regiochemistry was found, but our observations seem to suggest that the exclusive formation of compounds **11** and **21** is rather exceptional. The electronic and steric structures of the initially formed carbamoylmethyl radicals are likely responsible for the observed selectivity. Further experiments, including the evaluation of the possible role of the heteroatom on the radical centre, would appear to be desirable.

Experimental

IR spectra were recorded with a JASCO-IR-A-100 spectrophotometer. ^1H NMR spectra were determined with a JEOL JNM-PMX 60 (60 MHz) or a Varian XL-300 (300 MHz) spectrometer, and ^{13}C NMR spectra with a Varian XL-300 (75 MHz), for solutions in $CDCl_3$, and δ-values quoted are relative to tetramethylsilane. Exact mass (MS) determinations were obtained on a Hitachi M-80 instrument operating at 20 eV. GLC was carried out on a Shimadzu GC-14A gas chromatograph (helium carrier gas; capillary column at 220 °C). Chromatographic separation was performed with silica gel 60 PF_{254} (Merck) under pressure. Light petroleum refers to the fraction boiling in the range 40–60 °C except where stated otherwise.

Materials.—*N*-Methyl-*o*-(prop-1-enyl)aniline **1b**,[11] *o*-(prop-1-enyl)aniline **1c**,[12] methyl 3-(*o*-aminophenyl)propenoate **1d**,[13] *N*-methyl-*o*-(1-methylethenyl)aniline **1e**,[14] *o*-(1-methylethenyl)-aniline **1f**,[15] *o*-(1-phenylethenyl)aniline **1h**,[16] *N*-methyl-*o*-(1-phenylethenyl)aniline **1g**,[6] and *N*-methyl-*o*-(prop-2-enyl)aniline **16**[17] were prepared according to the reported procedure.

o-*Ethenyl*-N-*methylaniline* **1a**.—To a suspension of K_2CO_3 (16.7 g) and 2-[*o*-(aminophenyl)]ethanol[18] (3.68 g, 26.8 mmol) in acetone (50 cm^3) was added dropwise ethyl chloroformate (8.73 g, 80.5 mmol) and the mixture was refluxed for 2 h. After cooling, the inorganic material was removed by filtration and the filtrate was concentrated to give 2-[*o*-(ethoxycarbonylamino)phenyl]ethyl ethyl carbonate (7.35 g, 97%) as a crude oil, which was used for the next stage without further purification.

A solution of the carbonate (7.35 g, 26.1 mmol) in anhydrous diethyl ether (5 cm^3) was added dropwise to a suspension of lithium aluminium hydride (2.04 g, 53.7 mmol) in anhydrous diethyl ether (50 cm^3) at 0 °C and the mixture was refluxed for 1 h. Usual work-up gave 2-[*o*-(*N*-methylamino)phenyl]-ethanol[19] (4.06 g, quant.) as an oil.

By essentially the same procedure as that of Sabetay *et al.*,[18] KOH (*ca.* 5.0 g) was added to the aforementioned aminophenylethanol (3.90 g, 26.0 mmol) and the mixture was heated at 80–90 °C (bath temperature) for 10 min at 5 mmHg. Hydroquinone (50 mg) was added and the mixture was distilled to give the aniline **1a** (1.84 g, 62%), b.p. 59–61 °C/3 mmHg (lit.,[20] 108 °C/14 mmHg).

General Procedure for the Preparation of 2,2-*Dichloro*-N-[o-(*alk*-1-*enyl*)*phenyl*]*acetamides* **2a–h**.—A solution of dichloroacetyl chloride (1.58 g, 10.7 mmol) in diethyl ether (5 cm^3) was added dropwise to a solution of the aniline **1** (9.76 mmol) and triethylamine (1.09 g, 10.7 mmol) in diethyl ether (20 cm^3) at 0 °C, and the mixture was stirred at room temperature for 10 min and diluted with water. The organic layer was separated, washed successively with saturated aq. $NaHCO_3$ and brine, dried ($MgSO_4$) and concentrated. The residue was chromatographed on silica gel [hexane–AcOEt (7:1)] to give the acetanilide. The following compounds were thus obtained.

2,2-*Dichloro*-N-(o-*ethenylphenyl*)-N-*methylacetamide* **2a** (96%), an oil (Found: C, 53.9; H, 4.5; N, 5.75. $C_{11}H_{11}Cl_2NO$ requires C, 54.1; H, 4.5; N, 5.7%); $\nu_{max}(CCl_4)/cm^{-1}$ 1700; δ 3.36 (3 H, s, NMe), 5.41 (1 H, dd, *J* 11.5 and 2 Hz, one of $CH{=}CH_2$), 5.76 (1 H, s, $CHCl_2$), 5.82 (1 H, dd, *J* 17.5 and 2 Hz, one of $CH{=}CH_2$), 6.69 (1 H, dd, *J* 17.5 and 11 Hz, $CH{=}CH_2$) and 7.0–7.85 (4 H, m, ArH).

