

# Applications of Bismuth(III)salts in Organic Synthesis

# Anil Kumar

# Department of Chemistry, Govt. Degree College, Akhnoor-181201.

**Abstract:** In this review, recent applications of inexpensive and commercially available bismuth(III) salts in organic synthesis will be highlighted. The application of bismuth(III) salts in the development of new methodologies or synthetic routes that lead to compounds of pharmaceutical interest will be a matter of discussion. This review illustrates significant role of bismuth salts in the formation of carbon-carbon, carbon-nitrogen, carbon-sulphur bonds, as well as oxidation reaction, protection and deprotection of alcohols and carbonyl compounds.

**Keywords:** Bismuth (III) salts, aliphatics, alicyclics, catalytic reaction, amino acids, peptides, solid state.

## **1. Introduction**

Bismuth salts have been known since ancient times. The word bismuth has been derived form the German word "Weissmuth". Despite being a heavy metal, bismuth and bismuth (III) salts are considered safe, non-toxic and non-carcinogenic [1]. Bismuth an its compounds are being used in different medicinal preparations for over four hundred years. Bismuth has an electronic configuration of [Xe]4f<sup>14</sup>5d<sup>10</sup>6s<sup>2</sup>6p<sup>3</sup>, and due to the weak shielding of 4f-electrons, bismuth(III) salts exhibit Lewis acidity. Most of the bismuth (III) salts are relatively non-toxic, easy to handle and can tolerate small amounts of moisture. The use of inexpensive and easily available bismuth(III) salts in organic chemistry [2] as well as in medicinal chemistry [3] has been the focus of several reviews.

## 2. Carbon-carbon bond formation

The use of bismuth(III) chloride and bismuth(III) trifluoromethanesulfonate for the acylation of aromatic compound has been reported (Scheme-1) [4] Bismuth(III) triflate has been used as an efficient catalyst for the Friedel-Craft's acylation of variety of substituted benzenes[5,6].





The formation of para isomer was reported in all cases. The triedel-Craft's acylation of anisole catalysed by BiCl<sub>3</sub> as well as BiO<sub>3</sub> has been reported [7].

Bismuth(III) salts, particularly BiCl<sub>3</sub> form  $\pi$ -complexes with aromatic compounds and it has been suggested that this might facilitate solubility of BiCl<sub>3</sub> and hence lead to reaction in homogeneous conditions. The acylation of aromatic ethers under microwave irradiation catalyzed by bismuth(III) salts has also been reported (Scheme 2) [8].



The Lewis acid-**Setuysed** hydrative acylation of aromatic=compounds with carboxylic acids to afford aromatic ketones is an attractive Friedel-Crafts reaction; Shimade and co-workers reported the preparation of 1-tetralones in good to high yields by direct dehydrative cyclization of 4-arylbutyric acids in the presence of bismuth(III) salts (Scheme 3) [9].





#### Scheme-3

Further the reaction of ansole with different benzylating reagents and acetyl acetone with benzyl alcohols or cinnamyl alcohol in the presence of Bi(OTf)<sub>3</sub>.4H<sub>2</sub>O is reported (Scheme 4 and 6).



The cross-coupling reaction of acetals with allyltrimethylsilane is an efficient method for the synthesis of homoallyl ethers. Mohan and co-workers reported that  $Bi(OTf)_{3.x}H_2O$  could also catalyze the cross-coupling reaction at room temperature which provides an alternative method for the synthesis of homoallyl ethers (Scheme 6) [10].

$$\begin{array}{c} OR' \\ R \\ OR' \\ R \\ OR' \end{array} + Me_3Si \\ \hline \begin{array}{c} Bi(OTf)_3. xH_2O \\ (1.0 \text{ mole}\%) \\ \hline CH_2Cl_2, r.t \\ 5min. - 1.5h \\ \end{array} \\ \begin{array}{c} OR' \\ 69-96\% \end{array}$$

Scheme - 6 The electroreductiveBarbier type allylation of carbonyl compounds and imines has attracted much attention from both mechanistic and synthetic viewpoints. The electroreductiveallylation of imines with allyl bromides had been carried out in a Bi  $|BiCl_3|Bu_4NBr/THF - (Al-Anode) - (Pt cathode)$  system (Scheme-7) [11].



