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# A new modification of the Passerini reaction: a one-pot synthesis of α-acyloxyamides via sequential Kornblum oxidation/Passerini reaction

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**Abstract**-A novel approach for the synthesis of α-acyloxyamides is described. Benzylic substrates (halides or tosylates), under mild Kornblum conditions, are oxidized to give the corresponding aldehydes, which undergo a Passerini reaction with carboxylic acids and isocyanides to produce the corresponding α-acyloxyamides in excellent yields.

*Keywords*: Kornblum oxidation, Passerini reaction, α-acyloxyamides, benzylic substrates, dimethyl sulfoxide, carboxylic acids, isocyanides.

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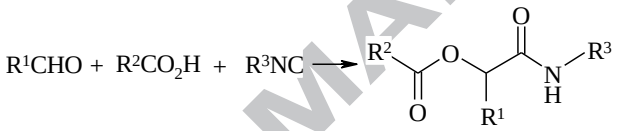
Multicomponent reactions (MCRs) have become important tools in synthetic chemistry due to their ability to provide efficient access to complex molecules from readily available starting materials, and their utility for the construction of interesting biologically active organic compounds and the rapid generation of drug-like molecule libraries.[1] Isocyanide-based MCRs are especially important in this area.[2,3]

The high efficiency of isocyanides in MCRs is due to their divalent C-atom and its distinct reactivity towards C($sp^2$ or sp) electrophilic centers. In transformations involving these entities, this divalent C-atom is converted into a tetravalent state, which makes the addition irreversible. It must be remembered that this property of isocyanides was essential in the successful development of classical MCRs such as the Passerini three-component reaction (P-3CR) as the first MCR involving isocyanides,[4] which combines an aldehyde, a carboxylic acid and an isocyanide to give an α-acyloxyamide (Scheme 1).




**Scheme 1.** The Passerini three-component reaction.

The Passerini reaction has been the subject of extensive research. Several modifications of the classic P-3CR have been described, including variation of one of the components or introduction of a linkage between two of them.[5] Some cases involve a catalyst such as Cu(II),[6] Al(III),[7] aluminum-organophosphate,[8] $SiCl_4$,[9] $Bi(OTf)_3$,[10] Ti(*i*-PrO)$_4$,[11] etc., or occur in the presence of an ionic liquid.[12] Through the application of these MCRs, interesting potential drug-like derivatives of the original α-acyloxyamide product and many different skeletons are now readily accessible.[5–13]

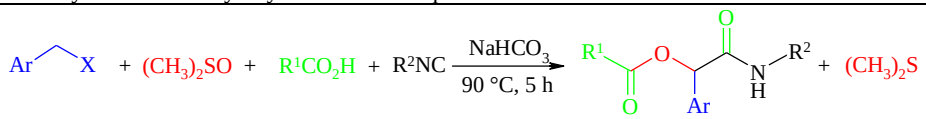
Although a large number of modifications of P-3CRs have been successfully explored, to the best of our knowledge, only one oxidative Passerini reaction has been reported, by Zhu et al., in which reactions between alcohols, isocyanides, and carboxylic acids in toluene in the presence of a catalytic amount of cupric chloride, sodium nitrite, and TEMPO, under an oxygen atmosphere, resulted in the formation of P-3CR adducts in good yields.[14]

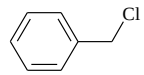
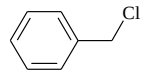
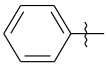
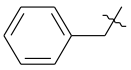
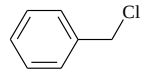
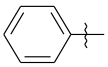
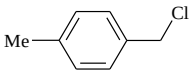
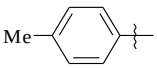
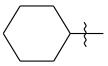
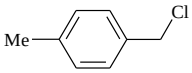
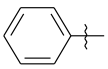
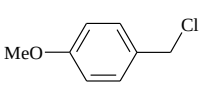
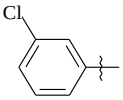
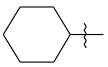
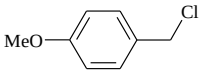
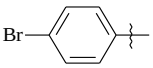
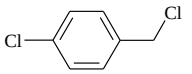
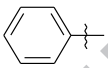
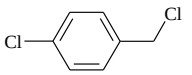
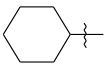
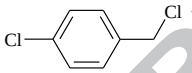
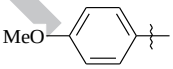
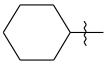
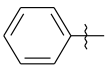
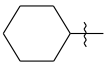
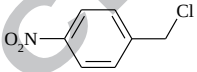
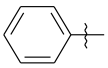
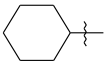
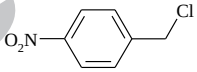
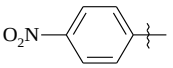
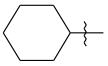
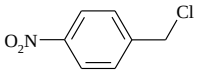
Considering the pharmacological significance of α-acyloxyamides, and as part of our continuing efforts on the development of new routes for the preparation of

