Review

Advances in indium triflate catalyzed organic syntheses

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Abstract

A short review on the use of $In(OTf)_3$ in organic syntheses has been described.
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Keywords: Indium triflate; Catalysis; Review; Lewis acid
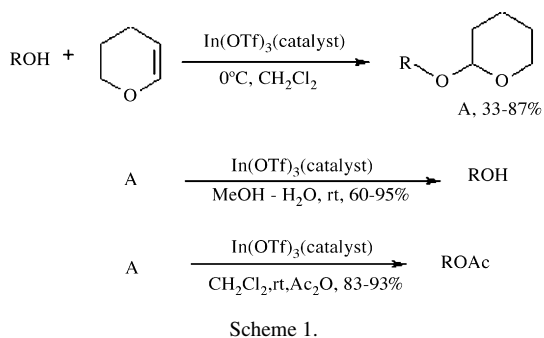
Contents

1. Introduction

Role of metal salts as Lewis acid catalysts in carbon–carbon bond formation and other organic transformations is well established [1]. Such catalyzed organic reactions are of great interest because of increased reactivities, selectivities and milder reaction conditions. Most of the conventional metal salts utilized as Lewis acids for many organic syntheses are not devoid of the disadvantage that most are moisture sensitive and must be used under strictly anhydrous conditions. The moisture sensitivity sometimes are so high that these react with water immediately rather than the substrates, hence getting hydrolyzed in the process before even taking part in the desired reaction. Moreover, their recovery and reuse also become formidable. Thus, search for moisture stable more efficient Lewis acids still continues.

Kobayashi et al. [2a,b] have categorized many metal salts on the basis of their relative stabilities in water. They, for the first time, made a correlation between the catalytic activity of a variety of metal salts in water through their 'Hydrolysis Constants' and 'Exchange Rate Constants' for substitution of inner-sphere water ligands from a model Mukaiyama–Aldol reaction.

During the last decade particularly after the review by Cintas [3], In(III) based carbon–carbon bond formation and other organic transformations have gained much attention. In

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Scheme 1.

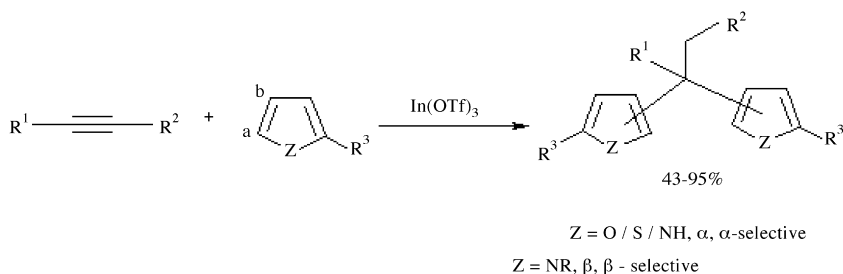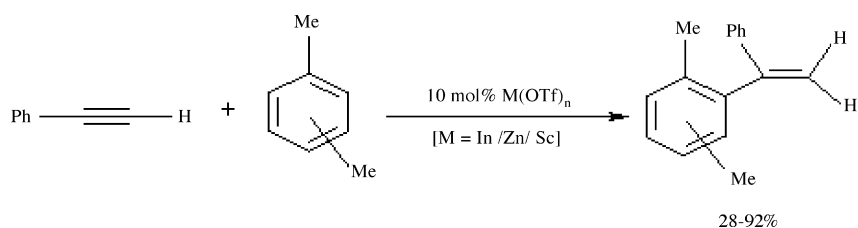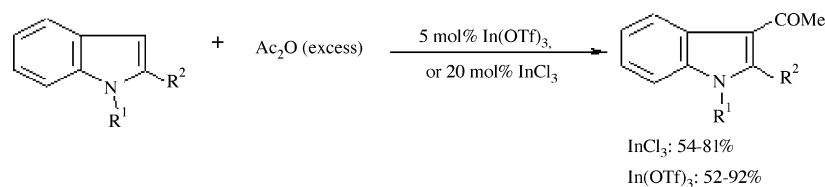## 2. In(OTf)$_3$ based organic syntheses

2.1. Formation of acetals and thioacetals

In(OTf)$_3$ has been used as a mild and chemoselective catalyst for the efficient and facile conversion of carbonyl compounds (aldehydes and ketones) into the corresponding 1,3-oxathiolanes [6]. Thioacetalisation of carbonyl compounds and transthioacetalisation of oxyacetals into thioacetals can also be achieved in high yields [7]. Tetrahydropyranylation of alcohols in dichloromethane and depyranylation of the products (A) in aqueous methanol or direct acetylation of the products (A) in the presence of acetic anhydride are possible using catalytic amount of In(OTf)$_3$ [8] (Scheme 1).

