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# *tert*-Butyl nitrite promoted transamidation of secondary amides under metal and catalyst free conditions†

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A mild and efficient method is demonstrated for the transamidation of secondary amides with various amines including primary, secondary, cyclic and acyclic amines in the presence of *tert*-butyl nitrite. The reaction proceeds through the *N*-nitrosamide intermediate and provides the transamidation products in good to excellent yields at room temperature. Moreover, the developed methodology does not require any catalyst or additives.



Amides are key functional groups that exist in many biomolecules, natural products, drugs, *etc*. (Fig. 1).[1] On the other hand, amides are also employed as valuable precursors in organic synthesis.[2] Transamidation is an important reaction that has received significant interest in recent years.[3] Owing to the high stability of amide bonds, transamidation of secondary amides was considered to be a more challenging task in organic synthesis.[3i–l] In this context, Garg and coworkers developed a two-step process for the efficient transamidation of secondary amides under mild conditions.[4] In the Garg method, secondary amides are converted into non-planar *N*-Boc amides in the first step and subjected to transamidation with different amines in the presence of nickel catalysts. Later, Szostak *et al.* demonstrated the activation of *N*-Boc and *N*-tosyl protected secondary amides using palladium catalysts.[5] Moreover, recently the Szostak group has also successfully demonstrated the base promoted transamidation and esterification of *N*-Boc and *N*-tosyl amides under metal free conditions.[6]

*N*-Nitrosamines are valuable precursors in organic synthesis.[7] In contrast, the applications of *N*-nitrosamides are less explored in synthetic organic chemistry.[8] In 1983, Garcia *et al.* reported that *N*-nitrosamides undergo transamidation efficiently with different amines in the absence of any catalysts or bases.[8e,f] However, this transamidation approach remained unpopular in organic synthesis because the preparation, isolation and handling of *N*-nitrosamides are quite difficult. In this context, our aim was to identify a suitable nitrosating reagent for the generation of *N*-nitrosamides *in situ* which can be directly subjected to the transamidation process without isolation of the *N*-nitrosamide intermediate. In fact, such protocols will reduce not only the number of steps but also tedious workup procedures. Moreover, it will be an alternative to *N*-Boc and *N*-tosyl amide mediated transamidation processes. In this context, here we report an efficient, metal and catalyst free method for the transamidation of secondary amides using *tert*-butyl nitrite at room temperature (Scheme 1).

The *N*-nitrosation of secondary amides has been achieved using different nitrosyl sources including $NaNO_2$-HCl,[8c] nitrogen oxide-NaOAc,[8e,f] nitrosyl chloride-KOAc,[9] nitrosyl tetrafluoroborate-pyridine,[10] and *tert*-butyl nitrite.[11] However, some of these reagents may not be suitable for the one-pot transamidation process. For example, the $NaNO_2$-HCl system requires aqueous acidic conditions which may lead to different side reactions. On the other hand, nitrogen oxide and nitrosyl

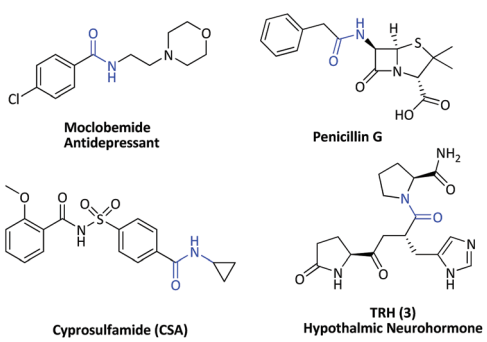


**Fig. 1** Some important natural products and drug molecules bearing amide bonds.

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†Electronic supplementary information (ESI) available: NMR spectra are available for all the products. See DOI: 10.1039/c8ob03010c

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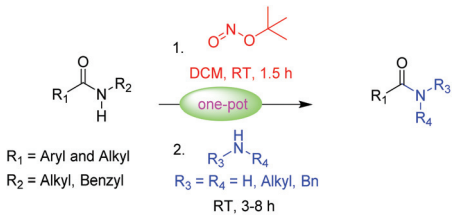