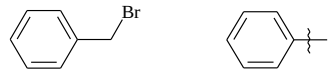
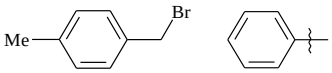
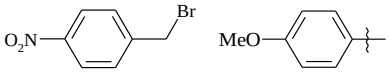
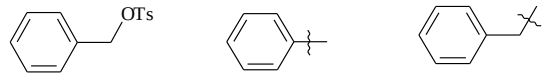
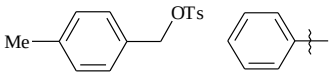
biologically active organic compounds,[15] herein, we describe a new approach for the synthesis of α-acyloxyamides, which is initiated by an *in situ* oxidation of benzylic substrates to the corresponding aldehydes. This procedure develops the flexibility of the Passerini reaction, while not limiting one of the substrates to being an aldehyde. It has additional advantages such as the use of more commercially accessible benzylic substrates compared to aldehydes, and the intermediate aldehydes do not need separation. This should be especially valuable in cases where the aldehydes are unstable (e.g., volatile, or susceptible to polymerization or hydrolysis).

Thus, under mild Kornblum oxidation conditions,[16] a solution of a benzylic substrate **1** in dimethyl sulfoxide in the presence of sodium bicarbonate at 90 °C was converted into the corresponding aldehyde within three hours,[17] as was indicated by TLC monitoring. Subsequently, the aldehyde prepared *in situ* was treated with a carboxylic acid **2** and an isocyanide **3** at 90 °C for two hours to afford the corresponding Passerini reaction products, α-acyloxyamides **4**, in 86–96% yields (with respect to the benzylic substrate **1**) (Table 1). All the reactions went to completion within five hours. $^{1}$H NMR analysis of the reaction mixtures clearly indicated formation of the corresponding α-acyloxyamides **4** in excellent yields.[18] All the products were characterized by melting point determination and from their $^{1}$H and $^{13}$C NMR spectral data.

