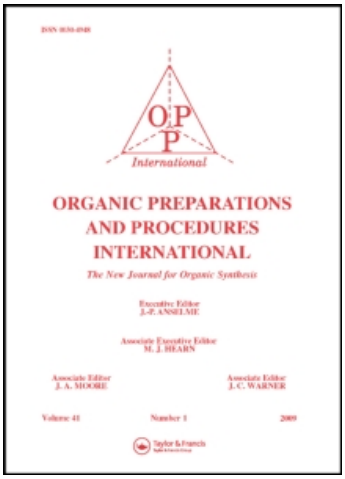

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PYRROLOQUINOLINES

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PYRROLOQUINOLINES

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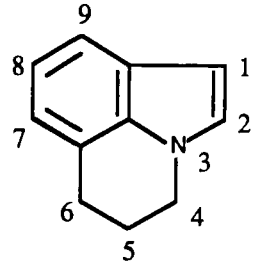
INTRODUCTION

1. *Scope and Arrangement*

Indole and quinoline ring systems occur in a multitude of naturally-occurring alkaloids and their synthetic chemistry has also been developed extensively. In some natural products, these ring structures are combined in various ways. In this review, we consider the pyrrolo-[3,2,1-ij]quinoline system **1**, in which the three rings are fused together, and their related benzo-derivatives **2**.



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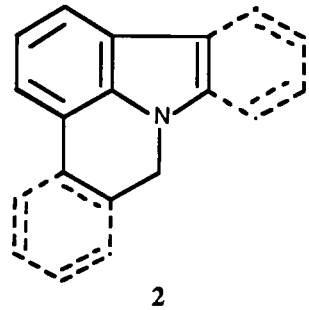
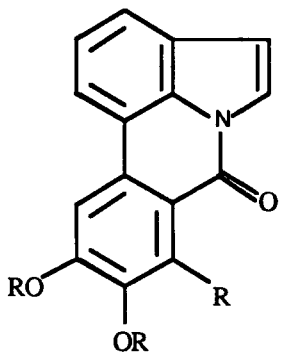


2

In most examples the indole ring system is intact, whilst the quinoline can be reduced (Section II) or in the form of a quinolone (Sections III and IV) or quinoline dione (Section V). Benzoanalogs of quinolones are also included, as are carbazole derivatives where relevant in each section.

2. *Naturally-occurring pyrroloquinolines*

Most of the alkaloids in this class have been isolated from the Amaryllidaceae species and especially the Crinum genus. Both the crinum alkaloids[1] and the amaryllidaceae alkaloids[2] have been reviewed



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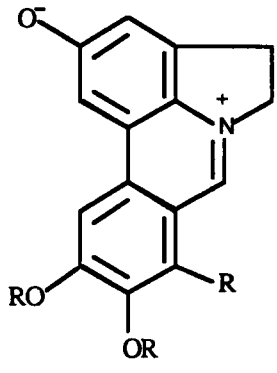

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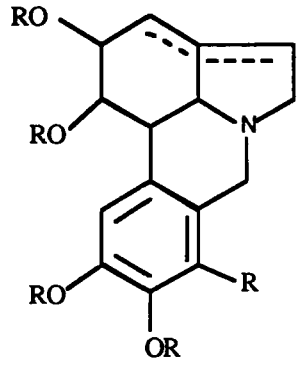


5

This review will not be concerned with types **4** or **5**, but only type **3**. Although earlier references are noted, literature coverage is essentially from 1966 to 1989.

3. *Biological activity*

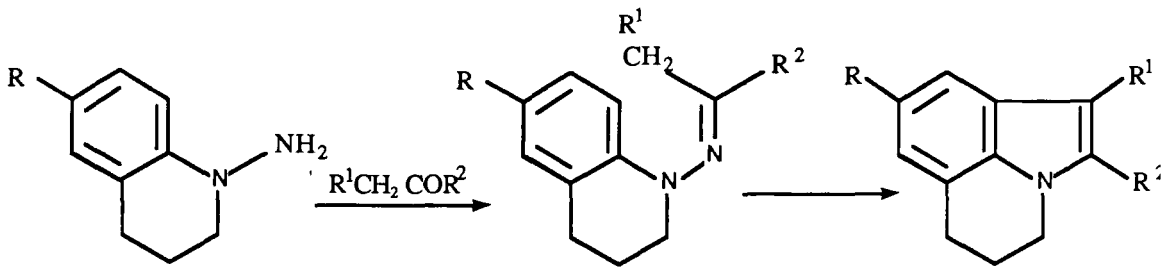
A variety of biological properties have been reported for relatively simple synthetic pyrroloquinolines. These include analgesic, antipyretic, anti-inflammatory and central nervous system stimulant activity reported for a range of tetrahydropyridopyrroloquinolines[3]. Some simpler compounds and their dibenzo analogs show antihypotensive, antidepressant and anticonvulsant activity.[4,5,6] Certain pyrroloquinolones lower blood pressure[7] and anti-tumour activity is shown by some related quinone derivatives.[8,9] Fungicidal properties are also shown by some hexahydropyrroloquinolinones.[10]

Many of the pyrroloquinoline alkaloids show a variety of similar effects, especially hippadine, which also produces reversible inhibition of fertility in male rats.[11] The reduced compound lycorine (related to structure **5**) acts against tumour cells and inhibits protein and DNA synthesis.[1]

I. PYRROLOTETRAHYDROQUINOLINES

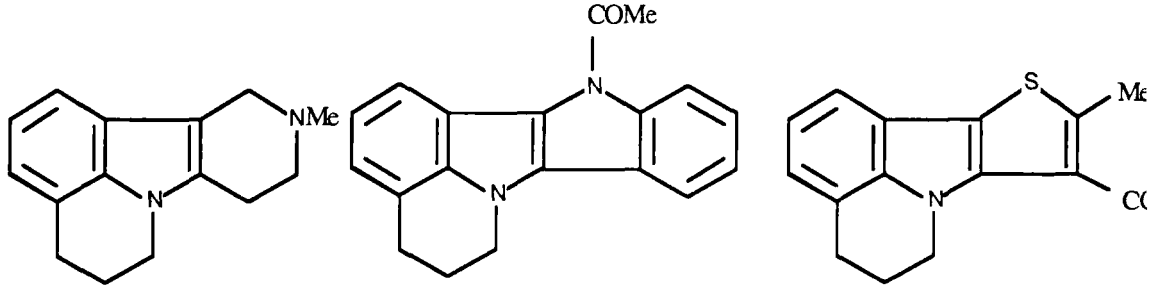
1. *Synthesis*

The Fischer synthesis provides the most general route[12] to these pyrroloquinolines, commencing with 1-aminotetrahydroquinolines **6**, which undergo reaction with methylene ketones to give hydrazones **7**, followed by indoles **8**.



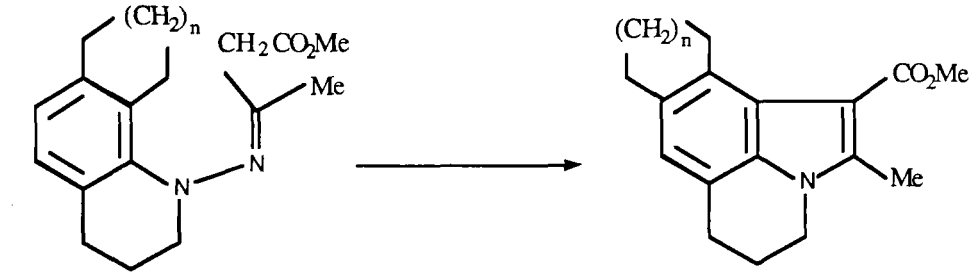
6 7 8

A variety of cyclic ketones can be used in this reaction. For example, cyclohexanone gives related tetrahydrocarbazolesl.[4] Similarly, the use of N-methyl piperidine-4-one, N-acetyldihydroindole-3-one and ethyl 3-hydroxy-5-methyl thiophene-4-carboxylate yields the products **9**, **10** and **11** respectively.[3,13]



9 10 11

In many cases, rearrangements occur with group migration allowing indole formation. For example, hydrazones **12** give pyrroloquinolines **13**, together with other products.[14,15]

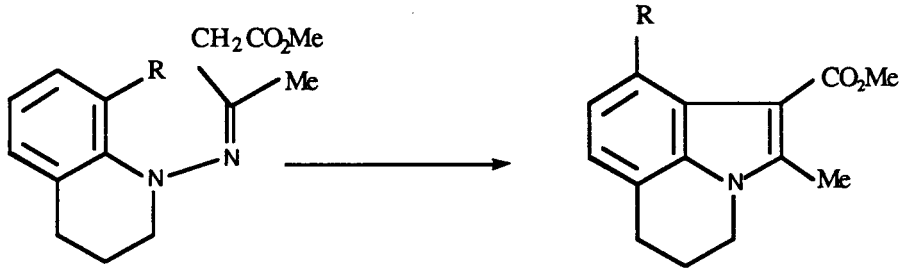


n= 1,2

12 13

In general, groups which have strong migratory aptitudes undergo migration, whilst groups which form stabilised cations are eliminated.[16-18] Consequently the phenyl-substituted hydrazone **14** gives the phenyl-

substituted product **15**, but the diethylaminoethyl derivative **16** is converted to the less-substituted product **17**.



