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Highly *Z*/*E* stereoselective approach to β-iodo aza Morita–Baylis–Hillman adducts

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Abstract—A multicomponent reaction between sulfonyl-protected imines, magnesium iodide, and acetylenic esters or ketones is described. The resulting β-iodo aza Morita–Baylis–Hillman adducts were obtained in good yields and excellent *Z*/*E* stereoselectivities. The reaction showed good tolerance for sulfonyl protecting groups, as well as for acetylenic ketones and esters. This work presents the first synthetic approach to β-iodo aza Morita–Baylis–Hillman adducts.

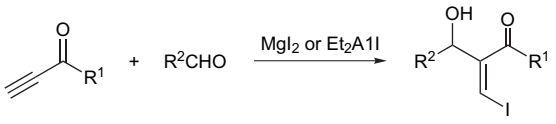
1. Introduction

Multicomponent reactions (MCRs) play a key role in organic chemistry due to the fact that highly complex structures can be formed in a simple one-pot process. Recent interest in MCRs has resulted in a number of elegant new reactions for the preparation of many new useful synthons.[1]

Recently, the Morita–Baylis–Hillman (MBH) reaction has received considerable interest from the synthetic community as a convenient reaction for the synthesis of allylic alcohols in an atom-economical fashion.[2] A number of useful Lewis base[3] and Lewis acid[4,5] catalyzed versions have been reported. Additionally, aza MBH reactions have been developed utilizing sulfonyl-protected imines, with useful asymmetric versions appearing only recently.[6,7]

An area of interest in our laboratory has been the synthesis of halo-aldol/halo-MBH adducts.[5,8] Such tandem C–C/C–X bond formation reactions lead to aldol or MBH products with an additional degree of functionalization not present in the classical versions of the respective reactions. The presence of a halogen in the product allows for a number of further transformations to be performed on the products, including eliminations, intermolecular and intramolecular displacements, and coupling reactions. Such transformations allow for the rapid synthesis of complex target molecules from simple starting materials.

One strategy we have recently employed involves the multicomponent coupling of α,β-acetylenic ketones or esters with aldehydes in the presence of a metal iodide (Et_2AlI or MgI_2) acting as Lewis acid promoter and halogen source (Scheme 1).[8,9] In particular, reactions utilizing MgI_2 have been found to be especially convenient since MgI_2 is a fairly air-stable solid that is easy to work with and is readily available.[9] Such reactions have led to the high stereoselective synthesis of β-iodo MBH adducts. In an effort to extend the scope of this reaction, we sought to utilize imines as electrophilic acceptors in order to obtain the corresponding aza MBH products. In this paper, we are pleased to report our initial results for the preparation of β-iodo aza MBH adducts.



Scheme 1.

2. Results and discussion

Initially, the tosyl imine of benzaldehyde was treated with 1.2 equiv 3-butyn-2-one and 1.1 equiv of MgI_2 in CH_2Cl_2 at 0 °C. The reaction was found to proceed to completion in 18 h to afford the product in 70% yield with exclusive *Z* stereoselectivity. The stereochemistry about the double bond was confirmed by 1D NOE spectroscopy in which an NOE enhancement was observed between the vinyl proton and the N–C*H*, and no enhancement was observed between the vinyl proton and the methyl group adjacent to the ketone.

Encouraged by these initial results, we sought to utilize esters to replace ketones since the resulting products would

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be the synthetically attractive di-protected β-amino acid derivatives. Fortunately, when methyl propiolate was used to replace 3-butyn-2-one, the yield was increased from 70 to 79%. It is noteworthy to mention that the crude reaction mixture was found to be cleaner for esters, possibly due to self-condensation of ketones. Further optimization experiments were thus conducted using methyl propiolate and the tosyl imine of benzaldehyde.

Initially, several solvents were screened and CH_2Cl_2 was found to be the best. Interestingly, the use of CH_3CN, THF, and toluene all resulted in the formation of no product at all. The NMR spectra (after aqueous workup) of crude reactions using these solvents were clean, containing only starting materials and β-iodo methyl acrylate, the result of quenching the iodo-allenolate intermediate. Increasing the reaction temperature from 0 °C to room temperature showed neither improvement in yield nor diminishment in stereoselectivity. The use of 1.5 equiv each of alkyne and MgI_2 did not result in a detectable increase in the rate of consumption of imine. With the above results in mind, all subsequent reactions were performed at 0 °C in CH_2Cl_2, utilizing 1.0 equiv imine, 1.2 equiv alkyne, and 1.1 equiv MgI_2.