Scheme-7



The alkylation of immonium ation to amines promoted by bismuth(III) chloride in aqueous media has been reported (Scheme 8) [12].



#### Scheme - 8

Wade and coworkers as well as Dubac and coworkers have used bismuth(III)chloride as a catalyst for Makaiyama-Aldol condensation. Bismuth(III) chloride (5 ml%) has been used as an efficient catalyst for the reaction of silylenol ethers with aldehyde at room temperature (Scheme 9) [13,14].



#### Scheme 9

Condensation of an aldehyde or ketone with an active methylene compound, the Knoevenagel reaction, is an important method to synthesize  $\alpha,\beta$ -unsaturated carboxylic acids and is typically catalysed by organic amines. A solvent free procedure for the Knoevenagel reaction catalysed by BiCl<sub>3</sub> is reported (Scheme 10) [15].



There have been reports of Reformastsky-type reactions in water using Zinc powder or tin-aluminium couple. The reaction of  $\alpha$ -halocarbonyl compounds with aldehydes in presence of BiCl<sub>3</sub>-Al occurs readily in water to yield the  $\beta$ -hydroxy compounds in good yields (Scheme-11) [16].



Scheme-11

Bismuth(III)chloride was found to be an efficient catalyst for the cyanation of aldehydes with TMSCN to form the cyanohydrin in good yield (Scheme 12). Aldehydes reacted faster than ketones, both gave very high yields fo the corresponding cyanohydrins.



# 3. Oxidationeusing Bismuth(III) salts

Bismuth nitrate impregnated on montmorillonite has been used for the oxidation of secondary alcohols to ketones (Scheme 13) [17].



The oxidation proceeds rapidly at room temperature without any pre-treatment of the catalyst or MW irradiation.  $Bi_2O_3$  also has been used for the oxidation of acyloins to diketones. The reaction can be carried out in acetic acid or with 2-ethoxyethanol as a co-solvent (Scheme 14) [18].



Scheme 14

This methodology has been used in the synthesis of several natural products including the conversion of 2-hydroxypulegone to diophenolene. Several oxidative transformations of the compounds containing a steroid ring are also known.

The oxidation to benzoins to benzils using bismuth nitrate in aqueous acetic acid has also been reported (Scheme 15) [19].



Scheme - 15

However, the oxidation of pyridoin and furoin was not successful. Recently, bismuth triflate has been used for the synthesis of bis(indolyl)methanes by grinding approach. The above compounds were obtained in 75-80% yield (Scheme 16) [20].



Further, the oxidation of  $\alpha$ -ketols to carboxylic acid has been achieved with bismuth mandelate and molecular oxygen as the oxidant (Scheme -17) [21].

$$\begin{array}{c} O \\ R^{1} \\ & \downarrow \\ OH \end{array} \xrightarrow{R^{2}} Bi(III) \text{ mandelate} \\ O_{2} \\ O_{2} \\ \\ O_{2}$$

## Scheme-17

Oxidation of the substrate is accelerated in the presence of DMSO. The main advantages of this system are cheap and clean oxidant, functional group selectivity. Since alcohols, germinal diols and olefins are not affected under the reaction conditions.

Suzuki et.al reported the antioxidation of cyclohexane catalyzed by insoluble bismuth sulfate in acidic solvents (Scheme 18) [22].



Major product of this reaction is 1-cyclopentene-1-carboxaldehyde. The product distribution was found to be dependent upon the solvent used. In benzene, the main products were cyclohexenol 2, and cyclohexenone 3. 4-methylcyclohexene was oxidized to the corresponding aldehyde in high selectivity while the presence of an alkyl group at 1,2 or 3-position retarded the reaction. The oxidative cleavage of substituted styrene oxides to corresponding carboxylic acid in DMSO is catalyzed by bismuth(III)mandelate (Scheme 19) [23,24]. The reaction is accelerated by the presence of electron donating groups on the phenyl ring. Further the oxidation of epoxides to cyclic carbonates using molecular oxygen as the oxidant and BiBr<sub>3</sub> as the catalyst has been reported (Scheme-20) [25].



