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# Rhenium complex-catalyzed acylative cleavage of ethers with acyl chlorides

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ABSTRACT

It was found that rhenium complex was an efficient catalyst for the acylative cleavage of C—O bond of ethers with acyl chlorides. When acyclic ethers were allowed to react with acyl chlorides in the presence of a catalytic amount of $ReBr(CO)_5$, acylative cleavage of C—O bond of acyclic ethers smoothly proceeded to give the corresponding esters in moderate to good yields. Similarly, cyclic ethers were acylative cleaved by acyl chlorides to give the corresponding chloro substituted esters in good yields by the use of $Re_2O_7$ catalyst.

## 1. Introduction

The cleavage of a C—O bond of ethers is a versatile reaction in organic synthesis and many methods have been developed.[1] Among them, the acylative cleavage of ethers with acyl chlorides is one of the attractive methods for the preparation of esters. Thus, it is of great interest in the development of the convenient and practical catalytic methods for the transformation of ethers into esters. There are some reports on the acylative cleavage of ethers with acyl chlorides using Lewis acids,[2] groups 5 and 6 transition metal complexes[3] and metals, such as Al[4] and Zn,[5] Pt,[6] and iodine.[7] However, these methods involved some disadvantages, such as the use of toxic, expensive, and unstable reagents or catalysts, the formation of mixture of products, and low yields of products.

Recently, Narasaka and our groups have shown the hitherto unknown capacity of $ReBr(CO)_5$, which is a relatively air-stable and water-tolerant compound, as an efficient catalyst for the various carbon—carbon formation.[8,9] During the course of our study on the catalytic use of the rhenium complex, it was found that the $ReBr(CO)_5$ complex acts as the catalyst on the acylative cleavage of the C—O bond of ethers with acyl chlorides giving the corresponding esters in moderate to good yields.[8f] Here, we would like to report the full results on the rhenium complex-catalyzed acylative cleavage of the C—O bond of acyclic and cyclic ethers with acyl chlorides.

## 2. Results and discussion

### 2.1. Rhenium-catalyzed reaction of acylic ethers with acyl chlorides

To determine the optimized reaction conditions, diisopropyl ether (**1a**) was treated with benzoyl chloride (**2a**) in the presence of a catalytic amount of rhenium complex under various reaction conditions and these results are shown in Table 1. When **1a** was allowed to react with **2a** in the presence of a catalytic amount of $ReBr(CO)_5$ (2.5 mol %) at 80 °C for 2 h in 1,2-dichloroethane solvent, the acylative cleavage of C—O bond of **1a** smoothly proceeded to give isopropyl benzoate (**3a**) in 90% yield (entry 4 in Table 1). The acylated product, **3a**, was not obtained when the reaction was carried out without rhenium complex or with lower reaction temperatures (25 °C and 60 °C) (entries 1–3). Even when the amount of $ReBr(CO)_5$ used as a catalyst was decreased; ester **3a** was obtained in 87% yield (entry 5). Although acylative cleavage of **1a** with **2a** was occurred, even using hexane and benzene instead of dichloroethane as the solvent, the best yield was obtained in dichloroethane solvent (entries 4, 6, and 7). The use of coordinating solvent, such as acetonitrile caused a distinct decrease in the yield of **3a** (entry 8). The other rhenium complexes, $ReCl(CO)_5$, $Re_2(CO)_{10}$, $CH_3ReO_3$, $ReCl_5$, and $Re_2O_7$, also functioned as a catalyst; however, the yields of **3a** slightly decreased (entries 9–13).

