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Review

Archive for Organic Chemistry

Arkivoc **2017**, part i, 1-25

$Sc(OTf)_3$ catalyzed carbon-carbon and carbon-heteroatom bond forming reactions: a review

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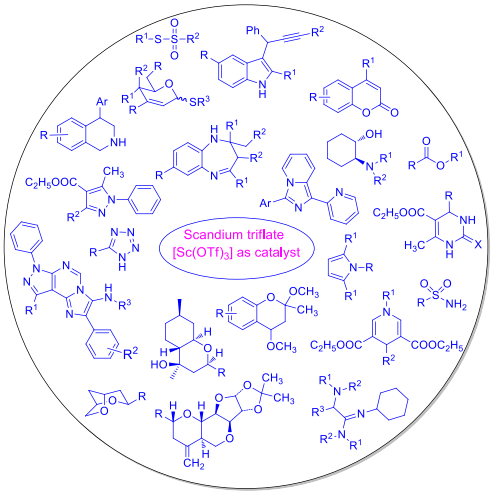
Received 09-05-2016 **Accepted** 11-08-2016 **Published on line** 12-04-2016

Abstract

In recent years scandium(III) trifluoromethanesulfonate [$Sc(OTf)_3$] has emerged as an efficient, mild, commercially available, inexpensive, water tolerant Lewis acidic catalyst in the formation of both carbon-carbon and carbon-heteroatom bonds, and thereby the formation of various biologically promising organic compounds. The present review summarizes the latest developments on $Sc(OTf)_3$-catalyzed organic transformations especially carbon-carbon and carbon-heteroatom bond forming reactions reported during the last decade.



Keywords: Lewis acid catalysis, scandium triflate, heterocycles

DOI: https://doi.org/10.24820/ark.5550190.p009.868

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1. Introduction

Carbon-carbon and carbon-heteroatom bond-forming reactions are the important tools of organic synthesis to afford structurally varied bioactive organic compounds.[1-3] Catalysts play an obvious role in such reactions and thus they find wide application. But the screening of suitable catalysts plays a crucial role among the other significant parameters during such chemical praxis.

The last decade has seen a great development in the use of triflate salts as catalysts for organic transformations.[4] Among triflate salts, the applications of scandium(III) trifluoromethanesulfonate [$Sc(OTf)_3$, scandium triflate] as a Lewis acid catalyst have increased rapidly in the variety of organic transformations that can be effected.[5-17] The catalytic applicability of this mild catalyst is well documented in the literature, especially in cycloaddition reactions,[18] Diels-Alder,[19] Ugi,[20] and Michael reactions.[21] Though the majority of the developed methods are based upon the ability of scandium to activate C=X π-bonds toward nucleophilic additions, more recently it has been found that scandium(III) can also activate C-X σ-bonds.[22]

In 1993, Kobayashi *et al*.[23] first demonstrated the use of $Sc(OTf)_3$ as a promising Lewis acid catalyst in organic synthesis. $Sc(OTf)_3$ is now commercially available and can be prepared easily from scandium oxide (Sc_2O_3) and aqueous trifluoromethanesulfonic acid (TfOH).[7] In general, most of the traditional Lewis acids are deactivated in the presence of water, but $Sc(OTf)_3$ is stable in an aqueous environment and can efficiently catalyze organic transformations in aqueous media. Moreover, $Sc(OTf)_3$ is well tolerated and worked efficiently as a Lewis acid catalyst in several other organic solvents. As the size of the scandium (Sc^{3+}) ion is smaller than those of the rare-earth elements forming triflate salts, $Sc(OTf)_3$ is a much more efficient Lewis acid catalyst than its congeners. Because of all these benefits the use of this unique catalyst has increased rapidly in organic synthesis especially in carbon-carbon and carbon-heteroatom bond forming reactions.[24]

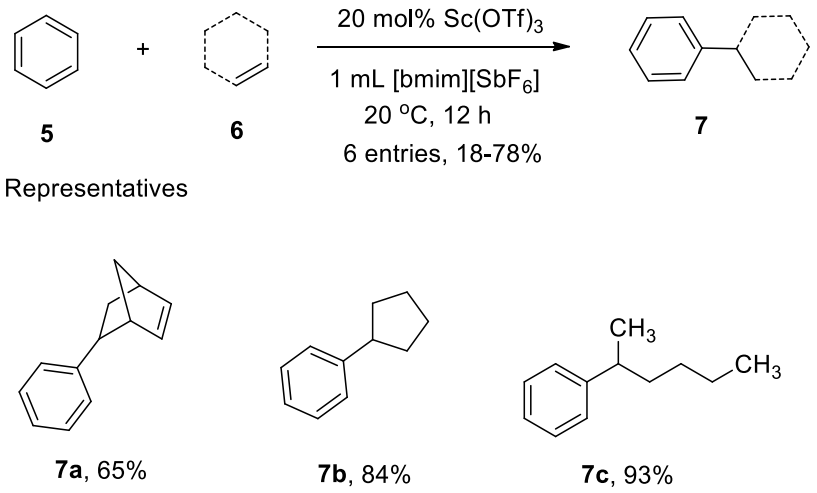
The present communication focuses on the catalytic application of $Sc(OTf)_3$ as a mild Lewis acid in organic synthesis, leading to carbon-carbon and carbon-heteroatom bond forming reactions, with up-to-date literature reported on this subject during the last decade.

The following Sections describe the catalytic applicability of scandium(III) triflate in organic synthesis.

2. Carbon-Carbon Bond-forming Reactions

2.1 Friedel-Crafts alkylation of aromatic compounds with alkenes

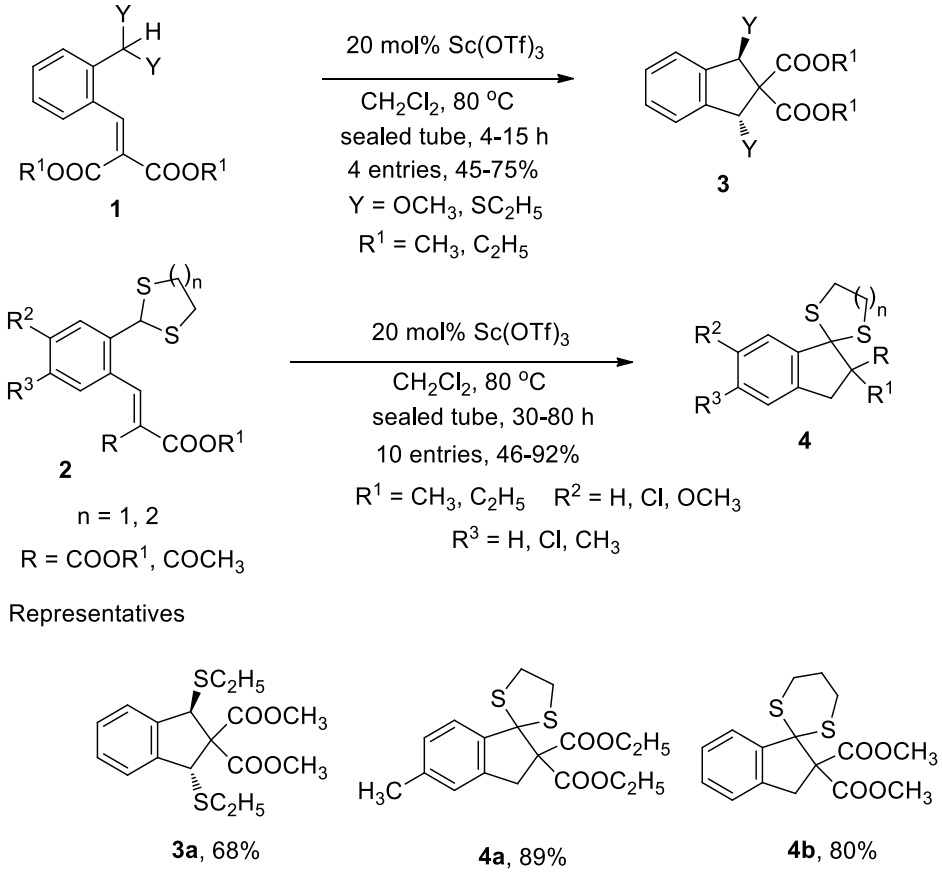
Scandium(III) triflate catalyzed Friedel-Crafts alkylation of aromatic compounds (**1**) with alkenes (**2**) to form the corresponding alkylated products (**3**) was demonstrated by Song *et al*.[25] (Scheme 1) in 1,3-dialkylimidazolium salts as hydrophobic ionic liquid solvents.



Scheme 1. Scandium(III) triflate catalyzed Friedel-Crafts alkylation.

2.2 Synthesis of 1,2-dihydroindane derivatives

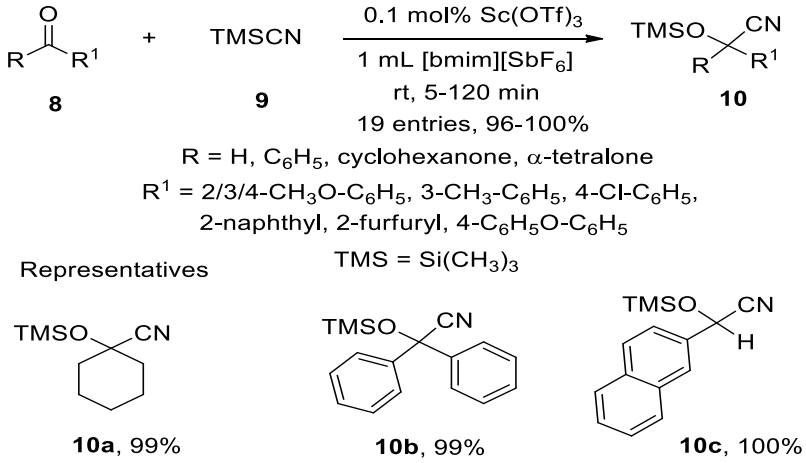
Alajarin *et al.*[26] designed a new carbon-carbon bond forming reaction leading to adjacent quaternary carbons to prepare 1,2-dihydroindane derivatives (**5**, **7**) in the presence of scandium(III) triflate as catalyst by the reaction of activated acetalic C-H bonds with benzylidenemalonate fragments (**4**, **6**) as electrophilic hydride acceptors. In this strategy both cyclic as well as acyclic acetal functions underwent smooth conversion to give the desired products (Scheme 2).



