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# Review on synthesis of acyclic and cyclic oxime ethers

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**Abstract:** Oxime ethers have attracted much attention due to their potential biological activities and wide variety of synthetic applications. Developing more efficient methods for synthesis of oxime ethers has been the subject of numerous of papers in recent years. This review surveys literature methods for the synthesis of acyclic and cyclic oxime ethers.

**Keywords:** oxime ether, isoxazoline, nitrile oxide, 1,3-dipolar cycloaddition**,** oxazete

## 1. Introduction

The name oxime ether is an abbreviation of oxy-imine ether. As one of the prominent medicinal motifs, the oxime ether group is featured in a large number of pharmaceutically important compounds and is widely applied in variety pesticides.[1,2] For example, oxiconazole **1** (Figure 1) with brand name of oxistat, is an antifungal drug marketed worldwide for the treatment of skin infections.[3-5] Fluvoxamine maleate **2** is used for treating obsessive compulsive disorder.[6-9] A series of novel thioaryl naphthylmethanone oxime ether analogs **3** exhibit excellent anticancer activities towards various cancer cells.[10] Roxithromycin **4** is a semi-synthetic macrolide antibiotic which was introduced in the 1980s, and is used to treat infections caused by bacteria.[11-14] Fenpyroximate **5** is a pesticide with oxime ether motif, this compound is very active against acaricide and widely used around the world.[15-17] Wang's group showed that a series of benzoylphenylureas **6** have excellent larvicidal activities against oriental armyworm.[18] These representative examples show that the oxime ether group offers very attractive options for drug design of various pharmacological agents, due to their relative ease of synthesis and their impressive medicinal chemistry applications.

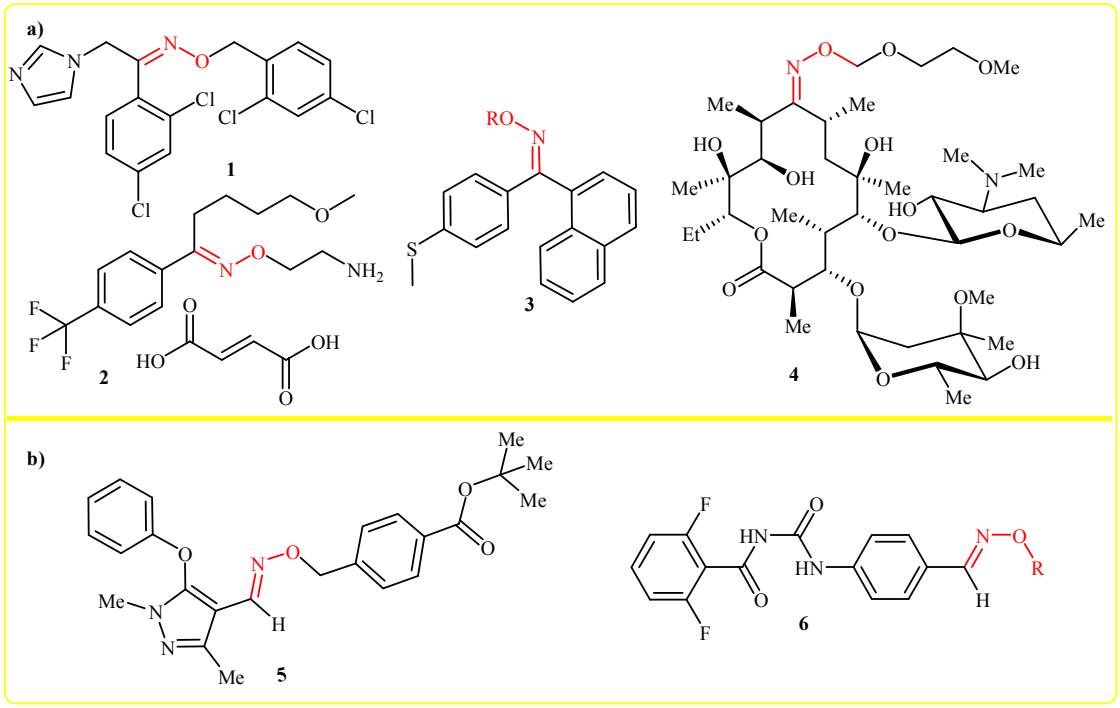


**Figure 1.** a) Some medicine containing oxime ether functional group; b) two pesticides containing oxime ether functional group.

Oxime ethers are important and versatile intermediates in organic synthesis. These compounds were successfully transformed into amines,[19-24] 1,2-aminoalcohols,[25] α- and β-amino acides,[26, 27] hydroxylamines,[28-41] nitriles,[42-44] pyridines,[45-50] benzofuranes,[51-53] indoles,[54, 55] pyrroles,[56, 57] pyrazines,[58] isoquinolines,[59, 60] isoxazolesm,[61-64] 8-hydroxytetrahydroquinolines,[65] aminocyclopentitols,[66] aziridines,[67] fluorenones,[68] diarylmethylidenefluorene and phenanthrene.[69] Furthermore, oxime ether is an elegant directing-group for activation of aromatic or vinylic C-H bonds for construction of new C-O, C-X and C-N bonds by metal-catalyzed cross-coupling reactions.[70-75] Considering the widespread synthetic applications and biological activities of oxime ethers and extensive attention on these compounds in recent years, especially in the field of metal-catalyzed cross-coupling reactions, there is an urgent need for a review article on the synthesis of titled compounds. In this review, we describe variety of methods for the synthesis of oxime ethers. We have classified these synthetic reactions based on the type (acyclic and cyclic), the starting materials (e.g. synthesis from oxime and alkyl halides, oxime and aryl halides, and oxime and epoxides) and the reactions type (e.g. cross-coupling reactions between oxime and arylboronic acids, and 1,3-dipolar cycloaddition of nitrile oxides to carbon-carbon double bonds). The most detailed discussion is focused on the synthesis of acyclic oxime ethers. It should be noted that we have not discussed synthesis of six membered cyclic oxime ethers, since it has recently been described in another publication.[76] To summarize, the main

methods for the synthesis of acyclic and cyclic (four and five membered cycles) oxime ethers is depicted in Figure 2.

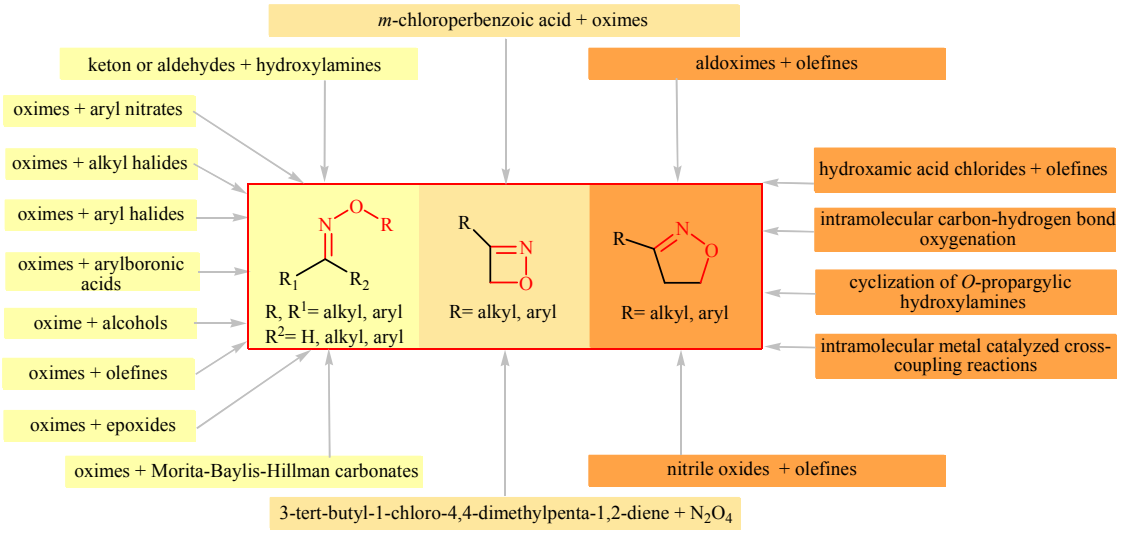
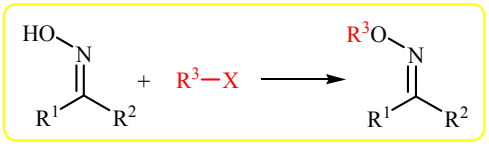



**Figure 2.** The main methods for synthesis of acyclic and cyclic oxime ethers.

## 2. Synthesis of acyclic oxime ethers

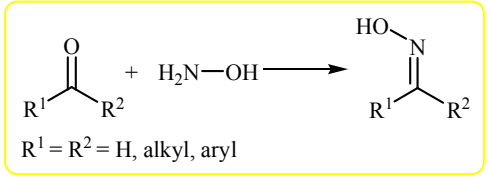
### 2.1. From oximes and alkyl halides

The best-known method for synthesis of acyclic oxime ethers is the reaction of oximes with alkyl- and aryl halides (Scheme 1).[76-95]




**Scheme 1.** Synthesis of oxime ethers from oximes and alkyl(aryl) halides.

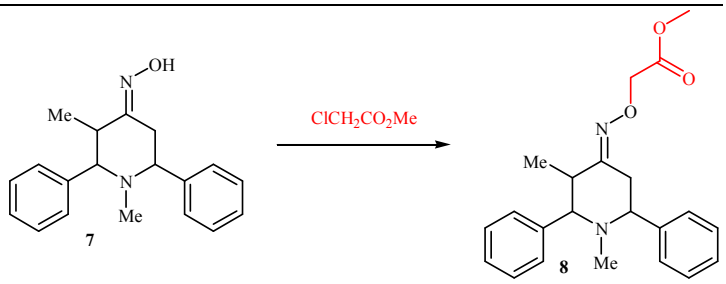
A safe method for the preparation of oximes involves reaction of carbonyl compounds (aldehydes and ketones) with hydroxyl amines (Scheme 2).[96-101] This type of reaction was introduced by Schiff [102] in 1864 and nowadays is the best choice for the synthesis of titled compounds.[103-105]




