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REVIEW ARTICLE

Microwave-induced ferrier rearrangement of hyroxy beta-lactams with glycals

Aparna Das[1,*], Ram Naresh Yadav[2], Bimal Krishna Banik[1,*]

[1] *Department of Mathematics and Natural Sciences, College of Sciences and Human Studies, Prince Mohammad Bin Fahd University, Al Khobar 31952, Saudi Arabia*

[2] *Veer Bahadur Singh Purvanchal University, Jaunpur 222003, India*

*** Corresponding authors:** Aparna Das, aparnadasam@gmail.com; Bimal Krishna Banik, bimalbanik10@gmail.com, bbanik@pmu.edu.sa

ABSTRACT

Microwave-induced organic methods are extremely useful in synthetic organic chemistry for the preparation of molecules. A combination of irradiation and high temperature is probably responsible to obtain the final product in an accelerated process. This review focuses on a crucial nucleophilic reaction using hydroxy beta-lactams as the starting compounds. Specifically, the reaction of *cis*- and *trans*-hydroxy beta-lactams with different types of glycals under microwave irradiation using iodine as the catalyst is explored. This reaction produces unstaturated glycosides through Ferrier Rearrangement.

Keywords: beta-lactams; synthesis; microwave; iodine; bismuth nitrate

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1. Introduction

The commercial preparation of beta-lactam antibiotics and penicillin was begun in the mid-1940s[1,2]. Numerous other structurally important beta-lactam antibiotics have been prepared since then[3,4]. These antibiotics are remarkably effective against infectious diseases[5,6]. Beta-lactams have additional applications for many humans. For example, they are serine protease and cholesterol inhibitors[7,8]. A few beta-lactams are used for the synthesis of diverse heterocycles of medicinal significance[9–11]. For instance, properly substituted hydroxy beta-lactams are used in the synthesis of anticancer drugs, Taxol and Taxotere[12–14]. The use of beta-lactams against human leukocyte elastase is known[15,16]. Our research group and others have explored the synthesis and biological evaluation of numerous anticancer beta-lactams[17–25]. Due to their promising biological activities, the preparation of new types of beta-lactams has been the target of researchers[26,27]. Many crucial procedures are available for the synthesis of beta-lactams[28]. These include Staudinger cycloaddition method[17,29–31], ester enolate-imine condensation[32,33], hydroxamate reaction[34], alkene-isocyanate procedure[35], and the alkyne-nitrone method[36,37], Catalytic asymmetric[38,39] and polymer-supported[40,41] preparation of beta-lactams are also achieved.

3-Hydroxy-2-azetidinones are crucial intermediates for the preparation of antibiotics, alkaloids, amino acids, and amino sugars. Chiral 3-acetoxy-1-(*p*-anisyl)-4-phenyl-2-azetidinone is an

intermediate present in the side chain of Taxol and Taxotere[12–14]. Our group has reported the preparation and biological evaluation of hydroxy beta-lactams in racemic and optically active forms as anticancer molecules[18,22,29]. It is known that chiral compounds do not demonstrate identical pharmacological and toxicological activities[42,43]. We have demonstrated diverse therapeutic potentials of different types of natural and non-natural compounds[44–62]. In our work, we have observed differences in the anticancer properties of two optically active beta-lactams having similar structures[22,63].

We describe here an acid-catalyzed stereospecific glycosides synthesis by reacting hydroxy beta-lactams with diverse glycals followig Ferrier rearrangement in a microwave oven. The stereoselectivity achieved in the glycoside forming process helps to have access to chiral compounds of definite stereostructures. A successful environmentally benign method is considered a part of green chemistry. Therefore, it is important to achieve highly stereocontrolled reactions and obtain greater yields of the products through catalytic methods. During our study, we have identified a few microwave-assisted organic reactions. Some reactions described in this review are conducted by microwave-mediated methods[64].

2. Microwave in organic synthesis

The application of microwave heating and irradiation in organic chemistry is a valuable method[65]. Recently, the use of microwave-mediated processes as an environmentally benign method for the preparation of organic substances has become extremely popular. The microwave-mediated method has several advantages compared to the regular heating procedure.

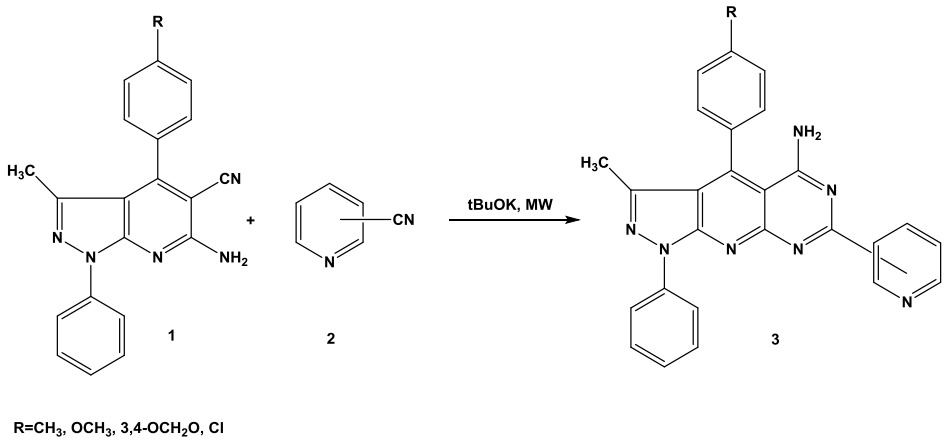
Many organic methods employing microwaves under homogeneous and heterogeneous conditions to synthesize heterocyclic molecules were reported[66–75]. Automated microwave is costly and therefore, it is not available in the undergraduate research lab. Therefore, a few research groups have been using a domestic microwave. However, the results from this study are comparable.

