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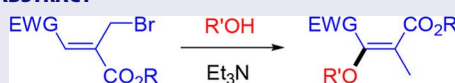
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ABSTRACT

An efficient coupling reaction of allyl bromides with various alcohols as nucleophiles activated by triethylamine, leading to functionalized vinyl ethers in good yields and with full stereoselectivity.

GRAPHICAL ABSTRACT



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KEYWORDS

Allyl bromides; oxygen nucleophiles; triethylamine; vinyl ethers

Introduction


The search for efficient synthetic methods to build C–O bonds is of great interest in organic chemistry, and vinyl ethers have attracted considerable attention for their potential applications: monomers for the synthesis of functional polymer materials,^[1] precursors of active pharmaceutical ingredients,^[2] and synthons for the formation of hemilabile mono-^[3] and diphosphine ligands.^[4] Moreover, such enol ethers can efficiently contribute in various reactions, such as cyclization,^[5] metathesis,^[6] Claisen rearrangement,^[7] addition of diverse X–H acids^[8] Heck reactions,^[9] and arylation.^[10] Among the existing methods to access vinyl ethers, the most recent progresses propose metal-catalyzed cross-coupling between vinyl(pseudo) halides and alcohols as a formal S_N2 reaction.^[11–13] Herein we report the S_N2' reaction between alcohols and an allyl bromide **1** activated by Et₃N affording enol ethers (Scheme 1).

Results and discussion

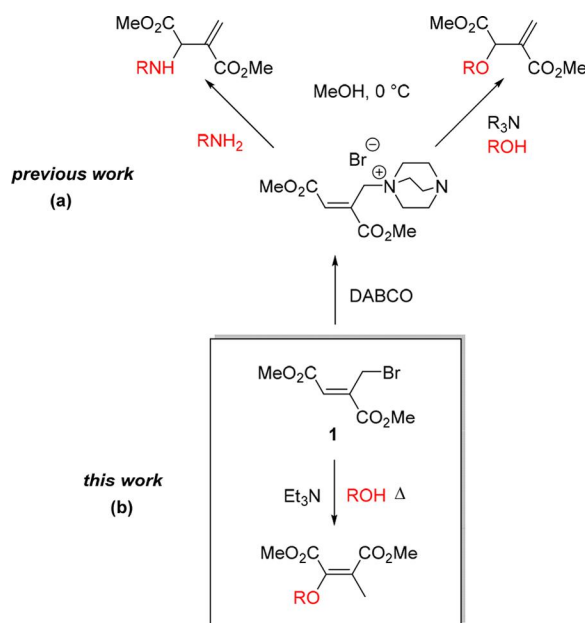
Dimethyl α-(bromomethyl)fumarate **1** has been used in the synthesis of alkenyl itaconic acids^[14] and organocatalytic α-alkylation of aldehydes.^[15] In this context, we have

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 Supplemental data (synthetic procedure for vinyl ethers **2**, vinyl ethers **4**, dimethyl 2-(bromomethyl)-3-phenoxy maleate **5**, methyl 1-benzyl-4-(benzylamino)-2,5-dihydro-5-oxo-1*H*-pyrrole-3-carboxylate **6** and copies of NMR spectra of compounds **2**, **4**, **5**, and **6**) can be accessed on the [publisher's website](#).

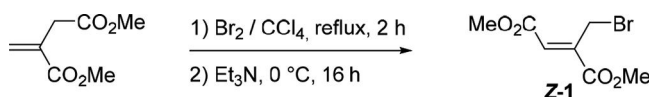
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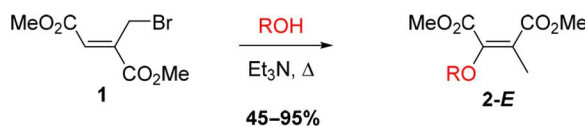
Scheme 1. Background (a) and the objective (b) of the current work.

previously described a simple and stereoselective synthesis of allyl bromide **1**,^[16] a trifunctional alkene from the addition of bromine to dimethyl itaconate, followed by dehydrobromination using triethylamine (Scheme 2).

