ient method of isomer separation is developed.

Experimental Section

Melting points were determined on a Mel-Temp capillary block melting point apparatus and are uncorrected. All compounds are homogenous by thin-layer chromatographic analysis with Analtech silica gel GF 250-μm TLC plates. ^{1}H NMR measurements were obtained on a Varian Associates EM-390 or CFT 20 spectrometer with tetramethylsilane as the internal standard in Me_2SO-d_6 solution. Mass spectral measurements were obtained on a Varian CH7 mass spectrometer.

Nitration of *s*-Triazolo[3,4-*a*]phthalazine (1). Method A.[1] To a solution of *s*-triazolo[3,4-*a*]phthalazine (6.8 g, 40 mmol) in sulfuric acid (80 mL, 98%) at 0 °C was added a solution of fuming nitirc acid (37 mL, 90%) in sulfuric acid (160 mL, 98%) over 5 min with stirring. The reaction was kept at 0 °C for 45 min, warmed to room temperature for 2 h, and poured over 2 L of crushed ice brought to pH 8 with ammonium hydroxide. The crude product was filtered, and the filtrate was washed with water and dried over P_2O_5 under vacuum, yielding 2.73 g, mp 260–268 °C. Four successive recrystallizations of this product from acetone gave the following melting points 268–278, 278–283, 280–286, 281–288 °C.

Method B. To a solution of potassium nitrate (100 g, 0.99 mol) in sulfuric acid (250 mL, 96%) was added *s*-triazolo[3,4-*a*]-phthalazine (30 g, 0.176 mol). A slight exotherm was noted as the reaction mixture turned light orange. After stirring overnight, the pale yellow reaction mixture was worked up as in method A to give the crude nitro product: 21.2 g of yellow solid. The isomers were separated by column chromatography on silica gel eluting with 2% methanol in chloroform and subsequent recrystallization of selected fractions. The 10-isomer (**2**) was eluted first, followed by the 8-isomer (**3**) and then the 7-isomer (**4**).

10-Nitro-*s*-triazolo[3,4-*a*]phthalazine (2) recrystallized from acetone: mp 300–301 °C; mass spectrum, *m/e* 215 (M^+); ^{1}H NMR δ 9.63 (s, 1, H_3), 9.16 (s, 1, H_6), 8.38 (AB, 2, H_7 and H_9), 8.03 (X, 1, H_8, $J_{AX} \cong J_{BX} \cong 8$).

Anal. Calcd for $C_9H_5N_5O_2$: C, 50.24; H, 2.34; N, 32.55. Found: C, 50.43; H, 2.27; N, 32.28.

8-Nitro-*s*-triazolo[3,4-*a*]phthalazine (3) recrystallized from acetonitrile: mp 334 °C; mass spectrum, *m/e* 215 (M^+); ^{1}H NMR δ 9.69 (s, 1, H_3), 9.24 (s, 1, H_6), 9.13 (X, 1, H_7, $J_{AX} + J_{BX} \cong$ 2–3 Hz), 8.71 (AB, 2, H_9 and H_{10}).

Anal. Calcd for $C_9H_5N_5O_2$: C, 50.24; H, 2.34; N, 32.55. Found: C, 49.72; H, 2.29; N, 32.57.

This material was not further purified but was subsequently fully characterized by derivatives **7** and **8**.

7-Nitro-*s*-triazolo[3,4-*a*]phthalazine (4) recrystallized from acetonitrile: mp 213–214 °C; mass spectrum, *m/e* 215 (M^+); ^{1}H NMR δ 9.68 (s, 1, H_3), 9.29 (s, 1, H_6), 8.83 (d, 1, H_{10}, J = 8 Hz), 8.52 (d, 1, H_8, J = 8 Hz).

Anal. Calcd for $C_9H_5N_5O_2$: C, 50.24; H, 2.34; N, 32.55. Found: C, 50.38; H, 2.37; N, 32.69.