Scheme 2. Scandium (III) triflate catalyzed synthesis of 1,2-dihydroindane derivatives.

2.3 Synthesis of α-(trimethylsilyloxy)nitriles

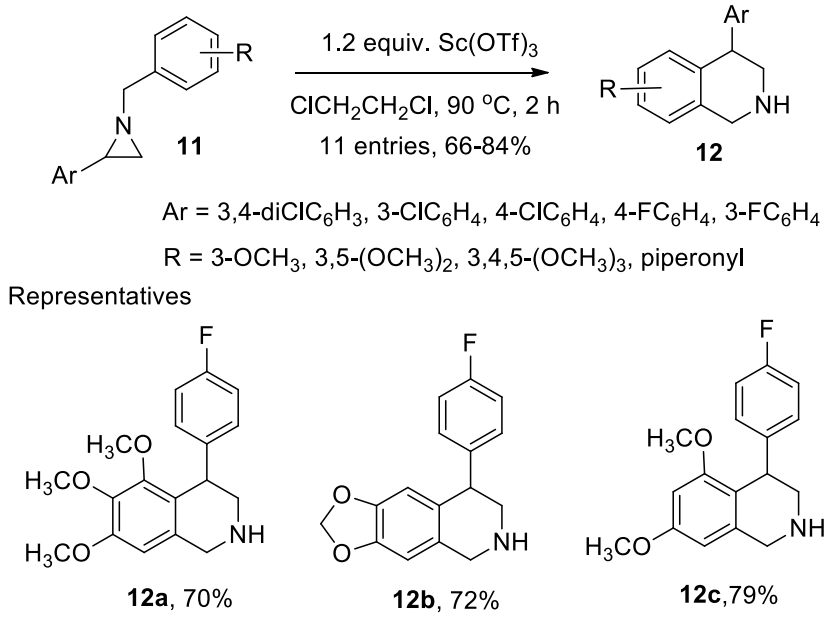
Park *et al.*[27] reported an environmentally benign, highly reactive $Sc(OTf)_3$ catalyzed cyanosilylation of carbonyl compounds (**8**) with trimethylsilyl cyanide (**9**) to prepare the corresponding α-(trimethylsilyloxy)carbonitriles (**10**) in [bmim][SbF_6] at room temperature (Scheme 3). They also successfully recovered and reused the ionic liquid containing $Sc(OTf)_3$ for several reaction cycles without any loss of catalytic activity.



Scheme 3. $Sc(OTf)_3$ catalyzed cyanosilylations of carbonyl compounds.

2.4 Synthesis of 4-substituted tetrahydroisoquinolines

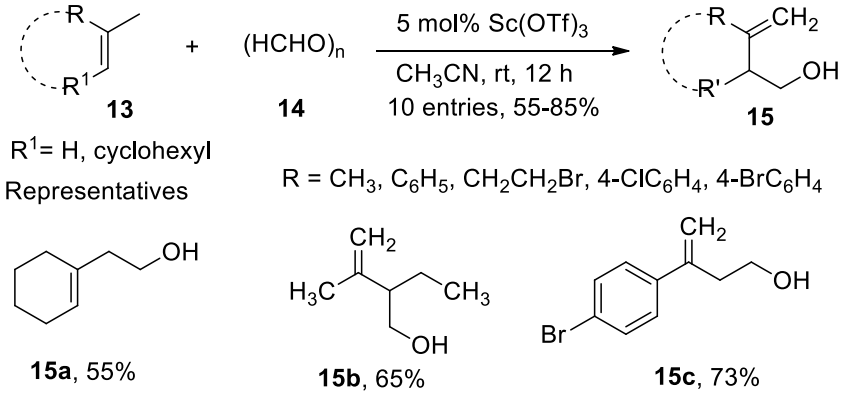
Tummanapalli *et al*.[28] demonstrated a novel, scandium(III) triflate catalyzed, facile, straight forward synthesis of 4-substituted tetrahydroisoquinolines (1**2**) *via* intramolecular ring expansion of aziridines (1**1**) in 1,2-dichloroethane at 90 °C (Scheme 4).



Scheme 4. Scandium(III) triflate catalyzed synthesis of 4-substituted tetrahydroisoquinolines.

2.5 Synthesis of primary homoallylic alcohols

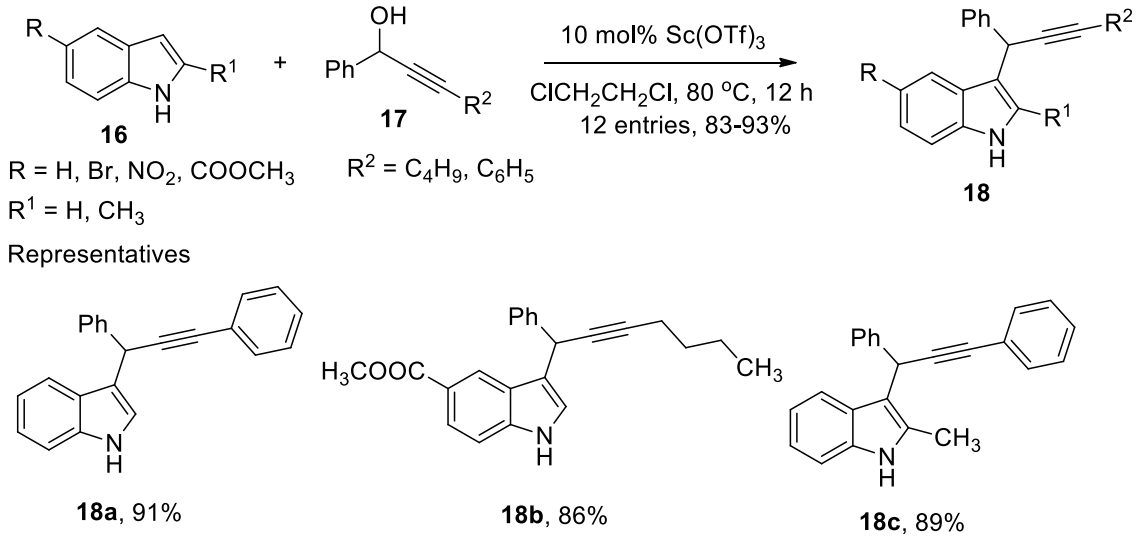
Sultana *et al*.[29] developed a simple protocol for the synthesis of primary homoallylic alcohols (**15**) from the reaction of alkenes (**13**) and paraformaldehyde (**14**) using scandium triflate as catalyst at room temperature (Scheme 5).



Scheme 5. $Sc(OTf)_3$-catalyzed synthesis of primary homoallylic alcohols.

2.6 Synthesis of 3-propargylated indoles

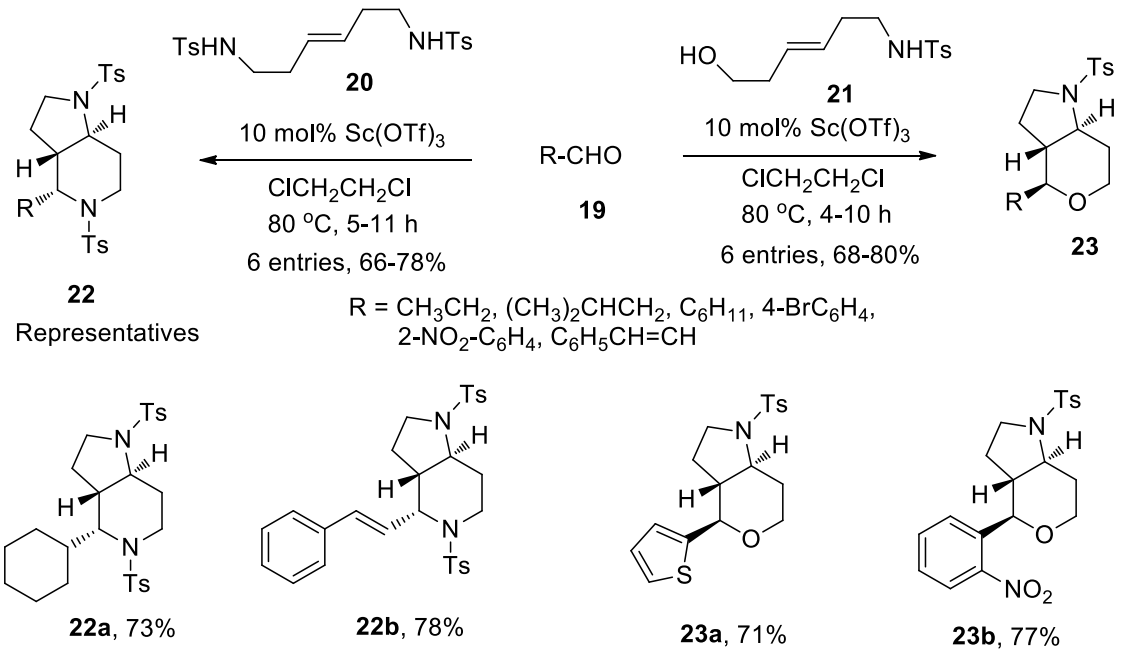
Yadav and his group (Scheme 6)[30] described a facile and efficient alkylation of indoles (16) with propargylic alcohols (17) to produce 3-propargylated indoles (18) in excellent yields using scandium triflate as catalyst in 1,2-dichloroethane at 80 °C.



Scheme 6. Scandium(III) triflate catalyzed alkylation of indoles with propargyl alcohols.

