Amines and amides

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REVIEW PERKIN 1

Covering: 1997
Previous review: *Contemp. Org. Synth.*, 1997, 4, 326

1 Introduction, scope and coverage

This review covers the literature published during 1997; references were obtained using the online science citation index in the same way as in previous years.[1] A consequence of this is that a few papers published at the end of 1996 are included in this review, and some papers published late in 1997 may not be included, but will be included in next year's review of this area. As with last year's review, papers dealing with the solid state or combinatorial synthesis of amines or amides have been omitted. The initial literature search produced over eight thousand references, so this review is necessarily highly selective. The approximately one hundred and seventy papers included are those which, in the opinion of the author, have general applicability or particular significance.

This year, the format of this review has been changed from a product-based subdivision to a methodology-based sectionalization. The main reason for this change is that many derivatives of amines and amides can be easily interconverted, which made the previous classification somewhat arbitrary. Thus for example, β-amino acids are easily cyclized to β-lactams. Throughout this review, the focus has been placed on methods which create a carbon-nitrogen bond, or in which an existing carbon-nitrogen bond is critical to the subsequent chemistry.

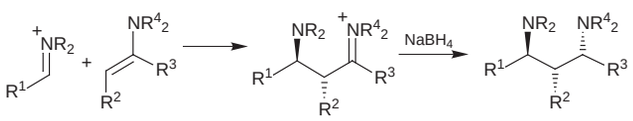
2 Addition of nucleophiles to imines and related species

The addition of nucleophiles (particularly those based on carbon or hydrogen) to imines and related compounds is rapidly developing into one of the most effective methods for the synthesis of amines. This section of the review has been divided into: methods which produce achiral or racemic amines; methodology which produces non-racemic amines from chiral imines; and chemistry which converts prochiral imines into chiral amines by the use of a chiral reagent, ligand, or catalyst. Both the addition of hydride to, and the hydrogenation of, imines are included in this section although the latter is not strictly a nucleophilic addition. A review of the asymmetric synthesis of amines by the addition of nucleophiles to imines, covering the literature up to early 1997 has been published.[2]

2.1 Methods giving achiral or racemic products

The addition of Me_3Si-CN to imines is catalysed by $Yb(OTf)_3$, and it is also possible to generate the imine *in situ* from an aldehyde and an amine.[3] $Yb(OTf)_3$, $Zr(OTf)_4$ and $Hf(OTf)_4$ also catalyse the addition of other nucleophiles such as silyl enol ethers and allyl stannanes to imines, and in the case of $Yb(OTf)_3$ do so chemoselectively in the presence of aldehydes.[4] The combination of $PhSCF_3$ and Et_3GeNa generates a synthetic equivalent to the trifluoromethyl anion which reacts with *N*-aryl imines to form α-trifluoromethyl imines.[5] Boron trifluoride–diethyl ether catalyses the reaction between an aldehyde, a primary amine and allyl(trimethyl)silane to form homoallylic amines.[6]

Aldehydes react with (trimethylsilyl)dialkylamines in the presence of lithium perchlorate to give an iminium ion to which organozinc reagents will add, producing tertiary amines. A wide variety of functional groups can be present in the organozinc reagent including esters, alkenes and alkynes, thus allowing the synthesis of functionalized amines by this methodology.[7] Similar chemistry involving trimethylsilylmethylmagnesium bromide or lithium trimethylsilylacetylide instead of an organozinc reagent has been reported as a method for the synthesis of silicon-containing amines.[8] A diastereoselective synthesis of 1,3-diamines by the reaction of an enamine with an iminium salt followed by sodium borohydride reduction has been reported as shown in **Scheme 1**.[9]



Scheme 1

In recent years, Katritzky has been exploring the synthetic utility of *N*-(α-alkylamino)benzotriazoles. These compounds react as imine equivalents, and reaction with a fluorinated Grignard reagent in the presence of boron trifluoride provides access to fluoroalkyl amines.[10] The addition of lithium propiolates to nitrones followed by reductive cleavage of the nitrogen-oxygen bond, partial reduction of the alkyne and cyclization has been used in a synthesis of γ-substituted-α,β-didehydro γ-lactams.[11]

2.2 Methods utilizing a chiral imine or related species

The Lewis acid induced asymmetric addition of organometallic reagents to imines bearing galactopyranosyl derived substituents on the nitrogen atom has been reported previously. This

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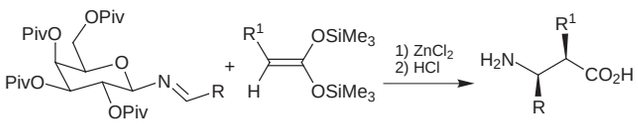
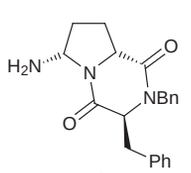
methodology has now been extended to the zinc chloride induced addition of silyl ketene acetals as shown in **Scheme 2**. Hydrolysis of the initial adducts produces enantio- and diastereo-merically pure β-amino acids.[12] Interestingly, if the same imine is allowed to react with the lithium enolate of *tert*-butyl phenylacetate, then the opposite stereoisomer at the stereocentre adjacent to the carboxylic acid is obtained.



Scheme 2

Full details have also been published of a procedure for the addition of organozinc compounds to ethyl valinate derived imines. The addition reaction proceeds with poor to excellent (>99 : 1) diastereoselectivity, but the conditions for the removal of the chiral auxiliary (LAH followed by H_5IO_6) are rather harsh and are likely to limit the applicability of the process.[13] Indium(III) induces the addition of allyl bromide to imines of (*S*)-valine methyl ester with a de of 90% or better, and hence provides ready access to homoallylic amines.[14] Addition of organolithium reagents to, or reduction of imines derived from, valinol or phenylglycinol and ferrocenyl aldehydes or ketones has been used in an asymmetric synthesis of a range of ferrocenyl amines. The amino alcohol derived auxiliaries could again be cleaved by treatment with H_5IO_6.[15] Imines can also be formed from phenylglycinol and a ketone, and they react with Grignard reagents in the presence of magnesium bromide with diastereoselectivities of 21–97%. The phenylglycinol auxiliary can be removed by oxidation with lead tetraacetate, providing a short synthesis of non-racemic α,α-disubstituted primary amines.[16]

The use of amine **1** as a chiral auxiliary for the addition of nucleophiles to imines has been reported. Thus condensation of **1** with benzaldehyde followed by the addition of a silyl ketene acetal in the presence of zinc bromide and cleavage of the auxiliary by acidolysis provides access to enantiomerically pure α-substituted β-amino acids.[17] In the presence of cerium(III) chloride, Grignard reagents react with imine **2** to give (*S*)-furfuryl amines after cleavage of the chiral auxiliary.[18] The addition of allylzinc bromide to bis-imine **3** has also been studied, and was found to produce the corresponding 1,2-diamine with the (*R*)-configuration at each new stereocentre.[19] In a reaction which presumably proceeds *via* an imine or iminium ion, treatment of an α-keto acid with an amine and an alkenyl or aryl boronic ester gives α-amino acids as shown in **Scheme 3**. If a chiral amine is used, then non-racemic amino acids can be prepared. In particular, phenylglycinol gives a >99% de and the chiral auxiliary can be removed (with concomitant reduction of any alkene present) by hydrogenation.[20] The diastereoselective addition of cyanide to *N*-(1-phenylethyl)imines has been used in an enantioselective synthesis of a cyclopentyl-derived (*S*)-aspartic acid (**Scheme 4**). After the initial cyanide addition, a 4.5 : 1 ratio of the two diastereomers in favour of the desired epimer is obtained. These can be separated and potassium carbonate can be used to re-equilibrate the unwanted diastereomer to a 4.5 : 1 mixture of epimers.[21]



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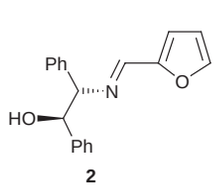

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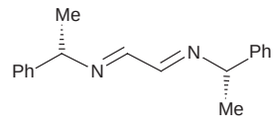
Isopropylcerium trichloride reacts with oxazolidine **4** to give (*R*)-2-(1-amino-2-methylpropyl)imidazole, a key intermediate



