

Reaction between 2-Phenylaziridines and 3-Halogenocyclohex-2-enones

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The reaction of 2-phenyl- or 2-(3,4-methylenedioxyphenyl)aziridine with 3-chlorocyclohex-2-enone give principally 3-(2-hydroxy-2-phenylethylamino)cyclohex-2-enone or 3-[2-hydroxy-2-(3,4-methylenedioxyphenyl)-ethylamino]cyclohex-2-enone, respectively, instead of the expected aziridines, in each case *via* the 2-chloro-derivative. 2-Benzyl-3-phenylaziridine and 2-chlorocyclohex-2-enone combine to form the expected 3-aziridinylcyclohex-2-enone, which does not easily undergo ring expansion with iodide ion.

THE reaction of 2-phenylaziridine (Ia) with 3-chlorocyclohex-2-enone (IIa) was expected to give the 3-aziridinylcyclohex-2-enone (IIIa), which would undergo ring-expansion with iodide ion (Scheme). Earlier work by Whitlock¹ had shown that aziridine (Ib) reacted with 3-chloro-2-(3,4-methylenedioxyphenyl)cyclohex-2-enone (IIb) in the presence of triethylamine as non-nucleophilic base to give the 3-aziridinylcyclohex-2-enone (IIIb), which was rearranged by iodide ion (*cf.* Scheme).

In the event, the product from the reaction of (Ia) and (IIa) consisted of a complex mixture from which two major components were isolated (t.l.c.). Both were $\alpha\beta$ -unsaturated ketones (u.v. and i.r. data), but showed i.r. absorption at *ca.* 3400 cm^{-1} and had exchangeable protons (¹H n.m.r.). The *N*-methylene protons were

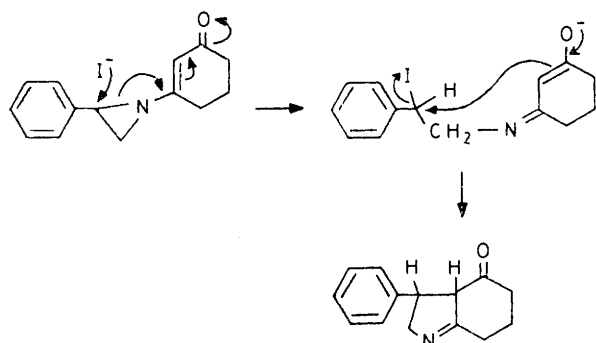
magnetically equivalent (unlike those of substituted aziridines). Analysis showed the two compounds to contain the elements of HCl and H₂O, respectively, in addition to the formula expected for (IIIa); they were identified as the chloro- and hydroxy-derivatives (IVa and b).

The formation of the open-chain chloro-compound (IVa) strongly implied that the initially formed aziridine (IIIa) was attacked by chloride ion at the benzylic position; subsequent hydrolysis would then give (IVb). It was shown that even t.l.c. partially converted (IVa) into (IVb), and that hydrolysis with hydroxide ion in ethanol quantitatively achieved the same conversion.

¹ H. W. Whitlock and G. L. Smith, *J. Amer. Chem. Soc.*, 1967, **89**, 3600.

The possibility that ring opening had occurred at the non-benzylic position of the aziridine (IIIa) to give the primary chloride (V) was eliminated by the ^1H n.m.r. spectrum of (IVa) in $(\text{CD}_3)_2\text{SO}$, in which the methylene signal, a double doublet (J 6.5 and 6.0 Hz) collapsed to a doublet (J 6.5 Hz) on deuteration. Clearly the vinyllogous amido-NH of structure (V) would exhibit coupling with only one proton. Similar conclusions about structure could be drawn from the ^1H n.m.r. spectra of the hydroxy-enone (IVb) in various solvents.