(Z)- *and* (E)-2,2-*Dichloro*-N-*methyl*-N-[o-(*prop*-1-*enyl*)-*phenyl*]*acetamide* **2b** (55%), an oil (Found: C, 55.6; H, 5.1; N, 5.4. $C_{12}H_{13}Cl_2NO$ requires C, 55.8; H, 5.1; N, 5.4%).

(Z)- *and* (E)-2,2-*Dichloro*-N-[o-(*prop*-1-*enyl*)*phenyl*]-*acetamide* **2c** (59%) (Found: C, 54.0; H, 4.6; N, 5.9. $C_{11}H_{11}Cl_2NO$ requires C, 54.1; H, 4.5; N, 5.7%); m.p. 144.5–146.0 °C (from hexane–AcOEt).

(E)-*Methyl* 3-[o-(2,2-*Dichloroacetamido*)*phenyl*]*propenoate* **2d** (quant.) (Found: C, 50.0; H, 3.8; N, 4.95. $C_{12}H_{11}Cl_2NO_3$ requires C, 50.0; H, 3.85; N, 4.9%); m.p. 137.5–138.5 °C [from THF–light petroleum].

2,2-*Dichloro*-N-*methyl*-N-[o-(1-*methylethenyl*)*phenyl*]-*acetamide* **2e** (82%) (Found: C, 55.8; H, 5.1; N, 5.5. $C_{12}H_{13}Cl_2NO$ requires C, 55.8; H, 5.1; N, 5.4%); m.p. 57.5–58.5 °C (from hexane).

2,2-*Dichloro*-N-[o-(1-*methylethenyl*)*phenyl*]*acetamide* **2f** (98%) (Found: C, 54.1; H, 4.8; N, 5.6. $C_{11}H_{11}Cl_2NO$ requires C, 54.1; H, 4.5; N, 5.7%); m.p. 43.5–44 °C (from hexane).

2,2-*Dichloro*-N-*methyl*-N-[o-(1-*phenylethenyl*)*phenyl*]-*acetamide* **2g** (47%) (Found: C, 63.9; H, 4.8; N, 4.3. $C_{17}H_{15}Cl_2NO$ requires C, 63.8; H, 4.7; N, 4.4%); m.p. 114.5–115.5 °C (from hexane).

2,2-*Dichloro*-N-[o-(1-*phenylethenyl*)*phenyl*]*acetamide* **2h**

(91%) (Found: C, 62.5; H, 4.2; N, 4.7. $C_{16}H_{13}Cl_2NO$ requires C, 62.8; H, 4.3; N, 4.6%); m.p. 84–85 °C [from light petroleum].

N-(o-*Ethenylphenyl*)-N-*methyl*-2,2-*bis*(*phenylthio*)*acetamide* **3**.—Benzenethiol (596 mg, 5.41 mmol) was added to a solution of sodium ethoxide in ethanol [prepared from sodium (124 mg, 5.41 mmol) and ethanol (15 cm^3)], and the mixture was stirred at room temperature for 10 min. A solution of the dichloride **2a** (600 mg, 2.45 mmol) in dichloromethane (5 cm^3) was added to the above solution and the mixture was stirred at room temperature for 16 h. After removal of the solvent, the residue was dissolved in water, and extracted with dichloromethane. The extract was dried ($MgSO_4$) and concentrated. The residue was chromatographed on silica gel [hexane–AcOEt (7:1)] to give the *acetamide* **3** (518 mg, 54%) (Found: C, 70.4; H, 5.4; N, 3.4. $C_{23}H_{21}NOS_2$ requires C, 70.55; H, 5.4; N, 3.6%); m.p. 91.5–92.5 °C (from hexane–AcOEt); $\nu_{max}(CHCl_3)/cm^{-1}$ 1665; δ 3.16 (3 H, s, NMe), 4.67 [1 H, s, C*H*(SPh)$_2$], 5.22 (1 H, dd, *J* 11 and 2 Hz, one of CH=C*H*$_2$), 5.64, (1 H, dd, *J* 18 and 2 Hz, one of CH=C*H*$_2$) and 6.3–7.7 (15 H, m, C*H*=CH$_2$ and ArH).

2-*Chloro*-N-*methyl*-N-[o-(1-*methylethenyl*)*phenyl*]-2-(*methylthio*)*acetamide* **4**.—Using a procedure similar to that described for the preparation of compounds **2a–h**, the aniline **1e** (1.50 g, 10.2 mmol) was treated with (methylthio)acetyl chloride (1.40 g, 11.2 mmol) and work-up gave N-*methyl*-N-[o-(1-*methylethenyl*)*phenyl*]-2-(*methylthio*)*acetamide* (1.22 g, 51%) (Found: C, 66.0; H, 7.5; N, 5.5. $C_{13}H_{17}NOS$ requires C, 66.35; H, 7.3; N, 5.95%); m.p. 54.5–56 °C [from hexane–light petroleum].

N-Chlorosuccinimide (187 mg, 1.40 mmol) was added by portions to a solution of the sulphide obtained above (300 mg, 1.27 mmol) in tetrachloromethane (10 cm^3) at 0 °C and the mixture was stirred at room temperature for 4 h. The precipitated succinimide was filtered off and the filtrate was concentrated to give the title acetamide **4** in quantitative yield, which was used immediately in the next stage.

