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Convenient Syntheses of Carboxylic Esters and Thiol Esters Using Acid Chlorides and Zinc Chloride

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Reaction of acid chlorides with primary alcohols, secondary alcohols, and aryl alcohols in the presence of a catalytic amount of zinc chloride gave the corresponding esters in high yields, whereas the reaction with tertiary alcohols failed to give the esters due to the fast solvolytic reactions of tertiary alcohols with hydrogen chloride generated from the reaction. The use of molecular sieves as a scavenger for hydrogen chloride was found to be moderately effective in the reaction of mesityl chloride with tertiary alcohols. Reaction of acid chlorides with thiols in the presence of zinc chloride in acetonitrile proceeded cleanly, yielding the corresponding thiol esters in high yields.

The combination of an organic acid chloride or an organic acid anhydride and a Lewis acid is very useful and well-known reagent in synthetic organic chemistry and has enjoyed its role as a source of an acylium ion in cleavage of acyclic and cyclic ethers since 1901.^{1,2} Besides the cleavage of ethers, these types of reagents have been recently utilized in the synthesis of β,γ -unsaturated ketones³ and aroyl azides.⁴ Furthermore, the reaction of acid chlorides in the presence of zinc chloride with aldehydes and ketones has been reported.⁵ However, the reaction of acid chlorides in the presence of Lewis acid with alcohols or thiols has been largely ignored by organic chemists, although syntheses of relatively sterically hindered carboxylic esters from acid chlorides with alcohols in the presence of silver cyanide⁶ or acetic anhydride with *t*-butyl alcohol in the presence of zinc chloride⁷ have been reported.

In connection with our continuous study directed toward development of new synthetic methods by use of transition metal salts,⁸ we had occasion to study the reaction of pivaloyl chloride with a highly hindered mesitol in the presence of 0.5 equiv of cupric bromide in acetonitrile at room temperature. The reaction proceeded smoothly, yielding 92% of mesityl pivate in 3 h, whereas the reaction in the absence of cupric bromide did not occur to an observable extent.

Since this initial discovery we have examined a number

of reactions to optimize the reaction condition by using an equimolar mixture of pivaloyl chloride and mesitol as a model study. The relative effectiveness of various metal salts was examined in acetonitrile at room temperature and is indicated in Table 1. Although zinc chloride and silver tetrafluoroborate were the most effective among various metal salts employed, zinc chloride was the reagent of the choice in view of the ready availability and the cheapness of zinc chloride. Furthermore, the esterification was complete within 10 min in the presence of 0.1 equiv of zinc chloride, indicating that zinc chloride effectively catalyzes the reaction of acid chloride with alcohols. Cupric bromide and aluminum chloride were also effective but less effective than zinc chloride. Other metal salts such as cupric cyanide, cuprous iodide, and nickel bromide did not give good results, even after stirring at room temperature for 24 h. Cupric acetate was totally ineffective and mesitol was recorded unchanged.

Although a number of useful and reliable methods for the preparation of carboxylic esters have been reported,⁹ there are only several methods available in the literature for the preparation of sterically hindered esters.^{6,10} The results obtained here indicate that the present procedure is very useful for the preparation of sterically hindered esters. Thus, we have studied the reaction with structurally different acids and alcohols to determine the scope and limitations

of the present procedure. Several experimental results are summarized in Table 2. In general, the reaction was carried out with an equimolar mixture of an acid chloride and an alcohol in the presence of 0.1 equiv of zinc chloride in acetonitrile at room temperature.