This reaction is catalyst and solvent specific. When the reaction was carried out in DMSO, only the carboxylic acid was obtained (Scheme-21).



Scheme-21

The oxidation of sulfides to sulfoxides using  $Bi(NO_3)_3.5H_2O$  in acetic acid as well as  $BiBr_3-Bi(NO_3)_3.5H_2O$  in benzene has been described (Scheme-22 and 23) [26-21].



Nitration 
$$R^1 - S - R^2 \xrightarrow{\text{BiBr}_3(5 \text{ mol}\%) - \text{Bi}(\text{NO}_3)_3.5\text{H}_2\text{O}(10 \text{ mol}\%)}_{\text{air, benzene, rt}} R^1 - \overset{\text{O}}{S} - R^2$$
  
Nitropyrazoles have been nitrated using Bismuth nitrate impregnated on montmorillonite K-10 in excellent yield by Ravi et.al (Scheme 24) [28].

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Bismuth nitrate impregnated on montmorilloniteK-10 has been used effectively for the synthesis of various nitro compounds of biological significance in high yield (Scheme 25) [29].



#### Scheme-25

Aromatic hydrocarbons are nitrated by Bi(NO<sub>3</sub>)<sub>3</sub>.5H<sub>2</sub>O impregnated on K-10 montmorillonite in the presence of acetic anhydride (Scheme 26) [30].



## Scheme-26

The choice of the solvent altered the product distribution e.g. the nitration of toluene gave the highest proportion of the para isomer in CCl<sub>4</sub> as the solvent. There was no nitrate product when acetonitrite was used as the solvent.

Bismuthnitrate impregnated on montmorillonite has further been used in microwave assisted facile nitration of  $\beta$ -lactams. This method, in principle, can provide 3,4-cis and trans nitro substituted  $\beta$ -lactams (Scheme 27) [31].



#### Scheme-27

Bi(NO<sub>3</sub>)<sub>3</sub>/SiO<sub>2</sub> has been established as an excellent and eco-friendly nitrating agent for Eugenol. Spectral analysis confirm that 5-nitroeugenol is the sole product in all the cases. No oxidized or isomerized product could be detected (Scheme 28) [32].



Bi(NO<sub>3</sub>)<sub>3</sub>.5H<sub>2</sub>O has been used smoothly at room temperature for the oxidative deprotection of 5,5acetals to the corresponding carbonyl compounds in good yield (Scheme 29) [33].



 $R^1$  = P-MeOC<sub>6</sub>H<sub>4</sub>, Me(CH<sub>2</sub>)<sub>6</sub>, Me(CH<sub>2</sub>)<sub>5</sub>,  $R^2$  = H, Me, Et,  $R_3$  = (CH<sub>2</sub>)<sub>2</sub>, (CH<sub>2</sub>)<sub>3</sub>, Ph, Et Common methods for deprotection of 5,5-acetals require the use of heavy metals such as copper(II), mercury(II), silver(I) and an oxidizing agent such as molecular halogen, N-halosuccinimides or MCPBA. Thus the use of relatively non-toxic bismuth salt offers significant improvement over these existing methods.

Ketoximes undergo deprotection in CH<sub>3</sub>CN acetone / H<sub>2</sub>O (3:6:1) in the presence of 20-40 mol% BiBr<sub>3</sub>/5mol% Bi(OTf)<sub>3</sub> (Scheme-30) [34].



#### Scheme-30

Bismuth(III) chloride and bismuth(III) triflate were found to be good catalyst for the cleavage of 2-tertbutoxy derivatives of thiophenes and furans. Bismuth(III) chloride was found to be a superior catalyst to ZnCl<sub>2</sub> and FeCl<sub>3</sub>.

In cleavage reactions catalysed by Bismuth(III) triflate, there is intermediacy of a tert-butyl cation (Scheme-31) [35].



Katritzky and coworkers developed BiCl<sub>3</sub> mediated methodology for the N-derivatization in aqueous Scheme - 32 media of L-proline and pipecolinic esters (Scheme 32) [36].

This is a useful synthesis of N-substituted  $\alpha$ -amino acids, which are important due to their use in the preparation and conformation studies of peptide analogs as well as biological activity.