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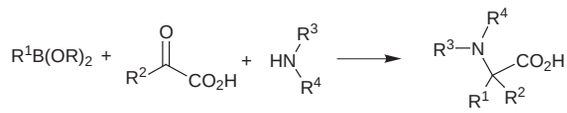
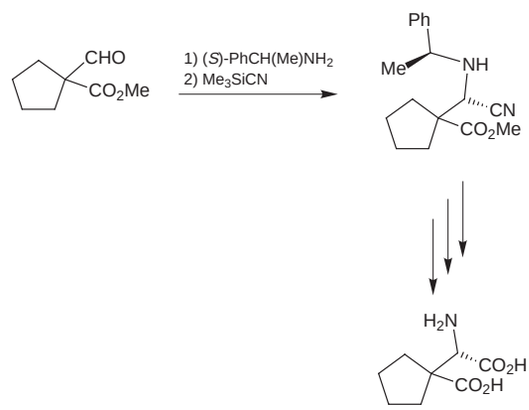
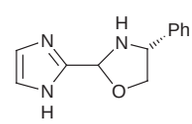


Scheme 3



Scheme 4



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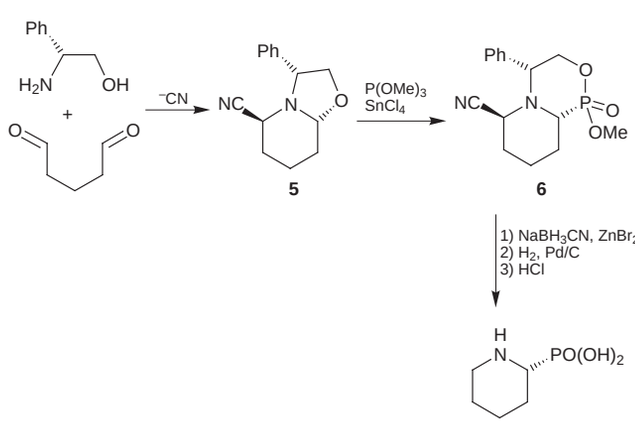
in the synthesis of protease inhibitors.[22] Phenylglycinol derived oxazolidines have also been used as chiral auxiliaries in an unusual asymmetric synthesis of piperidin-2-ylphosphonic acid as illustrated in **Scheme 5**. Thus condensation of phenylglycinol with pentane-1,5-dial in the presence of cyanide forms amino nitrile **5**, which in the presence of a Lewis acid reacts stereospecifically with trimethyl phosphite to give intermediate **6**. Subsequent reductive cleavage of the auxiliary and cyanide groups, and hydrolysis, provides piperidin-2-ylphosphonic acid.[23]

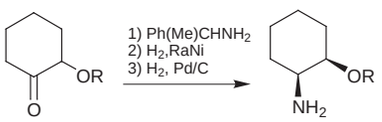


5 **6**

Scheme 5

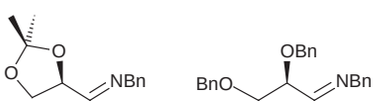
Amino ester imines of benzophenone are widely used in amino acid synthesis due to the acidifying effect of the imine on the α-hydrogen(s). However, it has now been shown that the same imines provide a convenient approach to the synthesis of *N*-alkyl amino acids. Thus reduction of the imine with sodium

cyanoborohydride gives an *N*-benzhydryl amino ester which reacts with an aldehyde and further sodium cyanoborohydride to give *N*-benzhydryl-*N*-alkyl amino esters from which the *N*-benzhydryl group can be cleaved by hydrogenation.[24] A diastereo- and enantio-selective synthesis of *syn*-β-aminocyclohexyl ethers from racemic α-alkoxycyclohexanones has been developed as shown in **Scheme 6**. Thus condensation of the cyclohexanone with 1-phenylethylamine gives an imine which is in equilibrium with an enamine. The latter is stereoselectively hydrogenated in the presence of Raney nickel to give a β-amino ether derivative from which the 1-phenylethylamino group can be removed by further hydrogenation over palladium. The main limitation of the methodology is likely to be the very slow (3 weeks) reduction of the enamine.[25] Similar methodology was used to prepare a variety of dimethylcyclohexylamines from the corresponding cyclohexanone, and again the hydrogenation of the imine is slow (8–10 days).[26]

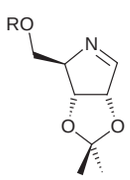


Scheme 6

The addition of vinylmagnesium bromide to imine **7** occurs with a de >98%, providing an asymmetric synthesis of (*S*)-vinylglycine after removal of the acetal protecting group and oxidation of the resulting diol.[27] Phenylmagnesium bromide reacts in the same way with imine **7**, but adds to the opposite face of imine **8**, thus allowing either enantiomer of phenylglycine to be prepared just by varying the diol protecting group.[28] Similarly, organometallic reagents react with imine **9** from the face opposite to the cyclic acetal.[29] The reduction of *N*-benzyl β-hydroxy imines by sodium cyanoborohydride is stereoselective, producing β-amino alcohols with a 4 : 1 to 14 : 1 *syn* to *anti* ratio.[30]



7 **8**



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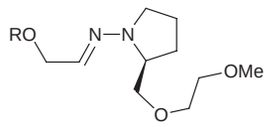
The groups of Denmark [31] and Enders [32] have simultaneously reported the asymmetric addition of organolithium reagents to α-heteroatom substituted hydrazones bearing a proline derived chiral auxiliary (**10** and **11** respectively). In both cases, the organolithium reagent preferentially reacted on the *re*-face of the imine, and the chiral auxiliary could be removed by hydrogenation over Raney nickel or by acylation followed by treatment with lithium in liquid ammonia, though the former method was reported to cause partial racemization in some cases. The utility of the SAMP auxiliary is that it can be used to control the stereochemistry of both the alkylation adjacent to a SAMP hydrazone, and the addition of a nucleophile to the hydrazone. Enders' group have exploited this versatility in the asymmetric synthesis of primary amines and piperidin-2-ones by the addition of organolithium reagents to RAMP/SAMP hydrazones and/or the alkylation of enolates of the RAMP/SAMP hydrazones.[33] The same approach was employed in an asymmetric synthesis of β-aminosilanes from ethanal,[34] and in



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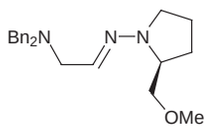

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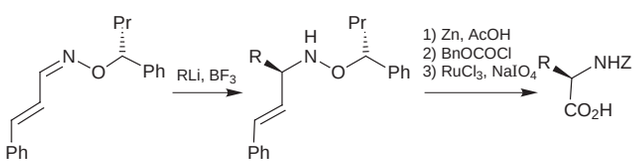
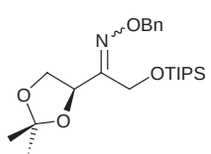
the preparation of chiral diamine derivatives of ferrocene from ferrocene dialdehydes. In the latter case, ee values of 90–98% and (±)/*meso* ratios of up to 95 : 5 were observed.[35]

Boron trifluoride induces the 1,2-addition of organolithium reagents to the (*R*)-*O*-(1-phenylbutyl)hydroxylamine oxime of cinnamaldehyde with a de >90%, and provides an asymmetric amino acid synthesis after subsequent manipulation as shown in **Scheme 7**.[36] The addition of organolithium reagents to oxime ether **12** has also been investigated. The sense of asymmetric induction was found to depend on the geometry of the oxime ether, with the (*E*)-isomer of the oxime giving the (*S*)-enantiomer of the amine adduct, whilst the (*Z*)-isomer of the oxime gave preferentially the (*R*)-enantiomer of the product. α-Substituted serine derivatives could be obtained from these chiral amines by cleavage of the acetal and oxidation of the resulting diol.[37] It is also possible to carry out radical additions to oxime ethers, and treatment of compound **13** with an alkyl iodide in the presence of tributyltin hydride and triethylborane results in diastereoselective addition of the radical to the oxime ether. Subsequent removal of the chiral auxiliary and cleavage of the nitrogen-oxygen bond provides an asymmetric amino acid synthesis.[38]



Scheme 7



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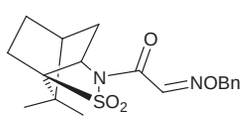

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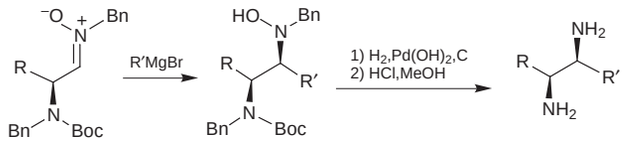
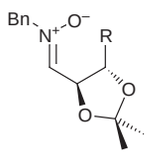
Grignard reagents add stereoselectively to enantiomerically pure *N*-Boc-*N*-benzyl-α-amino nitrones, providing a route to enantio- and diastereo-merically pure 1,2-diamines (**Scheme 8**).[39] The same group also reported that the stereochemistry of the addition of Grignard reagents to nitrone **14** depended on which metal salt was added. Thus zinc bromide favoured formation of the *syn*-isomer, whilst diethylaluminium chloride gave the *anti*-isomer. The products could be converted into 3-amino-1,2-diols.[40] Addition of cyanide to a variety of chiral nitrones has been investigated by Merino *et al.* All of the nitrones investigated had an oxygen atom attached to the α-carbon, and de values up to >90% were observed. A range of different