2.7 Synthesis of octahydro-1*H*-pyrrolo[3,2-*c*]pyridines and octahydropyrano[4,3-*b*]pyrroles

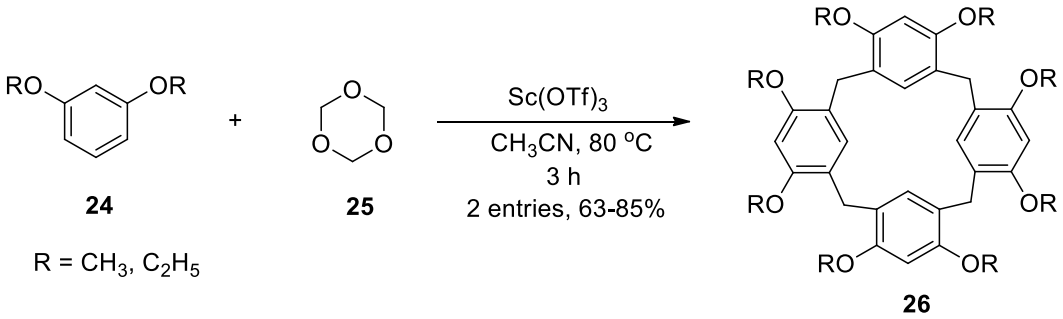
Reddy *et al*. (Scheme 7)[31] developed a new method for the synthesis of octahydro-1*H*-pyrrolo[3,2-*c*]pyridines (**22**) and octahydropyrano[4,3-*b*]pyrroles (**23**) selectively by means of intramolecular aza-Prins and Prins cyclization of aldehydes (**19**) and *bis*-homoallyl (**20**) and heteroallyl (**21**) derivatives respectively.



Scheme 7. Synthesis of octahydropyrrolo[3,2-*c*]pyridines and octahydropyrano[4,3-*b*]pyrroles.

2.8 Synthesis of resorcin[4]arene octaalkyl ethers

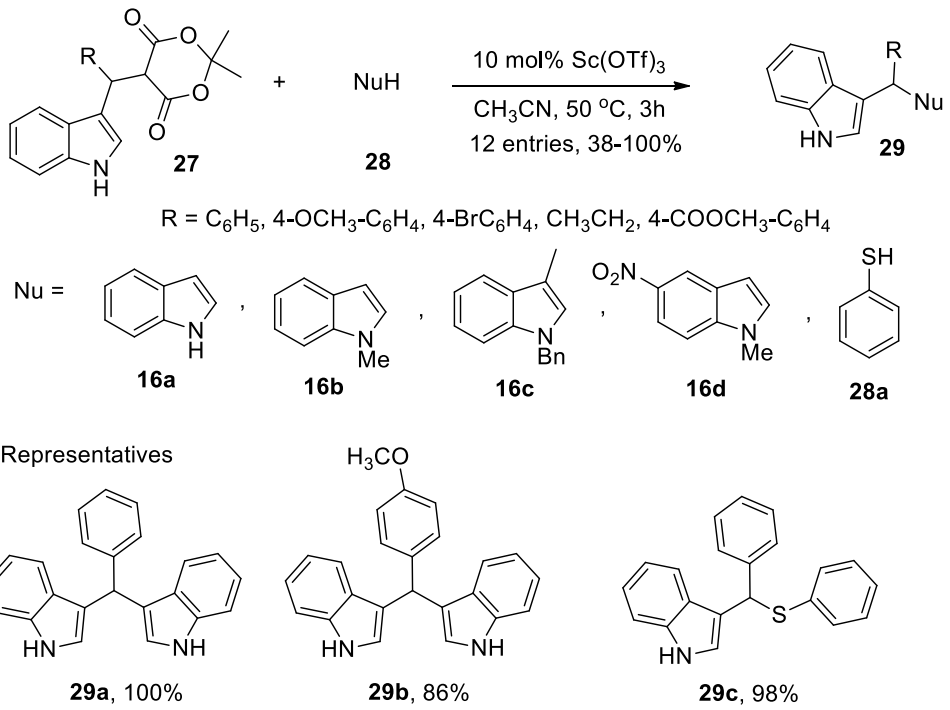
Morikawa *et al*.[32] demonstrated a simple and straightforward cyclocondensation of 1,3-dialkoxybenzenes (**24**) with 1,3,5-trioxane (**25**) to produce resorcin[4]arene octaalkyl ethers (**26**) in good yields using catalytic amount of $Sc(OTf)_3$ in acetonitrile at 80 °C (Scheme 8).



Scheme 8. Scandium(III) triflate catalyzed synthesis of resorcin[4]arene octaalkyl ethers.

2.9 Synthesis of indolemethane derivatives

Kerr and his group[33] developed an expedient and efficient method for the synthesis of indolemethane derivatives (**29**) from the reaction of indolylmethyl Meldrum's acids (**27**) with a variety of nucleophiles (**28**) *via* the nucleophilic displacement of the Meldrum's acid moiety in the presence of catalytic scandium triflate at 50 °C in acetonitrile as solvent (Scheme 9).

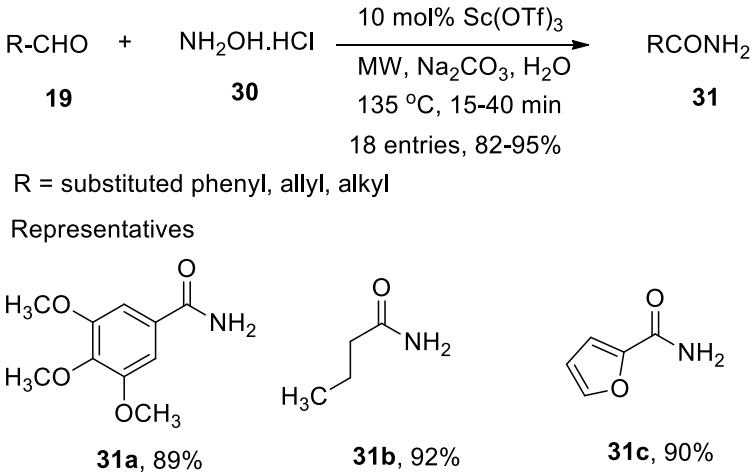


Scheme 9. Scandium(III) triflate catalyzed synthesis of indolemethane derivatives.

3. Carbon-Nitrogen Bond-forming Reactions

3.1 Synthesis of primary amides

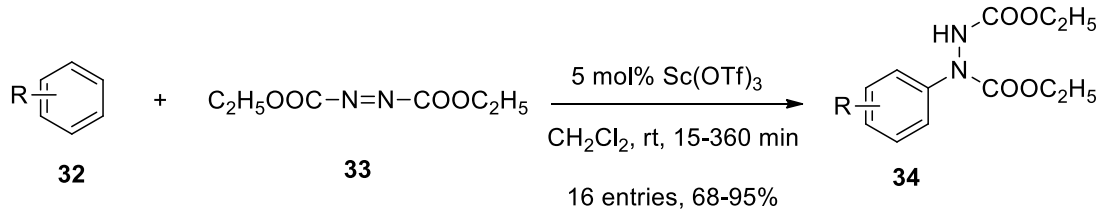
Allam *et al*.[34] described a versatile microwave-assisted synthetic protocol for the one-pot synthesis of primary amides (**31**) from aldehydes (**19**) and hydroxylamine hydrochloride (**30**) using scandium(III) triflate as a catalyst in water (Scheme 10).



Scheme 10. Scandium(III) triflate catalyzed synthesis of primary amides.

3.2 Synthesis of aryl hydrazides

Yadav *et al*.[35] reported a $Sc(OTf)_3$ catalyzed electrophilic amination of arenes (**32**) with diethyl azodicarboxylate (**33**) in dichloromethane at ambient temperature to afford the corresponding arylhydrazides (**34**) in high yields with high regioselectivity (Scheme 11).



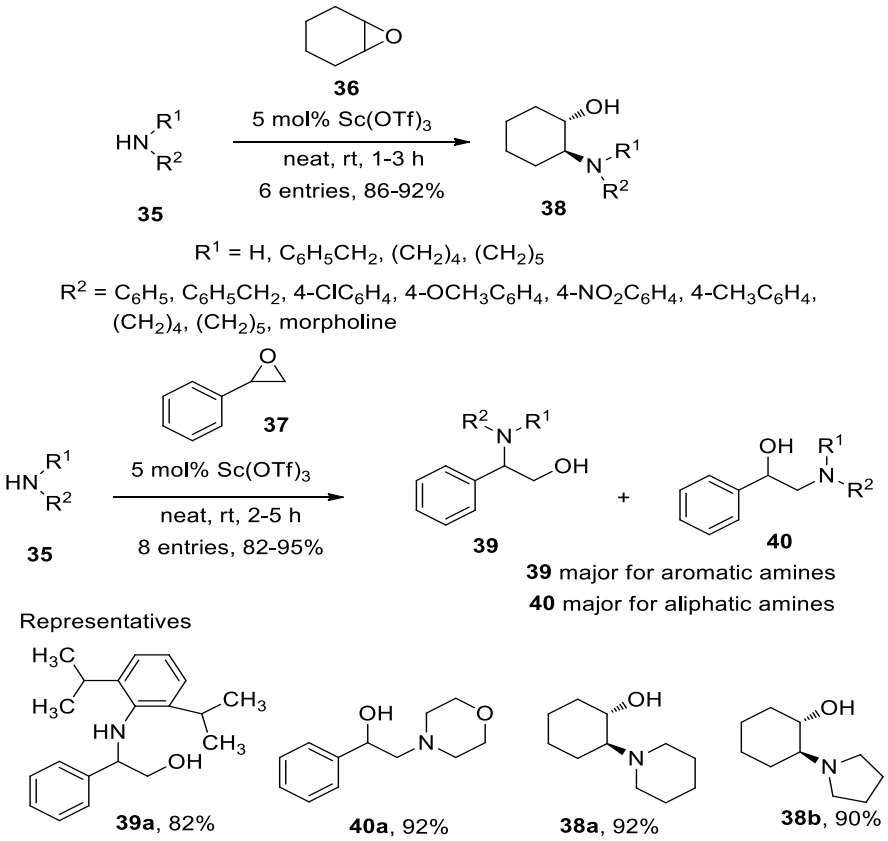
Scheme 11. $Sc(OTf)_3$ catalyzed electrophilic amination of arenes.

