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Multicomponent reactions (MCRs) of acetylenic compounds with isocyanides or triphenylphosphine–A Mini review

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- ✓ *Alkyl propiolates*

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Abstract

Acetylenic esters such as dimethylacetylenedicarboxylat (DMAD), diethyl acetylenedicarboxylat (DEAD) and alkyl propiolates are esters in which the ester groups are conjugated with a C-C triple bond. As such, these molecules are highly electrophilic, and widely employed as a dienophile in cycloaddition reactions. They have been used in muliticomponent reactions in the presence of nucleophilic compounds such as Isocyanides, Isoquinoline, Quinoline, Triphenylphosphine, trialkyl (aryl) phosphites and NH-acidic or OH-acidic compounds. This review mainly focuses on the research work reported in the scientific literature on the reaction this compounds with Isocyanides and Triphenylphosphine.

1. Introduction

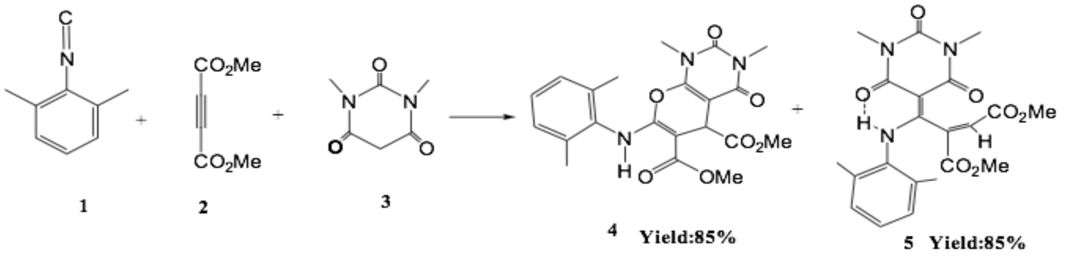
Acetylenic esters such as dimethyl acetylenedicarboxylat (DMAD), diethyl acetylenedicarboxylat (DEAD) and alkyl propiolates are esters in which the ester groups are conjugated with a C-C triple bond. These kind of molecules are highly electrophilic, and are mainly used as a dienophile in cycloaddition reactions [1].

The other uses of these compounds are the multi-component reactions (MCRs), that they are consist of a single synthetic operation.

MCRs are widely used in organic synthesis and chemical and pharmaceutical chemistry [2-12]. Because organic material is consumed and incorporated into the purpose of the combination, it is usually easy to clean products from MCRs. Maximum returns, reduced waste and eco-friendly products are the benefits of multi-component reactions. Therefore, start significant MCRs to prepare complex molecules of available materials. There are many ongoing efforts to develop new MCRs and many new 3- and 4-component reactions [13-14]. Acetylenic esters are reactive systems taking part in many chemical synthesis, for example, as Michael acceptors [15-18]. In recent years, there has been increasing interest in the application of acetylenic esters for multi-component synthesis [19-23]. Usually in addition to nucleophiles lacking an acidic hydrogen atom, it leads to a zwitterionic 1: 1 mediator [24-29]. It can finally lead to a stabilized product by further transformation [30-33]. It has been convincingly argued the formation of zwitterions from pyridine, amines, triphenylphosphine, dimethyl sulfoxide, isocyanides and phosphoranes. Triphenylphosphine and Isocyanide based on MCRs are very important in this regard [34-39].