First, the esterification of a highly hindered mesitoyl chloride with a variety of alcohols was studied. Reaction of mesitoyl chloride with methanol, isopropyl alcohol, phenol, and mesitol in the presence of 0.1 equiv of zinc chloride afforded the corresponding in essentially quantitative yields within 1 h at room temperature. Similar results were realized with pivaloyl chloride and caprylyl chloride. However, in the case of a highly hindered 2,6-di-*t*-butylphenol, the formation of a trace amount (<5%) of esters was indicated on tlc. Furthermore, difficulties were encountered in the cases of tertiary alcohols such as *t*-butyl alcohol and *t*-amyl alcohol. Reaction of mesitoyl chloride with an equimolar amount of *t*-butyl alcohol in the presence of 0.1 equiv of zinc chloride gave only 8% of *t*-butyl mesitoate in 1 h and similar results were obtained with pivaloyl chloride and caprylyl chloride. It is attributed to the fast solvolytic reactions of tertiary alcohols with hydrogen chloride generated from the reaction. The direct evidence for this assumption was obtained with 3-hydroxy-3-ethylheptane. The reaction of pivaloyl chloride with an equimolar amount of 3-hydroxy-3-ethylheptane in the presence of 0.1 equiv zinc chloride in acetonitrile at room temperature for 1.5 h afforded 3-chloro-3-ethylheptane in 51% yield.

In order to solve this problem, molecular sieves were added into the reaction mixture for absorption of hydrogen chloride.¹¹ When an excess amount of powered 4A molecular sieves was employed in the reaction, the reaction of mesitoyl chloride with *t*-butyl alcohol and *t*-amyl alcohol gave 71% of *t*-butyl mesitoate and 49% of *t*-amyl mesitoate, indicating the efficacy of molecular sieves as a scavenger for hydrogen chloride. Thus, the modified procedure may be useful for the preparation of sterically hindered esters under relatively mild conditions.

It is of interest to note that the reaction of most aroyl chlorides with alcohols in the presence of zinc chloride occurred rather slowly and the reaction depended critically on the electronic effects of the substituents on aroyl chlorides. Thus, reaction of *p*-methoxybenzoyl chloride with benzyl alcohol in the presence of 0.1 equiv of zinc chloride in acetonitrile gave 90% of benzyl *p*-methoxybenzoate in 1 h, whereas benzoyl chloride required 6 h for completion of the reaction. In the case of *p*-nitrobenzoyl chloride, the reaction proceeded to some extent, yielding only 23% of benzyl *p*-nitrobenzoate with recovery of 47% of *p*-nitrobenzoic acid in 12 h under the same conditions, indicating the importance of an acylium ion intermediate.^{10a}

Since carboxylic thiol esters have played an important role as active acylating agents in the synthesis of esters,¹² macrocyclic lactones,¹³ and ketones,¹⁴ we applied the present procedure for the preparation of thiol esters¹⁵ and we have found that the combination of acid chlorides and zinc chloride can be successfully utilized for the preparation of

various thiol esters.

The reaction was carried out under the same reaction conditions utilized in the preparation of carboxylic esters. The preparation of thiol esters has been performed on several structurally different acid chlorides utilizing primary, tertiary, and aryl thiols. Table 3 summarizes several experimental results. In general, the reaction proceeded rapidly and smoothly, yielding the corresponding thiol esters in high yields. For example, reaction of mesitoyl chloride with equimolar amounts of *n*-butyl mercaptan, *t*-butyl mercaptan, and thiophenol in the presence of 0.1 equiv of zinc chloride gave the corresponding thiol esters in high yields within 10 min at room temperature. However, the reaction of benzoyl chloride with *t*-butyl mercaptan in the presence of 0.2 equiv of zinc chloride did not proceed smoothly, yielding *S*-*t*-butyl benzothioate in 30% yield in 6 h at room temperature, whereas the reaction with *n*-butyl mercaptan was complete within 10 min.

Even though the present method has several limitations it should be useful for the preparation of carboxylic esters and thiol esters and has several advantages over previously known methods with respects to the high yield synthesis, the rapidity, and the simplicity. Although there are a number of synthetic methods to bring about such conversions, we consider the present method as useful addition to them.

