Table II. Relative Solubility of Imidazole in Chlorinated Hydrocarbon and Tetrahydrofuran Solvents

solvent	solubility[a] (mg/mL)	partition coeff[b]	ΔG^e (kcal/mol)
CH_2Cl_2	27.5	1.5×10^{-2}	0.0
$CHCl_3$	25.8	1.3×10^{-2}	+0.04,[c] +0.08[d]
CH_3CCl_3	0.08	7.1×10^{-4}	+3.5,[c] +1.8[d]
$CHCl_2CHCl_2$	37.4	2.2×10^{-2}	−0.2,[c] −0.2[d]
THF	41.3		−0.3[c]
2-MeTHF	18.0		+0.3[c]
2,5-Me_2THF	7.3		+0.8[c]
2,2-Me_2THF	10.5		+0.6[c]
2,2,5,5-Me_4THF	1.58		+1.7[c]
tetrahydropyran	16.1		+0.3[c]
dioxane	30.5		−0.1[c]
t-BuOMe	1.18		+1.9[c]

[a] [Imidazole] in a saturated solution of the solvent shown. [b] $[\text{Imidazole}]_{\text{organic solvent}}/[\text{imidazole}]_{\text{water}}$ by weight after vigorously stirring 1 M imidazole in water with an equal volume of organic solvent (25 °C). [c] From solubility. [d] From partition coefficient. [e] Solvation energies of imidazole in various solvents relative to methylene chloride.

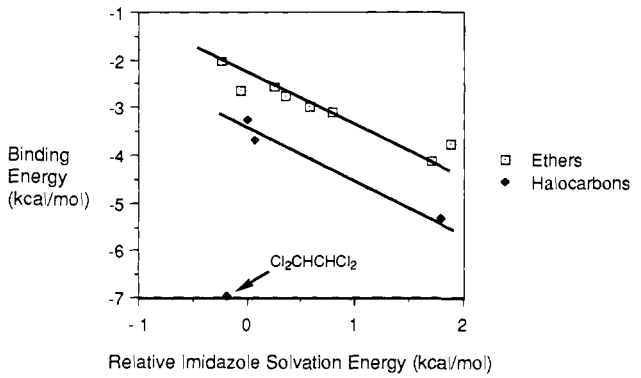

Figure 2. Correlation between 1/imidazole association and imidazole solvation in solvents listed in Table II.

Such interactions might include, for example, solute hydrogen bonding to the ethereal oxygen of the tetrahydrofuranoid solvents in inverse proportion to the extent of hindering methylation.

Considering the effect of specific solvent/solute interactions upon the position of the binding equilibrium above, we note that imidazole must undergo substantial desolvation as it is encapsulated by **1** during binding. Other effects being comparable, we would therefore expect a decrease in the extent of solvation of the free substrate imidazole to favor its binding to **1**. To measure the relative solvation free energy of imidazole in various solvents, we carried out the solubility and partition experiments summarized in Table II. Upon plotting the relative free energies of imidazole solvation against the association energies of **1** and imidazole (Figure 2), we find a strong inverse correlation ($R > 0.95$, slope $\cong -1.0$) for many of the solvents examined. The unitary relation of binding and imidazole solvation shows that the relative solvation energies of **1** and **2** are similar for most solvents of the same class and that it is differential imidazole solvation which is primarily responsible for shifting of the binding equilibrium.[5]

Some solvents, most notably 1,1,2,2-tetrachloroethane, lie well off the correlation lines in Figure 2. Such deviations imply differential solvation not of the substrate but of either the host **1** or the complex **2**.[6] We believe the best explanation of the 1,1,2,2-tetrachloroethane result lies in an especially poor solvation of **1**. In particular, we suggest that the binding cavity is sensitive to the size and shape of the solvent molecules and that large solvents do not penetrate and solvate it as well as smaller ones. By using solvents which do not interact favorably with internal binding cavities, a binding site need not be extensively desolvated to accept and bind a smaller or more appropriately shaped substrate. Complex formation should thus be favored in solvents whose molecular dimensions are large relative to those of the binding site.

In conclusion, the stability of our molecular complex is highly dependent upon the detailed structure of the solvent molecules employed. Binding is favored by solvents which selectively provide weak solvation of uncomplexed components or strong solvation of the molecular complex. Our results provide examples of the former mechanism of complexation enhancement. If our hypothesis of differential solvation of the substrate binding site as a function of solvent molecule size is correct, then other host molecules with preorganized three-dimensional cavities should exhibit increases in association constant in media composed of increasingly bulky solvent molecules. The solvent effects described here should facilitate the study of many molecular complexes which are not stable enough to be readily observed in more traditional organic solvents.[7]

(5) Using imidazole solvation energy as a standard, the somewhat tighter association of **1** and imidazole (Figure 2) in the chlorocarbon solvents implies an enhanced solvation of **2** or diminished solvation of **1** relative to the ethereal solvents.

