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Novel and Efficient Method for the Allylation of Carbonyl Compounds and Imines Using Triallylaluminum

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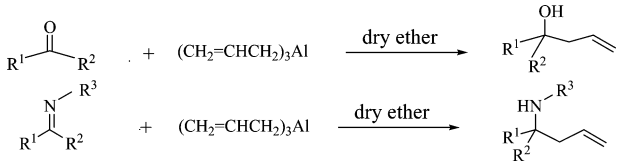

This is the first report of the use of triallylaluminum as a reagent for the allylation of carbonyl compounds and imines. The allylation of ketimines without additional metal catalyst is known so far only in the case of the Grignard reagent. Triallylaluminum is a useful alternative to provide the homoallylic amines in excellent yield upon addition to aldimines and ketimines. The significant reactivity of this reagent was confirmed by its reaction with a sterically rigid ketone such as adamantanone to provide 1-adamantyl-3-buten-1-ol in 98% yield. The chemoselectivity of triallylaluminum was demonstrated by using different ketoesters. It is noteworthy that triallylaluminum is prepared from allyl bromide and aluminum metal, and not from a Grignard reagent, and that the procedure is operationally simple, leading to good to excellent product yields.

The allylation of carbonyl derivatives and imines is of great interest in carbon−carbon bond-forming reactions as a result of the versatility of homoallylic alcohols and amines as synthetic intermediates.[1] Homoallylic alcohols are particularly valuable intermediates that have been used as building blocks of numerous macrolides and ionophore antibiotics.[2] Among the methods reported for the allylation of these compounds, the most common is a Barbier-type allylation in which allyl halide along with different metal sources are used,[3] and by the addition of an allylic Grignard-type reagent.[4] Over the past few decades, a number of organometallic reagents such as Grignard reagents, organolithiums,[5] organosilanes,[6] and organostannanes[7] have been developed for these transformations. The Grignard-type carbonyl addition of allyl halides has been developed in which organometallics derived from a number of metallic elements such as manganese,[8] zinc,[9] tin,[10] antimony,[11] magnesium,[12] cerium,[13] and bismuth are used.[14] Allylation reactions with organometallic reagents has largely been used in conjunction with aldehydes, but rarely for ketones, because of the difference in reactivity between these carbonyl groups. Wada and co-workers[14] reported on the allylation of aldehydes using allyl bromide and metallic bismuth [Bi(0)] or bismuth(III) chloride in the presence of metallic species such as Zn(0), Fe(0), and Al(0). However, the use of bismuth(III) chloride was not successful in the absence of Al(0) and Al(0) alone did not lead to the production of the desired products. Mukaiyama et al.[15] reported on the efficient allylation of aldehydes using allyldiethylaluminum, in which the organoaluminum was produced via the reaction of diethylaluminum chloride with allylmagnesium chloride. Albeit, aldehydes responded well with this reagent and ketones were unreactive. It is always important to examine the use of various elements in organic synthesis from the standpoint of their use in the future. Reports on the allylation of carbonyl compounds using organoaluminum reagents are relatively rare.[16] Thus, it would be desirable to develop an efficient allylation method for a wide variety of substrates with organoaluminum compounds in a Grignard-type addition, because aluminum is an inexpensive and convenient alternative to conventionally used metals, such as magnesium and zinc. In this paper, we wish to report on a convenient and efficient procedure for the allylation of aldehydes, ketones, and imines using triallylalumium (**2**) in excellent to good yields. It is noteworthy that the preparation of the triallylalumium reagent may be carried out conveniently using allyl bromide and aluminum metal,[17,18] a significant improvement over earlier methods which relied upon the reaction of a dialkylaluminum halide with allylmagnesium chloride.[19]

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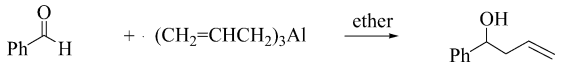
TABLE 1. Effect of the Loading of Triallylaluminum in a Reaction with Benzaldehyde



1a **2** **3a**

entry	**2** (mmol)	0 °C[a] **3a**[d] (%)	−30−0 °C[b] **3a**[d] (%)	−78−0 °C[c] **3a**[d] (%)
1	0.20	25	27	28
2	0.30	56	61	75
3	0.40	86	87	96
4	0.50	86	95	98

[a] All reactions were carried out with benzaldehyde (1.0 mmol in 3 mL dry ether) and triallylaluminum at 0 °C under nitrogen for 30 min. [b] All reactions were carried out with benzaldehyde (1.0 mmol in 3 mL dry ether) and triallylaluminum at −30 °C first, and the temperature was then gradually increased to 0 °C under nitrogen for 40 min. [c] All reactions were carried out with benzaldehyde (1.0 mmol in 3 mL dry ether) and triallylalumium at −78 °C first, and the temperature was then gradually increased to 0 °C under nitrogen for 90 min. [d] Isolated yields.