**Table 1** Direct synthesis of α-acyloxyamides **4** via sequential Kornblum oxidation/Passerini reaction.[a]

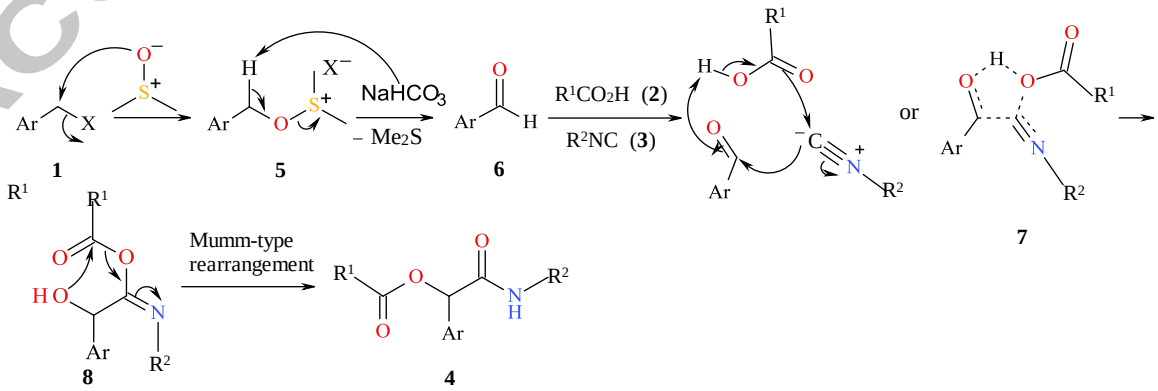



| | **1** | **2** | **3** | **4** | | |
|---|---|---|---|---|---|---|
| Entry | $ArCH_2X$ | $R^1$ | $R^2$ | **4**, Yield (%)[b] | mp (°C) | Lit. mp (°C) |
| 1 |  | $CH_3$ |  | **4a**, 93[c] | 75–76 | 77[19] |
| 2 |  |  |  | **4b**, 92 | 104–105 | 104[19] |
| 3 |  |  |  | **4c**, 94 | 149–150 | 148–150[20] |
| 4 |  |  |  | **4d**, 93 | 122–124 | 122–124[21] |
| 5 |  |  |  | **4e**, 91 | 151 | 151–153[20] |
| 6 |  |  |  | **4f**, 92 | 150–152 | 150–151[22] |
| 7 |  |  |  | **4g**, 90 | 125–127 | 125–126[22] |
| 8 |  |  |  | **4h**, 96 | 197 | 196–198[23] |
| 9 |  |  |  | **4i**, 93 | 199–200 | 198–200[21] |
| 10 |  |  |  | **4j**, 86 | 184–185 | 184–186[20] |
| 11 |  |  |  | **4k**, 93 | 149–150 | 148–150[24] |
| 12 |  |  |  | **4l**, 89 | 210–212 | 210–212[20] |
| 13 |  |  |  | **4m**, 90 | 114–115 | 114–116[21] |
| 14 |  |  |  | **4n**, 87 | 199–200 | 199–201[20] |
| 15 |  |  |  | **4o**, 91 | 113 | 112–114[24] |
| 16 |  |  |  | **4p**, 90 | 115–116 | 115–117[24] |
| 17 |  |  |  | **4b**, 93 | 104–105 | 104[19] |

| | | | | | |
|---|---|---|---|---|---|
| 18 |  | **4c**, 95 | 149–150 | 148–150[20] |
| 19 |  | **4e**, 92 | 151 | 151–153[20] |
| 20 |  | **4n**, 89 | 199–200 | 199–201[20] |
| 21 |  | **4b**, 95 | 104–105 | 104[19] |
| 22 |  | **4c**, 96 | 149–150 | 148–150[20] |
| 23 |  | **4e**, 94 | 151 | 151–153[20] |

[a]The reaction conditions were optimized for the synthesis of α-acyloxyamide **4c** as a model reaction (entry 3), which were used for the preparation of all the other α-acyloxyamides:
reaction temperature of 90 ºC for both the Kornblum oxidation and the Passerini reaction;
reaction time of 3 h for the Kornblum oxidation and 2 h for the Passerini reaction;
use of benzyl chloride, $NaHCO_3$, benzoic acid and *tert*-butyl isocyanide in a molar ratio of 1:1.1:1.2:1.2;
DMSO volume of 0.75 mL for 1 mmol of benzyl chloride.
[b]Isolated yields.
[c]α-Acyloxyamide **4a** was purified by column chromatography and α-acyloxyamides **4b**–**p** were purified through precipitation by adding water to the reaction mixture.[18]

A mechanistic explanation for this reaction is depicted in Scheme 2. At first, reaction of the benzylic substrate **1** with dimethyl sulfoxide gives the alkoxydimethylsulfonium intermediate **5**, which in the presence of $NaHCO_3$ undergoes elimination of dimethyl sulfide to form the corresponding aldehyde **6**. The next step involves the reaction between aldehyde **6**, carboxylic acid **2** and isocyanide **3** through a presumed cyclic transition state **7** to give intermediate **8**. Finally, an irreversible Mumm-type rearrangement (a transacylation) leads to the α-acyloxyamide **4** (Scheme 2).




**Scheme 2.** Proposed mechanism.

In conclusion, we have developed a new and straightforward approach for the synthesis of α-acyloxyamides of potential synthetic and pharmacological interest, via sequential Kornblum oxidation/Passerini reaction between benzylic substrates, dimethyl sulfoxide, carboxylic acids and isocyanides. The use of benzylic substrates in place of aldehydes, the good to excellent yields of the products and the easy work-up procedure are the main advantages of this method. To the best of our knowledge, this is the first report on the use of benzyl halides or benzyl tosylates instead of aldehydes as substrates for the preparation of α-acyloxyamides via the Passerini reaction.