Rigorous examination of the products of the reaction



SCHEME

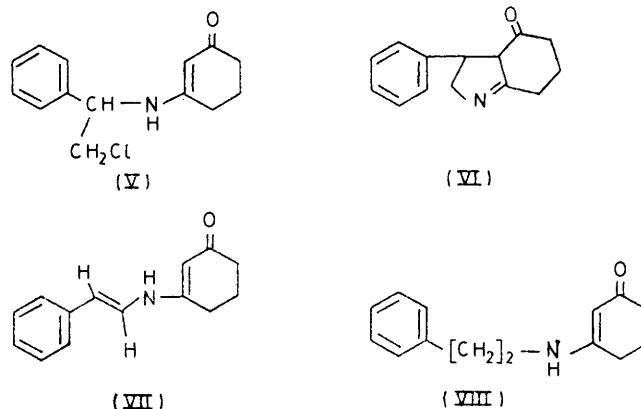
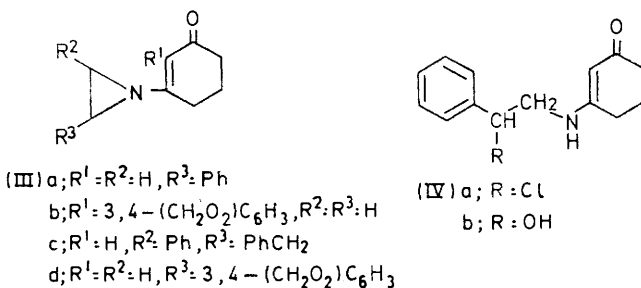
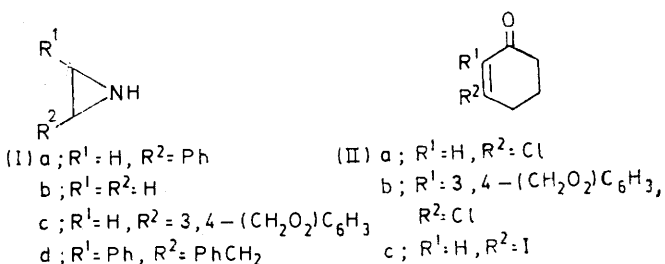
showed that no *N*-substituted aziridine (IIIa) was present nor was there any evidence to suggest that ring expansion had occurred to give (VI) or its tautomer (as in Scheme). In fact, only iodide¹⁻³ ion or azide ion⁴ is known to affect such a ring expansion.

Since no *N*-substituted aziridine (IIIa) was isolated, presumably because the 2-phenyl group makes the aziridine even more susceptible to nucleophilic attack, the next logical step was to study the reaction of the aziridine (Ia) with 3-iodocyclohex-2-enone (IIc). It was anticipated that initial formation of (IIIa) would be followed by iodide-catalysed ring-opening and subsequent closure (as in Scheme) to give the pyrroline (VI).

The iodo-ketone (IIc) was synthesised by a Finkelstein⁵ reaction with the chloro-ketone (IIa). The concentrations of halogeno-ketones in the mixture could easily be determined from the ^1H n.m.r. spectrum; the olefinic protons resonated at δ 6.83 (IIc) and 6.26 (IIa). Bulb-tube distillation at low pressure allowed pure iodo-ketone to be obtained on a small scale, but extensive decomposition occurred and it was decided that, for the purposes of reaction with (Ia), a mixture of the two would be used.

Accordingly, a mixture of iodo- and chloro-ketones (IIc and a) (4 : 1) was stirred with 2-phenylaziridine (Ia) and triethylamine. Only one product was isolated, an unstable yellow compound showing λ_{max} 349, 289, and 254 nm, and having ^1H n.m.r. absorptions corresponding to three olefinic protons, one a singlet and the other two mutually coupled doublets, J 15.0 Hz. On the basis of this and other evidence (see Experimental section) the

product was assigned the styrene structure (VII). Hydrogenation over palladised charcoal gave the dihydro-derivative (VIII), whose structure was confirmed by comparison with the product of unambiguous synthesis from 2-phenylethylamine and the chloro-ketone (IIa). In common with several other compounds encountered in this work, the styrene (VII) was unstable at room temperature, and subsequent attempts to isolate it were unsuccessful. As a photolysis of (VII) to give the required ring system seemed an attractive proposition, attempts were made to prepare (VII) by elimination from



(IVa) and (IVb) (with *t*-butoxide and toluene-*p*-sulphonic acid, respectively). These failed, as did a Finkelstein reaction with (IVa).