(6) No chemical change to **1** (e.g., formation of ammonium salts) seems to occur in tetrachloroethane: the same binding constants were found in methylene chloride solution whether or not **1** had been previously treated with 1,1,2,2-tetrachloroethane.

(7) This work was supported by Grant CHE86-05891 from the National Science Foundation and an American Cancer Society fellowship to K.T.C.

Glycidyl Derivatives as Chiral C_3 Synthons. Ring Opening Catalyzed by BF_3 Etherate

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Derivatives of glycidol such as the 4-nitrobenzoate[1] and arenesulfonates[2] possess widespread synthetic utility as chiral building blocks because of their stability and convenience in preparation. Since some glycidyl derivatives can be obtained in high optical purity,[3] we have sought to prepare some monoprotected 1,2-diols via nucleophilic opening reactions. Although a variety of Lewis acids have been found to be effective mediators of regio- and stereoselective epoxide openings,[4] no reports of Lewis acid mediated opening of glycidyl derivatives have appeared.[5] The well-known Ti(OPr-*i*)$_4$-mediated epoxy alcohol opening process[4g,h] *failed* to afford the desired opening of glycidyl derivatives **1-3** when benzyl alcohol, thiophenol, and various long-chain alcohols

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Table I. Opening of Glycidyl Derivatives with Nucleophiles Mediated by BF_3 Etherate[a]

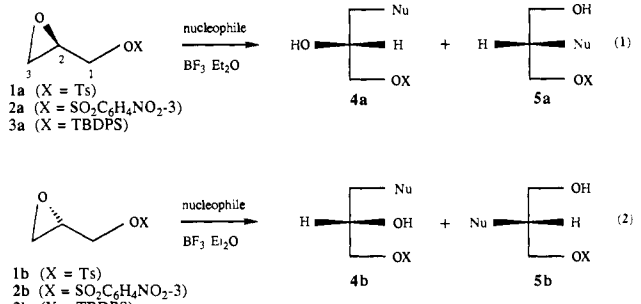
entry	epoxide	Nu	yield[b]	% 4/% 5[c]	HPLC R_t (min)[d] 4a	HPLC R_t (min)[d] 4b	% ee by HPLC[e] 4a	% ee by HPLC[e] 4b	% ee by NMR[f] 4a	% ee by NMR[f] 4b
1	**1a**	BnO	84	100/0	17.3[g]		96.2		95.0	
2	**1b**	BnO	81	100/0		19.3[g]		95.3		94.0
3	**1a**	PhS	81	100/0	13.4[h]		92.3			
4	**1b**	PhS	83	100/0		14.8[h]		95.3		
5	**1a**	$C_{16}H_{33}O$	80	100/0	17.2[i]		94.0 (97.7)[j]		~99	
6	**1b**	$C_{16}H_{33}O$	79	100/0		19.0[i]		95.7		~99
7	**2a**[k]	$C_{18}H_{35}O$[l]	75	100/0	21.5[m]		93.3			
8	**2b**[k]	$C_{18}H_{35}O$[l]	77	100/0		23.4[m]		88.4		
9	**2a**	$C_{16}H_{33}O$	83	100/0	21.1[m]		94.1			
10	**2b**	$C_{16}H_{33}O$	80	100/0		23.0[m]		86.9		
11	**2a**	$C_{18}H_{35}O$[n]	78	100/0	23.8[o]		92.1			
12	**2b**	$C_{18}H_{35}O$[n]	75	100/0		25.7[o]		85.6		
13	**3a**	BnO	70	89/11	15.1[p]		94.4		93.6	
14	**3b**	BnO	68	89/11		16.8[p]		95.3		94.5
15	**3a**	PhS	79	93/7	13.3[q]		93.7		92.5	
16	**3b**	PhS	78	93/7		14.6[q]		95.2		96.8
17	**3a**	$C_{16}H_{33}O$	74	90/10	20.7[r]		93.8		96.0	
18	**3b**	$C_{16}H_{33}O$	68	90/10		23.4[r]		95.3		98.0
19	**3a**	$C_{18}H_{35}O$[l]	72	90/10	22.4[p]		92.6			
20	**3b**	$C_{18}H_{35}O$[l]	70	90/10		25.4[p]		94.0		

