ONE-POT ESTERIFICATION AND AMIDE FORMATION VIA ACID-CATALYZED DEHYDRATION AND RITTER REACTIONS

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GRAPHICAL ABSTRACT

Abstract Esterification of carboxylic acid is achieved using acetonitrile as a water trap. Water liberated during esterification is consumed in cyanide hydrolysis, thereby driving the esterification to completion. Substrates having carboxylic acid and nitrile groups undergo intramolecular dehydration and rehydration to amido esters in the absence of acetonitrile. Cyano acids also undergo esterification and Ritter reaction in one pot when excess alcohol is used. For the first time, we have observed an interesting Ritter reaction of primary alcohols, leading to ester amide product in one pot.

[Supplementary materials are available for this article. Go to the publisher’s online edition of Synthetic Communications1 for the following free supplemental resource(s): Full experimental and spectral details.]

Keywords Amide; dehydration; esterification; hydrolysis; rehydration

INTRODUCTION

Esterification and Ritter reactions are important and useful synthetic transformations in both industry and academic research.[1] Many esters and amides are important intermediates in various industries such as pharmaceuticals, fine chemicals, aroma chemicals, plasticizers, and food.[2] There is always a need for new scientific methods to

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improve the yield of esters and amides. Conventionally esters are synthesized by refluxing the carboxylic acids with alcohol in the presence of a catalytic amount of acid.[3] However, there are several methods for the esterification under neutral, acidic, and alkaline conditions in the literature. Reagents such as diazomethane,[4] trimethylsilyl diazomethane,[5] methyl triazine,[6] and dialkyl formamidemethyldiacetal[7] are known to convert carboxylic acids to esters under neutral conditions. Besides these reagents, catalysts such as polymer-bonded dimethylamino-pyridines also used for facile esterification by N,N’-dicyclohexylcarbodiimide (DCC).[8] Ion exchange resin–facilitated esterification has also been reported.[9] Haworth methylation, which employs dimethylsulfate (DMS) and LiOH in tetrahydrofuran (THF),[10] proceeds well to furnish almost quantitative yield of esters without any side reactions. Under alkaline conditions, strong electrophiles such as alkyl iodides[11] or the corresponding mesylates or tosylates[12–15] are used for the esterification. These hard electrophiles such as methyl iodide and alkyl sulfonates are carcinogens and genotoxic, and dimethyl sulfate is extremely hazardous.[16–18] Salts such as Appel’s salt ((4,3-dichloro-1,2,3-dithiazolium chloride) and 2,6-lutidine are documented in literature for esterification under milder conditions.[19] Graphite bisulfate has been reported to play a dual role as acid catalyst and dehydrating agent.[20] Phosphonium anhydrides are also reported for esterifications.[21] Cyclic imides such as O-methyl caprolactam,[22] isourea ethers,[23] modified thiaynes, and S-propargyl xanthates[24] are a few more reagents that are used successfully for the esterification. Further, asymmetric esterification by organo catalysts[25] and enzymes for optically active esters was also reported in the literature.[26] Transesterification mediated by 1, 3-disubstituted tetraalkyldistannoxanes is also documented.[27] Ritter reactions provide an easy access for the synthesis of amides. This reaction is complementary to esterification. Here water is one of the substrates, whereas in esterification water is liberated as a side product. This reaction is normally carried out with alkyl/aryl cyanides and tert-alcohol/ester or an activated secondary alcohols in the presence of acid catalyst.[28] There is a recent review on the Ritter reaction that describes the importance of the Ritter reaction and the catalysts that bring about this transformation.[29a] Some of the classical methods involve catalysts like H2SO4,[29b] phosphotungstic acid (PTA, H3PW12O40),[30,31] Lewis acid, boron trifluoride,[32] and cobalt(II).[33] There are many alternative acidic reagents such as MeSO3H/Al2O3,[34] (CF3SO2)3,[35] and zeolites.[36] Many modified methods include Ph2C+ClSbCl6,[37] tertiary esters,[38] and trimethylsilyl cyanides.[39] Recently we have published an interesting one-pot reaction of esterification and Ritter reaction using tert-butyl methyl ether (TBME) as solvent and reagent.[40] To the best of our knowledge, there are no reports on the Ritter reaction of an unactivated primary alcohols so far.