N-*Methyl*-N-[o-(1-*methylethenyl*)*phenyl*]-2,2-*bis*(*phenylthio*)*acetamide* **5**.—Using a procedure similar to that described for the preparation for compound **3**, the *acetamide* **5** (462 mg, 37%) was obtained from the dichloride **2e** (800 mg, 3.1 mmol) and sodium benzenethiolate (6.8 mmol) (Found: C, 71.1; H, 5.85; N, 3.6. $C_{24}H_{23}NOS_2$ requires C, 71.1; H, 5.7; N, 3.45%); m.p. 75.5–77 °C (from hexane); $\nu_{max}(CCl_4)/cm^{-1}$ 1665; δ 1.99 (3 H, br s, CMe), 3.20 (3 H, s, NMe), 4.8–5.2 (2 H, m, C=CH$_2$), 4.88 [1 H, s, C*H*(SPh)$_2$] and 6.75–7.4 (14 H, m, ArH).

2-*Chloro*-N-*methyl*-N-[o-(1-*methylethenyl*)*phenyl*]-*acetamide* **6**.—Using a procedure similar to that described for the preparation of compound **2e**, the *acetamide* **6** (1.27 g, 80%) was obtained from the aniline **1e** (1.0 g, 6.80 mmol) and chloroacetyl chloride (845 mg, 7.48 mmol) as an oil (Found: C, 64.3; H, 6.5; N, 6.2. $C_{12}H_{14}ClNO$ requires C, 64.4; H, 6.3; N, 6.3%); $\nu_{max}(CCl_4)/cm^{-1}$ 1680; δ 2.03 (3 H, br s, CMe), 3.22 (3 H, s, NMe), 3.84 (2 H, s, COCH$_2$Cl), 4.95–5.1 (1 H, m, one of C=CH$_2$), 5.15–5.3 (1 H, m, one of C=CH$_2$) and 7.1–7.6 (4 H, m, ArH).

Radical Cyclisation of the Dichloroacetamide **2a**. *General Procedure A*.—A solution of Bu_3SnH (525 mg, 1.80 mmol) and AIBN (27 mg, 0.16 mmol) in dry toluene (40 cm^3) was added dropwise to a boiling solution of the acetamide **2a** (400 mg, 1.63 mmol) in dry toluene (20 cm^3) *via* a syringe during 40 min and the mixture was refluxed for a further 1 h. Then a further solution of Bu_3SnH (525 mg, 1.80 mmol) and AIBN (27 mg, 0.16 mmol) in toluene (5 cm^3) was added to this mixture, and the whole was refluxed for 2 h. After the solvent had been evaporated off, the residue was dissolved in diethyl ether (20 cm^3), 8% aq. KF (20 cm^3) was added, and the mixture was stirred at room temperature for 1 h. The organic layer was separated and the aq. layer was extracted with diethyl ether. The combined organic phases were dried ($MgSO_4$) and concentrated. The residue was chromatographed on silica gel [hexane–AcOEt (9:2)] to give 3,4-dihydro-1,4-dimethylquinolin-2(1*H*)-one **7a** (139 mg, 49%) as an oil[3] (Found: M^+, 175.0993. Calc. for $C_{11}H_{13}NO$: M, 175.0996); $\nu_{max}(CCl_4)/cm^{-1}$ 1680; δ 1.25 (3 H, d, *J* 7 Hz, 4-Me), 2.1–3.4 (3 H, m, 3-H$_2$ and 4-H), 3.33 (3 H, s, NMe) and 6.8–7.4 (4 H, m, ArH).

Radical Cyclisation of the Bis(*phenylthio*)*acetamide* **3**.—Following general procedure A, the bis(phenylthio)acetamide **3** (400 mg, 1.02 mmol) was treated with Bu_3SnH (327 mg, 1.12 mmol) and AIBN (17 mg, 0.1 mmol) in refluxing toluene for 5 h. Since TLC analysis of the reaction mixture showed that considerable amounts of the starting material still remained, a further solution of Bu_3SnH (327 mg) and AIBN (17 mg) in toluene (10 cm^3) was added dropwise during 15 min and the mixture was refluxed for 2 h. Work-up gave the quinolinone **7a** (91 mg, 50%) as an oil.

Radical Cyclisation of the Dichloroacetamide **2b**.—Following general procedure A, the acetamide **2b** (260 mg, 1.00 mmol) was treated with Bu_3SnH (322 mg, 1.1 mmol) and AIBN (16 mg, 0.1 mmol) twice and work-up gave 4-*ethyl*-3,4-*dihydro*-1-*methylquinolin*-2(1H)-*one* **7b** (195 mg, quant.) as an oil (Found: M^+, 189.1160. $C_{12}H_{15}NO$ requires M, 189.1153); $\nu_{max}(CCl_4)/cm^{-1}$ 1680; δ 0.91 (3 H, br t, *J* 7 Hz, CH$_2$*Me*), 1.2–3.0 (5 H, br m, 3-H$_2$, 4-H and C*H*$_2$Me), 3.32 (3 H, s, NMe) and 6.75–7.4 (4 H, m, ArH).