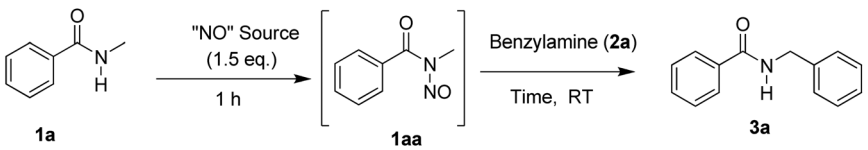

**Scheme 1** Transamidation of secondary amides in the presence of TBN.

chloride are gases that are difficult to prepare and handle. In this context, we have envisioned that nitrosyl tetrafluoroborate or *tert*-butyl nitrite might be more appropriate for one-pot transamidation reactions because these reagents are relatively milder and also commercially available.

At the outset, transamidation of *N*-methylbenzamide (**1a**) was investigated with benzylamine (**2a**) in the presence of nitrosyl tetrafluoroborate (Table 1, entries 1–4). Initially, the formation of *N*-nitrosamide (**1aa**) was assessed with nitrosyl tetrafluoroborate using crude $^1$H NMR. The reaction was performed in acetonitrile and dichloromethane solvents in the presence and absence of pyridine. The maximum yield of *N*-nitrosamide **1aa** (*i.e.* 92%) was obtained in acetonitrile in the presence of pyridine after 12 hours at room temperature (Table 1, entry 3). However, the other conditions gave relatively low yields of *N*-nitrosamide **1aa**. Nevertheless, after 12 hours, benzylamine (the external nucleophile) was added to the reaction mixture containing *N*-nitrosamide and the reaction mixture was allowed to stir for an additional 12 hours at room temperature. The transamidation proceeded efficiently in dichloromethane in the absence of pyridine and gave the desired product **3a** in 54% yield (Table 1, entry 2). However, only 20% yield of **3a** was obtained in acetonitrile (Table 1, entry 1). It is surprising to note that the transamidation process was significantly suppressed in the presence of pyridine in both acetonitrile and dichloromethane solvents (Table 1, entries 3 and 4).

*tert*-Butyl nitrite (TBN) is an acid free nitrating and nitrosating reagent used in many synthetic transformations.[12] Over the past few years, our research work has focused on the chemistry of *N*-nitrosamines[13] and we have reported solvent free synthesis of *N*-nitrosamines,[13a] radical dimerization of thiobenzamides[13d] and triazole formation of *o*-phenylenediamine[13f]

**Table 1** Optimization of transamidation of *N*-methylbenzamide[a]




| S. no. | Nitroso source | Solvent | Bases | Nitrosation Time (h) | Nitrosation Yield **1aa**[b] (%) | Transamidation Time (h) | Transamidation Yield **3a**[c] (%) |
|---|---|---|---|---|---|---|---|
| 1 | $NOBF_4$ | AcCN | — | 12 | 65 | 12 | 20 |
| 2 | $NOBF_4$ | DCM | — | 12 | 74 | 12 | 54 |
| 3 | $NOBF_4$ | AcCN | Py | 12 | 92 | 12 | 30 |
| 4 | $NOBF_4$ | DCM | Py | 12 | 74 | 12 | 15 |
| **5** | ***t*-BuONO** | **DCM** | — | **1** | **95** | **3** | **91** |
| 6 | *t*-BuONO | DCE | — | 1 | 91 | 3 | 87 |
| 7 | *t*-BuONO | THF | — | 1 | 50 | 12 | 15 |
| 8 | *t*-BuONO | Toluene | — | 1 | 35 | 12 | 30 |
| 9 | *t*-BuONO | Benzene | — | 1 | 30 | 12 | 17 |
| 10 | *t*-BuONO | AcCN | — | 1 | 80 | 12 | 60 |
| 11 | *t*-BuONO | 1,4-Dioxane | — | 1 | 70 | 12 | 40 |
| 12 | *t*-BuONO | MeOH | — | 1 | <5 | 12 | nr |
| 13 | *t*-BuONO | EtOH | — | 1 | <5 | 12 | nr |
| 14 | *t*-BuONO | $H_2O$ | — | 1 | <5 | 12 | nr |
| 15 | *t*-BuONO | Solvent-free | — | 1 | 97 | 12 | 47 |
| 16 | *t*-BuONO | DCM | Pyridine | 1 | 75 | 3 | 48 |
| 17 | *t*-BuONO | DCM | $Et_3N$ | 1 | 82 | 3 | 48 |
| 18 | *t*-BuONO | DCM | $Et(i\text{-}Pr)_2N$ | 1 | 78 | 3 | 44 |
| 19 | *t*-BuONO | DCM | DBU | 1 | 68 | 3 | 45 |
| 20 | *t*-BuONO | DCM | DABCO | 1 | 67 | 3 | 42 |
| 21 | iso-AmylONO | DCM | — | 1 | 70 | 3 | 51 |
| 22 | *n*-BuONO | DCM | — | 1 | 83 | 3 | 64 |
| 23 | *t*-BuONO | DCM | — | — | — | 6 | 10[d] |
| 24 | *t*-BuONO | DCM | — | — | — | 6 | nr[e] |