Concurrently with the above work, 2-(3,4-methylenedioxyphenyl)aziridine (Ic) had been made by a highly effective recent modification⁶ of the normal reduction of the corresponding ketoxime with lithium aluminium hydride. When the aziridine (Ic) reacted with chloro-ketone (IIa) and triethylamine, the products

² H. W. Heine, *J. Amer. Chem. Soc.*, 1963, **85**, 2743.

³ H. W. Heine, *Angew. Chem. Internat. Edn.*, 1962, **1**, 528.

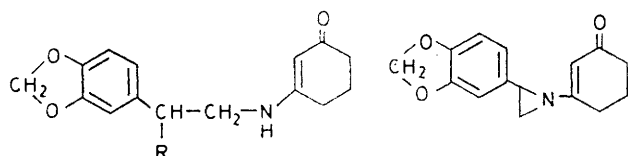
⁴ R. D. Guthrie and D. Murphy, *J. Chem. Soc.*, 1965, 3828.

⁵ A. H. Ford-Moore, *Org. Synth.*, Coll. Vol. 4, 1963, p. 84.

⁶ H. Tanida, T. Okada, and K. Kotera, *Bull. Chem. Soc. Japan*, 1973, **46**, 934.

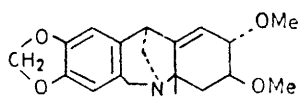
were the ethoxy-, methoxy-, and hydroxy-derivatives (IXa—c). By varying the isolation procedure (t.l.c. with methylene chloride alone) it was possible to show that (IXa) and (IXb) were artefacts from the solvents used in chromatography; with dichloromethane (IXc) was the sole product. As in the case of the earlier reaction of (Ia) with (IIa), no aziridine was isolated. The expected chloro-enone (IXd) could not be isolated either, suggesting that the methylenedioxy-group had further activated the benzylic position of the transient aziridine (X).

These disappointing observations meant that our primary objective, synthesis of the alkaloid manthine⁷ (XI) [for which the key step was to be the iodide-catalysed ring expansion of the aziridine (IIIc)], could not be achieved in this way. Nonetheless, we decided to test the generality of the iodide-catalysed rearrangement (Scheme) by making 3-(2-benzyl-3-phenylaziridinyl)-cyclohex-2-enone (IIIc) from *cis*-2-benzyl-3-phenylaziridine (Id) and the chloro-ketone (IIa). The aziridine

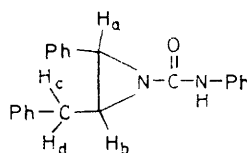


(IX) a; R: OEt
b; R: OMe
c; R: OH
d; R: Cl

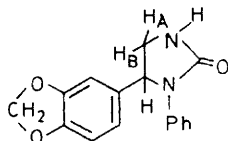
(X)



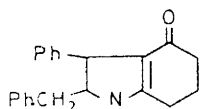
(XI)



(XII)



(XIII)



(XIV)

(Id) was chosen partly because we had found it to be⁸ much the most stable of the aziridines encountered in the present work, but principally because examination⁹ of the n.m.r. spectrum of the phenyl isocyanate derivative of (Id) strongly supported structure (XIII), with the aziridine ring intact. By contrast, the corresponding derivative of (Ic) was assigned the ring-expanded structure (XIII) on the basis of (i) the low chemical shift observed for the two magnetically non-equivalent *N*-methylene protons [δ 3.91 and 4.45, respectively; those

of the parent aziridine (Ic) resonate at δ 1.69 and 2.12] and (ii) the observed coupling constants (J_{gem} for aziridines is rarely above ± 3 Hz; the observed value is -11 Hz). No mechanism for the formation of (XIII) is suggested, since it is difficult to envisage a suitable mechanism which would also account for the failure of (XII) to undergo this rearrangement.

In the event, the reaction of the aziridine (Id) and the chloro-ketone (IIa) gave the expected aziridinylone (IIIc) in good yield, identified on the basis of aziridine-like characteristics of the ¹H n.m.r. spectrum (*cf.* the spectra in ref. 9), and the absence of a NH group (i.r. and ¹H n.m.r.). Iodide-catalysed rearrangement of (IIIc) in diethyl ketone gave only a small quantity of a very unstable compound having no olefinic protons (¹H n.m.r.). The compound appeared to be an $\alpha\beta$ -unsaturated ketone (u.v. and i.r. data) and had an exchangeable proton. The ¹H n.m.r. spectrum was consistent with structure (XIV), but further evidence is desirable.