Radical Cyclisation of the Dichloroacetamide **2c**.—Following general procedure A, the acetamide **2c** (180 mg, 0.73 mmol) was treated with Bu_3SnH (236 mg, 0.81 mmol) and AIBN (12 mg, 0.07 mmol) twice and work-up gave 4-*ethyl*-3,4-*dihydroquinolin*-2(1H)-*one* **7c** (102 mg, 79%) (Found: C, 75.3; H, 7.5; N, 8.0. $C_{11}H_{13}NO$ requires C, 75.4; H, 7.5; N, 8.0%); m.p. 130.5–132.5 °C (from hexane–AcOEt); $\nu_{max}(CCl_4)/cm^{-1}$ 1680; δ(300 MHz) 0.94 (3 H, t, *J* 7.4 Hz, CH$_2$*Me*), 1.5–1.75 (2 H, m, C*H*$_2$Me), 2.57 (1 H, dd, *J* 15.5 and 4 Hz, 3-H), 2.77 (1 H, dd, *J* 15.5 and 6.5 Hz, 3-H), 2.85 (1 H, ddd, *J* 13.5, 6.5 and 4 Hz, 4-H), 6.82 (1 H, dd, *J* 8 and 1.5 Hz, ArH), 7.01 (1 H, td, *J* 7.5 and 1.5 Hz, ArH), 7.16 (1 H, br d, *J* 8 Hz, ArH), 7.18 (1 H, td, *J* 7.5 and 1.5 Hz, ArH) and 8.63 (1 H, br, NH).

Radical Cyclisation of the Dichloroacetamide **2d**.—Following general procedure A, the acetamide **2d** (250 mg, 0.86 mmol) was treated with Bu_3SnH (278 mg, 0.95 mmol) and AIBN (14 mg, 0.09 mmol) twice and work-up gave *methyl* (1,2,3,4-*tetrahydro*-2-*oxoquinolin*-4-*yl*)*acetate* **7d** (190 mg, quant.) (Found: C, 65.8; H, 6.0; N, 6.4. $C_{12}H_{13}NO_3$ requires C, 65.7; H, 6.0; N, 6.4%); m.p. 114–115 °C (from hexane–AcOEt); $\nu_{max}(CHCl_3)/cm^{-1}$ 3400, 1730 and 1680; δ 2.3–3.1 (4 H, m, 3-H$_2$ and C*H*$_2$CO$_2$Me), 3.25–3.85 (1 H, m, 4-H), 3.62 (3 H, s, OMe), 6.7–7.3 (4 H, m, ArH) and 9.45–9.9 (1 H, br, NH).

Radical Cyclisation of the Dichloroacetamide **2e**.—Following general procedure A, the acetamide **2e** (350 mg, 1.35 mmol) was treated with Bu_3SnH (434 mg, 1.49 mmol) and AIBN (22 mg, 0.14 mmol) twice to give a mixture of four products, which was chromatographed on silica gel [hexane–AcOEt (7:1)]. The first fraction gave 3,4-dihydro-1,4,4-trimethylquinolin-2(1*H*)-one **9** (61 mg, 24%) as an oil;[3] $\nu_{max}(CCl_4)/cm^{-1}$ 1680; δ 1.29 (6 H, s, 4-Me$_2$), 2.47 (2 H, s, 3-H$_2$), 3.37 (3 H, s, NMe) and 6.9–7.45 (4 H, m, ArH).

The second fraction gave a mixture of 1,3,4,5-tetrahydro-1,4-

dimethyl- **11** and 1,3,4,5-tetrahydro-1,5-dimethyl-2*H*-1-benzazepin-2-one **10** (25 mg, 10%; 1:2) and the third fraction afforded 1,5-*dihydro*-1,4-*dimethy*-2H-1-*benzazepin*-2-*one* **8** (171 mg, 68%) (Found: C, 76.8; H, 7.05; N, 7.8. $C_{12}H_{13}NO$ requires C, 77.0; H, 7.0; N, 7.5%); m.p. 96–97 °C (from hexane); $\nu_{max}(CCl_4)/cm^{-1}$ 1665 and 1630; δ 1.97 (3 H, d, *J* 1.5 Hz, 4-Me), 3.23 (2 H, br s, 5-H_2), 3.49 (3 H, s, NMe), 5.6–5.7 (1 H, m, 3-H) and 7.1–7.3 (4 H, m, ArH); δ(75 MHz) 167.5, 152.9, 141.8, 135.5, 127.4, 127.2, 125.2, 121.7, 120.0, 38.0, 36.7 and 24.4. Compounds **10**[6] and **11** were identified by comparison of the ^1H NMR spectrum of the mixture and the GLC retention time with those of each authentic sample (for the preparation of authentic compound **11**, see below).