3.3 Synthesis of β-amino alcohols

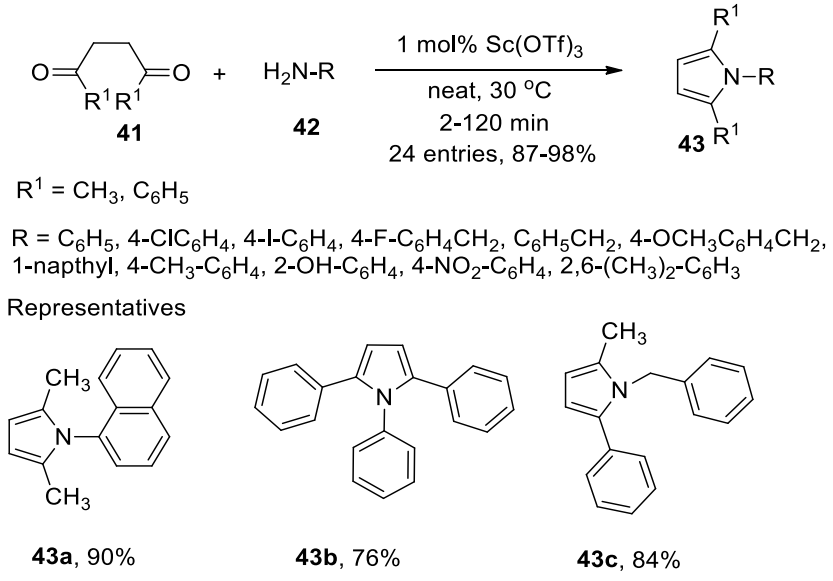
Placzek *et al*.[36] reported a simple, straight forward, efficient method for the synthesis of β-amino alcohols (**38**, **39**, **40**) *via* ring opening of epoxides (**36**, **37**) with amines (**35**) in the presence of a catalytic amount of $Sc(OTf)_3$ at room temperature under solvent-free conditions (Scheme 12).

3.4 Synthesis of N-substituted pyrroles

Chen *et al*.[37] developed a simple and efficient method for the synthesis of *N*-substituted pyrroles (**43**) by the Paal-Knorr condensation of various amines (**42**) with 1,4-diketones (**41**) using a catalytic amount of $Sc(OTf)_3$ under neat conditions at ambient temperature (Scheme 13). They also successfully recovered and reused the catalyst without significant loss in catalytic activity.



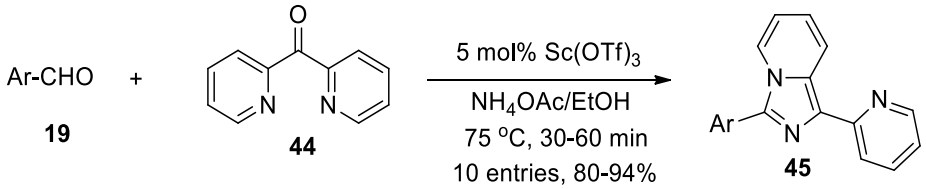
Scheme 12. Scandium(III) triflate catalyzed synthesis of β-amino alcohols.



Scheme 13. Scandium(III) triflate catalyzed synthesis of *N*-substituted pyrroles.

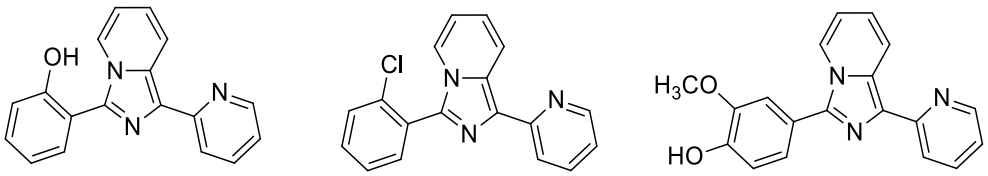
3.5 Synthesis of 1-pyridylimidazo-[1,5-a]-pyridines

Kottawar et *al*. (Scheme 14)[38] described a facile, mild and highly efficient protocol for the synthesis of 1-(2-pyridyl)imidazo[1,5-*a*]pyridines (**45**) from the reaction of various aromatic aldehydes (**19**) with di-2-pyridyl ketone (**44**) in presence of ammonium acetate in ethanol using scandium(III) triflate as catalyst.



Ar = C_6H_5, 4-OCH_3-C_6H_4, 4-CH_3-C_6H_4, 2-Cl-C_6H_4, 4-NO_2-C_6H_4, 3-OCH_3-4-OH-C_6H_3, 2-OH-C_6H_4, 3-OH-C_6H_4, 4-OH-C_6H_4,

Representatives

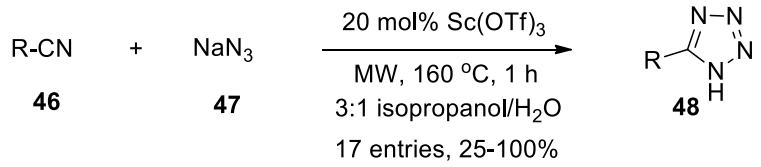


45a, 99% **45b**, 89% **45c**, 92%

Scheme 14. Scandium(III) triflate catalyzed synthesis of 1-pyridylimidazo[1,5-*a*]pyridines.

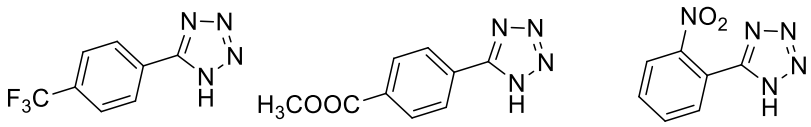
3.6 Synthesis of 5-substituted 1*H*-tetrazoles

Several 5-substituted 1*H*-tetrazoles (**48**) were synthesized by Coca *et al*.[39] (Scheme 15) *via* the [2+3] cycloaddition of sodium azide (**47**) with aryl nitriles, aliphatic nitriles, and vinyl nitriles (**46**) under the influence of microwave irradiation of 1 h at 160 °C in a 3:1 isopropanol / water mixture using scandium(III) triflate as catalyst.



R = C_6H_5, C_6H_4-CO, 2-Cl-C_6H_4, 4-CH_3-C_6H_4, 4-OCH_3-C_6H_4, 4-CF_3-C_6H_4, 4-Cl-C_6H_4, 4-Br-C_6H_4, 1-naphthyl

Representatives



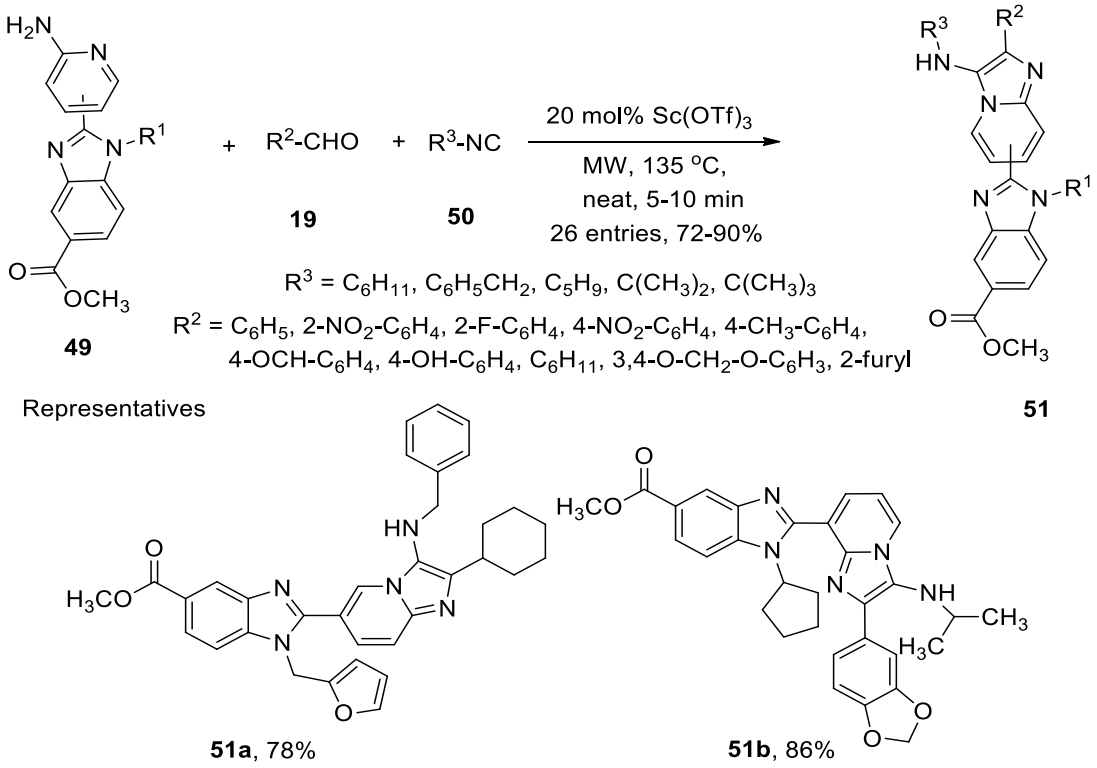
48a, 99% **48b**, 80% **48c**, 76%

Scheme 15. Scandium(III) triflate catalyzed synthesis of 5-substituted 1*H*-tetrazoles.

