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RESEARCH LETTER

Potassium carbonate as a green catalyst for Markovnikov addition of azoles to vinyl acetate in PEG

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A novel strategy to effect the Markovnikov addition between azoles and vinyl-acetate is described. The main attractive features of this process are a mild base catalyst, recyclability of solvent PEG, excellent yields of products, and easy workup procedure.

Keywords: Markovnikov addition; K_2CO_3; PEG; N-heterocycles; recyclability

Introduction

The current environmental concerns encourage the development of more efficient, economical and environmentally friendly reactions for chemical synthesis (*1*). In this context, a great deal of effort has gone into the development of efficient and environmentally acceptable alternatives for the synthesis of N-containing heterocyclic compounds. Markovnikov addition is one of the most useful carbon–carbon, carbon–oxygen, and carbon–nitrogen bond forming reactions. This method has been widely used for the synthesis of biologically active heterocyclic compounds in synthetic organic chemistry (*2–8*).

The N-heterocycle derivatives 1-[N-(N-heterocycle)]alkyl esters obtained are usually Pharmacological active (*9*, *10*) and may be applied as potential therapeutic alternatives like acaricides (*2,3*), antimicrobials (*4*), antitumor drugs (*5*), and (H^+–K^+)-ATPase inhibitors (*6*). However, the above-mentioned methods were generally promoted by harsh bases, strong acids, and high temperature (*11–13*), which would lead to environmentally hazardous residues and low yield of products. Nevertheless, the replacement of these hazardous conditions with the environmentally benign methodologies is one of the key areas of green chemistry.

Recently, K_2CO_3 catalyst in polyethylene glycol (PEG) (*14–17*) has gained recognition as favorable environmentally benign alternatives. Over the past few years, potassium carbonate (K_2CO_3) has been widely used as mild base catalyst in many organic reactions such as monomethylation reactions (*18*), O-alkylation (*19*), synthesis of 2*H*-chromenes (*20*), thiolysis of epoxides (*21*), Knoevenagel and Nitroaldol Condensation (*22*). Using potassium carbonate as green catalyst, our group has successfully carried out the synthesis of substituted 2-amino-4*H*-chromenes (*23*), thieno-pyrimidines (*24*), organomercurials (*25*), fused pyrimido derivatives (*26*), and thiohydantoins (*27*). The various articles reported reactions of K_2CO_3 not only showing its essentiality for a particular reaction but also depicting its other characteristics like solubility in water, mild character (*19*, *28*), easy availability, eco-friendly (*29*, *30*), and nontoxic nature (*18*, *23*). Thus potassium carbonate provides a mild basic medium for organic reactions to occur and get removed by easy separation by water.

During the past few years, the biologically compatible PEGs (*31*) were demonstrated to be a recyclable green reaction medium for various chemical reactions (*32–34*). Here, as an extension of this work, we discovered that K_2CO_3 in PEG could effectively catalyzed the C–N bond formation, namely the Markovnikov addition of N-heterocycles and vinyl acetate (*35–37*) to afford the corresponding N-heterocyclic derivatives in high yield under mild condition (Scheme 1).

In pursuit of our recent efforts on developing green methodologies (*38–47*), we evaluated the feasibility of Markovnikov addition of 4-nitroimidazole to vinyl acetate in different conditions, which was shown in Table 1. Initially, the reaction was carried with 4-nitroimidazole (1 mmol) and vinyl acetate (4 mmol) at room temperature in PEG 400 using K_2CO_3 as

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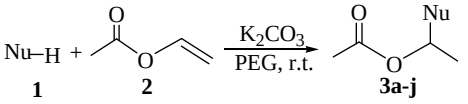


Scheme 1. Synthesis of Markonikov products using K_2CO_3 in PEG.

catalyst for 5 h to evaluate the product. The formation of product was monitored by thin layer chromatography (TLC).

To optimize the reaction conditions, the reaction was performed in a range of solvents such as DMSO, DMF, methanol, and various PEGs with K_2CO_3 in terms of both yield and reaction time (Table 1). We also tried different kind of bases such as L-proline, KOH, and NaOH but K_2CO_3 was found to be more effective (Table 1, entry 2). When the reaction was carried out in the absence of catalyst led to the Markovnikov adduct in very low yield (5%) even after longer reaction time.

Encouraged by these remarkable results, we tried the reaction with the imidazoles containing electron-withdrawing and electron-donating groups, high yield of product was obtained with the imidazole having electron-withdrawing groups. The reactivity decreases by the following order, 4-nitroimidazole, 4-methyl imidazole, and imidazole in agreement with their nucleophilicity (Table 2, entry 1, 6, and 7).