The reaction of diisopropyl ether (**1a**) and various acyl and aroyl chlorides **2** was carried out under the same reaction conditions as entry 4 in Table 1, and these results are shown in Table 2. The acylative cleavage of **1a** with various aroyl chlorides having

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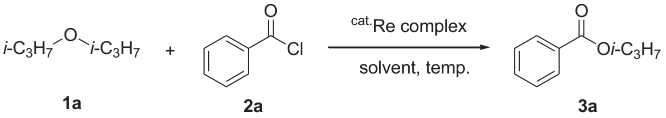
**Table 1**
Rhenium-catalyzed reaction of diisopropyl ether (**1a**) with benzoyl chloride (**2a**) under various reaction conditions[a]



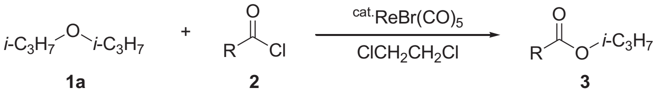
| Entry | Re complex | Solvent | Temp (°C) | Yield[b] (%) |
|---|---|---|---|---|
| 1 | None | $ClCH_2CH_2Cl$ | 80 | 0 |
| 2 | $ReBr(CO)_5$ | $ClCH_2CH_2Cl$ | 25 | 0 |
| 3 | $ReBr(CO)_5$ | $ClCH_2CH_2Cl$ | 60 | Trace |
| 4 | $ReBr(CO)_5$ | $ClCH_2CH_2Cl$ | 80 | 90 (76) |
| 5[c] | $ReBr(CO)_5$ | $ClCH_2CH_2Cl$ | 80 | 87 |
| 6 | $ReBr(CO)_5$ | $C_6H_{14}$ | 80 | 81 |
| 7 | $ReBr(CO)_5$ | $C_6H_6$ | 80 | 89 |
| 8 | $ReBr(CO)_5$ | $CH_3CN$ | 80 | 0 |
| 9 | $ReCl(CO)_5$ | $ClCH_2CH_2Cl$ | 80 | 74 |
| 10 | $Re_2(CO)_{10}$ | $ClCH_2CH_2Cl$ | 80 | 72 |
| 11 | $CH_3ReO_3$ | $ClCH_2CH_2Cl$ | 80 | 84 |
| 12 | $ReCl_5$ | $ClCH_2CH_2Cl$ | 80 | 69 |
| 13 | $Re_2O_7$ | $ClCH_2CH_2Cl$ | 80 | 80 |

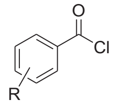
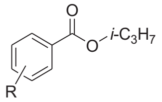
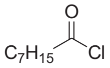
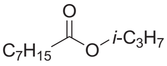
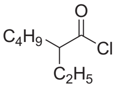
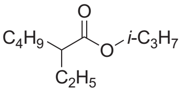
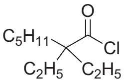
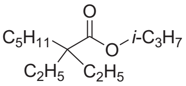
[a] Reaction condition: **1a** (0.6 mmol), **2a** (0.5 mmol), rhenium complex (2.5 mol %), and solvent (3.0 mL) for 2 h.
[b] Yields were determined by GC based on **2a**. The number in parenthesis shows the isolated yield.
[c] $ReBr(CO)_5$ (1.0 mol %) was used.

electron donating groups and halogen atom on aromatic ring proceeded to give the corresponding esters, **3b**,**e**,**f**, in moderate to good yields (entries 2, 5, and 6). For the reaction of 2- and 3-methylbenzoyl chloride, the yields of esters, **3c**,**d**, were slightly decreased (entries 3 and 4). In contrast, in the case of 4-nitrobenzoyl chloride substituted an electron withdrawing group, isopropyl 4-nitrobenzoate (**3g**) was not formed (entry 7). For the reaction, there is no significant reactivity difference between the aroyl and acyl chlorides. The reaction of **1a** with octanoyl chloride and 2-ethylhexanoyl chloride proceeded to give the esters **3h**,**i** in 77 and 72% yields, respectively (entries 8 and 9). When the 2,2-diethylheptanoyl chloride, which have a sterically hindered group, was used as an acyl chloride, no acylative cleavage product **3j** was formed (entry 10).

