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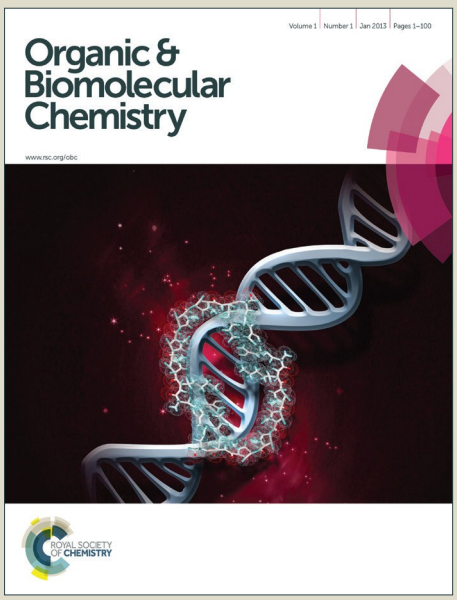

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COMMUNICATION

Chemoselective Room Temperature E1cB *N-N* Cleavage of Oxazolidinone Hydrazides from Enantioselective Aldehyde α-Hydrazination: Synthesis of (+)-1,4-Dideoxyallonojirimycin

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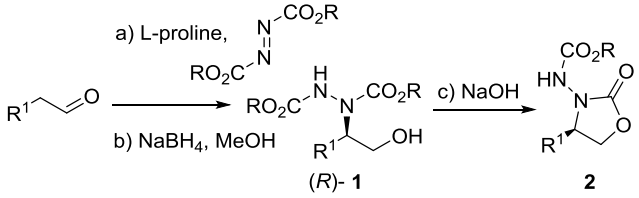
Jasmin Ferreira,[a] Sophie C. M. Rees-Jones,[a] Valerie Ramaotsoa,[a] Ath'enkosi Msutu[a] and Roger Hunter*[a]

Room temperature E1cB *N-N* cleavage of oxazolidinone hydrazides via *N*-alkylation with diethyl bromomalonate and potassium or caesium carbonate as base in acetonitrile is presented. The new method has a much improved chemoselectivity, which is illustrated by a concise total synthesis of the piperidine iminosugar (+)-1,4-dideoxyallonojirimycin.

The enantioselective organocatalyzed α-hydrazination reaction of an aldehyde with an azodicarboxylate first reported simultaneously by Jørgensen[1] and List[2] in 2002, has proved to be a powerful methodology for the production of α-hydrazino alcohols **1** in enantioenriched form following aldehyde reduction with sodium borohydride to limit racemization. The sense of product enantioselectivity as (*R*)- from L-proline has been rationalised as indicative of an assisted transition state,[3] while the alcohol **1** is invariably cyclised into its oxazolidinone hydrazide **2**, which can then undergo *N-N* reductive cleavage to afford the enantioenriched oxazolidinone, Scheme 1.

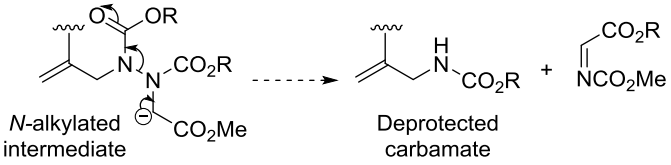


Scheme 1 Enantioselective Amination protocol[1,2]

The reaction[4] has been extended to the α-hydrazination of ketones[5] as well as to α-substituted aldehydes for the production of α-tertiary amine motifs.[6] In addition, the methodology has found extensive application as a key asymmetric step in several natural product syntheses[7] as well as tandem processes.[8] Although much effort has also gone into developing alternative catalysts,[9] by comparison, the manner in which the hydrazide *N-N* bond may be cleaved[10] to afford an α-amino alcohol ultimately has not received as much attention, particularly regarding chemoselectivity aspects. In this communication we present a room temperature, chemoselective and non-reductive procedure for the *N-N* cleavage of oxazolidinone hydrazides.