Experimental Section

NMR spectra were recorded with a Varian T-60A spectrometer, and chemical shifts are expressed as δ units relative to tetramethylsilane. Infrared spectra were recorded on a Perkin-Elmer 267 and the frequencies are given in reciprocal centimeters. Melting points were determined on an Electrothermal melting point apparatus and are uncorrected. Reported boiling points are those observed during distillation with a Kugelrohr apparatus and are uncorrected. Analytical thin-layer chromatography was performed on precoated silica gel plates (0.25 mm, 60F-254, E. Merck) and silica gel (activity III, 0.4526, ICN) was used for column chromatogra-

TABLE 1: Effect of Metal Salts on the Rate of Ester Formation in CH_3CN^a

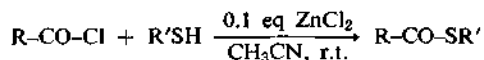
| Metal salt ^d | Time | Isolated yield, % ^e |
|---------------------------|--------|--------------------------------|
| CuBr_2 | 3 h | 92 |
| $\text{Cu}(\text{CN})_2$ | 24 h | 8 (84) |
| $\text{Cu}(\text{OAc})_2$ | 24 h | 0 (93) |
| CuI | 24 h | 20 (70) |
| ZnCl_2 | 10 min | 91 |
| NiBr_2 | 24 h | 35 (59) |
| AlCl_3 | 45 min | 89 |
| AgBF_4 | 10 min | 93 |

^a All reactions were carried out at room temperature using 1 equiv of pivaloyl chloride and 1.1 equiv of mesitol. ^b 0.5 equiv of metal salts were employed. ^c The numbers in parentheses indicate the isolated yield of the recovered mesitol.

TABLE 2: Ester Formation from Acid Chlorides and Alcohols in the Presence of 0.1 Equiv of ZnCl₂ in CH₃CN at Room Temperature

| Acid chloride | Alcohol | Time, h | Isolated yield, % ^a | BP, °C/mmHg ^b [mp, °C] | Lit. |
|------------------|-------------------------|---------|--------------------------------|--------------------------------------|-----------------------------|
| Pivaloyl | Mesitol | 0.1 | 93 | 89-93/0.95 | |
| | Cyclohexanol | 0.2 | 93 | 58-61/5.0 | |
| | 2-Octanol | 0.2 | 87 | 60-63/0.75 | 114/20 ¹⁶ |
| | β-Naphthol | 0.3 | 97 | [65-66] | [66-66.5] ^{10a} |
| | <i>t</i> -Amyl alcohol | 1.5 | 23 | 48-50/30 | 47-51/30 ^{8b} |
| | Benzyl alcohol | 0.1 | 95 | 62-65/1.3 | 60.5/1 ¹⁷ |
| Mesitoyl | Mesitol | 0.1 | 99 | [71-72] | [71-71.5] ^{10a} |
| | Methanol | 0.1 | 95 | 75-79/1.6 | 116/10 ^{10a} |
| | Isopropyl alcohol | 0.1 | 94 | 105-110/1.4 | 120-121/6-6.5 ¹⁸ |
| | Phenol | 0.1 | 92 | [36-37] | [37] ^{10a} |
| | <i>t</i> -Butyl alcohol | 1.0 | (71) | 93-97/1.6 | 114/2 ^{10a} |
| | <i>t</i> -Amyl alcohol | 1.0 | (49) | 92-95/1.0 | |
| Caprylyl | 3-Ethyl-3-pentanol | 4.0 | (53) | 137/6.5 | |
| | Mesitol | 0.1 | 97 | 165-169/1.4 | |
| Benzoyl | Cyclohexanol | 0.1 | 84 | 101-104/0.75 | 131-132/6 ¹⁹ |
| | Benzyl alcohol | 6 | 76 | 131-136/1.3 | 176-180/9 ²⁰ |
| 4-Methoxybenzoyl | Benzyl alcohol | 1 | 90 | 159-162/1.4 | 156-158/0.5 ²⁰ |
| 4-Nitrobenzoyl | Benzyl alcohol | 12 | 23 | [83-84] | [83-84.5] ²⁰ |

^aThe numbers in parentheses indicate the yields of esters using a 2:5 (w/w) mixture of mesitoyl chloride and 4A molecular sieves. ^bReported boiling points were those observed during distillation with a Kugelrohr apparatus and were uncorrected.