The representative results for the reaction of various ethers **1** with benzoyl chloride (**2a**) are shown in Table 3. For the reaction of dioctyl ether with **2a**, the yield of octyl benzoate (**3k**) was only 8% yield, however, the yield of the **3k** was improved by extending the reaction time and increasing the amount of the catalyst (entry 2). Allyl benzoate (**3l**) was formed by the reaction of diallyl ether with **2a**, in 68% yield (entry 3). For the reaction of dibenzyl ether, various complicated products including aromatic ketones, Friedel–Crafts acylation products, were formed (entry 4). For the reaction of the unsymmetric ethers, such as *tert*- and *sec*-butyl methyl ether and *tert*- and *sec*-butyl ethyl ether, it is interesting to note that the C–O bond of these ethers was selectively cleaved to give methyl and ethyl benzoate, **3n** and **3o**, in 66, 43, 65, and 75% yields, respectively (entries 5, 6, 8, and 9). In the case of *n*-butyl methyl ether, methyl benzoate (**3n**) was formed in low yield (27%) together with the formation of *n*-butyl benzoate (5%) under the longer reaction time and increasing the catalyst loading conditions.

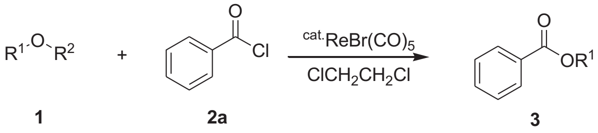
**Table 2**
Rhenium-catalyzed reaction of diisopropyl ether (**1a**) with acyl chlorides **2**[a]



| Entry | Acyl chloride | Product | | Yield[b] (%) |
|---|---|---|---|---|
| |  |  | | |
| 1 | R=H | | **3a** | 76 |
| 2[c] | 4-$CH_3$ | | **3b** | 66 |
| 3[c] | 3-$CH_3$ | | **3c** | 56 |
| 4[c] | 2-$CH_3$ | | **3d** | 41 |
| 5 | 4-$OCH_3$ | | **3e** | 87 |
| 6 | 4-Cl | | **3f** | 93 |
| 7 | 4-$NO_2$ | | **3g** | 0 |
| 8 |  |  | **3h** | 77 |
| 9[d] |  |  | **3i** | 72 |
| 10 |  |  | **3j** | 0 |

[a] Reaction condition: **1a** (0.6 mmol), **2** (0.5 mmol), $ReBr(CO)_5$ (2.5 mol %), and $ClCH_2CH_2Cl$ (3.0 mL) at 80 °C for 2 h.
[b] Isolated yields based on **2**.
[c] For 12 h.
[d] For 9 h.

**Table 3**
Rhenium-catalyzed reaction of ether **1** with benzoyl chloride (**2a**)[a]



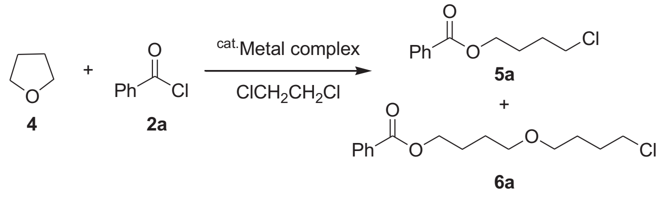
| Entry | $R^1$ | $R^2$ | Product | Yield[b] (%) |
|---|---|---|---|---|
| 1 | $i$-$C_3H_7$ | $i$-$C_3H_7$ | **3a** | 76 |
| 2[c,d] | $C_8H_{17}$ | $C_8H_{17}$ | **3k** | 35 |
| 3 | $CH_2{=}CHCH_2$ | $CH_2{=}CHCH_2$ | **3l** | 68 |
| 4 | $PhCH_2$ | $PhCH_2$ | **3m** | Trace |
| 5 | $CH_3$ | $t$-$C_4H_9$ | **3n** | 66 |
| 6 | $CH_3$ | $s$-$C_4H_9$ | **3n** | 43 |
| 7[d,e] | $CH_3$ | $C_4H_9$ | **3n** | 27[f] |
| 8 | $C_2H_5$ | $t$-$C_4H_9$ | **3o** | 65 |
| 9 | $C_2H_5$ | $s$-$C_4H_9$ | **3o** | 75 |

[a] Reaction condition: **1** (0.6 mmol), **2a** (0.5 mmol), $ReBr(CO)_5$ (2.5 mol %), and $ClCH_2CH_2Cl$ (3.0 mL) at 80 °C for 2 h.
[b] Isolated yields based on **2a**.
[c] For 5 h.
[d] $ReBr(CO)_5$ (12.5 mol %) was used.
[e] For 20 h.
[f] $^1H$ NMR yield. *n*-Butyl benzoate (5%) was also formed.