2.2. Aromatic electrophilic substitution

2.2.1. Friedel–Crafts reactions

Both InCl$_3$ and In(OTf)$_3$ were found to catalyze acetylation of indoles to the corresponding 3-acetyl indoles [9] (Scheme 2). Formation of bis-indolylmethane and indolylquinolide derivatives from the In(OTf)$_3$ catalyzed reactions are also reported.

A combination catalyst system from In(OTf)$_3$ and LiClO$_4$ had previously been found to be effective in acetylation of electron rich aromatics and alcohols [10]. The use of isoprenyl acetate as the acyl donor resulted in a cleaner conversion. Ding

accordance to the recent surge of interest in moisture stable metal triflates [2b,4], In(OTf)$_3$ has emerged as a promising catalyst for various types of organic reactions. Although, Frost and coworkers [5a] cited a few examples of In(OTf)$_3$ catalyzed organic reactions in their reviews on 'Indium catalyzed organic syntheses' and 'New applications of indium catalysis in organic synthesis' [5b] but no systematic compilations based only on In(OTf)$_3$ have so far been made. The present review is an attempt to summarize the published works concerning the use of In(OTf)$_3$ in organic syntheses. Reports where screening with different metal salts revealed lower efficacy of In(OTf)$_3$ compared to other Lewis acids are excluded from this dissertation.

Scheme 2.

Scheme 3.

Z = O / S / NH, α, α-selective

Z = NR, β, β - selective

Scheme 4.

Scheme 5.

Scheme 6.

et al. [11a] utilized In(OTf)$_3$ for Friedel–Crafts reaction of electron rich aromatic compounds with methyltrifluropyruvate in water. Report has been made of an efficient generation of alkyl benzophenones from alkyl benzene *via* In(OTf)$_3$ catalyzed microwave mediated Friedel–Crafts acylation [11b] with benzoyl chloride under solvent free condition.

Indium and other metal (Zn, Sc) triflates were used to catalyze Friedel–Crafts alkenylation of arenes using terminal and internal alkynes to afford *via* alkenyl cation intermediate, 1,1-diarylalkenes in high to excellent yields [12] (Scheme 3).

In(OTf)$_3$ has also been found to be a prominent catalyst for double addition of heterocyclic arenes to alkynes affording a 2:1 adduct where two heterocyclic arenes regioselectively attack the same carbon of alkynes [13a] (Scheme 4). Very recently, this catalyst has also been used for ene and Friedel–Crafts addition of electron rich alkenes and heteroaromatics, respectively, to activated α-imino esters [13b].

2.2.2. Other electrophilic substitutions

Aryl and alkyl sulfonylation of both activated and deactivated aromatic systems can be effected by 5–10 mol% of In(OTf)$_3$ [14,15a]. Efficacy of In(OTf)$_3$ or In(NTf$_2$)$_3$ as Lewis acid has also been established in other electrophilic aromatic substitutions like nitration and acylation reactions [15a]. Commercial In(OTf)$_3$ is also efficient for sulfamoylation of aromatic compounds [15b].

2.3. Coupling reaction of carbonyl and alkyne

1,5-Diketones were generated from a three component coupling reaction of terminal alkyne, α,β-unsaturated ketone and water in which In(OTf)$_3$ was utilised as a cocatalyst with a Ru-complex [CpRu(COD)Cl] [16].

[4 + 1] Annulation products and/or unsaturated ketones are, respectively, obtained by coupling alkynes and aldehydes in the presence of GaCl$_3$ or In(OTf)$_3$ [17] (Scheme 5).