Preliminary endeavors focused on product yield optimization. Initially, a reaction was carried out using benzaldehyde and triallylaluminum (0.2 mmol) in dry ether at 0 °C, which resulted in the production of the homoallylic alcohol in 25% yield after 30 min. With this information in hand, we then investigated the effect of temperature as well as the amount of triallylaluminum in an attempt to increase the product yield. A gradual increase in the amount of triallylaluminum used improved the yield considerably, and the results are tabulated (Table 1).

From Table 1, it is evident that 0.4 mmol of triallylaluminum is required to obtain the maximum yield. When the reaction was carried out at 0 °C, the product was formed in 86% yield, whereas adding triallylaluminum at −30 °C and then gradually increasing the temperature to 0 °C resulted in only a slight improvement in product yield. However, the maximum product yield (96%) was obtained by adding triallylaluminum at −78 °C, followed by a gradual increase in the temperature to 0 °C. The increased yield at lower temperatures can be attributed to the decreased reactivity of the aldehyde, which in turn facilitates the formation of a polymer-free product. Using additional equivalents (0.5 mmol) of triallylaluminum at 0 °C had no effect on the yield; on the other hand, only a small improvement was observed in product yield when the reaction was conducted at lower temperatures. With this encouraging result in hand, we further investigated the reaction of ketones. The latter reaction required a larger amount of triallylaluminum to proceed smoothly, but products were produced in excellent yield. As expected, an increase in the amount of triallylaluminum used led to an increase in product yields, even in the case of ketones.

These results suggest that a minimum of 0.4 mmol of triallylaluminum is needed in the allylation of aldehydes and 0.7 mmol is needed in the case of ketones to obtain the maximum product yield. The difference in the amount of triallylaluminum required to initiate these reactions may be explained on the basis of their reactivity. To further examine the versatility of this reagent, a range of carbonyl compounds were then allylated under the optimized conditions, and the results are summarized in Table 2.

Homoallylic alcohols were obtained in good to excellent yields, not only with aldehydes but also with ketones, by slight variations in the temperature. Both aromatic as well as aliphatic substrates were converted smoothly to the corresponding homoallylic alcohols in excellent yields. The results clearly show that a substituent on the phenyl ring, whether electron-donating or electron-withdrawing, had almost no influence on the reactions. Further, the position of the substituent on the phenyl ring almost does not affect product yield. For example, 4-chlorobenzaldehyde and 4-methylbenzaldehyde afforded allylated products in excellent yields. Similarly, the use of the corresponding 2-substituted benzaldehydes also resulted in excellent yields. In addition to other aldehydes, the bulkier 1-formyl naphthalene also gave an excellent yield while conducting the reaction by method B (−78 °C to 0 °C). On the other hand, almost all ketones furnished homoallylic alcohols in excellent yields at 20 °C. Sterically hindered ketones such as di-*tert*-butyl ketone, 2-acetyl naphthalene, and benzophenone also afforded excellent yields of product. Moreover, this reagent has been employed with cyclic ketones such as cyclohexanone to give the corresponding allylated products in almost quantitative yields. The significant reactivity of this reagent was confirmed in a reaction with a sterically rigid ketone, adamantanone. The higher yield obtained with adamantanone demonstrates the versatility of triallylaluminum as an efficient allylating agent for a wide range of substrates. It is noteworthy that this is the first and foremost example on the allylation of 2-adamantanone with an organometallic reagent and that these types of sterically rigid structures are helpful in studying face selectivity.[20] To the best of our knowledge, this constitutes an original report of a Grignard-type addition on the allylation of ketones using triallylaluminum.

The allylation of aldimines and ketimines provides a useful synthetic pathway to homoallylic amines, which are very useful intermediates in the syntheses of natural products.[21] Studies have also revealed that secondary and tertiary homoallylic amines such as zimelidine, norzimelidine, and so on, were neuronal norepinephrine and serotinine-uptake inhibitors of biological interest.[22] Homoallylic amines are generally prepared either by the addition of an organometallic reagent to an imine[23] or by the nucleophilic addition of allylmetal reagents in the presence of Lewis acids.[24] A variety of catalysts have been developed for the preparation of homoallylic amines using three-component coupling reactions[25] including lanthanide triflates[26] and bismuth triflate.[27] However, the high cost, substrate specificity, and