RESULTS AND DISCUSSION

Acid-catalyzed esterification is equilibrium controlled, therefore a large excess of alcohol is used and the reaction is not quantitative. If water is removed as azeotrope, then this equilibrium can be shifted toward product formation. We decided to see whether if water is removed from the reaction by the way of Ritter reaction, is it possible to do two reactions in one pot? If so, this would also drive the esterification
to completion, leading to dehydration and rehydration reaction in one pot. To achieve this, easily available acetonitrile was chosen as the dehydrating agent for the dehydration and rehydration reaction. When benzoic acid 1a is reacted with 1.1 equivalent of butyl alcohol (Table 1, entry 1) and acetonitrile (2 eq.) in the presence of 20% of concentrated sulfuric acid, butyl benzoate 1b is formed in 68% yield. Yield improved to 98% when 1.16 equivalent of sulfuric acid is used (Table 1, entry 5). Interestingly, when compared with the normal method of esterification, the presence of acetonitrile in reaction gives quantitative conversion of acid to ester (Table 1, entry 6). It is interesting to see that excess alcohol is not required for the completion of the reaction like in the conventional method. In the optimized conditions, several substrates were converted to their corresponding esters in almost quantitative yield (Table 2, entries 1–23). Many functional groups such as olefinic (5a), amido (6a), tert-hydroxyl (benzillic acid) (7a), halo (8a), and nitro (9a) esters are not affected during the reaction condition. Tryptophan gives a moderate yield of the corresponding ester (Table 2, entry 13). This is probably because of the strong acidic condition during the reaction. Even anhydrides are converted to corresponding esters under similar reaction conditions (Table 2, entry 14). Products 1b–21b (Table 2) are all for intermolecular dehydration and rehydration where acid is converted to ester and the acetonitrile group is converted to amide. When alcohol quantity is increased to about 2 equivalents, we obtained ester amide as the major product (entries 22b–23b) in the absence of acetonitrile. When alcohol quantity is increased to about 4 equivalents for cyano substrates, we observed an interesting esterification and Ritter reaction product. When n-butyl alcohol is reacted with cyanoacetic acid, it gave interesting n-butyl ester and 2-aminobutane Ritter product in moderate yield (entry 24, Table 3). To the best of our knowledge and available literature, this is the first example of Ritter reaction of an unactivated primary alcohol. When a secondary alcohol like isopropanol or cyclohexanol is used, it gave cyclohexyl ester and amide product (entries 25, 27, Table 3).

Table 1. Optimization of esterification of benzoic acid

| Entry | H$_2$SO$_4$ (mmol) | Temp. (°C) | Time (h) | Yield (%)$^b$ |
|-------|-------------------|------------|----------|----------------|
| 1     | 18                | 80–85°C    | 18       | 68             |
| 2     | 38                | 80–85°C    | 18       | 75             |
| 3     | 57                | 80–85°C    | 18       | 79             |
| 4     | 82                | 80–85°C    | 18       | 89             |
| 5     | 94                | 80–85°C    | 18       | 98             |
| 6     | 94                | 80–85°C    | 18       | 58$^c$         |

$^a$Benzoic acid (81 mmol), alcohol (90 mmol), and acetonitrile (162 mmol) are used for the experiments.

$^b$Isolated yield. All reactions were carried out for 16–18 h.