## 2.2. Rhenium-catalyzed reaction of cyclic ethers with acyl chlorides

Next, we examined the acylative cleavage of C–O bond of cyclic ethers with benzoyl chloride (**2a**) (Table 4). When **2a** was allowed to react with tetrahydrofuran (THF) (**4**) in the presence of a catalytic amount of $ReBr(CO)_5$ in 1,2-dichloroethane solvent, acylative cleavage of C–O bond of **4** with **2a** smoothly proceeded to give 4-chlorobutyl benzoate (**5a**) in 76% yield along with the formation of a small amount of 4-chlorobutoxy butyl benzoate (**6a**), 1:2 coupling product of **2a** and **4** (entry 1). In order to improve the yield of **5a**, **2a** was allowed to react with **4** using the other rhenium complexes instead of $ReBr(CO)_5$. In the contrast to that of acyclic ethers, the best yield was observed by the use of $Re_2O_7$ as the catalyst (entries 4–6 and 9).

**Table 4**
Metal complex-catalyzed reaction of THF (**4**) with benzoyl chloride (**2a**)[a]



| Entry | Metal complex | THF (**4**) (mmol) | Yield[b] (%) | |
|---|---|---|---|---|
| | | | **5a** | **6a** |
| 1 | $ReBr(CO)_5$ | 0.5 | 76 | 1 |
| 2 | $ReBr(CO)_5$ | 1.2 | 45 | 27 |
| 3 | $ReBr(CO)_5$ | 3.0 | 52 | 38 |
| 4 | $ReCl(CO)_5$ | 0.5 | 86 | 0 |
| 5 | $CH_3ReO_3$ | 0.5 | 90 | 0 |
| 6 | $Re_2O_7$ | 0.5 | 97 (92) | 2 |
| 7 | $Re_2O_7$ | 1.2 | 82 | 8 |
| 8 | $Re_2O_7$ | 3.0 | 69 | 14 |
| 9 | $Re_2(CO)_{10}$ | 0.5 | 17 | 14 |
| 10[c] | $Re_2(CO)_{10}$ | 1.2 | 44 | 26 |
| 11[c] | $Re_2(CO)_{10}$ | 3.0 | 30 | 32 |
| 12 | $MnBr(CO)_5$ | 1.2 | 7 | Trace |
| 13 | $MnBr(CO)_5$ | 3.0 | 26 | 3 |
| 14 | $ZnCl_2$ | 1.2 | 57 | 0 |
| 15 | $ZnCl_2$ | 3.0 | 69 | 0 |
| 16 | $FeCl_3$ | 1.2 | 21 | 2 |
| 17 | $FeCl_3$ | 3.0 | 17 | 2 |
| 18 | $BiCl_3$ | 1.2 | 75 | 14 |
| 19 | $BiCl_3$ | 3.0 | 71 | 7 |

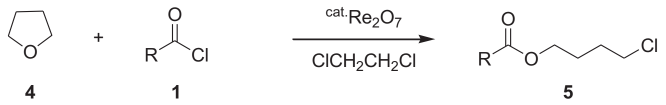
[a] Reaction condition: **2a** (0.6 mmol), metal complex (2.5 mol % for entries 1–5 and 7–19 and 1.3 mol % for entries 6–11), $ClCH_2CH_2Cl$ (3.0 mL) at 80 °C for 12 h.
[b] Yields were determined by GC based on **4** for entries 1, 4, 5, 6, and 9 and **2a** for entries 2, 3, 7, 8, and 10–19. The number in parenthesis shows the isolated yield.
[c] For 24 h.

