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Organomercury/Aluminum-Mediated Acylative Cleavage of Cyclic Ethers

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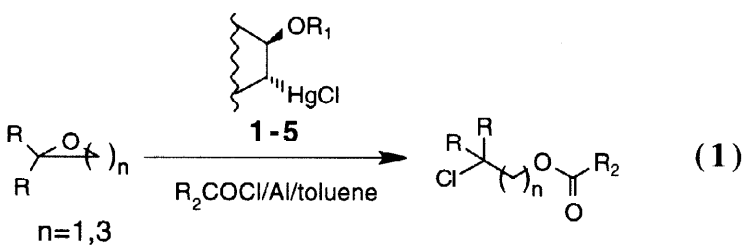
Abstract: Epoxides and tetrahydrofurans are cleaved with concomitant acylation to chloroalkyl esters using a reagent system composed of an organomercurial, aluminum metal and an acid chloride. The cleavage is promoted under mild conditions by a range of readily-available cyclic β-alkoxychloromercurials and acid chlorides. Using mainly tetrahydrofuran and cyclohexene oxide as substrates, the yield of isolated chloroesters ranged from 52 to 96%.
Keywords: Epoxides, Aluminum, Mercury, Acylation

Reactions which cleave cyclic ethers to haloesters[1,2] are an effective means of obtaining difunctional synthetic intermediates and are important for the removal of ethereal protecting groups.[3] Previous reports describe the acylative cleavage of cyclic ethers using reagent systems or conditions such as zinc metal,[4] $(Cp)_2YCl$,[5] $CoCl_2/CH_3CN$,[6,7] pressure,[8] Bu_2SnCl_2/Ph_3P,[9] $K[PtCl_3(C_2H_4)]$,[10] Pd^{II}/R_3SnX,[11] $Fe(CO)_5$,[12,13] $ZnCl_2$,[14] $Eu(dpm)_3$,[15] or hexabutylguanidinium chloride[16] in conjunction with acid chlorides. We report herein that a reagent system composed of an organomercurial, aluminum metal and an acid chloride is highly effective for the mild cleavage of epoxides and tetrahydrofurans to the corresponding chloroalkyl esters (**Eq 1**). The organo-



(1)

mercurials employed for the cleavage are a series of readily available *trans*-2-alkoxy-1-chloromercuricyclohexanes (**1-5**). These stable organomercurials are easily prepared by the alkoxymercuration[17,18,19] of cyclohexene with mercuric acetate followed by anion exchange with sodium chloride. Although the series of organomercurials selected for the present study (**1-5**)[20]

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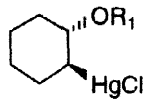
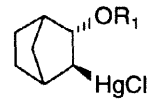
will promote the cleavage under mild conditions (toluene, 50°C) the addition of aluminum metal markedly increases both the reaction rate and yield of product.



1, R_1=CH_3
2, R_1=CH_2CH_3
3, R_1=$CH(CH_3)_2$
4, R_1=cyclohexyl



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The cleavage of tetrahydrofuran, 2-methyltetrahydrofuran (**Table 1**), cyclohexene oxide and styrene oxide (**Table 2**) was facilitated using a range of alkoxymercurials and acid chlorides. The yields of the

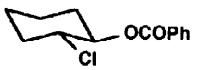
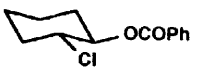
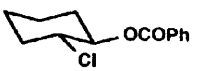
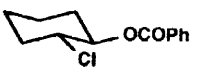
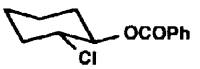
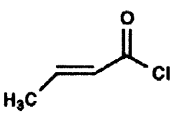
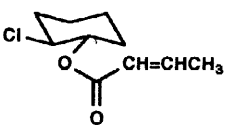
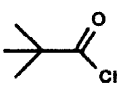
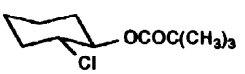
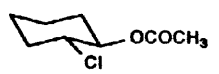
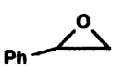
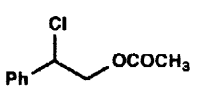
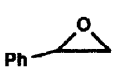
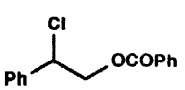
Table 1. Alkoxycycloalkylchloromercuri-Promoted Cleavage of THF's

