Indium(III) Chloride-Promoted Rearrangement of Epoxides: A Selective Synthesis of Substituted Benzylic Aldehydes and Ketones

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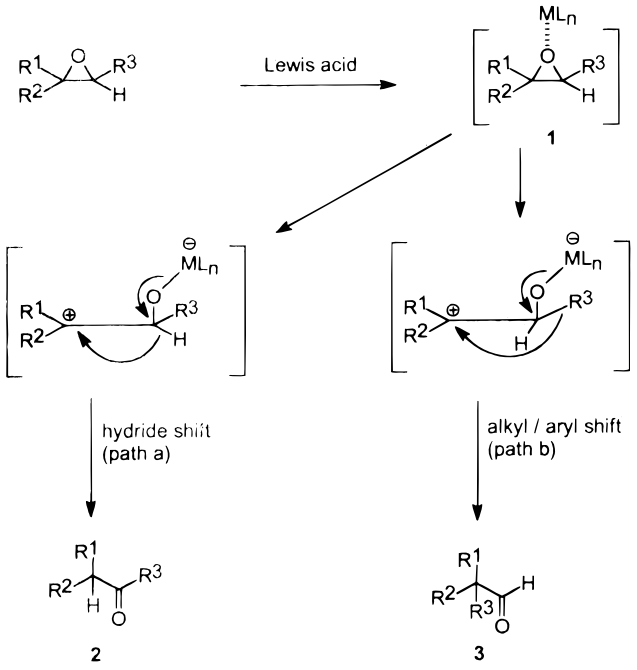
A simple and efficient procedure for the rearrangement of substituted epoxides catalyzed by $InCl_3$ has been developed. Aryl-substituted epoxides isomerize with complete regioselectivity to form a single carbonyl compound via cleavage of the benzylic C−O bond. The reactions are simple, fast, and high yielding. This procedure is very mild compared to those catalyzed with BF_3 and other Lewis acids and compatible with several acid-sensitive functionalities. This protocol provides a highly selective synthesis of substituted benzylic aldehydes and ketones. However, rearrangement of alkyl-substituted epoxides is not very selective.

Introduction

Epoxides are one of the most useful and versatile substrates in organic synthesis due to their high reactivity[1] and easy availability through a wide variety of methods,[2] often with high levels of relative and absolute stereocontrol.[3] One of the most frequently used atom-economical reactions of epoxides is their rearrangement to carbonyl compounds, and a number of reagents including a variety of Lewis acids[4] have been elaborated for this purpose. In principle, two types of rearrangements are possible for substituted epoxides depending on the migration pathways following Lewis acid promoted C−O bond cleavage (Scheme 1). The rearrangement of **1** with hydride (path a) or alkyl/aryl migration (path b) would lead to ketone **2** or aldehyde **3**, respectively. Unless there is a structural or a stereochemical bias, usually a mixture of products is obtained due to lack of regioselectivity in the ring opening step. Moreover, lack of chemoselectivity among various substituted epoxides limits the synthetic utility of this reaction in a multistep synthesis. Although, recent uses of palladium catalysts and metalloporphyrin in the isomerization of epoxides constitute a useful addition,[5] an efficient method for the regioselective rearrangement of epoxide to either aldehyde or ketone depending on the choice of Lewis acid is still well appreciated.

Scheme 1



In recent years, emergence of indium(III) chloride[6] as mild Lewis acid for effecting a variety of chemical transformations in chemo-, regio-, and stereoselective fashion prompted us to initiate a systematic study of this important reaction catalyzed by indium(III) chloride. We have discovered that $InCl_3$ is very efficient in effecting the rearrangement of epoxides to the corresponding carbonyl compounds very selectively (Scheme 2).