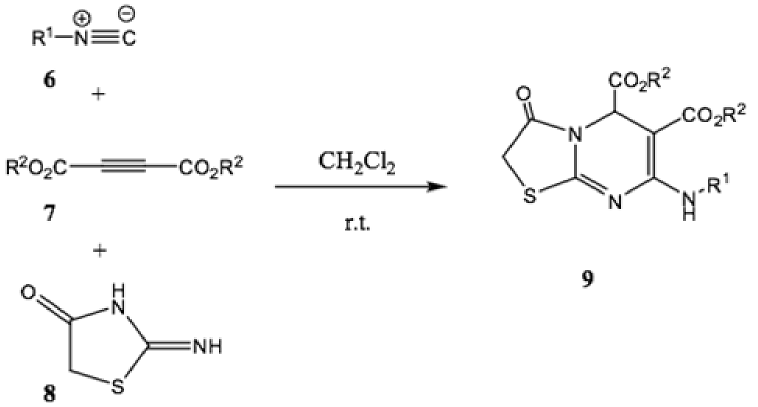
2. Reactions with Isocyanids

It has reported, that the reaction of alkyl isocyanides with dimethyl acetylenedicarboxylat (DMAD) and N, N' dimethylbarbituric acid can lead to the formation of 4H-pyrano [3, 2-d] pyrimidine derivatives. The isomeric products dimethyl 7-(2,6-dimethylphenylamino)-1,3-dimethyl-2,4-dioxo-4H-pyrano[3,2-d]pyrimidine-5,6-dicarboxylate **4** and dimethyl (E)-2-((2,6-dimethylphenylamino)-(1,3-dimethyl-2,4,6-trioxopyrimidine- 5-ylidene)-methyl)-but-2-enedioate **5** in a nearly 1:1 ratio can be obtained from The reaction of 2,6-dimethylphenyl isocyanide **1** with DMAD **2** in the presence of N,N' dimethylbarbituric acid **3** (Scheme 1) [40].



Scheme 1

We've had a lot of work in the field of multi-component reactions [41-44]. Continue this, we have recently described the synthesis of highly substituted 3-oxo-2, 3-dihydro-5H-thiazolo [3, 2 a] pyrimidines **9** via the one-pot, three-component reaction of an isocyanide **6** with a dialkyl acetylenedicarboxylate (DAAD) **7** and 2-imino-1, 3 thiazolidin-4-one **8** in good yields (Scheme 2) [45].



Product	R^1	R^2	Yielda (%)
9a	Cyclohexyl	Me	87
9b	Cyclohexyl	Et	70
9c	Cyclohexyl	i-Pr	75
9d	t-Bu	Me	85
9e	t-Bu	Et	78
9f	t-Bu	i-Pr	83
9g	1,1,3,3-Tetramethylbutyl	Me	85
9h	1,1,3,3-Tetramethylbutyl	Et	75
9i	1,1,3,3-Tetramethylbutyl	i-Pr	72

Scheme 2

A tentative mechanism for this transformation is proposed (figure 1):

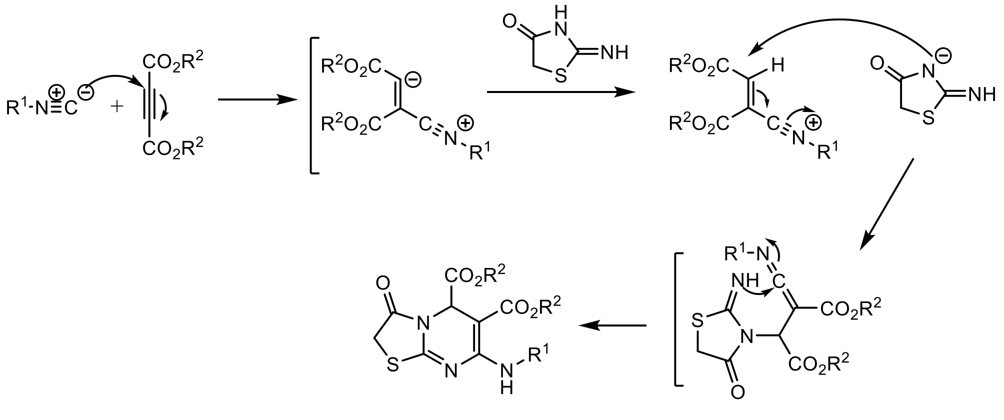
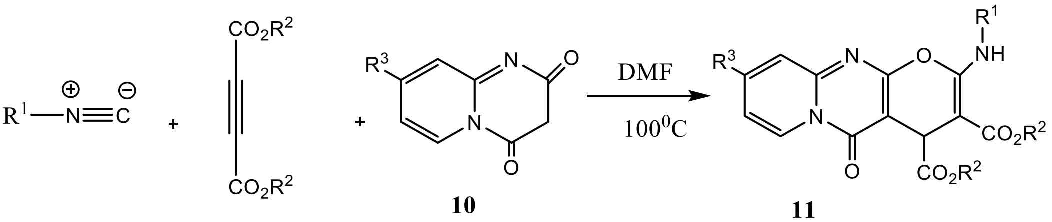