Reduction of the Nitro-*s*-triazolo[3,4-*a*]phthalazines. Reduction of the acetone-recrystallized crude nitro compound, which is a mixture of the 8- and 10-isomers (**3** and **2**) uses typical conditions. A 6.6-g (37 mmol) sample of the mixture was slurried in 100 mL of ethanol. To this was added 0.75 g of 10% Pd/carbon, and the mixture was hydrogenated at 50 psi on a Parr apparatus until uptake of hydrogen stopped. The reduction mixture was filtered and the filtrate was washed with ethanol (50 mL) and then with hot acetic acid. The ethanol filtrate and wash containing the 8-amino isomer (**7**) was evaporated to dryness and triturated with chloroform to remove any of the remaining 10-amino derivative (**5**). The combined chloroform triturate and acetic acid extract was evaporated and recrystallized from ethanol, yielding 10-amino derivative **5** (3.59 g). The chloroform-insoluble solid was recrystallized from acetonitrile to give the 8-amino isomer **7** (0.80 g): total yield 4.39 g (80%).

10-Amino-*s*-triazolo[3,4-*a*]phthalazine (5): mp 247–248 °C; mass spectrum, *m/e* 185 (M^+); ^{1}H NMR δ 9.41 (s, 1, H_3), 8.79 (s, 1, H_6), 7.53 (X, 1, H_8, $J_{AX} \cong J_{BX} \cong$ 8 Hz), 7.20 (AB, 2, H_7 and H_9), 6.89 (br s, 2, NH_2).

Anal. Calcd for $C_9H_7N_5$: C, 58.37; H, 3.81; N, 37.82. Found: C, 58.06; H, 3.92; N, 37.84.

8-Amino-*s*-triazolo[3,4-*a*]phthalazine (7): mp 274–275 °C; mass spectrum, *m/e* 185 (M^+); ^{1}H NMR δ 9.25 (s, 1, H_3), 8.70 (s, 1, H_6), 8.08 (d, 1, H_{10}, J = 8 Hz), 7.19 (dd, 1, H_9, J = 8 and 2 Hz), 7.02 (d, 1, H_7, J = 2 Hz), 6.10 (br s, 2, NH_2).

Anal. Calcd for $C_9H_7H_5$: C, 58.37; H, 3.81; N, 37.82. Found: C, 58.14; H, 3.72; N, 38.03.

7-Amino-*s*-triazolo[3,4-*a*]phthalazine (9) recrystallized from acetonitrile: mp 290 °C dec; mass spectrum, *m/e* 185 (M^+); ^{1}H NMR δ 9.38 (s, 1, H_3), 9.08 (s, 1, H_6), 7.51 (AB, 2, H_9 and H_{10}), 6.97 (X, 1, H_8, $J_{AX} \cong$ 8 Hz and $J_{BX} \cong$ 2 Hz), 6.62 (br s, 2, NH_2).

Anal. Calcd for $C_9H_7H_5$: C, 58.37; H, 3.81, N, 37.82. Found: C, 58.21; H, 3.74; N, 38.15.

Acetylation of the Amino-*s*-triazolo[3,4-*a*]phthalazines. Acetylation of the 8-amino derivative (**7**) is typical. A mixture 8-amino-*s*-triazolo[3,4-*a*]phthalazine (0.40 g, 2.1 mmol) and acetic anhydride (5 mL) was heated to a gentle reflux for 1 h, cooled to room temperature, and filtered. The product was washed with acetic anhydirde (2 mL) and ether (10 mL) and dried in vacuo to give the 8-acetamido derivative (**8**), 0.37 g (75%).

10-Acetamido-*s*-triazolo[3,4-*a*]phthalazine (6) recrystallized from acetic anhydride: mp 254–255 °C; mass spectrum, *m/e* 227 (M^+), 212 ($M^+ - CH_3$), 185 (M^+ – ketene; ^{1}H NMR δ 9.50 (s, 1, H_3), 8.95 (s, 1, H_6), 8.90 (X, 1, H_9, J_{AX} and $J_{BX} \cong$ 10 Hz), 7.78 (AB, 2, H_7 and H_8), 2.32 (s, 3, CH_3).

Anal. Calcd for $C_{11}H_9N_5O$: C, 58.14; H, 3.99; N, 30.82. Found: C, 58.07; H, 4.07; N, 30.87.