In(OTf)$_3$ [18a] also catalyzed the addition of active methylene compounds to terminal alkynes (Scheme 6). Interestingly welding grade acytylene gas has been utilized for In(OTf)$_3$ catalyzed efficient vinylation of β-keto esters at 100 °C in the presence of molecular sieves as additive under solventless condition [18b].

A catalytic alkynylation of carbonyl compounds promoted by a combination of In(III) salt and ^{i}Pr$_2$NEt, was developed by Shibasaki and coworkers [19] that is supposed to proceed *via* dual activation of both the soft nucleophiles (terminal alkynes) and hard electrophiles (aldehydes and ketones) (Scheme 7).

2.4. Diels–Alder reactions

A high catalytic activity of In(OTf)$_3$ was established by Frost and coworkers [20] in the hetero Diels–Alder reaction of benzaldehyde and Danishefsky's diene and this was further
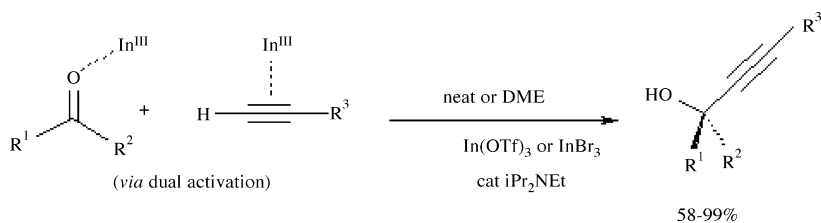
Scheme 7.
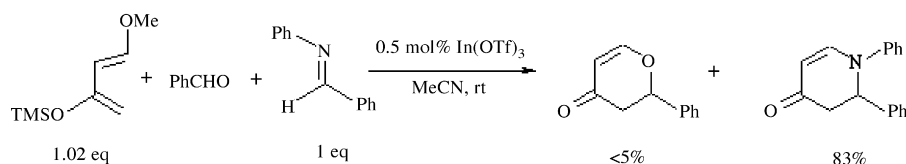
Scheme 8.

Scheme 9.

Scheme 10.

extended for the respective imino Diels–Alder reaction. The latter reaction proceeded at a faster rate than its oxygen counterpart as is evidenced from a competitive experiment of these two substrates. Thus, from a mixtur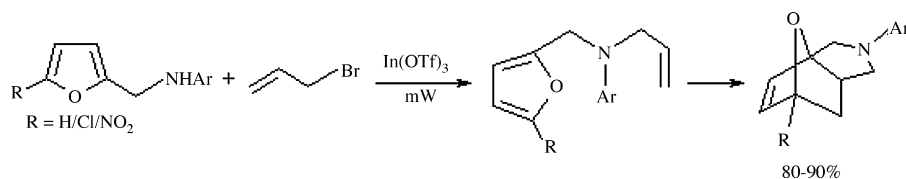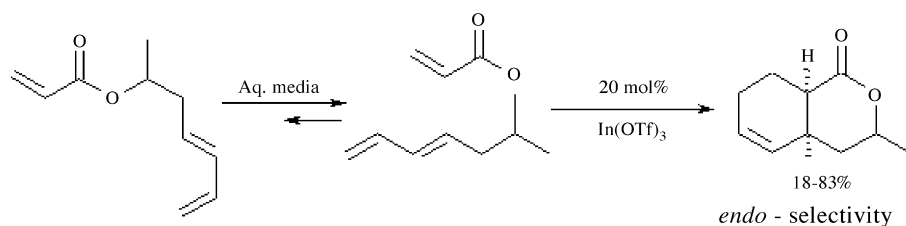e (1:1) of benzaldehyde and *N*-benzylidene aniline and Danishefsky's diene in the presence of 0.5 mol% of In(OTf)$_3$, the product arising from the reaction with imine was isolated in 83% yield indicating the possibility of a three-component coupling reaction (Scheme 8). A variety of *N*-heterocyclic compounds have thus been prepared by imino Diels–Alder reaction.

Sandhu and coworkers [21] employed this reagent for an intramolecular Diels–Alder reaction of furans under microwave

irradiation (Scheme 9). They have also utilized this Lewis acid for a [4 + 2] cyclo addition of chromone schiff's base [22].

