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Samarium-mediated mild and facile method for the synthesis of amides

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ABSTRACT

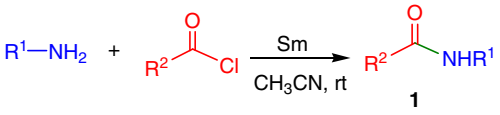
Samarium-mediated facile method for the formation of amide bonds by the reaction of acyl chlorides and amines is described. The reaction afforded high yields of the desired amides under mild and neutral conditions.

The amide bond is an important functional group in organic chemistry.[1] It plays a major role in the elaboration and composition of biological and chemical systems. Amides are traditionally synthesized by the reaction of amines with activated carboxylic acid derivatives.[2] Alternative approaches on the synthesis of amides are the Staudinger reaction,[3] the Schmidt reaction,[4] Beckmann rearrangement,[5] aminocarbonylation of haloarenes,[6] alkenes[7] and alkynes,[8] oxidative amidation of aldehydes,[9] hydrative amide synthesis with alkynes[10], and the amidation of thio acids with azides.[11] However, most of methods require the presence of a base or an acid and generate larger amounts of by-products. Thus, the synthesis of amides under neutral conditions and without the generation of by-products is a challenging goal.[12]

The synthesis of amides with acyl chlorides is generally conducted in the presence of tertiary amines, but the use of tertiary amines produces serious problem in the synthesis of peptide, such as epimerization or premature deblocking of protecting groups.[13] However, these problems can be solved easily by using a metal instead of tertiary amines.[14] The chemistry of samarium metal in organic synthesis has recently received increasing attention owing to its stability in water and air.[15] Our interests in utilizing samarium metal in organic synthesis have led us to investigate samarium-promoted reactions for the synthesis of amides.[16] As a continuation of our interest in samarium metal chemistry, herein we wish to report a mild and convenient methodology for the formation of amide bonds from amines and acid chlorides by using metallic samarium as a promoter under neutral conditions (Scheme 1).

Treatment of aniline with benzoyl chloride in the presence of samarium metal at room temperature resulted in the formation of the corresponding amide in 93% yield (Table 1, entry 1). The reactivity of acyl chlorides under the present conditions was examined and the results are summarized in Table 1. As can be seen from Table 1, The acyl chloride reacted smoothly with aliphatic primary and secondary amines or aromatic amines to afford the amides in excellent yields. Aromatic acyl chlorides with electron-withdrawing and electron-donating groups gave high yields of the amides (Table 1, entry 1–4, 8–11, 16–18). Aliphatic acyl chlorides also provided higher yields of the amides than aromatic acyl chlorides. The reaction proceeded efficiently even with highly sterically hindered pivaloyl chloride in high yields of the amides at room temperature (Table 1, entry 6). Especially, the reaction of pivaloyl chloride with sterically hindered *t*-BuNH$_2$ can also be performed smoothly to give the amide in 96% yield (Table 1, entry 15). However, the same reaction was carried out under the same reaction conditions using zinc or indium metal to give the amide in low yields.[14b] More importantly, the reaction proceeded smoothly in air at room temperature and carbon–carbon double bond, chloro, alkoxyl, ester, and nitro groups of the substrates were not affected under the reaction conditions. There was no need to activate samarium metal with pretreatment. The commercially available samarium was active enough to accomplish the reactions, and epimerization does not occur under the reaction conditions (Table 1, entry 21). We have also investigated the possibility that samarium could function catalytically. However, high yields of amides can only be achieved at least using 2/3 equiv of samarium. The control experiments were conducted by the reaction of aniline with benzoyl chloride using 1/3 equiv of samarium, or without samarium to give the corresponding product in 69% and trace yields, respectively.

In conclusion, we have developed a simple and facile method for the formation of amide bonds by using samarium metal as a promoter under mild and neutral reaction conditions.[17] The advantages of the present method include high yields of products, simple experimental procedure, avoiding poisonous solvent, no need for dry solvent and no activation or pretreatment of samarium metal,



1

Scheme 1. The synthesis of various amides.