8-Acetamido-*s*-triazolo[3,4-*a*]phthalazine (8): mp 329–330 °C; mass spectrum, *m/e* 227 (M^+); ^{1}H NMR δ 9.43 (s, 1, H_3), 8.98 (s, 1, H_6), 8.50 (d, 1, H_7, $J \cong$ 2), 8.37 (d, 1, H_{10}, J = 8 Hz), 8.01 (dd, 1, H_9, J = 8 and $\cong$2 Hz), 2.19 (s, 3, CH_3).

Anal. Calcd for $C_{11}H_9N_5O$: C, 58.14; H, 3.99; N, 30.82. Found: C, 57.71; H, 3.93; N, 30.95.

7-Acetamido-*s*-triazolo[3,4-*a*]phthalazine (10): mp 279–280 °C dec; mass spectrum, *m/e* 227 (M^+); ^{1}H NMR δ 9.51 (s, 1, H_3), 9.08 (s, 1, H_6), 7.99 (AB, 2, H_9 and H_{10}), 8.30 (X, 1, H_8, $J_{AX} + J_{BX} \cong$ 10 Hz), 2.23 (s, 3, CH_3); mass spectrum, *m/e* calcd for $C_{11}H_9N_5O$, 227.0807; *m/e* found, 227.0808.

Anal. Calcd for $C_{11}H_9N_5O$: C, 58.14; H, 3.99; N, 30.82. Found: C, 57.14; H, 3.90; N, 30.36.

Acknowledgment. The authors thank Professor Kevin Potts for interesting discussions during the course of this work, George Morton for information concerning NMR data, and Drs. Tom McKenzie and Ray Carhart for their help in the Huckel and CNDO calculations.

Registry No. **1**, 234-80-0; **2**, 83633-05-0; **3**, 21517-40-8; **4**, 83633-06-1; **5**, 83633-07-2; **6**, 83633-08-3; **7**, 83633-09-4; **8**, 83633-10-7; **9**, 83633-11-8; **10**, 83649-35-8.

Improved Synthesis of 1-Ethoxy-1,3-dihydroisobenzofuran, a Useful Precursor to Isobenzofuran

Randall J. Moss and Bruce Rickborn*

Department of Chemistry, University of California, Santa Barbara, California 93106

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Brown[1] was the first to observe that in situ generated carbenium ions could be reduced by $NaBH_4$, although solvolysis and elimination reactions may compete depending on the substrate used. Some time ago we applied this approach to the formation of 1,3-dioxolanes, using $NaBH_4$ in refluxing pyridine to trap the ions formed by solvolysis with ester neighboring group participation.[2]

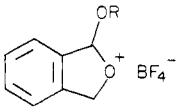
(1) Brown, H. C.; Bell, H. M. *J. Org. Chem.* **1962**, *27*, 1928. Bell, H. M.; Brown, H. C. *J. Am. Chem. Soc.* **1966**, *88*, 1473.

(2) Johnson, M. R.; Rickborn, B. *Org. Synth.* **1971**, *51*, 11. In this work, in situ generated 1,3-dioxolan-2-ylium ions derived from 2,3-butanediol were reduced in good yields by $NaBH_4$ in pyridine.

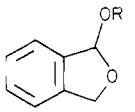
0022-3263/82/1947-5391$01.25/0

Raber[3] also has shown that tetraalkylammonium borohydride in CH_2Cl_2 can be used to reduce preformed 1,3-dioxolan-2-ylium fluoborates to the corresponding acetals in good yield.

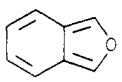
We have reported[4] that the acetal **2** is a convenient



1, R = CH_3, C_2H_5



2, R = CH_3, C_2H_5



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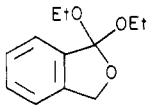
precursor of isobenzofuran **3**, through either strong base induced 1,4-elimination (which allows the isolation of solutions of **3**) or by acid-catalyzed reaction where transient **3** may be trapped with reactive dienophiles. In view of this utility, a high yield and general procedure for the formation of **2** and its analogues is desirable. Two syntheses of **2** (R = CH_3) have been reported. Tidwell and co-workers[5] obtained a 40% yield by diisobutylaluminum hydride reduction of phthalide, followed by conversion to the acetal in methanol. We formed **2** directly by laundry bleach oxidation of phthalyl alcohol in 60% yield under fairly high dilution conditions in pentane/methanol.[4] Neither method is general or attractive for larger scale preparations.[6]