Figure 1

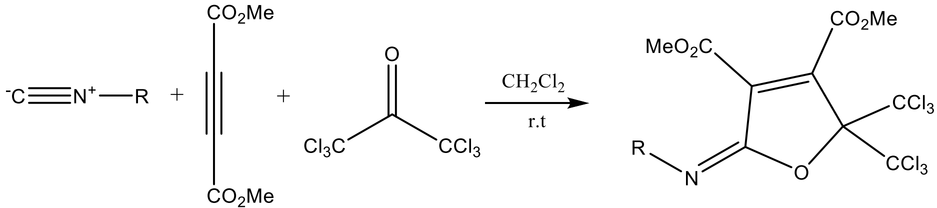
The synthesis of novel tricyclic fused pyrano[2,3-d]pyrido[1,2-a]pyrimidines **11** containing both biologically active pyridopyrimidine and pyranopyrimidine scaffolds, via the reaction of 2-Hpyrido[1,2-a]pyrimidine-2,4(3H)-dione **10**, isocyanides, and dialkylacetylenedicarboxylates are reported (Scheme 3) [46].



10 **11**

Scheme 3

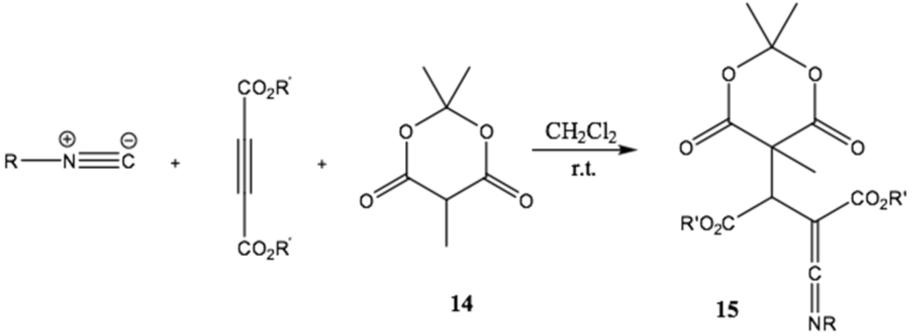
Resulted zwitterionic intermediate formed from reaction of isoncyanides and dimethyl acetylenedicarboxylate (DMAD), because of significant reactivity of isoncyanide toward DMAD, can undergo next reactions with different class of electrophilic reagent. This fact is a way to synthesis of more complex dompounds. These reactions have been studied by many number of research groups [47-49]. As an example for application of this class of reaction, a simple synthesis of trichloromethylated iminofuranes explained [50-52]. Trichloromethylated iminofuranes is produced by reaction of isocyanide and DMAD in the presence of hexachoroacetone (HCA) **12** in dry dichloromethane as solvent. In this reaction dimethyl 5-(tert-butylimino) -2, 2-bis (trichloromethyl)-2,5-dihydrofuran- 3,4-dicarboxylate **13** produced in 95% isolated yield (Scheme 4) [53].



12 **13**

Scheme 4

In spite of extensive developments in the chemistry of modified ketenes and isocyanates [54-55], little attention has been paid to the synthesis of ketenimines. A simple one-pot preparation of stable ketenimines was reported as a proton source nucleophile. In the preparation alkyl isocyanidesand dialkyl acetylenedicarboxylat is usedin the presence of 2,2,5-trimethyl-1,3-dioxane-4,6-dione (methyl Meldrum's acid) **14**. This three-component condensation reaction produces stable ketenimines **15** in fairly good yields (Scheme 5) [56].



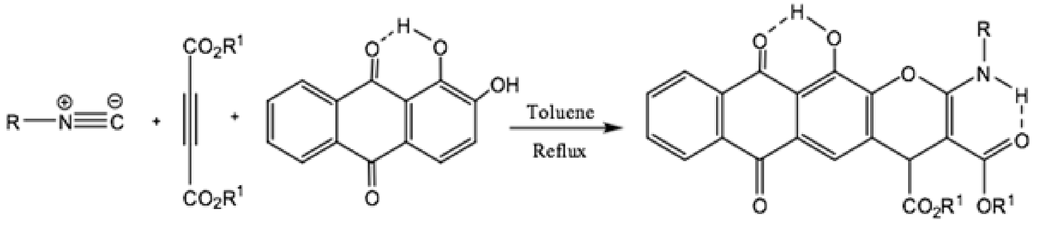
14 **15**

Scheme 5

In continuation of current interest in the development of new routes to heterocyclic and carbocyclic systems [57-61], a simple one-pot synthesis of functionalized alizarin derivatives was reported. Thus, the reactions of isocyanides with dialkyl acetylenedicarboxylates in the presence of alizarin **16** produces new functionalized dihydro-4H-naphtho [2, 3-g] - chromenes **17** in good yields (Scheme 6) [62].