**Scheme 2.** Synthesis of Oximes *via* condensation of carbonyl compound with hydroxyl amine.

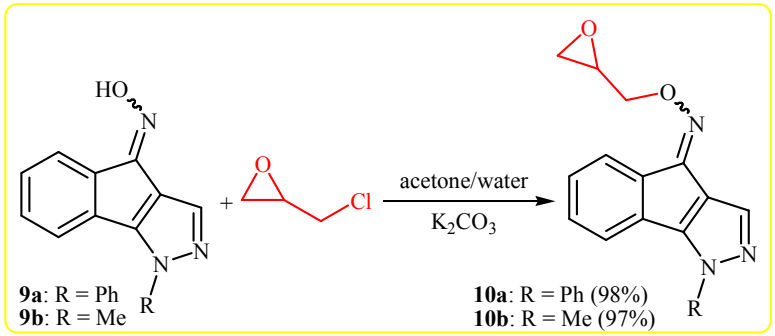
The alkylation of the oxygen atom of oxime moiety with alkyl halides has been performed using various base, such as sodium hydride,[76,77,80,82,90,91] sodium hydroxide,[86] potassium hydroxide,[81] and potassium carbonate.[87,93,94] As well as, the system Na/alcohol has also been utilized.[78,85,88] Using potassium carbonate as base and acetonitrile as solvent clearly accelerated the alkylation of the oxime moiety compared to other bases/solvents, and the desired products were synthesized in good yields (Table 1).[93]

**Table 1.** Synthesis of oxime ether **8** from oxime **7** and methyl 2-chloroacetate in the presence of $K_2CO_3$ in MeCN




| Entry | Solvent | Base | time (h) | Yield (%) |
|---|---|---|---|---|
| 1 | $CH_2Cl_2$ | $K_2CO_3$ | 24 | - |
| 2 | 1,4-Dioxan | $K_2CO_3$ | 24 | 15 |
| 3 | THF | $K_2CO_3$ | 18 | 30 |
| 4 | DMF | $K_2CO_3$ | 18 | 40 |
| 5 | Acetonitrile | $K_2CO_3$ | 8 | 70 |

Recently, an excellent method for generation of oxime ethers **10a,b** from oximes **9a,b** and epichlorhydrin have been reported by Cerra and co-workers using acetone/water/$K_2CO_3$ system (Scheme 3).[94]

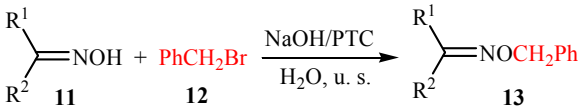



**Scheme 3.** Synthesis of oxime ethers **10a,b** from oximes **9a,b** and epichlorhydrin

Synthesis of oxime ethers from oximes and halides in the presence of phase transfer catalysis has been the subject of a number of papers. However, preparation of oxime ethers using this method resulted in poor to moderate yields of desired products.[80,81] In 2009, Li and co-workers reported the benzylation of oximes by combination of phase transfer catalysis and

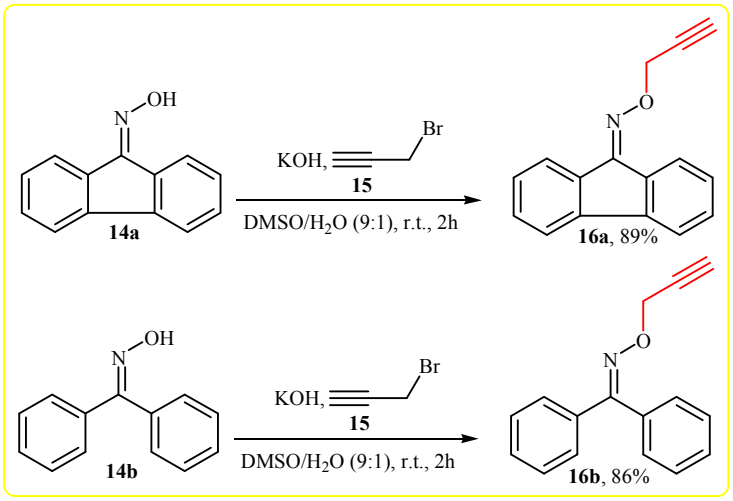
ultrasound irradiation. They tested several catalysts and solvents, and the system NaOH/benzyldimethytetradecylammonium chloride/$H_2O$ was found to be superior. Under optimized conditions, the reaction tolerates both electron-donating and electron-withdrawing groups in the phenyl ring of oxime and gave corresponding products in good to excellent yields (Table 2).[91] To compare the yields of product **3a** (78%) to the same reaction which was reported under toluene/$H_2O$/NaOH/TBAB by Wang et al (76.6%),[86] it can be concluded that the latter system is superior, due to the former method which was carried out under ultrasound irradiation.

**Table 2.** The reaction of oxime **11** with benzyl bromide in aqueous media catalyzed by benzyldimethyltetradecylammoniumchloride in combination with ultrasound irradiation.




| Entry | $R^1$ | $R^2$ | Time (min) | Product | Isolated yield (%) |
|---|---|---|---|---|---|
| 1 | 2-OMe-Ph | H | 30 | **13a** | 94 |
| 2 | 4-OMe-Ph | H | 35 | **13b** | 86 |
| 3 | 4-$NO_2$-Ph | H | 30 | **13c** | 78 |
| 4 | 2,4-di-Cl-Ph | H | 30 | **13d** | 96 |
| 5 | 2-Cl-Ph | H | 20 | **13e** | 68 |
| 6 | 3-Cl-Ph | H | 50 | **13f** | 89 |
| 7 | 4-Cl-Ph | H | 20 | **13g** | 73 |
| 8 | Ph | H | 30 | **13h** | 78 |
| 9 | 4-Cl-Ph | CH3 | 60 | **13i** | 90 |
| 10 | 4-OMe-Ph | CH3 | 60 | **13j** | 60 |

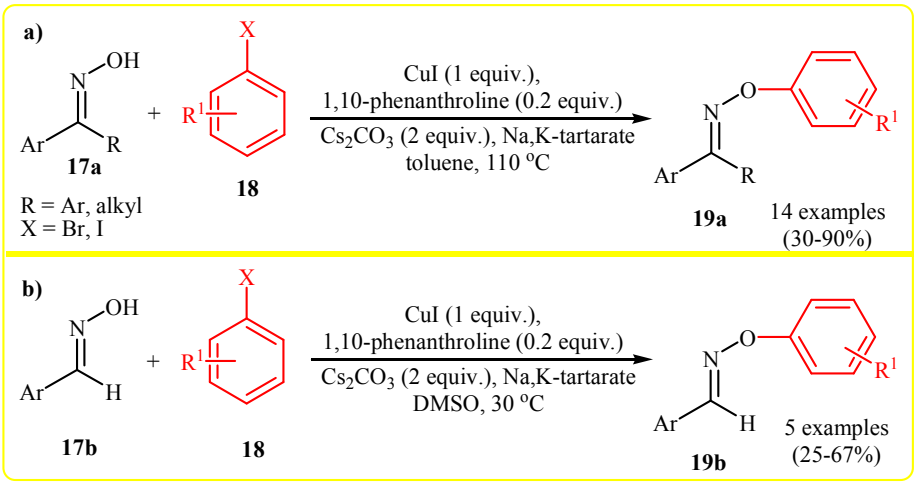
The attempts to synthesis of *O*-propargylated oximes **16a,b** with treatment of oximes **14** with propargyl bromide **15** in the presence of KOH in DMSO/$H_2O$ 9:1 resulted in products with higher than 86% yield (Scheme 4).[106]




**Scheme 4.** *O*-propargylation of oximes **14a,b**

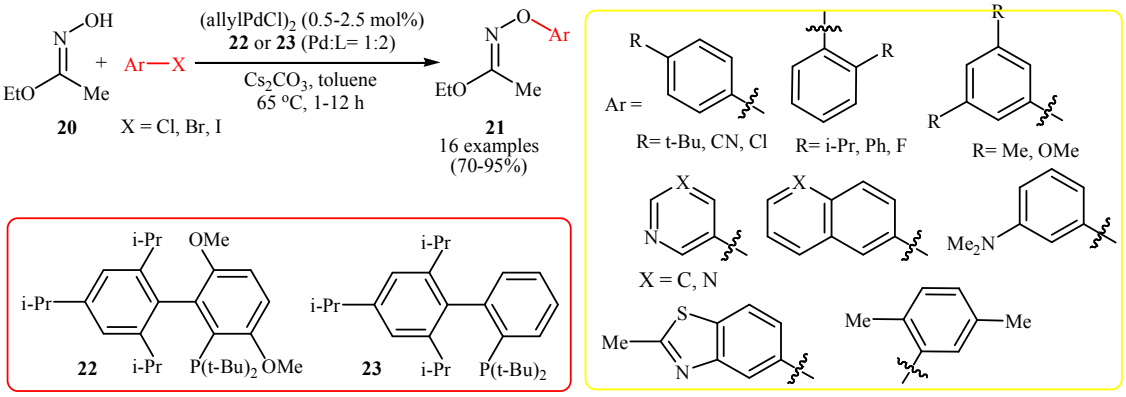
## 2. 2. From oximes and aryl halides

In 2007, the successful metal catalyzed cross-coupling of aryl halides with oximes have been reported by Maitra *et al.*[107] Oximes **17a,b** were found to undergo O-H arylation with various iodo- and bromoarenes **18** in the presence of CuI as catalyst, $Cs_2CO_3$ as base, Na- or K-tartrate as chelating agent, and 1,10-phenanthroline as a ligand in toluene or DMSO and gave corresponding *O*-aryl oximes **19a,b** in moderate to good yields (Scheme 5). Some important information of the reactions are listed below: 1) the reactions will not work with 1-iodo-4-methoxybenzene; 2) aldoximes compare to ketoximes gave lower yield of desired products; 3) the protocol is efficient for intramolecular cross-coupling reactions but not for intermolecular version; and 4) haloarenes bearing electron-withdrawing substituents gave higher yield of products than haloarenes with electron-donating substituents.