$^c$In the absence of acetonitrile.
Table 2. Esterification of carboxylic acids

| Product | % yield | Product | % yield | Product | % yield |
|---------|---------|---------|---------|---------|---------|
| ![Image 1b](88x87 to 117x329) | 98%     | ![Image 2b](146x278 to 185x338) | 97%     | ![Image 3b](187x474 to 227x533) | 99%     |
| ![Image 4b](187x87 to 227x130) | 98%     | ![Image 5b](187x278 to 227x363) | 98%     | ![Image 6b](187x474 to 238x533) | 98%     |
| ![Image 7b](240x474 to 292x556) | 99%     | ![Image 8b](240x87 to 310x139) | 98%     | ![Image 9b](312x87 to 352x175) | 98%     |
| ![Image 10b](312x278 to 357x380) | 97%     | ![Image 11b](312x474 to 361x542) | 98%     | ![Image 12b](363x87 to 414x184) | 98%     |
| ![Image 13b](363x278 to 423x346) | 60%     | ![Image 14b](363x474 to 409x585) | 99%     | ![Image 15b](396x433 to 442x602) | 98%     |

(Continued)
Table 2. Continued

| Product | % yield | Product | % yield | Product | % yield |
|---------|---------|---------|---------|---------|---------|
| ![Product 16b](image1) | 95%c | ![Product 17b](image2) | 95%c | ![Product 18b](image3) | 90%c |
| ![Product 19b](image4) | 85%c | ![Product 20b](image5) | 95%c | ![Product 21b](image6) | 95%c |
| ![Product 22b](image7) | 85%d | ![Product 23b](image8) | 80%d |

*a* All the reactions were carried with 80 mmol of substrate, 90 mmol of alcohol, and 162 mmol of acetonitrile with 94 mmol of sulfuric acid (98%) and yield referred to isolated yield.

*b* Alcohol (180 mmol) used for the experiments.

*c* 80 mmol of substrate, 190 mmol of alcohol, and 162 mmol of acetonitrile with 94 mmol of sulfuric acid (98%).

*d* 80 mmol of substrate, 190 mmol of alcohol with 94 mmol of sulfuric acid (98%) and yield referred to isolated yield.
Table 3. Esterification and Ritter reaction in one pot

| Entry | Substrate | Alcohol | Product | Yield (%) |
|-------|-----------|---------|---------|-----------|
| 24    | ![CN-COOH](image1) | ![CH3OH](image2) | ![N-(CH3)2-CONH](image3) | 53 |
| 25    | ![CN-COOH](image1) | ![i-PrOH](image4) | ![N-(i-Pr)2-CONH](image5) | 62 |
| 26    | ![CN-COOH](image1) | ![t-BuOH](image6) | ![N-(t-Bu)2-CONH](image7) | 60 |
| 27    | ![CN-COOH](image1) | ![Cyclohexanol](image8) | ![N-(Cyclohexyl)2-CONH](image9) | 50 |
| 28    | ![F-COOH](image10) | ![i-PrOH](image4) | ![N-(i-Pr)2-CONH](image5) | 58 |
| 29    | ![CN-COOH](image1) | ![i-PrOH](image4) | ![N-(i-Pr)2-CONH](image5) | 61 |

*All the reactions were carried with 80 mmol of substrate, 360 mmol of alcohol, and 94 mmol of sulfuric acid (98%). Yield refers to isolated yield.*

Very interestingly, when isobutyl alcohol is used for the reaction, tert-amido isobutyl ester is formed in one pot (entries 26, 28, 29, Table 3). A tentative mechanism for the acid-catalyzed reaction is given in Scheme 1.
CONCLUSIONS

Almost quantitative conversion of carboxylic acid to ester is achieved using acetonitrile as a water trap. Substrates having a nitrile group undergo intramolecular dehydration and rehydration to give amido ester in good yield. We have been able to show for the first time the Ritter reaction with unactivated primary alcohol. To sum up, we have developed a new reaction condition for esterification and hydrolysis in one pot in good yield, which should have great application in organic synthesis.