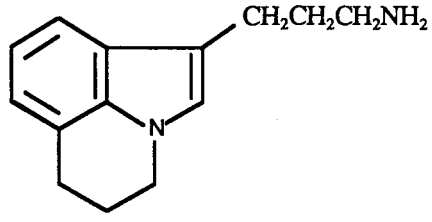
14 R= Ph

16 R= CH_2NEt_2

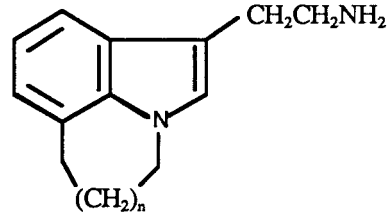
15 R= Ph

17 R= H

The Fischer synthesis has also been used to give tryptamine derivatives and analogs. The use of 1-piperideine (or its trimer) - effectively a source of 5-aminopentanal - directly gives the tryptamine homolog **18**.[19] The more common route involves 4-halobutanal and 5-halopentanal, which give tryptamine **19** and the homolog **18** respectively.[20,21] 1-Aminohexahydrobenzazocine has also been used in the preparation of the tryptamine derivatives **20**.[22]



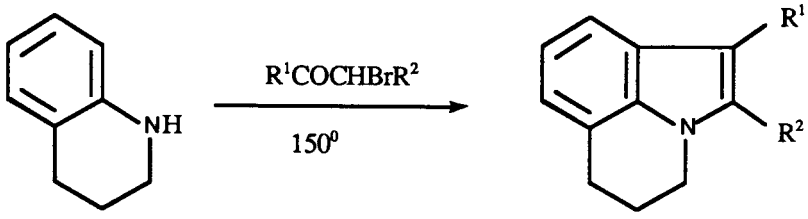
18



19 n = 1

20 n = 2

In these reactions the ammonia released in the Fischer synthesis displaces the halide ion to give the primary amine.

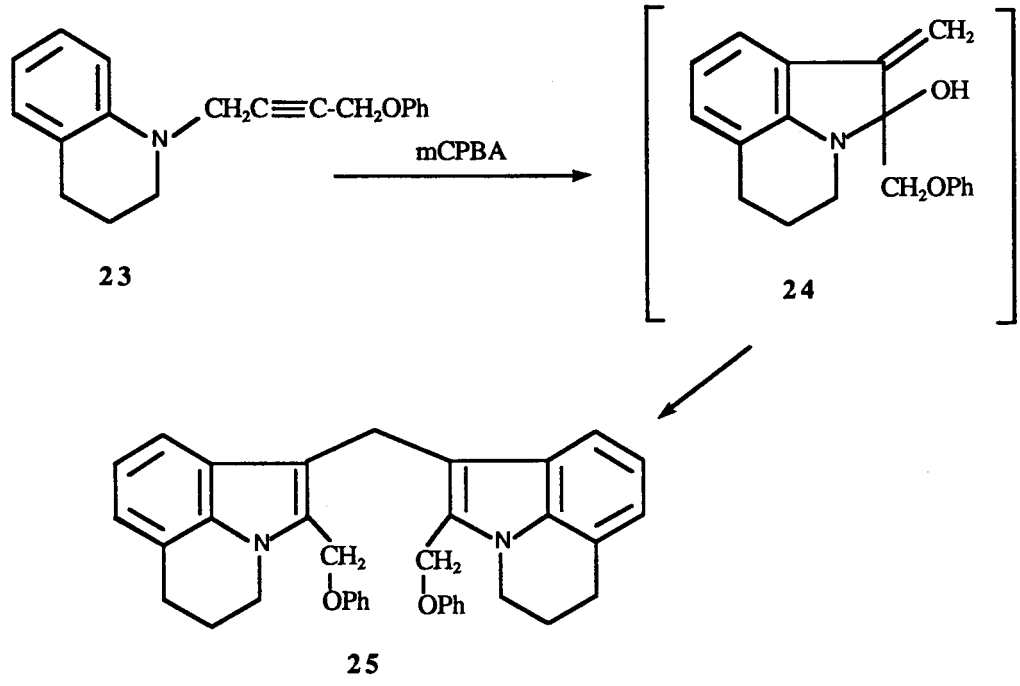


21 R^1, R^2 = Me

22 $R^1, R^2 = -(CH_2)_4-$

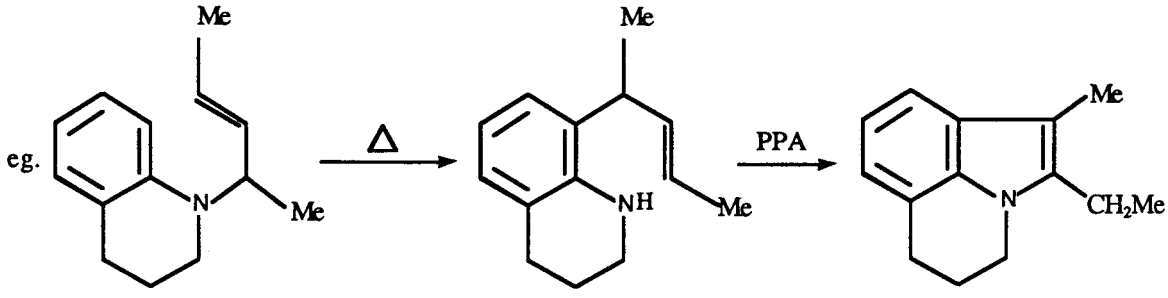
Other modes of indole formation have been used less frequently to convert tetrahydroisoquinolines to pyrroloquinolines. For instance, tetrahydroisoquinoline can be converted to compounds **21** and **22** by Bischler-type reactions using α-haloketones.[23]

Oxidative cyclization of an N-propargyl ether **23**, involving amine oxide rearrangement, gave the bis(pyrroloquinolyl) methane **25**, probably via the pyrroloquinolinol **24**.[24]



23 **24** **25**

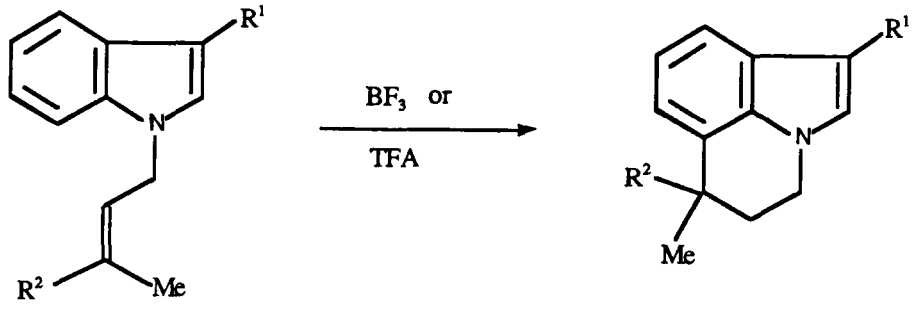
Claisen rearrangement of an N-allyl tetrahydroisoquinoline **26** gives the 8-allyl derivative **27** which can be cyclized by treatment with polyphosphoric acid, palladium chloride or ultraviolet irradiation, to give the pyrroloquinoline **28**.[25]

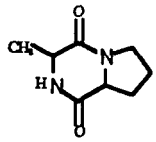


26 **27** **28**

In principle, any indole synthesis based on aniline starting materials could provide further examples of pyrroloquinolines.