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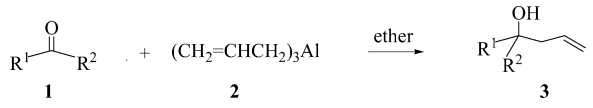
TABLE 2. Reaction of Aldehydes and Ketones with Triallylaluminum 2 To Generate Homoallylic Alcohols 3



1 2 3

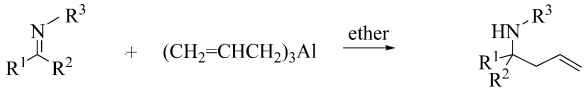
entry	1	R^1	R^2	3	Method A[a,b] (%)	Method B[b,c] (%)	Method C[b,d] (%)
1	**1a**	Ph	H	**3a**	86	96	
2	**1b**	4-ClC_6H_4	H	**3b**	83	98	
3	**1c**	4-MeC_6H_4	H	**3c**	83	97	
4	**1d**	2-ClC_6H_4	H	**3d**	84	96	
5	**1e**	2-MeC_6H_4	H	**3e**	79	96	
6	**1f**	$PhCH_2CH_2$	H	**3f**	99	95	
7	**1g**	2-thienyl	H	**3g**	82	50	
8	**1h**	1-naphthyl	H	**3h**	81	97	
9	**1i**	3-pentyl	H	**3i**	83	83	
10	**1j**	cyclohexyl	H	**3j**	99	98	
11	**1k**	Bu	H	**3k**	84	82	
12	**1l**	*t*-Bu	H	**3l**	70	90	
13	**1m**	Ph	CH_3	**3m**			98
14	**1n**	4-ClC_6H_4	CH_3	**3n**			98
15	**1o**	4-MeC_6H_4	CH_3	**3o**			95
16	**1p**	2-ClC_6H_4	CH_3	**3p**			97
17	**1q**	2-MeC_6H_4	CH_3	**3q**			96
18	**1r**	2-naphthyl	CH_3	**3r**			96
19	**1s**	$PhCH_2CH_2$	CH_3	**3s**			98
20	**1t**	$-(CH_2)_5-$		**3t**			98
21	**1u**	$-(CH_2)_4-$		**3u**			89
22	**1v**	Et	Et	**3v**			89
23	**1w**	Ph	Ph	**3w**			94
24	**1x**	*t*-Bu	*t*-Bu	**3x**			90
25	**1y**	2-adamantanone		**3y**			98

[a] Method **A**: all reactions were carried out with aldehyde (1.0 mmol in 3 mL dry ether) and triallylaluminum (0.4 mmol, 0.5 M × 0.8 mL) at 0 °C under nitrogen for 30 min. [b] Isolated yields. [c] Method **B**: all reactions were carried out with aldehyde (1.0 mmol in 3 mL dry ether) and triallylaluminum (0.4 mmol, 0.5 M × 0.8 mL) at −78 °C first and then increased to 0 °C gradually under nitrogen for 90 min. [d] Method **C**: all reactions were carried out with ketone (1.0 mmol in 3 mL dry ether) and triallylaluminum (0.7 mmol, 0.5 M × 1.4 mL) at 20 °C under nitrogen for 30 min.

sensitivity of these reagents limit these processes. The allylation of ketimines without the need for a metal catalyst is not known so far, except for the case of a Grignard reagent.[28] Thus, under the optimized conditions developed for the carbonyl compounds, we initiated a study of allylation reactions of aldimines as well as ketimines. To evaluate the efficiency of this reagent and to further explore the scope of the reagent, a series of substituted aldimines and ketimines were subjected to the allylation reaction using triallylaluminum. Both aldimines and ketimines reacted with equal efficiency to afford products smoothly and in excellent yield (Table 3).

Although there was no significant difference in product yields, the reaction temperatures required varied depending on the reactivity pattern of the substrates. In the case of aldimines, the substituent on the nitrogen atom was a key determinant of reactivity. For example, the reaction of *N*-methylimine derived from benzaldehyde required 0 °C, the *N*-naphthylimine derived from the same compound required 20 °C, and the other aldimines reacted at room temperature to afford the maximum product yields. On the other hand, ketimines react with

TABLE 3. Reaction of Aldimines and Ketimines with Triallylaluminum 2 To Generate Homoallylic Amines 5



