Preparation and Allylation of Enamides and Enecarbamates Generated via Iron(0) Reduction of Oximes and Derivatives

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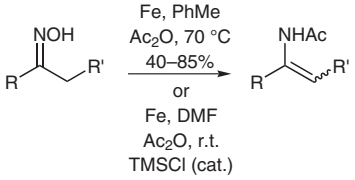
Dedicated for the 65th Birthday of Marty Semmelhack

Abstract: Reductive acylation of oximes and oxime carbonates can be effected with iron powder in the presence of an acid chloride or a chloroformate to produce enamides or enecarbamates.

Key words: acylations, *N*-acyliminium ions, addition reactions, allylations, reductions



Scheme 1

Enamides have been widely used as intermediates in organic synthesis. For example, enamides are valuable precursors for generation of *N*-acyliminium ions,[1,2] and have also been utilized recently in asymmetric hydrogenation reactions to prepare *N*-acyl derivatives of chiral amines.[3] Enamides have been synthesized by a variety of methods, including the Curtius rearrangment,[4] by direct addition of amides to alkynes,[5] and by transition-metal-promoted coupling of vinyl halides with amides.[6] Another common method for the preparation of enamides is simply to N-acylate an imine with an acid halide or an acid anhydride.[7] However, this methodology is only practicable for synthesis of tertiary enamides derived from *N*-alkyl- or *N*-aryl-substituted imines due to the difficulty in condensing ammonia with aldehydes or ketones to form simple unsubstituted imines.[8]

In 1975 Barton et al. demonstrated that ketoximes, as well as their *O*-acyl and *O*-alkyl derivatives, could be converted into secondary acetyl enamides in excellent yields by heating at reflux with acetic anhydride in pyridine.[9] A free radical mechanism was postulated for this transformation. In addition, it was found that treatment of ketoximes with acetic anhydride in the presence of stoichiometric amounts of metals such as Cr(II) and Ti(III) also affords the corresponding enamides. More recently, Burk and coworkers reported that acetyl enamides can be prepared in moderate to good yields from ketoximes by heating at 70 °C in toluene–acetic anhydride in the presence of inexpensive iron powder (Scheme 1).[10] In an improvement of this methodology, Zhang and Casalnuovo found that these reactions can be effected at room temperature if *N*,*N*-dimethylformamide is used as solvent, and also that the reaction is initiated by addition of a catalytic amount of chlorotrimethylsilane.[3g] It might be noted that to date the methodology has only been applied to ketoximes derived from aryl or hindered ketones.[3g,10]

As part of some ongoing projects which involve the use of enamides and enecarbamates as *N*-acyliminium ion precursors,[2] we became interested in possibly extending the scope of this methodology to acylating agents other than acetic anhydride. Moreover, since acetamides can be difficult to cleave, especially in the presence of sensitive functionality, we considered the possibility of effecting this transformation to enable generation of enecarbamates bearing more readily removable acyl protecting groups. In this paper are described some of our studies in this area.

Initial exploratory experiments were conducted with α-tetralone oxime (**1**; Scheme 2). It was found that treatment of **1** with iron powder and benzoyl chloride (2 equiv) in tetrahydrofuran at room temperature led to the desired enamide **2** in good yield. Similarly, with ethyl chloroformate and benzyl chloroformate the enecarbamates **3** and **4**, respectively, were generated. Since aldehyde oximes had not previously been used in this type of reductive acylation, aldoxime **5** was treated under the same conditions with ethyl chloroformate and benzyl chloroformate to produce enecarbamates **6** and **7**, respectively, in moderate isolated yields. We believe some loss of product is probably occurring here via enecarbamate hydrolysis on chromatography (vide infra).

We also investigated applying this methodology to ketoxime systems, which would produce non-conjugated enamide and enecarbamate products. For example, when 4-phenylcyclohexanone oxime (**8**) was subjected to the standard reduction conditions using benzoyl chloride, the enamide **10** was produced in only low irreproducible isolated yields (19–48%; Scheme 3). We speculated that the problem here might be due the HCl generated in the process, which may accelerate the in situ hydrolysis of the acid-sensitive product **10** by adventitious moisture.