An alternative approach to these compounds is the cyclization of suitable indole derivatives in which the quinoline formation is the key step. This strategy is the second most general one used for the synthesis of pyrroloquinolines. Acid-catalyzed cyclizations of both N- and 7-allyl indoles allow formation of the additional six-membered rings. The indoles **29** and **31** undergo direct cyclization with boron trifluoride etherate to give the products **30** and **32** respectively.[26,27]

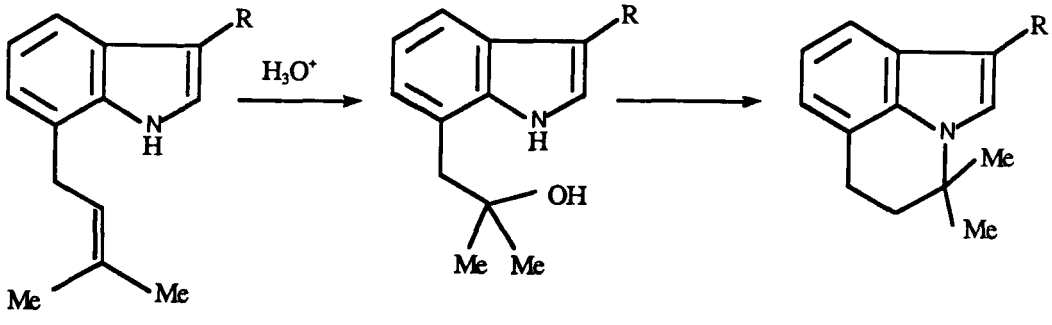


29 R^1 =  R^2 = Me **30**

31 R^1 = CO_2Me, R^2 = $-(CH_2)_3OCH_2Ph$ **32**

33 R^1 = CHO, R^2 = Me **34**

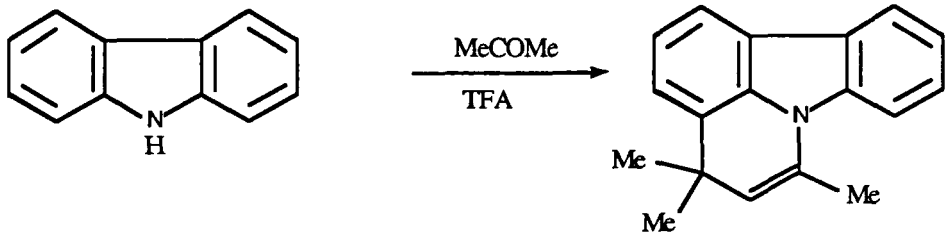
The latter product **32** is used to construct the skeleton of the teleocidin alkaloids. A similar triflûoroacetic acid-catalyzed cyclization of alkenyl compound **33** to pyrroloquinoline **34** has been shown to proceed via



35 **36** **37**

an intermediate trifluoroacetate derivative resulting from an addition reaction.[28] Acid-catalyzed cyclization can also occur from a 7-alkenyl indole **35** via the intermediate alcohol **36**, to give pyrroloquinoline **37**.[8] In this sequence, the indole 3-substituent is an indolylquinone and the compounds are members of the asterriquinone pigments.

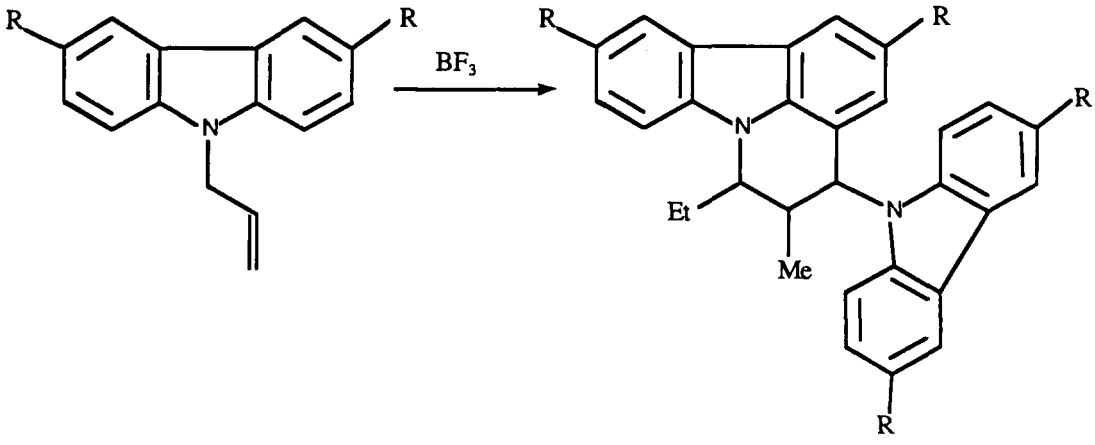
Carbazole undergoes reaction with acetone and trifluoroacetic acid to give a good yield of the unsaturated pyrroloquinoline **38**, resulting from reactivity at C1 and N9.[29]



37 **38**

Similar reactions involving formaldehyde and acetone mixtures give rise to cyanine dyes containing related aromatic structures.[30]

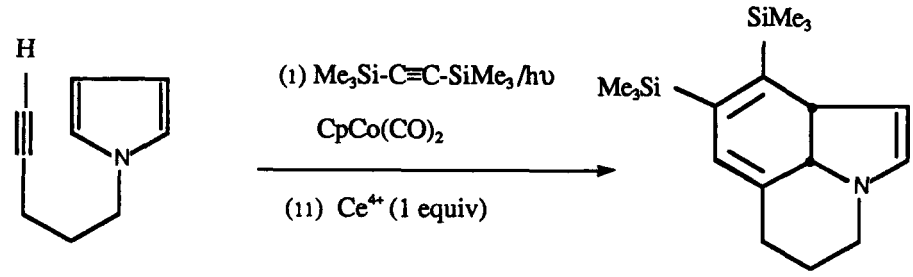
N-Allyl carbazoles **39** and **41** dimerise on treatment with boron trifluoride etherate to give good yields of compounds **40** and **42** respectively.[31]



39 R = H **40** R = H

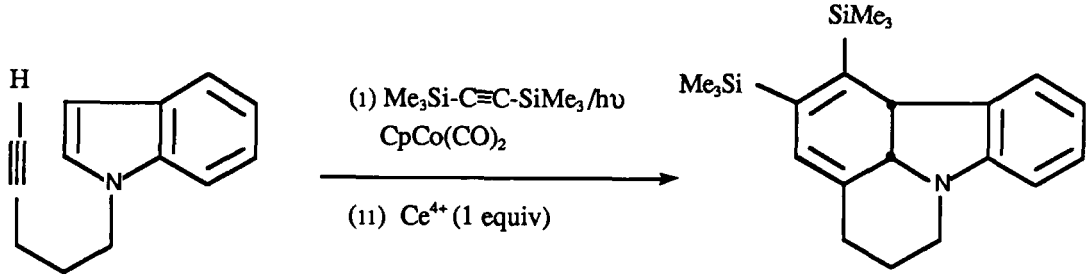
41 R = Cl **42** R = Cl

A novel cobalt-mediated [2+2+2] cycloaddition of alkynes to alkynyl-substituted pyrroles has recently been developed by Vollhardt and co-workers. For example, irradiation of the alkynyl pyrrole **43** and bis-trimethylsilylacetylene in the presence of cyclopentadienyl cobalt dicarbonyl formed the dihydro-pyrroloquinoline cobalt complex, which on oxidative demetalation gave the free ligand **44**.[32]