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Table 1
Synthesis of amides from acyl chlorides and amines by using samarium metal at room temperature[a]

Entry	Amine	Acyl chloride	Time (h)	Yield[b] (%)
1	$PhNH_2$	PhCOCl	2	93 (**1a**)
2	$PhNH_2$	*p*-ClC_6H_4COCl	2	95 (**1b**)
3	$PhNH_2$	*p*-$MeOC_6H_4COCl$	3	90 (**1c**)
4	$PhNH_2$	*p*-MeC_6H_4COCl	2	92 (**1d**)
5	$PhNH_2$	$PhCH_2COCl$	3	99 (**1e**)
6	$PhNH_2$	*t*-BuCOCl	2	97 (**1f**)
7	$PhNH_2$	$CH_3(CH_2)_6COCl$	2	97 (**1g**)
8	*p*-$MeOC_6H_4NH_2$	*p*-ClC_6H_4COCl	4	95 (**1h**)
9	*p*-$MeOC_6H_4NH_2$	*p*-$MeOC_6H_4COCl$	4	92 (**1i**)
10	*p*-$ClC_6H_4NH_2$	*p*-$MeOC_6H_4COCl$	4	91 (**1j**)
11	*p*-$MeC_6H_4NH_2$	*p*-$MeOC_6H_4COCl$	3	89 (**1k**)
12	*t*-$BuNH_2$	PhCOCl	3	85 (**1l**)
13	*t*-$BuNH_2$	*p*-ClC_6H_4COCl	3	87 (**1m**)
14	*t*-$BuNH_2$	*p*-$O_2NC_6H_4COCl$	2	92 (**1n**)
15	*t*-$BuNH_2$	*t*-BuCOCl	2	96 (**1o**)
16	*c*-Hexylamine	PhCOCl	3	94 (**1p**)
17	*c*-Hexylamine	*p*-MeC_6H_4COCl	2	92 (**1q**)
18	*c*-Hexylamine	PhCH=CHCOCl	2	96 (**1r**)
19	Morpholine	PhCOCl	4	82 (**1s**)
20	Morpholine	*t*-BuCOCl	3	85 (**1t**)
21	(S)-Ethyl 2-amino-3-phenylpropanoate	PhCOCl	2	83 (**1u**)

[a] Reaction conditions: samarium (4 mmol), acid chloride (6 mmol), amine (6 mmol).
[b] Isolated yields.

the applicability of a wider range of substrates and the neutral conditions. The procedure will also provide alternative opportunities for the preparation of the amides.