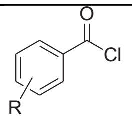
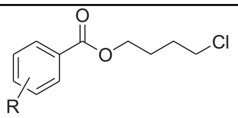
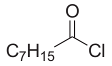
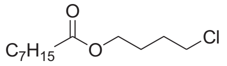
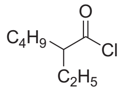
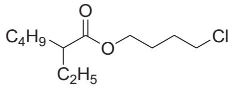
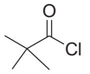
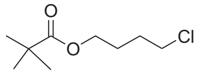
It has been reported that the THF (**4**) was acylative cleaved by acyl halides in the presence of various Lewis acids giving the corresponding 4-chloro substituted esters.[2–7] However, to the best of our knowledge, there are only a few reports on the formation of 1:2 coupling product of acyl halides and **4**.[3b,10] Thus, in order to improve the selectivity of **6a**, THF (**4**) was treated with benzoyl chloride (**2a**) in the presence of various Lewis acids. The selectivity of **6a** was improved by the increasing the amount of **4**, when $ReBr(CO)_5$, $Re_2O_7$,[11] and $Re_2(CO)_{10}$ were used as a catalyst (entries 2, 3, 7, 8, 10, and 11). The highest yield of **6a** was attained by the use of $ReBr(CO)_5$. The use of $MnBr(CO)_5$ catalyst led to the distinct decrease of the yields of both **5a** and **6a** (entries 12 and 13). In the case of $ZnCl_2$ and $FeCl_3$, which are often used as a catalyst, the yields of **6a** were very low (entries 14–17). In the case of $BiCl_3$ (entries 18 and 19), **6a** was obtained in 14% yield when 2.0 equiv amount of **4** was used.

The reaction of cyclic ethers with various acyl and aroyl chlorides carried out under the same reaction conditions as that of entry 6 in Table 4, and the representative results are shown in Table 5. On the reaction of aroyl chlorides except 4-nitrobenzoyl chloride, the acylative cleavage of THF (**4**) efficiently proceeded to give the corresponding 4-chlorobutyl benzoates **5b**–**f** in good to excellent yields (entries 2–7). Similarly, 4-chloro substituted esters **5h**–**j** were obtained by the reaction of **4** with the corresponding acyl chlorides in moderate to good yields (entries 8–10).

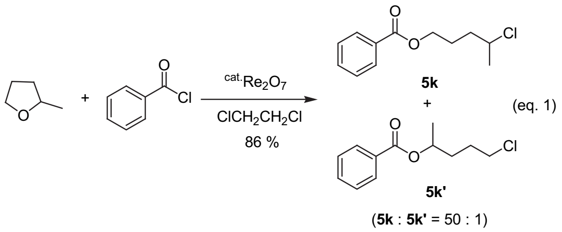
On the C–O bond cleavage of 2-methyltetrahydrofuran, the C–O bond attached to the methyl substituent was predominantly cleaved to form 2-chloropentyl benzoate (**5k**) in 86% yield with the formation of a small amount of 5-chloropentan-2-yl benzoate (**5k′**) (Eq. 1). In the case of 3-phenyltetrahydrofuran, 4-chloro-3-phenylbutyl benzoate (**5l**) was predominantly obtained in 73% yield along the formation of 4-chloro-2-phenylbutyl benzoate (**5l′**) (7%) (Eq. 2). This rhenium complex catalytic system is also effective for the acylative cleavage of the C–O bond of 3-, 4-, and six-membered cyclic ethers to give the 2-, 3-, and 5-chloro substituted esters in 46, 70, and 52% yield, respectively (Eqs. 3–5).

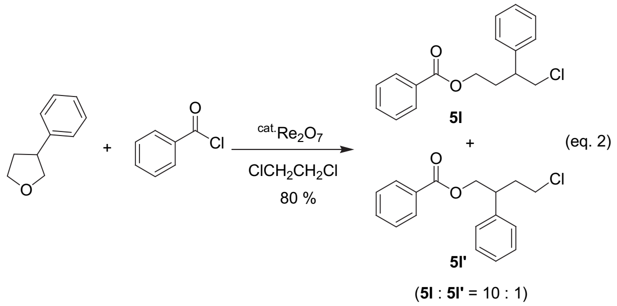
**Table 5**
Rhenium-catalyzed reaction of THF (**4**) with acyl chlorides **1**[a]