The difficulties encountered in the present work lead us to believe that the rearrangement of the Scheme may only be applicable to aziridines which do not possess a 2-phenyl group.

EXPERIMENTAL

T.l.c. refers to separations on silica gel plates or columns, according to scale. Spectra were run in ethanol (u.v.), as liquid film (i.r.), and as solutions in CDCl₃ (60 MHz ¹H n.m.r.) unless otherwise stated.

3-(2-Chloro-2-phenylethylamino)cyclohex-2-enone (IVa) and its Hydroxy-analogue (IVb).—2-Phenylaziridine¹⁰ (1.8 g), triethylamine (3.0 ml), and tetrahydrofuran (50 ml) were stirred under nitrogen during addition of a solution of 3-chlorocyclohex-2-enone¹¹ (2.0 ml) in tetrahydrofuran (50 ml). After 4 days, the triethylamine hydrochloride was filtered off and the solution was evaporated to dryness. T.l.c. (5% methanol in chloroform) gave (i) the *enone* (IVa) (0.98 g) as an oil (Found: M^+ , 249.092036 and 251.089085. C₁₄H₁₇³⁵ClNO requires M , 249.091763. C₁₄H₁₇³⁷ClNO requires M , 251.088911), λ_{max} 288 nm ($\log \epsilon$ 4.328), ν_{max} 3420 and 1585 cm⁻¹; δ 7.50—7.20 (5 H, m, ArH), 5.60 (1 H, s, NH), 5.33 (1 H, s, olefinic H), 5.25—5.10 (1 H, m, CHCl), 3.61 (2 H, dd, J 6.0 and 6.5 Hz, CH₂N), and 2.55—1.70 (6 H, m, methylene); δ [(CD₃)₂SO] 3.61 (2 H, dd, J 6.0 and 6.5 Hz, collapsing to 2 H, d, J 6.5 Hz on addition of D₂O) and 3.40br (1 H, s, NH, exchangeable with D₂O), m/e 251 (12%), 249 (30), 213 (9), 156 (7), 138 (20), 134 (20), 124 (100), and 121 (50); and (ii) the *enone* (IVb) (0.5 g), white prisms, m.p. 175—176° (from chloroform) (Found: C, 72.5; H, 7.25; N, 6.1%; M^+ , 231.125921. C₁₄H₁₇NO₂ requires C, 72.8; H, 7.4; N, 6.05%; M , 231.125562); λ_{max} 290 nm ($\log \epsilon$ 4.389); ν_{max} (Nujol) 3320br, 3180, and 1585 cm⁻¹; δ [(CD₃)₂SO] 7.50—7.20 (5 H, m, ArH), 7.00br (1 H, t, NH, exchangeable with D₂O), 5.57 (1 H, d, J 5.0 Hz, OH, exchangeable with D₂O), 4.90 (1 H, s, olefinic H), 4.72 (1 H, t, J 5.5 Hz, CH·O), 3.12 (2 H, dd, J 5.0 and 5.5 Hz, CH₂N, collapsing to 2 H, d, J 5.5 Hz on addition of D₂O), and 2.40—1.50 (6 H, m, methylene), m/e 231 (18%), 213 (10), 156 (10), 149 (8), 124 (100), 123 (100), and 121 (60).

⁹ K. Kotera, Y. Matsukawa, H. Takahashi, T. Okada, and K. Kitahonoki, *Tetrahedron*, 1968, **24**, 6177.

¹⁰ A. Hassner and C. Heathcock, *Tetrahedron*, 1964, **20**, 1039.

¹¹ L. Bateman and F. W. Shipley, *J. Chem. Soc.*, 1955, 1996.

⁷ Y. Inubushi, H. M. Fales, E. W. Warnhoff, and W. C. Wildman, *J. Org. Chem.*, 1960, **25**, 2153.

⁸ B. W. Singer, Ph.D. Thesis, Leeds.