Substrate scope was examined with regard to alkyne, protecting group, and aldehyde component of the imines. The results are summarized in Table 1. Interestingly, several *N*-sulfonyl protecting groups were found to be effective in this process. No obvious difference between tosyl (Ts) and benzenesulfonyl (Bs) protecting groups was noted. The methanesulfonyl (Ms) group also worked, however, the yield was somewhat diminished when compared to Ts and Bs substrates. Not surprisingly, *N*-benzyl and *N*-aryl imines failed to react in this system.

With regard to alkyne substrate scope, both the methyl ketone and the methyl esters worked well, however, as previously mentioned, the ester gave somewhat better yield and cleaner reaction mixtures than the ketone. Interestingly, the use of ethyl propiolate (entry 3, Table 1) resulted in the formation of the product in high yield but with somewhat diminished stereoselectivity.

It is worth noting that imines prepared from electron-rich and electron-deficient aldehydes both worked well. Furthermore, heteroaromatic imines (entries 10 and 11, Table 1) also provided satisfactory results. Unfortunately, aliphatic and α,β-unsaturated imines worked poorly, reacting only very sluggishly and affording low yields. Attempts to devise a process that is more amenable to these substrates are currently underway.

In summary, a convenient synthesis of β-iodo aza MBH products is reported. The reaction shows good substrate scope and high yields and stereoselectivities were obtained for a number of examples. These products give rise to numerous implications for the synthesis of novel β-amino acids for peptide and catalysis studies.

3. Experimental

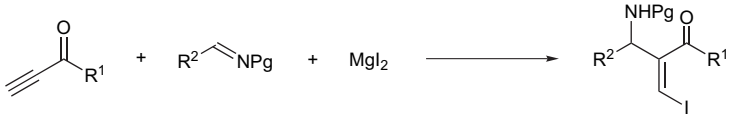
3.1. General

All reactions were performed in oven-dried glassware. Dichloromethane was dried by passing through an alumina column under an N_2 atmosphere. Imines were prepared according to standard procedures.[10] All other chemicals were commercially available and used without further purification. Stoichiometries were calculated based on the purities reported by the manufacturers. Flash chromatography was performed on silica gel (Merck 60, 230–400 mesh). Melting points were taken in open capillaries and are reported uncorrected. IR spectra were recorded as CH_2Cl_2 deposits on a NaCl disk. NMR spectra were recorded on a Varian Mercury NMR spectrometer operating at 300 MHz (^{1}H) and 75 MHz (^{13}C). Shift values are reported in parts per million and are referenced based on TMS or solvent for ^{1}H and ^{13}C, respectively. All spectra were recorded in $CDCl_3$.

3.2. Procedure for synthesis of β-iodo aza MBH products

Into an oven-dried vial was added imine (0.5 mmol) and MgI_2 (0.55 mmol). The vial was fitted with a septum and

Table 1. Results of the synthesis of β-iodo aza Morita–Baylis–Hillman adducts



Entry	R^1	R^2	Pg	*Z*/*E*[a]	Yield[b]
1	Me	Ph	Ts	>20:1	70
2	OMe	Ph	Ts	>20:1	79
3	OEt	Ph	Ts	17:1	84
4	OMe	Ph	Ms	>20:1	68
5	OMe	4-Me–C_6H_4	Bs	>20:1	72
6	OMe	4-MeO–C_6H_4	Ts	>20:1	77
7	OMe	3-BnO–C_6H_4	Ts	>20:1	73
8	OMe	4-F–C_6H_4	Ts	>20:1	82
9	OMe	4-Cl–C_6H_4	Bs	>20:1	84
10	OMe	2-Furyl	Ts	>20:1	83
11	OMe	2-Thienyl	Ts	>20:1	78

[a] Determined by ^{1}H NMR analysis of crude reaction mixture.
[b] Yield of analytically pure sample after purification via flash chromatography. >20:1 means that no minor isomer was observed.

flushed with nitrogen. Dichloromethane (3 mL) was then added and the reaction was allowed to stir at 0 °C for 20 min, at which time the alkyne (0.60 mmol) was added in one portion via a syringe. The reaction was allowed to stir at 0 °C for 18 h before being quenched with 5 mL 1 N HCl. The reaction mixture was extracted with dichloromethane (3×10 mL) and the combined organic layers were washed with brine and dried with Na_2SO_4. The mixture was concentrated under reduced pressure and purified via flash chromatography (EtOAc/hexane, 1:5) to afford the pure product.