Bismuth(III) acetate has been used as catalyst for synthesizing azlactones from aromatic aldehydes (Scheme 33) [37].



Subramanyam et.al synthesized substituted arylphosphonates/Phosphinates via bismuth(III)chloride mediated Michaelis-Arbuzov reaction (Scheme 34) [38].



Bismuth nitrate was used as a catalyst for the diasteroselective construction of anti-4,5-disubstituted-1,3-dioxolanes via two component hemiacetaloxa-conjugate addition of  $\gamma$ -hydroxy- $\alpha$ , $\beta$ -unsaturated ketones with paraformaldehyde (Scheme 35) [39].



A highly efficient Hosomi-Sakurai reaction of Aromatic aldehydes catalyzed by Montmorillonite doped with bismuth(III) salts has been reported by Elizanov et.al. The reaction was performed on different substituted aromatic aldehydes with allyl and methallyltrimethyl-silanes (Scheme-36) [40].



Scheme-36

Bismuth nitrate on neutral alumina has been used for the new and one-pot solid supported synthesis of 1,2,4,6-tetraaryl-1,4-dihydropyridines in the solid state in (56-76%) yield (Scheme 37) [41].



Bismuth nitrate on Neutral alumina has further been employed in the synthesis of 4-Aryl-2,6dicoumarinylpyridines under solventless conditions (Scheme 38)[42]. Same catalyst has been used in the solid supported synthesis of 2,4,6-triarylpyridines (Scheme 39) [43].



Scheme-39

An efficient method has been developed for the synthesis of biologically active 1,5-benzodiazepines in one pot by reacting o-phenylenediamine and ketones catalyzed by bismuth nitrate pentahydrate at room temperature. The use of MW irradiation not only enhanced the yield of the product but also significantly reduced the reaction time. Tolerance of the present method towards various functionalities present in the substrate viz. methyl, halides, cyano, carbonyl and nitro groups generalizes scope of the present method (Scheme 40) [44].



## Scheme 40

#### **Carbon-Oxygen bond formation**

The bismuth(III) bromide catalyzed reductive homocoupling of carbonyl compounds and heterocoupling of a carbonyl compound with an alkoxysilane to afford the corresponding symmetrical and unsymmetrical ethers has been reported (Scheme 41 and 42) [45].

$$\begin{array}{c}
\stackrel{O}{R} \xrightarrow{} R' + Et_{3}SiH \xrightarrow{} BiBr_{3} (1-3 \text{ mol}\%) \xrightarrow{} R \xrightarrow{} P \xrightarrow{} R' \xrightarrow{}$$

 $R^1 = H, CH_3, CH_3CH_2$  $R^2 = PhCH_2, 2-Octyl$ 

Scheme - 42

#### **Benzylation of alcohols**

An effective bismuth triflate catalyzed benzylation of 2,4-pentanediones was performed using various free benzyl alcohols and dicarbonyl compounds [46]. The products have been isolated in good to



#### Scheme - 43

Various substituted warfarin derivatives were synthesized by the Bi(OTf)<sub>3</sub> catalyzed benzylation of 4hydroxy coumarin. (Scheme-44) [47].



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Scheme-44
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Several arenes were also benzylated by employing benzyl alcohol and benzyl acetate as the electrophiles. Yields were slightly lower for the benzyl alcohol as compared to the benzyl acetate (Scheme-45).

Ph 
$$OR$$
 +  $Bi(OTf)_3(1 \text{ mol}\%)$  Ph  $R = H, R = Ac$   
Scheme = 45

Various propargylic alcohols were treated with allyltrimethylsilane in the presence of BiCl<sub>3</sub> as the catalyst and the substituted 1,5-enynes were obtained inhigh yields (Scheme 46) [48]. BiCl<sub>3</sub> is also an efficient catalyst for the propargylation of several electron rich arenesandheteroarenes.



#### Scheme - 46

When the acetates were derived from chiral  $\alpha$ -branched para methoxybenzylic alcohols, high facial diasteroselectivities were observed [49]. The reaction with the silylenol ether derived from t-butyl

methyl keotne afforded the corresponding ketone with a good yield and a high diastereoselectivity (Scheme 47).