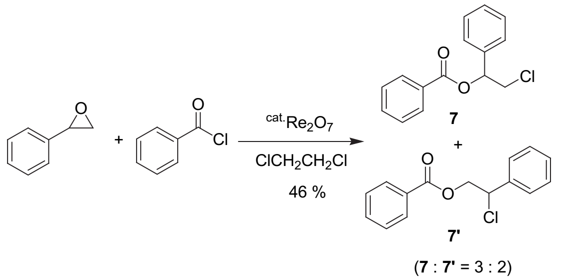


| Entry | Acyl chloride **1** | Product **5** | | Yield[b] (%) |
|---|---|---|---|---|
| |  |  | | |
| 1 | R=H | | **5a** | 92 |
| 2 | 4-$CH_3$ | | **5b** | 85 |
| 3 | 3-$CH_3$ | | **5c** | 90 |
| 4 | 2-$CH_3$ | | **5d** | 78 |
| 5 | 4-$OCH_3$ | | **5e** | 65 |
| 6 | 4-Cl | | **5f** | 69 |
| 7 | 4-$NO_2$ | | **5g** | 0 |
| 8 |  |  | **5h** | 78 |
| 9 |  |  | **5i** | 83 |
| 10 |  |  | **5j** | 66 |

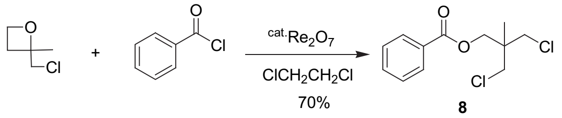
[a] Reaction condition: **1** (0.6 mmol), **4** (0.5 mmol), $Re_2O_7$ (1.3 mol %), and $ClCH_2CH_2Cl$ (3.0 mL) at 80 °C for 12 h.
[b] Isolated yields based on **4**.



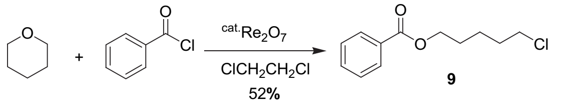




(eq. 3)



(eq. 4)



(eq. 5)

For the reaction of dioctyl ether with **1a**, the formation of 1- and 2-chlorooctane and 1-octene were identified with GC analysis. In order to clarify the reaction pathway, a stoichiometric reaction of dioctyl ether with $ReBr(CO)_5$ was carried out at 80 °C. However, 1- and 2-chlorooctane and 1-octene were not formed and dioctyl ether was recovered. The results on the reaction of *tert*- and *sec*-butyl methyl ether, *tert*- and *sec*-butyl ethyl ether, and 2-methyltetrahydrofuran were consistent with the fact that C–O bond, which gives more stable carbocations species is cleaved. Narasaka and Kusama have already reported the rhenium complex-catalyzed the Friedel–Crafts acylation of aromatic compounds having an electron donating group, such as methoxy and methyl with acyl chlorides giving the corresponding alkyl aryl ketones.[8a] In the manuscript, they suggested that coordinatively unsaturated complex, $ReBr(CO)_4$, which was generated in situ the decarbonylation of CO coordinated with $ReBr(CO)_5$, acts as a Lewis acid catalyst.[12]

From these results, we proposed that the decarbonylation of CO coordinated with $ReBr(CO)_5$ to form the $ReBr(CO)_4$, was the first step at the catalytic reaction. Acyl and aroyl chlorides were reacted with the $ReBr(CO)_4$. The addition of generated species to the oxygen of ether **2** followed by the elimination of chloride anion produced the oxonium salt. The cleavage of the carbon–oxygen bond of oxonium salt gave the ester **3**. On the other hand, the reaction pathway including the generation of acyl cation ($RCO^+$) and aroyl cation ($ArCO^+$), which were generated by the reaction of acyl and aroyl chlorides and $ReBr(CO)_4$, can not be ruled out.

## 3. Conclusion

In summary, we developed a new catalytic method for the acylative and aroylative cleavage of the C–O bond of ethers with acyl and aroyl chlorides giving the esters in moderate to good yields.