43 **44**

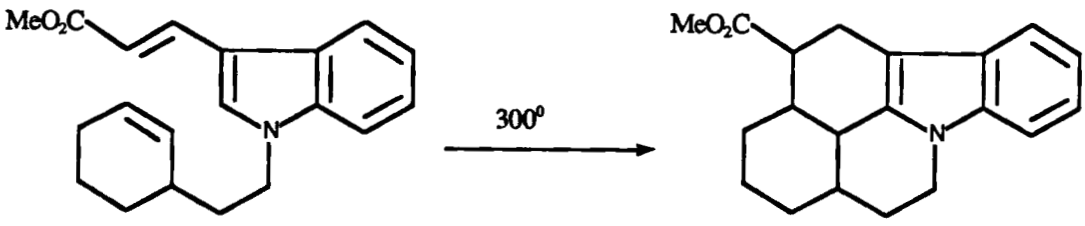
Oxidation with 6-8 equivalents of ceric ammonium nitrate gives the corresponding aromatic indole derivative. This cycloaddition reaction has been extended to describe the conversion of alkynyl indoles **45** into their related carbazole derivatives **46**.[33]



45 **46**

Intramolecular Diels-Alder reactions can build two additional rings onto an indole, such as compound **47**, to produce the indoloquinoline **48**.[34]

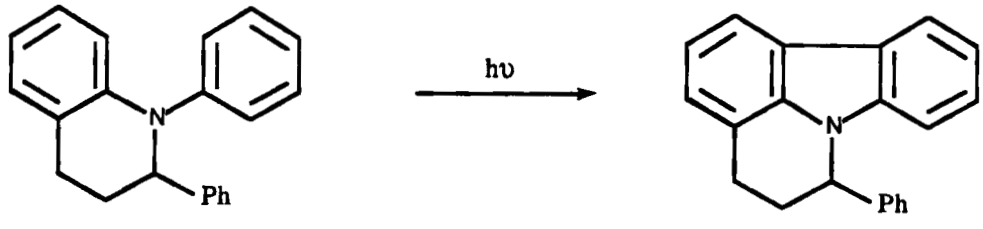
In the two previous classes of cyclization reactions the corresponding N-acyl derivatives have also been investigated and shown to give the related 4-oxopyrroloquinolines (see Section III).



47 48

Alternatively, the carbazole ring can be constructed by photochemical oxidative coupling of an N-aryl tetrahydroquinoline derivative.[35]

e.g. **49** → **50**

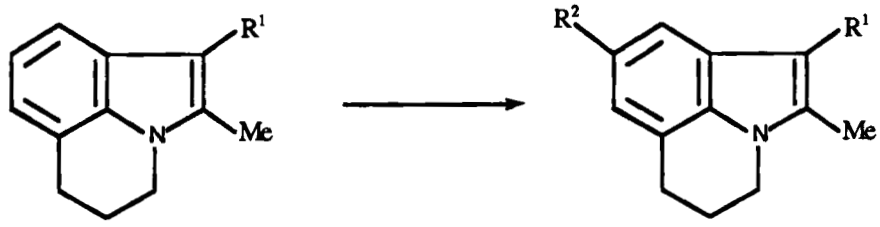


49 50

2. *Reactions*

(i) Electrophilic substitution

Pyrrolotetrahydroquinolines behave in the same manner as other indoles towards electrophiles. The dimethyl compound **21** undergoes bromination,[36] sulfonation[37] and chlorosulfonation[38] at the indole 5-



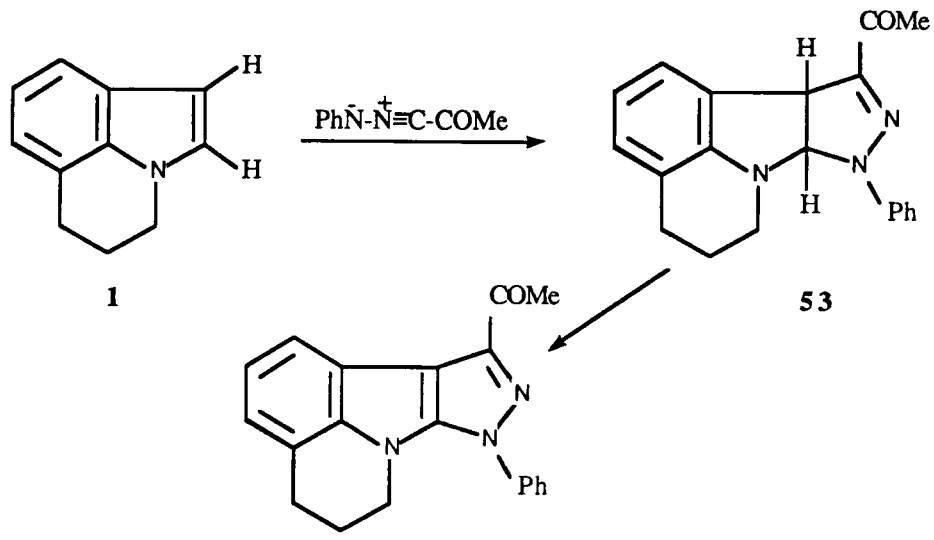
21 $R^1 = Me$ **51** $R^1 = Me$, $R^2 = Br$, SO_3H or SO_2Cl

52 $R^1 = CO_2Et$

position to give compounds **51**. In contrast, nitration of the indole-3-ester **52** gave a mixture of 4- and 6-nitro-substituted products.[39]

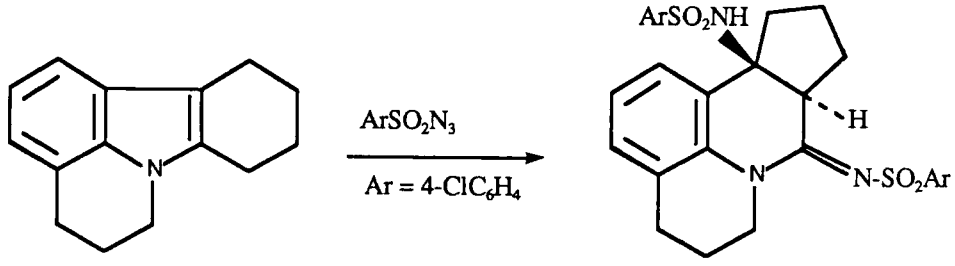
(ii) Cycloadditions

Nitrilimines undergo 1,3-dipolar cycloaddition across the indole 2,3-double bond of pyrrolotetrahydroquinolines, e.g. **1** in a manner similar to that of simple N-methyl indoles, to give the cycloadducts **53**, which can be oxidised to the related indoles **54** using chloranil.[40]



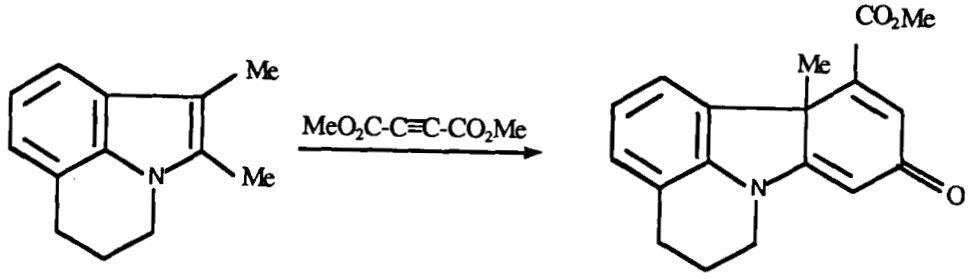
1 **53** **54**

4-Chlorobenzenesulfonyl azide undergoes addition to compound **22** to give the product **55**, which results from indole ring-opening.[41]



22 **55**

Dimethylacetylene dicarboxylate reacts at the indole 3-position in a Michael addition process. Pyrroloquinolines with 2,3-dialkyl substituents behave in the same manner as N-methyl indole. For example, compound **21** gives the tetracyclic product **56**.[42]

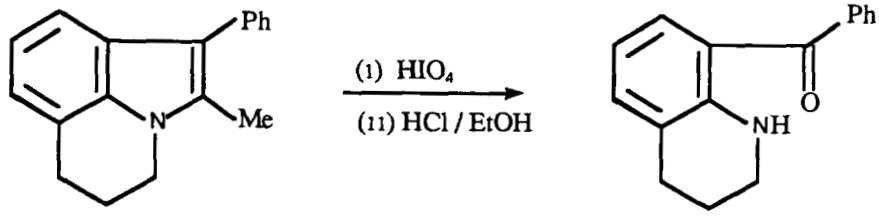


21 56

In contrast, tetracyclic compounds such as **22** give additional products resulting from indole ring-opening.[43-45] Methyl propiolate adds in a similar manner to dimethyl acetylenedicarboxylate, giving a variety of different products under different reaction conditions.[46]