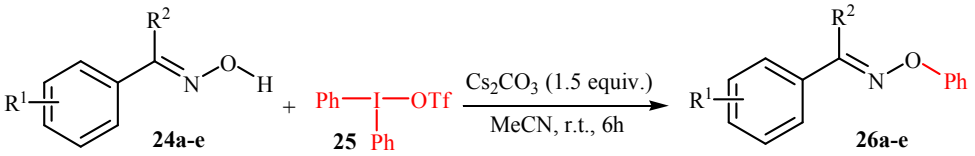

**Scheme 5.** a) Cross-coupling of ketoximes with haloarenes/ catalytic CuI in refluxing toluene; b) cross-coupling of aldoximes with haloarenes/ catalytic CuI in DMSO at 30 °C.

Following this work, Buchwald research team in 2010, has investigated the *O*-arylation of ethyl acetohydroximate **20** with aryl chlorides, bromides, and iodides using $(allylPdCl)_2$ as catalyst, *t*-BuBrettPhos **22** or *t*-BuXPhos **23** as ligand in toluene at 65 °C. This method has several advantages such as good to excellent yields, short reaction time and broad substrate scope (Scheme 6). Key to the success of this reaction was the use of bulky biarylphosphine ligands **22** and **23**, which promote C-O reductive elimination under relatively mild conditions.[108]




**Scheme 6.** Palladium-catalyzed O-arylation of ethyl acetohydroximate.

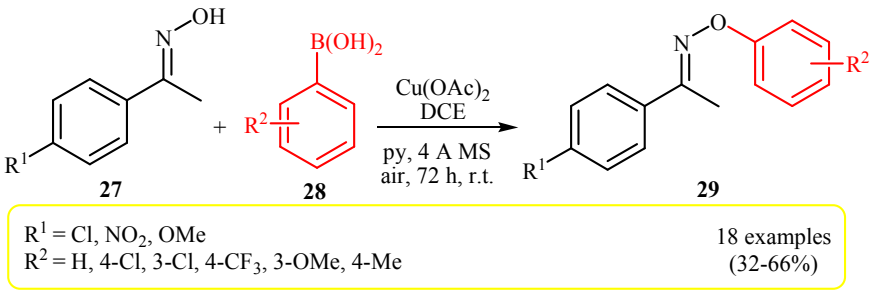
With the objective of designing a comprehensive protocol to *O*-arylation of oximes, the scope of electrophilic partners was extended to diaryliodonium salts under transition-metal-free cross-coupling conditions. Several bases and solvents were tested and the system *t*-BuOK/DMF at room temperature was found to be superior. Under optimized conditions, both electron-donating and electron-withdrawing groups on either coupling partners were well tolerated and gave desired products in good to high yields (40-96% for 26 examples).[109] The use of $Cs_2CO_3$ as base and acetonitrile as solvent provided the same products in comparable yields (Scheme7).[110]




**Scheme 7.** The *O*-arylation of oximes **1a-e** with diaryliodonium salts

### 2.3. From oximes and arylboronic acids

In 2009, Meyer and Feng research teams independently reported the copper catalyzed O-arylation of oximes with arylboronic acids.[111,112] The reaction was undertaken at room temperature using $Cu(OAc)_2$ as catalyst and pyridine as base in DCE. This method afforded *O*-arylated acetophenone oximes **29** in moderate yields with various *meta* and *para*-substituent arylboronic acids **28** (Scheme 8). It should be noted that using polystyrene supported copper catalyst **30** (Figure 3) in aforementioned reaction gave relatively better results.[113]




**Scheme 8.** $Cu(OAc)_2$-mediated *O*-arylation of oximes **27** with phenylboronic acids **28**

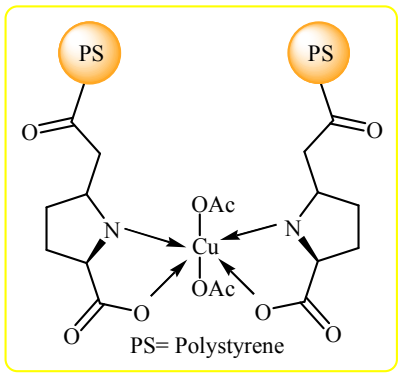
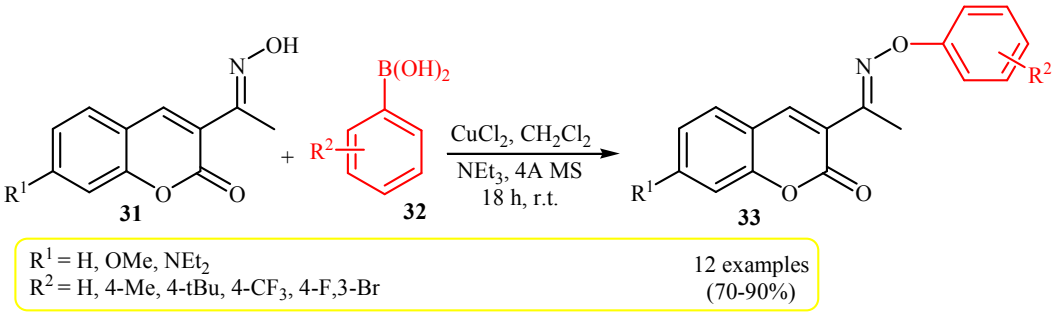



**Figure 3.** Chemical structure of polymer supported copper **30**.

Recently, Bora and co-workers investigated the efficiency of different bases in titled reaction and showed the system $Cu(OAc)_2/Cs_2CO_3$ in DMF gave corresponding *O*-arylated acetophenone oximes **33** in higher yields than previous methods.[114] Coumarins **31** were found to undergo efficient *O*-arylation with various arylboronic acids **32** in the presence of $CuCl_2$ as catalyst and $NEt_3$ as base (Scheme 9). This system shows good reactivity for a range of arylboronic acids. *Para* electron-rich aryl boronic acids and phenylboronic acid worked well under these reaction conditions. *Meta*- and *para* electron-deficient arylboronic acids gave coupling products in moderate to good yields.[115]




**Scheme 9.** $CuCl_2$-promoted *O*-arylation of (hydroxyimino)ethylcoumarin **31** with arylboronic acids **32**

To develop an efficient protocol for the synthesis of *O*-aryl oximes *via* cross-coupling reaction, Mulla and co-workers have investigated the *O*-arylation of acetophenone oximes with arylboronic acids in the presence of recyclable and heterogeneous copper fluorapatite

(CuFAP) catalyst in methanol, and good to high yields of desired products was observed (61-96% for 30 examples). A plausible catalytic cycle is depicted in Figure 4.[116]

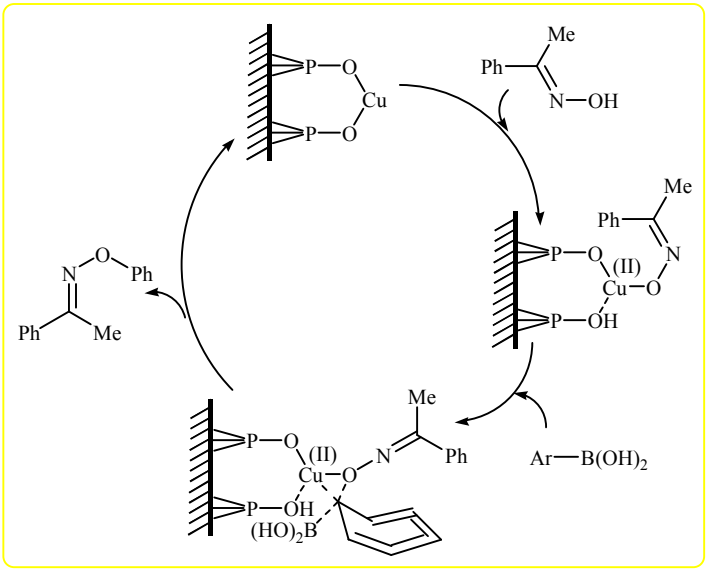
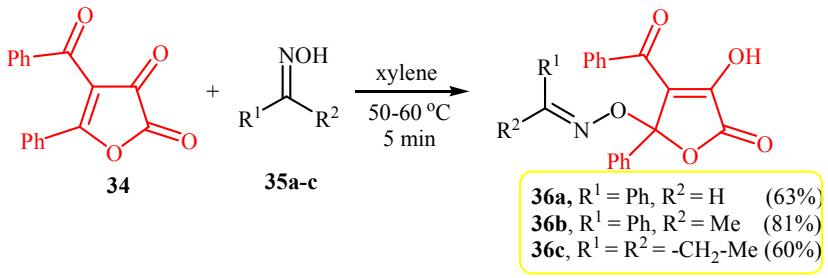



**Figure 4.** Plausible catalytic cycle for *O*-arylation of acetophenone oximes with arylboronic acids

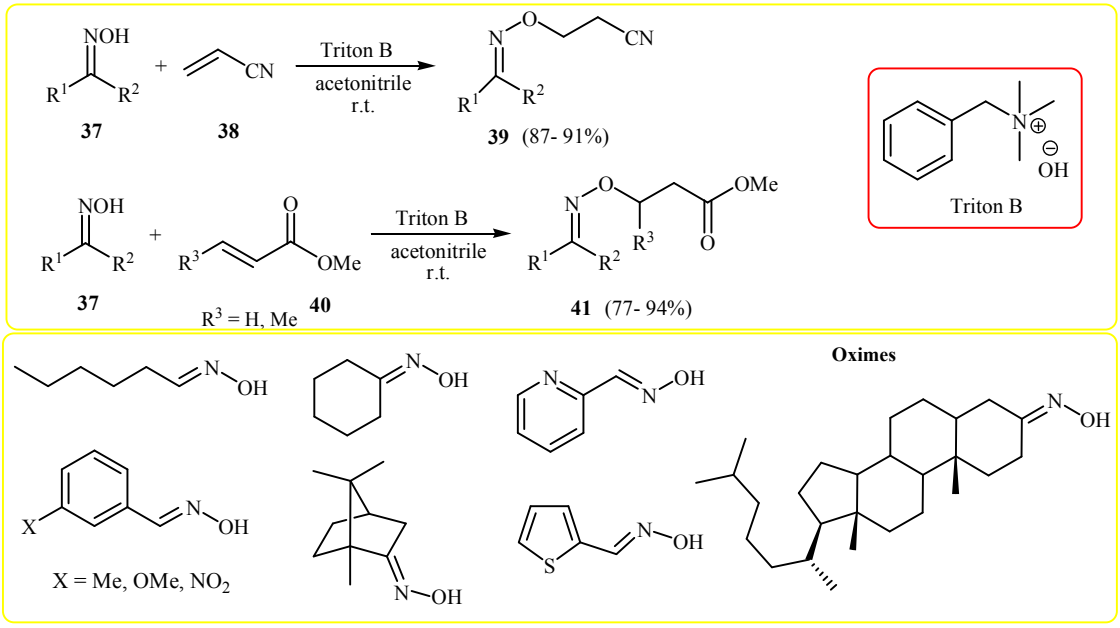
## 2.4. From oximes and olefins

Preparation of oxime ethers from oximes and olefins has been the subject of a number of papers. One of the earliest report of the successful formation of allylic oxime ethers *via* Michael addition of oximes have been published by Akcamur and Kollenz in 1987 (Scheme 10).[117] This reaction showed an attractive route for the conversion of oximes into oxime ethers in good to high yields at mild reaction conditions and short reaction times.