3. Results and discussion

Since this paper describes microwave-assisted nucleophilic reactions, some other examples of microwave-assisted synthesis *via* nucleophilic pathways are given here. Microwave-induced synthesis of pyrimidines, benzoselenophenes, xanthenes, steroids, thiazoles, imines, tetrazoles, triazoles, quinolines, indolizine, beta-lactams, Schiff bases, furans, quinoxaline, and coumarins are conducted. It is obvious that the nucleophilicity of the functional group has improved under microwave irradiation. Very few of these syntheses are given here.

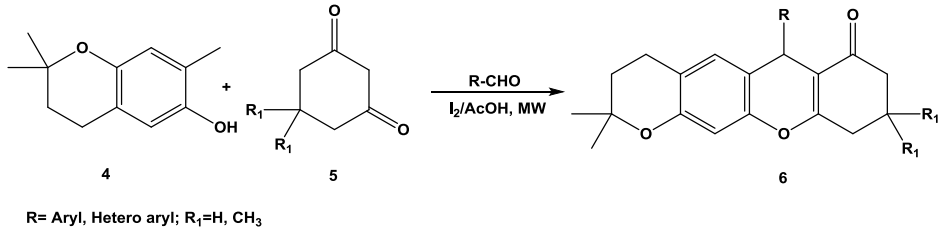
Pyrimidine is an aromatic organic molecule containing two nitrogen atoms at positions 1 and 3 of the ring. The pyrimidine nucleus is present in natural products and diverse synthetic compounds[76–78]. Because of the medicinal importance of pyrimidines, they have received attention from the scientific community.

Acosta et al. reported the microwave-mediated synthesis of new pyrazolo-pyrido[2,3-d]pyrimidines[79]. This was performed under solvent-free conditions *via* a cyclo condensation process between *o*-aminonitriles and cyanopyridines in the presence of tBuOK. The reaction between *ortho*-aminonitrile **1** (2-aminopyridine-3-carbonitrile) and 4-cyanopyridine **2** is given in **Scheme 1**. Many parameters were examined to identify the best conditions for the preparation of pyrimidine derivatives (**3**) by altering the media, temperature, and power of the microwave.



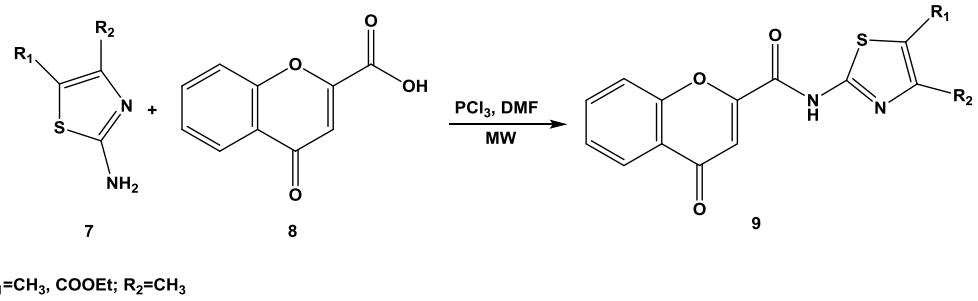
Scheme 1. Microwave-mediated synthesis of pyrimidine derivatives.

Scheme 2 demonstrated the one-pot procedure for the three-component condensation of 2,2-dimethylchroman-7-ol (**4**), aryl/heteroaryl aromatic aldehydes and 1,3-cyclohexadione/5,5-dimethyl-1,3-cyclohexanedione (**5**) for the preparation of pyranoxanthenes (**6**). To identify the best reaction conditions, the reaction was performed under various conditions. The results demonstrated that the microwave irradiation method was the best and faster.



Scheme 2. Synthesis of pyranoxanthenes.

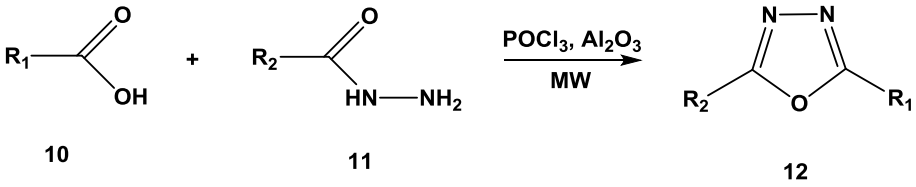
Cagide et al. reported the preparation of 4-oxo-*N*-(substituted-thiazol-2-yl)-4H-chromene-2-carboxamides (**9**) from chromone-2-carboxylic acid by two amidation procedures[80]. This new chromone–thiazole mixed molecules have served as ligands for human adenosine receptors. The preparation of chromone–thiazole hybrids is shown in **Scheme 3**. The chromone carboxamide derivatives were obtained by a condensation method that requires the activation of the chromone-2-carboxylic phosphorous oxychloride followed by the addition of the aminothiazole to the formed acyl chloride in a method mediated by microwave.



Scheme 3. Preparation of 4-oxo-N-(substituted-thiazol-2-yl)-4H-chromene-2-carboxamides.

Farshori et al. reported a fast and solvent-free preparation of 2,5-disubstituted-1,3,4-oxadiazoles (**12**) from fatty acid hydrazide in a microwave[81]. **Scheme 4** demonstrates the reaction. In this method, the reactions were performed by irradiating a mixture of fatty acid hydrazide with carboxylic acid, supported on neutral alumina, using phosphorus oxychloride. To identify the conditions for the preparation of oxadiazole bearing an

alkanyl/alkenyl/hydroxyalkenyl group, the molar proportions of the reagents, microwave irradiation time, and the power level of the microwave were examined.