3. Reactions with Triphenylphosphine

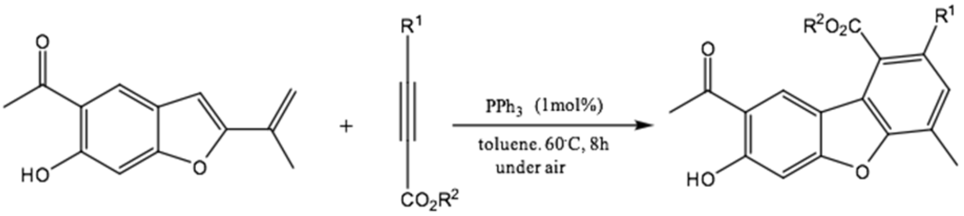
Aligned with the use of acetylene esters for the synthesis of new compounds [63-67], herein, we report a simple and efficient one-step procedure for the synthesis of novel dibenzofuran derivatives. In this regard, we use the Diels–Alder reaction of euparin, a natural product isolated from the roots of Petasites hybridus, and different activated acetylenes in the presence of catalytic PPh_3 (Scheme 7). The reaction between euparin **18**, a natural product isolated from the roots of Petasites hybridus, and dimethyl dialkyl acetylenedicarboxylate or alkyl propiolate **19** in the presence of PPh_3 affords the corresponding dibenzofuran **20** derivatives in high to excellent yields (Scheme 7) [68].



16 **17**

Entry	R	R^1	Product	Yield/%
1	Cyclohexyl	Me	17a	90
2	Cyclohexyl	Et	17b	88
3	2,5-Dimethylphenyl	Me	17c	85
4	2,5-Dimethylphenyl	Et	17d	80
5	1,1,3,3-Tetramethylbutyl	Me	17e	90
6	1,1,3,3-Tetramethylbutyl	Et	17f	87

Scheme 6



18 **19** **20**

Scheme 7

A proposed mechanism for this evolution is shown (figure 2):

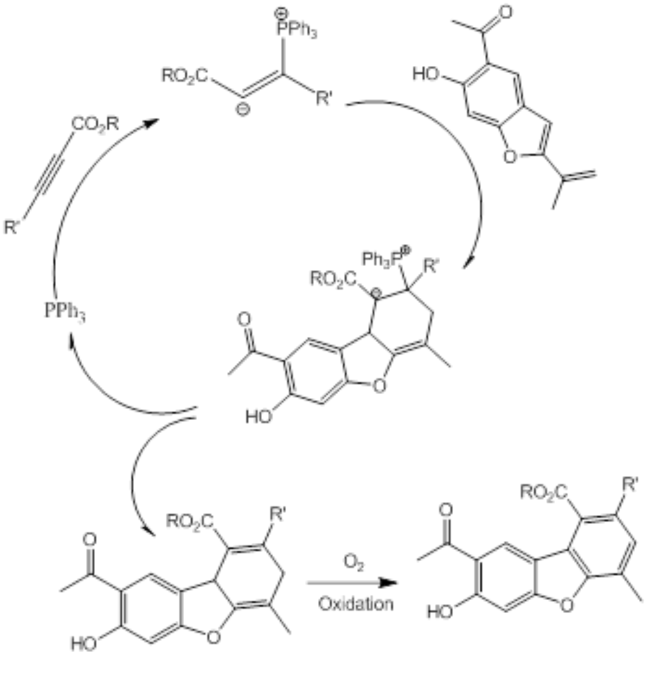
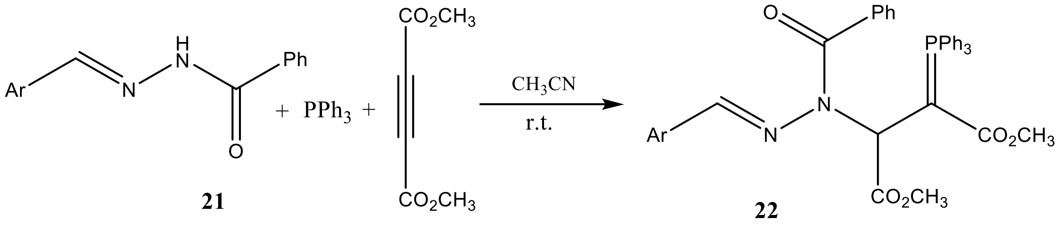