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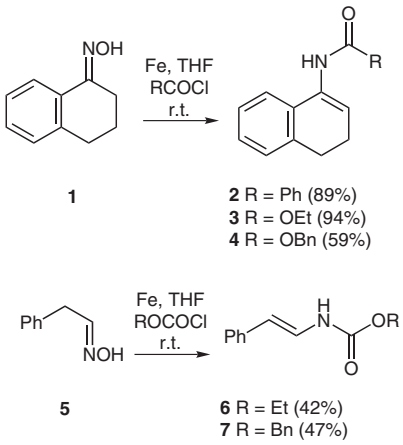
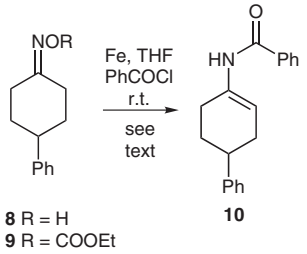


Scheme 2

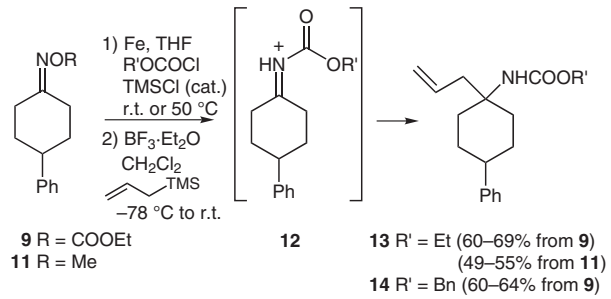


Scheme 3

Therefore, in an attempt to alleviate this difficulty, oxime **8** was first converted to oxime carbonate **9** with ethyl chloroformate. Reaction of carbonate **9** with benzoyl chloride and iron powder using a catalytic amount of TMSCl as initiator[3g] gave enamide **10** in a more reproducible 45% yield. Unfortunately, none of the corresponding enecarbamate could be isolated from the reductive acylation of **9** with ethyl chloroformate. During the course of these experiments, however, it became clear to us that the main problem is the general tendency of non-conjugated secondary (NH) enamides like **10** to hydrolyze upon chromatography to the corresponding ketone. It should also be noted that in our experience tertiary (*N*-alkyl and *N*-aryl) enamides generated from aliphatic ketones have been found to be significantly more resistant to hydrolysis, and are amenable to chromatographic purification.[2]

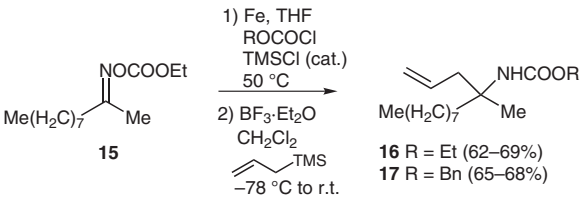
In order to make use of the non-conjugated ketone-derived enamide systems generated by this methodology it became apparent that chromatography should be avoided. Thus, we explored effecting direct alkylations of the *N*-acyliminium ions generated from the crude enamides. Treatment of oxime carbonate **9** with iron powder and ethyl chloroformate at room temperature in tetrahydrofuran containing a catalytic amount of TMSCl produced the corresponding enecarbamate. After a brief aqueous workup, the product was immediately exposed to allyltrimethylsilane–boron trifluoride etherate in dichloromethane (Scheme 4). The latter operation leads to *N*-acyliminium ion **12**, which undergoes alkylation to afford product **13** in 60–69% isolated overall yields for the two steps. Similarly, carbonate **9** could be converted to Cbz-protected allylation product **14** in 60–64% yields using benzyl chloroformate.

We have also examined the possibility of using oxime methyl ethers in this process. The reaction of methoxime **11** with iron powder/ethyl chloroformate at room temperature as done with **9** proved to be sluggish, and heating at 50 °C was therefore required. Allylation of the crude enecarbamate product then afforded ethyl carbamate **13** in somewhat lower yields than those obtained with the oxime carbonate **9** (49–55% for two steps).



Scheme 4

Finally, an acyclic ketoxime system was also examined in this two-step operation. Oxime carbonate **15** derived from 2-decanone was found to undergo reductive acylation with ethyl chloroformate under the standard conditions, although in this case it was advantageous to conduct the reaction at 50 °C (Scheme 5). Allylation of the crude enecarbamate afforded **16** in 62–69% overall isolated yields for the two steps. The transformation with **15** could also be carried out with benzyl chloroformate to generate the Cbz-substituted allylic system **17** in 65–68% yields.