## **Synthesis of Pyrroles**

Bi(OTf)<sub>3</sub> was found to be an efficient catalyst for the synthesis of substituted pyrroles from 2propynylamine and methylene active compounds [50]. The reaction could be applied to various  $\beta$ dicarbonyl compounds (Scheme 48). However with  $\beta$ -ketoesters, the reaction afforded 3-acylpyrroles only.



#### **Other reactions**

1) Glycosylation reaction: Bi(OTf)<sub>3</sub> is used as a catalyst for the glycosylation of a silvl donor with a thioethylgalactosyl acceptor (Scheme-49). In this case, the desired disaccharide was obtained with nearly complete  $\alpha$ -steroselectivity ( $\alpha/\beta = 20$ :1) [51].



## 2. Wagner – Meerwein rearrangement

Bi(OTf)<sub>3</sub> as a catalyst for the Wagner-Meerwein rearrangement of lupine derivatives with expansion of ring E and formation of an additional O-containing ring was reported [52]. This process has also been extended to other terpenes, such as (-) caryophyliene oxide (Scheme 50). Bi(OTf)<sub>3</sub> was also used as a catalyst for the direct conversionof corticosteroids into highly functionalized 17-ketosteroids by cleavage of the C17-dihydroxyacetone side chain [53].



#### Scheme-50

#### **Alkylation reaction**

A diastereoselective construction of syn-1,3-dioxanes was reprted via bismuth nitrate catalyzed two component hemiacetal/oxaconjugate addition reaction [54].  $\delta$ -Trialkylsilyloxy and  $\delta$ -hydroxy $\alpha$ , $\beta$ -unsaturated aldehydes and ketones react with alkyl aldehydes in a highly efficient and stereoselective manner (Scheme 51). The analogous process with nitric acid provided the 1,3-dioxane in significantly lower yield, thus illustrating the superiority of bismuth salts.



The same authors reported a one-pot diastereoselective sequential two component etherification followed by an oxa-conjugate addition [55]. Reaction of an anomericacetal with a trimethylsilyloxydiene yielded a bis(tetrahydropyran) core used in the total synthesis of (+) – leucascandrolide A (Scheme 52).



# [3+2] Cycloaddition reaction

 $Bi(OTf)_3$  has been used in the (3+2) cycloaddition reaction of a series of substituted N-tosylazridines with nitriles [56]. Under these condition,  $Bi(OTf)_3$  appeared to be the best promoter among various Lewis acids and various imidazolines were obtained under mild reaction condition (Scheme-53).



#### Scheme-53

One example of enanlioselective cyclization is the synthesis of (2R)-2,5-diaryl-2,3-dihydropyrano[2,3-b]quinolin-4-ones by the reaction of 3-acetyl-4-aryl-carbostyril and an aldehyde in the presence of Bi(OTf)<sub>3</sub> and L-proline (Scheme 54) [57]. It is believed that Bi(OTf)<sub>3</sub> L-proline complex is formed in situ.





# Synthesis of Furans and Peroxides

Bi(OTf)<sub>3</sub> has been employed for tandem condensation reaction of acyloins and  $\beta$ -diketones or  $\beta$ -keto esters to afford highly substituted furans in good yield (Scheme 55) [58]. It has also been used in the synthesis of both 1,1-dihydroperoxides and symmetrical and unsymmetrical 1,2,4,5-tetraoxanes (Scheme 56) [59].



## Some recent reactions

Zhang et.al reported an efficient 1,6-allylation addition of para-quinonemethides to allylboronic acid pinacol ester catalyzed by bismuth(III)triflate. The reaction proceeds smoothly in high yields under mild conditions with only 05 - 5 mol% catalyst (Scheme 57) [60].



1,3-Cyclohexanedione has been reacted with numerous aldehydes using bismuth iodide as a catalyst giving rise to the formation of octahydroxanthenes. The reaction is proposed to follow Michael-type condensation with highly reactive diketone followed by facile dehydration to form the cyclic product (Scheme 58) [61].



#### Scheme-58

N-Amino-2-Pyridone derivatives have been synthesized in 88-98% yield in the presence of  $Bi(NO_3)_3.5H_2O$  as a catalyst (Scheme 59) [62].