Catalytic Reduction of the Benzazepin-2-one **8**.—A solution of compound **8** (50 mg, 0.26 mmol) in methanol (10 cm^3) was hydrogenated in the presence of 10% Pd-C (50 mg) under pressure (4 kg/cm^2) for 40 h. After the catalyst had been removed by filtration, the filtrate was concentrated. The residue was chromatographed on silica gel [hexane–AcOEt (7:1)] to give the unchanged starting material **8** (6 mg recovery) and 1,3,4,5-*tetrahydro*-1,4-*dimethyl*-2H-1-*benzazepin*-2-*one* **11** (35 mg, 69%) as an oil (Found: M^+, 189.1158. $C_{12}H_{15}NO$ requires M, 189.1153); $\nu_{max}(CCl_4)/cm^{-1}$ 1665; δ 1.04 (3 H, d, *J* 6 Hz, 4-Me), 1.7–3.1 (5 H, m, 3-H_2, 4-H and 5-H_2), 3.34 (3 H, s, NMe) and 7.0–7.5 (4 H, m, ArH).

Radical Cyclisation of the Dichloroacetamide **2f**.—Following general procedure A, the acetamide **2f** (400 mg, 1.63 mmol) was treated with Bu_3SnH (525 mg, 1.80 mmol) and AIBN (27 mg, 0.16 mmol) twice to give 1,5-*dihydro*-4-*methyl*-2H-1-*benzazepin*-2-*one* **12** (124 mg, 44%) (Found: C, 76.3; H, 6.4; N, 8.1. $C_{11}H_{11}NO$ requires C, 76.3; H, 6.4; N, 8.1%); m.p. 158.5–160.5 °C (from hexane–AcOEt); $\nu_{max}(CCl_4)/cm^{-1}$ 1675; δ 2.01 (3 H, d, *J* 1 Hz, 4-Me), 3.28 (2 H, s, 5-H_2), 5.6–5.8 (1 H, m, 3-H), 6.85–7.35 (4 H, m, ArH) and 8.9–9.3 (1 H, m, NH). Other minor products were not characterised.

Radical Cyclisation of the 2-*Chloro*-2-(*methylthio*)*acetamide* **4**. *General Procedure B*.—A solution of Bu_3SnH (407 mg, 1.40 mmol) and AIBN (21 mg, 0.13 mmol) in dry toluene (40 cm^3) was added dropwise to a boiling solution of the acetamide **4** (345 mg, 1.27 mmol) in toluene (20 cm^3) during 1 h and the mixture was then refluxed for 9 h. After work-up as described in procedure A, the crude material was chromatographed on silica gel [hexane–AcOEt (15:1)]. The first fraction gave 3,4-*dihydro*-1,4,4-*trimethyl*-3-*methylthioquinolin*-2(1H)-*one* **13** (81 mg, 27%) (Found: C, 66.1; H, 7.3; N, 6.1. $C_{13}H_{17}NOS$ requires C, 66.35; H, 7.3; N, 5.95%); m.p. 77.5–79 °C (from hexane); $\nu_{max}(CCl_4)/cm^{-1}$ 1670; δ 1.25 and 1.53 (3 H each, both s, 4-Me_2), 2.13 (3 H, s, SMe), 3.19 (1 H, s, 3-H), 3.38 (3 H, s, NMe) and 6.8–7.4 (4 H, m, ArH).

The second fraction gave the 1-benzazepinone **8** (113 mg, 48%).

Radical Cyclisation of the Bis(*phenylthio*)*acetamide* **5**.—Following procedure B, the acetamide **5** (400 mg, 0.99 mmol) was treated with Bu_3SnH (316 mg, 1.08 mmol) and AIBN (16 mg, 0.1 mmol) to give the 1-benzazepin-2-one **8** (83 mg, 45%). Other minor products were not characterised.

Radical Cyclisation of the Chloroacetamide **6**.—Following procedure B, the acetamide **6** (400 mg, 1.71 mmol) was treated with Bu_3SnH (797 mg, 2.74 mmol) and AIBN (42 mg, 0.26 mmol) and work-up gave the quinolinone **9** (60 mg, 19%) and a mixture of the benzazepinones **10** and **11** (1:1; 78 mg, 24%).

Radical Cyclisation of the Dichloroacetamide **2g**.—Following procedure A, the acetamide **2g** (400 mg, 1.25 mmol) was treated with Bu_3SnH (400 mg, 1.38 mmol) and AIBN (21 mg, 0.1 mmol) twice and work-up gave 1,3,4,5-tetrahydro-1-methyl-5-phenyl-2*H*-1-benzazepin-2-one **14a** (275 mg, 88%), m.p. 102–103.5 °C (from hexane) (lit.,[8] 102–104 °C); $\nu_{max}(CCl_4)/cm^{-1}$ 1665; δ 2.3–2.6 (4 H, m, 3- and 4-H_2), 3.28 (3 H, s, NMe), 4.0–4.45 (1 H, m, 5-H) and 6.55–7.7 (9 H, m, ArH).