In the original work by Jørgensen and List[1,2] both groups made use of the dibenzyl derivative of the azodicarboxylate in conjunction with *N-N* hydrogenolysis using Raney nickel or a sequence of Pd/C followed by Zn as the catalysts. Raney nickel has primarily been the reagent of choice for *N-N* cleavage despite its pyrophoric and environmentally toxic nature. Other sequences specifically for the motif in question involve trifluoroacetic anhydride / samarium diiodide on the di-Boc derivative[11] and $NaNO_2$ / HCl on the deprotected hydrazide.[12] Less common reagents that have been used for a broader range of hydrazides, some with alkyl rather than acyl groups, include Na or Li / NH_3,[13] B_2H_6,[14] $NiCl_2{\cdot}2H_2O$-LiDTBB,[15] R_3Si-H,[16] SmI_2[17] as well as oxidative *N-N* cleavage.[18]

In 2009, inspired by earlier work by others on non-reductive *N-N* cleavage in both cyclic and acyclic systems, Magnus reported[19] a potentially elegant solution via an E1cB protocol. Acyclic allylic hydrazides derived via an ene reaction between an alkene and an azodicarboxylate underwent *N*-alkylation with methyl bromoacetate and Cs_2CO_3 in acetonitrile at 50 °C. Following isolation, the *N*-alkylated product underwent *N-N* cleavage to the carbamate via an E1cB mechanism by refluxing in acetonitrile with further Cs_2CO_3 over 18h, Scheme 2.



Scheme 2 The E1cB step in the Magnus *N-N* cleavage protocol

Subsequent work by Magnus[20] on the synthesis of α-carbamoyl ketals required the use of the stronger base NaH (in diglyme at 50 °C) in order to achieve full conversion. Moreover, Magnus did not report an application of his conditions to oxazolidinone hydrazides formed from the enantioselective α-amination reaction of Jørgensen and List. We have recently reported acetals as a new

[a] Chemistry Department, University of Cape Town, Rondebosch 7701, Cape Town, South Africa.
E-mail: Roger.Hunter@uct.ac.za; Fax: +27 21 650 5195; Tel: + 27 21 650 2544.
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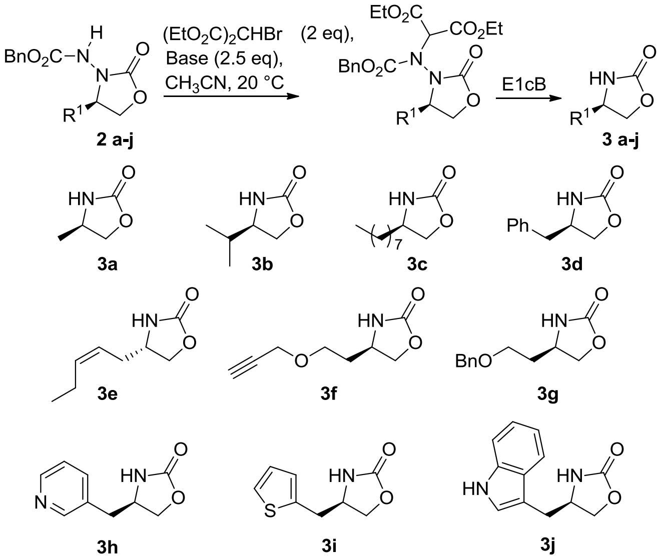
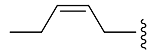
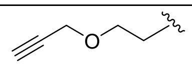
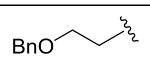
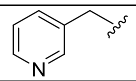
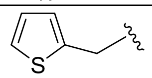
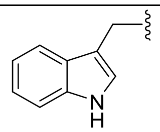
chemotype for enantioselective α-hydrazination using a combination of aqueous monochloroacetic acid and proline tetrazole as promotors,[21] in which conversion of the hydrazide to the corresponding oxazolidinone was required, and implementation of the Magnus protocol selected. To this end, decanal was chosen as a model substrate, which was converted into its α-hydrazino alcohol in near quantitative yield and in 92% ee using dibenzyl azodicarboxylate (DBAD) as the aminating electrophile under the conditions shown in Scheme 1 (R = Bn; $R^1 = C_8H_{17}$) according to the original work.[1,2] The oxazolidinone hydrazide **2c** (see Table 1) was easily obtained using NaOH in MeOH at room temperature following the borohydride step. However, application of the Magnus protocols[19,20] to oxazolidinone hydrazide **2c** gave poor results; Cs_2CO_3 and either methyl or ethyl bromoacetate in acetonitrile at reflux over two days failed to convert any of the starting material, while sodium hydride and ethyl bromoacetate in DMF at 50 °C for 24h afforded only a modest yield of 46% (after isolation and chromatography), with a much lower yield (16%) using diglyme as solvent. Green[22] has recently also pointed out difficulties with the Magnus procedure for α-hydrazinated intermediates in their synthetic studies on Sphingosine analogues. Consideration of the Magnus methodology (Scheme 2) reveals that the reaction sequence demands a good electrophile in the *N*-alkylation step, as well as stabilization of the intermediate carbanion in the E1cB step for fast turnover. Hence, it was decided to study the use of diethyl bromomalonate as the alkylating agent in the hope of accommodating improvements in each of the steps. To our satisfaction, the reaction with the model oxazolidinone hydrazide **2c** with diethyl bromomalonate and either Cs_2CO_3 or K_2CO_3 in acetonitrile at room temperature gave the desired oxazolidinone **3c** in good to excellent yield (see entry 3 in Table 1). Optimization of the reaction with respect to the equivalents of reagent, solvent and temperature revealed the use of 2 equivalents of bromide and 2.5 equivalents of base in acetonitrile at room temperature to be optimal. Hydrazides from DBAD proved to furnish higher yields of oxazolidinone than those from DIAD (Diisopropyl azodicarboxylate) or D*t*-BAD (Di-*tert*-butyl azodicarboxylate).