The polar solvents such as ethanol afforded better yields than non-polar solvents. Halli et.al performed three component synthesis of  $\alpha$ -arylglycinescatalysed by Bismuth (Scheme 60) [63].

This method provides a general, atom-economic route to various N-protected  $\alpha$ -arylglycines starting from readily available amides, glyoxalates and (hetero) arenes with water as the only by- product.

## Cleavage of phenolic methoxy methyl ethers

A simple and efficient method for removal of phenolic methoxymethyl ethers in the presence of 30 mol% of bismuth trichloride in acetonitrile/water has been described by Best et.al (Scheme 61) [64].



New polycyclic hexahydrobenzo[c]acridines were synthesized in excellent yields by intramolecular [4+2] cycloaddition reactions of aldimines derived from aromatic amines and 2-(4-Methylpent-3-en-1-yl) benzaldehyde in acetonitrile in the presence of 10 mol% of bismuth(III)chloride. The reaction is highly diastereoselective, giving cis-fused benzoacridine derivatives preferentially (Scheme 62) [65].



# Scheme - 62

An efficient one-pot multicomponent synthesis of 3,4-dihydropyridin-2(1H)-ones/Thiones has been achieved with bismuth(III)sulfate trihydrate under solvent free conditions (Scheme 63) [66].



 $R = H, 4-OCH_3, 4-NO_2, 2-NO_2$ 

Polyhydroquindligestare source of valuable drugs. They demonstrate activity for treatment of angina pectoris and 6 hypertension [67]. Bismuth(III)bromide is an efficient catalyst for the one-pot multicomponent synthesis of polyhydroquinolines via the Hantzsch reaction (Scheme 64) [68].



#### Scheme-64

Murai et.al performed bismuth(III) catalyzed dehydrative etherification and thioetherification of phenolic hydroxy groups (Scheme 65) [69]



Scheme - 65

The reaction required equimolar amounts of the two readily available substrates that generated  $H_2O$  as the only by-product.

Bi(OTf)<sub>3</sub> has also been employed for the exo-oleifinisomerisation of  $\alpha$ -benzoyl  $\beta$ -styryl sulfones. The reactions were performed in CH<sub>3</sub>NO<sub>2</sub> and afforded  $\alpha$ -benzoyl  $\alpha$ -cinnamyl sulfones in moderate to good

yields (Scheme 66) [70]. This catalyst has also been used for tendem Meyer-Schuster rearrangement and 1,4-addition to the resulting vinyl ketone (Scheme 67) [71].



This tandem reaction can be extended to an intramolecular version, featuring a one pot dihydroquinolone synthesis.

The reaction of 2-(2-arylphenyl)vinylethers gave substituted phenanthrenes in the presence of catalytic amount of bismuth(III)triflate in excellent yields. The reaction was also applied to the construction of other polycyclic aromatic hydrocarbons, such as chrysene, helicene and pyrene having a phenanthrene backbone via regioselective cyclization (Scheme 58) [72].



Scheme - 68

# **Polysubstituted Cyclopentenones**

Lempenauer et.al has performed catalytic rearrangement of diallyl alcohols comprising a cyclic enol ether in the presence of bismuth(III)triflate under very mild conditions (Scheme 69) [73].



In this case, Bismuth(III)triflate was found to be very active catalyst for the ring rearrangement of a range of tertiary allylic alcohols to afford polysubstitutedcyclopentenones with a high degree of diasteseoselectivity.

Prakash et.al et.al has synthesized microwave induced bismuth(III)nitrate catalyzed synthesis of linear indoloquinolines. In this reaction, minimal quantity of THF is sued as solvet. This methodology provides different linear indolo[2,3-b]quinolines in good yields (Scheme 70) [74].



#### Scheme - 70

Recently, Cornella et.al. has shown that bismuth maneuvers between different oxidation states enabling access to unique redox cycles that can be harnessed in the context of organic synthesis.[75]

# Conclusion

Bismuth(III) compounds show remarkable potency as catalysts in Organic synthesis due to their nontoxicity and ease of handling. In nutshell, Bi(III) compounds ( $BiX_3$ , X= Cl, Br, NO<sub>3</sub>, OTf etc.) are moisture and air- tolerant Lewis acids which have widely used as green catalysts in diverse organic synthesis.

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