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PERSPECTIVE

Indium Metal-Induced Reactions: Synthesis of Biologically Active Molecules

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Indium metal has proven to be extremely useful in our research for the synthesis of diverse organic compounds of significant importance.

Keywords: Indium metal, Catalysis, Carbohydrate, Reduction, β-Lactam.

INTRODUCTION

Indium metal is highly economical and readily available. Our research group has been conducting indium-mediated useful reactions for the past 20 years. Synthesis of diverse organic molecules in chiral and achiral form is realized by indium-induced reactions very effectively. Most of these reactions are performed in aqueous ethanol. Moreover, indium-mediated reactions can be easily performed under microwave irradiation and ultrasound-mediated methods making these processes are attractive. Illustrative examples are provided here that include various compounds of interests.

RESULTS AND DISCUSSION

Indium metal is a versatile reagent in organic synthesis and readily available with extremely low cost. Importantly, this metal is not sensitive in water like most of the other metals. It has been possible to conduct numerous successful reactions with indium-mediated reactions for the preparation of diverse organic compounds. A short summary of reactions that are developed in our laboratory is discussed in this perspective.

Synthesis of β-lactams: Indium metal was used for the preparation of 3-unsubstituted β-lactams by the reaction of an imine with ethylbromoacetate. A variety of imines derived from monocyclic benzene derivatives were successfully employed. In some examples, a mixture of β-lactams and β-amino esters were produced [1]. However, β-amino esters were cyclized to the β-lactams by treatment with Grignard reagent. This method was then successfully applied for the preparation of 3,4-disubstituted β-lactams with varying stereochemistry using substituted ethylbromoacetate and imines [2]. A similar reaction with imines derived from multicyclic amines and carbonyl compounds failed to produce any β-lactams or β-amino esters. Indium and the bromoester formed a complex initially and this oragno indium species then reacted with the carbon center of the imines producing β-amino esters that cyclized to β-lactams under the reaction conditions. The author has been conducting research on the chemistry of β-lactams for the past 25 years [3].

Glycosylation reactions: Stereospecific synthesis of oxygen glycosides was performed with diverse alcohols and glycals using indium as a catalyst [4]. In general, this type of reaction required Lewis acids. The protective groups in glycal had a strong influence on the success of this reaction. For example, acetoxy protecting glycals had found to be better than the benzyl ether protecting group. Although the reasons for this superior activity of acetate-protected glycals over benzyl ether protected glycals were not established, it is believed that the leaving group properties of the acetate group are responsible for the success of these reaction. The electron withdrawing acetate group served as a better leaving group compared to the benzyl group and this facilitates the migration of the alkene bond to the 2, 3-position of the sugar and completes the reaction. However, the orientation of the 3-acetate group was very crucial. While glucose derived glycal was found to react successfully, galactose-derived glycal was partially effective. This observation was explained because of the anchimeric assistance exerted by the C-3 acetate group present in glucose derived glycal. Similar anchimeric assistance was not possible with glycal derived from galactose.

The stereochemistry of the resulting O-glycosides was found to be α. The mechanism of this indium-induced reaction was not established. This was explained by the bottom side attack of the alcohol to the anomeric carbon of the glycal. The top phase attack of the alcohol was not permissible because of steric hindrance and severe electronic repulsive forces at the transition state between the pyranose oxygen in the ring system of the sugar and the oxygen of the nucleophilic alcohol. The stereochemistry at the anomeric center was confirmed by a hydrogenation experiment. Reduction of the alkene bond in 2,3-unstatuared derivative produced a compound in which a low coupling constant (1-2 Hz) of the anomeric proton was detected. This method was subsequently used for the chiral resolution of *trans*-2-phenylcyclohexanol.

Indium metal was able to thioglycosylate bromoglucose peracetate at room temperature and this reaction was stereosepecific. The products were α-glycosides [5].

In contrast to the above observation, indium-mediated reaction of *trans* 3-hydroxy β-lactams derived from polyaromatic compounds with bromoglucose derivative produced two diasteroemeric separable β-O-glycosides [6]. The complete reversal of sterochemical results with bromo sugar with β-lactam alcohol derivatives was an interesting study. The O-glycoside bonds in the two diastereomers after separatio were cleaved by mild acid to the corresponding hydroxy β-lactams in both enantiomeric forms. These types of β-lactams were demonstrated unprecedented anticancer activity with great deal of selectivity against numerous cancer cell lines [7]. This study indicated that indium-mediated reaction of glycal with β-lactam alcohol and that of bromo sugar with alcohol followed different reaction pathways which are not well established [8]. Nevertheless, they produced stereo defined crucial molecules that are not reported in the literature previously.

Reduction of the aromatic nitro groups: Indium metal in the presence of ammonium chloride in aqueous ethanol was used for the successful reduction of the aromatic nitro groups and this reaction produced aromatic amines in excellent yield [9]. This reaction was found to be chemoselective. Many other reducible groups remained intact during this reduction process. Nitro groups present in polyaromatic compounds were reduced by this method to produce polyaromatic amines. Notably, this method was investigated using different amounts of starting nitro compounds (10 mg to 10 g). These polyaromatic amines were used for the preparation of diverse molecules that have demonstrated anticancer as well as antibacterial properties [10]. Other metal-induced methods for the reduction of nitro groups present in the polyaromatic molecules were difficult to achieve. For example, zinc and tin in the presence of acids failed to produce the required amines.