(iii) Oxidation

One important use of tetrahydropyrroloquinolines (e.g. **57**) is as precursors of 8-acyl tetrahydroquinolines (e.g. **58**). The reaction involves oxidative cleavage of the indole ring followed by hydrolysis of the amide group.[47]

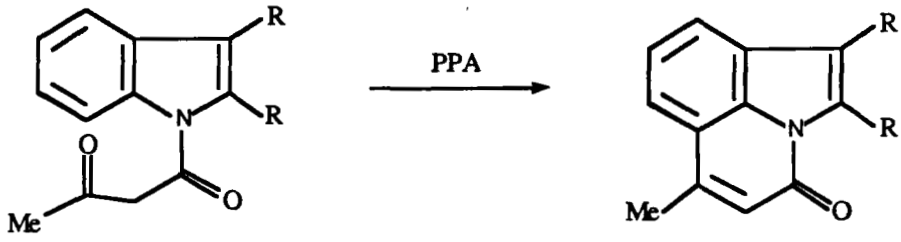


57 58

II. 4-OXOPYRROLOQUINOLINES

1. *Synthesis*

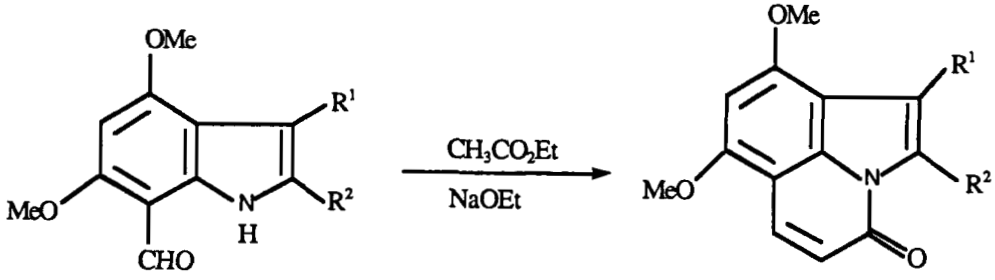
Compounds of this structural class are relatively rare. The earliest synthesis makes use of the polyphosphoric acid cyclization of N-acetoacetyl-indoles **59**, **61**, to give the pyrroloquinolones **60**, **62** respectively.[48]



59 R = Me **60**

61 R , R = -$(CH_2)_4$- **62**

This class is now more accessible as the result of the recent conversion of 7-formyl indoles **63** to pyrroloquinolines **64** by reaction with ethyl acetate and sodium ethoxide.[49]



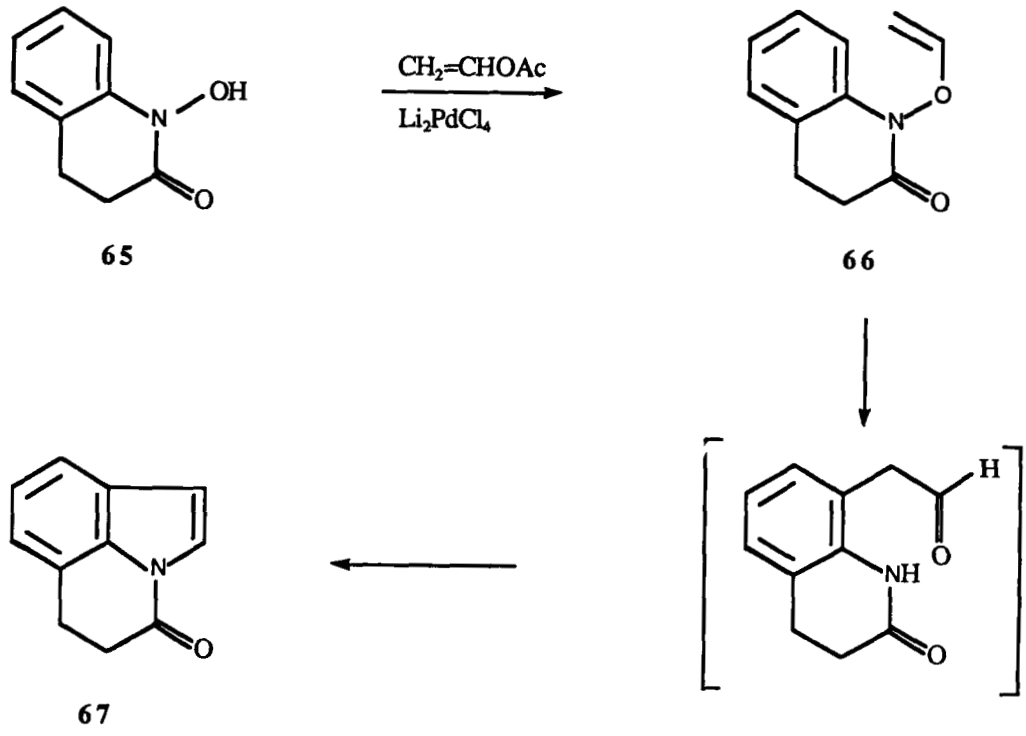
63 R^1 , R^2 = H , Ph , -$(CH_2)_4$- **64**

R^1 = Me , R^2 = H

R^1 = H, R^2 = Ph or 4-BrC_6H_4

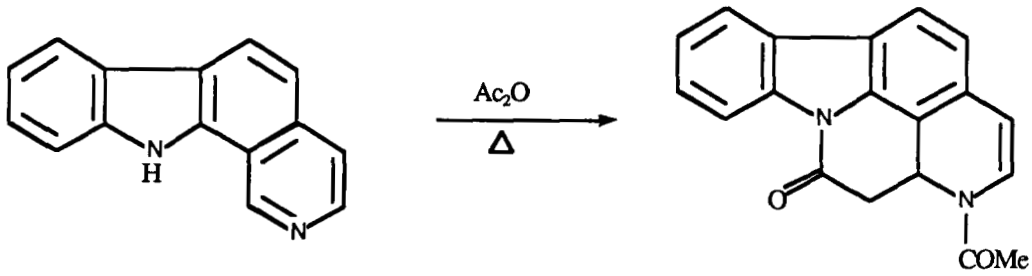
The more reduced pyrroloquinolone **67** has been prepared by treatment of the hydroxamic acid **65** with vinyl acetate in the presence of lithium tetrachloropalladate, via a hetero-Cope rearrangement of the intermediate **66**.[50]

The cobalt mediated [2+2+2] cycloaddition described in Section II can also be applied to the synthesis of pyrroloquinolones. The corresponding N-aryl derivatives of alkynyl pyrrole **43**[32] and indole **45**[33] give oxo-derivatives of compounds **44** and **46** respectively. The related intramolecular Diels-Alder reaction[34] can also be applied to the N-acyl derivatives.



65 66 67

In a rather special example, the pyridocarbazole **68** reacts in refluxing acetic anhydride to give the pentacyclic product **69**.[51]

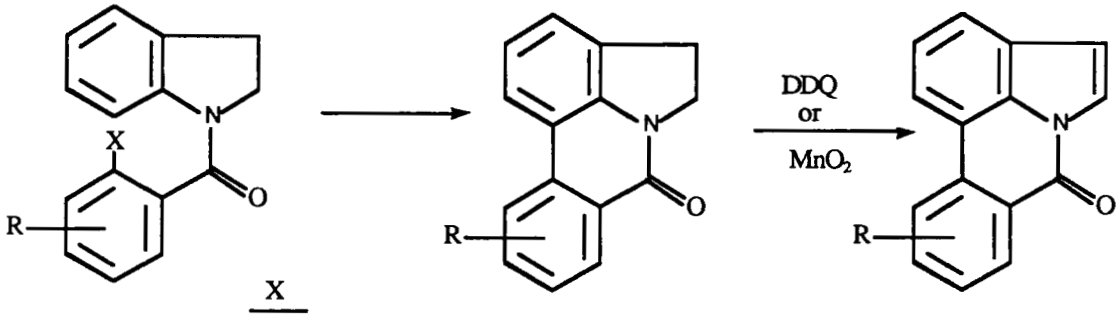


68 69

The benzo-analogs of the 4-oxopyrroloquinolines form the class of pyrrolophenanthridones, which include a number of important alkaloids. The isolation and characterization of these alkaloids have been reviewed recently.[1] Here we are concerned with general synthetic routes to the structural class as a whole, rather than detailed syntheses of specific alkaloids.