Recently, we have reported a general methodology for the selective synthesis of allyl ethers^[17] from the use of 1,4-diazabicyclo[2.2.2]octane^[18,19] in the direct nucleophilic substitution of the brominated derivative **1**.^[16] Alcoholysis of the resulting quaternary ammonium salt produce the functionalized allyl ethers in the presence of triethylamine in $\text{S}_{\text{N}}2'$ reaction in good yields (Scheme 1). We have also examined the sequence involving the use of the ammonium salt previously described with quite bulky secondary amines in methanol to afford allyl amines.^[20] On the basis of this work, it has been observed that 1,4-diazabicyclo[2.2.2]octane (DABCO) plays an important role in the stereoselectivity of the obtained product either, allyl amines or allyl ethers, by the formation of a co-nucleophile



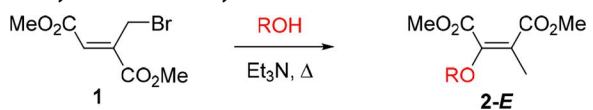
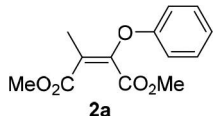
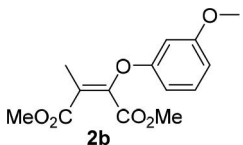
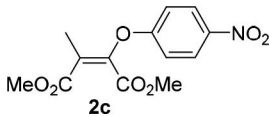
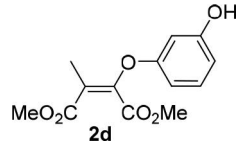
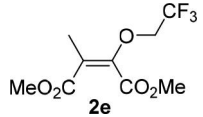
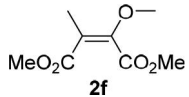
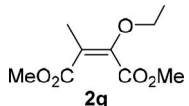
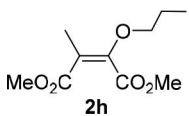
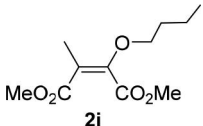
Scheme 2. A convenient pathway for synthesis of allyl bromide **1**.



Scheme 3. Vinylic etherification of brominated derivative **1**.

which can be easily dislodged by conjugate addition of heteroatom nucleophiles. In continuation of this study, we enhance herein the reaction shown in [Scheme 1](#) by implementing an effective protocol for the synthesis of α,β -difunctionalized vinyl ethers **2** by the

Table 1. Stereoselective synthesis of *E*-vinyl ethers.

				
Entry	R	Vinyl ethers	Time (h)	Yields ^a (%)
1	C ₆ H ₅	 2a	2	95
2	<i>p</i> -MeOC ₆ H ₄	 2b	1	90
3	<i>p</i> -NO ₂ C ₆ H ₄	 2c	1,5	92
4	<i>m</i> -OHC ₆ H ₄	 2d	1,5	70
5	CF ₃ CH ₂	 2e	11	60
6	Me	 2f	17	65
7	Et	 2g	22	51
8	nPr	 2h	76	48
9	nBu	 2i	144	45
10	tBu	no reaction	144	–

^aIsolated yields after purification by column chromatography on silica gel.

combination of allyl bromide **1** with various alcohols without addition of DABCO. A mixture of two regioisomers has been formed after being stirred for 5 min which allyl ethers disappeared in favor of vinyl ethers by spontaneous isomerization.^[21] The coupling reaction was activated by the use of triethylamine leading to the formation of vinyl ethers **2** with total stereoselectivity in favor of *E*-isomer with good yields ranging from 45 to 92%. The vinyl ethers **2** could be used as monomers for the synthesis of polyamides or polyesters. Geometry of the resulting multisubstituted vinyl ethers **2** was examined by NOE experiments by considering the trend of the through-space coupling between two atom groups OR and methyl to develop a rational criterion for the structural assignment of the vinyl ethers **2** as well are significantly in favor of the corresponding *E*-configuration (Scheme 3).

Substituents at the *meta* and *para* positions of the aryl group of substituted phenol derivatives have shown that they are more reactive than the aliphatic alcohols such as MeOH, EtOH, *n*-PrOH, and *n*-BuOH and the corresponding vinyl ethers **2a–d** have been obtained in good yields with low reaction time (1–2 h) (Table 1).