Entry	Substrate	Mercurial	RCOCl	Product	% Yield
1	THF	1	PhCOCl	$PhCOO(CH_2)_4Cl$	53
2	THF	2	PhCOCl	$PhCOO(CH_2)_4Cl$	52
3	THF	3	PhCOCl	$PhCOO(CH_2)_4Cl$	88
4	THF	1	CH_3COCl	$CH_3COO(CH_2)_4Cl$	77
5	THF	1	$ClCH_2COCl$	$ClCCH_2COO(CH_2)_4Cl$	80
6	2-CH_3THF	1	PhCOCl	$PhCOO(CH_2)_3CH_3CHCl$	58

haloesters ranged from 52% to 96%. Since a significant factor in the cleavage reaction is the solubility of the alkoxymercurial, the solvent of choice was toluene due to its ability to dissolve the mercurial, relative inertness and higher boiling range.

A variety of alkoxymercurials were shown to cleave tetrahydrofuran[21] and its 2-methyl derivative with the ispropoxychloromercury derivative (**3**) giving the highest yield of 4-chlorobutyl ester. Variations in the acid chloride components will also cause yields to vary. Higher yields of chlorobutyl esters were realized with acetyl chloride or chloroacetyl chloride while trichloroacetyl chloride gave only complex mixtures and no ester product, presumably due to the highly reducing nature of the reaction mixture. Variations in the alkoxy group of the mercurial do not markedly change its effectiveness in epoxide cleavages to 2-chlorocyclohexylesters (**Entries 1-8, Table 2**). Although the yield of *trans*-2-chlorobenzoyloxycyclohexane is lower when employing the norbornyl mercurial (**5**) than in the other examples, a case may be made for the steric influence of the norbornyl group. The acylative cleavage of cyclohexene oxide with *trans*-crotonyl chloride to *trans*-2-chloro 1-(*trans*-crotonyloxy)cyclohexane (**Entry 6, Table 2**) is significant in organic synthesis since β-haloalkyl crotonates have been proposed as useful substrates in the synthesis of lactones via intramolecular free-radical

Table 2. Alkoxycycloalkylchloromercury-Promoted Cleavage of Epoxides

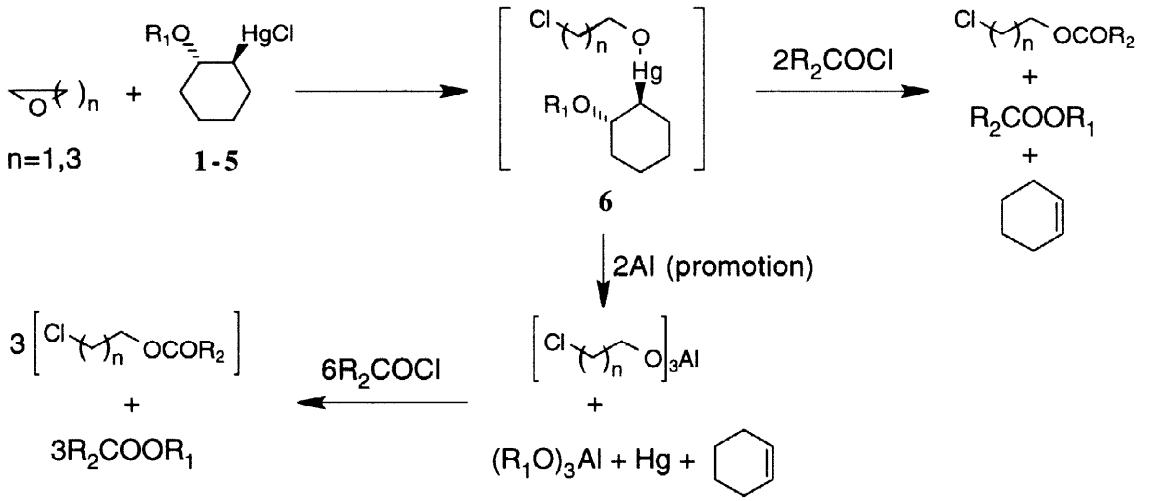
Entry	Substrate	Mercurial	RCOCl	Product	% Yield[1]
1	cyclohexene oxide	1	PhCOCl		60
2	cyclohexene oxide	2	PhCOCl		61
3	cyclohexene oxide	3	PhCOCl		77
4	cyclohexene oxide	4	PhCOCl		67
5	cyclohexene oxide	5	PhCOCl		47
6	cyclohexene oxide	1			60
7	cyclohexene oxide	1			52
8	cyclohexene oxide	1	CH_3COCl		53
9		1	CH_3COCl		96
10		1	PhCOCl		63[2]