Results and Discussion

The epoxides employed in this study are, in general, prepared from the corresponding olefins by reaction with *m*-chloroperoxybenzoic acid following standard procedures.[2,5] The conjugated carbonyl compounds and ni-

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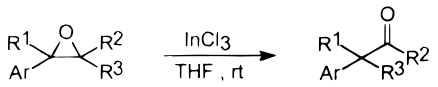
Scheme 2



R^1, R^2, R^3 = H, alkyl or phenyl

troolefin are epoxidized by treatment with alkaline hydrogen peroxide.[7] A few aryl-substituted epoxides are obtained by reaction of the corresponding aldehydes and ketones with dimethylsulfonium methylide.[8]

The experimental procedure for rearrangement of epoxide is very simple. The epoxide was stirred with a suspension of $InCl_3$ in THF at room temperature for a certain period of time as required to complete the reaction (TLC). Usual workup and evaporation of solvent followed by purification through column chromatography furnished the pure product. In general, 0.5−0.6 equiv of $InCl_3$ was found to give the best results although in a couple of reactions a larger amount has been required.

A wide range of structurally varied epoxides were subjected to rearrangement with $InCl_3$ by this procedure to provide the corresponding carbonyl compounds in good yields as summarized in Table 1. 1-Aryl-, 1,1-diaryl-, and 1,1-alkyl, aryl-substituted epoxides (entries 1−10) underwent rearrangement by exclusive hydride shift (path a) to give the respective aryl-substituted acetaldehydes as the only isolable compounds. The rearrangement of tri- and tetrasubstituted aryl epoxides is greatly influenced by the nature of the substituent at the carbon β to the aryl ring. In a competition between hydrogen and methyl or substituted methyl, hydrogen migration occurred selectively producing the corresponding methyl ketones (entries 11−14). However, methyl migration is observed in substrates bearing two CH_3 groups at the β-position (entry 21). In aromatic ring fused and aryl-substituted cycloalkene oxides exclusively the β-hydrogens migrate giving the corresponding ketones (entries 22−24). On the other hand, phenyl beats hydrogen as observed in the isomerization of stilbene oxides (entries 15 and 16) to the corresponding diphenylacetaldehydes. These reactions are often problematic with other Lewis acids due to competitive hydride vs phenyl migration.[4a,9] Acetyl and benzoyl groups also migrate in preference to hydrogen in the reactions of 1-acetyl- and 1-benzoylstyrene oxides (entries 17 and 18).[10] However, the benzoyl epoxide (entry 18) after initial benzoyl shift undergoes retroaldol cleavage with loss of the formyl group to give the final ketone. This observation, though interesting, is not unusual.[11] The substitution of electron withdrawing groups such as CO_2Me and NO_2 at the β-carbon retards the usual isomerization to carbonyl compounds through hydride or alkyl shift possibly because of less electron density at the β-center, and thus the oxirane ring is cleaved by the nucleophilic attack of the Cl^- ion from $InCl_3$ to produce the corresponding chloro compounds (entries 19 and 20). The loss of nitro group in the cleavage of nitroepoxides (entry 20) is not unexpected.[7b] The isomerization of nonaromatic epoxide to the carbonyl compound by the present procedure is not very encouraging. Presumably, the incipient carbocation formed by the initial cleavage of epoxide (Scheme 1) is much less stabilized in alkyl-substituted epoxide compared to that in the aryl substituted one. Thus, in the α-pinene oxide (entry 25) where the carbocation is better stabilized on a tertiary center, rearrangement leads to the expected aldehyde,[12] but in cyclohexene oxide (entry 26) where such stabilization is absent chlorohydrin is formed by the nucleophilic attack of the Cl^- ion. The methylenecyclohexane oxide (entry 27) being an intermediate case leads to a mixture of products containing cyclohexanealdehyde, chlorohydrin, and some other unidentified compounds, and in general, it was found that nonaromatic epoxides without having any strong carbonium stabilizing component afforded a mixture of products of which chlorohydrin is the main constituent. Use of solvents of different polarity also does not make any difference.