**Acknowledgement**

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**References and notes:**

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18. *General procedure for the preparation of compounds **4a**–**p***: A mixture of the appropriate benzylic substrate (1 mmol) and $NaHCO_3$ (0.092 g, 1.1 mmol) in DMSO (0.75 mL) was stirred for 3 h at 90 ºC. Next, the appropriate carboxylic acid (1.2 mmol) and isocyanide (1.2 mmol) were added to the mixture and stirring was continued at 90 ºC for 2 h. After completion of the reaction, the mixture was cooled to ambient temperature. Next, $H_2O$ (2 mL) was added to the mixture and stirring was continued for 15 min at ambient temperature. The resulting precipitate was filtered, washed with $H_2O$, dried, and recrystallized from *n*-hexane/EtOAc

(3:1) to afford the pure product **4**. [α-Acyloxyamide **4a** was purified by column chromatography using *n*-hexane-EtOAc (4:1) as eluent].

Selected NMR spectral data:

**2-(*tert*-Butylamino)-2-oxo-1-phenylethyl benzoate (4c)** $^{1}$H NMR (300.1 MHz, DMSO-$d_6$): $\delta$ 1.22 [9H, s, C(CH$_3$)$_3$], 6.09 (1H, s, OCH), 7.35 (1H, t, $J$ = 7.2 Hz, CH), 7.42 (2H, dd, $J$ = 7.4, 7.1 Hz, 2CH), 7.53 (2H, dd, $J$ = 7.5, 7.4 Hz, 2CH), 7.61 (2H, d, $J$ = 7.6 Hz, 2CH), 7.65 (1H, t, $J$ = 7.1 Hz, CH), 7.97 (1H, s, NH), 8.04 (2H, d, $J$ = 7.5 Hz, 2CH). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta$ 28.32 [C(*C*H$_3$)$_3$], 50.41 [*C*(CH$_3$)$_3$], 75.45 (OCH), 127.16 (2CH), 128.37 (CH), 128.38 (2CH), 128.73 (2CH), 129.33 (C), 129.34 (2CH), 133.50 (CH), 136.21 (C), 164.91 and 167.00 (2C=O). **2-(Cyclohexylamino)-1-(4-methylphenyl)-2-oxoethyl 4-methylbenzoate (4d)**: $^{1}$H NMR (300.1 MHz, DMSO-$d_6$): $\delta$ 1.00–1.76 [10H, m, CH(C*H*$_2$)$_5$], 2.29 and 2.37 (6H, 2s, 2CH$_3$), 3.45–3.55 (NCH), 6.00 (1H, s, OCH), 7.20 (2H, d, $J$ = 7.9 Hz, 2CH), 7.33 (2H, d, $J$ = 8.0 Hz, 2CH), 7.45 (2H, d, $J$ = 7.9 Hz, 2CH), 7.91 (2H, d, $J$ = 8.0 Hz, 2CH), 8.14 (1H, d, $J$ = 7.7 Hz, NH). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta$ 20.72 and 21.15 (2CH$_3$), 24.36, 24.44, 25.10, 32.02 and 32.20 (5CH$_2$), 47.61 (NCH), 75.24 (OCH), 126.60 (C), 127.11 (2CH), 128.93 (2CH), 129.26 (2CH), 129.41 (2CH), 133.18, 137.80 and 143.86 (3C), 164.92 and 166.94 (2*C*=O). **2-(*tert*-Butylamino)-2-oxo-1-phenylethyl 4-chlorobenzoate (4h)**: $^{1}$H NMR (300.1 MHz, DMSO-$d_6$): $\delta$ 1.21 [9H, s, C(CH$_3$)$_3$], 6.08 (1H, s, OCH), 7.48 (2H, d, $J$ = 8.4 Hz, 2CH), 7.53 (2H, t, $J$ = 7.6 Hz, 2CH), 7.62 (2H, d, $J$ = 8.4 Hz, 2CH), 7.66 (1H, t, $J$ = 7.4 Hz, CH), 8.00 (1H, s, NH), 8.02 (2H, d, $J$ = 7.8 Hz, 2CH). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta$ 28.26 [C(*C*H$_3$)$_3$], 50.45 [*C*(CH$_3$)$_3$], 74.69 (OCH), 128.45 (2CH), 128.72 (2CH), 128.95 (2CH), 129.18 (C), 129.34 (2CH), 133.15 (C), 133.55 (CH), 135.16 (C), 164.80 and 166.67 (2C=O). **2-(Cyclohexylamino)-1-(4-methoxyphenyl)-2-oxoethyl 4-chlorobenzoate (4j)**: $^{1}$H NMR (300.1 MHz, DMSO-$d_6$): $\delta$ 0.99–1.77 [10H, m, CH(C*H*$_2$)$_5$], 3.45–3.51 (1H, NCH), 3.82 (3H, s, OCH$_3$), 6.05 (1H, s, OCH), 7.05 (2H, d, $J$ = 8.5 Hz, 2CH), 7.47 (2H, d, $J$ = 8.2 Hz, 2CH), 7.61 (2H, d, $J$ = 8.2 Hz, 2CH), 7.99 (2H, d, $J$ = 8.5 Hz, 2CH), 8.24 (1H, d, $J$ = 7.7 Hz, NH). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta$ 24.32, 24.41, 25.09, 31.98 and 32.18 (5CH$_2$), 47.68 (NCH), 55.49 (OCH$_3$), 74.46 (OCH), 114.01 (2CH), 121.29 (C), 128.45 (2CH), 128.88 (2CH), 131.58 (2CH), 133.16 and 135.24 (2C), 163.42 (C–O) 164.49 and 166.63 (2C=O). **2-(Cyclohexylamino)-2-oxo-1-phenylethyl 4-chlorobenzoate**