In(OTf)$_3$ [23] efficiently catalyzes the intramolecular Diels–Alder reaction of a suitable ester-tethered compound in a mixture of H$_2$O and 2-PrOH furnishing the corresponding cyclo adduct in good yields with perfect *endo*-selectivity (Scheme 10).

An interesting dual Diels–Alder behaviour of 2-azetidinone-tethered aryl imines (as azadienophile or azadiene) has been reported in the presence of different Lewis acids, with best diastereoselectivity based on In(OTf)$_3$. Cyclopentadiene, 2,3-dimethyl-1,3-butadiene or 3,4-dihydro-2*H*-Pyran led to cyclo adducts arising from inverse electron-demand condensation
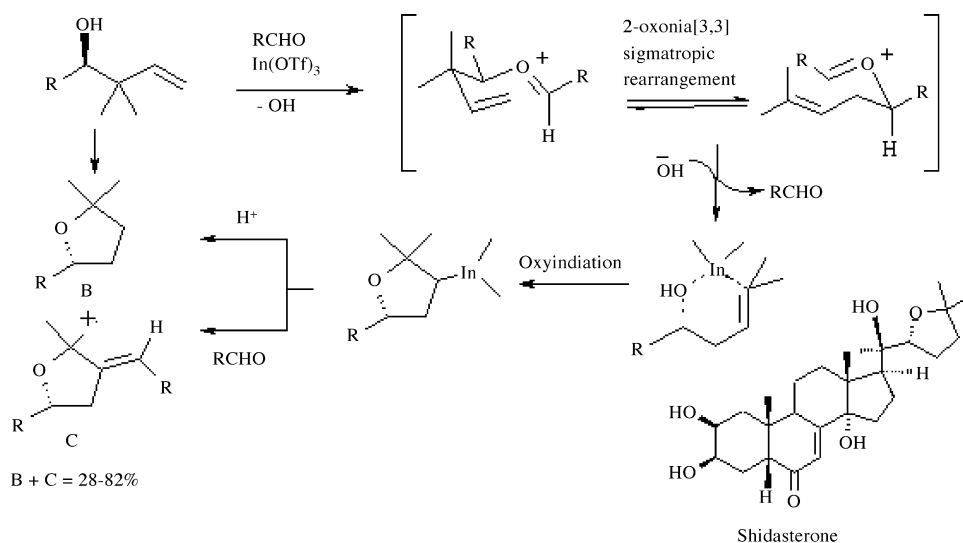
Scheme 11.

Scheme 12.
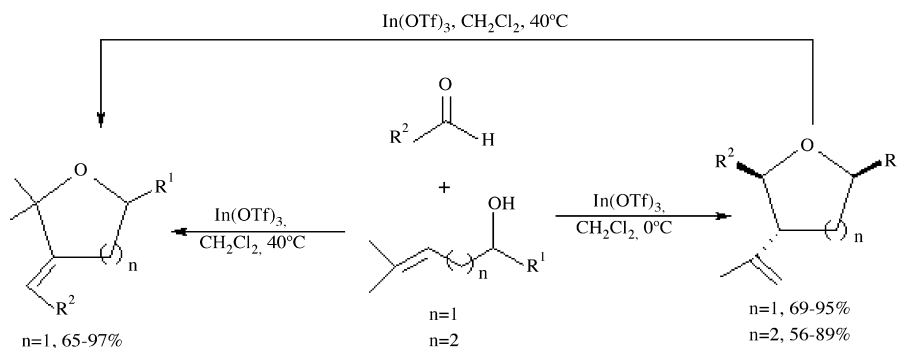
involving the β-lactam tethered aryl imine as the hetero diene component and this is the first report for preparation of functionalized indolizine from β-lactams [24].

2.5. Formation of tetrahydrofuran and pyran rings

Loh et al. [25] first discovered the In(OTf)₃ catalyzed stereoselective formation of tetrahydrofuran by a tandem 2-oxonia [3,3]-sigmatropic rearrangement/cyclisation sequence from the corresponding γ-adduct homoallylic alcohol (Scheme 11). They have also applied this method for construction of the side chain tetrahydrofuran moiety in an antitumor ecdysteroid called shidasterone [26].