3-Iodocyclohex-2-enone (IIc).¹²—3-Chlorocyclohex-2-enone (1.5 g), sodium iodide (5.0 g), and acetone (250 ml) were refluxed for 24 h. Evaporation and extraction of the residue into dichloromethane gave a solution which was shown to contain two components (4:1). Careful distillation (bulb-tube) of a small quantity gave the *iodo-enone* as a pale yellow oil, b.p. 110—120° at 0.1 τ (oven temperature; much decomposition) (Found: M^+ , 221.954347. C_6H_9IO requires M , 221.954408); δ 6.83 (1 H, m, olefinic H) and 3.05—1.75 (6 H, m, methylene); m/e 222 (100%), 194 (20), 132 (10), 130 (16), 128 (15), 127 (10), 112 (20), 104 (18), 102 (28), 95 (90), 67 (65), and 55 (36). This preparation was difficult to reproduce; the addition of a catalytic amount of toluene-*p*-sulphonic acid is recommended.

3-(Styrylamino)cyclohex-2-enone (VII).—2-Phenylaziridine (0.34 g), triethylamine (2 ml), tetrahydrofuran (50 ml), and a mixture of 3-chloro- and 3-iodo-cyclohex-2-enones (0.65 g) (1:4) were stirred under nitrogen for 4 days. Evaporation followed by trituration with ether left a gum which was dissolved in dichloromethane and separated (t.l.c.) to give the styrene (0.28 g) as a deep yellow glass which decomposed on attempted crystallisation or sublimation; λ_{max} 254, 289, and 349 nm ($\log \epsilon$ 3.623, 3.760, and 4.013); ν_{max} (CHCl₃) 3 260 and 1 590 cm^{-1} ; δ 8.0br (1 H, s, NH, exchangeable with D₂O), 7.51—7.00 (5 H, m, ArH), 7.04 (1 H, d, J 15 Hz, olefinic H), 6.21 (1 H, d, J 15 Hz, olefinic H), 5.53 (1 H, s, olefinic H), and 2.60—1.65 (6 H, m, methylene), m/e 213 (10%), 152 (20), 188 (20), 186 (50), 184 (15), 180 (25), 172 (70), 159 (22), 152 (70), 138 (100), 123 (80), and 119 (100). Hydrogenation of the styrene (VII) over 10% palladium-charcoal gave 3-(2-phenylethylamino)cyclohex-2-enone, identical with authentic material (see below).

3-(2-Phenylethylamino)cyclohex-2-enone (VIII).—Freshly distilled 3-chlorocyclohex-2-enone (1.0 g), triethylamine (2 g), 2-phenylethylamine (0.98 g), and tetrahydrofuran (50 ml) were stirred under nitrogen for 3 days. Work-up in the usual way gave the *enone* (0.2 g) as an oil, b.p. 180—183° at 0.05 Torr (Found: M^+ , 215.13043. $C_{14}H_{17}NO$ requires M , 215.131007); λ_{max} 289 nm ($\log \epsilon$ 4.305); ν_{max} 3 440, 1 620, and 1 600 cm^{-1} ; δ 7.45—7.05 (5 H, m, ArH), 5.25br (1 H, s, NH, exchangeable with D₂O), 5.19 (1 H, s, olefinic H), 3.31 (2 H, t, J 7.0 Hz, CH₂-N), 2.86 (2 H, t, J 7.0 Hz, ArCH₂), and 2.45—1.70 (6 H, m, methylene), m/e 215 (40%), 149 (60), 124 (100), 122 (42), 118 (15), 105 (80), 104 (100), 91 (50), 84 (30), and 77 (60).