Radical Cyclisation of the Dichloroacetamide **2h**.—Following procedure A, the acetamide **2h** (350 mg, 1.14 mmol) was treated with Bu_3SnH (366 mg, 1.25 mmol) and AIBN (18 mg, 0.11 mmol) twice. After work-up, the crude material was chromatographed on silica gel [hexane–AcOEt (7:1)]. The first fraction gave *o*-(1-phenylethenyl)acetanilide **15** (88 mg, 33%), m.p. 123–125 °C [from acetonitrile–light petroleum (b.p. range 80–110 °C)] (lit.,[16] 122 °C); $\nu_{max}(CHCl_3)/cm^{-1}$ 3410 (NH) and 1690; δ 1.75 (3 H, s, COMe), 5.30 (1 H, d, *J* 2 Hz, one of C=CH_2), 5.79 (1 H, d, *J* 2 Hz, one of C=CH_2), 6.75–7.7 (9 H, m, ArH) and 7.9–8.2 (1 H, m, NH).

The second fraction gave 1,3,4,5-tetrahydro-5-phenyl-2*H*-1-benzazepin-2-one **14b** (115 mg, 43%), m.p. 183–184 °C (from acetonitrile–hexane) (lit.,[9] 180–182 °C); $\nu_{max}(CHCl_3)/cm^{-1}$ 3390 (NH) and 1670; δ 2.1–2.8 (4 H, m, 3- and 4-H_2), 3.95–4.6 (1 H, m, 5-H), 6.6–7.6 (9 H, m, ArH) and 8.1–8.35 (1 H, br, NH).

2,2-*Dichloro*-N-*methyl*-N-[o-(*prop*-2-*enyl*)*phenyl*]*acetamide* **17a**.—To a solution of the aniline **16** (2.0 g, 13.6 mmol) in dichloromethane (20 cm^3) was added dichloroacetic acid (1.93 g, 14.9 mmol) and then a solution of dicyclohexylcarbodiimide (DCC) (3.07 g, 14.9 mmol) in dichloromethane (20 cm^3) at room temperature, and the mixture was stirred at the same temperature for 16 h. The precipitated dicyclohexylurea was filtered off and the filtrate was washed with saturated aq. $NaHCO_3$, dried (Na_2SO_4) and concentrated. The residue was chromatographed on silica gel [hexane–AcOEt (20:1)] to give the *amide* **17a** (1.43 g, 41%) as an oil (Found: C, 55.5; H, 5.1; N, 5.55. $C_{12}H_{13}Cl_2NO$ requires C, 55.8; H, 5.1; N, 5.4%); $\nu_{max}(CCl_4)/cm^{-1}$ 1695; δ 3.2–3.55 (2 H, m, CH_2CH=CH_2), 3.25 (3 H, s, NMe), 4.85–5.35 (2 H, m, CH=CH_2), 5.55–6.3 (1 H, m, CH=CH_2), 5.72 (1 H, s, $CHCl_2$) and 7.1–7.5 (4 H, m, ArH).

N-*Methyl*-2,2-*bis*(*phenylthio*)-N-[o-(*prop*-2-*enyl*)*phenyl*]-*acetamide* **17b**.—Using a procedure similar to that described for the preparation of compound **17a**, the aniline **16** (723 mg, 4.91 mmol) was treated with bis(phenylthio)acetic acid (1.36 g, 4.91 mmol) and DCC (1.12 g, 5.40 mmol) in dichloromethane (5 cm^3) at room temperature overnight, and work-up gave the *amide* **17b** (572 mg, 29%) as an oil (Found: C, 70.8; H, 5.9; N, 3.4. $C_{24}H_{23}NOS_2$ requires C, 71.1; H, 5.7; N, 3.45%); $\nu_{max}(CCl_4)/cm^{-1}$ 1665; δ 3.0–3.3 (2 H, m, CH_2CH=CH_2), 3.17 (3 H, s, NMe), 4.7–5.2 (2 H, m, CH=CH_2), 4.71 [1 H, s, CH(SPh)$_2$], 5.55–6.2 (1 H, m, CH=CH_2) and 6.6–7.6 (14 H, m, ArH).

2-*Chloro*-N-*methyl*-N-[o-(*prop*-2-*enyl*)*phenyl*]*acetamide* **17c**.—Using a procedure similar to that described for the preparation of compound **17a**, the aniline **16** (1.83 g, 12.4 mmol) was treated with chloroacetic acid (1.29 g, 13.7 mmol) and DCC (2.82 g, 13.7 mmol) in dichloromethane (40 cm^3), and work-up gave the *amide* **17c** (1.32 g, 48%) as an oil (Found: C, 64.6; H, 6.8; N, 6.6. $C_{12}H_{14}ClNO$ requires C, 64.4; H, 6.3; N, 6.3%); $\nu_{max}(CCl_4)/cm^{-1}$ 1675; δ 3.2–3.5 (2 H, m, CH_2CH=CH_2), 3.22 (3 H, s, NMe), 3.71 (2 H, s, $COCH_2$), 4.8–5.3 (2 H, m, CH=CH_2), 5.6–6.3 (1 H, m, CH=CH_2) and 7.0–7.5 (4 H, m, ArH).