Apart from imidazole derivatives, other N-heterocycles such as pyrrole, benzotriazole, and benzimidazole also exhibited high Markovnikov addition activity.

Among these the benzotriazole reacted fairly faster due to its strong nucleophilicity (Table 2, entry 4).

It is important to stress that the solvent was recycled and reused for 3–4 runs. After completion of the reaction (monitored by TLC), the reaction mixture was extracted with solvent ether, since PEG is immiscible with solvent ether. In PEG phase, ethanol (10 mL) was added and passed through a very short pad of silica gel and activated charcoal. The colorless organic layer was evaporated under reduced pressure. PEG was further dried under high vacuum and used for the next run (Table 3).

Table 1. Effect of solvents and catalysts on Markovnikov addition between azoles and vinyl acetate[a].

Entry	Solvent	Catalyst	Time (h)	Yield (%)[b]
1	PEG 400	Catalyst-free	24	5
2	PEG 400	K_2CO_3	5	98
3	PEG 200	K_2CO_3	7	90
4	DMSO	K_2CO_3	15	72
5	DMF	K_2CO_3	10	85
6	Methanol	K_2CO_3	15	52
7	PEG 400	L-proline	14	20
8	PEG 400	NaOH	14	55
9	PEG 400	KOH	12	70

[a]Reaction conditions: 4-nitroimidazole (1.0 mmol), vinyl acetate (4.0 mmol), K_2CO_3 (0.3 mmol), solvent PEG 400 (3 mL); (room temperature).
[b]Isolated yields.

Experimental section

General

The materials were purchased from Sigma-Aldrich and Merk that used without any purification. All reactions and purity of N-heterocycles were monitored by TLC using aluminum plates coated with silica gel (merk) using hexane-ethylacetate (80:20) as an eluent. The isolated products were further purified by column chromatography using silica gel (100–200 meshes), purchased from RFCL Pvt. Ltd. New Delhi, India. ^{1}H NMR and ^{13}C NMR (300 and 75 MHz, respectively) spectra were recorded on Bruker Avance Spectrospin 300 (300 MHz). All NMR samples were run in $CDCl_3$ and chemical shifts are expressed as δ relative to internal Me_4Si. IR spectra were obtained on Perkin Elmer FT-IR spectrometer spectrum-2000 using potassium bromide pellets or as liquid films.

Typical procedure for the Markovnikov addition:

A mixture of azoles (1 mmol), vinyl acetate (4 mmol), and K_2CO_3 (0.3 mmol) in PEG 400 (3 mL) was stirred at room temperature for the time indicated in Table 2. After completion of the reaction (monitored by TLC), the reaction mixture was diluted with diethyl ether (10 mL) (PEG being insoluble in ether). The organic layer was washed with saturated solution of NaCl (20 mL) and dried over anhydrous sodium sulfate (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was purified by silica gel column chromatography using ethylacetate: hexane (20:80) as an eluent. The structure of products was confirmed by IR, ^{1}HNMR, ^{13}CNMR, and HRMS/mass spectral data. All the products are known compounds.

1-[1-(4-Nitroimidazole)]-ethyl acetate (3a): White solid; melting point 83–85°C; IR (KBr): 3134, 1745, 1546, 1510 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz, δ, ppm): 7.99 (s, 1H), 7.71 (s, 1H), 6.74 (q, 1H, $J = 6.98$Hz), 2.11 (s, 3H), 1.85 (d, 3H, $J = 6.15$Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ ppm): 169.28, 148.74, 135.27, 117.12, 77.52, 20.93, 20.16. HRMS: m/z calculated for $C_7H_9N_3O_4$ [M^+]: 199.0593; found: 199.0386.

Table 2. K_2CO_3 catalyzed Markovnikov addition of azoles with vinyl acetate[a].