[a] Reaction conditions: Substrate (1 mmol) and TBN (1.5 equiv.) stirred in appropriate solvents (5 mL) for appropriate times after which benzylamine (2.2 equiv.) was added. [b] Crude yield was obtained using $^1$H NMR. [c] Isolated yield. [d] TBN was added to the pre-stirred solution of *N*-methylbenzamide and benzylamine. [e] *N*-Methylbenzamide was added to the pre-stirred solution of benzylamine and TBN.

using *tert*-butyl nitrite. Recently, *N*-nitrosation of secondary amides using *tert*-butyl nitrite has been demonstrated by Bhanage *et al.*[11] Therefore, the one-pot transamidation reaction was investigated with *tert*-butyl nitrite in different solvents.

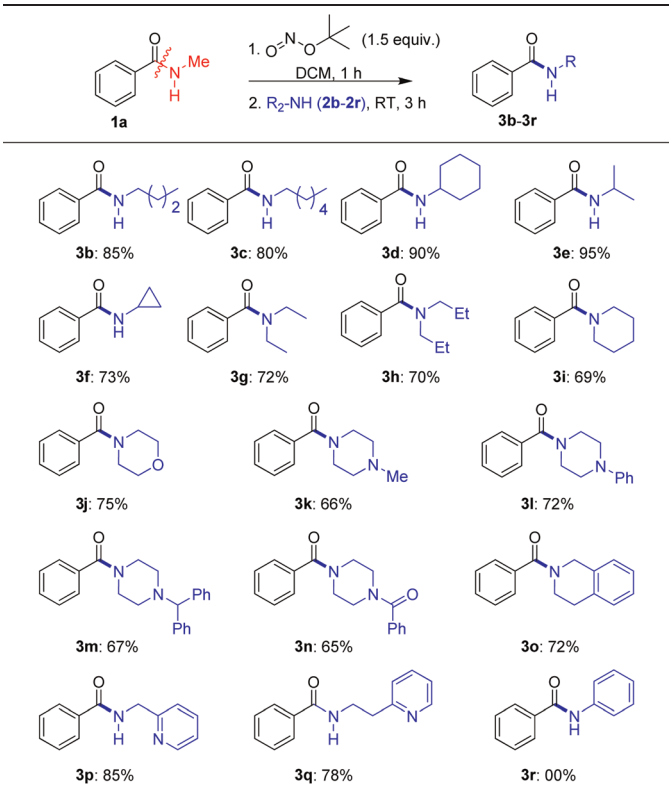
Initially, the conversion of *N*-methylbenzamide into the corresponding *N*-nitrosamide (**1aa**) was assessed in the presence of TBN in different solvents including dichloromethane, dichloroethane, THF, toluene, benzene, acetonitrile and dioxane using $^1$H NMR (Table 1, entries 5–11). Among them, dichloromethane was found to be the most efficient which provided the *N*-nitrosamide **1aa** in 95% yield within 1 h (Table 1, entry 5). On the other hand, polar protic solvents such as methanol, ethanol and water failed to provide the *N*-nitrosamide **1aa** (Table 1, entries 12–14). As reported by Bhanage *et al.* the *N*-nitrosamide **1aa** was obtained in 97% yield under solvent free conditions (Table 1, entry 15). Nevertheless, after 1 hour, benzylamine (2.2 equiv.) was added to the reaction mixture containing *N*-nitrosamide and the reaction mixture was allowed to stir up to 12 h at room temperature. To our delight, the one-pot transamidation was successfully achieved with 91% yield in dichloromethane within 3 h while other conditions gave the desired product **3a** in low yields. It is also worth mentioning that the transamidation product **3a** was obtained only in 47% yield under solvent free conditions even after 12 h at room temperature (Table 1, entry 15).