2-*Chloro*-N-*methyl*-N-[o-(*prop*-2-*enyl*)*phenyl*]*propionamide* **17d**.—To a solution of the aniline **16** (500 mg, 3.40 mmol) pyridine (857 mg, 10.8 mmol), and 4-(dimethylamino)pyridine (DMAP) (42 mg, 0.34 mmol) in toluene (20 cm^3) at 0 °C was

added a solution of 2-chloropropionyl chloride (474 mg, 3.74 mmol) in toluene (5 cm^3) and the mixture was stirred at 0 °C for 30 min. The organic layer was washed with saturated aq. $NaHCO_3$ (20 cm^3), dried (Na_2SO_4) and concentrated. The residue was chromatographed on silica gel [hexane–AcOEt (9:2)] to give the *amide* **17d** (324 mg, 40%) as an oil (Found: M^+, 237.0947. $C_{13}H_{16}ClNO$ requires M, 237.0920); $\nu_{max}(CCl_4)$/cm^{-1} 1675; δ 1.56 (3 H, d, *J* 7.0 Hz, CMe), 3.1–3.5 (2 H, m, $CH_2CH{=}CH_2$), 3.23 (3 H, s, NMe), 4.02 and 4.24 (total 1 H, both q, *J* 7.0 Hz, COCH), 4.8–5.3 (2 H, m, $CH{=}CH_2$), 5.5–6.4 (1 H, m, $CH{=}CH_2$) and 6.9–7.5 (4 H, m, ArH).

2-*Chloro*-N-*methyl*-2-*phenyl*-N-[o-(*prop*-2-*enyl*)*phenyl*]-*acetamide* **17e**.—Using a procedure similar to that described for the preparation of compound **17d**, the aniline **16** (600 mg, 4.08 mmol) was treated with 2-chloro-2-phenylacetyl chloride (847 mg, 4.48 mmol), pyridine (1.03 g, 13.0 mmol) and DMAP (50 mg, 0.41 mmol) in toluene (30 cm^3), and work-up gave the *amide* **17e** (492 mg, 40%) (Found: C, 72.2; H, 6.4; N, 5.0. $C_{18}H_{18}ClNO$ requires C, 72.1; H, 6.05; N, 4.7%); m.p. 46.5–48.5 °C (from hexane–AcOEt); $\nu_{max}(CCl_4)$/cm^{-1} 1680; δ 2.55–2.85 and 3.3–3.55 (total 2 H, both m, $CH_2CH{=}CH_2$), 3.19 (3 H, s, NMe), 4.6–6.3 (3 H, m, $CH{=}CH_2$), 5.09 and 5.21 (total 1 H, both s, COCH) and 6.5–7.5 (9 H, m, ArH).

Radical Cyclisation of the Dichloroacetamide **17a**.—Following procedure A, the acetamide **17a** (410 mg, 1.59 mmol) was treated with Bu_3SnH (508 mg, 1.75 mmol) and AIBN (26 mg, 0.18 mmol) twice and work-up gave the 2*H*-1-benzazepin-2-one **11** (131 mg, 48%) as an oil, along with two unidentified, oily products (67 mg and 10 mg).

Radical Cyclisation of the Bis(*phenylthio*)*acetamide* **17b**.—Following procedure B, the acetamide **17b** (316 mg, 0.78 mmol) was treated with Bu_3SnH (249 mg, 0.857 mmol) and AIBN (13 mg, 0.08 mmol). After work-up, the crude material was chromatographed on silica gel [hexane–AcOEt (10:1)]. The first fraction gave the unchanged starting material **17b** (80 mg, 25% recovery). The second fraction gave N-*methyl*-2-*phenylthio*-N-[o-(*prop*-2-*enyl*)*phenyl*]*acetamide* **17f** (22 mg, 9%) (Found: C, 72.4; H, 6.6; N, 4.6. $C_{18}H_{19}NOS$ requires C, 72.7; H, 6.4; N, 4.7%); m.p. 43–45 °C (from hexane–AcOEt); $\nu_{max}(CCl_4)$/cm^{-1} 1665; δ 3.19 (3 H, s, NMe), 3.2–3.6 (2 H, m, $CH_2CH{=}CH_2$), 3.39 (2 H, s, CH_2SPh), 4.8–5.3 (2 H, m, $CH{=}CH_2$), 5.5–6.3 (1 H, m, $CH{=}CH_2$) and 6.8–7.8 (9 H, m, ArH).

The third fraction gave 3,4,5,6-*tetrahydro*-1-*methyl*-3-*phenylthio*-1-*benzazocin*-2(1H)-*one* **21** (102 mg, 47%) (Found: C, 72.7; H, 6.6; N, 4.8. $C_{18}H_{19}NOS$ requires C, 72.7; H, 6.4; N, 4.7%); m.p. 127.5–129.5 °C (from hexane–AcOEt); $\nu_{max}(CCl_4)$/cm^{-1} 1665; δ 1.9–3.0 (6 H, m, 4-, 5- and 6-H_2), 3.27 (3 H, s, NMe), 3.79 (1 H, dd, *J* 9 and 4 Hz, 3-H) and 7.0–7.4 (9 H, m, ArH).