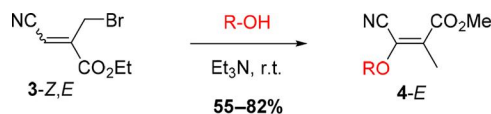
Notably, the reaction seems to be inhibited by the size of the alkyl primary alcohol groups. The use of a bulky tertiary alcohol (such as *tert*-butyl alcohol) as an oxygen nucleophile is ineffective even after 144 h of stirring at reflux. It seems to be affected by the steric hindrance of the alcohol which attenuates its nucleophilicity (entry 10, Table 1).

To expand the field of the reactivity of allylic bromides toward oxygen nucleophiles, we have tried the coupling reaction of ethyl 2-(bromomethyl)-3-cyanoacrylate **3**^[18] as a mixture of two isomers *Z* and *E* with a series of aliphatic and aromatic alcohols activated by the presence of triethylamine. A mixture of two regioisomers was observed and a complete isomerization of allyl ethers into vinyl ethers **4** was formed in *E*-configuration with good yields (Scheme 4).

The aryl-substituted phenol has shown that they are more reactive than the aliphatic alcohols such as *n*-PrOH and *n*-BuOH and the corresponding vinyl ethers **4a–d** have been obtained in good yields with moderate reaction time (5–10 h) (Table 2). Moreover, the steric hindrance of the alcohol attenuates its nucleophilicity in the case of (*o,o',p*)-tribromophenol and (*o,o',p*)-trinitrophenol (entries 8 and 9—Table 2). The very low reactivity of HC≡C–CH₂OH (entries 7—Table 2) is attributed to the attracting effect of the two hybridized carbons sp.

Theoretical calculations have been made on the ethyl 3-cyano-2-phenoxybut-2-enoate **4a** show that the configuration *E* is more stable compared to the corresponding *Z*-isomer. The geometry optimizations in vacuo, were performed with Jaguar 7.6^[22] at the DFT level, using the B3P86^[23] hybrid density functional, the basis set used for computations was 6–31 + g**. ^[24] As expected, the *E*-isomer **4a** has the lowest energy and therefore the most stable structure (Fig. 1).

Such functionalized enol ethers can be of interest as building blocks to synthesize elaborated molecules. Thus we have used compound phenoxymaleate **2a** as starting

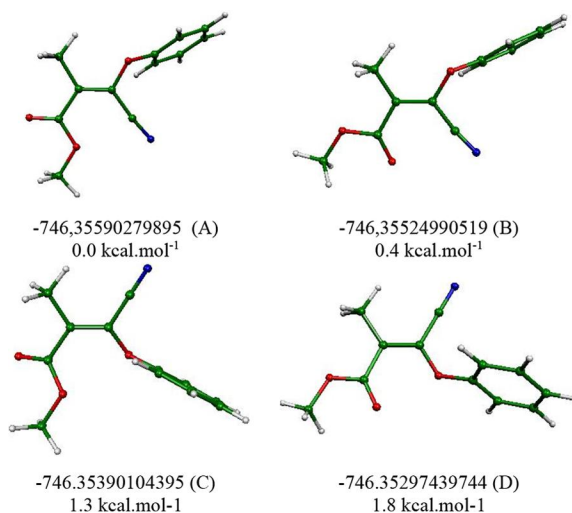


Scheme 4. Direct synthesis of vinyl ethers **4**.

Table 2. Total stereoselective synthesis of vinyl ethers.

Reaction scheme: $\text{NC}-\text{CH}=\text{CH}-\text{CO}_2\text{Et}$ (**3-Z,E**) $\xrightarrow[\text{Et}_3\text{N, r.t.}]{\text{R-OH}}$ $\text{NC}-\text{CH}=\text{CH}-\text{CO}_2\text{Me}$ (**4-E**)

Entry	R	Vinyl ethers	Time (h)	Yields ^a (%)
1	C ₆ H ₅	 4a	10	82
2	<i>p</i> -MeOC ₆ H ₄	 4b	5	80
3	<i>m</i> -OHC ₆ H ₄	 4c	6	60
4	C ₆ H ₅ CH ₂	 4d	10	75
5	<i>n</i> -C ₃ H ₇	 4e	24	50
6	<i>n</i> -C ₄ H ₉	 4f	20	55
7	C ₃ H ₃	No reaction	60	—
8	<i>o,p,p'</i> -Br ₃ C ₆ H ₂	No reaction	65	—
9	<i>o,p,p'</i> -NO ₂ C ₆ H ₂	No reaction	70	—

^aIsolated yields after purification by column chromatography on silica gel.**Figure 1.** Calculated energy difference for *Z*- and *E*-**4a** isomers.