This group has also developed a convergent approach for the synthesis of various tetrahydropyran and furan ethers from In(OTf)₃ catalyzed condensation of aldehyde and homoallylic alcohol or its chain elongated homologue via [3,5]-oxonium-ene type cyclization [27] as shown in Scheme 12. It is interesting to note that the reaction course is temperature dependent, and the skeletal rearrangement is limited to the skeletal reorganization of tetrahydrofurans but not the tetrahydropyran counterparts.

A diastereoselective construction of *cis*-2,6-disubstituted tetrahydropyran ring is also possible via In(OTf)₃ catalyzed intramolecular [2,5]-oxonium-ene cyclization from homoallylic alcohol and aldehyde [28a]. This has also been applied for the formation of a common intermediate towards the total synthesis of zampanolide and dactylolide [28a]. Further application of these techniques by the same group led to atom-economic one-pot diastereoselective synthesis of *cis*-2,6-disubstituted tetrahydropyran via self-tandem carbonyl-ene followed by intramolecular [2,5]-oxonium-ene cyclization from aldehyde and methylene cyclohexane [28b]. In(OTf)₃ has also

been employed for α- to α-allyl transfer from linear homoallylic alcohol to aldehyde via a 2-oxonia [3,3]-sigmatropic rearrangement [28c].

2.6. Asymmetric synthesis

This water compatible Lewis acid has been observed to render a moderate selectivity in comparison to that of InCl₃ in stereoselective asymmetric aldol reaction of formaldehyde with glycose-derived silyl enol ether in water [29]. Report has been made of a bifunctional catalyst system based on a chiral nucleophile and an achiral Lewis acid [M(OTf)$_n$, where M = In/Zn/Sc/Al/Ag/La/Mg] to afford optically enriched β-lactam products in high yields [30]. A Pd-In(OTf)₃ catalyst system was found to be more effective for dimerization of vinylarenes compared to the generally used cationic Pd(II) catalysts [31]. This method has also been utilized by Bedford et al. [32] in the asymmetric dimerization of styrene.

In(OTf)₃ catalyzes the conversion of branched homoallylic alcohols to the thermodynamically preferred linear regioisomer [33]. This has also been utilized for the construction of steroidal side chains with *anti*-Cram stereoselectivity. This has further been extended for the enantioselective and α-regioselective allylation of various aldehydes based on a homoallylic sterol-In(III) Lewis acid reagent system [34]. A variety of ketones were allylated by diallyl dibutyltin in the presence of 10 mol% In(OTf)₃ [35]. The homoallylation reactions have been exploited by other groups in successful enantioselective (60–93% ee) formation of homoallylic alcohols from the condensation of aldehydes and allyltributylstannane in the presence of a combination catalyst based on In(III)–PYBOX complex in solution [36a,b] (Scheme 13) and in ionic liquid medium with

Scheme 13.

X=Ar, 68-92%
X=OCOR, 85-92%
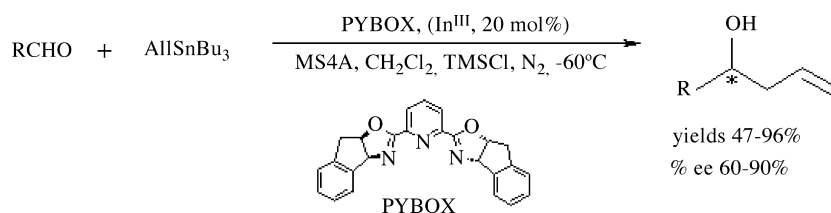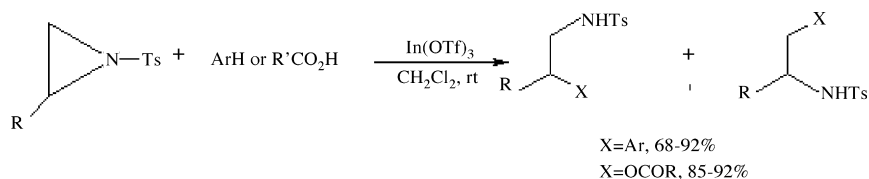
Scheme 14.

an increase in enantioselectivity in the later case [37a]. The In(III)–PYBOX system has also been used in catalytic enantioselective Mukaiyama–Aldol reaction [37b].