4. Simultaneous Carbon-Carbon and Carbon-Nitrogen Bond-forming Reactions

4.1 Synthesis of benzimidazolyl imidazo[1,2-*a*]pyridines

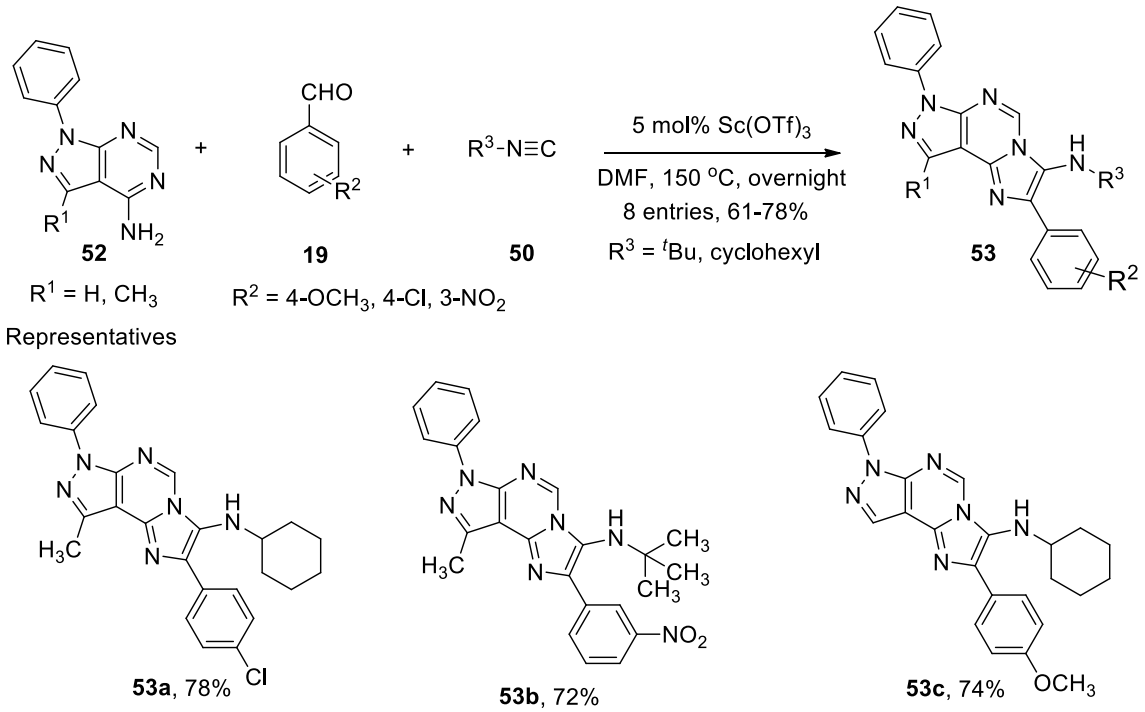
Maiti *et al*.[40] developed a microwave irradiated novel environmentally benign, one-pot three-component reaction protocol for the synthesis of biologically interesting benzimidazolyl-imidazo[1,2-*a*]pyridines (**51**) employing variously substituted benzimidazole-linked amino pyridines (**49**), aldehydes (**19**), and isocyanides (**50**) in presence of catalytic amount of scandium(III) triflate under solvent-free conditions (Scheme 16).



Scheme 16. Synthesis of benzimidazolyl imidazo[1,2-*a*]pyridines.

4.2 Synthesis of imidazo[1,2-*c*]pyrazolo[3,4-*e*]pyrimidines

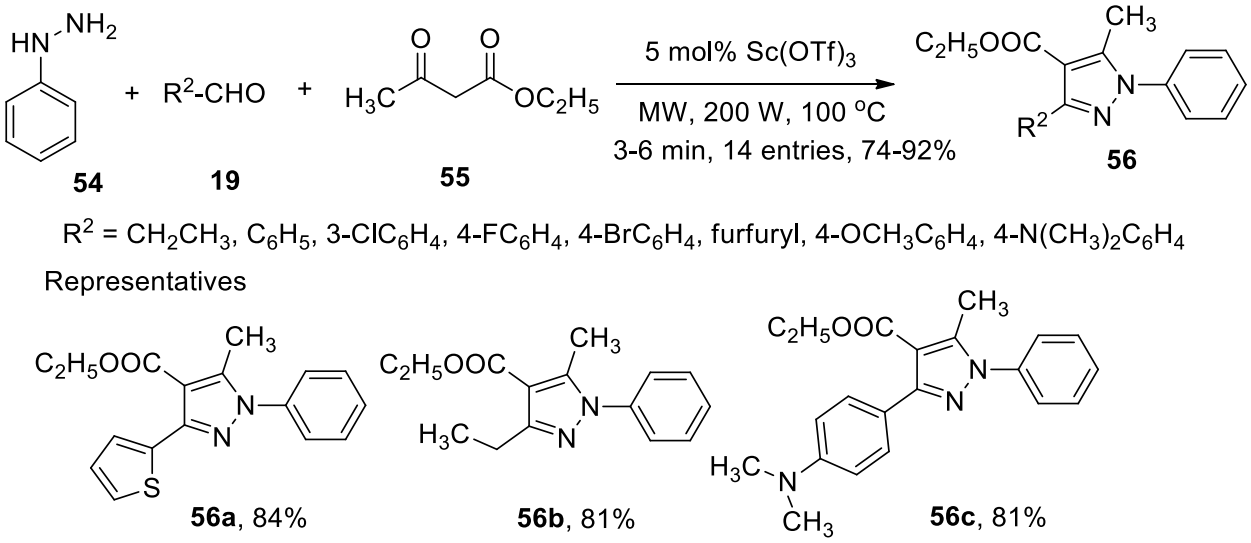
A facile, one-pot three-component scandium triflate catalyzed condensation reaction between aminopyrazolo[3,4-*d*]pyrimidine (**52**), aldehyde (**19**), and isocyanide (**50**) was developed by Agrebi *et al*.[41] for the synthesis of a series of fluorescent imidazo[1,2-*c*]pyrazolo[3,4-*e*]pyrimidines (**53**) in good to excellent yields at 150 °C (Scheme 17).



Scheme 17. Synthesis of imidazo[1,2-*c*]pyrazolo[4,3-*e*]pyrimidines.

4.3 Synthesis of functionalized pyrazoles

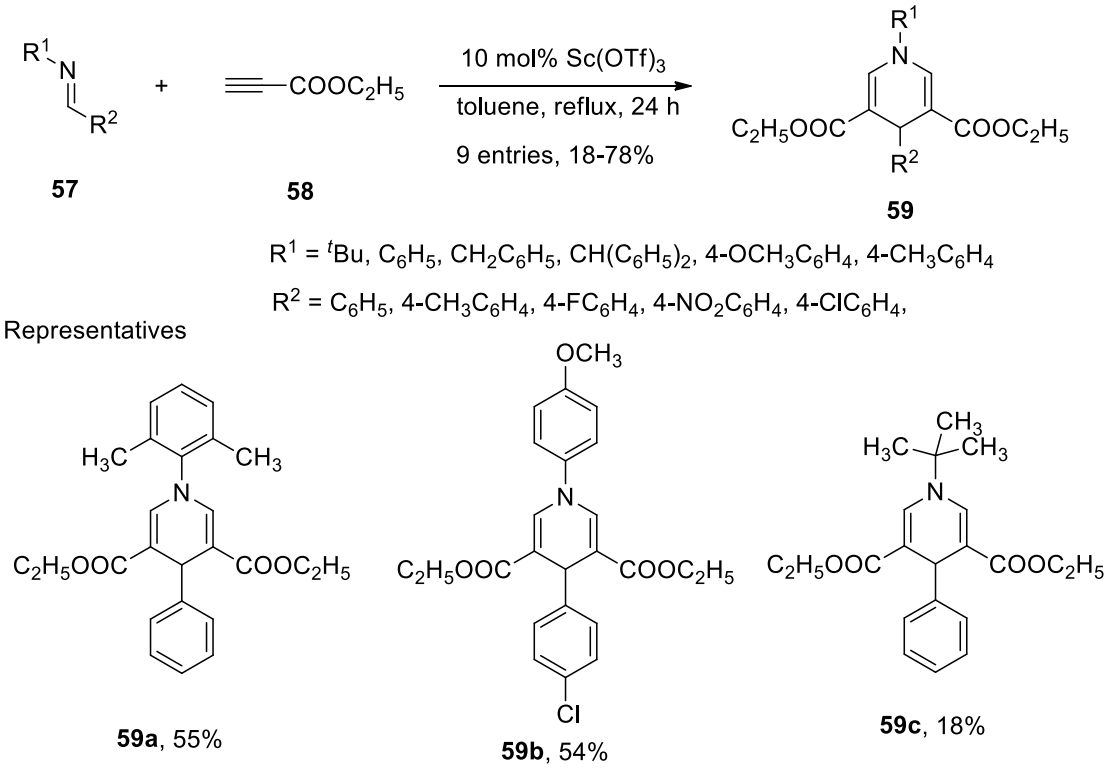
Kumari *et al*.[42] reported an efficient, facile, straight forward, microwave irradiated rapid, and environmentally benign scandium triflate catalyzed synthesis of functionalized pyrazoles (**56**) by the reaction of phenyl hydrazine (**54**), aldehydes (**19**) and ethyl acetoacetate (**55**) under neat conditions (Scheme 18).



Scheme 18. Scandium triflate catalyzed synthesis of functionalized pyrazoles.

4.4 Synthesis of *N*-substituted 1,4-dihydropyridine derivatives

Kikuchi *et al*.[43] developed a facile and straight forward method for the synthesis of *N*-substituted 1,4-dihydropyridine derivatives (59) from the reaction of imines (**57**) with ethyl propiolate (**58**) using catalytic amount of scandium(III) triflate in toluene or benzotrifluoride under reflux conditions (Scheme 19).