These reactions are usually fast and high yielding. Requirement of $InCl_3$ in these reactions is not stoichiometric, rather catalytic; however an optimum amount is necessary. Most significantly $InCl_3$ is unique in pushing the reaction to one direction providing one compound exclusively or predominantly; the best comparison being the rearrangement of 1-phenyl-2-nitro-1,2-epoxypropane (entry 20) where a BF_3-catalyzed reaction furnished a mixture of five compounds.[7] Moreover, its mode of action in epoxide rearrangement is possibly different from other Lewis acids, e.g., BF_3, SbF_5, and MABR [methylaluminum bis(4-bromo-2,6-di-*tert*-butyl phenoxide)] produced the ring-contracted 1-phenylcyclopentanealdehyde from the reaction of 1-phenyl-1,2-epoxycyclohexane with complete selectivity,[4b] whereas $InCl_3$ afforded 2-phenylcyclohexanone as the major product (entry 24) with less than 5% of the ring contracted aldehyde. In another example, *trans*-stilbene oxide produced a mixture of the corresponding ketone and aldehyde in varying amounts with $MgBr_2$,[4d] while by the present procedure the aldehyde was obtained as the sole product (entry 15). Again, *trans*-1-benzoyl-2-phenylethylene oxide was isomerized to the respective keto aldehyde by $BF_3{-}Et_2O$,[4a] but $InCl_3$ provides the corresponding deformylated product (entry 18) as the only isolable compound. $InCl_3$ is mild enough not to induce any isomerization in allyl and propargyl moieties (entries 6 and 7). Acid sensitive functionalities such as methoxyl (entries 2, 3, and 13) and carbomethoxyl (entries 10 and 19) remained unaffected under the reaction conditions.

From the results of rearrangement of a variety of epoxides employed in this study, it was found that aryl-substituted epoxides isomerize with complete regioselectivity via cleavage of the benzylic C−O bond and this isomerization process follows a uniform pattern of migration. Thus, between the groups present at the β-position of the aryl ring, hydrogen migrates in preference to methyl or substituted methyl, whereas phenyl, acetyl, and benzoyl precede hydrogen. This unique selectivity provided by this $InCl_3$-catalyzed procedure makes this reaction a viable alternative to Wacker oxidation of vinylarenes[13] and homologation of carbonyl compounds

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(12) Lewis, J. B.; Hedrick, G. W. *J. Org. Chem.* **1965**, *30*, 4271.

Table 1. Rearrangement of Epoxide with $InCl_3$ in THF

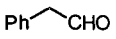
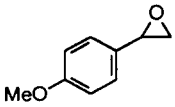
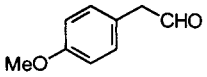
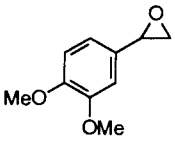
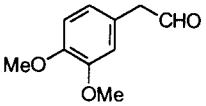
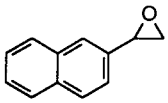
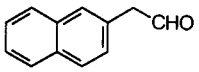
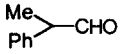
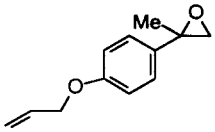
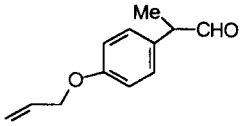
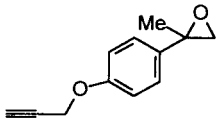
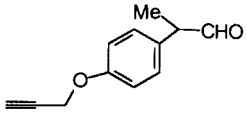
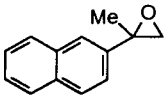
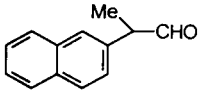
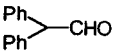
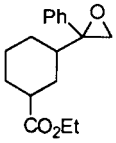
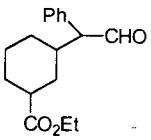
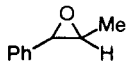
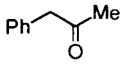
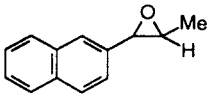
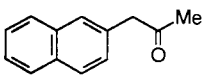
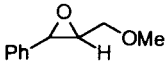
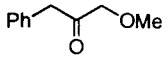
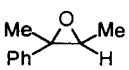
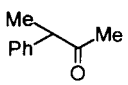
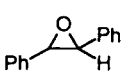
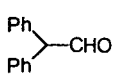
entry	epoxide	time	product	yield(%)[a]	ref
1		15 min		85	14
2		10 min		90	
3		10 min		91	
4		15 min		89	5
5		30 min		86	14
6		30 min		95	
7		30 min		92	
8		15 min		88	5
9		15 min		90	14
10		50 min[b]		88	
11		50 min		90	14
12		45 min		88	5
13		1 h[c]		65	15
14		1 h		86	16
15		25 min		90	14