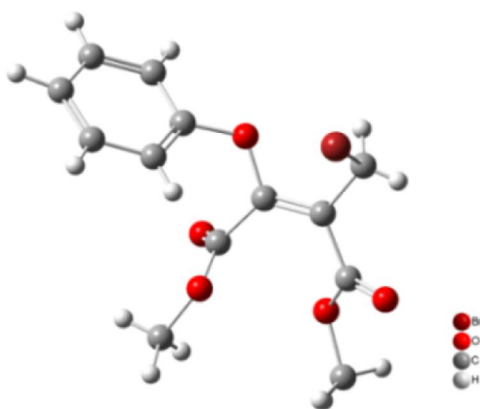
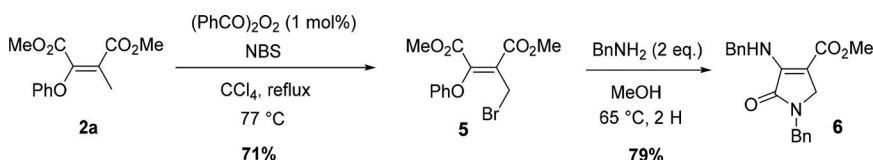


Figure 2. X-ray crystal structure of Z-5. CCDC 1548610.^[27]



Scheme 5. Efficient synthesis of 3,4-disubstituted pyrrolin-2-one **6**.

material to access caspase-3 inhibitors with a pyrrolinone scaffold.^[25] For this, we first performed radical bromination of **2a** with *N*-bromosuccinimide under classical conditions (in refluxing tetrachloride with benzoyl peroxide^[26] as initiator) to give allyl bromide Z-5 in 71% yield (Scheme 4). The stereochemistry of the functionalized allyl bromide **5** was determined based on the X-ray diffraction analysis (Fig. 2). The molecular structure shows that the double bond in **5** is in *Z*-configuration, and the phenoxyl group is attached to the β -carbon of the carbonyl group and the methylene bromide atom is connected to the α -carbon, which suggests that the bromination of **2a** is achieved in a *syn*-fashion. It can be deduced that the vinyl ether **2** has an *E*-configuration by analogy with the preceding result. Therefore Z-3, was reacted with an excess of benzylamine, to undergoes two consecutive transformations: aza-Michael addition-cyclization reactions to produce the target bioactive 3,4-disubstituted pyrrolin-2-one **6**^[25] (Scheme 5).

Experimental

All reagents and solvents were purchased from commercial sources and used as received without further purification. Reactions were routinely performed under nitrogen atmosphere with magnetic stirring. All reactions were monitored by TLC on silica gel plates (Fluka silica gel 60 F₂₅₄, Merck) and visualized by a 254 nm UV lamp and potassium permanganate aqueous solution. The crude products were purified using column chromatography on silica gel; Fluka silica gel 70-230 mesh was used. ¹H, ¹³C NMR, and NOESY spectra were recorded on a Bruker AC 400 MHz spectrometer at 400 and 77 MHz, for ¹H, ¹³C, and NOESY respectively in deuterated chloroform as solvent and tetramethylsilane as internal standard. The chemical shifts (δ) and coupling constants (*J*) are respectively, expressed in parts per million (ppm) and Hertz (Hz). All NMR spectra were

acquired at room temperature. Multiplicity of peaks is indicated by the following: s, singlet; d, doublet; t, triplet; q, quartet; quint, quintuplet; sext, sextuplet, and m, multiplet. High resolution mass spectroscopy data were recorded on a micromass quadrupole time-of-flight instrument with an electrospray source in the EI or ESI mode in the university of Rouen and GC-MS analyses were performed with a GCMS-QP2010S (Shimadzu) instrument with a GC-2010 equipped with a 30 m capillary column (Supelco, SLBTM-5ms, fused silica capillary column, 30 m \times 0.25 mm \times 0.25 mm film thickness), which was used with helium as the vector gas in the university of Rennes 1.