**Scheme 10.** Formation of allylic oxime ethers **36** *via* Michael addition of oximes **35**.

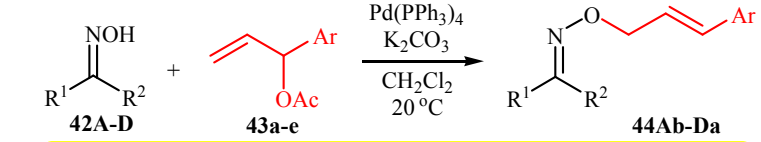
Meshram *et al*. expanded the efficiency of this method by using the Triton B as a nonmetallic organic base. All of aliphatic and aromatic oximes **37** with both electron-donating and electron-withdrawing substituents in treatment with α,β-unsaturated nitriles **38** or α,β-unsaturated esters **40** gave corresponding oxime ethers in good to high yields (Scheme 11).[118]




**Scheme 11.** Triton B–catalyzed Michael addition of oximes to electron-deficient alkenes.

A robust process for the synthesis of allylic oxime ethers involves the reaction of oximes with $\pi$-allyl metal complexes.[119-121] Treatment of oximes **42** with α,β-unsaturated acetates **43** in the presence of $Pd(PPh_3)_4$ as catalyst gave allylic oxime ethers **44** in good to high yields (Table 3). Interestingly, when the reaction was carried out under the $[IrCl(cod)]_2/Et_2Zn$/THF system, instead of oxime ethers **44**, the branched oxime ethers **45** was observed as desired products in good to high yields ( Scheme 12).[119] Previously, this result has been reported by Takeuchi in allylic amination.[122]

**Table 3.** Palladium-Catalyzed Reaction of **42A–D** with Acetates **43a–e**[a]



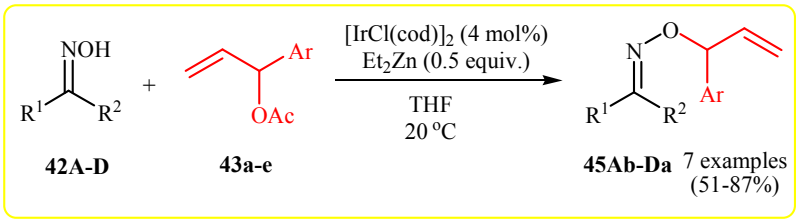

A: $R^1$ = Ph, $R^2$ = H
B: $R^1$ = Ph, $R^2$ = Ph
C: $R^1 = R^2 = (CH_2)_5$
D: $R^1 = CO_2Me$, $R^2$ = H

a: Ar = Ph
b: Ar = 4-$F_3C$-Ph
c: Ar = 4-OMe-Ph
d: Ar = 1-Naph
e: Ar = 2-Naph

| Entry | Oxime | Acetate | Product | Yield (%) |
|---|---|---|---|---|
| 1 | **42A** | **43b** | **44Ab** | 80 |
| 2 | **42A** | **43c** | **45Ac** | 71 |
| 3 | **42A** | **43d** | **45Ad** | 81 |
| 4 | **42A** | **43e** | **45Ae** | 73 |

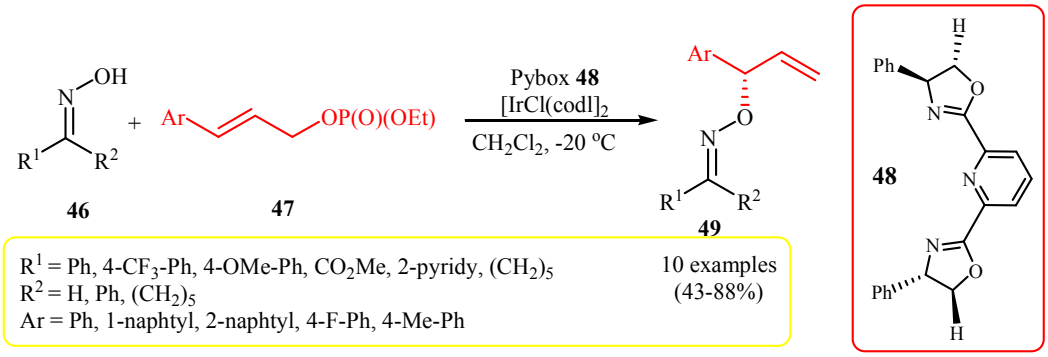
| | | | | |
|---|---|---|---|---|
| 5 | **42B** | **43a** | **45Ba** | 67 |
| 6 | **42C** | **43a** | **45Ca** | 51 |
| 7 | **42D** | **43a** | **45Da** | 75 |

[a]Reactions were carried out with **42A–D** (1 equiv) and **43a–e** (1.5 equiv) in the presence of $Pd(PPh_3)_4$ (6 mol%) and $K_2CO_3$ (1 equiv) in $CH_2Cl_2$.



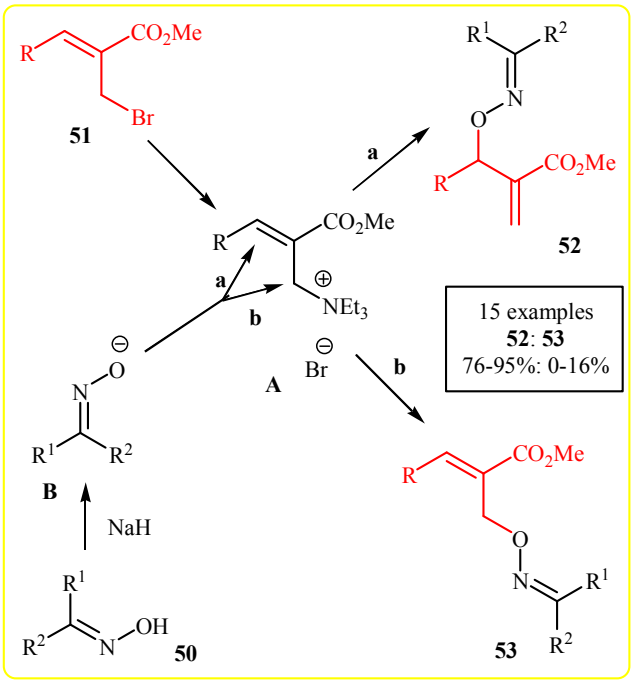

**Scheme 12.** Iridium-catalyzed reaction of **42A–D** with acetates **43a–e**.

With the objective of designing a comprehensive protocol to high regio- and enantioselective synthesis of the branched oxime ethers, the scope of electrophilic partners were extended to α,β-unsaturated phosphates (Scheme 13). The $[IrCl(cod)]_2$/pybox **48**/ $Ba(OH)_2{\cdot}H_2O/CH_2Cl_2$ system was found to be optimal for this reaction. Notably, the system works well for the allylic substitution of phosphates with amines.[120,123]



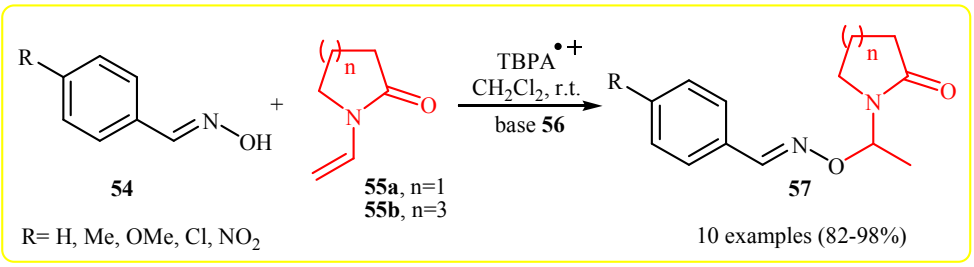

**Scheme 13.** Iridium-pybox-catalyzed allylic substitution with oximes

Oximes **50** underwent Baylis-Hillman reaction with allyl bromides **51** in the presence of sodium hydride and triethyl amine, and the regioselective products **52** were formed in good yields. The mechanism of the reaction involves the deprotonation of oxime by NaH to generate oxime anion **B** and subsequent reaction of **B** with intermediate **A** (derived from the allyl bromide **51** and $NEt_3$) *via* path **a** and path **b** to produce regioselective oxime ethers **52** and **53** (Scheme 14).[124]