Scheme 5

In conclusion, we have developed reductive acylation methodology, which allows one to convert readily available oximes and oxime carbonates to a variety of enamides and enecarbamates using inexpensive reagents. Isolation of products in the case of conjugated systems is feasible by chromatography. However, with non-conjugated compounds it is preferable to simply use the crude material for further transformations.

Preparation of Oxime Ethyl Carbonates; General Procedure
To a solution of the oxime (3.17 mmol) in THF (10 mL) were added TEA (2.21 mL, 15.9 mmol) and ethyl chloroformate (6.34 mmol) at 0 °C. The reaction mixture was stirred at 0 °C until TLC showed no starting material remained. The mixture was then diluted with water and the aqueous layer was extracted with CH_2Cl_2. The extract was dried over $MgSO_4$ and evaporated. The residue was purified by silica gel flash column chromatography to provide the O-acylated oxime.

4-Phenylhexanone Oxime Ethyl Carbonate (9)
Yield: 96%; white solid; mp 43–44 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.11–7.26 (m, 5 H), 4.27 (q, *J* = 7.0 Hz, 2 H), 3.31–3.37 (m, 1 H), 2.68–2.78 (m, 2 H), 2.26 (dt, *J* = 4.9, 13.7 Hz, 1 H), 1.92–2.12 (m, 3 H), 1.57–1.73 (m, 2 H), 1.29 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (90 MHz, $CDCl_3$): δ = 167.0, 154.1, 144.8, 128.4, 126.5, 64.4, 53.3, 43.1, 33.6, 32.8, 31.6, 26.3, 14.2.

HRMS (ESI+): *m*/*z* [MH^+] calcd for $C_{15}H_{20}NO_3$: 262.1443; found: 262.1472.

2-Decanone Oxime Ethyl Carbonate (15)
Yield: 85%; light yellow oil (mixture of geometric isomers).

Major Geometric Isomer
^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.23 (q, *J* = 7.1 Hz, 2 H), 2.26 (t, *J* = 7.3 Hz, 2 H), 1.92 (s, 3 H), 1.49 (m, 2 H), 1.20–1.31 (m, 13 H), 0.81 (t, *J* = 6.4 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 166.3, 153.9, 64.263, 35.5, 31.7, 29.1, 29.0, 28.99, 26.1, 22.5, 14.9, 14.2, 13.9.

Minor Geometric Isomer
^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.24 (q, *J* = 7.1 Hz, 2 H), 2.36 (t, *J* = 8.0 Hz, 2 H), 1.96 (s, 3 H), 1.41–1.49 (m, 2 H), 1.22–1.32 (m, 13 H), 0.83 (t, *J* = 6.9 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 166.8, 154.0, 64.3, 31.7, 30.1, 29.3, 29.1, 29.0, 25.6, 22.5, 19.8, 14.2, 14.0.

HRMS (ESI+): *m*/*z* [MH^+] calcd for $C_{13}H_{26}NO_3$: 244.1913; found: 244.1907.

4-Phenylcyclohexanone *O*-Methyloxime (11)
To a solution of 4-phenylcyclohexanone (4.00 g, 22.96 mmol) in EtOH (60 mL) were added pyridine (2.80 mL, 34.34 mmol) and methoxylamine hydrochloride (2.30 g, 27.54 mmol) at r.t. The reaction mixture was stirred for 1 h until TLC showed no starting material remained. Sat. NH_4Cl was then added and the aqueous phase was extracted with CH_2Cl_2. The extract was dried over $MgSO_4$ and evaporated. The residue was purified by flash column chromatography (25–50% EtOAc–hexanes) to provide the product **11** (4.48 g, 96%) as a colorless oil.

^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.34–7.38 (m, 2 H), 7.25– 7.28 (m, 3 H), 3.93 (s, 3 H), 3.43–3.48 (m, 1 H), 2.81 (tt, *J* = 3.0, 12.1 Hz, 1 H), 2.58–2.62 (m, 1 H), 2.29 (dt, *J* = 4.7, 13.6 Hz, 1 H), 2.05–2.15 (m, 2 H), 1.91 (dt, *J* = 5.1, 13.8 Hz, 1 H), 1.61–1.82 (m, 2 H).

^{13}C NMR (90 MHz, $CDCl_3$): δ = 158.5, 145.5, 128.3, 126.5, 126.1, 60.8, 43.5, 33.9, 32.8, 31.7, 24.6.

HRMS (APCI+): *m*/*z* [MH^+] calcd for $C_{13}H_{18}NO$: 204.1388; found: 204.1369.