The most general approach uses N-aroyl dihydro-indole precursors and the key step becomes the

linking of the aryl group to the dihydro-indole C7 position to afford the dihydropyrrolophenanthridones **75**. Such intramolecular biphenyl formation can be achieved in several ways, such as diazonium coupling[52, 53] (e.g. **70** → **71** → **75**), ultraviolet irradiation of aryl halides[54] (e.g. **72** or **73** → **75**) or palladium acetate coupling[55] (e.g. **74** → **75**). Subsequent dehydrogenation using dichlorodicyanoquinone or oxidation using manganese dioxide converts the dihydro derivatives **75** into the pyrrolophenanthridones **76**.

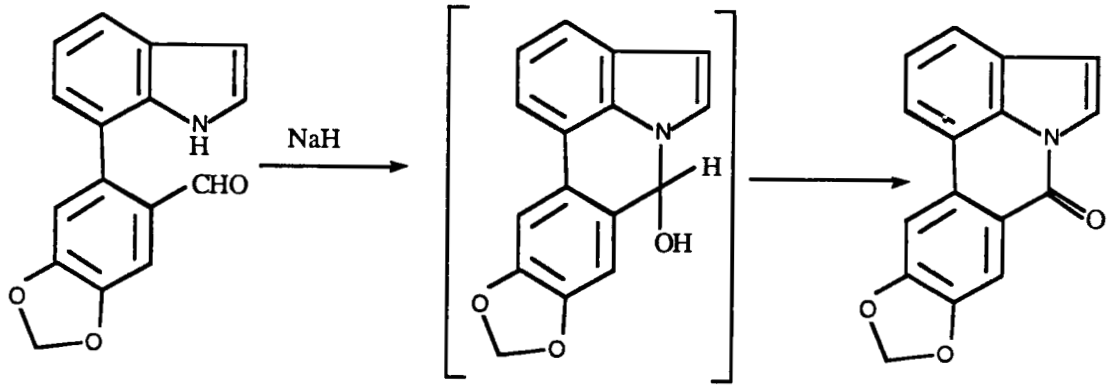


	X
70	NH_2
71	N_2^+
72	Br
73	I
74	H

75 **76**

R = H or various oxygenated substituents

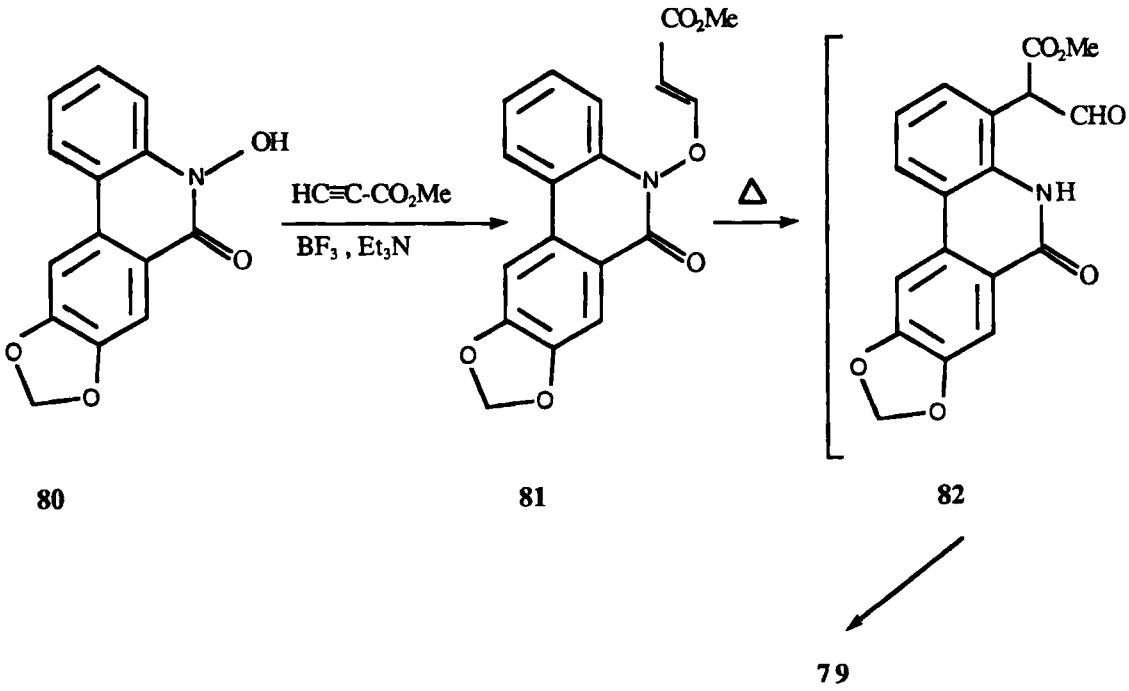
The natural product hippadine has also been synthesised by a lengthy procedure in which a 7-indolyl benzaldehyde derivative **77** is constructed.



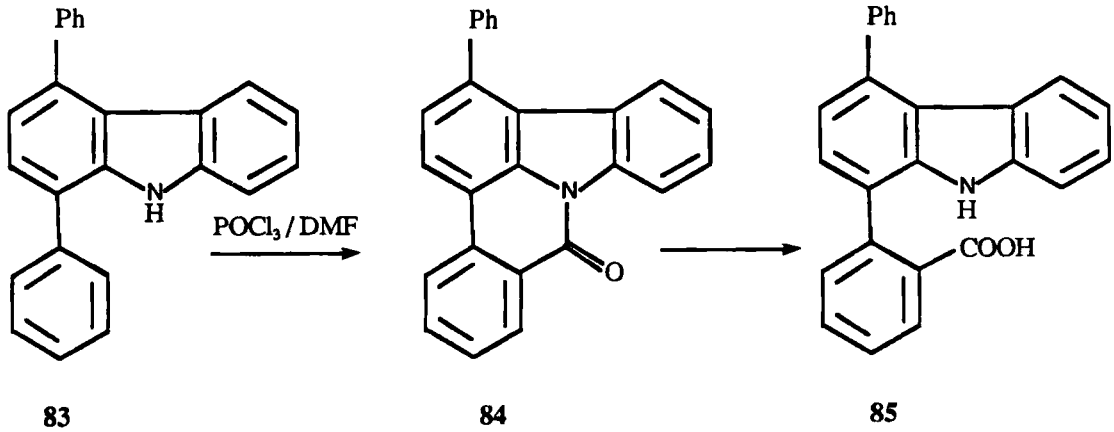
77 **78** **79**

Thus the crucial cyclization becomes the attack of an indole nitrogen anion on an aldehyde to give an intermediate alcohol **78**, which undergoes ready oxidation to the pyrrolophenanthridone alkaloid, hippadine **79**.[56]

Another route to hippadine uses an N-hydroxyphenanthridone precursor **80**. Treatment with boron



trifluoride, methyl propiolate and triethylamine affords the derivative **81**, which undergoes thermal rearrangement to an intermediate malonyl phenanthridone **82**, which subsequently undergoes cyclization, ester hydrolysis and decarboxylation to give hippadine **79**.[57]

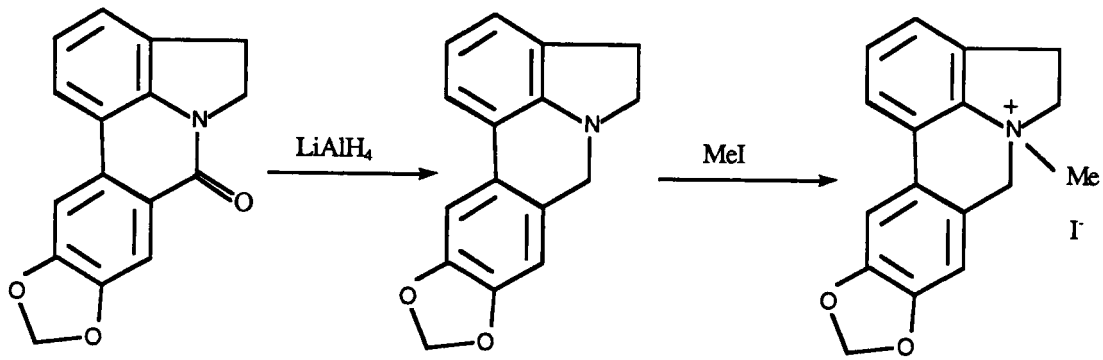


The related indolophenanthridone **84** has been generated from 1,4-diphenylcarbazole **83** under vigorous Vilsmeier conditions of phosphoryl chloride and dimethylformamide in refluxing o-dichlorobenzene.[58]