The facile preparation[7] of ethylphthalidium salt **1** suggested that direct reduction might provide a convenient source of **2** (R = C_2H_5). In initial attempts, we found that addition of **1** to $NaBH_4$ in ethanol gave both **2** and orthoester **4** while the use of dimethoxyethane solvent gave



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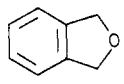

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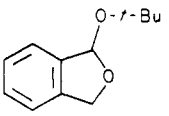
essentially only phthalan **5** in a vigorous reaction. To avoid solvolysis and overreduction, we turned to pyridine as solvent, since this had given good results in our earlier study involving 1,3-dioxolan-2-ylium ions.[2] While this did prevent overreduction, yields of only 30% could be reproducibly obtained. The fate of the remainder of the salt **1** has not been completely determined, but a major proportion is converted to phthalide, perhaps by ethylation of pyridine. The limited solubility of $NaBH_4$ in pyridine (ca. 3 g/100 g)[8] does not allow much flexibility in concentration of reducing agent in this medium.

The solubility of $NaBH_4$ in DMF (18 g/100 g)[8] allows greater control of this variable and suggested that this property might be turned to advantage. In the event, we found that even 1 mol of $NaBH_4$/mol of **1** gave excellent yields of **2** contaminated only by a small amount (ca. 2%) of **5**. Further, this overreduction can be eliminated by the addition of 1 mol of pyridine/mol of $NaBH_4$ used. The details of this successful approach are given under Experimental Section.

Lithium aluminum hydride in ethereal solvents gives negligible yields of **2**, leading instead to **5**. Use of a stoichiometric amount of this reagent simply caused formation of **5** and recovery of phthalide (from hydrolysis of **1**). Changing the order of addition did not materially affect the results. This implied that the alane formed on initial hydride transfer to **1** functions as a strong Lewis acid in the same manner as borane. To diminish this Lewis acidity, we turned to alkoxy-substituted derivatives of $LiAlH_4$. This approach also proved to be successful but led to an unexpected observation; although overall yields of acetal are high, the product using $LiAlH(O\text{-}t\text{-}Bu)_3$ or Red-Al, $NaAlH_2(OCH_2CH_2OCH_3)$, contained approximately 15% of material in which the alkoxy group was derived from the reducing agent (**6** and **7**, respectively;



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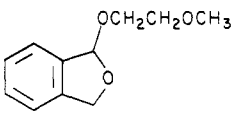

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identification based on NMR and GC/MS.) The mechanism of formation of these products is unknown. They do not represent a serious contaminant, since they can be converted to the desired acetal if heated in the appropriate alcohol with strong acid catalyst. The obvious way to avoid this additional step is to use $LiAlH_4$ modified by ethoxide substituents.

The addition of anhydrous ethanol to $LiAlH_4$ solutions was monitored by H_2 evolution to attain the stoichiometry of $LiAlH_2(OEt)_2$, **8** and this reagent was examined in some detail for reduction of **1**. Addition of an ether solution of **8** to a CH_2Cl_2 slurry of **1** (R = Et) gave **2** in moderate yield, but relatively more **5** is formed with this order of mixing. Addition of the solid **1** to the hydride solutions gave excellent yields of monomeric product, where the only contaminant is again **5**, with the amount dependent on the ratio of reducing agent and solvent employed. The results of several runs are shown in Table I.

Table I

8/1 (mol)	reduction of 1 by 8[a] solvent (mL)[b]	time, h	2/5[c]	yield,[d] %
1.1	Et_2O (6.6), CH_2Cl_2 (20)	5	63/37	92
1.2	Et_2O	44	88/12	96
1.1	Et_2O	6	93/7	89
1.1	THF	1.5	95/5	73
1.9	THF	2	98/2	85
2.3	THF	2.3	100/0	82
2.0	THF	1	100/0	95
2.2	Et_2O (13), CH_2Cl_2 (20)	16	93/7	94
2.3	Et_2O	6[e]	98/2	87

[a] Approximately 1 g of **1** was used in each run, added as a solid to the hydride solution cooled in an ice bath. The bath was then removed and stirring was continued for the time indicated, with quenching by addition of Rochelle's salt solution (30%) or 10% NaOH. [b] The volume of solvent used was 30 mL where not otherwise indicated. [c] Determined by VPC with a Carbowax 6M column. [d] Based on theory for acetal, using the weight of crude product after rotary evaporation; these residues showed no indication by NMR or VPC of products other than **2** and **5**. [e] Shorter times in Et_2O led to incomplete reaction, as shown by NMR peaks corresponding to phthalide and the ring-opened ethyl ester from hydrolysis of **1**; by visual inspection, this appears to be associated with the slow dissolution of **1** in this solvent.