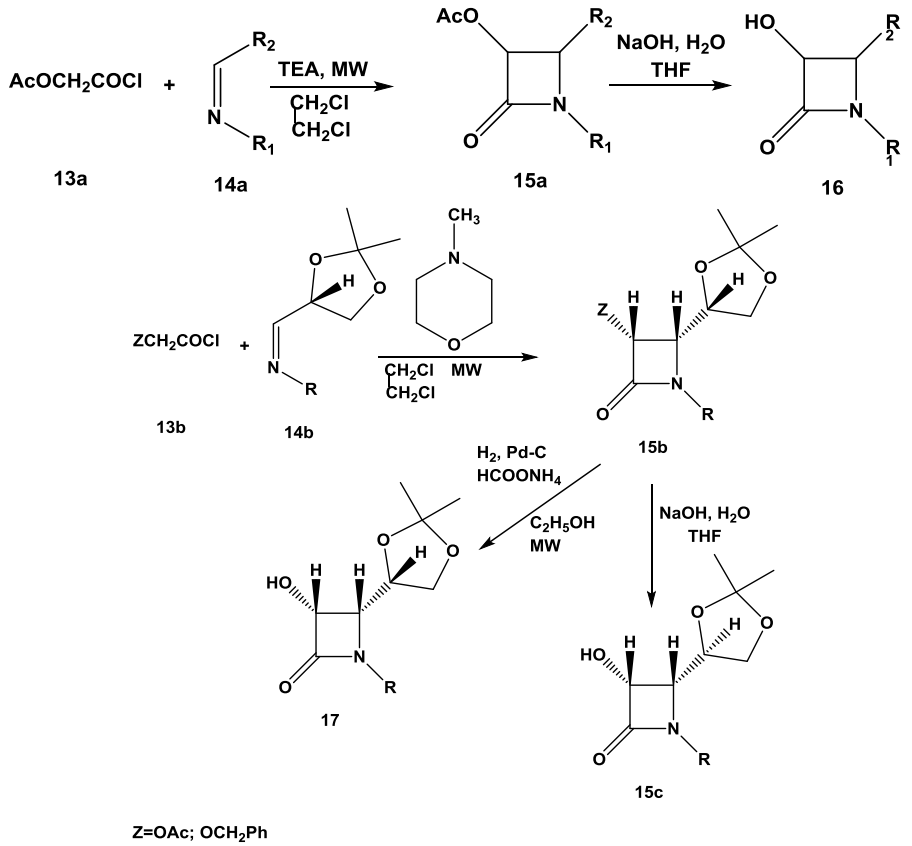


10 11 12

Scheme 4. Preparation of disubstituted-1,3,4-oxadiazoles.

3.1. Stereocontrolled synthesis of 3-hydroxy beta-lactams

In this paper, reactions of hydroxy beta-lactams with glycals are investigated. L-Glyceraldehyde acetonide and D-threonine were used for beta-lactam synthesis[82]. Further studies using D-glyceraldehyde acetonide and aldehydes obtained from other carbohydrates gave beta-lactams[83,84]. The reaction of acetoxyacetyl chloride and benzyloxyacetyl chloride (**13a** and **13b**) with the chiral imine **14b** in the presence of triethylamine gave *cis* beta-lactams in 70–80% yields. The acetoxy functionality in **13** was hydrolyzed to produce **16** and **17** with aqueous methanolic alkali to hydroxy compound without rupturing the ring. Alternatively, the benzyloxy group in beta-lactam **15b** on hydrogenation with 10% Pd/C and ammonium formate gave the chiral hydroxy beta-lactam **17**. The beta-lactam formation method with acid chloride was also conducted in a domestic microwave and identical results were seen. However, N-methylmorpholine was chosen to be a superior base than triethylamine. Dichloroethane was found to be a superior solvent than dichloromethane under microwave-induced method[85,86]. In another method, racemic hydroxy beta-lactam with an aromatic group at C4 group was synthesized from acetoxy beta-lactam **15a** and hydrolysis of the ester functionality. Hydrogenation of the benzyloxy beta-lactam **15b** with the aromatic group at C4 did not produce racemic hydroxy beta-lactam[12,87] (**Scheme 5**).

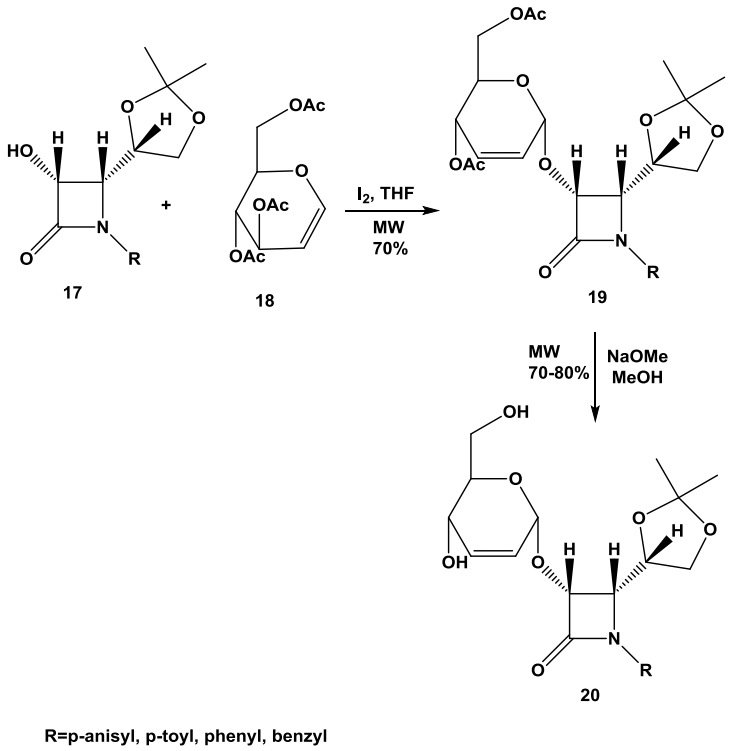


Scheme 5. Synthesis of 3-hydroxy beta-lactams.