General procedure for the preparation of vinyl ethers 2

To a stirred solution of dimethyl α -(bromomethyl)fumarate **1** (0.5 g, 1.83 mmol) and alcohols (1.83 mmol, 1 equiv.) was added dropwise triethylamine (0.5 mL, 3.66 mmol) in DCM (5 mL) under a nitrogen atmosphere. The mixture was then stirred vigorously at 39.6 °C for 1–144 h. Next, the mixture was diluted with DCM (20 mL) and washed successively with 2N HCl solution and water. The organic layer was dried over anhydrous Na_2SO_4 and concentrated in vacuo. The crude product, thus obtained was purified by column chromatography on silica gel to afford pure vinyl ethers **2**.

Dimethyl 2-methyl-3-phenoxy maleate 2a

Colorless liquid; yield: (0.26 g, 95%); eluent CH_2Cl_2 /petroleum ether, 60:40; ^1H NMR (CDCl_3 , δ ppm, J Hz): 7.23 (t, 2H, $J = 8.00$), 7.00 (t, 1H, $J = 8.00$), 6.90 (d, 2H, $J = 8.00$), 3.75 (s, 3H), 3.59 (s, 3H), 1.92 (s, 3H); ^{13}C NMR (CDCl_3 , δ ppm): 168.3 (CO_2Me), 163.0 (CO_2Me), 155.9 (aromatic =C), 144.2 (=C), 129.7 (2 aromatic =CH), 126.6 (=C), 123.4 (aromatic =CH), 116.3 (2 aromatic =CH), 52.5 (OCH_3), 52.4 (OCH_3), 13.6 (CH_3). HRMS calcd. for $[\text{M}+\text{H}]^+$ $\text{C}_{13}\text{H}_{15}\text{O}_5$: 251.0905; found: 251.0902.

Typical procedure for the synthesis of vinyl ethers 4

General procedure for aryl alcohols

To a solution of ethyl (*Z,E*)-2-(bromomethyl)-3-cyanoacrylate **3** (0.3 g, 1.38 mmol) and aryl alcohols (1.38 mmol, 1 equiv.) was added dropwise triethylamine (0.38 mL, 2.76 mmol) in CH_3CN (5 mL) under a nitrogen atmosphere. The mixture was then stirred vigorously at 25 °C for 5–10 h. Next, the excess of solvent was concentrated under reduced pressure, and the organic residue obtained was purified by chromatography on silica gel using a mixture of petroleum ether/ AcO_2Et (80:20) as eluent to provide ethyl (*E*)-3-cyano-2-methyl-3-aryloxyacrylate **4**.

Ethyl (*e*)-3-cyano-2-methyl-3-phenoxyacrylate 4a

Yellow liquid; yield: (0.26 g, 82%); ^1H NMR (CDCl_3 , δ ppm, J Hz): 7.39 (t, 2H, $J = 8.00$), 7.21 (t, 1H, $J = 8.00$), 7.05 (d, 2H, $J = 8.00$), 4.35 (q, 2H, $J = 8.00$), 2.10 (s, 3H), 1.37 (t, 3H, $J = 8.00$); ^{13}C NMR (CDCl_3 , δ ppm): 164.7 (CO_2Et), 154.3 (aromatic =C), 131.7 (=C), 130.0 (aromatic =CH), 125.3 (=C), 118.2 (aromatic =CH), 112.2 (CN), 62.1 (OCH_2), 14.0 (CH_3), 12.8 (CH_3). HRMS calcd. for $[\text{M}]^+$ $\text{C}_{13}\text{H}_{13}\text{NO}_3$: 231.08954; found: 231.08914.

Conclusion

In summary, we have developed a simple and convenient method for stereoselective synthesis of functionalized enol ethers by Et_3N -activated sequential $\text{S}_{\text{N}}2'$ reaction/isomerization from various alcohols and allyl bromides obtained from Morita–Baylis–Hillman adducts. This simple procedure involves simple starting materials and the obtained vinyl ethers can offer an access to pyrrolinones.

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