Figure 2

For preparation of stable phosphorus ylides, it has been reported a new method on the base of reaction between PPh_3, electron-deficient acetylenic compounds (for example dimethyl acetylenedicarboxylate) and different nucleophiles (N-H, O-H, or C-H acids like benzoylhydrazones) [69-72]. For example the corresponding ylide **22** was prepared via the reaction of benzoylhydrazones **21** with dimethyl acetylenedicarboxylate with triphenylphosphine (Scheme 8) [73].

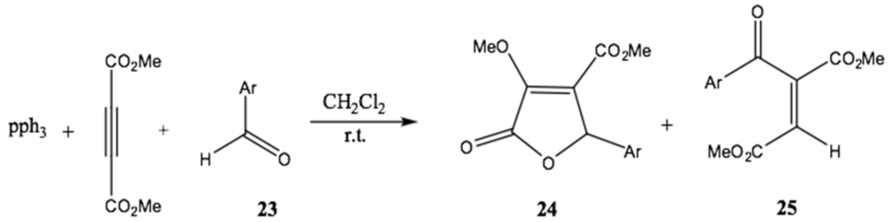


21 **22**

Scheme 8

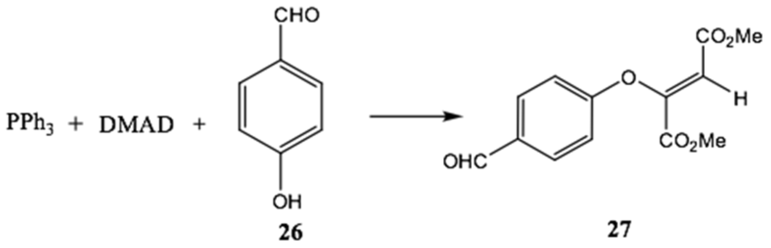
For developing of MCRs by use of zwitterion species, the reaction probability of 1, 3- dipoles with aryl aldehydes were studied. In this case, instead of expected products unsaturated γ-butyrolactones and highly substituted enones with PPh_3 created. In these reactions, the role of PPh_3 is a mediator [74-75]. Reaction between aryl aldehyde **23**, dimethyl acetylenedicarboxylate in the presence of Ph_3P is done as well at room temperature in dichloromethane as solvent at 24h. The presence of two products (unsaturated γ-butyrolactone **24** and highly substituted enone **25**) is indicated according to ^{1}H-NMR and ^{13}C- NMR (Scheme 9).

Product **27** also is resulted of reaction between DMAD and p-hydroxybenzaldehyde **26** in the presence of PPh_3 in moderate yield (Scheme 10) [76].