EXPERIMENTAL

Reagents and solvents were obtained from commercial sources and used without further purification. Thin-layer chromatography (TLC) was performed on precoated Merck silica-gel 60 F254 plates. Compounds were visualized using ultraviolet (UV) light (254 nm). Column chromatography refers to flash column chromatography on silica gel (200–300 mesh, Merck). NMR spectra were recorded on Jeol LA-300, Bruker AV400 spectrometer in CDCl$_3$. Tetramethylsilane [TMS (Me$_4$Si); $\delta = 0.00$ ppm] served as internal standard for $^1$H NMR. Chemical shifts were determined relative to the residual solvent peaks (CHCl$_3$, $\delta = 7.26$ ppm for hydrogen atoms, $\delta = 77.0$ for carbon atoms). The following abbreviations are used to indicate signal multiplicity: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; and bs, broad singlet. Mass spectra were measured with Micromass Q-Tof (ESI-HRMS) and GC/MS were measured with GCMSQP 2010 Plus gas chromatography mass spectrometer (Shimadzu).
Procedure for Ester (Table 2, entries 1–12)

Sulfuric acid (98%, 94 mmol, 5 ml) was added to a well-stirred mixture of acid (80 mmol), and alcohol (90 mmol), and acetonitrile (162 mmol), at room temperature. The mixture was slowly heated to 80–85 °C and maintained at 80–85 °C for 16–18 h. The reaction mixture was cooled and added to 20% sodium carbonate solution (100 mL). The reaction mass was extracted in CH₂Cl₂ (50 mL × 2). The combined organic layer was washed with water (100 mL), dried over sodium sulfate, and concentrated under reduced pressure to obtain corresponding ester (97–99%) as the only product. Most examples gave NMR pure product. Where minor impurities are formed, the product was purified over silica gel using ethyl acetate–petroleum ether (1:9).

Procedure for Amino Acid/Anhydrides (Table 2, entries 13–15)

Sulfuric acid (98%, 94 mmol, 5 ml) was added to a well-stirred mixture of amino acid (80 mmol, or anhydride), alcohol (180 mmol), and acetonitrile (162 mmol) at room temperature, and the temperature was maintained at 80–85 °C for 16–18 h. The reaction mixture was cooled and added to 20% sodium carbonate solution (100 mL). The reaction mass was extracted in CH₂Cl₂ (50 mL × 2). The combined organic layer was washed with water (100 mL), dried over sodium sulfate, and concentrated under reduced pressure to obtain the product (60–99%). Most examples gave the pure product. Where minor impurities are formed, the product was purified over silica gel using ethyl acetate–petroleum ether (1:9).

Procedure for Cyano Acid (Table 2, entries 16b–21b)

Sulfuric acid (98%, 94 mmol, 5 ml) was added to a well-stirred mixture of cyano acid (80 mmol) in alcohol (90 mmol) and acetonitrile (162 mmol) at room temperature, and temperature was maintained at 80–85 °C for 16–18 h. The reaction mixture was cooled and added to 20% sodium carbonate solution (100 mL). The reaction mass was extracted in CH₂Cl₂ (50 mL × 2). The combined organic layer was washed with water (100 mL), dried over sodium sulfate, and concentrated under reduced pressure to obtain the product (85–95%). Most examples gave NMR pure product. Where minor impurities are formed, the product was purified over silica gel using ethyl acetate–petroleum ether (1:9).

Butyl 2-Cyano-3-(3,4-dimethoxyphenyl)acrylate[41] (17b)

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.14 (s, 1H, C=CH), 7.80 (s, 1H, Ar-H), 7.47 (d, J = 8.4 Hz, 1H, Ar-H), 7.28 (s, 1H, Ar-H), 4.31 (t, J = 6.6 Hz, 2H, OCH₂), 3.96 [s, 6H, (OCH₃)₂], 1.76–1.71 (m, 2H, -CH₂), 1.49–1.43 (m, 2H, -CH₂), 0.98 (t, J = 7.4 Hz, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃) δ (ppm): 163.1, 154.5, 153.6, 149.2, 127.8, 124.5, 116.2, 56.0, 55.9, 30.5, 19.0, 13.6. MS (m/z, %): 289 (100%); HRMS calc. for C₁₆H₁₉NO₄Na: 312.1212; found: 312.1212(M + Na).