Scheme 8



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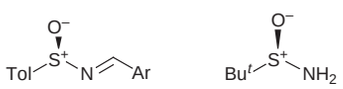
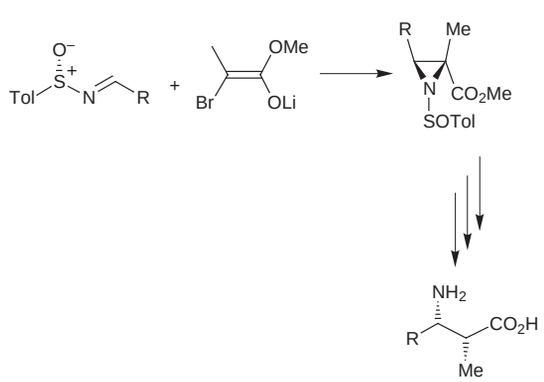
cyanide sources were investigated, but best results were obtained using Me_3Si-CN or diethylaluminium cyanide.[41]

The addition of benzylmagnesium bromide to sulfinimine **15** occurs preferentially from the *si*-face, resulting in the formation of (*S*)-1-aryl-2-phenylethylamines with ee values of 75–94%. However, attempts to extend the chemistry to the use of methylmagnesium bromide were unsuccessful.[42] The addition of the lithium enolate of methyl 2-bromopropionate to sulfinimines also occurs stereospecifically as shown in **Scheme 9** and allows the synthesis of chiral aziridine carboxylic acid derivatives, γ-amino alcohols and α-alkyl-β-amino acids.[43] An asymmetric synthesis of α-amino phosphonic acids by the addition of phosphites to chiral sulfinimines has also been reported. The key nucleophilic addition reaction occurs with a de of 85–97%.[44] An asymmetric synthesis of *tert*-butylsulfinamide **16** has been reported, and the addition of Grignard reagents to sulfinimines derived from compound **16** and an aldehyde occured with a de of up to 84%.[45]



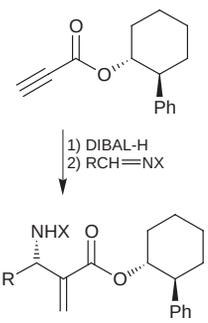
15 **16**



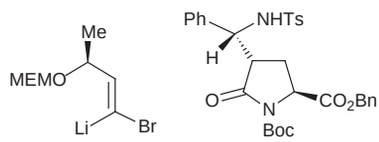
Scheme 9

2.3 Methods utilizing a chiral catalyst, ligand, or reagent

The synthesis of homoallylic amines by the addition of allylboranes with two chiral groups attached to the boron atom to *N*-(trimethylsilyl)imines has been investigated. Nine different chiral boranes were examined, and predictably, the structure of the chiral groups had a major influence on the degree of asymmetric induction, with ee values of 14–92% being observed. The best results were observed with the complex formed from triallylborane and *N*-tosyl norephedrine.[46] An enantioselective nitrogen version of the Baylis–Hillman reaction has been reported (**Scheme 10**). The synthesis starts with a chiral ester of propiolic acid which is reduced by DIBAL-H and the resulting enolate trapped stereoselectively with an imine. Diastereoselectivities of up to 85% were observed.[47] Full details of an asymmetric α-amino acid synthesis in which the chiral vinyllithium reagent **17** is added to an *N*-sulfonylimine have been reported. The synthesis is completed by ozonolysis of the alkene and removal of the *N*-protecting group.[48] Addition of the γ-enolate of benzyl *N*-Boc-pyroglutamate to *N*-tosyl benzylidenimine gives predominantly compound **18**, which can be transformed into a variety of other amino acids.[49]

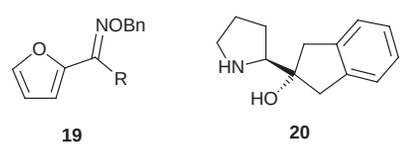


Scheme 10

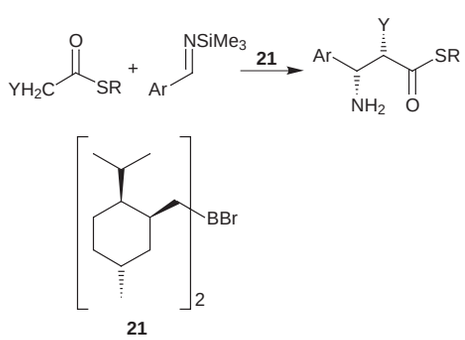


17 **18**

The asymmetric reduction of oximines **19** to furanyl amines by borane in the presence of chiral ligand **20** has been used as the first step in an asymmetric α-amino acid synthesis. The other geometrical isomer of the oximine gave the other enantiomer of the furanyl amine. Ozonolysis of the furan ring completed the amino acid synthesis.[50] Bromoborane **21** induces an enantio- and diastereo-selective aldol type reaction between a thioester and an *N*-$SiMe_3$-imine, as illustrated in **Scheme 11**, to give a β-amino thioester.[51]



19 **20**



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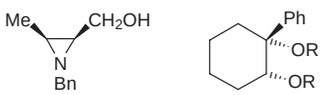
Scheme 11

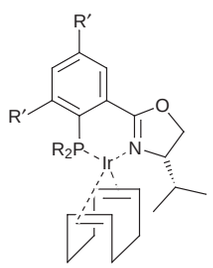
Aziridine **22** catalyses the asymmetric addition of diethylzinc to *N*-(diphenylphosphinoyl)imines with ee values of up to 87%.[52] Full details have been reported of the use of amino acid derived catechol catalysts for the asymmetric addition of organolithium reagents to imines as discussed in last year's review.[53] A variety of 1-phenylcyclohexane-*cis*-1,2-diol derivatives **23** have also been investigated as ligands for the asymmetric addition of organolithium reagents to imines. The best ee reported was 43% when R = CH_2CH_2OMe.[54] Denmark *et al.* have previously reported [1,2] the use of sparteine as a chiral catalyst for the addition of organolithium reagents to imines.

The use of this catalyst for the asymmetric 1,2-addition of organolithium reagents to cinnamyl imines has been investigated, and the nature of the *N*-protecting group was found to have a profound influence on both the magnitude and direction of asymmetric induction. Best ee values were obtained using a *p*-methoxyphenyl group, though it was not possible to remove this protecting group from the allylic amines. The other protecting groups investigated (triphenylmethyl and trimethylsilyl) gave lower ee values, though by employing an *N*-trimethylsilyl group the adducts could be transformed into the corresponding α-amino acids by oxidative cleavage of the alkene.[55]



22 **23**

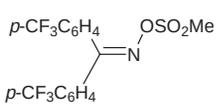
The complexation of chiral β-amino sulfides to diethylzinc has been shown to give a catalyst which allows the enantioselective reduction of dihydroisoquinolines by borane.[56] Catalyst **24** has been developed for the asymmetric hydrogenation of cyclic and acyclic imines. Ee values of up to 89% were obtained using just 0.1 mol% of the catalyst.[57]



24

3 Other syntheses of amines and amides from imines and their derivatives

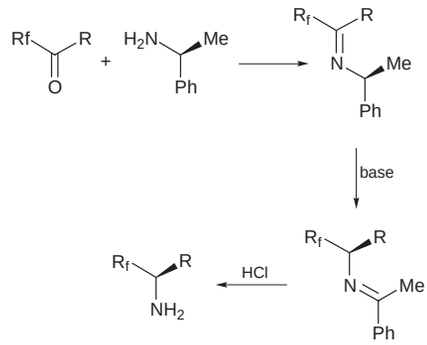
This section covers the chemistry of imines that does not conveniently fit into any other section of the review. A general method for the synthesis of primary amines based on the reaction of Grignard reagents with oxime derivative **25** has been reported. Compound **25** acts as a source of NH_2^+, the addition reaction is catalysed by copper cyanide, and the primary amine is liberated by treatment of the initial adduct with hydrochloric acid.[58]