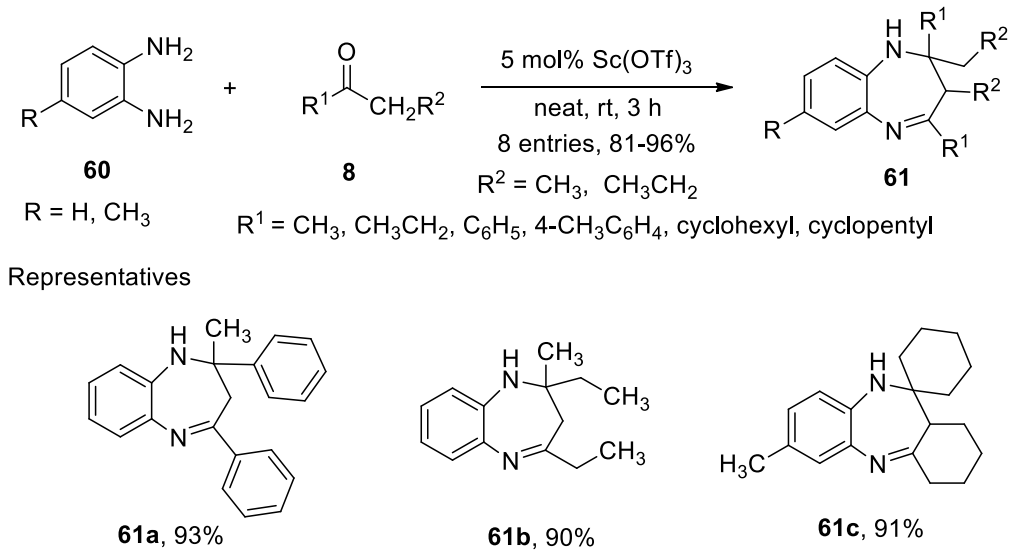
Scheme 19. Scandium(III) triflate catalyzed synthesis of 1,4-dihydropyridines.

4.5 Synthesis of 2,3-dihydro-1*H*-1,5-benzodiazepines

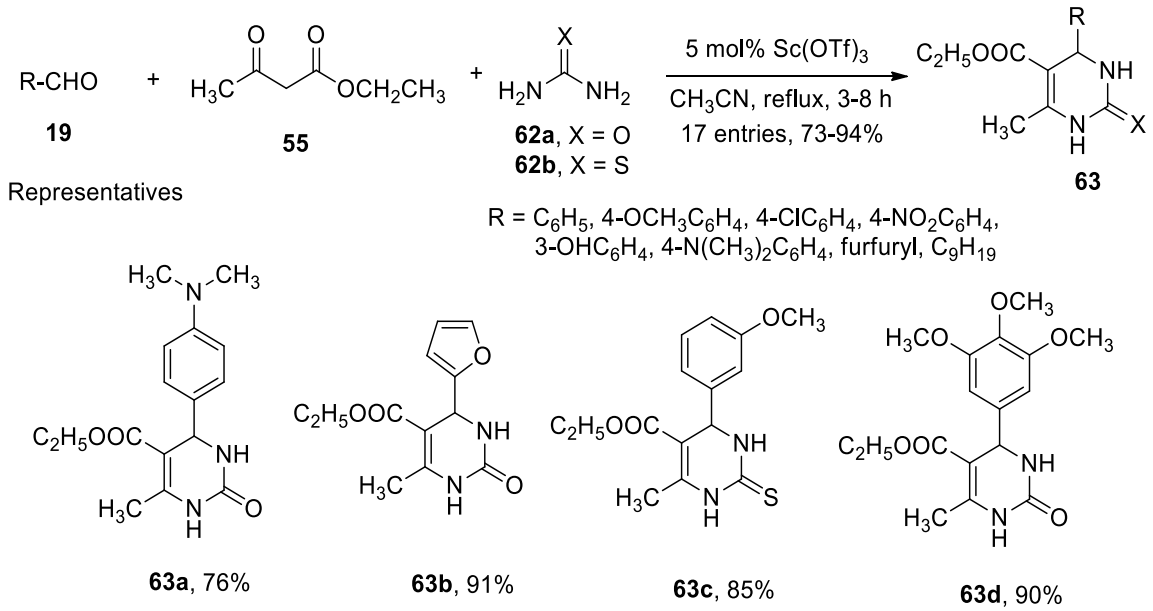
De *et al*.[44] demonstrated a mild, simple and efficient method for the synthesis of 2,3-dihydro-1*H*-1,5-benzodiazepines (**61**) with good yields from the reaction of *o*-phenylenediamines (**60**) and ketones (**8**) in the presence of a catalytic amount of $Sc(OTf)_3$ under neat conditions at room temperature (Scheme 20).

4.6 Synthesis of 3,4-dihydropyrimidin-2(1*H*)-ones

De and his group[45] developed another scandium(III) triflate catalyzed protocol for the synthesis of 3,4-dihydropyrimidin-2(1*H*)-ones (**63**) *via* the three component Biginelli reaction of aldehyde (**19**), β-ketoester (**55**), and urea (**62a**) or thiourea (**62a)** in acetonitrile under reflux conditions (Scheme 21).



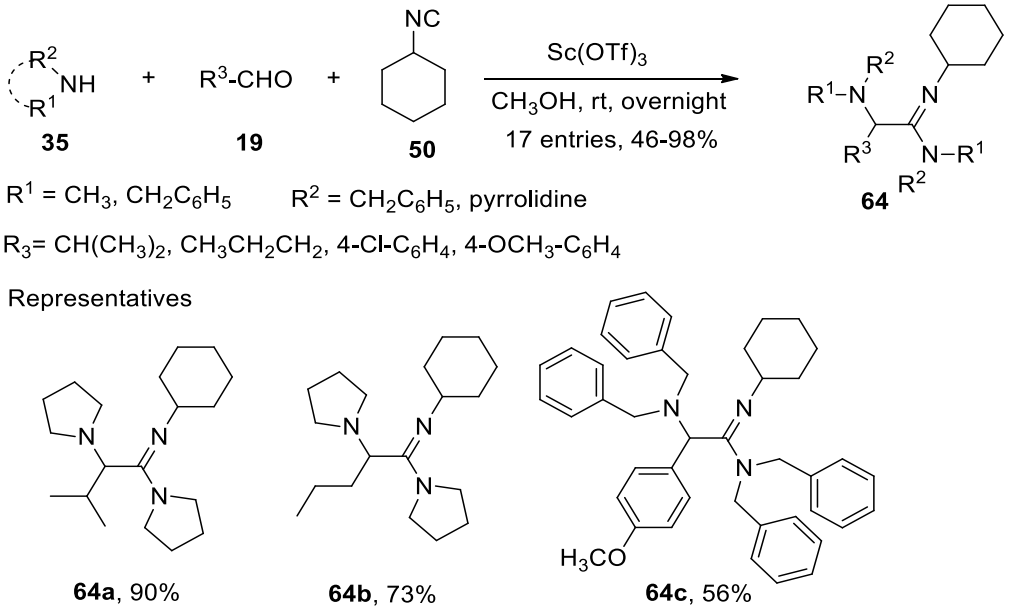
Scheme 20. Scandium(III) triflate catalyzed synthesis of 2,3-dihydro-1*H*-1,5-benzodiazepines.



Scheme 21. Scandium(III) triflate catalyzed synthesis of 3,4-dihydropyrimidin-2(1*H*)-ones.

4.7 Synthesis of α-amino amidines

α-Amino amidines (**64**) were synthesized by Keung *et al*.[46] *via* a simple, efficient and straightforward three-component reaction of amines (**35**), aldehydes (**19**), and isonitrile (**50**) using scandium(III) triflate as catalyst in methanol at room temperature (Scheme 22).

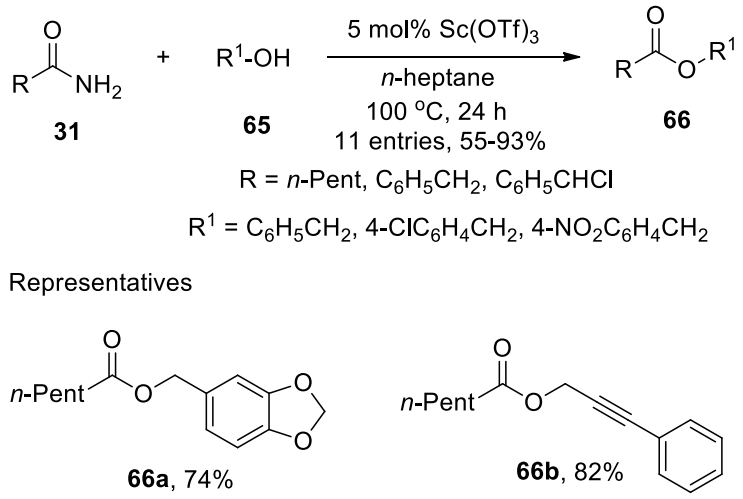


Scheme 22. Scandium(III) triflate catalyzed synthesis of α-amino amidines.

5. Carbon-Oxygen Bond-forming Reactions

5.1 Synthesis of esters

Atkinson *et al*.[47] demonstrated a scandium(III) triflate catalyzed protocol for the synthesis of esters (**66**) from the reaction of various primary amides (**31**) and alcohols (**65**) in *n*-heptane at 100 °C (Scheme 23).

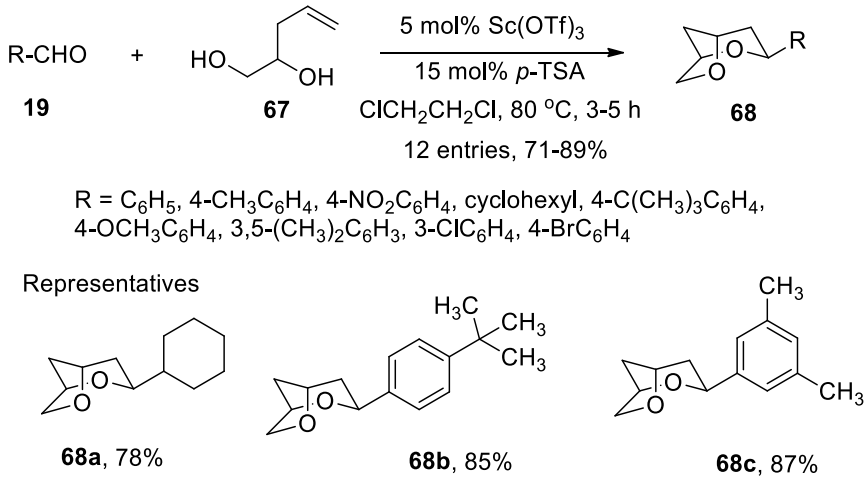


Scheme 23. Scandium(III) triflate catalyzed synthesis of ester using primary amides.