2-(3,4-Methylenedioxyphenyl)aziridine (Ic).—A solution of the oxime (m.p. 157—159°; lit.¹³ 156—157°) of 3',4'-methylenedioxyacetophenone (1 g) in tetrahydrofuran (20 ml) was slowly added to a solution of lithium aluminium hydride (1 g) in tetrahydrofuran (25 ml) and *N*-methylbutylamine (2.25 g). The mixture was refluxed for 1 h, and the excess of hydride was destroyed with ice-water. The organic layer was extracted into dichloromethane; the extract was dried (K₂CO₃) and evaporated to give the aziridine (0.88 g) as a pale yellow oil which decomposed on attempted distillation; λ_{max} 3 430 cm^{-1} ; δ 6.90—6.66 (3 H, m, ArH), 5.91 (2 H, s, O-CH₂-O), 2.93 (1 H, dd, J 6.0 and 3.5 Hz), 2.12 (1 H, d, J 6.0 Hz), 1.69 (1 H, d, J 3.5 Hz), and 1.40br (1 H, s, NH, exchangeable with D₂O); *phenyl isocyanate derivative* (XIII), white prisms, m.p. 145—147° (Found: C, 68.2; H, 4.8; N, 10.25. $C_{16}H_{13}N_2O_3$ requires C, 68.2; H, 4.95; N, 9.95%); ν_{max} (CHCl₃) 1 700 and 1 670 cm^{-1} ; δ 3.91 (1 H, dd, J 8.5 and 11.0 Hz, CH_B-N), 4.45 (1 H,

dd, J 8.5 and 11.0 Hz, CH_A-N), 5.43 (1 H, t, J 8.5 Hz, PhCH), 5.93 (2 H, s, O-CH₂-O), 6.70—6.83 (3 H, m, ArH), 6.95—7.63 (5 H, m, ArH), and 11.79br (1 H, s, NH, exchangeable with D₂O).

3-[2-Ethoxy-2-(3,4-methylenedioxyphenyl)ethylamino]cyclohex-2-enone (IXa) and its Methoxy- and Hydroxy-analogues (IXb and c).—2-(3,4-Methylenedioxyphenyl)aziridine (0.98 g), triethylamine (3.0 g), 3-chlorocyclohex-2-enone (0.48 g), and tetrahydrofuran (30 ml) were stirred for 6 days under nitrogen. The solution was filtered, diluted with ether, washed with sodium carbonate (2N), dried, and separated [t.l.c.; 5% methanol in chloroform (containing 0.67% ethanol)] to give (i) the *ethoxy-enone* (0.13 g) as an oil, which decomposed upon attempted distillation (Found: M^+ , 303.147048. $C_{17}H_{21}NO_4$ requires M , 303.145979), ν_{max} 3 420, 1 610, and 1 585 cm^{-1} , δ 6.95—6.80 (3 H, m, ArH), 5.94 (2 H, s, O-CH₂-O), 5.4br (1 H, t, NH), 5.12 (1 H, s, olefinic H), 4.40 (1 H, t, J 6.5 Hz), 3.48 (2 H, t, J 6.5 Hz, CH₂-N), 3.29 (2 H, q, J 6.0 Hz, O-CH₂), 2.55—1.67 (6 H, m, methylene), and 1.19 (3 H, t, J 6.0 Hz), m/e 303 (60%), 273 (70), 258 (16), 251 (16), 242 (8), 241 (12), 213 (40), 192 (44), 181 (80), and 67 (100); (ii) the *methoxy-enone* (IXb) (0.10 g) as an oil, also unstable to distillation (Found: M^+ , 289.132399. $C_{16}H_{19}NO_4$ requires M , 289.131399), ν_{max} 3 420, 1 610, and 1 585 cm^{-1} , δ 6.90—6.70 (3 H, m, ArH), 5.99 (2 H, s, O-CH₂-O), 5.12 (1 H, s, olefinic H), 5.01 (1 H, m, NH), 4.29 (1 H, t, 6.0 Hz), 3.24 (3 H, s, OCH₃), 3.23 (2 H, t, J 6.0 Hz, CH₂-N), and 2.55—1.62 (6 H, m, methylene), m/e 289 (2.1%), 166 (32.1), 165 (42.1), 154 (10.6), 150 (100), 139 (20), 121 (66), and 111 (67); and (iii) the *hydroxy-enone* (IXc) (0.17 g), as white prisms, m.p. 171—172° (from chloroform) (Found: C, 65.25; H, 6.0; N, 4.8%; M^+ , 275.115749. $C_{15}H_{17}NO_4$ requires C, 65.5; H, 6.25; N, 5.1%, M , 275.114684), ν_{max} (Nujol) 3 420, 1 615, and 1 585 cm^{-1} , δ (C₆D₆-N) 8.71 (1 H, s, OH, exchangeable with D₂O), 7.65—6.80 (3 H, m, ArH), 5.95 (2 H, s, O-CH₂-O), 5.61 (1 H, s, olefinic H), 5.20 (1 H, t, J 6.0 Hz, NH, exchangeable with D₂O), 3.32 (2 H, t, J 6.0 Hz, CH₂-N, collapsing to 2 H, d, J 6.0 Hz on addition of D₂O), and 2.55—1.55 (6 H, m, methylene), m/e 275 (5%), 257 (53), 256 (50), 228 (15), 215 (24), 214 (36), 201 (30), 200 (30), 183 (32), 182 (50), 173 (100), 171 (100), 160 (57), 150 (60), and 149 (100).