3.2.1. Table 1, entry 1. Isolated as a white solid. Mp=145–147 °C. FTIR: 3275.7, 1700.5 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.68–7.64 (m, 2H), 7.28–7.23 (m, 5H), 7.14–7.10 (m, 2H), 6.58 (d, *J*=1.2 Hz, 1H), 5.73 (d, *J*=8.7 Hz, 1H), 5.21 (d, *J*=8.7 Hz, 1H), 2.41 (s, 3H), 2.17 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 203.6, 149.5, 143.7, 137.1, 136.9, 129.7, 128.9, 128.2, 127.2, 126.4, 81.9, 62.7, 30.5, 21.6. HRMS (MNa^+): expected: 477.9944, found: 477.9948.

3.2.2. Table 1, entry 2. Isolated as a white solid. Mp=95–97 °C. FTIR: 3286.5, 1728.5 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.69–7.66 (m, 2H), 7.29–7.22 (m, 5H), 7.17–7.12 (m, 2H), 7.07 (d, *J*=0.6 Hz, 1H), 5.85 (d, *J*=9.3 Hz, 1H), 5.32 (d, *J*=9.3 Hz, 1H), 3.59 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.2, 143.7, 140.6, 137.3, 137.2, 129.7, 128.7, 128.0, 127.1, 126.2, 89.1, 61.9, 51.9, 21.6. HRMS (MNa^+): expected: 493.9893, found: 493.9895.

3.2.3. Table 1, entry 3. Isolated as a white solid. Mp=97–98 °C. FTIR: 3286.7, 1719.5 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.69–7.66 (m, 2H), 7.29–7.22 (m, 5H), 7.18–7.14 (m, 2H), 7.02 (d, *J*=0.9 Hz, 1H), 5.80 (d, *J*=9.3 Hz, 1H), 5.31 (d, *J*=9.3 Hz, 1H), 4.11–4.01 (m, 2H), 2.42 (s, 3H), 1.09 (t, *J*=6.9 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 164.9, 143.7, 140.5, 137.4, 137.3, 129.7, 128.6, 128.0, 127.1, 126.2, 88.7, 62.0, 61.5, 21.6, 13.8. HRMS (MNa^+): expected: 508.0050, found: 508.0053.

3.2.4. Table 1, entry 4. Isolated as a colorless oil. FTIR: 3284.9, 1722.7 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.40–7.28 (m, 6H), 5.70 (d, *J*=9.3 Hz, 1H), 5.52 (d, *J*=9.3 Hz, 1H), 3.72 (s, 3H), 2.87 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.7, 142.6, 137.3, 129.0, 128.4, 126.5, 88.9, 61.9, 52.2, 41.9. HRMS (MNa^+): expected: 417.9580, found: 417.9586.

3.2.5. Table 1, entry 5. Isolated as a white solid. Mp=104–105 °C. FTIR: 3287.0, 1719.8 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.81–7.77 (m, 2H), 7.60–7.45 (m, 3H), 7.11 (d, *J*=0.9 Hz, 1H), 7.07–6.96 (m, 4H), 5.68 (d, *J*=9.0 Hz, 1H), 5.32 (d, *J*=9.0 Hz, 1H), 3.60 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.5, 140.8, 140.2, 138.0, 134.2, 132.8, 129.4, 129.1, 127.1, 126.1, 88.9, 61.8, 51.9, 21.0. HRMS (MNa^+): expected: 498.9893, found: 498.9899.

3.2.6. Table 1, entry 6. Isolated as a colorless oil. FTIR: 3283.5, 1719.6 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.69–7.65 (m, 2H), 7.30–7.26 (m, 2H), 7.07–7.02 (m, 3H), 6.80–6.75 (m, 2H), 5.63 (d, *J*=9.0 Hz, 1H), 5.26 (d, *J*=9.0 Hz, 1H), 3.76 (s, 3H), 3.61 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.5, 159.3, 143.7, 140.9, 137.2, 129.7, 129.3, 127.5, 127.2, 114.1, 88.4, 61.5, 55.2, 51.9, 21.6. HRMS (MNa^+): expected: 523.9999, found: 523.9983.