2.7. Opening of 3-membered heterocycles

In(OTf)$_3$ efficiently catalyzes the C-arylation of aziridines leading to regiocontrolled formation of β-arylamines [38]. Aziridines also react smoothly with carboxylic acids in the presence of catalytic amount of In(OTf)$_3$ affording β-amino acetates and benzoates in high yields and regioselectivity [39] (Scheme 14). InCl$_3$ or In(OTf)$_3$ catalyzed opening of epoxides are also possible with thiols leading to the generation of β-hydroxy sulfides [40].
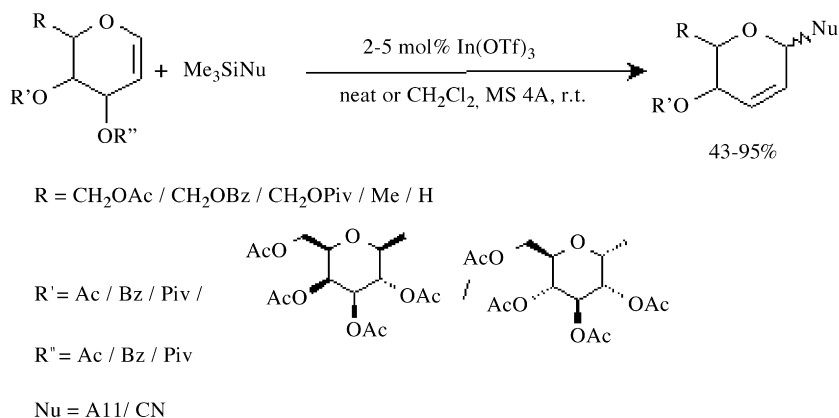
2.8. Miscellaneous reactions

Apart from the aforementioned reactions In(OTf)$_3$ has also been applied for a number of other organic syntheses. It is an efficient catalyst for acylation of a variety of alcohols including sugars, and amines [41]. An efficient expeditious chemoselective recyclable conversion of aldehydes to acylals has been developed by our group in the presence of very small amount of In(OTf)$_3$ [42]. This efficiently catalyzes the insertion reaction of various α-diazoketones with aliphatic and aromatic hydroxy compounds or benzenethiol providing the corresponding insertion products [43]. This aquacompatible Lewis acid has also been utilized for Pictel–Spengler reactions of tryptophan methyl ester and tryptamine with aliphatic and aromatic aldehydes under microwave condition [44]. A high *anti*-diastereofacial selective route to β-aminoalcohol was developed *via* lanthanide triflate promoted In- or Zn-mediated allylation of α-amino aldehyde in aqueous medium [45].

High regioselective Markonikov-type addition of thiolacetic acid to non-activated olefins is catalyzed by InCl$_3$ or In(OTf)$_3$ [46]. In some linear olefins, In(III) also catalyzes selective isomerization of carbon–carbon double bonds, thus finally yielding a mixture of isomeric addition products. Intra- and intermolecular addition of thiols to non-activated olefins is possible *via* In(OTf)$_3$ catalyzed regioselective hydrothiolation reaction [47].
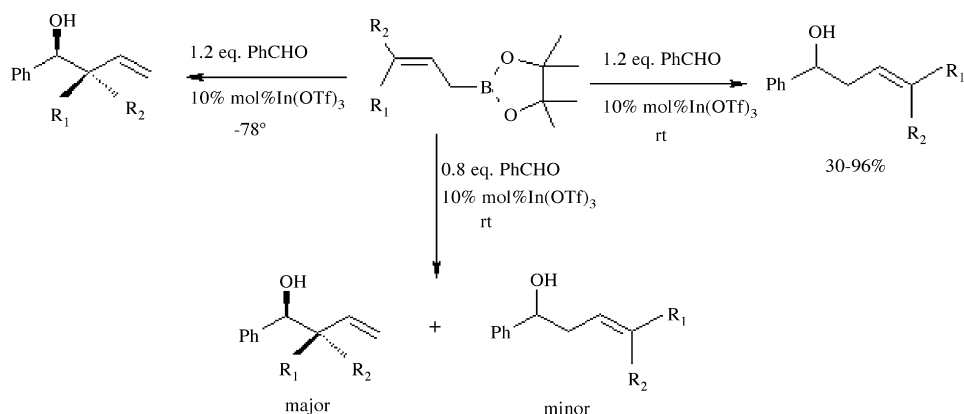
Report has been made of the one-pot synthesis of 3,4-dihydropyrimidinones based on In(OTf)$_3$ in solvent by our research group [48a] and on solid support under microwave induced condition by Perumal and coworkers [48b]. We have further utilized In(OTf)$_3$ in the catalyzed efficient one-pot multicomponent synthesis of α-amino phosphonates from aldehydes, amine and diethyl phosphite [49]. Very recently, use of this moisture stable catalyst has been made in the catalyzed synthesis of 2-substituted benzimidazoles from *o*-phenylenediamine and aldehyde under solvent free condition at room temperature [50].