**Scheme 14.** The proposed mechanism for the formation of **52** *via* Baylis-Hillman reaction of **50** with **51**.

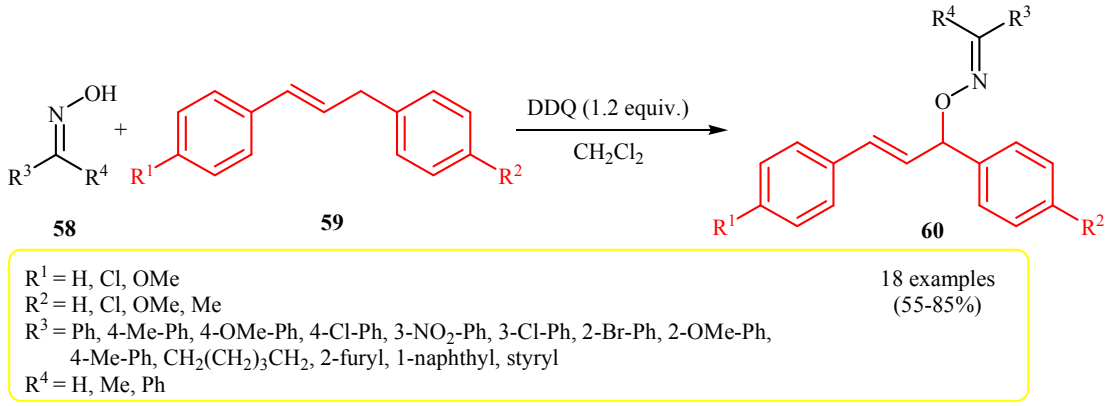
Jia and co-workers established an efficient protocol for radical cation promoted *O*-alkylation of oximes with *N*-vinyllactams. They showed treatment of oximes **54** with *N*-vinyllactam **55** in the presence of tris(4-bromophenyl)aminium hexachloroantimonate ($TBPA^{+\bullet}$ $SbCl6^{-}$) as an initiator and 2,6-di-tertbutyl-pyridine **56** as base afforded corresponding *O*-alkylated oxime ethers **57** in high to excellent yields at ambient temperature (Scheme 15). Generally, both electron-donating and electron withdrawing groups in the phenyl ring periphery of oximes were well tolerated.[125] The use of cerium (IV) ammonium nitrate (10 mol%) in acetonitrile provided the same products in comparable yields (73-95% for 15 examples).[126]




**Scheme 15.** $TBPA^{+\bullet}$ $SbCl6^{-}$ initiated addition of oxime **54** with N-vinyllactam **55**

Direct generation of oxime ethers from allylic C($sp^3$)-H bonds and oxime without a metal catalyst was reported by Bao and co-workers. They tested several oxidants and solvents, and the 2,3-dichloro-5,6-dicyanoquinone (DDQ)/$CH_2Cl_2$ system was found to be superior. Mechanistically, the reaction involves hydride transfer from the allylic position to DDQ. Good yields were achieved in reaction with both oximes involving electron-donating and

electron-withdrawing substituents (Scheme 16).[127] Following this work, the same group in 2013, extended their methodology to C-O bond formation between oximes and isochroman.[128]

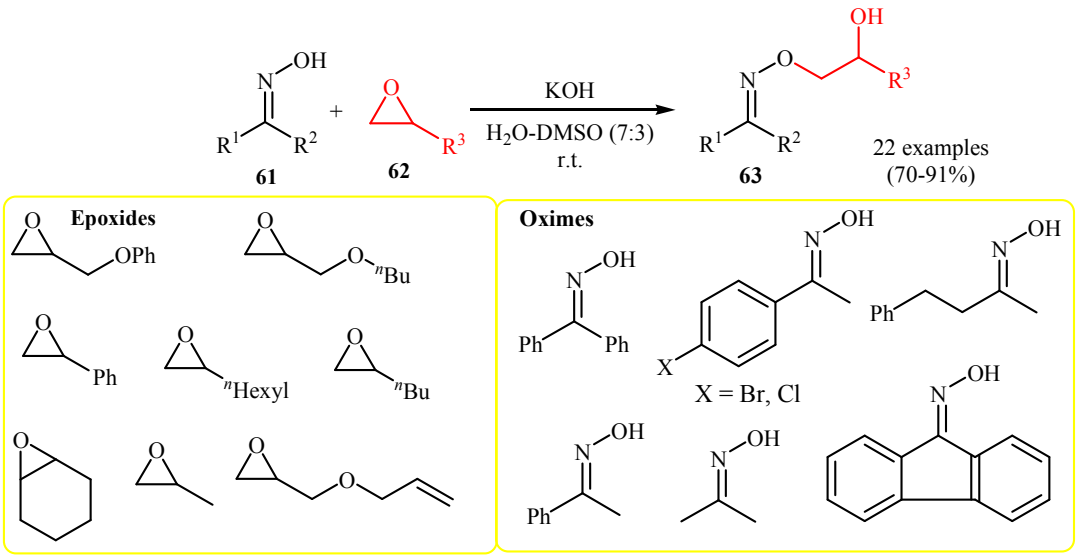



**Scheme 16.** Formation of oxime ether with oxime **58** and 1,3-diphenylpropene **59**

### 2.5. From oxime and epoxides

The nucleophilic substitution reaction of oxygen atom of oximes with epoxides for preparation of oxime ethers has been the subject of a number of papers. However, in primary reports a mixture of oxime ethers and nitrones have been examined for this reaction in various conditions, such as base and solvent types, etc.[129-133]

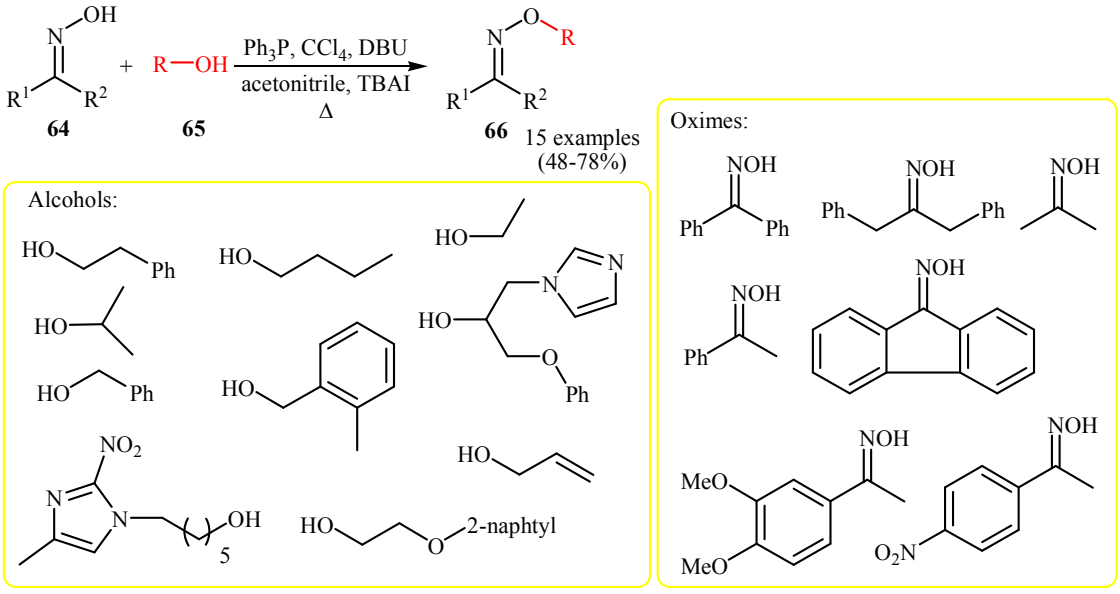
In 2008, Soltani reported a highly efficient regio- and diastereoselective synthetic methodology for preparation of β-hydroxy oxime *O*-ethers **63** *via* the *O*-alkylation of oxime anions **61** with epoxides **62**. This aqueous-mediated reaction carried out in the presence of KOH as base and gave corresponding *E*-oxime ethers **63** in good to high yields (Scheme 17). Interestingly, in contrast to previous methods, using this methodology gave no products derived from reaction of the nitrogen atom of oximes on epoxides, even in trace amounts.[134] Recently, Crich's research team performed the same reaction in DMF with a series of epoxides and acetophenone oximes.[135]




**Scheme 17.** β-hydroxy oxime *O*-ethers synthesized by the ring opening of epoxides with oximes.

## 2.6. From oximes and alcohols

One-pot *O*-alkylation of oximes with alcohols employing $Ph_3P/CCl_4$/DBU/TBAI catalyst system in refluxing acetonitrile was reported in 2010. A wide range of alcohols were efficiently transformed into oximes in good yields (Scheme 18). It is worth to note that the methodology showed excellent regioselectivity for generation of *Z*-isomers. The selectivity of this method was demonstrated *via* a competitive reaction of a mixture consisting of primary and secondary alcohols. The results showed high selectivity for the *O*-alkylation of oximes using the primary alcohols rather than the secondary analogues.[136]

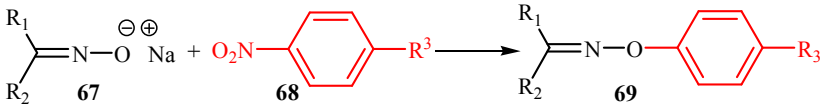


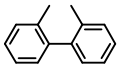
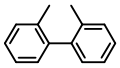
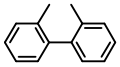
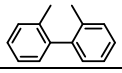

**Scheme 18.** One-Pot *O*-alkylation of oximes *via* alcohols in Refluxing Acetonitrile.

### 2.7. From oximes and aryl nitro compounds

Bauman demonstrated that oximes can be converted to oxime ethers by treatment with 4-substituted aryl nitro compounds **68** in sodium methoxide at room temperature. The reaction tolerates aryl oximes and gave corresponding *O*-aryl oximes in moderate yields, but extension of the reaction to aldoximes and alkyl ketoximes bearing a hydrogen at α-position was failed (Table 4).[137]

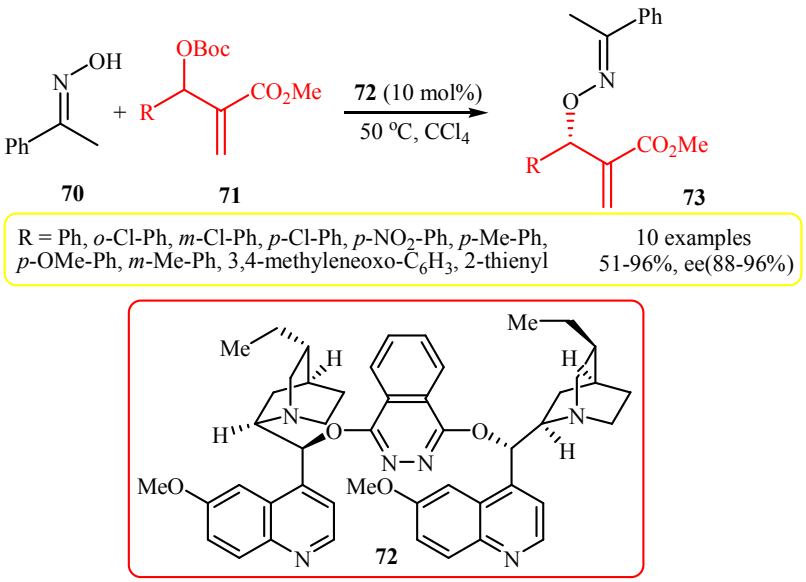
**Table 4.** Synthesis of oxime ethers **69** *via* treatment of oximes **67** with **68**.