25

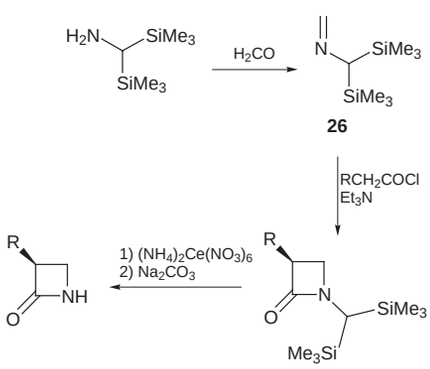
A biomimetic synthesis of (*RS*)-trifluoroalanine has been developed in which ethyl trifluoropyruvate is condensed with (*RS*)-1-phenylethylamine and treated with a base to isomerize the resulting imine into conjugation with the phenyl ring. Subsequent hydrolysis releases (*RS*)-trifluoroalanine.[59] The methodology has also been extended to provide an asymmetric synthesis of fluoroalkyl secondary amines and β-fluoroalkyl-β-amino acids by the use of enantiomerically pure 1-phenylethylamine as shown in **Scheme 12**.[60] A synthesis of (*S*)-thienylalanines in which *E. coli* ATCC11303 is used to transfer ammonia from aspartic acid to thienyl-α-keto acids has been reported.[61]

Reaction of bis(trimethylsilyl)methylamine with formaldehyde results in the formation of imine **26**, the first stable, isolable, monomeric imine of formaldehyde. Imine **26** was found to undergo [2 + 2] cycloaddition reactions with ketenes and



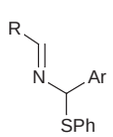
Scheme 12

aminoketenes, giving β-lactams as shown in **Scheme 13**. If the acid chloride precursor of the ketene was chiral, then asymmetric induction was observed at the new stereocentre created during the cycloaddition. Finally, the bis(trimethylsilyl)methyl protecting group could be removed by treatment with ceric ammonium nitrate.[62] It seems likely that more synthetic applications of imine **26** will be reported in the near future. 1-(Thiophenyl)benzyl imines **27** have also been used in [2 + 2] cycloaddition reactions with ketenes giving β-lactams from which the (thiophenyl)benzyl protecting group could be removed by treatment with $K_2S_2O_8$.[63]



26

Scheme 13

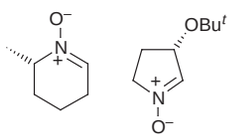


27

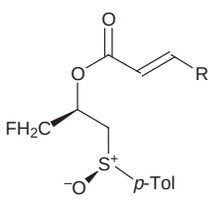
The ketene for a [2 + 2] cycloaddition reaction with an imine is usually prepared from an acid chloride. However, it is also possible to obtain the ketene by the Wolff rearrangement of an amino acid derived diazoketone.[64] In recent years, the group of Palomo have developed an unusual asymmetric amino acid synthesis which starts with a stereoselective [2 + 2] cycloaddition reaction between an *N*-1-phenylethyl imine and an alkoxyketene. The β-lactam is subsequently oxidized to an *N*-carboxyanhydride. In the latest development of this work, the synthesis of both enantiomers of the unnatural amino acid *tert*-leucine and their incorporation into dipeptides has been reported.[65] A number of other asymmetric syntheses of β-lactams by the [2 + 2] cycloaddition of ketenes and chiral imines have been reported.[66]

Nitrone **28** undergoes [3 + 2] cycloaddition reactions with alkenes in which the new carbon-carbon bond is formed *trans* to the methyl group. Subsequent reductive cleavage of the nitrogen-oxygen bond provides *trans*-2,6-disubstituted piperidines.[67] Similarly, nitrone **29** reacts with alkenes to give, after reductive cleavage of the nitrogen-oxygen bond, chiral γ-amino alcohols typically with a 2:1 to 3:1 ratio of diastereomers at

the newly formed stereocentre adjacent to the nitrogen atom.[68] Achiral, cyclic nitrones have also been shown to react with enantiomerically pure vinyl sulfoxides, again giving chiral γ-amino alcohols after reductive cleavage of of the sulfoxide auxiliary and nitrogen-oxygen bond.[69] An intramolecular nitrone [3 + 2] cycloaddition was the key step in an asymmetric synthesis of fluoromethyl substituted β-amino acids. Thus sulfoxides **30** undergo a Pummerer rearrangement to the corresponding aldehydes which can subsequently be converted to nitrones with *N*-benzylhydroxylamine. After the resulting cycloaddition, hydrogenolysis of the nitrogen-oxygen bond reveals the β-amino acids.[70]

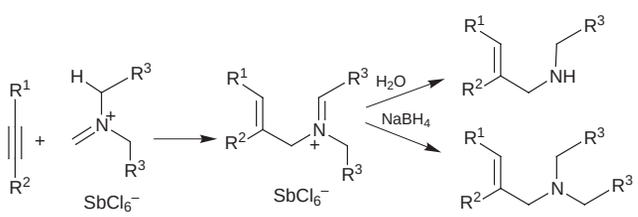


28 **29**



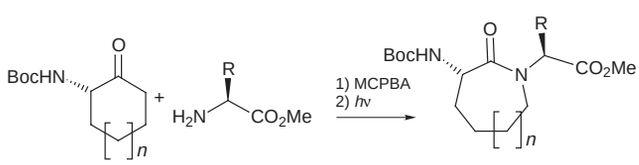
30

A stereoselective synthesis of allylic amines by an ene-reaction between an alkyne and an iminium salt has been reported as shown in **Scheme 14**. If the reaction is worked-up hydrolytically, then secondary amines are produced, whilst a reductive work-up allows the preparation of tertiary amines.[71]



Scheme 14

A method for the ring expansion of cyclic ketones to cyclic amides which are of use as conformationally constrained peptide analogues has been reported as shown in **Scheme 15**. Thus condensation of a cyclic ketone with an amino ester gives the corresponding imine which is converted to an oxaziridine with MCPBA and photolysed to give the amide.[72] Oxidation of *N*-alkyl or *N*-aryl imines with sodium perborate also produces oxaziridines which rearrange with migration of the imine side-chain from carbon to nitrogen to produce *N*,*N*-disubstituted amides.[73]



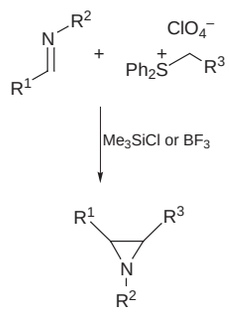
Scheme 15

4 Aziridine chemistry

4.1 Formation of aziridines

This section covers methods which were reported specifically for the synthesis of aziridines. A number of procedures in which an aziridine was first formed, and then ring opened have also been published, and are included in the following section. Benonite clay catalyses the formation of aziridines from amines and 1,2-dibromides.[74]

The reaction of carbonyl compounds with sulfur ylids to give epoxides is a well known reaction. A nitrogen analogue of this reaction has now been reported as shown in **Scheme 16**. Thus reaction of an imine with an allylic, prop-2-ynylic, or amide stabilized sulfonium salt in the presence of a Lewis acid was found to give the corresponding aziridines. In some cases, the reaction was stereoselective, giving only the *cis*-isomer of the aziridine, but in other cases a 1 : 1 mixture of the *cis* and *trans*-isomers was produced. In addition to achiral examples, a camphor derived, chiral sulfur ylid was employed and formed aziridines with an ee of up to 70%.[75]

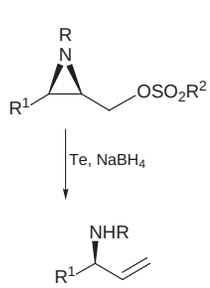


Scheme 16

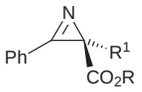
A Michael addition followed by intramolecular substitution of ammonia onto an α-bromo-α,β-unsaturated carboxylate was employed in a synthesis of ethyl (2*R*,3*S*)-3-vinylaziridine-2-carboxylate.[76] Nitroalkenes react with $NsONHCO_2Et$ in the presence of calcium oxide to form α-nitroaziridines. The mechanism is again likely to be a Michael addition of the hydroxylamine derivative followed by intramolecular displacement of the *p*-nitrobenzenesulfonate (nosylate) group.[77]

4.2 Formation of amines and amides by the ring opening of aziridines or aziridinium ions

Azirine **31** reacts with methylmagnesium bromide to give the corresponding aziridine with the methyl and carboxylate groups *cis* to one another. Subsequent hydrogenation of the aziridine ring occurs with cleavage of the benzylic carbon-nitrogen bond to give α-substituted-β-methyl phenylalanine derivatives, and can be controlled to give either diastereomer of the product.[78] Samarium iodide induces the reductive ring opening of 2-acylaziridines by selective cleavage of the C2-N bond, providing a regioselective synthesis of β-amino acids.[79] Reaction of aziridinemethanol sulfonate esters with tellurium and sodium borohydride results in ring opening of the aziridine and elimination of the sulfonate to produce allylic amines as shown in **Scheme 17**.[80] Vinyl phosphonates react with Ph-I=NTs in the presence of copper(II) triflate to give aziridinylphosphonates which can be hydrogenated to give α-amino phosphonic acids.[81]