Encouraged, we have studied the effect of the different bases including pyridine, $Et_3N$, $Et(i\text{-}Pr)_2N$, DBU and DABCO in the transamidation process in dichloromethane (Table 1, entries 16–20). It was observed that in the presence of a base, not only nitrosamide formation but also the transamidation process was significantly suppressed. Furthermore, the transamidation reaction was investigated with other alkyl nitrites such as iso-amyl nitrite and *n*-butyl nitrite in dichloromethane. These reagents gave the desired product **3a** in 70% and 83% yields, respectively (Table 1, entries 21 and 22). Furthermore, transamidation of *N*-methylbenzamide was executed in two other conditions in dichloromethane using *tert*-butyl nitrite (TBN), namely, (i) TBN was added to the pre-stirred solution of *N*-methylbenzamide and benzylamine (Table 1, entry 23) in dichloromethane and (ii) *N*-methylbenzamide was added to the pre-stirred solution of benzylamine and TBN (Table 1, entry 24) in dichloromethane. In the first case, the desired product **3a** was obtained only in 10% yield while the latter conditions failed to yield the desired transamidation product.

Having established the optimized conditions (Table 1, entry 5), transamidation of *N*-methylbenzamide was investigated with various amines in the presence of *tert*-butyl nitrite (Table 2). Initially, *N*-methylbenzamide was subjected to transamidation with different primary amines such as *n*-butyl, *n*-hexyl, cyclohexyl, iso-propyl and cyclopropyl-amines under optimized conditions. All these reactions proceeded smoothly at room temperature while the desired transamidation products were obtained in 73–95% yields (Table 2, **3b–3f**). Similarly, cyclic and acyclic secondary amines such as diethylamine, dipropylamine, piperidine, morpholine, *N*-methyl piperazine, *N*-phenyl piperazine, *N*-benzhydryl piperazine, *N*-benzoyl piperazine and 1,2,3,4-tetrahydroisoquinoline participated in the transamidation process smoothly and gave the desired products in good to excellent yields (Table 2, **3g–3o**). Interestingly, heteroaromatic amines such as 2-aminomethyl pyridine and 2-aminoethyl pyridine also acted as efficient nucleophiles in the transamidation process and provided the desired products **3p** and **3q** in 85% and 78% yields, respectively. However, transamidation was not successful when aniline was used as a nucleophile, perhaps due to less nucleophilicity of the amine (Table 2, **3r**).

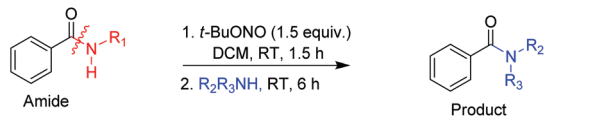
The scope of the developed methodology was further examined with different *N*-alkyl benzamides under optimized conditions (Table 3). Similar to *N*-methylbenzamides, *N*-ethyl (**1b**), *N*-propyl (**1c**), *N*-butyl (**3b**), *N*-hexyl (**3c**) and *N*-benzylbenzamides (**3a**) also underwent nitrosation followed by transamidation very efficiently with different primary and secondary amines (cyclic and acyclic) under optimized conditions (Table 3). These reactions provided the desired transamidation products in 54–88% yields. Moreover, irrespective of the substitutes present on the aryl ring (*i.e.* electron donating or withdrawing), nitrosation followed by transamidation occurred smoothly to provide the desired products in good to excellent yields (Table 4, **5a–5r**).