23 **24** **25**

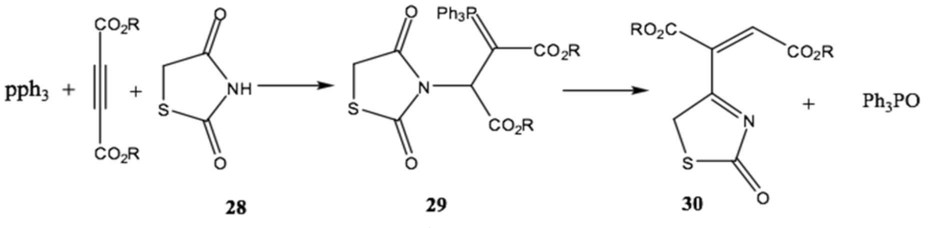
Scheme 9



26 **27**

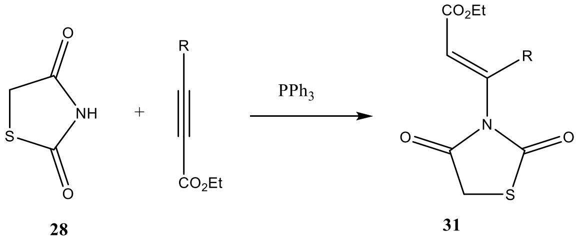
Scheme 10

As part of current studies on synthesis of sulfurcontaining organic compounds [77-79], an efficient procedure for chemoselective synthesis of functionalized thiazol-2(5H)-ones from reaction between thiazolidine-2, 4-diones and acetylenic esters in the presence of triphenylphosphine (Ph_3P) was reported. The reaction of dialkyl acetylenedicarboxylates with thiazolidine-2, 4-dione **28** in the presence of Ph_3P at r.t. in ethyl acetate was completed within a few hours, and phosphoranes **29** were produced in good yields. The ^{1}H-NMR spectra of the reaction mixtures clearly showed the formation of the phosphorane **29**, and no other productscould be detected. These ylides were subjected to intramolecularWittig reaction in boiling toluene, without further purification, to produce dialkyl 2-(2, 5-dihydro-2- oxothiazol-4-yl) fumigates **30** in good yields (Scheme 11) [80-81]. The reaction of thiazolidine-2,4-dione **28** with alkyl propiolates in the presence of Ph_3P proceeded spontaneously at r.t. in toluene and was finished within 1-4 h. 1H and ^{13}C-NMR spectra of the reaction mixtures clearly indicated the presence of product **31** (Scheme 12) [82].



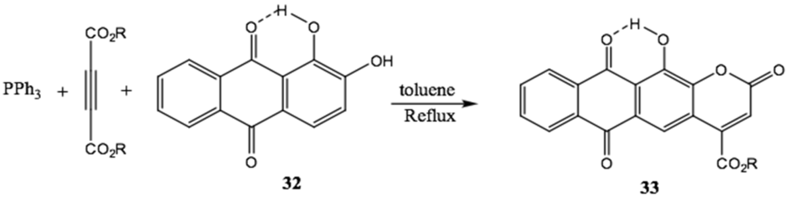
28 **29** **30**

Scheme 11

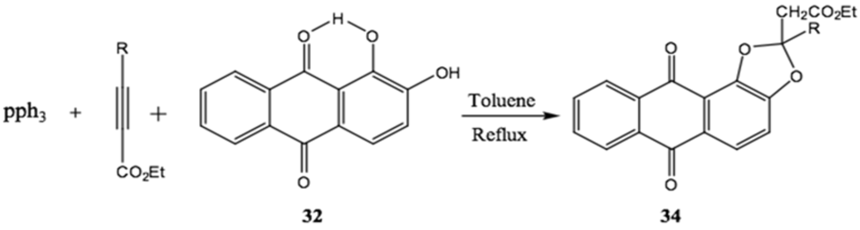


Scheme 12

In continuation of current interest in the development of new routes to heterocyclic and carbocyclic systems [83-85], a simple one-pot synthesis of functionalized alizarin derivatives was reported. Thus, the reaction of alizarin **32** with dialkyl acetylenedicarboxylates in the presence of Ph$_3$P produced functionalized dihydro-2H-naphtho [2, 3-g] - chromenes **33** in good yields (Scheme 12). When alkyl propiolates were employed as the acetylenic component, the reaction afforded **34** (Scheme 13 and 14) [86].

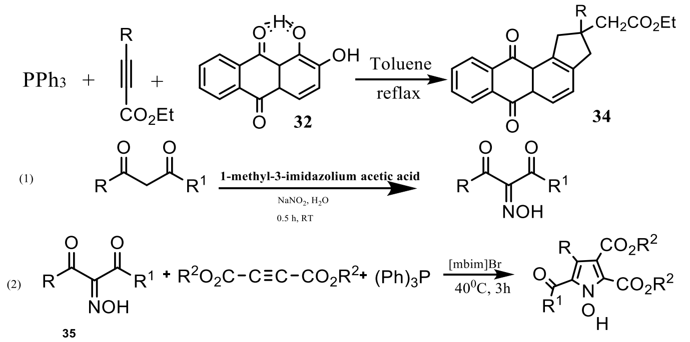


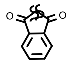
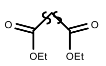
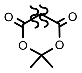
Scheme 13

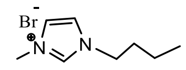


Scheme 14

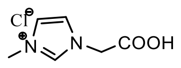
In continuation of our studies in using according to green chemistryfor the synthesis of organic compounds, we report the condensation reaction between α-oximinoketone **35** and dialkyl acetylene-dicarboxylate in the presence of triphenylphosphine to afford substituted pyrroles **36** under ionic liquid conditions in good yields. The advantages of the present procedure are decrease reaction time, high yield products, green and recyclable solvent for the synthesis of oximes and pyrroles. (Scheme 15) [87].