TABLE 3: Preparation of Thiol Esters from Acid Chlorides and Thiols in the Presence of 0.1 Equiv ZnCl₂ in CH₃CN at Room Temperature

| Acid Chloride (R) | Thiol (R') | Time, h | Yield, % ^a (RCOSR') | BP, °C/mmHg ^b [mp, °C] | Lit. |
|--|---|---------|-----------------------------------|--------------------------------------|-----------------------|
| 2,4,6-(CH ₃) ₃ -C ₆ H ₂ | (CH ₃) ₃ C | 0.1 | 95 | [42-43] | [42-42] ²¹ |
| | C ₆ H ₅ | 0.1 | 99 | [53-54] | [54-55] ²¹ |
| | CH ₃ (CH ₂) ₃ | 0.1 | 98 | 88-91/1.0 | |
| (CH ₃) ₃ C | (CH ₃) ₃ C | 0.2 | 93 | 61-63/20 | |
| | C ₆ H ₅ | 0.1 | 98 | 76-80/0.7 | 80/0.1 ^{15a} |
| CH ₃ (CH ₂) ₆ | (CH ₃) ₃ C | 0.1 | 90 | 61-64/1.3 | |
| | CH ₃ (CH ₂) ₃ | 0.1 | 97 | 74-77/1.2 | |
| C ₆ H ₅ | (CH ₃) ₃ C | 6 | 30 ^c | 66-68/1.0 | 110/28 ²² |
| | CH ₃ (CH ₂) ₃ | 0.2 | 82 | 88-91/0.7 | 125/5 ²³ |
| <i>p</i> -MeO-C ₆ H ₄ | (CH ₃) ₃ C | 2 | 97 | 88-91/0.7 | |

^aIsolated yields. ^bReported boiling points were those observed during distillation with a Kugelrohr apparatus and were uncorrected. ^c0.2 equiv of ZnCl₂ was employed.

phy.

Since the reactions performed are all similar in many respects, typical reactions will be described as specific examples.

Preparation of Methyl Mesitoate. To a mixture of mesitoyl chloride (248 mg, 1.4 mmol) and anhydrous zinc chloride (20 mg, 0.15 mmol) in dry acetonitrile (5 ml) was added methanol (58 mg, 1.5 mmol). The reaction mixture was stirred at room temperature for 5 min, taken up in methylene chloride (20 ml × 3), washed successively with saturated NaHCO₃ solution (20 ml), saturated NaCl solution (20 ml), dried over MgSO₄, and filtered. Removal of methylene chloride afforded the crude product, which further distilled with a Kugelrohr apparatus to yield pure methyl mesitoate (231 mg, 95%). bp 75-79°C/1.6 mm, Lit.^{10a} 116/10 mm.

Preparation of *t*-Butyl Mesitoate in the Presence of Molecular Sieves. To a mixture of mesitoyl chloride

(202 mg, 1.1 mmol), anhydrous zinc chloride (17 mg, 0.13 mmol), baked powdered 4A molecular sieves (520 mg) in dry acetonitrile (5 ml) was added *t*-butyl alcohol (89 mg, 1.2 mmol). The reaction mixture was stirred at room temperature for 1 h, diluted with methylene chloride (40 ml), and filtered to remove molecular sieves. The filtrate was washed with saturated NaHCO₃ solution (20 ml) and saturated NaCl solution (20 ml) and dried over MgSO₄. Removals under reduced pressure afforded the crude product, which was distilled with a Kugelrohr apparatus to yield pure *t*-butyl mesitoate (171 mg, 71%). bp 93-97/1.6 mm, Lit.^{10a} 114/2 mm.

Preparation of *S-t*-Butyl 2,4,6-Trimethylbenzothioate.