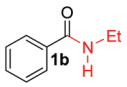
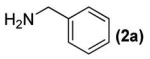
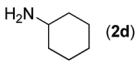
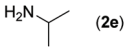
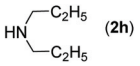
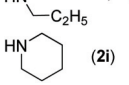
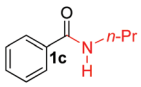
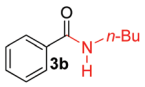
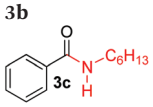
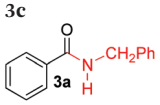
**Table 2** One-pot transamidation of *N*-methylbenzamide with different amines[a,b]




[a] Reaction conditions: Substrate (1 mmol), TBN (1.5 equiv.) and amine (2.2 equiv.) in DCM (5 mL) at room temperature. [b] Isolated yields.

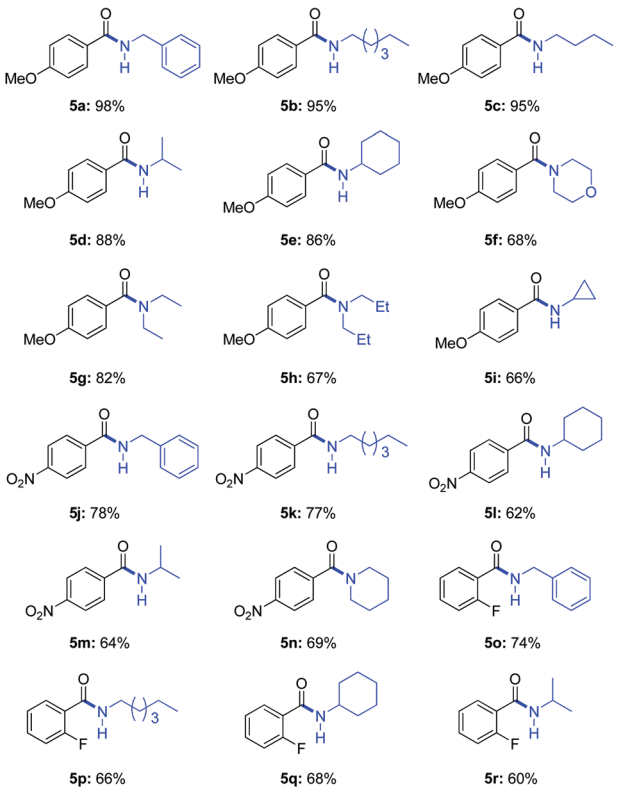
**Table 3** One-pot transamidation of *N*-alkyl benzamides with different amines[a,b]