4 2 5

entry	4	R^1	R^2	R^3	5	0 °C[a,b] (%)	50 °C[b,c] (%)
1	**4a**	Ph	H	Ph	**5a**	98	
2	**4b**	4-$MeOC_6H_4$	H	Ph	**5b**	98	
3	**4c**	4-ClC_6H_4	H	Ph	**5c**	98	
4	**4d**	1-naphthyl	H	Ph	**5d**	95	
5	**4e**	2-thienyl	H	Ph	**5e**	98	
6	**4f**	Ph	H	Bn	**5f**	98	
7	**4g**	2-thienyl	H	Bn	**5g**	98	
8	**4h**	Ph	H	CH_3	**5h**	96[d]	
9	**4i**	Ph	H	1-naphthyl	**5i**	98	
10	**4j**	Ph	CH_3	Ph	**5j**		98
11	**4k**	4-MeC_6H_4	CH_3	Ph	**5k**		96
12	**4l**	4-ClC_6H_4	CH_3	Ph	**5l**		98
13	**4m**	2-naphthyl	CH_3	Ph	**5m**		98
14	**4n**	2-thienyl	CH_3	Ph	**5n**		94
15	**4o**	Ph	CH_3	Bn	**5o**		98
16	**4p**	2-naphthyl	CH_3	Bn	**5p**		98
17	**4q**	Ph	Ph	H	**5q**		98[e]
18	**4r**	CH_3	CH_3	Ph	**5r**		97[e]

[a] All reactions were carried out with aldimine (1.0 mmol in 3 mL dry ether) and triallylaluminum (0.6 mmol, 0.5 M × 1.2 mL) at 20 °C under nitrogen for 30 min. [b] Isolated yields. [c] All reactions were carried out with ketimine (1.0 mmol in 3 mL dry ether) and triallylaluminum (0.9 mmol, 0.5 M × 1.8 mL) at 50 °C under nitrogen for 1 h. [d] Reaction was carried at 0 °C for 30 min. [e] Reaction was carried at 50 °C for 30 min.

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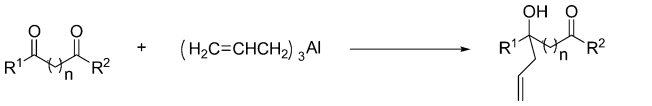
SCHEME 1



n = 0; R^1 = Ph; R^2 = OCH_3; **6**

n = 1; R^1 = CH_3; R^2 = OCH_3; **7**

n = 0; R^1 = Ph; R^2 = OCH_3; **8**

n = 1; R^1 = CH_3; R^2 = OCH_3; **9**

triallylaluminum to afford homoallylic amines in excellent yields at 50 °C. Irrespective of the pattern of substitution, either on nitrogen or on the phenyl ring, excellent product yields were observed with all ketimines. However, slight variations in reaction times were observed for ketimines derived from symmetrical ketones such as benzophenone and acetone. For example, with these substrates, only a 30 min reaction time was needed to generate products in quantitative yield. In another variation, the chemoselectivity of this reagent was confirmed by extending the scope of this reagent to keto esters under different reaction conditions (Scheme 1). The α-keto ester **6** reacts with triallylaluminum at 20 °C to afford product **8** in 86% yield after 30 min, whereas in the case of the *β*-ketoester **7**, the reaction did not proceed at the same temperature. A series of experiments conducted at different temperatures enabled us to establish the optimum conditions, and when the reaction was carried out at −40 °C for 30 min, product **9** was obtained in 88% yield, with a recovery of 9% of the starting material.

Compared to other allyl reagents, the preparation of triallylaluminum is relatively easy, and the experimental procedures are simple and convenient with short reaction times. For example, the preparation of allylsamarium reagents from samarium metal and allyl bromide is more expensive, and the reactivity of the reagent is also limited to simple aldehydes and ketones with longer reaction times. Similarly, the preparation of allylzinc is also troublesome, and the use of allyltin reagents is environmentally harmful due to its toxic nature.

In conclusion, we report on a simple and general procedure for the allylation of carbonyl derivatives using triallylaluminum. The reaction can be applied to a wide variety of aldehydes, ketones, aldimines, and ketimines. To the best of our knowledge, this is the first report of the use of triallylaluminum as a reagent for the allylation of carbonyl compounds and imines. The simple procedures, high reaction rates, and excellent product yields described here for the preparation of homoallylic alcohols as well as amines using triallylaluminum, make this method an alternative to existing processes. The main features of this reaction are (1) the triallylaluminum is prepared from allyl bromide and aluminum metal, not from a Grignard reagent; (2) this is the first report of the use of triallylaluminum as a reagent in the allylation of carbonyl compounds and imines; (3) a broad substrate scope including sterically hindered ketones such as adamantanone, imines, and keto esters; (4) the allylation of ketimines without any metal catalyst is not known so far except with Grignard reagent; and (5) the procedure is operationally simple and provides good to excellent product yields.

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Supporting Information Available: Copies of ^1H and ^{13}C NMR spectra for homoallylic alcohols and amines, detailed X-ray information of 1-adamantyl-3-buten-1-ol. This material is available free of charge via the Internet at http://pubs.acs.org.

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