To a mixture of mesitoyl chloride (163 mg, 0.9 mmol) and anhydrous zinc chloride (15 mg, 0.11 mmol) in dry acetonitrile (4 ml) was added *t*-butyl mercaptan (110 μl, 1.0 mmol). The reaction mixture was stirred at room temperature for

5 min, diluted with methylene chloride (60 ml), washed with saturated NaHCO₃ solution (20 ml), saturated NaCl solution (20 ml), dried over MgSO₄, and filtered. Removal of methylene chloride under reduced pressure afforded the crude product, which further distilled with a Kugelrohr apparatus to yield pure *S-t*-butyl 2,4,6-trimethylbenzothioate (200 mg, 95%). bp 95–99/0.9 mm, mp 42–43°C; *Lit*²¹ mp 42–43°C.

Reaction of Pivaloyl Chloride with 3-Ethyl-3-heptanol in the Presence of 0.1 equiv of Zinc Chloride. To a solution of pivaloyl chloride (173 mg, 1.4 mmol) and anhydrous zinc chloride (19 mg, 0.14 mmol) in dry acetonitrile (4 ml) was added 3-ethyl-3-heptanol (209 mg, 1.4 mmol). The reaction mixture was stirred at room temperature for 1.5 h, diluted with methylene chloride (40 ml), washed with saturated NaHCO₃ solution (20 ml) and saturated NaCl solution (20 ml), dried over MgSO₄, and filtered. Removal of solvents under reduced pressure afforded the crude product, which was subjected to silica gel column chromatography with hexane-ethyl acetate (5:1) as an eluant to yield 3-chloro-3-ethylheptane (118 mg, 51%). NMR (CDCl₃) δ 0.9–1.2 (m, 9H), 1.2–2.1 (m, 10H). IR (film) 2960, 1462 cm⁻¹. The spectral data were identical with those of an authentic sample.

Spectral Data of Unknown Esters and Thiol Esters. Mesityl pivalate: NMR (CCl₄) δ 1.41 (s, 9H), 2.09 (s, 9H), 6.73 (s, 2H); IR (film) 1740 cm⁻¹. Cyclohexyl pivalate: NMR (CCl₄) δ 1.00–2.17 (m, 10H), 1.25 (s, 9H), 4.43–5.10 (m, 1H); IR (film) 1730 cm⁻¹. *t*-Amyl mesitoate: NMR (CCl₄) δ 1.02 (t, 3H, *J*=7), 1.62 (s, 6H), 1.80 (q, 2H, *J*=7), 2.34 (s, 9H), 6.80 (s, 2H); IR (film) 1720 cm⁻¹. 3-Ethyl-3-pentyl mesitoate: NMR (CCl₄) δ 0.94 (t, 9H, *J*=7), 1.95 (q, 2H, *J*=7), 2.24 (s, 3H), 2.33 (s, 6H), 6.80 (s, 2H); IR (film) 1720 cm⁻¹. Mesityl caprylate: NMR (CCl₄) δ 0.68–2.13 (m, 23H), 2.24 (t, 2H, *J*=7), 4.44–4.97 (m, 1H); IR (film) 1725 cm⁻¹. *S-n*-Butyl 2, 4, 6-trimethylbenzothioate: NMR (CCl₄) δ 0.83–1.97 (m, 7H), 2.31 (s, 9H), 2.90–3.22 (t, 2H, *J*=7), 6.77 (s, 2H); IR (film) 1675 cm⁻¹. *S-t*-Butyl 2, 2-dimethylpropanethioate: NMR (CCl₄) δ 1.14 (s, 9H), 1.41 (s, 9H); IR (film) 1675 cm⁻¹. *S-t*-Butyl octanethioate: NMR (CCl₄) δ 0.67–1.83 (m, 13H), 1.43 (s, 9H), 2.35 (t, 2H, *J*=7); IR (film) 1690 cm⁻¹. *S-n*-Butyl octanethioate: NMR (CCl₄) δ 0.57–2.23 (m, 20H), 2.51 (t, 2H, *J*=7), 2.85 (t, 2H, *J*=7); IR (film) 1700 cm⁻¹.

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