3.2. Glycosylation of hydroxyl beta-lactams

Some glycals on reaction with water, phenol, and alcohol in the presence of an acid form 2,3-unsaturated pyranosides, and this reaction is called Ferrier rearrangement[88]. Acids include hydrochloric acid, sulfuric acid, $BF_3.Et_2O$, $SnBr_4$, $EtAlCl_2$, and iodonium dicollidinium perchlorate are used for this purpose[89,90]. The reaction of tri-O-acetyl-D-glucal was performed with the glycolic acid ester using $BF_3.Et_2O$ as the catalyst[91]. Based upon our research on iodine-induced methods, we conducted glycosylation of numerous alcohols following the Ferrier rearrangement[92,93]. Tri-O-acetyl-D-glucal in reaction with the glycolic acid ester and iodine gave a single glycoside. Hydrogenation[94] indicated the stereochemistry of the anomeric bond and it was found to be an axial glycoside[95]. This procedure opens up a new possibility of application in beta-lactam science.

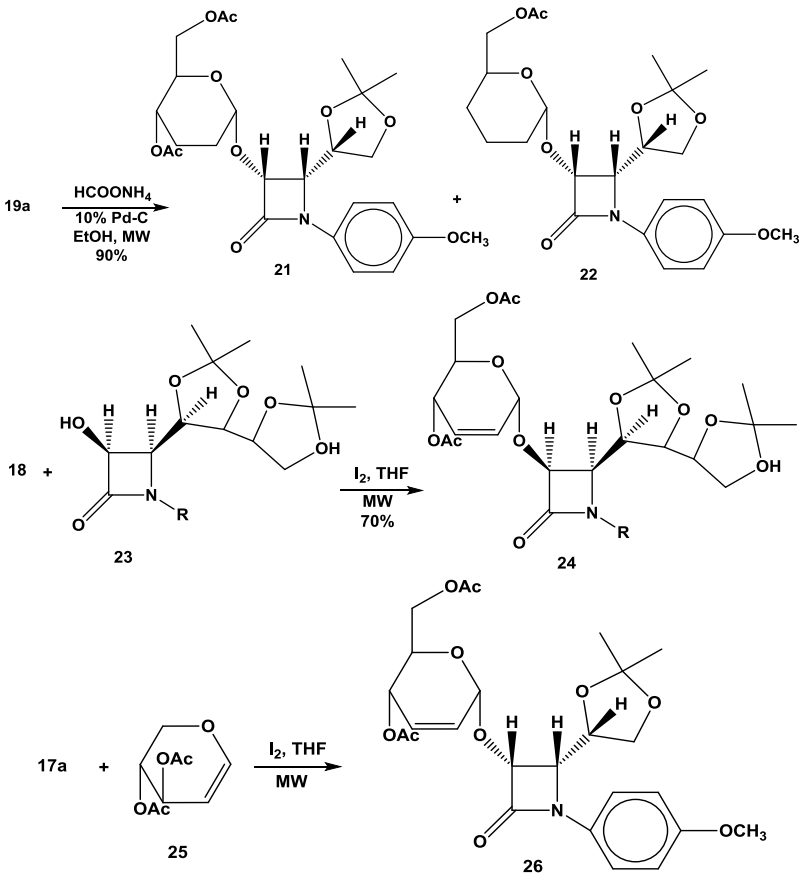
The results on iodine-induced glycosylation of alcohols are intriguing and therefore, we applied this procedure to optically active 3-hydroxy beta-lactam **17** with three asymmetric centers and commercial glycal **18**. These chiral beta-lactams **17** are synthesized following cycloaddition of **13** with **14b** and subsequent base-mediated hydrolysis (**15b**-**17**). A single glycoside **19** was formed in all of these examples in the presence of iodine. The reaction of **19** with sodium methoxide gave the dihydroxy molecule **20** in an excellent yield (**Scheme 6**).



Scheme 6. Glycosylation of optically active beta-lactams.