The two most significant observations are the effect of solvent, where improvement in the ratio of **2**/**5** is found on going from CH_2Cl_2 to Et_2O to THF, and, curiously, the dimunition of overreduction by increasing the proportion

(3) Raber, D. J.; Guida, W. C. *Synthesis* **1974**, 808. The simpler ethylene glycol derivatives were used in this study.

(4) Naito, K.; Rickborn, B. *J. Org. Chem.* **1980**, *45*, 4061. See also Makhlouf, M. A.; Rickborn, B. *Ibid.* **1981**, *46*, 2734.

(5) Rynard, C. M.; Thankachan, C.; Tidwell, T. T. *J. Am. Chem. Soc.* **1979**, *101*, 1196.

(6) The dibal procedure is widely used for formation of lactols from lactones but fails when the lactone has limited solubility under the low-temperature conditions needed to effect selective reduction.

(7) Meerwein, H.; Borner, P.; Fuchs, O.; Sasse, H. J.; Schrodt, H.; Spille, J. *Chem. Ber.* **1956**, *89*, 2060.

(8) The booklet "Sodium Borohydride", Thiokol/Ventron, Danvers, MA, 1979, provides a very useful compendium of the properties and reactions of $NaBH_4$.

of reducing agent. It may be that the additional mole of 8 serves to complex the $AlH(OEt)_2$ formed on initial hydride transfer, lowering its Lewis acidity and ability to interact with acetal. The use of a twofold molar excess of 8 in THF constitutes the second useful procedure for formation of **2**.

We have also briefly explored the use of $LiAlH(OEt)_3$ in THF but find that it offers no advantage compared to 8 and even leads to traces (1–2%) of **5**.

The use of **1** (R = CH_3) gave similar results with the various reducing agents described, in somewhat lower yields. Because of the greater difficulty in preparing the methylated salt, it is preferable in this system to make ethylated **2** and transacetalize to obtain other derivatives.

A few experiments were carried out with **1** as the $SbCl_6^-$ salt. While acetal is formed, the counterion is also reduced (e.g., vigorous reaction with $NaBH_4$) to a black solid, presumably antimony. Other counterions have not been examined.

The procedures described here for the reduction of **1** have also been successfully applied to some naphthalene analogues and should have wide generality.

Experimental Section

Dichloromethane was distilled from $LiAlH_4$,[9] ether from CaH_2, and THF from sodium and benzophenone ketyl. Reagent grade DMF was stored over Linde 3A molecular sieve, and pyridine was stored over KOH pellets.

The *O*-ethylphthalidium tetrafluoroborate, **1** was prepared by Meerwein's procedure.[7]

For small-scale reactions, stock solutions of $LiAlH_4$ in ether and THF were prepared by refluxing for 3 h and using the clear supernatant without filtration. Mixed hydride reagents were formed by addition of anhydrous ethanol (distilled from Mg) and measuring H_2 evolution by gas buret.

The following procedures illustrate the preferred methods for formation of **2**.

(A) $NaBH_4$. To a 50-mL three-necked flask equipped with a magnetic stirrer, thermometer, and condenser with N_2 inlet was added 10 mL of DMF, 0.78 mL (9.6×10^{-3} mol) of pyridine, and 0.38 g (1.0×10^{-2} mol) of $NaBH_4$. Brief stirring at room temperature effected solution. The mixture was then immersed in an ice bath, and 2.0 g (8.0×10^{-3} mol) of **1** (R = CH_2CH_3) was added (powder funnel, N_2 flow) over a period of 20 min. After the mixture was stirred an additional 10 min, 5 mL of water was added dropwise (some H_2 evolution) followed by 30 mL of saturated NaCl solution. This mixture was extracted four times with 20-mL portions of pentane; the combined pentane phase was washed with water and brine, dried over K_2CO_3, and rotary evaporated to give 1.2 g (89%) of **2**. The ^1H NMR of this material was identical with that of an analytically pure sample, and VPC gave no indication (≤1%) of **5**.