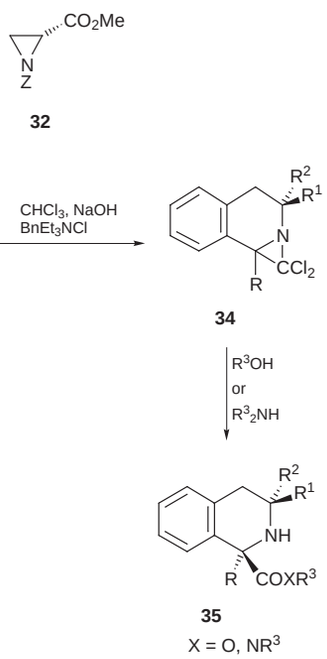


Scheme 17



31

Treatment of *N*,*N*-dialkyl phenylglycinols with mesyl chloride gives an aziridinium ion which is ring opened by methylamine regioselectively by cleavage of the benzylic carbon-nitrogen bond to give chiral diamines.[82] Arylmethyl amines react with two equivalents of *N*-trifluoromethanesulfonyl aziridines to give bis[(2-trifluoromethylsulfonamido)ethyl] amines.[83] Aziridine **32** is ring opened by amines to provide an asymmetric synthesis of 2,3-diaminopropanoic acids.[84] The addition of dichlorocarbene to imines **33** gives dichloroaziridine derivatives **34** which undergo ring opening when treated with alcohols or amines to give derivatives of 1,2,3,4-tetrahydroisoquinoline-1-carboxylic acid **35** (**Scheme 18**), which are versatile, conformationally constrained, phenylalanine derivatives.[85]



32

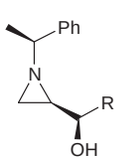
33 **34**

35

X = O, NR^3

Scheme 18

The ring opening of aziridines **36** by acetic acid occurs at the least substituted carbon atom, providing a stereocontrolled synthesis of β-amino-γ-hydroxy acetates.[86] Ring opening of both aziridine **37** and the corresponding *trans*-substituted aziridine by acid nucleophiles (TFA and methanesulfonic acid) occurs exclusively at the allylic carbon-nitrogen bond, and provides access to γ-hydroxy-δ-amino-α,β-unsaturated esters.[87] Protected, enantiomerically pure epoxy alcohols can be converted into the corresponding aziridines with inversion of configuration, by treatment with sodium azide followed by triphenylphosphine. Subsequent acylation of the aziridine and ring opening by phosphonic acid derivatives provides a route for the synthesis of enantiomerically pure, amide containing surfactants.[88] Acylation of chiral aziridine carboxamide derivatives **38** results in spontaneous rearrangement to 4-imido-4,5-dihydrooxazoles which can be hydrolysed to provide a diastereo- and enantio-controlled synthesis of β-hydroxy-α-amino acids (**Scheme 19**).[89]



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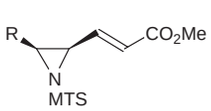
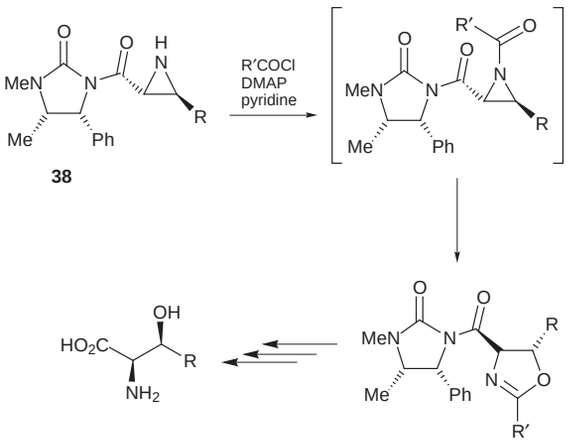


MTS = 2,4,6-trimethylphenylsulfonyl

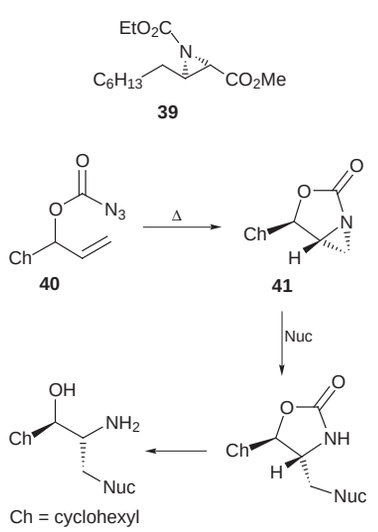
37



38

Scheme 19

In the presence of amberlyst-15 ion exchange resin, sodium bromide reacts stereoselectively with aziridine **39** by cleavage of the C2-N bond, giving the corresponding α-bromo-β-amino acid derivative.[90] Organocuprates react with *N*-phosphorylated aziridines at the least hindered carbon atom, providing a route for the synthesis of primary *sec*-alkylamines.[91] Thermolysis of acyl azide **40**, results in the predominant formation of aziridine **41** as shown in **Scheme 20**. Subsequent ring opening of the aziridine occurs at the primary carbon atom, and further deprotection provides a stereospecific synthesis of β-amino alcohols.[92] The ring opening of aziridines **42** by a range of heteroatom based nucleophiles (Cl^-, BnS^-, N_3^-, RCO_2^-, MeO^- and I^-) provides a synthesis of *erythro*-β-substituted aspartic acid derivatives. Most of the examples reported employed racemic aziridines **42**, but the *N*-methanesulfonyl derivative could be obtained enantiomerically pure, and thus allowed an asymmetric synthesis of β-substituted aspartates.[93] Reaction of bis-aziridine **43** with nucleophiles can lead to the formation of *cis*-2,5-disubstituted pyrrolidines or *cis*-3,6-disubstituted piperidines by ring opening of one of the aziridines followed by intramolecular reaction of the resulting nitrogen nucleophile with the other aziridine. Under S_N2 reaction conditions, pyrrolidines are formed since the nucleophile reacts at the terminal end of the aziridine, whilst under S_N1 reaction conditions, the nucleophile reacts at the other carbon atom of the aziridine ring leading to piperidines.[94]



39

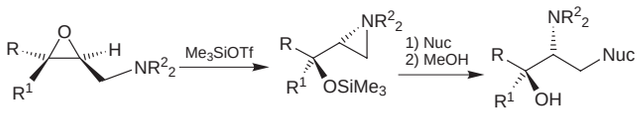
40 **41**

Ch = cyclohexyl

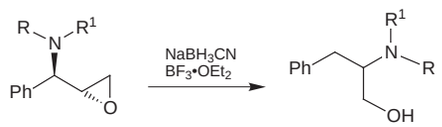
Scheme 20

Reaction of an enantiomerically pure tertiary amino epoxide with trimethylsilyl trifluoromethanesulfonate results in an aza-Payne rearrangement to the corresponding trimethylsilyloxy aziridinium ion which can subsequently be regiospecifically

42 43



Scheme 21

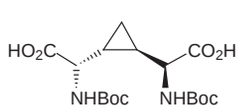


Scheme 22

ring-opened by nitrogen based nucleophiles, providing access to 2,3-diamino alcohols as shown in **Scheme 21**.[95] Thiiranium salts also react with primary amines and imines derived from amino acids, producing β-amino sulfides.[96] An aza-Payne rearrangement also occurs when 1-phenyl-1-oxiranylmethanamines are treated with sodium cyanoborohydride and boron trifluoride, the aziridinium salt being reduced by cleavage of the benzylic carbon-nitrogen bond to form β-phenylethylamines as shown in **Scheme 22**.[97]

5 Synthesis of amines and amides using the Evans and related auxiliaries

Over recent years, Evans has developed a versatile synthesis of α-amino acids based on the asymmetric amination of enolates bearing an imide chiral auxiliary. This methodology has been used in the preparation of the arylglycine derivatives found in vancomycin,[98] in the synthesis of cyclopropane-1,2-bis(glycine) derivatives such as **44**,[99] and in the preparation of β-substituted prolines.[100] An asymmetric β-amino acid synthesis in which the Evans auxiliary is reacted with $BzNHCH_2Cl$ has been reported by Hintermann and Seebach as shown in **Scheme 23**.[101]



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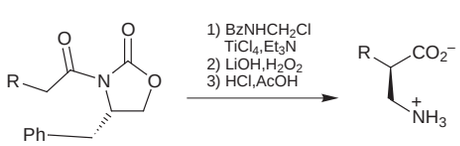
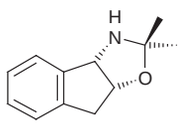


Scheme 23

A variation on the Evans synthesis starts with an α,β-unsaturated ester attached to the chiral auxiliary. Michael addition of an organocuprate, followed by electrophilic amination then allows the enantio- and diastereo-selective synthesis of β-substituted α-amino acids. In recent years, this approach has been used by Hruby and co-workers to prepare a variety of constrained amino acids. In the latest developments, the synthesis of all four stereoisomers of β-isopropyl-2′,6′-dimethyltyrosine,[102] β-isopropyltyrosine,[103] β-(isopropyl)-phenylalanine,[104] and β-methyl-3-(2′-naphthyl)alanine[105] have been reported.