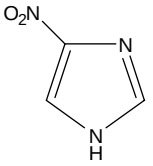
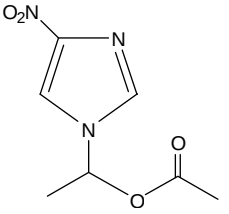
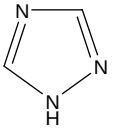
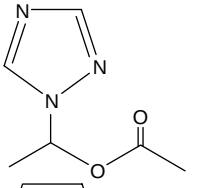
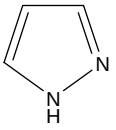
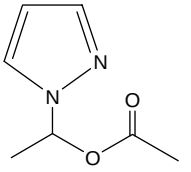
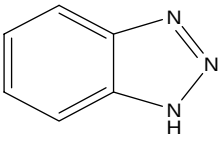
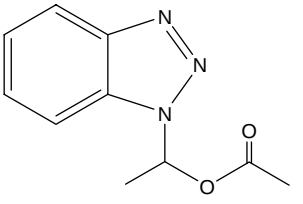
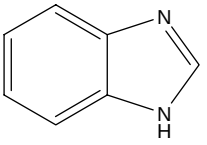
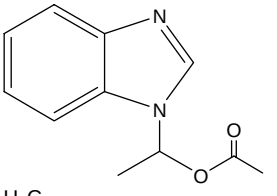
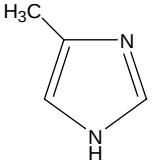
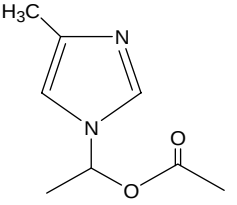
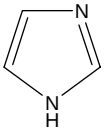
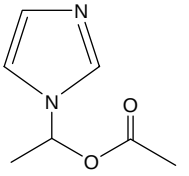
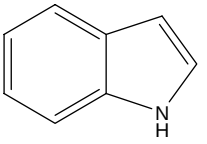
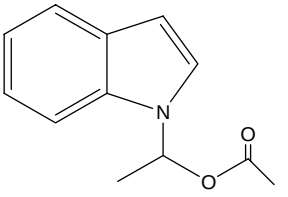
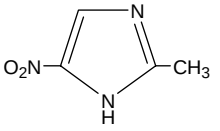
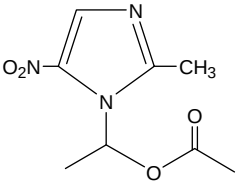
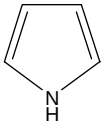
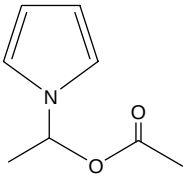
Entry	Nu–H	Products	Time (h)	Yield (%)[b]
1			5	**(3a)** 98
2			8	**(3b)** 96
3			6	**(3c)** 92
4			5	**(3d)** 85
5			7	**(3e)** 81
6			9	**(3f)** 78
7			30	**(3g)** 75
8			6	**(3h)** 82
9			6	**(3i)** 88

Table 2 (*Continued*)

Entry	Nu–H	Products	Time (h)	Yield (%)[b]
10			10	**(3j)** 80

[a]Reaction conditions: azoles (1.0 mmol), vinyl acetate (4.0 mmol), K_2CO_3 (0.3 mmol), solvent PEG 400 (3 mL); (room temperature).
[b]Isolated yields.

1-(1-Triazole)-ethyl acetate (3b): Yellowish oil. IR (film): 3120, 3005, 1750, 1490, cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 8.31 (s, 1H), 7.93(s, 1H), 6.84 (q, 1H, $J=6.28$ Hz), 2.10 (s, 3H), 1.87 (d, 3H, $J=6.21$Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 169.39, 150.91, 145.02, 76.6, 20.95, 19.22. HRMS: *m/z* calculated for $C_6H_9N_3O_2$ [M^+]: 155.0695 found 156.1031.

1-(1-Pyrazole)-ethyl acetate (3c): Colorless oil. IR (film): 3125, 3000, 1748, 1495 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 7.90(s, 1H), 7.52 (s, 1H,), 6.77 (q, 1H, $J=6.23$Hz), 6.28 (t, 1H), 2.04 (s, 3H), 1.72 (d, 3H, $J=6.25$Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 169.63, 141.03, 130.58, 106.28, 79.42, 21.23, 19.62. HRMS: *m/z* calculated for $C_7H_{10}N_2O_2$ [M^+]: 154.0742 found 154.0634.

1-(1-Benzotriazolyl)-ethyl acetate (3d): Colorless oil. IR (film): 3130, 2998, 1752, 1501 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 8.01 (d, 1H, $J=8.23$Hz). 7.77 (d, 1H, $J=8.3$), 7.50 (m, 1H), 7.43 (m, 2H), 2.09 (d, 3H, $J=6.4$Hz), 2.04 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 168.20, 130.63, 124.48, 117.56, 91.71, 19.12, 15.84. HRMS: *m/z* calculated for $C_{10}H_{11}N_3O_2$ [M^+]: 205.0851 found 205.0764.

1-(1-Benzimidazolyl)-ethyl acetate (3e): Colorless oil. IR (film): 3095, 1744, 1640, 1490 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 8.07 (s, 1H), 7.83 (m, 1H), 7.57 (m, 1H), 7.30 (m, 2H), 6.97 (q, 1H, $J=6.35$Hz), 2.02 (s, 3H), 1.91 (d, 3H, $J=6.41$Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 168.72, 140.21, 139.46, 132.35, 120.17, 108.56, 20.28, 16.90. HRMS: *m/z* calculated for $C_{11}H_{12}N_2O_2$ [M^+]: 204.0899 found 204.1476.