2. *Reactions*

The reactivity of 4-oxopyrroloquinolines has so far not been investigated in detail. However, hydrolysis of the amide bond of the indolophenanthridone **84** has been used to generate a 1-(o-carboxyphenyl) carbazole **85**.[59]

Reduction of the carbonyl group of the dihydropyrrolophenanthridone **86** has been carried out using lithium aluminium hydride to yield the amine **87**, which has been quaternized with methyl iodide to give the salt **88**.[60]



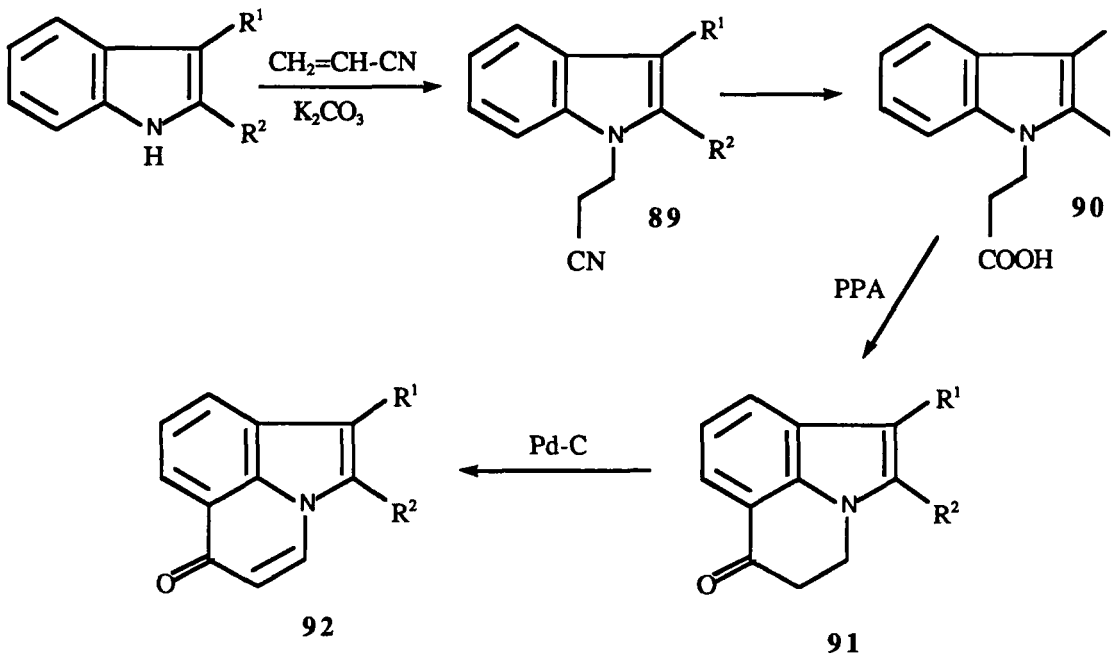
86 **87** **88**

III. 6-OXOPYRROLOQUINOLINES

1. *Synthesis*

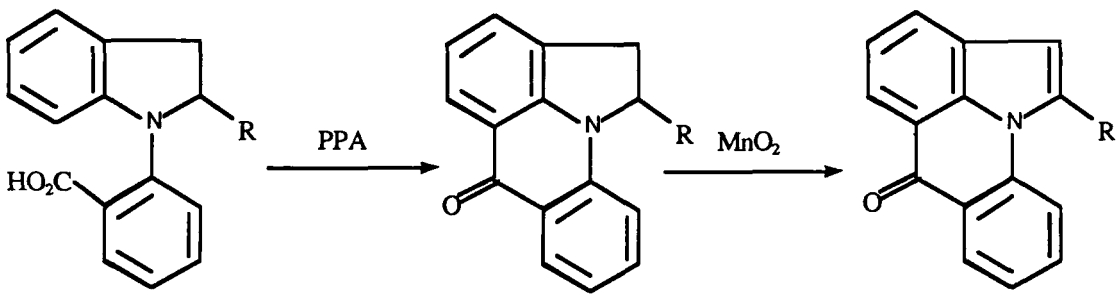
The general synthetic route to these compounds involves the polyphosphoric acid cyclization of an indol-1-yl propionic acid **90** on to the 7- position.[7, 60-63] The starting materials are usually obtained from the related nitriles **89**, following the Michael addition of indoles to acrylonitrile. In some cases, dehydrogenation of the dihydro derivatives **91** has yielded the 6-oxopyrroloquinolines **92**.[63]

Commonly, both R^1 and R^2 are aryl, alkyl or cycloalkyl, so cyclization can only proceed to the indole C7



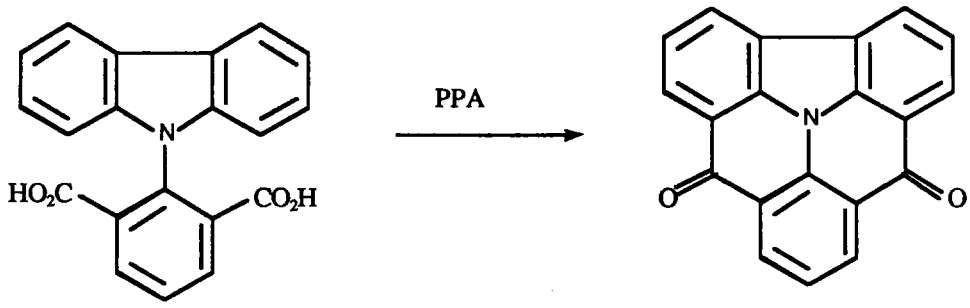
position. If R^1=H and R^2=alkyl, cyclization occurs at C2, but where R^1=H and R^2=CO_2Me, cyclization still takes place at C7.[64] Related cyclization has been carried out on the carbazole system.[65]

The benzo analogs, the pyrrolo-acridones **95** have been synthesized by a similar cyclization, but this time on the indoline derivatives **93** rather than the indoles themselves. Oxidation of the indoline products **94** with manganese dioxide affords the indoles **95**.[66]