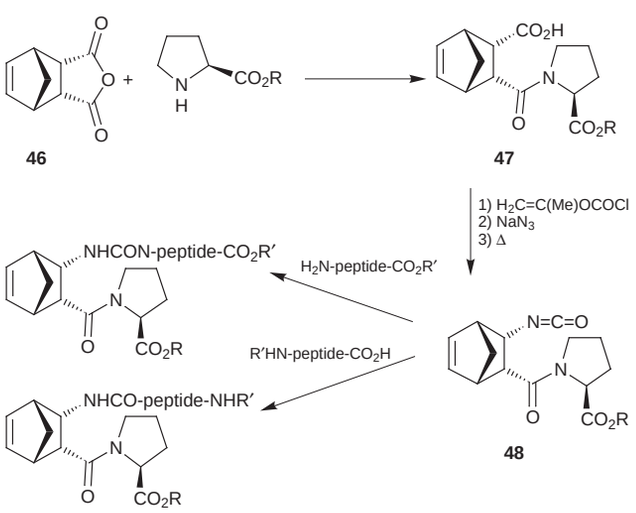
A number of modifications of the Evans auxiliary have been developed in recent years, and Zheng *et al.* have described the use of compound **45** as an auxiliary for asymmetric amino acid synthesis. Compound **45** is reacted with an acid chloride to form an imide which can be deprotonated to generate an enolate which reacts with TsON(Li)Boc to give an α-*N*-Boc-amino derivative with asymmetric induction. The chiral auxiliary can be removed by acidolysis to give the desired amino acid.[106]



45

6 Synthesis of amines and amides using the Curtius and related rearrangements

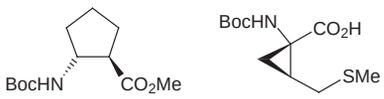
A Curtius rearrangement was the key step in a synthesis of peptides and pseudo-peptides containing an *endo*-(2*S*,3*R*)-2-amino-3-carboxynorborn-5-ene residue. The synthesis started from *endo*-norborn-5-ene-2,3-dicarboxylic anhydride **46** which reacted with proline esters as reported previously to give the corresponding enantio- and diastereo-merically pure acids **47**.[107] A Curtius rearrangement subsequently gave isocyanates **48** which reacted with the *N*- or *C*-terminus of peptides to give pseudo-peptides and peptides respectively as shown in **Scheme 24**.[108] The same approach could be used to prepare pseudo-peptides incorporating a β-aminocyclopropane carboxylic acid; a difficult amino acid to prepare and incorporate into peptides since the amino acid contains an electron donating and an electron withdrawing group on the cyclopropane ring and so is prone to ring opening.[109]



46 **47** **48**

Scheme 24

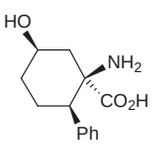
One of the most versatile ways of using a Curtius rearrangement for asymmetric amino acid synthesis is to start with a chiral ester of a malonate derivative. Alkylation (or dialkylation) of the malonate enolate occurs with asymmetric induction, and the subsequent manipulations can allow the Curtius rearrangement to be carried out on either carbonyl (or nitrile) of the malonate derivative, thus giving access to both enantiomers of an α-amino acid from the same precursor. This approach has been used to prepare α-allyl-α-amino acids[110] and (*S*)-α-methylaspartic acid.[111] Amongst the other amino acids that have been prepared by Curtius rearrangements are enantiomerically pure methyl *trans*-2-[*N*-(Boc)amino]cyclopentane-1-carboxylate **49**,[112] (1*S*,3*R*)-1-aminocyclopentane-1,3-dicarboxylic acid,[113] β-fluoro-α-aminocyclopropane carboxylic acid,[114] both diastereomers of 2,3-methanomethionine **50**,[115]



49 **50**

and enantiomerically pure *cis*-3-amino- (or 3-methylamino)-2,2-dimethylcyclobutane carboxylic acids.[116]

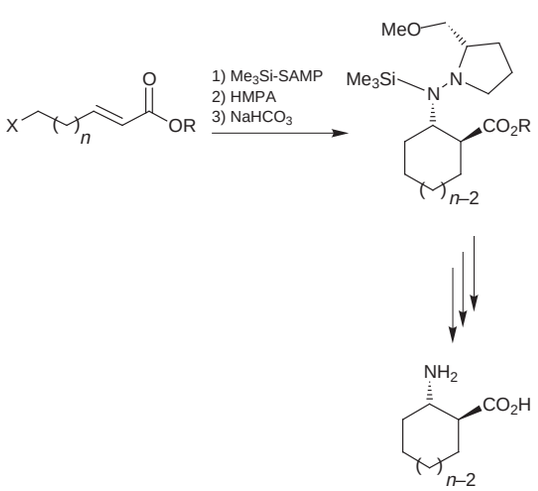
The synthesis of both racemic and enantiomerically pure 4-amino[2.2]paracyclophanes as well as *N*-alkylated derivatives has been achieved, employing a Curtius rearrangement on the corresponding racemic or enantiomerically pure paracyclophane-4-carboxylic acid.[117] An asymmetric synthesis of cyclic γ-hydroxy-α-amino acid **51** has been reported in which the amino group is formed from the corresponding amide *via* a Hoffman rearrangement.[118] A Hoffman rearrangement was also employed to convert asparagine derivatives into 2,3-diaminopropionic acids.[119]



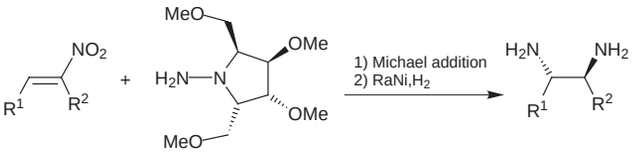
51

7 Synthesis of amines and amides by Michael additions

Over recent years, Davies and co-workers have developed an asymmetric synthesis of β-amino acids based on the asymmetric Michael addition of chiral amines [usually *N*-(1-phenylethyl)benzylamine] to α,β-unsaturated esters. This chemistry was used in a formal total synthesis of thienamycin.[120] Davies has also reported the use of *N*-(1-phenylethyl)allylamine as the nucleophile in this reaction, and has shown that unsaturated β-amino acids can be prepared from this reagent. However, six steps are required to remove the allyl and 1-phenylethyl groups from the nitrogen atom which will limit the utility of the process.[121] Chiral hydrazines also undergo asymmetric Michael additions, and the reaction of Me_3Si-SAMP with α,β-unsaturated esters bearing a leaving group in the ω-position, leads to cyclic β-amino acids (**Scheme 25**).[122] The Michael addition of hydrazine **52** to nitroalkenes followed by hydrogenation provides a route for the diastereo- and enantio-selective synthesis of 1,2-diamines as shown in **Scheme 26**.[123] A tandem Michael addition approach to the synthesis of pyrrolidines from nitroalkenes and allylic amines has also been developed (**Scheme 27**). The adducts could be further transformed into azanorbornanes.[124]