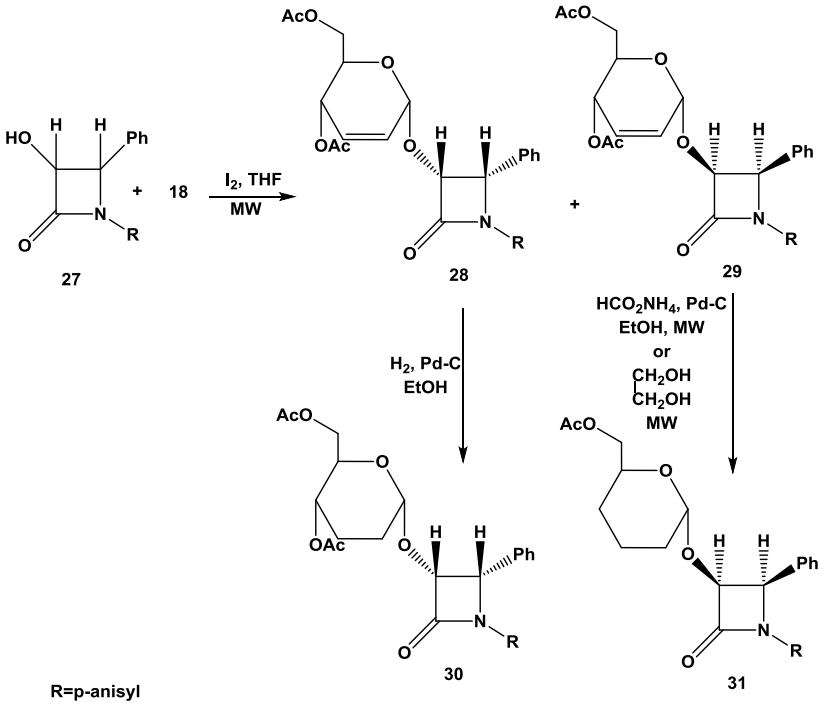
To know the stereochemistry of the anomeric carbon, analysis of the unsaturated glycoside **19** was not helpful. Therefore, to establish the configuration of the product, **19** was hydrogenated with ammonium formate in the presence of Pd/C. This reaction was also performed in a microwave oven[86]. Two compounds **21** and **22** were obtained from this reaction. The proton NMR data of the sugar-containing compounds **21** and **22** showed a small coupling constant data for the anomeric hydrogens. This value indicates that the glycosidic bond is axial in nature.

The glycosylation method was extended to another hydroxy beta-lactam **23** that has five chiral carbons. The reaction of **23** with **18** as shown above produced **24**. However, the reaction of **23** with **18** produced other products in a small amount (**Scheme 7**). An alpha-glycopyranoside was obtained by the reaction between **17** with glycal **25** that lacks functionality at C-5 center (**Scheme 7**).



Scheme 7. Hydrogenation of glycosides and glycosylation with L-sugar.

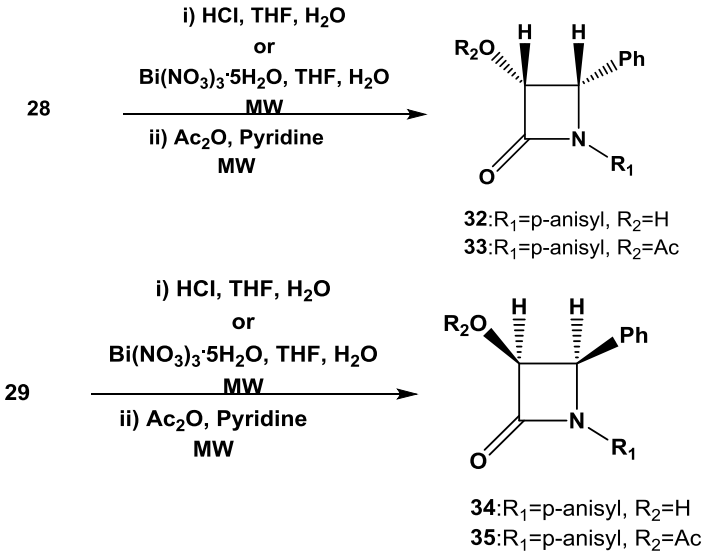
The single glycoside synthesis from chiral 3-hydroxybeta-lactams **17** and **23** opened up the possibility of preparing two enantiomers of racemic hydroxy-beta-lactams. To implement the process, glycosylation of racemic *cis*-3-hydroxy-4-phenyl-2-azetidinone (**27**) was performed. On reaction with triacetate of glucal, **27** produced a mixture of two diastereomers **28** and **29** in 55:45 ratio (**Scheme 8**).



Scheme 8. Chiral resolution.

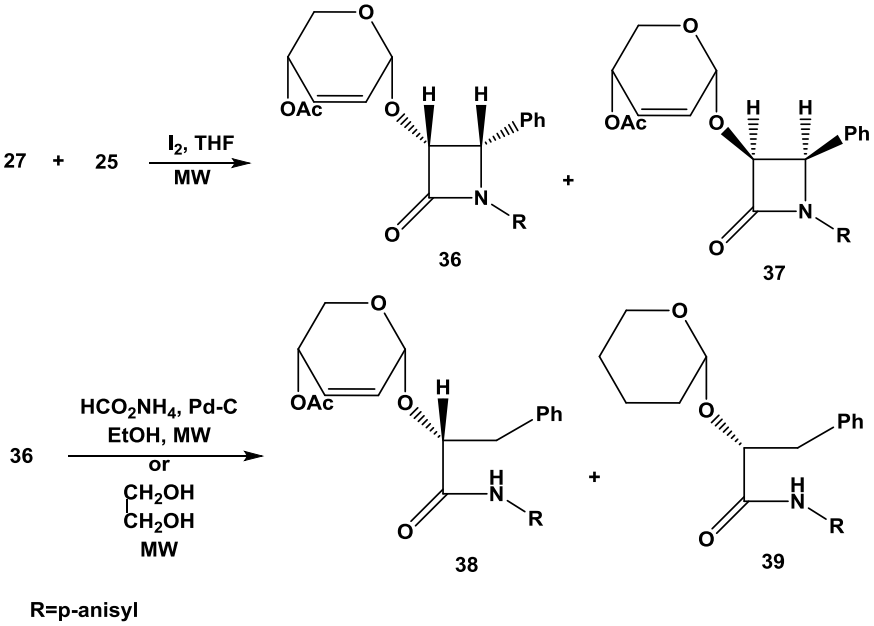
Hydrogenation of **29** gave a trideoxyglucoside **31**. The small coupling constant of the anomeric hydrogen confirmed the product as alpha-glycoside (**Scheme 8**).