[1]Yields are of isolated products unless otherwise noted (See Experimental).
[2]A 2:1 mixture of the secondary/primary chloro isomer was detected by ^1H NMR analysis (See Experimental).

cyclizations.[22] The acylative cleavage of styrene oxide and 2-methyltetrahydrofuran introduce regiochemical considerations. The acylative cleavage of styrene oxide which employed benzoyl chloride provided both isomeric phenethyl benzoates (**Entry 10, Table 2**), however the secondary chloro regioisomer is favored over the primary according to ^1H NMR analysis. Similarly the cleavage of 2-methyltetrahydrofuran with 2-chloromercuri-1-methoxycyclohexane/aluminum gave 4-chloropentyl benzoate (**Entry 6, Table 1**). The fact that both cases provide the more substituted secondary chloro isomer suggest an initial S_N1-type mechanism for chloride formation.

The overall general mechanism responsible for the cleavage process is not immediately obvious especially when the role of aluminum as a promoter is considered. A plausible overall reaction pathway is proposed in **Scheme 1**. Mercury-mediated cleavage of the cyclic ether by the 2-chloromercurialkoxycyclohexane reagent results in an alkoxycyclohexyl(chloroalkoxy)mercurial (**6**). Although the organomercurichlorides are not usually

Scheme 1. Suggested Reaction Pathways for Acylative Cleavage.



considered as strongly electrophilic compounds, one may presume that the ring-opening step is facilitated by ring strain.[23] Intermediate **(6)** undergoes aluminum-mercury exchange[24] and disproportionation to both a chloroalkylaluminum alkoxide and the alkoxide derived from the mercury reagent together with elemental mercury and cyclohexene. Subsequent attack of both the alkoxides[25] on the acid chloride results in the esters.

Gas chromatographic monitoring of tetrahydrofuran cleavage promoted by benzoyl chloride and 2-methoxychloromercuricyclohexane/aluminum confirmed the appearance of the by-products methyl benzoate and cyclohexene which were consistent with the proposed mechanism. In order to ascertain the extent of aluminum as a promoter, GC analyses were also employed to monitor cleavage reactions in the absence of aluminum **(Table 3)** and using aluminum as a promoter **(Table 4)**. GC analyses of the non-aluminum-promoted reaction **(Table 3)** indicated the appearance of 4-chlorobutyl benzoate (*Rt*=12.77 min) after 2 h, while in the presence of aluminum, the chloroester was first detected at the 1.5 h mark **(Table 4)**. Secondly the ratio of the concentration of 4-chlorobutyl benzoate to that of methyl benzoate (*Rt*=7.44 min) was 1:1.2 when the experiment was conducted overnight without aluminum. In contrast GC analyses of the aluminum-promoted reaction showed a ratio of 2:1 in favor of 4-chlorobutyl benzoate over the same time period **(Table 4)**. Isolated yields (%) of the benzoates also support this ratio. In the absence of aluminum the ratio of the isolated yields for the chlorobenzoate relative to methyl benzoate was 1:1.4 (28%:40%). In the presence of aluminum, the ratio is closer to 2.1:1 (53%:25%) during a 14-16 h reaction period. Since the production of methyl benzoate was a competing reaction for production of the chloroester, it was anticipated that the employment of excess benzoyl chloride should increase the yield of the chloroester. This was confirmed when the yield of 4-chlorobutyl benzoate, as calculated from the weight of the isolated compound, increased to 76% when using a slight excess (2.2 eq) of benzoyl chloride. GC analysis also showed increased peak areas for both 4-chlorobutyl benzoate and methyl benzoate at the 4 h mark as compared to the standard reaction for the same time period. The rate of formation of the chloroester as well as methyl benzoate was also higher for the reaction employing excess acid chloride. Both products appeared at the 0.5 h interval as compared to the 1.5 h mark for the reaction utilizing only one equivalent of benzoyl chloride. However, the relative ratios of esters remained at 2:1.