Desulphurisation of Compound **21**.—A suspension of the sulphide **21** (49 mg, 0.16 mmol) and Raney nickel (*ca.* 1 g) in ethanol (5 cm^3) was refluxed for 2 h. After the catalyst had been removed by filtration, the filtrate was concentrated and the residue was chromatographed on silica gel [hexane–AcOEt (5:1)] to give 3,4,5,6-tetrahydro-1-methyl-1-benzazocin-2(1*H*)-one **20** (27 mg, 89%), m.p. 58.5–59.5 °C (from light petroleum) (lit.,[10] 60–61.5 °C); $\nu_{max}(CHCl_3)$/cm^{-1} 1630; δ 1.1–3.0 (8 H, br m), 3.30 (3 H, s, NMe) and 7.0–7.4 (4 H, m, ArH).

Radical Cyclisation of the Chloroacetamide **17c**.—Following procedure B, the acetamide **17c** (300 mg, 1.34 mmol) was treated with Bu_3SnH (429 mg, 1.47 mmol) and AIBN (22 mg, 0.13 mmol) and the crude material was chromatographed on silica gel [hexane–AcOEt (9:2)]. The first fraction gave an unidentified, oily product (15 mg), the second fraction gave the 2*H*-1-benzazepinone **11** (96 mg, 38%) and the third fraction afforded the 1-benzazocin-2(1*H*)-one **20** (112 mg, 44%).

Radical Cyclisation of the Chloropropionamide **17d**.—Following procedure B, the acetamide **17d** (250 mg, 1.05 mmol) was treated with Bu_3SnH (337 mg, 1.16 mmol) and AIBN (17 mg, 0.11 mmol). After work-up, the crude material was chromatographed on silica gel [hexane–AcOEt (15:1)]. The first fraction gave 1,3,4,5-*tetrahydro*-1,3,4-*trimethyl*-2H-1-*benzazepin*-2-*one* **18** (123 mg, 58% as a stereoisomeric mixture in the ratio 68:32, determined by 300 MHz ^1H NMR spectroscopy) as an oil (Found: M^+, 203.1298. $C_{13}H_{17}NO$ requires M, 203.1308); $\nu_{max}(CCl_4)$/cm^{-1} 1665; δ 0.9–1.2 (6 H, m), 1.7–3.2 (4 H, m), 3.33 (3 H, s, NMe) and 6.9–7.4 (4 H, m, ArH).

The second fraction gave 3,4,5,6-*tetrahydro*-1,3-*dimethyl*-1-*benzazocin*-2(1H)-*one* **22** (78 mg, 36%) as an oil (Found: M^+, 203.1288. $C_{13}H_{17}NO$ requires M, 203.1308); $\nu_{max}(CCl_4)$/cm^{-1} 1665; δ 1.04 (3 H, d, *J* 6 Hz, 3-Me), 1.4–2.9 (7 H, m), 3.27 (3 H, s, NMe) and 7.0–7.4 (4 H, m, ArH).

Radical Cyclisation of the Chloro(*phenyl*)*acetamide* **17e**.—Following procedure B, the acetamide **17e** (350 mg, 1.16 mmol) was treated with Bu_3SnH (374 mg, 1.28 mmol) and AIBN (19 mg, 0.12 mmol). After work-up, the crude material was chromatographed on silica gel [hexane–AcOEt (10:1)]. The first fraction gave one of the diastereoisomeric isomers of 1,3,4,5-*tetrahydro*-1,4-*dimethyl*-3-*phenyl*-2H-1-*benzazepin*-2-*one* **19** (48 mg, 16%) (Found: C, 81.5; H, 7.55; N, 5.5. $C_{18}H_{19}NO$ requires C, 81.5; H, 7.2; N, 5.3%); m.p. 121–122.5 °C (from hexane); $\nu_{max}(CCl_4)$/cm^{-1} 1670; δ 1.05 (3 H, d, *J* 6 Hz, 4-Me), 2.1–3.1 (3 H, m), 3.38 (3 H, s, NMe), 3.75–3.9 (1 H, m) and 6.9–7.6 (9 H, m, ArH).

The second fraction gave *another isomer of compound* **19** (21 mg, 7%) as an oil (Found: M^+, 265.1440. $C_{18}H_{19}NO$ requires M, 265.1465); $\nu_{max}(CCl_4)$/cm^{-1} 1670; δ 0.76 (3 H, d, *J* 6 Hz, 4-Me), 2.3–3.5 (4 H, m), 3.36 (3 H, s, NMe) and 6.9–7.5 (9 H, m, ArH).

The third fraction gave 3,4,5,6-*tetrahydro*-1-*methyl*-3-*phenyl*-1-*benzazocin*-2(1H)-*one* **23** (196 mg, 63%) (Found: C, 81.3; H, 7.6; N, 5.6. $C_{18}H_{19}NO$ requires C, 81.5; H, 7.2; N, 5.3%); m.p. 119–120 °C (from hexane); $\nu_{max}(CCl_4)$/cm^{-1} 1665; δ 1.1–3.7 (7 H, m), 3.27 (3 H, s, NMe) and 6.8–7.5 (9 H, m, ArH).

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