The diastereomeric compounds **28** and **29** were isolated by column chromatography. These were reacted with aqueous hydrochloric acid or bismuth nitrate to deblock the sugar component. The hydroxy beta-lactams **32** and **34** were formed and acetates **33** and **35** were prepared (**Scheme 9**). Hydrochloric acid acted well in the breakage of the sugar in **29** and **28**. An aqueous solution of bismuth nitrate also worked well to obtain products **32** and **34**.



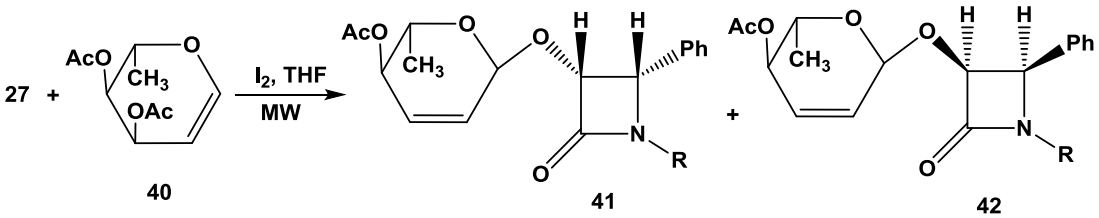
Scheme 9. Preparation of chiral hydroxyl-beta-lactams.

The reaction of **27** with **25** following the same method produced **36** and **37** in equal proportions. Catalytic hydrogenation of **36** and **37** produced amides **38** and **39** through the scission of the beta-lactam rings. The cleavage of the beta-lactam ring during hydrogenation was also identified by Ojima and our group[12,87]. Importantly, deacetoxylation was noted. Microwave irradiation of **36** and **37** gave the same products (**Scheme 10**).



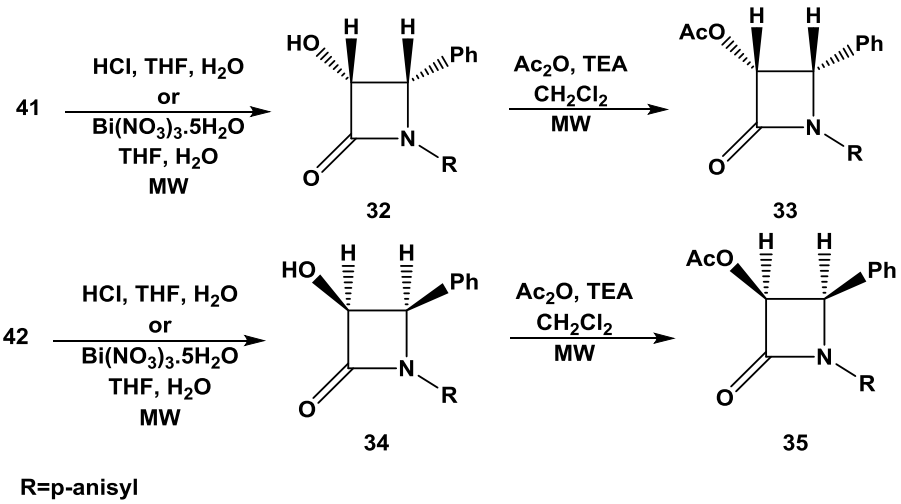
Scheme 10. Hydrogenation experiment on glycosylated beta-lactams.

To expand the chemistry, rhamnal acetate **40** was allowed to react with **27.** As usual, two compounds **41** and **42** were formed (**Scheme 11**). Following the previous results as described herein, the alpha-anomeric bond was identified in products **41** and **42**.



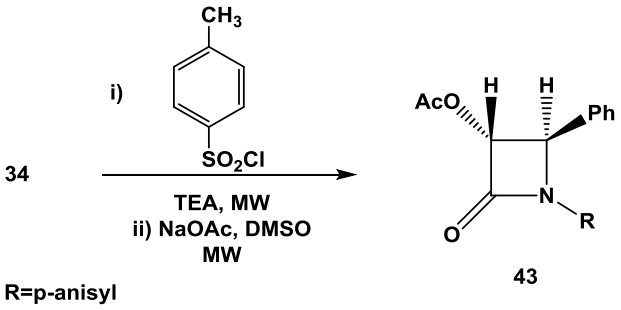
Scheme 11. Glycosylation of *cis*-hydroxy-beta-lactam.

Dilute hydrochloric acid-induced treatment converted the two diastereomers **41** and **42** into enantiomerically pure 3-hydroxy-2-azetidinones **32** and **34**. The breakage of the anomeric bond was also performed with an aqueous bismuth nitrate solution. Due to the ring strain in beta-lactams, ring cleavage is possible with strong acid treatment. The 3-hydroxy beta-lactams **32** and **34** were used to prepare acetoxy compounds **33** and **35** by reaction with acetic anhydride in the presence of pyridine (**Scheme 12**). Compound **33** was used for the preparation of the side chain of taxol and Taxotere[96].



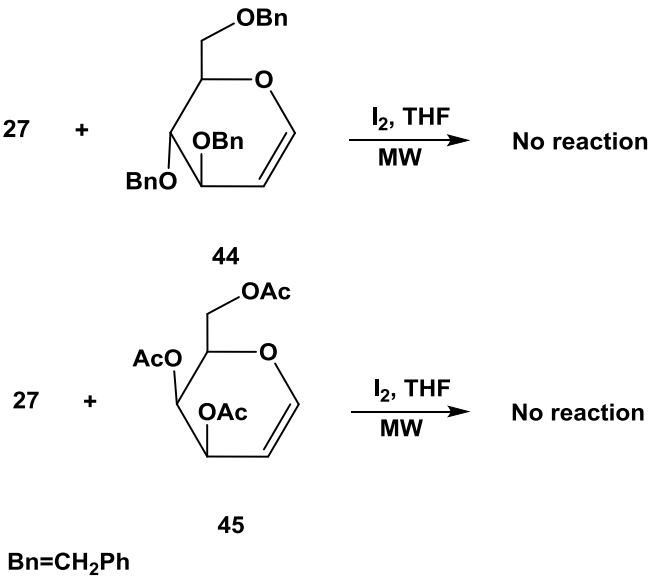
Scheme 12. Preparation of Taxol and Taxotere side chains.