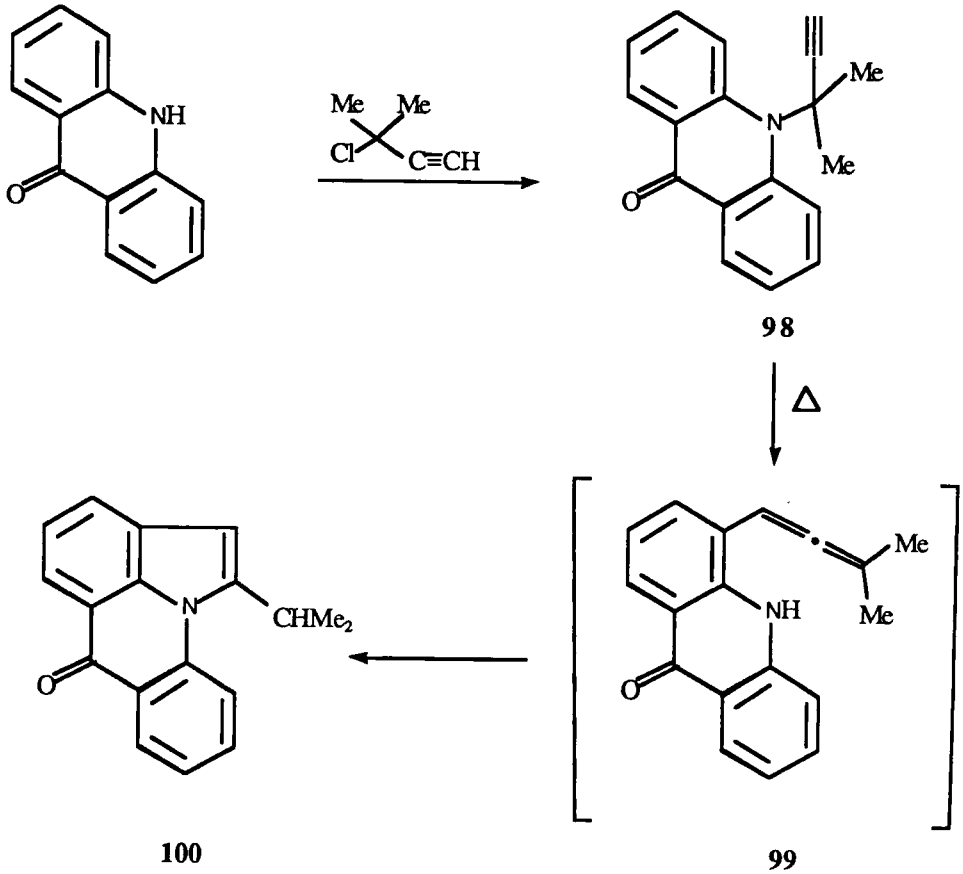
93 R = H , Me **94** **95**

A similar, but double cyclization of the carbazole derivative 96 yields the fused heterocyclic system **97**.[67]



96 97

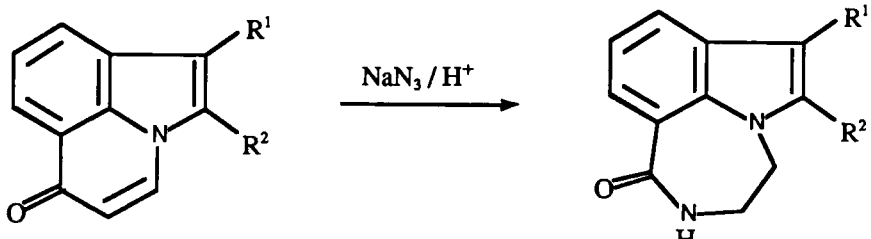
The pyrrolo-acridone **100** has been prepared from acridone by reaction with 3-chloro-3-methylbut-1-yne under phase transfer conditions. Presumably, reaction occurs initially at nitrogen to give the derivative **98**, which undergoes Claisen rearrangement to the allene intermediate **99**, which in turn allows cyclization to the indole **100**.[68]



100 98 99

2. *Reactions*

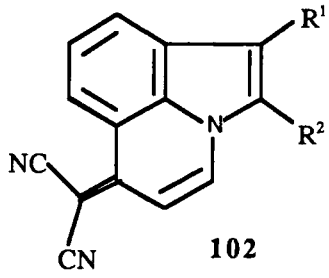
The 6-oxo group shows normal carbonyl properties, such as oxime formation.[7] Whilst Beckmann rearrangement of the oximes does not appear to have been investigated, Schmidt reaction of the 6-oxo



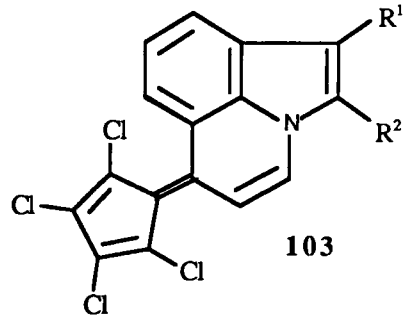
92 R^1, R^2 = H, alkyl, cycloalkyl **101**

compounds **92** readily affords pyrrolo-benzodiazepinones **101**, which are of pharmacological interest.[60,61]

Carbonyl reactivity also allows reaction of the oxo-compounds **101** with malononitrile and



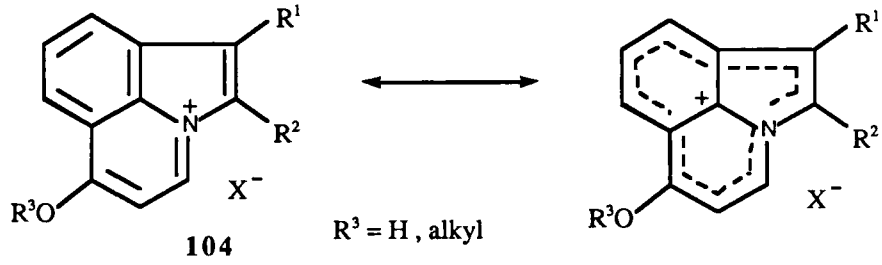
102



103

tetrachlorocyclopentadiene to give the products **102** and **103** respectively.[69]

Treatment of oxo-compounds **92** with trifluoroacetic acid or trialkyloxonium fluoroborates



104

R^3 = H, alkyl

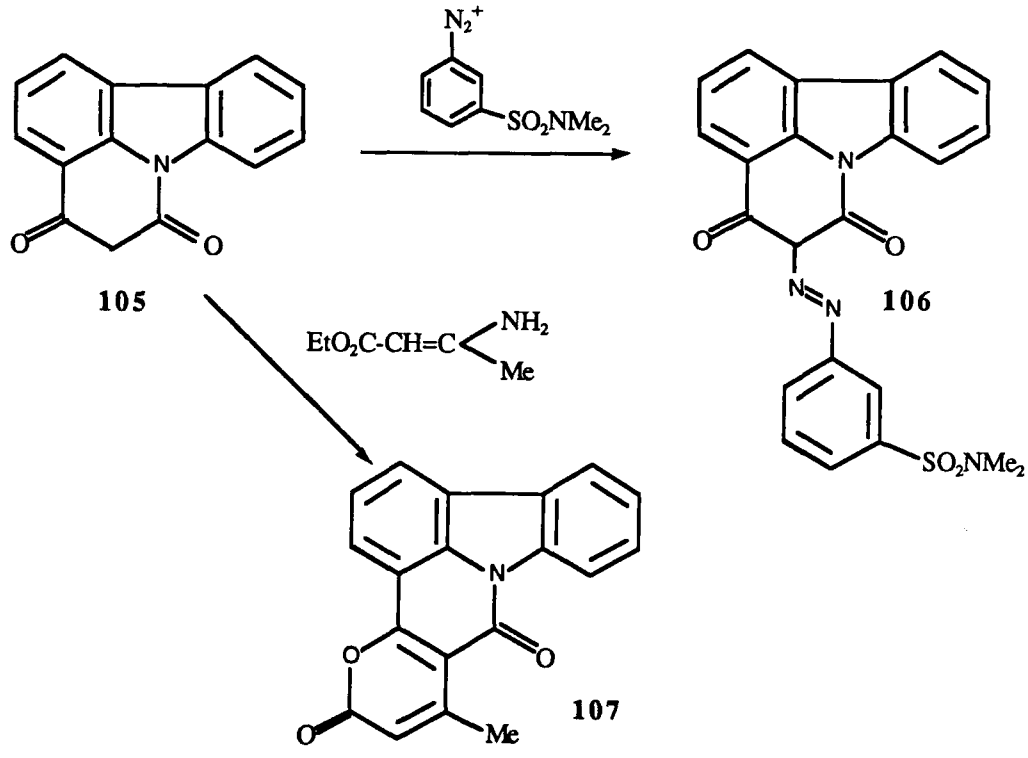
X = CF_3CO_2, BF_4

generates the fully aromatic azapseudophenalenone salts **104**.[69,70]

Similar reactions have been carried out on the related thione, generated from compounds **92** by treatment with phosphorus pentasulfide.[70] ^{1}H and ^{13}C n.m.r. studies of the salts **104** show a downfield shift for all the signals in the molecule.[71]

IV. 4,6-DIOXOPYRROLOQUINOLINES

Simple 4,6-dioxopyrroloquinolines are not known, but the related carbazoles **105** have been reported.[72-74] In the absence of detail it can be assumed that these arise from carbazole and malonyl dichloride. These β–dicarbonyl compounds **105** undergo typical coupling reactions and have been used as precursors of a disperse azo dye **106**[72] and a highly fluorescent coumarin **107**.[73, 74]



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