We have also successfully established the efficacy of this Lewis acid catalyst in the stereoselective C-glycosylation of glucal derivative in the presence of allytrimethylsilane and trimethylsilyl cyanide in solution as well as under solvent free condition [51] (Scheme 15).

Synthesis of 4-substituted homoallylic alcohols are prepared *via* In(OTf)$_3$ catalyzed tandem crotylboration-[3,3]-sigmatropic rearrangement from aldehydes and *E* or *Z*-crotylboronates [52]. The course of the reaction is however, dependent on the temperature as well as on the amount of the aldehyde. Thus, at temperature as low as −78 °C the 2-substituted homoallylic alcohol is formed from benzaldehyde and *E* or *Z*-crotylboronate but the 4-substituted product is formed with very high regioselectivity at room temperature. Use of substoichiometric amount of aldehyde generated a mixture of these two products with the 2-sustituted one as the major product (Scheme 16).
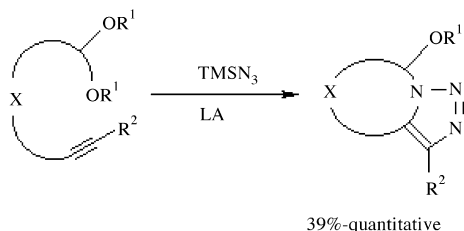

43-95%

R = CH$_2$OAc / CH$_2$OBz / CH$_2$OPiv / Me / H

R' = Ac / Bz / Piv /

R" = Ac / Bz / Piv

Nu = A11 / CN

Scheme 15.

Scheme 16.

R = *n*-C$_5$H$_{11}$, Ph

39%-quantitative

Scheme 17.

Azidation reaction of dialkoxy acetal derivatives with TMSN$_3$ is catalyzed by (1–5) mol% In(OTf)$_3$ furnishing a mixture of α-azido ether as the major product from aliphatic acetals and *gem*-diazide as the major product from aromatic acetals. In the same report syntheses of bicyclic 1,2,3-triazolo-heterocyclic compounds *via* In(OTf)$_3$ [53] catalyzed tandem azidation-intramolecular 1,3-dipolar cycloaddition of alkynyl acetals have also been mentioned (Scheme 17).

Very recently, In(OTf)$_3$ has been employed for stereoselective rearrangement of aryl substituted cyclopropyl carbinol derivatives under sonication for generation of substituted conjugated all-*trans* butadienes [54]. Chiral *N*-acylhydrazones undergo highly diastereoselective fluorine-initiated addition of allylsilanes in the presence of In(OTf)$_3$ following a mechanism involving dual activation in which the nucleophilic component is a hypervalent allylfluorosilicate and the electrophilic component is a chiral *N*-acylhydrazone–In(III) chelate [55].

3. Conclusion

Although in a few of the above reports mechanistic studies of some transformations have been made but the status of inter-mediate In-species has not been well addressed. Two examples of In(III) *N*-heterocyclic carbene triflate complexes are reported those could represent structural models for the poorly defined catalyst In(OTf)$_3$ [56a,b]. The foregoing discussions reveal the

still increasing interest of this Lewis acid. Further exploitation of its potential in other organic transformations is expected to come out in near future.

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