5.2 Synthesis of 2,6-dioxabicyclo[3.2.1]octane derivatives.

Reddy *et al*. (Scheme 24)[48] demonstrated an efficient straight forward strategy for the synthesis of aryl and alkyl substituted 2,6-dioxabicyclo[3.2.1]octane derivatives (**68**) *via* an intramolecular Prins cyclization of

aldehydes (**19**) with pent-4-ene-1,2-diol (**67**) in the presence of 5 mol% scandium triflate and 15 mol% *p*-toluenesulfonic acid in dichloroethane at 80 °C.



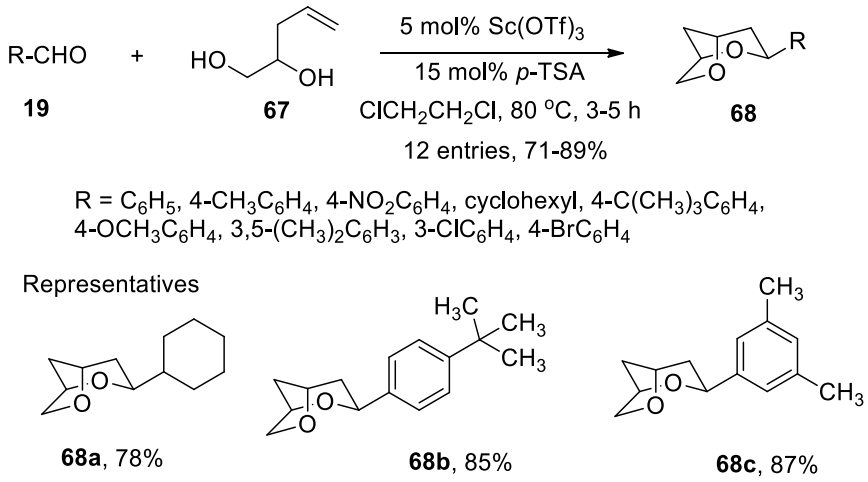
Scheme 24. Scandium(III) triflate catalyzed synthesis of 2,6-dioxabicyclo[3.2.1]octanes.

5.3 Synthesis of sugar fused pyranopyran derivatives

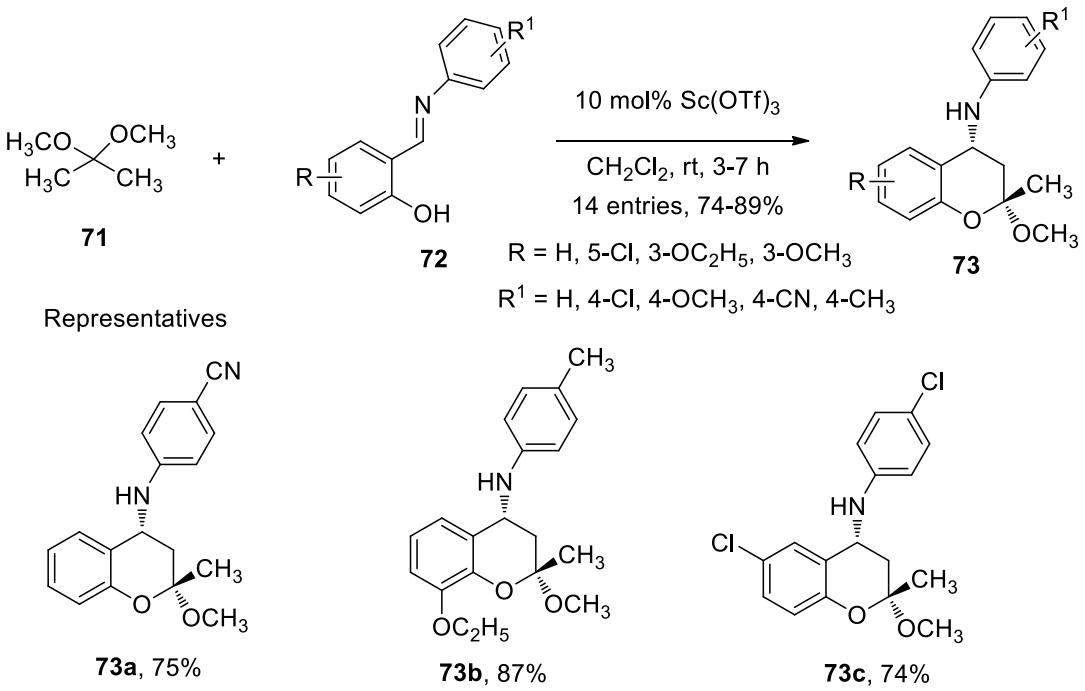
Tandem ene-Prins cyclization between an aldehyde (**19**) and *O*-prenyl derivative of a sugar aldehyde (**69**) was successfully coupled by Reddy *et al*. (Scheme 25)[49] using a catalytic amount of scandium triflate (10 mol %) at ambient temperature in dichloromethane to produce a novel series of sugar fused pyranopyran derivatives (**70**) in good to excellent yields with high enantioselectivity.

5.4 Synthesis of 3,4-dihydro-4-amino-2-methoxy-2-methyl-2*H*-1-benzopyrans

Yadav and his group (Scheme 26)[50] demonstrated a simple, facile and efficient protocol for the diastereoselective synthesis of 3,4-dihydro-4-amino-2-methoxy-2-methyl-2*H*-1-benzopyrans (**73**) with good yields from the reaction of 2,2-dimethoxypropane (**71**) with a variety of *o*-hydroxybenzaldimines (**72**) in the presence of a catalytic amount of scandium triflate at ambient temperature.



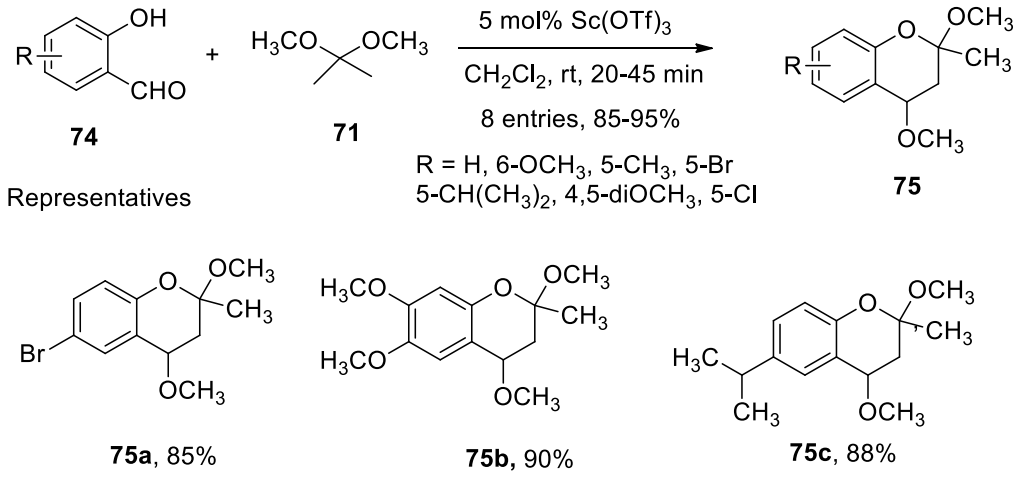
Scheme 25. Synthesis of sugar fused pyranopyran derivatives.



Scheme 26. Synthesis of 3,4-dihydro-4-amino-2*H*-1-benzopyrans.

5.5 Synthesis of 2,4-dimethoxy-2-methyl chromans

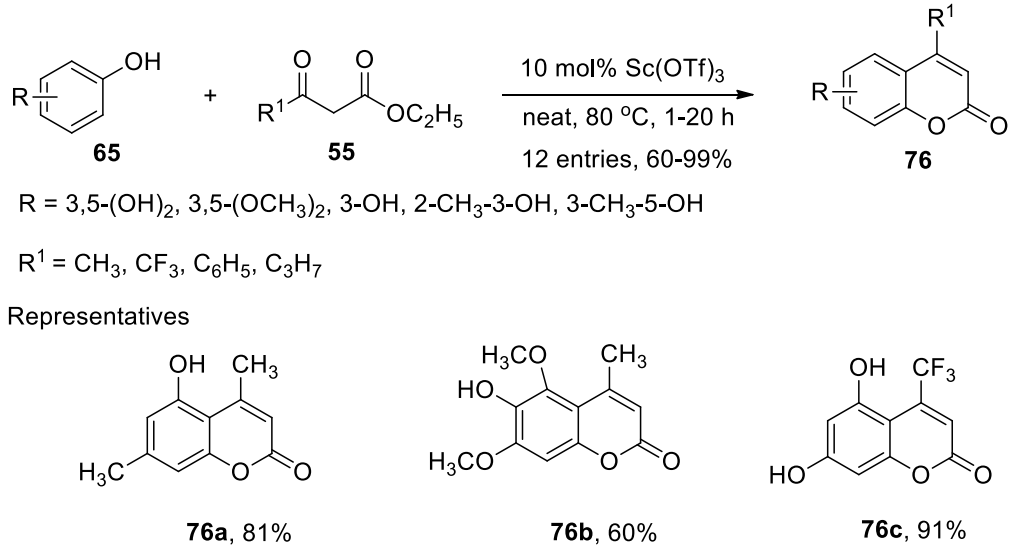
Yadav and his group (Scheme 27)[51] also reported a novel facile, straight forward, efficient procedure for the synthesis of a new class of 2,4-dimethoxy-2-methyl chromans (**75**) in high yields *via* an unusual cyclocondensation of *o*-hydroxybenzaldehydes (**74**) with 2,2-dimethoxypropane (**71**) using a catalytic amount of scandium triflate at room temperature.