Table 3. Gas Chromatographic Analysis of THF Cleavage without Aluminum: Relative Rates of Appearance of Products.[1]

Time/hr	$[PhCOO(CH_2)_4Cl]^{2,3}$	$[PhCOOCH_3]^{2,4}$	$[PhCOO(CH_2)_4Cl]/[PhCOOCH_3]$	$[Cyclohexene]^{2,5}$
0.5	-	-	-	-
1	-	-	-	0.69
1.5	-	-	-	-
2	4.5	4.5	1 : 1	0.75
2.5	7.1	9	1 : 1.2	27.2
3	14.2	16.2	1 : 1.1	30.6
4	25.8	20.8	1 : 0.8	36.7
16	48.9	59.1	1 : 1.2	74.2

[1] Reaction was carried out on 125 mg (1.73 mmol) THF, 1.2 eq (0.69 g) *trans*-2-chloromercuri-1-methoxycyclohexane, 1 eq (200.8 μL) PhCOCl in toluene (100°C).
[2] The concentration (mM) was calculated relative to that of *p*-xylene (5% internal standard, R_t = 4.87 min.).
[3] R_t = 12.77 min.; Percent yield of pure isolated product is 28.3%.
[4] R_t = 7.44 min.; Percent yield of pure isolated product is 40%.
[5] R_t = 2.52 min.

Table 4. Gas Chromatographic Analysis of THF Cleavage with Aluminum: Relative Rates of Appearance of Products.[1]

Time/hr	$[PhCOO(CH_2)_4Cl]^{2,3}$	$[PhCOOCH_3]^{2,4}$	$[PhCOO(CH_2)_4Cl]/[PhCOOCH_3]$	$[Cyclohexene]^{2,5}$
0.5	-	-	-	-
1	-	-	-	2.2
1.5	9.29	-	-	7.6
2	22.2	12.8	1 : 0.58	23.3
2.5	27.5	18.9	1 : 0.69	26.2
3	44	26	1 : 0.61	31.2
3.5	55	30	1 : 0.55	79.4
4	91	50	1 : 0.55	151

[1] Reaction was carried out on 125 mg (1.73 mmol) THF, 1.2 eq (0.69 g) *trans*-2-chloromercuri-1-methoxycyclohexane, 1 eq (200.8 μL) PhCOCl, 0.4 eq (18.6 mg) Al foil.
[2] The concentration (mM) was calculated relative to that of *p*-xylene (5% internal standard, R_t = 4.90 min.).
[3] R_t = 12.74 min.; Percent yield of pure isolated product is 53%.
[4] R_t = 7.44 min.; Percent yield of pure isolated product is 25%.
[5] R_t = 2.47 min.

In order to determine the importance of the organomercurial (R_t = 13.65 min.) to the progress of the reaction, a control reaction[26] was conducted under similar molar quantities of substrate, reagents and conditions for the same period (16 h) but in the absence of alkoxycyclohexylchloromercurial. GC analysis of the reaction mixture after 16 h revealed the absence of any peaks corresponding to R_t = 12.77 min. (4-chlorobutyl benzoate) or R_t = 7.44 min. (methyl benzoate).