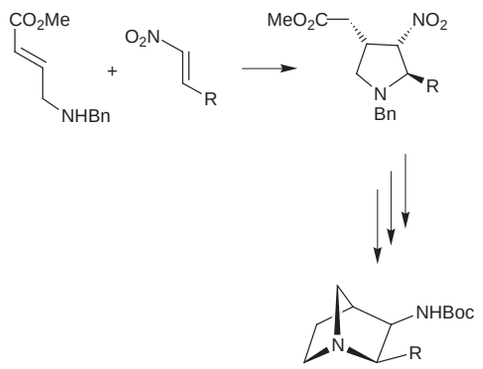


Scheme 25



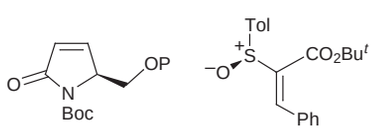
52

Scheme 26

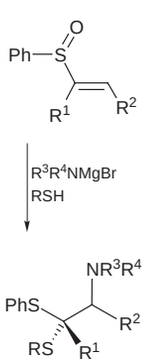


Scheme 27

The stereochemistry of the addition of lithium dialkylamides to γ-substituted α,β-unsaturated esters has been investigated, and the nature of the γ-substituent was found to determine the stereochemistry of the conjugate addition.[125] Michael addition of benzylamine or *N*-benzylhydroxylamine to lactams **53** occurs diastereoselectively *anti*- to the alkoxymethyl substituent. If *N*-benzylhydroxylamine is used as the nucleophile, then treatment of the product with $TiCl_3$ gives the corresponding benzylamine.[126] The Michael addition of amines onto dimethyl 2-(phenylseleno)fumarate provides 2-phenylseleno-3-amino-succinates, the de values of which vary from 0 to 62% depending on the nature of the amine, and favour formation of the *anti*-diastereomer.[127] An enzyme (3-methyl aspartase) has been reported to catalyse the *anti*-addition of primary and secondary amines to 2-alkyl fumaric acids, providing access to enantio- and diastereo-merically pure 3-alkyl aspartic acid derivatives.[128] Amines undergo Michael addition to enantiomerically pure sulfoxide **54**, providing a synthesis of β-amino acids with ee values of 49–89% after removal of the sulfoxide group (by treatment with samarium iodide) from the initial adducts.[129] Vinyl sulfoxides react with magnesium amides in the presence of thiols to give β-amino-dithioacetals as shown in **Scheme 28**.[130]



53 **54**

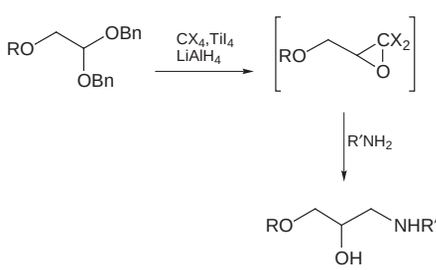


Scheme 28

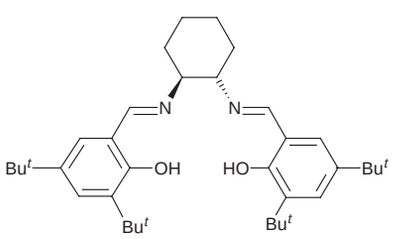
In the presence of copper cyanide, trimethylsilyl chloride and lithium chloride, α-(*N*-Boc-amino)organolithium reagents undergo Michael additions to α,β-unsaturated esters, thioesters, and imides, providing access to γ-amino carboxylic acid derivatives.[131] Enantiomerically pure *N*-Boc-α-(trimethylstannyl)-amines can be transmetallated with butyllithium in the presence of (−)-sparteine, and the resulting chiral lithium enolates react with a variety of electrophiles including: carbon dioxide, alkyl halides, and α,β-unsaturated carbonyl compounds to give precursors of α-, β-, and γ-amino acids respectively in 25–94% ee.[132]

8 Transition metal mediated processes

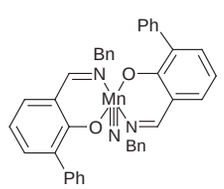
Reaction of a β-alkoxy acetal with a tetrahalomethane in the presence of titanium(IV) iodide and lithium aluminium hydride gives an epoxide intermediate which can be trapped by a primary amine to produce 2-hydroxy-3-alkoxy amines as shown in **Scheme 29**.[133] In recent years, Jacobsen and co-workers have shown that the manganese complexes of chiral salen-ligands such as **55** will catalyse the asymmetric epoxidation of alkenes. In an extension of this work, it has now been shown that just 2 mol% of the chromium(III) chloride complex of **55** will induce the desymmetrization of cyclic, *meso*-epoxides by trimethylsilylazide. Subsequent reduction of the azido group provides *trans*-β-amino alcohols with ees of 96–99%.[134] The manganese salen complex **56** reacts with styrene and trifluoroacetic anhydride to give *N*-trifluoroacetyl 2-amino-1-phenylethanol.[135]



Scheme 29



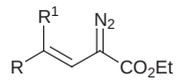
55



56

Reaction of a β-phenyl-α,β-didehydroamide with an aromatic amine in the presence of 2-methylpropanal, oxygen and a polyaniline supported cobalt(II) salen catalyst results in the formation of *anti*-β-arylamino-α-hydroxy amides, and has been used in a stereoselective synthesis of bestatin analogues.[136] Treatment of a lithium dialkylamide with an organocuprate produces an intermediate amidocuprate complex which can be oxidized by oxygen to produce a tertiary amine.[137] Reiser and co-workers have developed an asymmetric synthesis of 3-aminocyclopropane-1,2-dicarboxylic acid derivatives which can be incorporated into peptides. The key steps in the synthesis are the cyclopropane formation by addition of a copper carbenoid to an *N*-acyl pyrrole, followed by ozonolysis of the remaining double bond within the pyrolidine ring.[138]

Reaction of diazoesters **57** with aniline in the presence of rhodium acetate results in insertion of the rhodium carbenoid into the aniline N-H bond, giving the corresponding *N*-phenyl-



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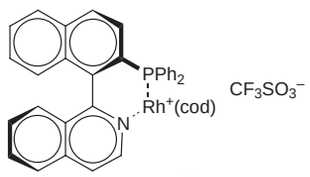

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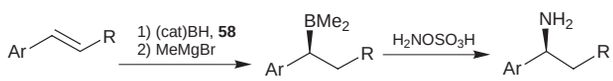
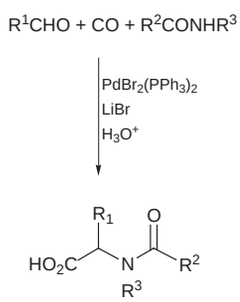


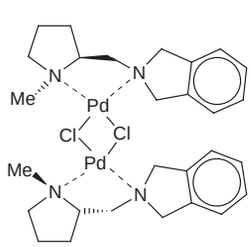
Scheme 30

amino ester.[139] A rhodium catalyst [Rh(cod)Cl_2] was also found to be effective in the hydroaminomethylation of alkenes (*i.e.* reaction of the alkene with a primary or secondary amine, CO and H_2), thus providing a method for the conversion of primary amines to secondary amines, and secondary amines to tertiary amines.[140] Catalyst **58** has been found to catalyse the asymmetric addition of catecholborane to aromatic alkenes as shown in **Scheme 30**. Subsequent replacement of the catechol group by methyl groups and treatment of the resulting trialkylborane with hydroxylamine sulfate produces amines with ee values of 77–98%.[141]

Palladium catalysis has been employed in a novel synthesis of racemic *N*-acyl α-amino acids.[142] Thus reaction of a primary or secondary amide with an aldehyde and carbon monoxide in the presence of $PdBr_2(PPh_3)_2$, LiBr and acid results in coupling of the three components to give *N*-acyl amino acids as shown in **Scheme 31**. One of the difficulties with polyamino compounds is obtaining selectivity for reaction at just one of the many NH and NH_2 groups present. However, reaction of a polyamine with an aryl bromide in the presence of $PdCl_2$(dppf) and sodium *tert*-butoxide results in arylation of just one primary amino group.[143] An asymmetric catalyst (**59**) for the rearrangement of allylic imidates to allylic amides has been reported by Overman and co-workers. The best ee obtained was 55%.[144]



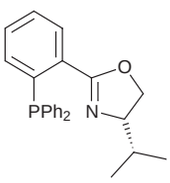
Scheme 31



59

Reaction of a racemic allyl acetate with phthalimide in the presence of palladium(0) and ligand **60** gives *N*-phthaloyl allylic

amines without allylic transposition and with high ee values. The alkene unit of the products can be oxidatively manipulated, and the phthaloyl group removed to give either α- or β-amino acids.[145] A very similar process has been reported by Sudo and Saigo, using chiral ligand **61** and benzylamine as the nucleophile.[146] Similarly, reaction of glycine derivative **62** with a (BINAP)Pd(0) catalyst and sodium malonates gives β-carboxy-aspartic acids with up to 86% ee.[147] Another class of ligands for palladium catalysed allylic amination are represented by structure **63**, and were found to give ee values >99.5% when ethyl 1,3-diphenylprop-2-enyl carbonate was reacted with benzylamine in the presence of 3 mol% of the palladium complex of **63**. However, high enantiomeric excesses were only obtained when tetrabutylammonium fluoride was also added to the reaction mixture.[148] Reaction of the benzophenone imine of amino-acetonitrile with a vinyl epoxide in the presence of $Pd(PPh_3)_4$ results in the formation of ε-hydroxy-γ,δ-unsaturated α-amino acid precursors by allylic ring opening of the epoxide by a formal nitrile enolate.[149]