3.2.7. Table 1, entry 7. Isolated as a colorless oil. FTIR: 3286.0, 1729.4 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.68–7.65 (m, 2H), 7.39–7.31 (m, 5H), 7.26–7.23 (m, 2H), 7.18–7.12 (m, 1H), 7.06 (d, *J*=0.9 Hz, 1H), 6.85–6.81 (m, 1H), 6.77–6.70 (m, 2H), 5.87 (d, *J*=9.3 Hz, 1H), 5.29 (d, *J*=9.3 Hz, 1H), 4.94 (s, 2H), 3.58 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.3, 158.9, 143.7, 140.4, 138.9, 137.2, 136.6, 129.8, 129.7, 128.5, 128.0, 127.5, 127.1, 118.7, 114.5, 112.7, 89.3, 69.9, 61.8, 51.9, 21.5. HRMS (MNa^+): expected: 600.0312, found: 600.0316.

3.2.8. Table 1, entry 8. Isolated as a white solid. Mp=109–110 °C. FTIR: 3285.2, 1721.1 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.68–7.65 (m, 2H), 7.29–7.26 (m, 2H), 7.16–7.10 (m, 2H), 7.06 (d, *J*=0.9 Hz, 1H), 6.96–6.90 (m, 2H), 5.95 (d, *J*=9.3 Hz, 1H), 5.29 (d, *J*=9.3 Hz, 1H), 3.60 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.3, 163.9, 160.6, 143.9, 140.4, 137.0, 133.2, 129.8 (2C), 128.0, 127.1 (2C), 115.7, 115.4, 89.3, 61.4, 51.9, 21.6. ^{19}F NMR (282.3 MHz, $CDCl_3$): −114.3 (m). HRMS (MNa^+): expected: 511.9799, found: 511.9808.

3.2.9. Table 1, entry 9. Isolated as a white solid. Mp=105–106 °C. FTIR: 3282.7, 1728.8 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.80–7.76 (m, 2H), 7.61–7.54 (m, 1H), 7.52–7.45 (m, 2H), 7.23–7.19 (m, 2H), 7.14 (d, *J*=0.9 Hz, 1H), 7.12–7.07 (m, 2H), 6.03 (d, *J*=9.6 Hz, 1H), 5.33 (d, *J*=9.6 Hz, 1H), 3.60 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.2, 140.1, 140.0, 135.8, 134.0, 133.0, 129.2, 128.8, 127.6, 127.0, 89.9, 61.5, 52.0. HRMS (MNa^+): expected: 513.9347, found: 513.9348.

3.2.10. Table 1, entry 10. Isolated as a white solid. Mp=91–92 °C. FTIR: 3280.8, 1722.6 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.69–7.65 (m, 2H), 7.28–7.23 (m, 3H), 7.19 (d, *J*=0.9 Hz, 1H), 6.22 (dd, *J*=2.1 Hz, 3.3 Hz, 1H), 6.12–6.10 (m, 1H), 5.84 (d, *J*=9.3 Hz, 1H), 5.41 (d, *J*=9.3 Hz, 1H), 3.69 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.0, 149.7, 143.7, 142.7, 138.7, 137.1, 129.6, 127.1, 110.6, 107.9, 90.3, 56.4, 52.0, 21.5. HRMS (MNa^+): expected: 483.9686, found: 483.9688.

3.2.11. Table 1, entry 11. Isolated as a white solid. Mp=105–108 °C. FTIR: 3285.7, 1729.2 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): 7.69 (d, *J*=8.4 Hz, 2H), 7.29 (d, *J*=8.1 Hz, 2H), 7.21–7.18 (m, 2H), 6.88–6.85 (m, 1H), 6.77–6.76 (m, 1H), 5.95 (d, *J*=9.6 Hz, 1H), 5.49 (d, *J*=9.6 Hz, 1H), 3.66 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): 165.1, 143.9, 141.6, 139.6, 137.1, 129.8 (2C), 127.2 (2C), 127.1, 125.8, 125.0, 90.5, 58.9, 52.0, 21.6. HRMS (MNa^+): expected: 499.9458, found: 499.9456.

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