Table 3. Recycling of PEG 400[a].

No. of cycles[a]	Fresh	Run 1	Run 2	Run 3	Run 4
Yield (%)[b]	98	98	97	96	93
Time (h)	5	5	5	5	5

[a]Reaction conditions: 4-nitroimidazole (1.0 mmol), vinyl acetate (4.0 mmol), K_2CO_3 (0.3 mmol), solvent PEG 400; (room temperature).
[b]Isolated yields.

1-(1-4-Methylimidazole)-ethyl acetate (3f): Light yellowish oil. IR (film): 3095, 1744, 1640, 1490 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 7.69 (s, 1H), 7.10 (s, 1H), 6.61 (q, 1*H*, $J=6.21$ Hz), 2.04 (s, 3*H*), 2.01 (s, 3*H*), 1.63 (d, 3*H*, $J=6.12$ Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 169.98, 136.91, 136.12, 114.01, 76.25, 21.35, 20.60, 14.21. HRMS: *m/z* calculated for $C_8H_{12}N_2O_2$ [M^+]: 168.0899 found 168.0301.

1-(1-Imidazole)-ethyl acetate (3g): Yellow oil. IR (film): 3116, 1743, 1493 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 7.79 (s, 1*H*), 7.23 (d, 1*H*, $J=5.11$ Hz), 7.12 (d, 1*H*, $J=6.73$ Hz,), 6.67 (q, 1*H*, $J=6.45$ Hz), 2.48 (s, 3*H*), 1.75 (d, 3*H*, $J=6.51$ Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 169.78, 135.01, 128.31, 117.62, 76.20, 21.06, 20.17. HRMS: *m/z* calculated for $C_7H_{10}N_2O_2$ [M^+]: 154.0742 found 155.0112.

1-(1-Indole)-ethyl acetate (3h): Colorless oil. IR (film): 3110, 1742, 1570, 1498 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 7.68 (m, 1*H*), 7.51 (m, 1*H*), 7.46 (m, 2*H*), 7.18 (d, 1H, $J=6.28$ Hz), 6.78 (d, 1H, $J=6.30$ Hz), 6.52 (q, 1*H*, $J=6.32$ Hz), 2.03(s, 3*H*), 1.73 (d, 3*H*, $J=6.42$ Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 163.32, 131.21, 126.73, 124.15, 118.63, 107.24, 85.33, 18.72, 15.25. HRMS: *m/z* calculated for [M^+]: $C_{12}H_{13}NO_2$ 203.0946 found 203.0472.

1-(1-2-Methyl-5-nitroimidazole)-ethyl acetate (3i): White solid. melting point 137–138°C. IR (KBr): 3138, 1753, 1540, 1508 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 7.91 (s, 1*H*), 6.68 (q, 1*H*, $J=6.32$ Hz), 2.49 (s, 3*H*), 2.10 (s, 3*H*,), 1.73 (d, 3*H*, $J=6.33$ Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 169.61, 148.57,

147.01, 119.82, 76.39, 21.12, 20.49, 13.48. HRMS: m/z calculated for $C_8H_{11}N_3O_4$ [M^+]: 213.0750 found 213.0295.

1-(1-Pyrrole)-ethyl acetate (3j): Colorless oil. IR (film): 3110, 1742, 1570, 1498 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz, δ, ppm): 6.72 (d, 2*H*, $J = 2.5$ Hz), 6.29 (q, 1*H*, $J = 6.23$ Hz), 6.11 (d, 2*H*, $J = 6.35$ Hz), 2.08 (s, 3*H*), 1.69 (d, 3*H*, $J = 6.25$ Hz). ^{13}C NMR ($CDCl_3$, 75 MHz, δ, ppm): 169.71, 121.32, 120.08, 110.19, 108.25, 79.11, 21.15, 19.12. HRMS: m/z calculated for $C_8H_{11}NO_2$ [M^+]: 153.0790 found 153.2031.

Conclusion

In conclusion, we have developed a novel and efficient protocol for the Markovnikov addition using K_2CO_3 as catalyst in PEG 400 under mild reaction conditions with excellent yields. This strategy is quite general and it works with a broad range of azoles. Furthermore, this method exhibits a simple and green procedure to avoid the use of toxic solvents and catalysts. The ease of mildness of this method provides an attractive route to the synthesis of N-heterocycle derivatives which may use as pharmacological alternatives.

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