	R	R^1	R^2	yield%
a	Me	Ph	Me	94
b	Me	Ph	Et	92
c	Ph	Ph	Me	92
d	Ph	Ph	Et	90
e	Me	OEt	Me	92
f	Me	OEt	Et	90
g^a	Me	Me	Me	95
h	Me	Me	Et	87
i			Me	86
j			Et	87
k			Me and Et	No reaction
m			Me and Et	No reaction



1-methyl-3-buthylimidazolium bromide [mbim]Br

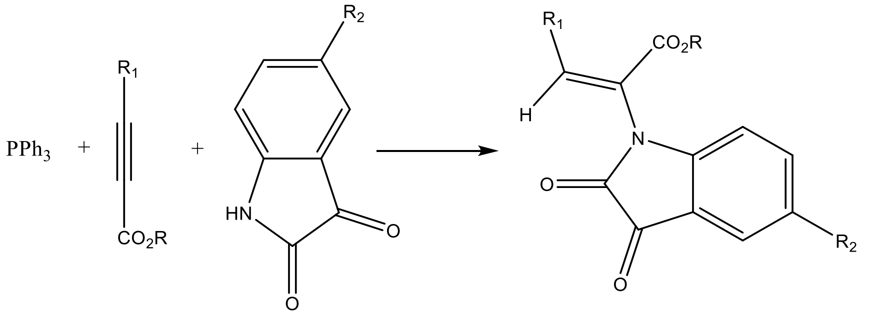


1-methyl-3-imidazolium acetic acid

[a] 5g, yiled 95% in [bmim]Br for the first time, 89% in the second run, and 88% in the third run with recycled il.

Scheme 15

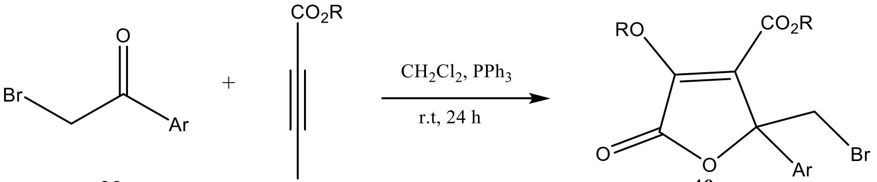
In 2014 Sheikhi and et al reported products **38**through the standard Michael addition of the triphenylphosphineto the b-carbon of the electron-deficient alkyne (alkyle propiolate) andisatin derivatives **37** leads to products **38** under the correct reaction condition in satisfactory yields (Scheme 16) [88].



R: Me, Et R_1:H, Ph R_2: H, F

Scheme 16

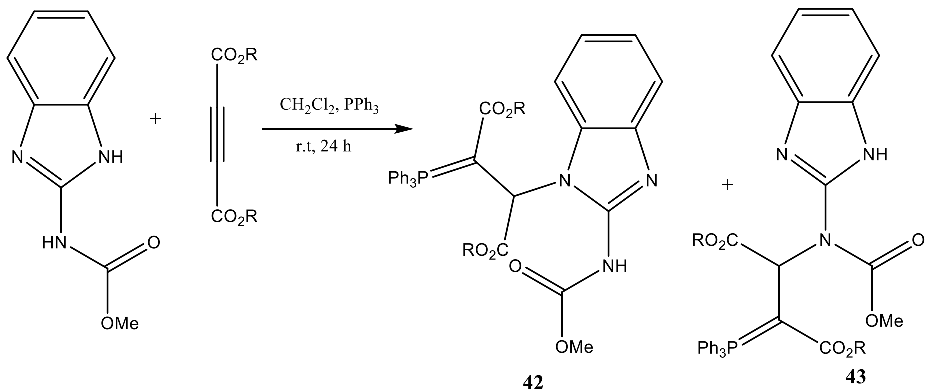
M. Anary-Abbasinejad and et al in 2012 reported One-pot, three-component reaction of different phenacyl bromides **39** and dialkylacetylenedicarboxilates was carried out with triphenylphosphine and functionalized oxodihydrofuran derivatives **40** in excellent yields. Nucleophilic addition of triphenylphosphine to activated acetylene is well known to produce a reactive zwitterionin intermediate, [89-91] which may be trapped by acidic organic compounds such an alcohols. Reaction between dimethyl acetylenedicarboxylate and triphenylphosphine in the presence of alcohols has been reported to produce phosphorus ylide as an intermediate or final product. When a-hydroxy carbonyl compounds were used, the intramolecular Wittig reaction of the ylide intermediate afforded oxygen heterocycles.[92-93] Similar reactions have been developed for the synthesis of a variety of carbocycles and heterocycles, using N-H, [94] O-H, S-H [95] and C-H [96] acidic compounds to trap the zwitterionic DMAD-PPh3 intermediate in condition of our precision studies on the reaction of PPh_3-DMAD zwitterions with organic acidic compounds [97]. Here was reported that the reaction of different phenacyl bromides, dialkyl acetylenedicarboxylates, and triphenylphosphine afforded oxodihydrofuran derivatives in good yields. Treatment of dimethyl acetylenedicarboxylate (DMAD) with triphenylphosphine and 4-chlorophenacyl bromide in dichloromethane at room temperature after 24 h afforded methyl 2-(bromomethyl)2-(4-chlorophenyl)4-methoxy-5-oxo-2,5-dihydrofuran-3-carboxylate **40** in 88% yield (Scheme 17) [98].