## 4. Experimental section

### 4.1. Reagents

2-Ethylhexanoyl and 2,2-diethylheptanoyl chlorides were prepared by reaction of thiony chloride with the corresponding acids. 2,2-Diethylheptanoic acid was prepared by the Grignard reaction of ethyl propionate with ethyl magnesium bromide followed by the carboxylation with HCOOH in the presence of sulfonic acid in acetic acid solution.[13] 3-Phenyltetrahydrofuran was synthesized by the intramolecular cyclization of 2-phenyl-1,4-butanadiol, which was prepared by the reduction of 2-phenylsuccinic acid with $LiAlH_4$.[14,15] Other chemical agents were obtained commercially and were purified if necessary.

### 4.2. General procedure for rhenium-catalyzed reaction of acyclic ethers with acyl chlorides

A 1,2-dichloroethane (3.0 mL) solution of acyl chloride (0.6 mmol), ether (0.5 mmol), and $ReBr(CO)_5$ (2.5 mol %) was stirred under an atmosphere of nitrogen at 80 °C for 2 h. After the reaction was complete, $H_2O$ was added to the reaction mixture and extracted with ethyl acetate. The organic layer was dried with $MgSO_4$. The resulting mixture was filtered, and the filtrate was concentrated. Purification of the residue by silica gel column chromatography afforded ester. The structures of the products were assigned by their $^1H$ and $^{13}C$ NMR, and mass spectra. The product was characterized by comparing its spectral data with those of authentic sample or previous reports **3a**,[16] **3b**,[17] **3c**,[18] **3d**,[17] **3e**,[17] **3f**,[17] **3k**,[17] **3l**,[3] **3n**,[19] and **3o**.[19] The structures of the products (**3h** and **3i**) were assigned by their $^1H$ and $^{13}C$ NMR, IR, and mass spectrum.

*Compound* **3h**: $^1H$ NMR (270 MHz, $CDCl_3$) $\delta$ 4.99 (sept, $J$=6.3 Hz, 1H), 2.24 (t, $J$=7.6 Hz, 2H), 1.63–1.56 (m, 2H), 1.30–1.20 (m, 14H), 0.86 (t, $J$=6.8 Hz, 3H). $^{13}C$ NMR (68 MHz, $CDCl_3$) $\delta$ 173.5, 67.3, 34.7, 31.6, 29.0, 28.9, 25.0, 22.6, 21.8, 14.0. IR (KBr) 2929, 2857, 1735, 1467, 1375, 1178, 1110 cm$^{-1}$.

*Compound* **3i**: $^1H$ NMR (270 MHz, $CDCl_3$) $\delta$ 5.02 (sept, $J$=6.2 Hz, 1H), 2.24–2.14 (m, 1H), 1.68–1.42 (m, 4H), 1.27–1.20 (m, 10H), 0.84 (t, $J$=7.3 Hz, 6H). $^{13}C$ NMR (68 MHz, $CDCl_3$) $\delta$ 175.9, 67.1, 47.5, 31.8, 29.5, 25.5, 22.6, 21.8, 14.0, 11.8. IR (KBr) 2961, 2929, 2858, 1731, 1462, 1380, 1262, 1180, 1109, 808, 676 cm$^{-1}$.

### 4.3. General procedure for rhenium-catalyzed reaction of cyclic ethers with acyl chlorides

A 1,2-dichloroethane (3.0 mL) solution of acyl chloride (0.6 mmol), cyclic ether (0.5 mmol), and $Re_2O_7$ (1.3 mol %) was stirred under an atmosphere of nitrogen at 80 °C for 2 h. After the reaction was complete, $H_2O$ was added to the reaction mixture and extracted with ethyl acetate. The organic layer was dried with $MgSO_4$. The resulting mixture was filtered, and the filtrate was concentrated. Purification of the residue by silica gel column chromatography afforded ester. The product was characterized by comparing its spectral data with those of authentic sample or previous reports **5a**,[5c] **5b**,[17] **5c**,[2i] **5e**,[17] **5f**,[20] **5j**,[20] **5k**,[21] **6**,[3b] **7**,[22,23] **7'**,[22,23] and **9**.[3] The structures of the products (**5d**, **5h**, **5i**, **5l**, and **8**) were assigned by their $^1H$ and $^{13}C$ NMR, IR, and mass spectrum.