The *trans*-beta-lactam **43** was obtained from *cis*-hydroxy beta-lactam **34** through tosylation and inversion with sodium acetate. NMR studies using chiral NMR shift reactant [Pr(hcf)$_3$] indicated that **43** is enantiomerically pure (**Scheme 13**).



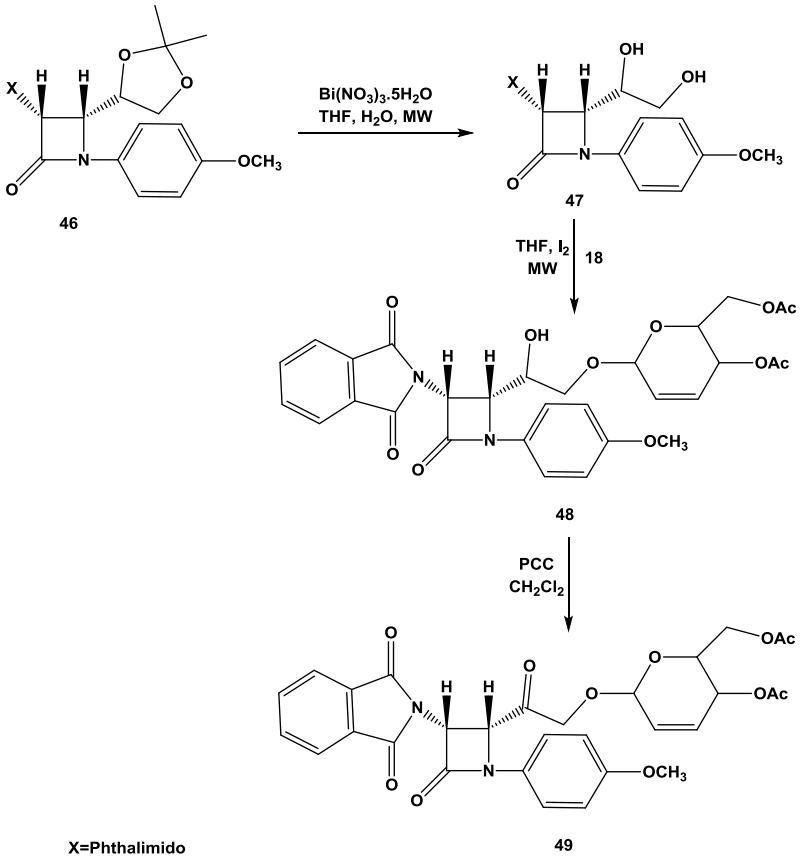
Scheme 13. Chiral *trans* beta-lactam.

To examine the method further, other glycals were also tested. A reaction with 3,4,5-tri-O-acetyl D-galactal (**45**) with alcohol **27** failed to produce the desired glycosides (**Scheme 14**). In addition, 3,4,5-tri-O-benzyl D-glucose (**44**) also did not produce the desired compounds.



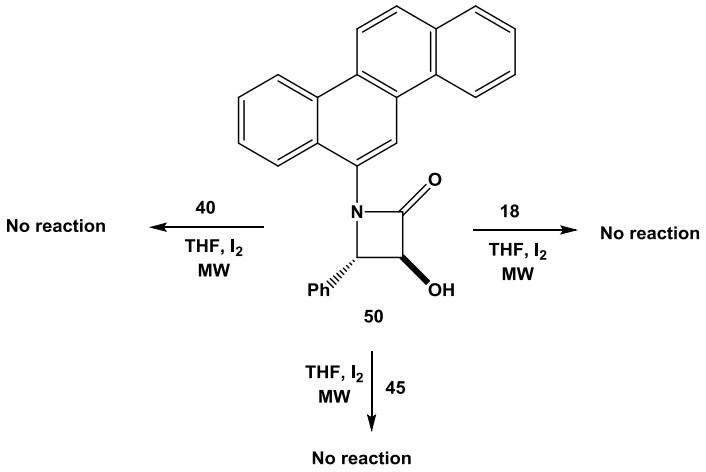
Scheme 14. Attempted glycosylation with different sugars.

The phthalimido alcohol **47** was made by acid-induced deprotection of **46** with bismuth nitrate. The diol **47** was subsequently reacted with **18** in the presence of the catalytic amount of iodine. The product was **48**. The primary alcohol reacted with **18**. This observation was confirmed by oxidation of **48** to **49**, a ketone (**Scheme 15**).



Scheme 15. Selective glycosylation.

Glycosylation of *trans*-hydroxy-beta-lactam **50** derived from chrysene with numerous glycals **18**, **40**, and **45** did not yield produced glycosides (**Scheme 16**). The *cis*-beta-lactam gave the desired products with a few glycals regardless of the absolute configuration at the C–3 and C–4 centers of the ring.



Scheme 16. Glycosylation of alcohol with chrysene-beta-lactam.