(B) $LiAlH_2(OEt)_2$. A 1-L three-necked flask was fitted with a mechanical stirrer, condenser with N_2 inlet, and addition funnel. THF, 450 mL, was added, followed by 9.3 g (0.24 mol) of $LiAlH_4$. The cloudy solution was cooled in an ice bath and 25.7 mL (0.44 mol) of anhydrous ethanol in 50 mL of THF was added over a period of 1 h. With continued cooling and stirring, 25 g (0.10 mol) of **1** (R = CH_2CH_3) was added rapidly through a powder funnel. The ice bath was removed, and stirring was continued for 2 h. Again with cooling, the excess hydride was quenched by the dropwise addition of 150 mL of 10% NaOH, after which the liquid phase was decanted from the precipitated aluminum salts and rotary evaporated to remove most of the THF. The residue was taken up in 50 mL of ether, washed with water and brine, dried over K_2CO_3, and again rotary evaporated to give 16.0 g of crude material, which contained only traces of **5** by VPC analysis. Distillation gave 14.3 g (87%) of pure **2**, bp 77–79 °C (2.0 Torr).[10]

Acknowledgment. Support by a grant from the University of California Cancer Research Coordinating Committee is gratefully acknowledged.

Registry No. **1** (R = C_2H_5), 487-97-8; **2** (R = C_2H_5), 75802-19-6; **3**, 270-75-7.

(9) A referee has questioned our use of dichloromethane as a solvent (when so specified) for reductions of the salt **1** and also the use of $LiAlH_4$ to dry this solvent, stating "Halogenated hydrocarbons are known to react with explosive violence with reactive complex metal hydrides." On the first point, CH_2Cl_2 is the only common solvent that will dissolve a moderate amount of **1** without potentially or in practice reacting with it. On the second point, we shared the referee's concern but have now used this technique for over a year without experiencing any difficulty, after first learning of it as a method for preparing ultra-dry CH_2Cl_2 from Professor W. C. Kaska of this department. While a still pot of CH_2Cl_2 to which solid $LiAlH_4$ has been added has been maintained at reflux for weeks without incident, we do not advocate this as a general procedure for drying halogenated hydrocarbons.

(10) Some decomposition of **2** may occur when distillation is done at higher temperatures, evidenced by the formation of viscous pot residue.

Use of Ethylaluminum Dichloride as a Catalyst for the Friedel–Crafts Acylation of Alkenes

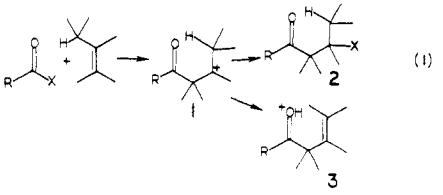
Barry B. Snider* and Andrew C. Jackson

Department of Chemistry, Brandeis University, Waltham, Massachusetts 02254

Received May 3, 1982

Introduction

Friedel–Crafts acylation of alkenes is an old and well-known reaction.[1,2] A reactive acylating agent reacts with an alkene to give an intermediate cation **1**, which can react



(1)

with nucleophiles, usually chloride, to give **2** or undergo a 1,5-proton shift to give the protonated β,γ-unsaturated ketone **3**. Since both the starting alkenes and product β,γ-unsaturated ketones are sensitive to the protic acid produced in this reaction, aliphatic Friedel–Crafts acylation has been much less useful than its aromatic counterpart.

Reaction of acid halides with alkenes in the presence of Lewis acids usually leads to β-chloro ketones. Treatment of the crude reaction mixture with base provides a good yield of α,β-unsaturated ketone.

The best synthesis of β,γ-unsaturated ketones involves the reaction of acylium salts with alkenes in the presence of a hindered base[3,4] or the reaction of alkenes with zinc chloride in a large excess of acyl anhydride as solvent.[5–7] Acylium salts are not attractive starting materials, and the zinc chloride procedure is only applicable to acetic anhy-

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