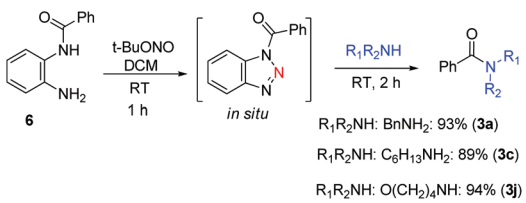

| Entry | Amide | Nucleophile | Product | Yield (%) |
|---|---|---|---|---|
| 1 |  |  (2a) | 3a | 83 |
| 2 | 1b |  (2d) | 3d | 79 |
| 3 | 1b |  (2e) | 3e | 76 |
| 4 | 1b |  (2h) | 3h | 58 |
| 5 | 1b |  (2i) | 3i | 62 |
| 6 |  | 2a | 3a | 85 |
| 7 | 1c | 2d | 3d | 81 |
| 8 | 1c | 2e | 3e | 80 |
| 9 | 1c | 2h | 3h | 57 |
| 10 | 1c | 2i | 3i | 63 |
| 11 |  | 2a | 3a | 86 |
| 12 | 3b | 2d | 3d | 82 |
| 13 | 3b | 2e | 3e | 74 |
| 14 | 3b | 2h | 3h | 56 |
| 15 | 3b | 2i | 3i | 60 |
| 16 |  | 2a | 3a | 88 |
| 17 | 3c | 2d | 3d | 85 |
| 18 | 3c | 2e | 3e | 78 |
| 19 | 3c | 2h | 3h | 56 |
| 20 | 3c | 2i | 3i | 60 |
| 21 |  | 2d | 3d | 54 |
| 22 | 3a | 2e | 3e | 72 |
| 23 | 3a | 2i | 3i | 63 |

[a] Reaction conditions: Substrate (1 mmol), TBN (1.5 equiv.) and amine (2.2 equiv.) in DCM (5 mL) at RT. [b] Isolated yields.

**Table 4** One-pot transamidation of various *N*-methylbenzamides with different amines[a,b]


[a] Reaction conditions: Substrate (1 mmol), TBN (1.5 equiv.) and amine (2.2 equiv.) in DCM (5 mL) at RT. [b] Isolated yields.

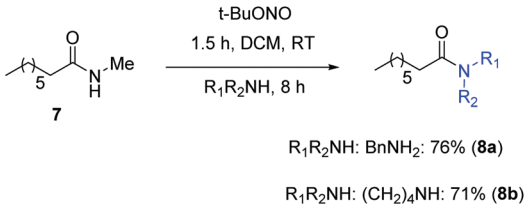



**Scheme 2** Transamidation of *N*-benzoyl *o*-phenylenediamine *via* *N*-acyl benzimidazole.

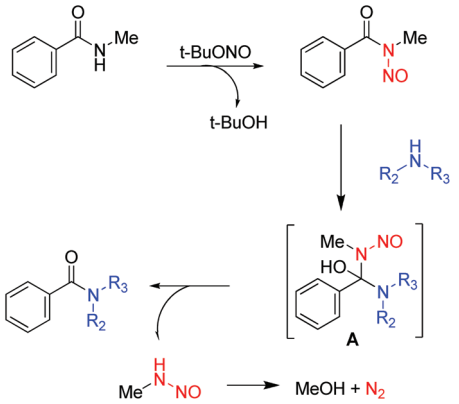
It is also interesting to note that the transamidation of *N*-benzoyl *o*-phenylenediamine (**6**) with different amines was achieved with excellent yields (Scheme 2). The reaction proceeds through the *N*-acyl benzimidazole intermediate under optimized conditions.

Having explored the transamidation of various *N*-substituted benzamides, an alkyl secondary amide, *i.e.* *N*-methyloctanamide (**7**), was subjected to transamidation with benzylamine and pyrrolidine under optimized conditions. To our delight, the one-pot transamidation proceeded smoothly in the presence of *tert*-butyl nitrite to provide the desired products **8a** and **8b** in high yields (Scheme 3).

A proposed mechanism for the transamidation process is shown in Scheme 4. In the first step, the secondary amide undergoes *N*-nitrosation to form *N*-nitrosamide in the presence of *tert*-butyl nitrite. In the second step, *N*-nitrosamide undergoes nucleophilic addition with an external amine to form the intermediate **A**. This unstable intermediate undergoes elimination to provide the desired product and alkyl *N*-nitrosamine.




**Scheme 3** Transamidation of *N*-methyloctanamide.




**Scheme 4** Proposed mechanism for the transamidation reaction.

In conclusion, a mild and practical method for the one-pot transamidation of various secondary amides was demonstrated with different amines in the presence of *tert*-butyl nitrite. The reaction proceeds through the *N*-nitrosamide intermediate. All the reactions were carried out at room temperature to obtain the desired products in good to excellent yields. Moreover, the developed methodology does not require any catalyst, acidic medium or high temperature which demonstrates the broad scope of the methodology.

## Conflicts of interest

There are no conflicts to declare.

## Acknowledgements

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