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17. *General procedure for the homocoupling of terminal alkynes*: To a stirred suspension of samarium powder (4 mmol) in CH_3CN (10 mL) were added acid chloride (6 mmol) and amine (6 mmol) successively at room temperature. The reaction mixture was stirred at room temperature for given time (Table 1). The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was filtered and the solid was washed with dichloromethane. The solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (5:1) to afford the corresponding amides **1** in excellent yields. All the compounds were reported in the literatures and were identified by melting points, IR, ^1H NMR, and ^{13}C NMR spectra.
Compound **1a**: mp 162.7–163.2 °C (lit.[18] 163 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.89 (s, 1H), 7.86 (d, J = 9.0 Hz, 2H), 7.66–7.15 (m, 8H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ: 165.9, 137.6, 135.1, 131.7, 129.3, 129.1, 128.5, 126.9, 124.7, 120.0, 118.4, 114.9; IR (KBr) ν: 3344, 1656, 1599 cm^{-1}.
Compound **1b**: mp 200.8–201.1 °C (lit.[19] 199.5–200 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.94 (s, 1H), 7.85 (d, J = 9.0 Hz, 2H), 7.66–7.12 (m, 7H); IR (KBr) ν: 3351, 1654, 1598 cm^{-1}.
Compound **1c**: mp.173.4–174.1 °C (lit.[20] 170–171 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.85 (d, J = 12.0 Hz, 2H), 7.82 (s, 1H), 7.64–6.95 (m, 7H), 3.88 (s, 3H); IR (KBr) ν: 3338, 2959, 1656, 1599 cm^{-1}.
Compound **1d**: mp 141–142 °C (lit.[21] 145 °C). ^1H NMR (300 MHz, $CDCl_3$) δ: 7.89 (s, 1H), 7.78–7.14 (m, 9H), 2.42 (s, 3H); IR (KBr) ν: 3340, 2958, 1657, 1597 cm^{-1}.
Compound **1e**: mp 118.5-119.3 °C (lit.[22] 117–118 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.89 (s, 1H), 7.43–7.07 (m, 10H), 3.73 (s, 2H); IR (KBr) ν: 3285, 1658, 1601 cm^{-1}.
Compound **1f**: mp 132–133 °C (lit.[23] 132 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.46 (m, 2H), 7.23 (m, 3H), 7.05 (m, 1H), 1.28(s, 9H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ: 176.5, 138.1, 128.9, 124.1, 120.2, 39.5, 27.6; IR (KBr) ν: 3314, 2986, 2963, 2931, 1654, 1596 cm^{-1}.
Compound **1g**: mp 50–52 °C (lit.[24] 48–51 °C); ^1H NMR (500 MHz, $CDCl_3$) δ: 8.03 (br s, 1H), 7.53 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 15.5 Hz, 2H), 7.06 (t, J = 7.0 Hz, 1H), 2.31 (t, J = 15.0 Hz, 2H), 1.71–1.66 (m, 2H), 1.34–1.21 (m, 8H), 0.87 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 172.2, 138.2, 128.9, 124.2, 120.2, 37.8, 31.8, 29.3, 29.1, 25.8, 22.7, 14.1; IR (KBr) ν: 3313, 2925, 2850, 1656, 1600 cm^{-1}.
Compound **1h**: mp 207.3–207.9 °C (lit.[25] 209.3–210 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.81 (d, J = 12.0 Hz, 2H), 7.67 (s, 1H), 7.54–7.45 (m, 4H), 6.93 (d, J = 9.0 Hz, 2H), 3.82 (s, 3H); IR (KBr) ν: 3346, 2957, 1648, 1599 cm^{-1}.
Compound **1i**: mp 201–202 °C (lit.[26] 200–203 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.84 (d, J = 12.0 Hz, 2H), 7.65 (s, 1H), 7.53 (d, J = 12.0 Hz, 2H), 7.0–6.89 (m, 4H), 3.88 (s, 1H), 3.83 (s, 1H); IR (KBr) ν: 3327, 2955, 1647, 1605 cm^{-1}.
Compound **1j**: mp 205–206 °C (lit.[25] 206.2–208.5 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.80 (d, J = 9.0 Hz, 2H), 7.65 (s, 1H), 7.54–7.44 (m, 4H), 6.91 (d, J = 9.0 Hz, 2H), 3.82 (s, 3H); IR (KBr) ν: 3355, 2969, 1655, 1606 cm^{-1}.
Compound **1k**: mp 153.1–153.8 °C (lit.[27] 156–157 °C). ^1H NMR (300 MHz, $CDCl_3$) δ: 7.88 (s, 1H), 7.81 (d, J = 9.0 Hz, 2H), 7.50 (d, J = 12.0 Hz, 2H), 7.13 (d, J = 9.0 Hz, 2H), 6.92 (d, J = 9.0 Hz, 2H), 3.82 (s, 2H), 2.32 (s, 3H); IR (KBr) ν: 3340, 2962, 1652, 1601 cm^{-1}.
Compound **1l**: mp 134–135 °C (lit.[28] 134–135 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.70 (d, J = 9.0 Hz, 2H), 7.44–7.39 (m, 3H), 5.96 (s, 1H), 1.46 (s, 9H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ: 166.8, 136.0, 131.2, 128.3, 126.7, 51.7, 28.8; IR (KBr) ν: 3326, 2979, 1636, 1579 cm^{-1}.
Compound **1m**: mp 136–137 °C (lit.[29] 137–138 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 7.81 (d, J = 9.0 Hz, 2H), 7.54–7.44 (m, 2H), 6.0 (s, 1H), 1.47 (s,9H); IR (KBr) ν: 3357, 2979, 1636, 1592 cm^{-1}.
Compound **1n**: mp 158–159 °C (lit.[29] 159–160 °C); ^1H NMR (300 MHz, $CDCl_3$) δ: 8.24 (d, J = 9.0 Hz, 2H), 7.87 (d, J = 9.0 Hz, 2H), 6.09 (s, 1H), 1.49 (s, 9H); IR (KBr) ν: 3308, 2969, 1638, 1600 cm^{-1}.
Compound **1o**: mp 117–118 (lit.[30] 118–118.7 °C); ^1H NMR (300 MHz, $CDCl_3$) δ:

5.30(s, 1H), 1.26 (s, 9H), 1.12 (s, 9H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ: 178.9, 52.3, 40.2, 30.1, 28.9; IR (KBr) ν: 3376, 2967, 2931, 1638 cm^{-1}.

Compound **1p**: mp 147–148 °C (lit.[31] 148–149 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.76 (d, J = 12.0 Hz, 2H), 7.46 (m, 3H), 5.97 (s, J = 12.0 Hz, 1H), 4.02–3.98 (m, 1H), 2.07–1.20 (m, 10H); IR (KBr) ν: 3308, 2935, 1628, 1577 cm^{-1}.

Compound **1q**: mp 150–151 °C (lit.[32] 152–153 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.65 (d, J = 12.0 Hz, 2H), 7.23 (d, J = 12.0 Hz, 2H), 6.05 (d, J = 9.0 Hz, 1H), 3.98–3.95 (m, 1H), 3.38 (s, 3H), 2.04–1.16 (m, 10H); IR (KBr) ν: 3324, 2973, 2937, 1627, 1573 cm^{-1}.

Compound **1r**: mp 176–177 °C (lit.[33] 179.2–180.2 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.61 (d, J = 18.0 Hz, 1H), 7.51–7.48 (m, 2H), 7.36–7.33 (m, 3H), 6.38 (d, J = 18.0 Hz, 1H), 3.93–3.90 (m, 1H), 2.02–1.13 (m, 10H); IR (KBr) ν: 3277, 2916, 1654, 1618, 1496 cm^{-1}.

Compound **1s**: mp 74–75 °C (lit.[34] 73–74 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.46 (m, 5H), 3.77–3.45 (m, 8H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ: 170.2, 135.1, 129.6, 128.6, 127.0, 66.7, 43.2; IR (KBr) ν: 2855, 1628, 1579 cm^{-1}.

Compound **1t**: mp 72.1–72.8 °C (lit.[23] 73 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.50 (m, 8H), 1.12 (s, 9H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ: 177.6, 68.1, 47.3, 40.7, 29.5; IR (KBr) ν: 2966, 2855, 1615, 1419 cm^{-1}.

Compound **1u**: mp 104–105 °C (lit.[35] 100–102 °C); $[\alpha]_D^{20}$ +0.20° (c 0.56, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.51 (d, J = 2.0 Hz, 2H), 7.50–7.14 (m, 8H), 6.61 (d, J = 7.0 Hz, 1H), 5.09–5.05 (m, 1H), 4.21 (q, J = 7.0 Hz, 2H), 3.31–3.22 (m, 2H), 1.28 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 171.8, 166.9, 136.0, 134.1, 131.9, 129.6, 128.8, 128.7, 127.3, 127.1, 61.8, 53.7, 38.1, 14.3; IR (KBr) ν: 3363, 3069, 2960, 2932, 1749, 1641, 1602 cm^{-1}.

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