Procedure for Cyano Acid (Table 2, entries 22b–23b)

Sulfuric acid (98%, 94 mmol, 5 ml) was added to a well-stirred mixture of cyano acid (80 mmol) in alcohol (190 mmol) at room temperature, and temperature was
maintained at 80–85 °C for 16–18 h. The reaction mixture was cooled and added to 20% sodium carbonate solution (100 mL). The reaction mass was extracted in CH₂Cl₂ (50 mL × 2). The combined organic layer was washed with water (100 mL), dried over sodium sulfate, and concentrated under reduced pressure to obtain the product (95–98%). Most examples gave NMR pure product. Where minor impurities are formed, the product was purified over silica gel using ethyl acetate–petroleum ether (1:9).

**Methyl 3-Amino-2-cyclohexylidene-3-oxo-propanoate[^41] (22b)**

\[^1\]H NMR (400 MHz, CDCl₃) δ (ppm): 5.89 (d, J = 32.9 Hz, 2H, NH₂), 3.76 (s, 3H, OCH₃), 2.61 [t, J = 5.8 Hz, 2H, (CH₂) (Cyclic)], 2.44 [t, J = 5.9 Hz, 2H, (CH₂) (Cyclic)], 1.72–1.59 [m, 6H, (CH₂)₃]; \[^13\]C NMR (100 MHz, CDCl₃) δ (ppm): 168.4, 166.2, 160.1, 123.1, 51.9, 33.4, 31.8, 28.4, 28.1, 26.0. HRMS (m/z): calc. for C₁₀H₁₅NO₃Na: 220.0950. Found: 220.0949 (M + Na).

**Procedure for Cyano Acid (Table 3, entries 24b–29b)**

Sulfuric acid (98%, 94 mmol, 5 ml) was added to a well-stirred mixture of cyano acid (80 mmol) in alcohol (360 mmol) at room temperature, and the temperature was maintained at 80–85 °C for 16–18 h. The reaction mixture was cooled and added to 20% sodium carbonate solution (100 mL). The reaction mass was extracted in CH₂Cl₂ (50 mL × 2). The combined organic layer was washed with water (100 mL), dried over sodium sulfate, and concentrated under reduced pressure to obtain the product (50–62%). Most examples gave NMR pure product. Where minor impurities are formed, the product was purified over silica gel using ethyl acetate–petroleum ether (1:9).

**Butyl 3-(Sec-butylamino)-3-oxopropanoate:[^41] (24b)**

\[^1\]H NMR (400 MHz, CDCl₃) δ (ppm): 4.14 [t, 2H, (OCH₂)], 3.97–3.90 (m, 1H, CH), 3.29 (s, 2H, CO-CH₂), 1.67–1.60 (m, 2H, CH₂), 1.53–1.43 (m, 2H, CH₂), 1.41–1.33 (m, 2H, CH₃), 1.25 (s, 1H, NH₂), 1.13 (s, 2H, CH₂), 0.95–0.89 (m, 3H, CH₃); \[^13\]C NMR (100 MHz, CDCl₃) δ (ppm): 169.8, 164.2, 65.3, 46.7, 41.2, 30.4, 29.4, 20.2, 19.0, 13.6, 10.2. HRMS calc. for C₁₁H₂₁NO₃ Na: 238.1419. Found: 238.1415 (M + Na).

**SUPPORTING INFORMATION**

Full experimental details, \[^1\]H and \[^13\]C NMR spectra, GC/MS, and HRMS (entries 1b–29b) can be found via the Supplementary Content section of this article’s Web page.

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