| Entry | $R^1$ | $R^2$ | $R^3$ | Yield (%) | Entry | $R^1$ | $R^2$ | $R^3$ | Yield (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | -$(CH_2)_5$- | | $CO_2Me$ | 7 | 7 | Ph | Ph | PhCO | 66 |
| 2 | -$(CH_2)_5$- | | CN | 22 | 8 | Ph | Ph | CHO | 59 |
| 3 | Me | Me | $CO_2Me$ | 12 | 9 |  | | $CO_2Me$ | 31 |
| 4 | Me | Me | CN | 24 | 10 |  | | CN | 73 |
| 5 | Ph | Ph | $CO_2Me$ | 28 | 11 |  | | PhCO | 37 |
| 6 | Ph | Ph | CN | 61 | 12 |  | | CHO | 48 |

### 2.8. From oximes and Morita–Baylis–Hillman (MBH) carbonates

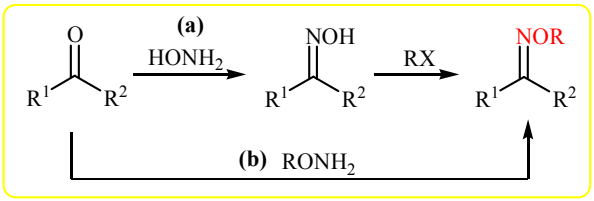
An excellent method for generation of oxime ethers is the reaction of oximes with Morita–Baylis–Hillman (MBH) carbonates. Chen and co-workers showed acetophenone oxime **70** and MBH carbonates **71** in the presence of commercially available hydroquinidine 1,4-phthalazinediyl diether **72** as a chiral catalyst at 50 $^oC$ gave *O*-allylic alkylated acetophenone oxime **73** in moderate to excellent yields with high enantiomeric excess (Scheme 19). However, the MBH carbonates bearing alkyl-substitution were incompatible in this type of reaction.[138]




**Scheme 19.** Asymmetric *O*-allylic alkylation of acetophenone oxime **70** with MBH carbonates **71**

## 2.9. From condensation of carbonyl compounds with aminooxy groups

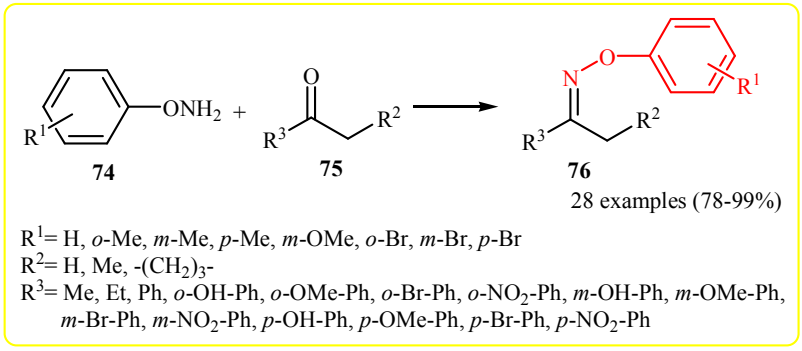
As it is mentioned in section 2.2., oximes are synthesizes from the reaction of carbonyl compounds (ketones or aldehydes) with hydroxyl amines, in a two-step reaction (Scheme 20, route a). The reaction of carbonyl compounds with aminooxy groups is a one-step route for the synthesis of titled compounds (Scheme 20, route b). [139,140] However the synthesis of aminooxy groups requires another step.[141-144]



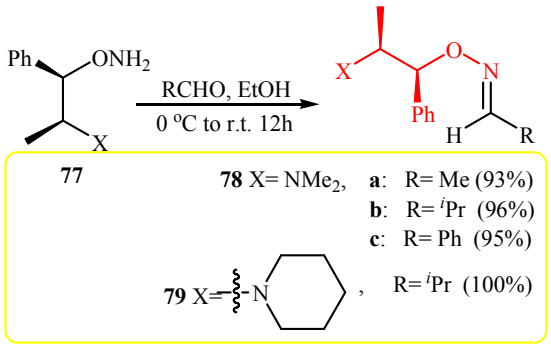

**Scheme 20.** General route for synthesis of oxime ethers

Synthesis of oxime ethers *via* condensation of carbonyl compounds with aminooxy groups are well described in the literature.[145-156] This reaction is usually conducted in various solvents and in the presence of an acid, which influence the yield of oxime ethers. Usual solvents which have been used are water,[145-147] methanol,[148,149] ethanol,[150] aqueous tetrahydrofuran,[146,147] chloroform[151] and common acids include hydrochloric acid,[147,148] acetic acid,[148,150] piperazine-*N,N′*-bis(2-ethanesulfonic acid),[152] pyridinium *para*-toluenesulfonate.[151]

The system MeOH/aqueous HCl gave excellent yield for the synthesis of *O*-aryl oximes at room temperature (Scheme 21).[148] The reaction of carbonyl compounds with benzyl hydroxyamines in absolute ethanol without catalyst gave superior result for preparation of *O*-benzyl oximes (Scheme 22).[153]




**Scheme 21.** Preparation of oxime ethers **7Aa–7He** in the presence of aqueous HCl as catalyst in MeOH




**Scheme 22.** Preparation of oxime ethers **78** and **79** in EtOH without catalyst

Indeed, the reaction rate is accelerated by using acid catalyst in aforementioned reactions. The acidic conditions are not compatible with biological systems and can damage biomolecules.[157] In 2008, to overcome this difficulty, Dirksen and Dawson introduced aniline as an efficient catalyst for condensation of carbonyl group with aminooxy group at neutral pH.[158] Crisalli and Kool[159] reported anthranilic acids and 3,5-diaminobenzoic acid (Figure 5) as superior catalysts for oxime ether formation under neutral pH conditions. Using the same catalysts, Palandoken and co-workers synthesized sugar oxime ether surfactant **85** in moderate to excellent yields (Scheme 23).[149]

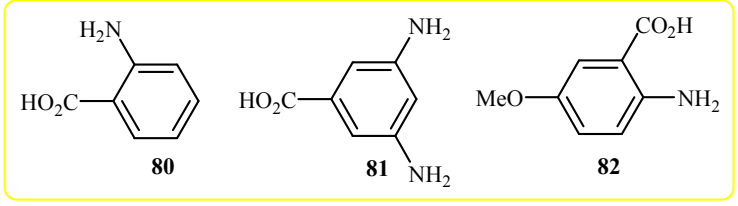
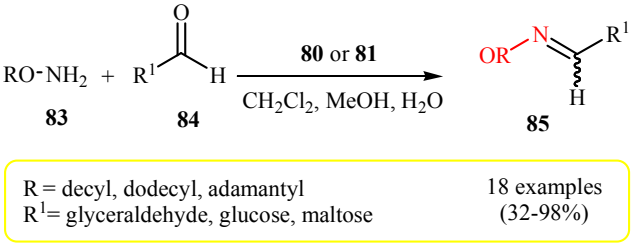


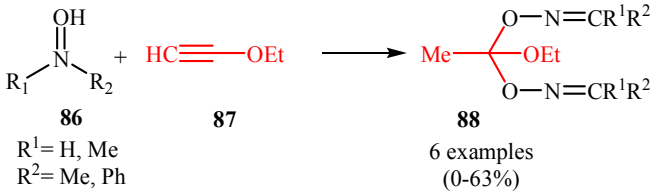

**Figure 5.** Chemical structure of anthranilic acids and 3,5-diaminobenzoic acid.




**Scheme 23.** Sugar oxime ether surfactant (SOESurf) synthesis.

## 2.10. Miscellaneous

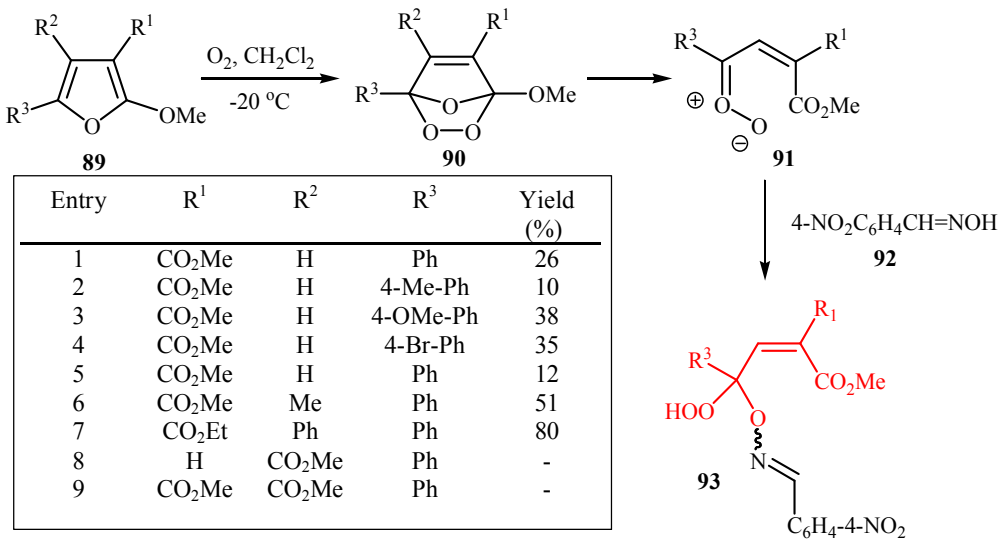
Reaction of oximes with acetylenes is a potential route for synthesis of novel oxime ethers. The example for this type of reaction have been reported by Tigchelaar-Lutjeboer *et al*. It is shown that, the reaction of oximes **86** with ethoxyethyne **87** at 75-90 $^{o}$C rise to the formation of *di*-oxime ethers **88** in moderate yields (Scheme 24). However the products are unstable compounds and in the case of aldoximes, corresponding *di*-oxime ethers were decomposed immediately.[160]