Table 1. (Continued)

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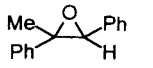
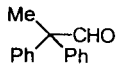
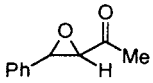
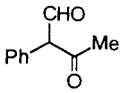
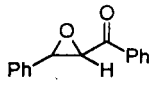
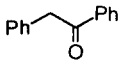
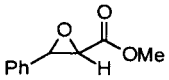
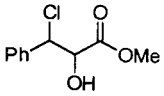
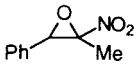
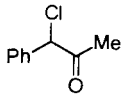
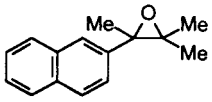
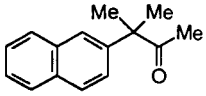
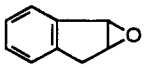
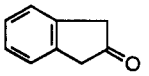
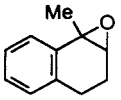
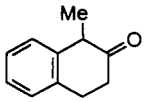
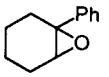
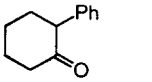
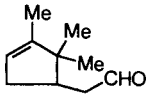
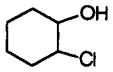
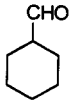
16		1 h		86	
17		1 h[c]		72	10a
18		3.5 h[c]		75	14
19		2 h[c]		80[d]	
20		1.5 h[c]		70	
21		6 h[c]		78	
22		45 min		85	14
23		45 min		87	14
24		40 min		88	14
25		20 min[d]		85	12
26		50 min[b]		90	14
27		30 min[c]		50[e]	14

[a] The yields refer to pure isolated products, properly characterized by spectral data. [b] 1.6 Equivalent of $InCl_3$ has been used. [c] The reaction was carried out by refluxing with 1 equivalent of $InCl_3$. [d] The product was a 2:1 mixture of *syn* and *anti* diastereoisomers (NMR). [e] Chlorohydrin and other unidentified products are also formed.

to higher member through traditional methods. The exact reason for this selectivity offered by $InCl_3$ is not very clear; however, its mild Lewis acid character may be an important factor.

Conclusion

This $InCl_3$-promoted procedure provides an efficient methodology for a highly regioselective isomerization of aryl-substituted epoxides and demonstrates a useful protocol for the high yield synthesis of benzylic aldehydes and ketones with complete predictability. The other notable advantages offered by this procedure are (a) fast reaction (usually 10 to 50 min), (b) mild reaction condition (room temperature), (c) simplicity in operation (no special apparatus or technique required), and (d) mild nature of $InCl_3$ in comparison to BF_3 and other Lewis acids and its compatibility with sensitive functionalities such as

(13) Wacker oxidation of β-methylstyrene gave a 3:1 mixture of 1-phenyl-2-propanone and 1-phenyl-1-propanone: Kienan, E.; Seth, K. K.; Lamed, R. *J. Am. Chem. Soc.* **1986**, *108*, 3474.

OMe, CO_2Me, and double and triple bonds. We believe this method will find many useful applications in organic synthesis.

Experimental Section

General. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ solution at 300 and 75 MHz, respectively. Analyses were done on a Perkin-Elmer 2400 autoanalyzer. Indium(III) chloride was purchased from Aldrich and used as such. Tetrahydrofuran (THF) was distilled over potassium−benzophenone immediately before use. Thin-layer chromatography was done on precoated silica gel plates (E. Merck). Silica gel (60−120 mesh, SRL, India) was used for column chromatography.

Epoxides used in this investigation were prepared either from olefins or from carbonyl compounds following reported procedures.[2,5,7,8] A few epoxides which were not previously reported in the literature have been identified from their ^1H NMR spectra and analysis. These data are included in Supporting Information.