*Compound* **5d**: $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.85–7.82 (m, 2H), 7.37–7.26 (m, 2H), 4.35 (t, $J$=6.0 Hz, 2H), 3.62 (t, $J$=6.2 Hz, 2H), 2.41 (s, 3H), 1.99–1.91(m, 4H). $^{13}C$ NMR (100 MHz, $CDCl_3$) $\delta$ 166.7, 138.1, 133.7, 130.0, 128.2, 127.7, 126.6, 64.0, 44.5, 29.2, 26.1, 21.3. IR (KBr) 2960, 2873, 1783, 1718, 1602, 1575, 1488, 1458, 1383, 1293, 1254, 1142, 1081, 1051, 999, 738, 696, 654 cm$^{-1}$.

*Compound* **5h**: $^1H$ NMR (270 MHz, $CDCl_3$) $\delta$ 4.08 (t, $J$=5.8 Hz, 2H), 3.55 (t, $J$=5.9 Hz, 2H), 2.27 (t, $J$=7.3 Hz, 2H), 1.86–1.71 (m, 4H), 1.59 (t, $J$=7.0 Hz, 2H), 1.86–1.77 (m, 4H), 1.76–1.48 (m, 2H), 1.26 (br, 8H), 0.86 (t, $J$=6.2 Hz, 3H). $^{13}C$ NMR (68 MHz, $CDCl_3$) $\delta$ 173.8, 63.3, 44.4, 34.2, 31.6, 29.0, 28.8, 26.0, 24.9, 22.5, 14.0 (one signal is missing due to overlap). IR (KBr) 2928, 2856, 1736, 1462, 1166, 1105 cm$^{-1}$.

*Compound* **5i**: $^1H$ NMR (270 MHz, $CDCl_3$) $\delta$ 4.11 (t, $J$=5.8 Hz, 2H), 3.56 (t, $J$=6.1 Hz, 2H), 2.30–2.20 (m, 1H), 1.91–1.73 (m, 4H), 1.69–1.38 (m, 4H), 1.36–1.16 (m, 4H), 0.88 (t, $J$=7.6 Hz, 6H). $^{13}C$ NMR

(68 MHz, $CDCl_3$) $\delta$ 176.4, 63.1, 47.4, 44.4, 31.8, 29.6, 29.2, 26.1, 25.4, 22.6, 13.9, 11.8. IR (KBr) 2959, 2873, 1732, 1460, 1171 $cm^{-1}$.

*Compound* **5l**: $^1H$ NMR (270 MHz, $CDCl_3$) $\delta$ 7.98 (d, *J*=7.0 Hz, 2H), 7.54–7.24 (m, 8H), 4.53–4.41 (m, 2H), 3.56–3.48 (m, 1H), 3.42–3.29 (m, 2H), 2.46–2.05 (m, 2H). $^{13}C$ NMR (100 MHz, $CDCl_3$) $\delta$ 166.4, 139.9, 133.0, 128.8, 128.4, 127.9, 127.3, 68.3, 42.5, 42.2, 35.3.

*Compound* **8**: $^1H$ NMR ($CDCl_3$, 400 MHz): $\delta$ 8.04–8.01 (m, 2H), 7.61–7.57 (m, 1H), 7.48–7.44 (m, 2H), 4.31 (s, 2H), 3.68–3.62 (m, 4H), 1.22 (s, 3H). $^{13}C$ NMR ($CDCl_3$, 100 MHz): $\delta$ 165.9, 133.3, 129.5, 129.4, 128.4, 66.7, 48.0, 41.0, 18.8. IR (KBr): 3063, 2975, 1724, 1451, 1272, 1111, 710 $cm^{-1}$.

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## Supplementary data

Supplementary data for new compounds of $^1H$ and $^{13}C$ NMR spectra. Supplementary data can be found in the online version, at doi:10.1016/j.tet.2011.07.072.

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