2-Benzyl-N-(3-oxocyclohex-1-enyl)-3-phenylaziridine (IIIc).—2-Benzyl-3-phenylaziridine, m.p. 44—45° (lit.¹⁴ 44—45°) [characterised as the phenylcarbonyl derivative (XII), m.p. 122—123° (lit.⁹ 123—125°), ν_{max} 3 270 and 1 662 cm^{-1} , δ * 7.50—6.80 (15 H, m, ArH), 6.65br (1 H, s, NH, exchangeable with D₂O), 3.84 (1 H, d, $J_{ab} \pm 6.0$ Hz, H_a), 2.85 (1 H, m, $J_{bd} + 6.5$, $J_{bc} + 5.0$, $J_{ab} \pm 6.0$ Hz, H_b), 2.61 (1 H, dd, $J_{cd} - 11.5$, $J_{bc} + 5.0$ Hz, H_c), and 2.46 (1 H, dd, $J_{cd} - 11.5$, $J_{bd} + 6.5$ Hz, H_d)], (2 g) was treated as above with 3-chlorocyclohex-2-enone (1.2 g) and triethylamine (2 ml). The usual work-up followed by t.l.c. (benzene-ether, 10:1) gave the *N*-substituted aziridine (1.1 g), b.p. 160—163° at 1.5 Torr, as an oil (Found: M^+ , 303.162446. $C_{21}H_{21}NO$ requires M , 303.162306); λ_{max} 279 nm ($\log \epsilon$ 4.356); ν_{max} 1 635 and 1 590 cm^{-1} , δ 1.55—2.38 (6 H, m, methylene), 2.43—2.63 (3 H, m), 3.28 (1 H, m, PhCH), 5.42 (1 H, s, olefinic H), and 6.89—7.48 (10 H, m, ArH), m/e 303 (46%), 275 (12), 264 (13), 245 (24), 244 (20), 212 (12), 211 (50), 210 (100), 209 (38), 206 (20), 192 (30), and 190 (37).

¹² An alternative preparation has been reported (E. Piers and I. Nagakura, *Synth. Comm.*, 1975, 5, 193).

¹³ G. Maneli, *Gazzetta*, 1909, 39, 165.

¹⁴ K. Kotera and K. Kitahonoki, *Org. Synth.*, 1968, 48, 20.

* Part of the spectrum analysed by NMRCAI for a four-spin system.

Attempted Rearrangement of the Aziridine (IIIc) into 8-Benzyl-9-phenyl-7-azabicyclo[4.3.0]non-1(6)-en-2-one.— A solution of the aziridine (IIIc) (1.5 g), sodium iodide (2.1 g), and diethyl ketone (10 ml) was refluxed under nitrogen for 3 days. Evaporation and chromatography of the complex product gave an oil (0.1 g), believed to be the bicyclo[4.3.0]-

nonenone (XIV); λ_{max} 300 nm ($\log \epsilon$ 4.029); ν_{max} 3 240 and 1 595 cm^{-1} ; δ 1.8—2.5 (6 H, m, methylene H), 2.78 (2 H, d, 7.0 Hz, PhCH_2), 3.9 (2 H, m), 6.2br (1 H, s, NH, exchangeable with D_2O), and 6.9—7.40 (10 H, m, ArH).

[6/831 Received, 30th April, 1976]