General Procedure for Rearrangement of Epoxides. Representative Procedure. A solution of styrene oxide (240 mg, 2 mmol) in THF (2 mL) was added to a stirred suspension of $InCl_3$ (265 mg, 1.2 mmol) in THF (3 mL) at room temperature (25 °C) under nitrogen, and stirring was continued for 15 min for a complete reaction (TLC). The reaction mixture was quenched with brine and extracted with ether. The ether extract was dried over Na_2SO_4 and evaporated to leave a crude product which was purified by column chromatography over silica gel to afford pure phenylacetaldehyde (204 mg, 85%) which is easily identified by comparison of its spectra (IR and NMR) with those of an authentic sample.[14]

This procedure is followed for the rearrangement of all the epoxides included in Table 1. Many of these products are known compounds and were easily characterized by comparison with authentic samples. Those which are unknown have been identified by their spectral (IR, ^1H and ^{13}C NMR) and analytical data. These data are presented below in order of their entries in Table 1.

2-(4-Methoxyphenyl)ethanal (entry 2):IR (neat) 1515, 1610, 1725 cm^{-1}; ^1H NMR δ 3.69 (d, J = 2.4 Hz, 2H), 3.80 (s, 3H), 6.94 (d, J = 6.3 Hz, 2H), 7.13 (d, J = 6.9 Hz, 2H), 9.72 (t, J = 2.4 Hz, 1H); ^{13}C NMR δ 48.3 (CH_2), 53.9 (CH_3), 112.5 (C), 113.0 (2 CH), 126.2 (C), 129.3 (2 CH), 198.3 (CH). Anal. Calcd for $C_9H_{10}O_2$: C, 71.97; H, 6.72. Found: C, 72.15; H, 6.78.

2-(3,4-Dimethoxyphenyl)ethanal (entry 3): IR (neat) 1520, 1610, 1725 cm^{-1}; ^1H NMR δ 3.63 (d, J = 2.4 Hz, 2H), 3.87 (s, 6H), 6.70−6.89 (m, 3H), 9.73 (t, J = 2.4 Hz, 1H); ^{13}C NMR δ 50.1 (CH_2), 55.8 (2 CH_3), 110.3 (C), 111.4 (CH), 112.4 (CH),121.0 (CH), 126.8 (C), 124.0 (C), 199.5 (CH). Anal. Calcd for $C_{10}H_{12}O_3$: C, 66.64; H, 6.72. Found: C, 66.82; H, 6.74.

2-[4-(2-Propenoxy)phenyl]propanal (entry 6): IR (neat) 1510, 1580, 1610, 1650, 1720 cm^{-1}; ^1H NMR δ 1.40 (d, J = 6.9 Hz, 3H), 3.56 (m, 1H), 4.51−4.53 (m, 2H), 5.25−5.43 (m, 2H), 5.97−6.10 (m, 1H), 6.92 (d, J = 9 Hz, 2H), 7.11 (d, J = 9 Hz, 1H), 9.62 (d, J = 0.9 Hz, 1H); ^{13}C NMR δ 14.5 (CH_3), 52.0 (CH), 68.7 (CH_2), 115.3 (2 CH), 117.5 (CH_2), 133.1 (CH), 158.0 (C), 129.71 (2 CH), 130.5 (C), 200.9 (CH). Anal. Calcd for $C_{12}H_{14}O_2$: C, 75.75; H, 7.42. Found: C, 75.48; H, 7.46.

2-[4-(2-Propynoxy)phenyl]propanal (entry 7): IR (neat) 1510, 1610, 1720 cm^{-1}; ^1H NMR δ 1.34 (d, J = 7.2 Hz, 3H), 2.45 (t, J = 2.4 Hz, 1H), 3.51 (m, 1H), 4.62 (d, J = 2.4 Hz, 2H), 6.92 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 6.6 Hz, 2H), 9.57 (d, J = 0.9 Hz, 1H); ^{13}C NMR δ 15.0 (CH_3), 52.5 (CH), 56.2 (CH_2), 76.0 (CH), 97.4 (C), 115.0 (2 CH, C), 129.9 (2 CH, C), 201.4 (CH). Anal. Calcd for $C_{12}H_{12}O_2$: C, 76.56; H, 6.43. Found: C, 76.78; H, 6.54.