A range of oxazolidinone hydrazides **2a-j** varying the R^1 group were then prepared using the conditions shown in Scheme 1; structures of the oxazolidinone products are shown in Table 1. Yields for the hydrazination were generally in excess of 90% and ees greater than 85% except for the pyridine and thiophene derivatives **2h** and **2i** respectively, which gave low ees at room temperature. These could be improved to 62% for the pyridine case **2h** by using L-proline tetrazole at 0 °C, while the thiophene reaction pleasingly improved to 85% ee running the reaction with L-proline at 0 °C (see supplementary data). The library of hydrazides **2a-j** were then subjected to the new *N-N* cleavage conditions and the results shown in Table 1. A number of chemoselectivity issues deserve mentioning. Firstly, several of the substrates would not be candidates for existing methodology using hydrogenolytic *N-N* cleavage, particularly using Raney nickel (entries 5-7 and 9). Secondly, the conditions avoid the use of the strong base sodium hydride,[20] which presents hazard issues on a large scale; moreover, the reaction works efficiently at room temperature. The difference in reaction time for the two bases was also interesting, with Cs_2CO_3-mediated reactions generally faster than those run using K_2CO_3, most likely due to a greater basicity. Similarly, entry 10 could be carried out without protecting the indole nitrogen, presumably in view of the moderately basic nature of the base. Importantly, reactions run at higher temperature gave lower yields, tlc studies suggesting a side reaction of the bromomalonate reagent. Finally, no loss of enantioselectivity going from the oxazolidinone hydrazides **2a-j** (ees given in brackets in Table 1) to the cleaved oxazolidinones **3a-j** was incurred, within the accuracy of the chiral HPLC determination.

Table 1 Scope of the bromomalonate-based methodology for *N-N* cleavage[a]