In summary, the employment of alkoxycycloalkylchloromercurials (**1-5**) in conjunction with acid chlorides and aluminum metal is an effective reagent system for the acylative cleavage of epoxides and tetrahydrofurans to chloroalkyl esters. When compared to existing reagent systems, the alkoxycycloalkylchloromercurials are easily and safely prepared in large quantities and high yields at moderate cost ($0.23/g) and are shelf-stable. Furthermore, a wide range of acid chlorides and organomercurials may be employed for the cleavage reaction. The cleavage reactions may be conducted under mild conditions and are devoid of the tars and polymeric by-products that can complicate the purification procedure. The workup of the reaction mixtures and the removal of the non-halogenated by-products is easily facilitated and the overall process affords the chloroester products[27] in modest to good yields. Using either substituted epoxides or 2-substituted tetrahydrofurans as substrates, the attack of chloride is at the more substituted carbon thereby suggesting an S_N1-type mechanism.

Experimental

General Methods. ^1H and ^{13}C NMR spectra were recorded on a Bruker AMX 500 with $CDCl_3$ as the solvent. IR spectra (cm^{-1}) were recorded using a Mattson Galaxy 5000 FT instrument. Gas chromatographic analyses were carried out with a Hewlett Packard 5890 instrument equipped with an HP1 FID detector, 12x0.2 mm column and He carrier gas. Glass-backed silica gel plates (E. Merck 5715) were used for thin-layer chromatographic anlyses which utilized anisaldehyde stain or UV lamp for visualization after development. Kieselgel 60 (E. Merck, 7734, 70-230 mesh) was employed for standard gravity column chromatographic separations while flash-column chromatographic separations utilized Kieselgel 60 (E. Merck 9385, 230-400 mesh). Heavy duty food-grade aluminum foil was prepared for the reaction by cutting into 5 mm wide strips, coiling the strips about a 5 mm glass rod and degreasing the coils by rinsing in ether. Tetrahydrofuran (THF) was distilled from sodium/benzophenone and toluene was distilled from sodium prior to use. The solvents used in chromatographic separations were ACS grade and were used as commercially supplied. Celite filtrations were done using Celite 521. The organomercurials **1-5** were prepared by the method of Brown.[19] Their spectral data are in agreement with expected values. High resolution mass spectral determinations were performed at the Nebraska Center for Mass Spectrometry, Lincoln, Nebraska.

4-Chlorobutyl acetate (Entry 4, Table 1) Typical Procedure. *trans*-2-Chloromercuri-1-methoxycyclohexane (**1**) (0.55 g, 1.65 mmol) was dissolved in distilled toluene (5 mL). To this solution, freshly distilled tetrahydrofuran (100 mg, 1.4 mmol) was added, followed by acetyl chloride (260 μL, 3.4 mmol) then aluminum foil (15 mg, 0.55 mmol). A cold finger condenser was then fitted to the reaction flask and the mixture was warmed at 50°C (18 h). Thin layer chromatographic analysis (hexane/ether, 1:1) was used to monitor the product formation. The reaction mixture was then cooled to room temperature, diluted with ether and filtered through Celite.[28] The concentrated filtrate was then purified by chromatography on silica gel

(hexane/ether, 9:1) followed by Kugelröhr distillation to give the chloroester (160 mg, 77%); TLC: *Rf*=0.11 (hexane/ether, 9:1); b.p. 103-104°C/1.0 mmHg (lit. b.p. 92-93°/20 mmHg.)[10]

4-Chlorobutyl benzoate (Entries 1-3, Table 1): Purification of the oily chloroester (390 mg, 53%) was achieved by column chromatography (hexanes/ether, 9:1) followed by Kugelröhr distillation. TLC: R_f=0.38 (hexane/ether, 9:1); b.p. 128-129°C/1.0 mmHg (lit. b.p. 142-144°/5 mmHg).[12]

4-Chlorobutyl chloroacetate (Entry 5, Table 1): The oily chloroester was column-chromatographed (hexanes/ether, 9:1) then Kugelröhr-distilled (203 mg, 80%); TLC: R_f=0.09 (hexane/ether, 9:1); b.p. 185-187°C/0.9 mmHg; ^{1}H NMR ($CDCl_3$): δ 4.20-4.18 (t, 2H); 4.03 (s, 2H); 3.54-3.52 (t, 2H); 1.83-1.81 (m, 4H). ^{13}C NMR ($CDCl_3$): δ 167.5, 65.4, 44.3, 40.8, 28.9, 25.8. FTIR (KBr, CH_2Cl_2): 1755 cm^{-1}; HRMS calcd for C_9H_8ClO (M–$COCH_2Cl$) 107.0263, found 107.0263.