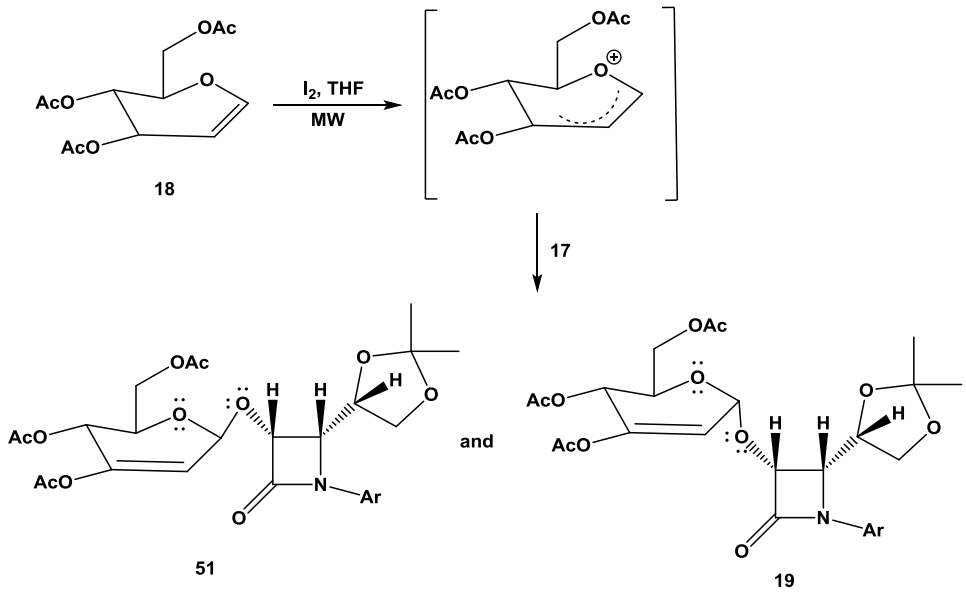
3.3. Mechanism of glycosylation reaction

The pathways of Ferrier rearrangement were studied[89,91]. One of these pathways involved an allylic isomerization of the unsaturated sugar **18** to a 2,3-didehydro derivative. To identify the mechanism, NMR monitoring of the reaction was performed. The glycosylation was made faster using a higher concentration of catalyst iodine. However, an excess of more than 30% of iodine produced a mixture of products. Keeping the reaction in the presence of iodine for a longer period reduced the yield of the products because many of the hydroxy beta-lactams were recovered from the reaction.

The ^{13}CNMR data indicated the peaks for the final compound, glycal, and hydroxy beta-lactam. No peaks due to iodinated intermediates or products were seen. It was argued that the intermediates are transformed into the product very fast. Interestingly, it was reported that the reaction of **18** with alcohol with N-iodosuccinimide or iodonium dicollidinium perchlorate produces 2-deoxy-2-beta-iodo-3-alpha-glycoside[97].

To know the role of iodine in this reaction, some other catalysts were tested. Nitrate salts of iron, copper, potassium, and zinc in reaction with alcohol and glycal produced no products. Hydrochloric acid, sulfuric acid, borontrifluoride, and stannic chloride were adopted in the reaction of diverse alcohols with a type success. A few neutral catalysts N-iodosuccinimide, N-bromosuccinimide, and iodonium dicollidinium perchlorate were employed. In some examples, the products were halogenated O-glycosides[97]. However, these catalysts were not used for the glycosylation of beta-lactam alcohol (e.g., **17** and **27**).

The formation of alpha-isomers from this reaction was new conceptually. For example, the reaction between **17** and **18** was studied with catalytic proportions of hydroiodic acid. The desired product **19** was isolated in less than 10% yields. Moreover, an aqueous solution of iodine did not produce glycosides **28** and **29**. This indicated a complex formation between iodine and the alkene group of the glycal **18**. The proton NMR spectral data of glycal **18** and iodine (10 mol%) in $CDCl_3$ showed a change at the olefinic portion of the glycal. These experiments suggested an allylic isomerization of glycal **18** with an intermediate dehydro sugar is involved followed by the nucleophilic pathway[63].



Scheme 17. Mechanism of the glycosylation.

These observations suggested an axial attack by the –OH group of the beta-lactam (**17**) on the anomeric carbon of the allylic carbonium ion of the glycal **19**. Such an attack through the equatorial pathway would have produced the beta-glycoside **51** (**Scheme 17**). This pathway was supported since no spectral data corresponding to beta-glycosides was detected by ^{13}CNMR.

Glycals **18**, **25**, and **40** gave stereoselective diastereomers. The glycals **45** and **44** synthesized from D-galactose and D-glucose were not fruitful. This failure indicated the role of the protective functionalities and their configurations. The configuration of the stereocenters of compounds **18** and **50** is identical with different protective groups. It seemed that the acetoxy group at C–4 of the sugar is suitably oriented for helping glycosylation. In contrast, glycal **45** obtained from D-galactose had an acetoxy functionality at C4 of the sugar unit with the opposite configuration. This participation was partially stopped in a nonparticipating group at C–4 of the sugar. For example, the galactal-derived glycals underwent Ferrier rearrangement under restricted situations[98].

4. Conclusion

Microwave-induced rearrangement reactions of racemic hydroxy beta-lactams with diverse glycals in the presence of iodine produce diastereomeric alpha-glycosides through an environmentally benign method. The synthesis of oxygen glycosides fused with a beta-lactam ring has not been demonstrated by any other groups. These glycosides were hydrolyzed to enantiomeric beta-lactams. This method proceeded well with chiral hydroxy-beta-lactams as well. Microwave-assisted hydrogenation was useful in the determination of the configuration of the anomeric center. The configuration and nature of the groups in sugar and beta-lactams were critical for effective glycosylation. The methods and compounds described here should find numerous applications in chemistry and chemical biology research.

Acknowledgments

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Conflict of interest

The authors declare no conflict of interest.

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