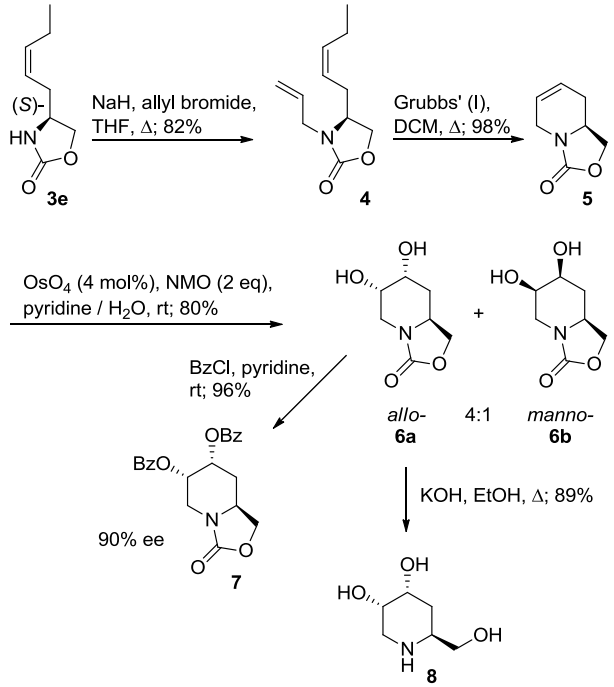


Entry	R^1	Prod	Base[b]	Time (h)	Yield[d] (%)	ee of 3[e,f] (%)
1	Me	**3a**	**A**	20	89	90[g]
			B	48	61	(90)
2	*i*-Pr	**3b**	**A**	20	95	92[g]
			B	48	62	(91)
3	*n*-Oct	**3c**	**A**	48	64	88[g]
			B	23	90	(92)
4	Bn	**3d**	**A**	3	82	91
			B	20	84	(90)
5		**3e**[c]	**A**	5	84	90[h]
			B	24	84	(88)
6		**3f**	**A**	4	81	87[g]
			B	24	80	(87)
7		**3g**	**A**	5	84	92
			B	24	82	(90)
8		**3h**	**A**	2	90	61
			B	24	75	(62)[i]
9		**3i**	**A**	6	81	88
			B	24	81	(85)[j]
10		**3j**	**A**	2	76	84
			B	24	78	(85)

[a] Substrates **2a-j** were synthesized according to the conditions in Scheme 1 (full data in supplementary information) on a 0.26 mmol scale of reaction with stoichiometries as indicated. [b] A = Cs_2CO_3; B = K_2CO_3. [c] Reaction carried out with D-proline to obtain the (*S*)-enantiomer as major. [d] Isolated yields after column chromatography. [e] Measured by chiral HPLC on a Daicel Chiralcel OD or a Daicel Chiralpak AD column. [f] ees of hydrazides **2a-j** shown in brackets. [g] ee established via the Cbz derivative. [h] ee established via

dibenzoate **7** (Scheme 3). [i] Reaction run with L-proline tetrazole at 0 °C. [j] Reaction run with L-proline at 0 °C.

The application of the methodology to substrates incorporating heteroaromatic rings such as thiophene opens up access to medicinal chemistry libraries for diversity-oriented synthesis[23] that would not be accessible via existing Raney nickel-based methodology. By way of illustration of a target-based synthesis with diversity-oriented synthesis possibilities, the product **3e** (entry 5) obtained from a commercially available olefin and enriched in its (*S*)-enantiomer from D-proline, was used in a concise total synthesis of the piperidine iminosugar 1,4-dideoxyallonojirimycin, Scheme 3. Such targets have shown promise in recent years as mimics in carbohydrate-related metabolic pathways, with possible therapeutic application as anti-diabetic, anti-cancer and anti-bacterial agents.[24] To this end, reaction of **3e** (obtained by E1cB elimination in 84% on a 0.25 mmol scale; 64% on a 3.3 mmol scale) with allyl bromide and sodium hydride in THF at reflux for 12 h furnished the *N*-allylated derivative **4** in 82 % yield. Subsequent ring-closing metathesis[25] using the Grubbs' first-generation RCM catalyst in DCM furnished the bicycle **5**[26] in virtually quantitative yield after a flash-filtration. Finally, dihydroxylation with catalytic OsO_4 and NMO as co-oxidant in a pyridine / water mix produced *allo*- and *manno*-1,4-dideoxynojirimycins as oxazolidinones **6a** and **6b** respectively in an 80% combined yield and a 4:1 ratio.[26,27] The ee of the major diol **6a**[26] as ascertained via its dibenzoate **7**, was found to be 90% by chiral HPLC, which was in good agreement with that of oxazolidinone hydrazide **2e** (88%). Oxazolidinone **6a** was then hydrolysed to the target unprotected iminosugar **8** in 89% yield by refluxing in ethanolic KOH for 12 h. The NMR data obtained for **8** agreed with those from the literature.[27c] To conclude on this part, we have achieved a concise six-step synthesis of (+)-1,4-dideoxyallonojirimycin in high overall yield and in a highly enantioenriched form from a commercially available starting material. This synthesis would not have been possible using existing methods because of chemoselectivity issues.



Scheme 3 Synthesis of (+)-1,4-dideoxyallonojirimycin **8**

In conclusion, building on an earlier E1cB protocol by Magnus,[19,20] an improved set of conditions based on bromomalonate rather than bromoacetate as activator has been developed for converting oxazolidinone hydrazides into their corresponding oxazolidinones under non-reductive conditions. The methodology allows use of either Cs_2CO_3 or K_2CO_3 as a green base in acetonitrile, and *N*-*N* cleavage proceeds at room temperature. Importantly, it significantly extends the chemoselectivity profile of organocatalytic enantioselective hydrazination methodology using an azodicarboxylate electrophile for converting aldehydes into chiral, non-racemic oxazolidinones in high ee overall. Such chiral α-secondary amine motifs provide pivotal building blocks in medicinal and pharmaceutical chemistry programmes.

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