**Scheme 24.** Addition of oximes to ethoxyethyne

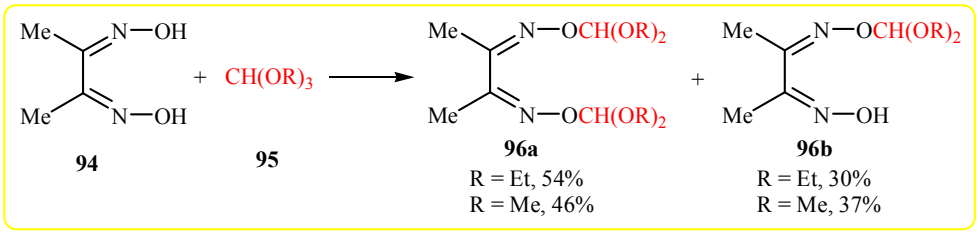
Reaction of oximes with cyclic peroxides is an efficent route for the synthesis of oxime ethers. 1-Methoxy-2,3,7-trioxa-bicyclo[2.2.1]hept-5-enes **90**, derived from photooxygenation of 2-methoxyfurans **89**, were converted into oxime ether hydroperoxides **93** by treatment of 4-nitrobenzaldehyde oxime **92**. The reaction proceed *via* the oxygen nucleophilic trapping reaction of intermediate carbonyl oxides **91**. However, in the most cases the products are unstable and rearrange into *N*-hydroperoxy alkylnitrones (Table 5).[161]

**Table 5.** Reaction of oximes with cyclic peroxides.




| Entry | $R^1$ | $R^2$ | $R^3$ | Yield (%) |
|---|---|---|---|---|
| 1 | $CO_2Me$ | H | Ph | 26 |
| 2 | $CO_2Me$ | H | 4-Me-Ph | 10 |
| 3 | $CO_2Me$ | H | 4-OMe-Ph | 38 |
| 4 | $CO_2Me$ | H | 4-Br-Ph | 35 |
| 5 | $CO_2Me$ | H | Ph | 12 |
| 6 | $CO_2Me$ | Me | Ph | 51 |
| 7 | $CO_2Et$ | Ph | Ph | 80 |
| 8 | H | $CO_2Me$ | Ph | - |
| 9 | $CO_2Me$ | $CO_2Me$ | Ph | - |

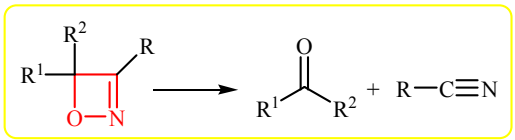
Treatment of methylglyoximes **94** with trialkyl orthoformate **95** resulted in the formation of a mixture of corresponding bis-*O*-alkylated oximes **96a** in moderate yields and mono-*O*-alkylated analogues **96b** in poor yields (Scheme 25).[162]




**Scheme 25.** Synthesis of *O*-dialkoxymethyloximes from oximes and trialkyl orthoformate

## 3. Synthesis of four membered cyclic oxime ethers (4H-1,2-oxazete)

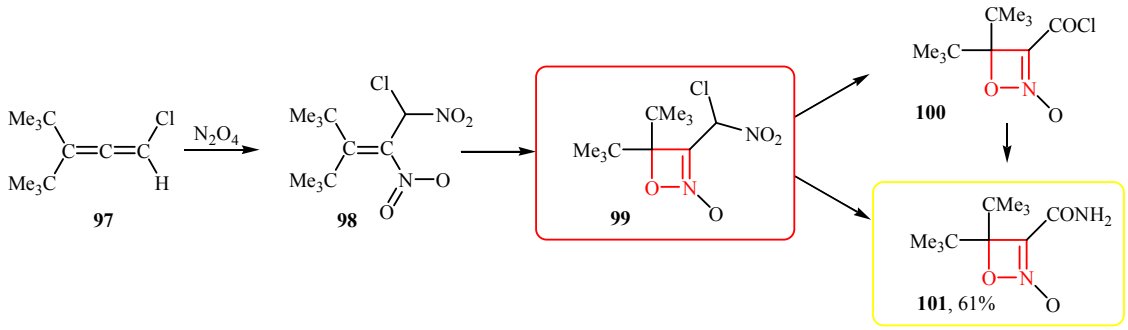
Generally, 4H-1,2-oxazetes and their highly strained derivatives are known as reactive intermediates in thermal and photochemical reactions. These compounds undergo facile fragmentation to carbonyl compounds and nitrile oxides (Scheme 26).[163-168]



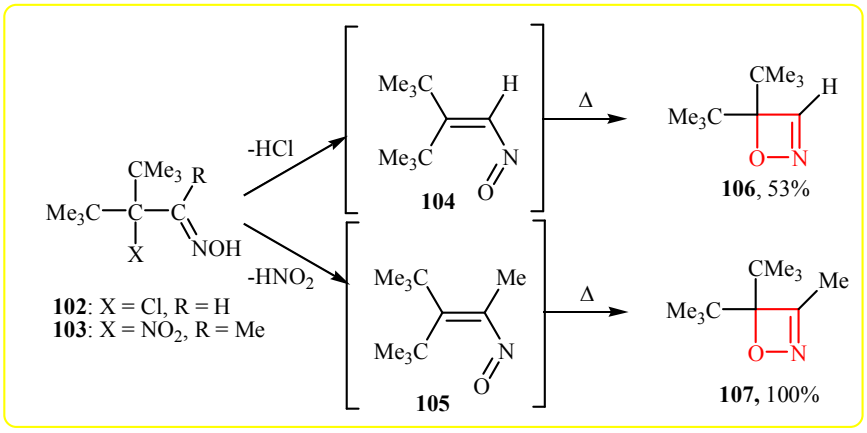
**Scheme 26.** Fragmentation of 4H-1,2-oxazetes to carbonyl compounds and nitrile oxides.

However, there are some reports for preparation of relatively stable derivatives of 4H-1,2-oxazetes. Wiese and Berndt reported two different route for generation of two family of stable 4H-1,2-oxazete derivatives[169,170]: 1) treatment of 3-*tert*-butyl-1-chloro-4,4-

dimethylpenta-1,2-diene **97** with $N_2O_4$ readily gave crystalline oxazete *N*-oxide **99**. This compound undergoes partial transformation to **100** and stable **101** (Scheme 27); 2) Elimination of H-X from oximes **102** and **103**, followed by intramolecular cyclization of unsaturated nitro intermediates **104** and **105**, gave corresponding stable oxazete **106** and **107** in good to excellent yields (Scheme 28).

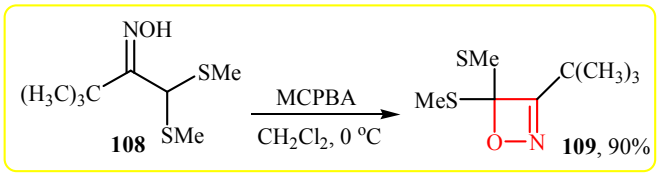



**Scheme 27.** Synthesis of stable oxazetes from 3-*t*-butyl-1-chloro-4,4-dimethylpenta-1,2-diene




**Scheme 28.** Synthesis of stable oxazetes from oximes.

Corkins and co-workers reported an efficient protocol for the synthesis of stable 3-*tert*-butyl-4,4-bis-(methylthio)-4H-1,2-oxazete **109** (Scheme 29). Addition of *m*-chloroperbenzoic acid to oxime **108** in $CH_2Cl_2$ leads directly to oxazete **109** after 16 hours at 0 °C in 90% yield.[171]



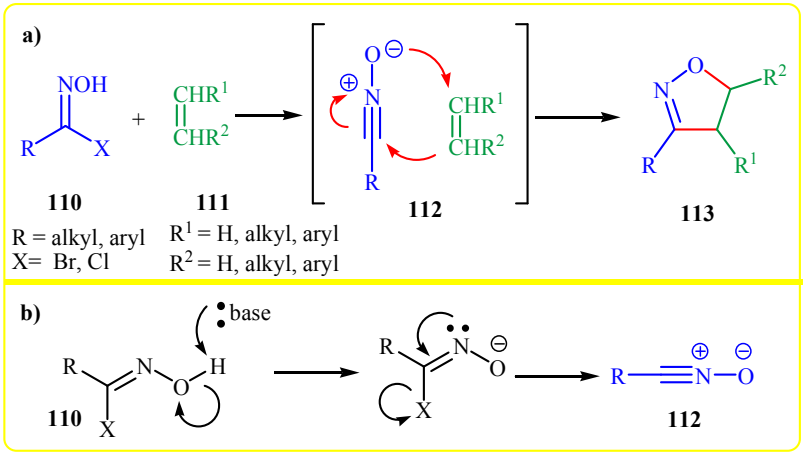

**Scheme 29.** Synthesis of stable oxazete **109** from oxime **108**.

## 4. Synthesis of five membered cyclic oxime ethers

### 4.1. 1,3-dipolar cycloaddition of nitrile oxides to cabon-carbon double bonds

The 1,3-dipolar cycloaddition of nitrile oxides to C=C bond is a fundamental tool and straightforward route to isoxazoline rings.[172-228] The same reaction with carbon-carbon triple bonds is one of the most efficient protocols for generation of isoxazoles.[229-232] Hydroxamic acid chlorides **110** are the commonly used precursors for generation of nitrile oxides **112**. Nitro compounds can also serve as convenient nitrile oxides precoursors.[211-220] However, nitro compounds are commonly used for the synthesis of isoxazoline *N*-oxides[233-246]. The conversion of **110** to **112** and 1,3-dipolar cycloaddition of **112** to alkenes **111** usually carried out in a one-pot reaction sequence (Scheme 30a). This reaction is conducted with various bases, such as $NaHCO_3$,[172,173] $KHCO_3$,[174] $AgNO_3$[173] and $NEt_3$.[174-180] The sequence in Scheme 30b shows how hydroxamic acid chlorides can be converted into nitrile oxides.