**(4k)**: $^{1}$H NMR (300.1 MHz, DMSO-$d_6$): $\delta$ 0.99–1.78 [10H, m, CH(C$H_2$)$_5$], 3.46–3.53 (1H, NCH), 6.08 (1H, s, OCH), 7.48 (2H, d, $J$ = 8.5 Hz, 2CH), 7.53 (2H, t, $J$ = 7.7 Hz, 2CH), 7.62 (2H, d, $J$ = 8.5 Hz, 2CH), 7.66 (1H, t, $J$ = 7.4 Hz, CH), 8.03 (2H, dd, $J$ = 7.2, 1.1 Hz, 2CH), 8.27 (1H, d, $J$ = 7.8 Hz, NH). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta$ 24.30, 24.39, 25.08, 31.96 and 32.17 (5$CH_2$), 47.69 (NCH), 74.75 (OCH), 128.47 (2CH), 128.73 (2CH), 128.93 (2CH), 129.12 (C), 129.40 (2CH), 133.23 (C), 133.60 (CH), 135.02 (C), 164.83 and 166.46 (2C=O).

**2-(Cyclohexylamino)-1-(4-methoxyphenyl)-2-oxoethyl 4-nitrobenzoate (4n)**: $^{1}$H NMR (300.1 MHz, DMSO-$d_6$): $\delta$ 1.00–1.77 [10H, m, CH(C$H_2$)$_5$], 3.43–3.54 (1H, NCH), 3.81 (3H, s, $CH_3$), 6.21 (1H, s, OCH), 7.05 (2H, d, $J$ = 8.5 Hz, 2CH), 7.88 (2H, d, $J$ = 8.4 Hz, 2CH), 8.02 (2H, d, $J$ = 8.5 Hz, 2CH), 8.27 (2H, d, $J$ = 8.4 Hz, 2CH), 8.39 (1H, d, $J$ = 7.6 Hz, NH). $^{13}$C NMR (75.5 MHz, DMSO-$d_6$): $\delta$ 24.26, 24.34, 25.05, 31.91 and 32.13 (5$CH_2$), 47.78 (NCH), 55.45 ($OCH_3$), 74.35 (OCH), 113.99 (2CH), 121.04 (C), 123.54 (2CH), 128.05 (2CH), 131.63 (2CH), 143.37 and 147.40 (2C), 163.48 (C–O) 164.33 and 165.98 (2C=O).


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**A new modification of the Passerini reaction: a one-pot synthesis of α-acyloxyamides via sequential Kornblum oxidation/Passerini reaction**

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