39 **40**

Scheme 17

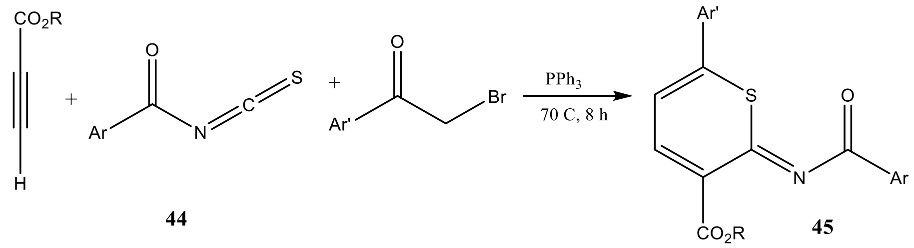
Mohebati and et al in 2016 reported the three-component and chemoselective reaction between dialkylacetylenedicarboxylates and triphenylphosphine in the presence of carbendazim **41** produces highly functionalized, phosphorus ylides **42**and **43** in excellent yields (Scheme 18) [99].



41 **42** **43**

Scheme 18

Shikholeslami-Farahani and et al in 2014 reported the synthesis of thiopyran derivatives **45** via three-componentreaction of alkyl propiolate, benzoylisothiocyanate **44** and a-haloketones in the presence of triphenylphosphine under solvent-free conditions at 70 ◦C (Scheme 19) [100].

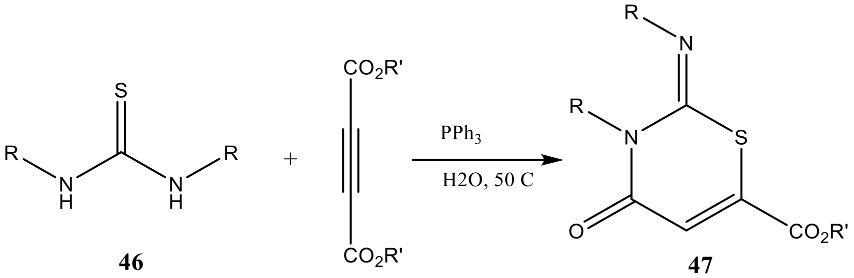


44

45

Scheme 19

Yavari and et al in 2010 reportedA one-pot synthesis of alkyl 3, 4-dihydro-4- Oxo-2H-1, 3-thiazine-6-carboxylates **47** from dialkyl acetylenedicarboxylates and N,N0-dialkylthioureas **46** in the presence of triphenylphosphine (Scheme 19) [101].



46

47

Scheme 20

4. Conclusion

Acetylenic esters such as dimethyl acetylenedicarboxylat (DMAD), diethyl acetylenedicarboxylat (DEAD) and alkyl propiolates are esters in which the ester groups are conjugated with a C-C triple bond. As such, these molecules are highly electrophilic, and widely employed as a dienophile in cycloaddition reactions. They have been used in mulliticomponent reactions in the presence of nucleophilic compounds such as Isocyanides, Triphenylphosphine, trialkyl (aryl) phosphites. That can lead to heterocyclic products or open chain products in good yield.

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