Reductive cyclizations: The method of the reduction of the aromatic nitro group with indium/ammonium chloride was extended for reductive cyclization toward the synthesis of many heterocycles [11]. Intramolecular reductive cyclization was performed with indium and ammonium chloride. The concept of reductive cylization was important. For example, a suitable substituted carbonyl group or electron deficient alkene functional group present in molecules were forced to undergo a cyclization reaction with the aromatic amino group that was generated in the same molecule by indium metal. All these reaction indicated a powerful role of indium as a reducing agent and also as a promoter for intramolecular nucleophilc addition reaction.

Reduction and subsequent intramolecular rearrangement: Reduction of the aromatic nitro group in β-lactam by indium metal was used for the preparation of heterocycles. For example, 3-(2-nitrophenoxy)-4-phenyl-2-azetidinone on reaction with indium in ethanol produced oxazines in excellent yield [12]. The nitro group was reduced first to an amino group. The resulting amino group attacked the 4-membered β-lactam ring through a nucleophilic pathway and opened the ring. Subsequent protonation produced oxazine. The success of this reaction was due to the strain present in the β-lactam ring. A similar reaction of 3-phenoxy-4-(2-nitrophenoxy)-2-azedidinone on reduction rearrangement produced a different type of heterocyclic compound.

Reduction of imines: Indium metal was used for the reduction of an imine bond and these reactions produced secondary amino compounds [13]. Depending upon the structure of the imines, a monoamine or a diamine was produced. Monoamines were the exclusive product when sterically hindered imines were used. In contrast, diamines were the product when steric crowding was not present in the imines. These observations were explained by a unique mechanism [14]. A single electron transfer from indium metal to the carbon-nitrogen bond produced an ion-radical. This initially formed ion radical followed multiple pathways. Another electron transfer from the metal produced reactive dianion and on protonation this dianion produced the monoamine as the only product. A coupling of the ion radical with another ion radical and subsequent protonation produced the diamine. Such coupling was not possible when sterically hindered imines were used as the substrates and therefore, products were the monoamines. A dimeric product was formed when the stability of the ion radical was high enough so that a coupling can take place. The stability of the ion radical was enhanced by the addition of a species (for example a Lewis acid) that can coordinate with the ion radical.

Condensed heterocycles: Isatin on reaction with 4-hydoxyproline in the presence of indium produced 3-pyrrole substituted 2-oxo indole system in excellent yield [15]. The amino and the carboxyl group reacted with the keto group of isatin in the presence of indium and formed an intermediate [16]. This intermediate formed azomethine ylide *via* decarboxylation. A dehydration reaction at high temperature was followed to yield a conjugated product. A 1, 5-proton shift occurred to afford the more stable zwitter ion intermediate which was easily transformed to the most stable product to gain aromatic character [17]. These series of reactions confirmed the capability of indium metal as an activator in catalyzing several processes.

Pyrrole synthesis: Reaction of primary amines with hexane 2,5-dione in the presence of indium metal produced 2,5-disubstituted pyrroles in excellent yield [18]. Unlike many other examples, strong acids were not required for this reaction. A series of nucleophilic attack by the amino group to the keto group and proton loss finally produced pyrrole following a mechanism advanced by Paal-Knorr. The pyrrole synthesis without the substituents was achieved using 2,5-dimethoxy-

tetrahydrofuran instead of hexanedione as the starting material. The dimethoxytetrahydrofuran on reaction with primary amine in the presence of indium metal afforded N-substituted pyrroles in excellent yield. This reaction produced reactive 1,4-butanedialdehyde in the media which upon condensation with primary amines finally produced N-substituted pyrroles [19]. Sterically hindered and less basic pyrroles also produced products, but these reactions required longer reaction time. Microwave irradiation method was found to accelerate the formation of pyrroles.

Microwave-induced method: Some of the reactions described herein with indium metal were performed in domestic and automated microwave oven. Significant acceleration of reaction rates was observed. In many instances, the isolation of products became easier than the conventional methods. The advantages of microwave-induced reactions over conventional methods were discussed in our previous publications [20].

Ultrasound-induced method: Like microwave-induced method, some reactions described here were conducted by indium-induced ultrasound-mediated process and yielded the products in excellent yield [21].

Conclusion

We demonstrated indium-induced reactions for the synthesis of diverse compounds under environmentally benign conditions [22]. Most of the indium-mediated reactions as described above produced products with high yields. Isolation of products from the reaction mixture was very convenient. Some reactions proceeded in water with 5 mol % of indium. The methods by indium were environmentally friendly. Considering the budgetary restrictions, our indium-mediated reactions are the perfect examples to cut down the cost of research maintaining the standard of good publications. In addition, unlike many other metal-mediated reactions, it was also found that indium-catalyzed reactions tolerated microwave-irradiation and ultrasound. On this basis, some of the indium-induced reactions under microwave irradiation and ultrasound-induced process were completed within minutes instead of hours/days. This study indicated that indium has many roles depending upon the type of reactions. It has great affinity to bromo compound, electron releasing properties and Lewis acid activities. A few compounds prepared by indium-mediated reactions demonstrated anticancer and antibacterial activities. In addition, the pertinent references with indium-induced method and related reaction by other methods should be very useful to prepare diverse organic compounds of biological interests along with the study of reaction mechanisms. These studies have a close correlation to our other studies on iodine-catalyzed reaction, cycloadddition reaction, β-lactams and polyaromatic compounds [23].

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