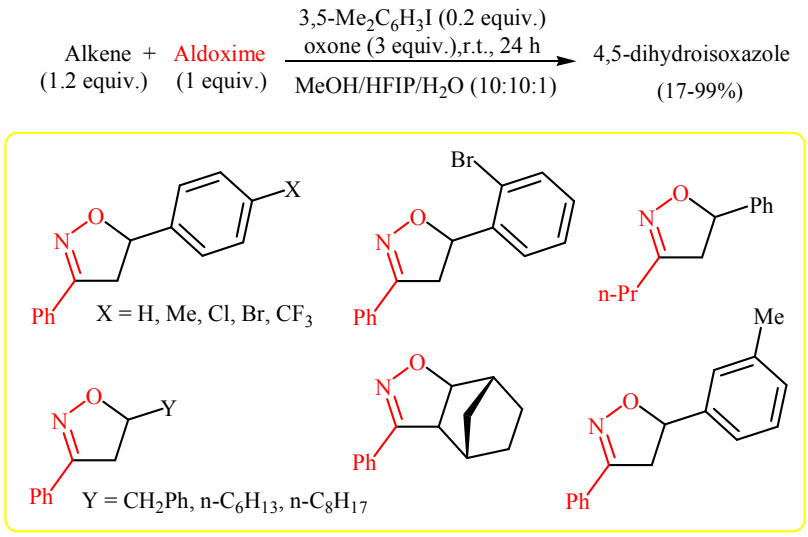
It is noteworthy that nitrile oxide can be generated *in situ* from the corresponding aldoxime by chlorination with bleach and then dehydrochlorination.[221-228]




**Scheme 30. a)** 1,3-dipolar cycloaddition of nitrile oxides to C=C bond; b) The mechanism of generation of nitrile oxides from hydroxamic acid chlorides.

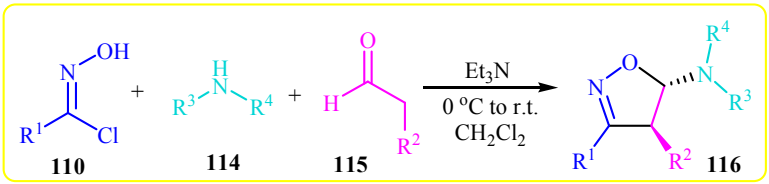
The impressive methods have been developed for direct use of aldoximes instead of hydroxamic acid chlorides as nitrile oxides precursor. In these methods, generation of nitrile oxides and then a 1,3-dipolar cycloaddition to alkenes performed in single step using hypervalent iodine as oxidants.[247-253] Recently, a more robust protocol for the synthesis of 4,5-dihydroisoxazoles was introduced by Yoshimura *et al*. They have exemplified the direct reaction of aldoximes with variety of alkenes in the presence of iodoarenes as catalyst and oxone as a terminal oxidant (Scheme 31). The similar reaction with alkynes gave the corresponding isoxazoles.[251] In 2014, Yan improved the efficiency of Yoshimura protocol by using potassium chloride/oxone as oxidation system in water.[252]

Following Yan, Bharate and co-workers have investigated the same reaction using DBU/NCS/DMF system and achieved better results.[253]




**Scheme 31.** Iodine catalyzed generation of nitrile oxides from oximes and their cycloaddition with alkenes.

Recently, Wang and co-workers reported a beautiful three component reaction for the synthesis of 4,5-dihydroisoxazole rings with secondary amine at C-5 position (Scheme 32). Most of the applied secondary amines failed to participate in the reaction, whereas, pyrrolidine was well tolerated. Mechanistically, the reaction involves: 1) The formation of nitrile oxide by hydroxamic acid chloride **110** and dialkylamine **114**; 2) condensation of aldehyde **115** with pyrrolidine to give enamine; 3) 1,3-dipolar cycloaddition of nitrile oxides to cabon-carbon double bonds of enamine to produce 4,5-dihydroisoxazole rings **116** containing dialkylamino moiety at C-5 position in good to excellent yields (77-99% for 18 examples).[254]

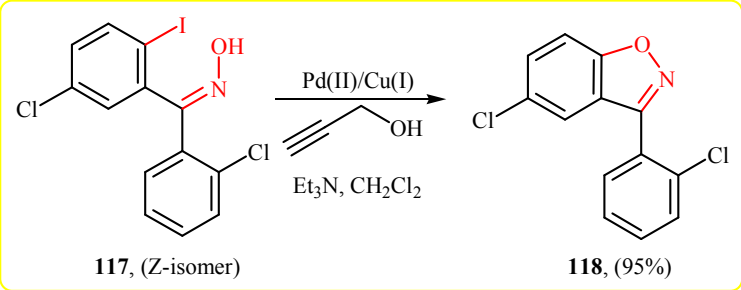



**Scheme 32.** Tri-component reaction for the synthesis of 4,5-dihydroisoxazole rings.

### 4.2. Intramolecular metal catalyzed cross-coupling reactions

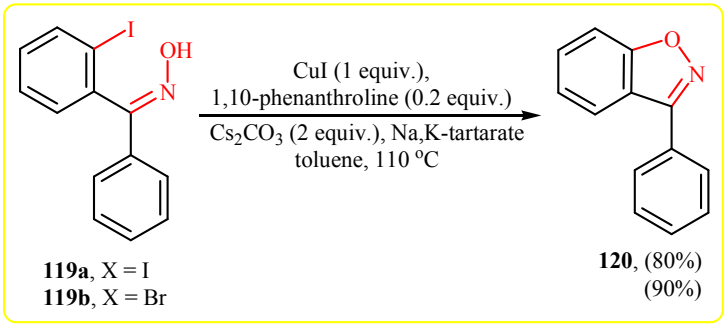
Metal catalyzed cross-coupling reaction is a straightforward route for formation of =N-O-R linkage *via* reaction of oximes (=N-OH) with an electrophilic partner (X-R). The

intramolecular version of this reaction is a highly effective protocol for generation of isoxazolines. The first example, was reported by Coffen and co-workers in 1984.[255] Coupling of iodooxime **117** with propargyl alcohol using $Pd(PPh_3)_2Cl_2/CuI/Et_3N/CH_2Cl_2$ system, resulted in isoxazoline **118** in 95% yield (Scheme 33).




**Scheme 33.** Synthesis of isoxazoline **118** from iodo oxime **117** via metal-catalyzed cross-coupling reaction.

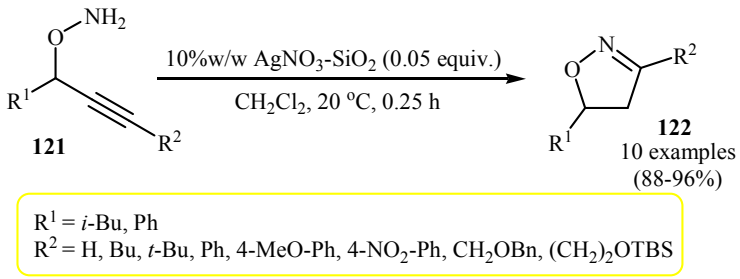
Subsequently Wailes reported the same cyclization using $CuI/Et_3N/CH_2Cl_2$ system and obtained isoxazoline **120** in 80% yield. Two other examples utilizing this protocol is depicted in Scheme 34.[107]




**Scheme 34.** Intramolecular cross-coupling of ketoximes **119a**,**b** with catalytic CuI in refluxing toluene.

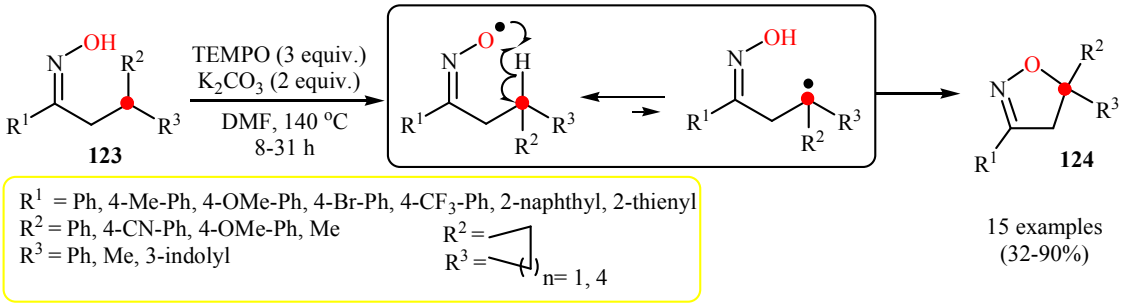
### 4.3. Miscellaneous

In 2010, Knight and co-workers were able to take advantage of a new route for the synthesis of isoxazolines in their efforts to develop novel cyclization of *O*-propargylic hydroxylamines. It was shown that hydroxylamines **121** in treatment with 10% w/w silver nitrate/silica gel as a catalyst in $CH_2Cl_2$ at 20 °C underwent intramolecular hydroamination to give corresponding isoxazolines **122** in good to excellent yields (Scheme 35).[256]




**Scheme 35.** Synthesis of isoxazolines **122** from hydroxylamines **121**.

Regioselective intramolecular carbon-hydrogen bond oxygenation at β-position of oxime moiety with activation of hydroxyl group is the newest route for generation of isoxazolines which have been introduced by Chiba *et al.* in 2013. The cyclization has been conducted using 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) in DMF and gave desired products in moderate to high yields (Scheme 36). Mechanistically, it involves: 1) the reaction of oxime moiety with TEMPO to give iminoxyl radical; and 2) 1,5-H radical shift of iminoxyl radical result in the formation of corresponding isoxazoline.[257]




**Scheme 36.** TEMPO-mediated C-H oxygenation of oximes **123**.

## 5. Conclusion

This review provides concise overview on the synthesis of acyclic and cyclic oxime ethers. The new strategies in this area such as synthesis of oxime ethers *via* metal-catalyzed cross-coupling reactions and intramolecular carbon-hydrogen bond oxygenation has further potential for development. We believed that the highly versatile and novel procedures for the synthesis of oxime ethers will be attainable in the near future.

## References

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# Review on synthesis of acyclic and cyclic oxime ethers

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