4-Chloropentyl benzoate (Entry 6, Table 1): The oily chloroester was column-chromatographed (hexane/ether, 14:1) then Kugelröhr-distilled (0.7 g, 58%) TLC: R_f=0.18 (hexane/ether, 14:1); b.p. 140-142°C/0.9 mmHg (lit. b.p. 129-132°/0.45 mmHg).[13]

***trans*-2-Chlorocyclohexyl benzoate (Entries 1-5, Table 2):** Column chromatography (hexane/ether, 14:1) and Kugelröhr-distillation gave the chloroester as a yellow oil (730 mg, 60%); TLC: R_f=0.32 (hexane/ether, 14:1); b.p. 186-187°C/0.5 mmHg. The NMR and IR spectral data were consistent with previously reported values.[7]

2-Chlorocyclohexyl-*E*-2-butenoate (Entry 6, Table 2): The chloroester (304 mg, 60%) was purified by column chromatography (hexanes/ether, 20:1) followed by Kugelröhr distillation. TLC: R_f=0.17 (hexane/ether, 20:1); b.p. 135-136°C/0.9 mmHg. The NMR and IR spectral data were consistent with previously reported values.[7]

***trans*-2-Chlorocyclohexyl pivalate (Entry 7, Table 2):** Column chromatography on silica gel (hexane/ether, 9:1) followed by Kugelröhr-distillation provided the oily pivalate (186 mg, 52%). TLC: R_f=0.18 (hexane/ether, 9:1); b.p. 114-115°C/0.45 mmHg. ^{1}H NMR ($CDCl_3$): δ 4.69-4.67 (m, 1H, J=5 Hz); 3.79-3.78 (m, 1H, J=5 Hz); 2.11 (m, 1H); 1.983-1.98 (m, 1H); 1.66-1.61 (m, 3H); 1.32-1.25 (m, 3H); 1.14 (s, 9H). ^{13}C NMR ($CDCl_3$): δ 178.5, 75.1, 60.5, 38.6, 34.5, 30.3, 27, 24.3, 23. FTIR (KBr, CH_2Cl_2): 1730 cm^{-1}; HRMS Calcd for $C_6H_{10}Cl$ (M–*tert*-$BuCO_2$) 117.0471, found 117.0450.

***trans*-2-Chlorocyclohexyl acetate (Entry 8, Table 2):** Purification of the oily chloroester (95 mg, 53%) was achieved by column chromatography (hexanes/ether, 20:1) followed by Kugelröhr distillation. TLC: R_f=0.23; (hexane/ether, 10:1). b.p. 110°C/0.5 mmHg. The NMR and IR spectral data were consistent with previously reported values.[7]

2-Chloro-2-phenethyl acetate (Entry 9, Table 2): Column chromatography on silica gel (hexane/ether, 30:1) gave the oily secondary chloride as the only regioisomer (158 mg, 96%). TLC: R_f=0.2 (hexane/ether, 30:1). The IR and NMR spectral data were consistent with previously reported values.[4]

2-Chloro-2-phenethyl benzoate and 2-chloro-1-phenethyl benzoate (Entry 10, Table 2): Column chromatography (hexanes/ether, 25:1) afforded the mixture of regioisomeric esters (134 mg, 63%) in a 2:1 ratio in favor of the secondary chloride as confirmed by ^{1}H NMR analysis; . TLC: R_f=0.27 (hexane/ether, 15:1). The spectral data for both compounds were consistent with previously reported values.[29]

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