2-(3-Carboethoxycyclohexyl)-2-phenylethanal (entry 10): IR (neat) 1450, 1600, 1730 (broad) cm^{-1}; ^1H NMR δ 1.27 (t, J = 7 Hz, 3H), 1.54−2.32 (m, 10H), 3.66 (m, 1H), 4.15 (q, J = 7 Hz, 2H), 7.17−7.58 (m, 5H), 9.70 (d, J = 3.3 Hz, 1H); ^{13}C NMR δ 14.1 (CH_3), 21.9 (CH_2), 25.0 (CH_2), 28.2 (CH_2), 30.9 (CH_2), 38.9 (CH), 42.5 (2 CH), 127.2 (CH), 128.66 (CH), 128.9 (CH), 129.3 (CH), 129.4 (CH), 132.9 (2 C), 175.1 (C), 200.3 (CH). Anal. Calcd for $C_{17}H_{22}O_3$: C, 74.41; H, 8.09. Found: C, 74.63; H, 8.18.

2,2-Diphenylpropanal (entry 16): IR (neat) 1450, 1490, 1600, 1725 cm^{-1}; ^1H NMR δ 1.78 (s, 3H), 7.10−7.40 (m, 10H), 9.93 (s, 1H); ^{13}C NMR δ 22.5 (CH_3), 65.5 (C), 126.4 (2 C), 127.5 (2 CH), 127.7 (4 CH), 128.1 (4 CH), 199.6 (CH). Anal. Calcd for $C_{15}H_{14}O$: C, 85.67; H, 6.72. Found: C, 85.36; H, 6.54.

Methyl (3-chloro-2-hydroxy-3-phenyl)propionate (a 2:1 mixture of syn:anti by NMR) (entry 19): IR (neat) 700, 1455, 1495, 1600, 1750, 3300−3600 (braod) cm^{-1}; ^1H NMR δ 3.08 (broad, 1H), 3.74 (s, <3H), 3.86 (s, <3H), 4.54 (d, J = 2.4 Hz, <1H), 4.66 (d, J = 4.2 Hz, <1H), 5.23 (d, J = 4.2 Hz, <1H), 5.33 (d, J = 2.4 Hz, <1H), 7.33−7.54 (m, 5H). Anal. Calcd for $C_{10}H_{11}O_3Cl$: C, 56.06; H, 5.18. Found: C, 55.76; H, 5.28.

3-Chloro-3-phenyl-2-propanone (entry 20): IR (neat) 700, 1350, 1610, 1720 cm^{-1}; ^1H NMR δ 2.22 (s, 3H), 5.35 (s, 1H), 7.37−7.49 (m, 5H); ^{13}C NMR δ 25.7 (CH_3), 66.5 (C), 127.8 (2 CH, C), 129.0 (2 CH, C), 200.1 (C). Anal. Calcd for C_9H_9OCl: C, 64.27; H, 5.40. Found: C, 64.36; H, 5.42.

3-Methyl-3-naphthyl-2-butanone (entry 21): IR (neat) 1600, 1650, 1710 cm^{-1}; ^1H NMR δ 1.61 (s, 6H), 1.96 (s, 3H), 7.28−7.30 (m, 1H), 7.51 (m, 2H), 7.88−7.96 (m, 4H); ^{13}C NMR δ 25.0 (2 CH_3), 25.6 (CH_3), 52.6 (C), 124.1 (CH), 124.5 (CH), 125.9 (CH), 126.2 (CH), 127.5 (CH), 127.6 (CH), 128.4 (CH), 132.2 (C), 133.4 (C), 141.5 (C), 211.3 (C). Anal. Calcd for $C_{15}H_{16}O$: C, 84.86; H, 7.60. Found: C, 85.04; H, 7.70.

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Supporting Information Available: Spectroscopic and analytical data for epoxides (entries 3, 6, 7, 10, and 13) included in Table 1 (1 page). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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