Preparation of Enamides and Enecarbamates; General Procedure
To a solution of oxime (0.62 mmol) in THF (3 mL) were added iron powder (173 mg, 3.10 mmol) and ethyl chloroformate (0.49 mL, 4.96 mmol), or benzoyl chloride (0.58 mL, 4.96 mmol), or benzyl chloroformate (0.71 mL, 4.96 mmol) at r.t. After stirring overnight, the reaction mixture was diluted with sat. $NaHCO_3$ solution and subsequently extracted with Et_2O. The combined organic layers were dried over $MgSO_4$ and concentrated in vacuo. Basic alumina (II–III) chromatography (hexanes–EtOAc) of the residue afforded the enecarbamate or enamide product.

Compounds **6** and **7** have been previously prepared.[11]

***N*-(3,4-Dihydronaphthalen-1-yl)benzamide (2)**
Yield: 89%; white solid; mp 147–148 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.89–7.91 (m, 2 H), 7.50–7.60 (m, 4 H), 7.21–7.24 (m, 4 H), 6.66 (br s, 1 H), 2.85 (t, *J* = 7.6 Hz, 2 H), 2.46 (dt, *J* = 5.3, 7.6 Hz, 2 H).

^{13}C NMR (90 MHz, CD_2Cl_2): δ = 166.7, 137.3, 135.3, 132.4, 132.1, 132.0, 129.0, 128.2, 127.9, 127.4, 126.8, 121.3, 120.6, 28.0, 22.7.

HRMS (APCI+): *m*/*z* [MH^+] calcd for $C_{17}H_{16}NO$: 250.1231; found: 250.1242.

(3,4-Dihydronaphthalen-1-yl)carbamic Acid Ethyl Ester (3)
Yield: 94%; white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.17–7.22 (m, 4 H), 6.33 (br s, 1 H), 6.12 (br s, 1 H), 4.21 (q, *J* = 7.1 Hz, 2 H), 2.78 (t, *J* = 7.6 Hz, 2 H), 2.35–2.40 (m, 2 H), 1.31 (t, *J* = 7.1 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 154.7, 136.9, 131.6, 131.3, 127.8, 127.5, 126.4, 120.4, 116.3, 61.1, 27.7, 22.1, 14.5.

HRMS (APCI+): *m*/*z* [MH^+] calcd for $C_{13}H_{16}NO_2$: 218.1181; found: 218.1181.

(3,4-Dihydronaphthalen-1-yl)carbamic Acid Benzyl Ester (4)
Yield: 59%; white solid.

^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.35–7.41 (m, 5 H), 7.16–7.22 (m, 4 H), 6.36 (br s, 1 H), 6.28 (br s, 1 H), 5.20 (s, 2 H), 2.79 (t, *J* = 7.6 Hz, 2 H), 2.26 (dt, *J* = 5.1, 7.6 Hz, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 154.4, 136.8, 136.2, 131.5, 131.3, 128.5, 128.2, 127.8, 127.5, 126.4, 120.4, 116.5, 66.9, 27.7, 22.1.

HRMS (ESI+): *m*/*z* [MH^+] calcd for $C_{18}H_{18}NO_2$: 280.1338; found: 280.1358.

Styrylcarbamic Acid Ethyl Ester (6)
Yield: 42%; white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.11–7.38 (m, 6 H), 6.67 (d, *J* = 10.8 Hz, 1 H), 5.92 (d, *J* = 14.5 Hz, 1 H), 4.17 (q, *J* = 6.7 Hz, 2 H), 1.17 (t, *J* = 6.4 Hz, 3 H).

Styrylcarbamic Acid Benzyl Ester (7)
Yield: 47%; white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.14–7.37 (m, 11 H), 6.74 (d, *J* = 10.8 Hz, 1 H), 5.96 (d, *J* = 14.6 Hz, 1 H), 5.18 (s, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 153.5, 136.1, 135.8, 128.6, 128.4, 128.2, 126.3, 125.3, 123.9, 110.9, 67.4.