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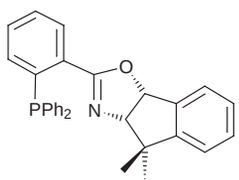

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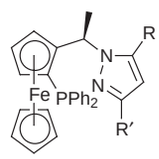

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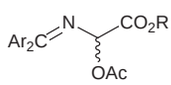
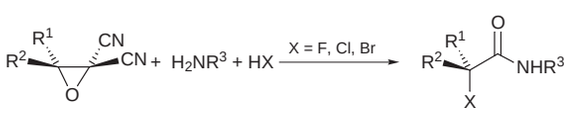


62

9 Derivatization of amines and amides

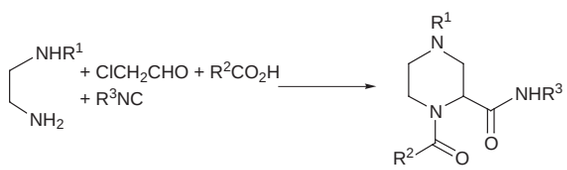
Only those methods that are not suitable for inclusion in earlier sections of this review are included here. Primary aromatic amines are selectively monomethylated by dimethyl carbonate in the presence of faujasite X- and Y-type zeolites.[150] Hydroxamic acids are oxidized by sodium periodate to acyl nitroso compounds which react with primary amines to give amides by elimination of NO.[151] Ultrasound has been found to induce the reaction between enantiomerically pure ferrocenyl acetates and primary amines, producing C_2-symmetric ferrocenylamines.[152] Vinyl epoxides react with amines stereo- and regio-specifically, reaction occurring exclusively at the allylic end of the epoxide and with inversion to give β-amino alcohols.[153] Treatment of 2,3-epoxy amines with magnesium bromide or iodide results in the formation of 3-hydroxyazetidines by regioselective opening of the epoxide ring by halide at the end remote to the amine, followed by intramolecular displacement of the halide.[154] Reaction of an α,α dicyano-epoxide with an amine and a halogen acid provides a route for the synthesis of α-halo amides as illustrated in **Scheme 32**.[155]



Scheme 32

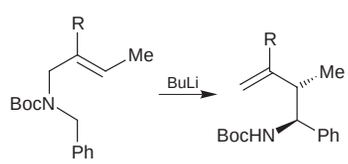
N-Alkyl 2,4-dinitrobenzenesulfonamides undergo Mitsunobu reactions with alcohols in the presence of triphenylphosphine and diethyl azodicarboxylate and react with alkyl halides in the presence of potassium carbonate to give *N*,*N*-dialkyl 2,4-dinitrobenzenesulfonamides. The 2,4-dinitrophenylsulfonyl group can be removed under relatively mild conditions with thioacetic acid or propylamine, giving a synthesis of secondary amines.[156] Chiral cyanohydrins are readily available by a variety of methodologies, and it has been shown that they undergo a Mitsunobu reaction with *N*-Boc(β-trimethylsilylethylsulfonyl)-amine/PPh_3/DEAD to give chiral amino nitrile derivatives, which on treatment with dilute hydrochloric acid are hydrolysed and deprotected to give α-amino acids.[157] A variety of enantio- and diastereo-merically pure β-amino alcohols including ephedrine derivatives have also been prepared from chiral cyanohydrins.[158]

Full details have been reported of Myers' recently disclosed asymmetric amino acid synthesis in which the pseudo-ephedrine amide of glycine (or *N*-alkyl- or *N*-Boc-glycine) is deprotonated and alkylated. In addition to allowing the synthesis of simple amino acids *via* an α-anion synthon, the glycine unit can also be dialkylated on nitrogen and carbon, thus allowing the asymmetric synthesis of cyclic α-amino acids.[159] A synthesis of racemic piperazine-2-carboxamides by an Ugi reaction involving a 1,2-diamine, chloroethanal, an isocyanide, and a carboxylic acid has been reported as shown in **Scheme 33**.[160]

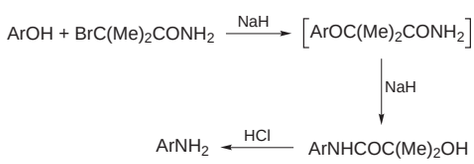


Scheme 33

Treatment of an *N*-Boc-*N*-benzyl allylic amine with butyllithium results in a diastereoselective [2,3]aza-Wittig rearrangement to give Boc-protected amines (**Scheme 34**).[161] A synthesis of aryl amines from phenols has been developed which employs a Smiles rearrangement as shown in **Scheme 35**.[162]



Scheme 34



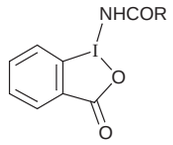
Scheme 35

10 New methods in peptide synthesis

Nature synthesizes peptides and proteins starting from the *N*-terminal amino acid. In contrast, the chemical synthesis of peptides and proteins conventionally starts with the *C*-terminal amino acid to avoid racemization. It has been shown however, that the cysteine protease clostripain can be used to catalyse the synthesis of peptides from amino esters starting from the *N*-terminal amino acid.[163] Subtilisin BPN has been shown to couple peptides containing non-coded amino acids and peptidomimetics.[164] Ultrasound irradiation has been reported to be advantageous in peptide couplings using unprotected amino acids and anhydrides, *N*-carboxyanhydrides, or active esters.[165]

11 Miscellaneous methods

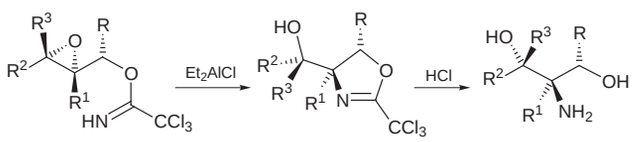
The allylic amination of alkenes can be accomplished with Ts-N=S=N-Ts, and this was used in a synthesis of chiral azanoradamantanes.[166] Reagent **64** reacts with adamantane in the presence of benzoyl peroxide to give *N*-adamantyl amides,



64

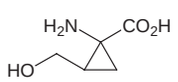
and with *N*,*N*-dimethyl aryl amines to give *N*-(*N*-aryl-*N*-methyl)-aminomethyl amides.[167] Aryllithium reagents and aromatic Grignard reagents react with allyl azide on the terminal nitrogen to give an intermediate which on acidolysis provides aromatic amines.[168]

A procedure for the asymmetric synthesis of β-amino alcohols from epoxides starts from enantiomerically pure epoxy alcohols. Formation of the corresponding trichloroacetimidate followed by diethylaluminium chloride induced cyclization and hydrolysis gives the β-amino alcohols as shown in **Scheme 36**. In suitable cases, the products can be further manipulated into α-substituted serine derivatives.[169]



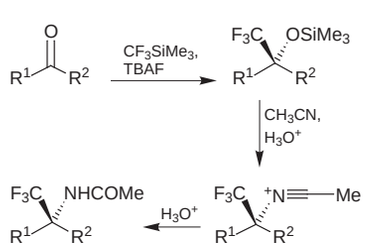
Scheme 36

A synthesis of (1*S*,2*R*)-1-aminocyclopropane-1,2-dicarboxylic acid (2,3-methanoaspartic acid) derivatives (and their enantiomers) in which the 2-carboxylate group is introduced as a 1,2-diol to prevent problems with ring-opening of the cyclopropane ring has been reported.[170] A very similar approach in which 2-hydroxymethyl-1-amino-cyclopropane carboxylic acid **65** is used as a precursor to 2,3-methanoaspartic acid has also been reported. Compound **65** is incorporated into a peptide by standard methodology, then the hydroxymethyl group is oxidized by sodium periodate–ruthenium trichloride to give the conformationally constrained aspartic acid derivative.[171]



65

Aromatic amines can be produced by the reduction of aromatic azides with Bakers' yeast.[172] Ketones react with trifluoromethyltrimethylsilane in the presence of TBAF to give silyl ethers which undergo a Ritter reaction with acetonitrile to produce trifluoromethylated amines as shown in **Scheme 37**.[173]



Scheme 37

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