Scheme 27. Synthesis of 2,4-dimethoxy-2-methyldihydrobenzopyrans.

5.6 Synthesis of coumarins

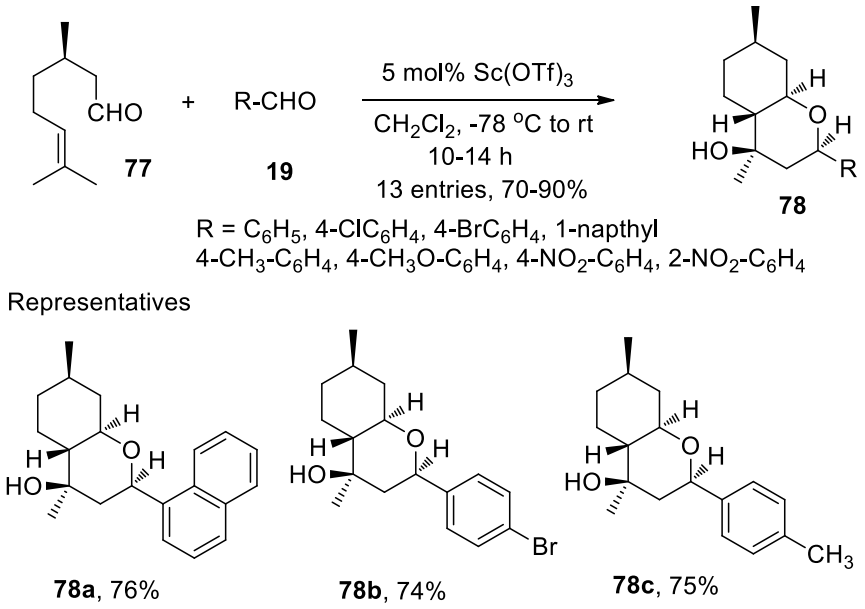
Jung *et al*.[52] demonstrated the application of scandium(III) triflate as an efficient catalyst for the synthesis of coumarins (**76**) *via* the Pechmann condensation of a variety of phenols (**65**) and β-ketoesters (**55**) under neat conditions at 80 °C (Scheme 28).



Scheme 28. Scandium(III) triflate catalyzed synthesis of coumarins.

5.7 Synthesis of octahydro-2*H*-chromen-4-ols

Yadav *et al*. (Scheme 29)[53] demonstrated a tandem ene-Prins cyclization between (R)-citronellal (**77**) and aldehydes (**19**) using $Sc(OTf)_3$ as a catalyst at ambient temperature to furnish octahydro-2*H*-chromen-4-ols (**78**) in good to excellent yields with high cis-selectivity.

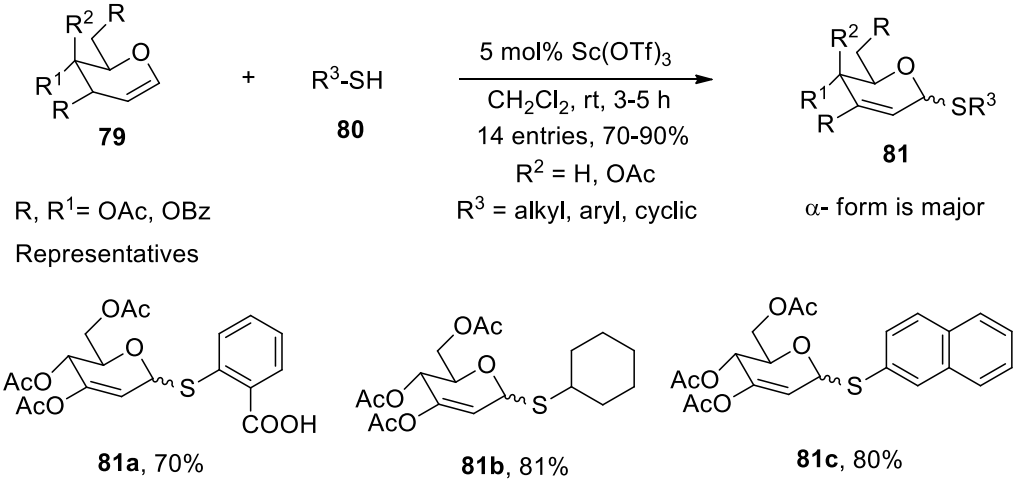


Scheme 29. Scandium(III) triflate catalyzed synthesis of octahydro-2*H*-chromen-4-ols.

6. Carbon-Sulfur Bond-forming Reactions

6.1 Synthesis of 2,3-unsaturated thioglycosides

Another scandium(III) triflate catalyzed method was developed by Yadav *et al*. (Scheme 30)[54] for the selective synthesis of 2,3-unsaturated thioglycosides (**81**) with good yields *via* the thioglycosidation of 3,4,6-tri-*O*-acetyl or benzoyl-D-glycals (**79**) with various thiols (**80**) in dichloromethane at room temperature.

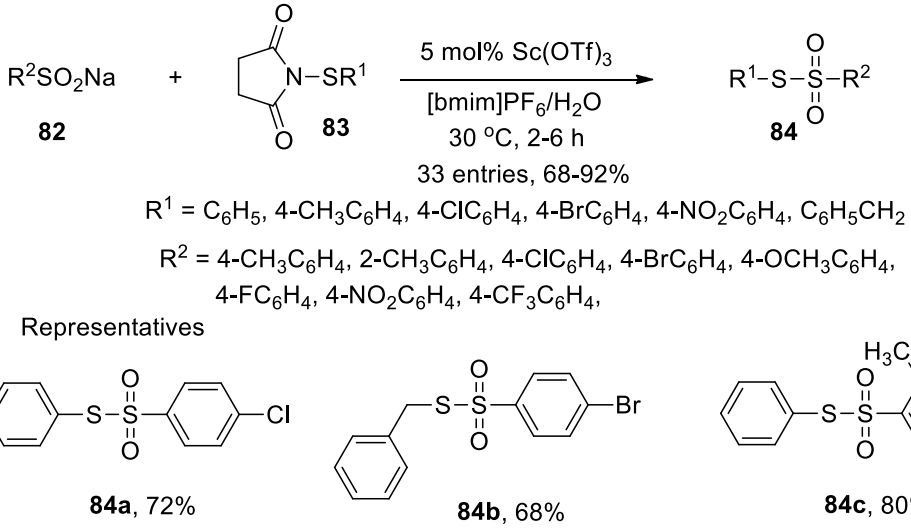


Scheme 30. Scandium(III) triflate catalyzed synthesis of 2,3-unsaturated thioglycopyranosides

7. Sulfur-Sulfur Bond-forming Reactions

7.1 Synthesis of thiosulfonates

$Sc(OTf)_3$-catalyzed sulfenylation of sodium sulfinates (**82**) with *N*-(organothio)succinimides (**83**) in ionic liquids and water mixture as cosolvent was demonstrated by Liang *et al*. (Scheme 31)[55] to afford thiosulfonates (**84**) in moderate to excellent yields at ambient temperature. They also successfully recovered and reused the ionic liquid containing $Sc(OTf)_3$ for several reaction cycles without any significant loss of catalytic activity.

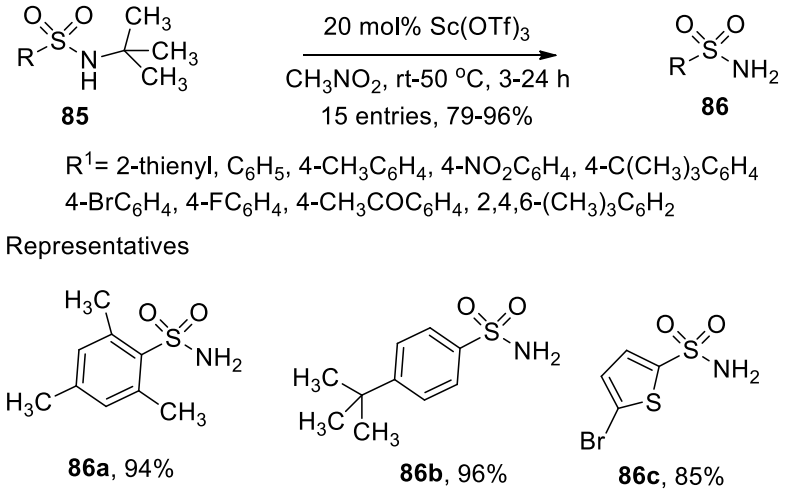


Scheme 31. Scandium(III) triflate catalyzed synthesis of thiosulfonates.

8. Other Reactions

8.1. Deprotection of tert-butyl aryl sulfonamides

Mahalingam *et al*.[56] developed a mild and high-yielding method for removal of a variety of *tert*-butyl protecting group from the *N*-substituted aryl sulfonamides (85) to form the corresponding sulfonamides (86) utilizing $Sc(OTf)_3$ as catalyst in nitomethane at ambient temperature (Scheme 32).



Scheme 32. Scandium(III) triflate catalyzed deprotection of *tert*-butyl aryl sulphonamides.

9. Conclusions

The present review offers an up-to-date literature on the latest developments of $Sc(OTf)_3$-catalyzed organic transformations specially carbon-carbon and carbon-heteroatom bond forming reactions reported during the last decade. Therefore the present review will surely make some impacts on the on-going developments of triflate salts catalyzed organic transformations as it is one of the thrusting areas for today's organic methodologists worldwide.

10. Acknowledgements

The author is thankful to the Indus International University, Una, Himachal Pradesh, India for support and the Kartha Education Society, Mumbai, India for financial help. Special thanks are due to Mr. Hridaynath Bhattacharjee, Research Scholar, Department of Chemistry, University of Saskatchewan, Saskatoon, Canada, for his able assistance throughout.

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This paper is dedicated to **Prof. (Dr.) Goutam Brahmachari** who is pictured above.