Addition of Allyltrimethylsilane to Enamides; General Procedure
To a solution of *O*-acyloxime (0.30 mmol) in THF (3 mL) were added iron powder (51 mg, 0.91 mmol) and ethyl chloroformate (344 μL, 3.58 mmol) or benzyl chloroformate (0.514 mL, 3.61mmol). One drop of TMSCl was then added to the reaction mixture, which was stirred (at r.t. for **9** or at 50 °C for **11** and **15**) until all the starting material was consumed. The mixture was diluted with sat. $NaHCO_3$ solution and was extracted with Et_2O. The combined organic layers were dried over Na_2SO_4, and concentrated. The residue was dissolved in CH_2Cl_2 (3 mL) and cooled to –78 °C. Allyltrimethylsilane

(190 μL, 1.19 mmol) and $BF_3{\cdot}Et_2O$ (36 μL, 0.286 mmol) were added to the solution. After stirring at –78 °C for 5 min and at r.t. overnight, the mixture was diluted with sat. NH_4Cl solution and the aqueous phase was extracted with CH_2Cl_2. The organic extract was dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by silica gel flash column chromatography to provide the adduct.

(1-Allyl-4-phenylcyclohexyl)carbamic Acid Ethyl Ester (13)
Yield: 60–69% for two steps from **9**; pale yellow oil.

^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.20–7.33 (m, 5 H), 5.79–5.86 (m, 1 H), 5.06–5.12 (m, 2 H), 4.45 (br s, 1 H), 4.12 (q, *J* = 6.9 Hz, 2 H), 2.47–2.53 (m, 3 H), 2.25 (br d, *J* = 13.2 Hz, 2 H), 1.63–1.80 (m, 4 H), 1.43 (dt, *J* = 3.6, 13.6 Hz, 2 H), 1.27 (t, *J* = 7.1 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 154.9, 146.7, 133.7, 128.3, 126.7, 126.0, 118.1, 60.1, 53.9, 43.9, 43.7, 34.8, 29.0, 14.6.

HRMS (APCI+): *m*/*z* [MH^+] calcd for $C_{18}H_{26}NO_2$: 288.1964; found: 288.1953.

(1-Allyl-4-phenylcyclohexyl)carbamic Acid Benzyl Ester (14)
Yield: 60–64% for two steps; white solid; mp 57–58 °C.

^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.22–7.41 (m, 10 H), 5.79–5.87 (m, 1 H), 5.06–5.17 (m, 4 H), 4.61 (br s, 1 H), 2.49–2.55 (m, 3 H), 2.28 (d, *J* = 13.2 Hz, 2 H), 1.60–1.80 (m, 4 H), 1.45 (dt, *J* = 3.4, 13.6 Hz, 2 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 154.6, 146.6, 136.7, 133.5, 128.5, 128.3, 128.0, 127.99, 126.7, 126.1, 118.3, 66.1, 54.1, 44.0, 43.7, 34.8, 29.0.

HRMS (APCI+): *m*/*z* [MH^+] calcd for $C_{23}H_{28}NO_2$: 305.2120; found: 305.2124.

(1-Methyl-1-octylbut-3-enyl)carbamic Acid Ethyl Ester (16)
Yield: 62–69% for two steps; pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 5.66–5.80 (m, 1 H), 5.01–5.07 (m, 2 H), 4.46 (br s, 1 H), 4.01 (q, *J* = 7.0 Hz, 2 H), 2.24–2.45 (m, 2 H), 1.45–1.68 (m, 2 H), 1.15–1.28 (m, 18 H), 0.84 (t, *J* = 6.9 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 154.8, 133.9, 118.2, 60.0, 54.7, 43.0, 38.7, 31.8, 29.9, 29.5, 29.2, 24.4, 23.5, 22.6, 14.6, 14.1.

HRMS (ESI+): *m*/*z* [MH^+] calcd for $C_{16}H_{32}NO_2$: 270.2433; found: 270.2408.

(1-Methyl-1-octylbut-3-enyl)carbamic Acid Benzyl Ester (17)
Yield: 65–68% for two steps; pale yellow oil.

^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.27–7.36 (m, 5 H), 5.68–5.78 (m, 1 H), 5.03–5.07 (m, 4 H), 4.58 (br s, 1 H), 2.28–2.46 (m, 2 H), 1.50–1.68 (m, 2 H), 1.19–1.30 (m, 15 H), 0.87 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 154.5, 136.8, 133.7, 128.4, 128.0, 127.9, 118.4, 65.9, 54.9, 43.0, 38.7, 31.8, 29.9, 29.5, 29.2, 24.3, 23.5, 22.6, 14.1.

HRMS (APCI+): *m*/*z* [MH^+] calcd for $C_{21}H_{34}NO_2$: 332.2590; found: 332.2596.

Acknowledgment

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