[a] Ring-opening reactions were carried out in dichloromethane (except for entries 5 and 6, in which chloroform was used) in the presence of catalytic (~5–10 mol %) BF_3 etherate for ~18 h at room temperature (entries 5–12) or 4 °C (entries 1–4 and 13–20). Reactions with the glycidyl arenesulfonates **1** and **2** used 1.1–1.4 equiv of nucleophile. Reactions with glycidyl *tert*-butyldiphenylsilyl (TBDPS) ether **3** used 0.83 equiv of nucleophile to suppress formation of byproduct NuOTBDPS. [b] The percent yield refers to the major regioisomer obtained after workup and flash chromatography. [c] The ratio of regioisomers was determined by reverse-phase HPLC (4.6 × 250 mm C_{18} Carbosphere) of the crude reaction mixture. [d] Retention times were recorded on a Perkin-Elmer Model 410 HPLC equipped with a LC235 diode array detector and LCI 100 recorder. [e] The % ee was determined by chiral HPLC (Pirkle type IA column, 4.6 × 250 mm, J.T. Baker) of the crude (*R*)-(+)-MTPA esters[14] derived from **4**. In each case, baseline separation of the diastereomeric Mosher esters was achieved. [f] The % ee was determined by 400-MHz ^1H NMR analysis of the crude (*R*)-(+)-MTPA ester derived from **4**. To calculate % ee, the methoxy peaks of the two diastereomers were used for entries 1 and 2, whereas the AB quartets of the CH_2OTs, CH_2OBn, and $CH_2SC_6H_5$ were used for entries 5-6, 13-14, and 15-16, respectively. For entries 17 and 18, the CH_2OMTPA peaks of the bis-Mosher ester (prepared from desilylated **4**) were used to calculate % ee (Guivisdalsky, P. N., Bittman, R., unpublished results). In each case, baseline separations of the respective peaks in the two diastereomers were attained. [g] Flow 0.65 mL/min, hexanes–*i*-PrOH 85:15. [h] Flow 0.70 mL/min, hexanes–*i*-PrOH 80:20. [i] Flow 0.40 mL/min, hexanes–*i*-PrOH 90:10. [j] The ring-opened product was recrystallized three times from ether–hexanes prior to conversion to the (*R*)-(+)-MTPA ester. [k] The % ee of the starting material can be strikingly enhanced (to 99%) by multiple recrystallizations from ethanol (ref 9). Based on the literature[9] $[\alpha]^{25}_D$ of (+)-**2b**, the % ee values of the lots of (−)-**2a** and (+)-**2b** we used are ca. 92 and 89, respectively. [l] Oleyl, $C_{18}H_{35}O$ (Δ^9 cis). [m] Flow 0.50 mL/min, hexanes–*i*-PrOH 90:10. [n] Petroselinyl, $C_{18}H_{35}O$ (Δ^6 cis). [o] Flow 0.45 mL/min, hexanes–*i*-PrOH 90:10. [p] Flow 0.65 mL/min, hexanes–*i*-PrOH 100:0. [q] Flow 0.75 mL/min, hexanes–*i*-PrOH 100:0. [r] Flow 0.45 mL/min, hexanes–*i*-PrOH 100:0.

were used as nucleophiles. Other Lewis acids such as $TiCl_4$, $AlCl_3$, and $ZnCl_2$ gave complex reaction mixtures. We report here the first use of BF_3 etherate as an efficient catalyst for the opening of glycidyl derivatives **1–3** with a variety of nucleophiles.[6] The high regio- and stereoselectivity of the BF_3-mediated reaction makes this new methodology attractive for the preparation of chiral monoprotected vic-diols from glycidyl derivatives.

Nucleophilic ring-opening reactions of (*R*)- and (*S*)-**1–3** with catalytic amounts of BF_3 etherate are outlined in eq 1 and 2, and the results are summarized in Table I. The regioselectivity (attack at C_3 vs C_2) is very high (≥89:11). Both arenesulfonates gave exclusive formation of the C_3-opened product **4**, whereas the TDBPS ether **3** gave slightly lower regioselectivity with the nucleophiles we used. Although BF_3 etherate is a known desilylating agent when used in excess,[7] no desilylation of **3** occurred under the conditions employed. The two methods used for determination of % ee of **4** agreed with the exception of opening by hexadecanol, in which the % ee estimated by ^1H NMR was anamolously high. With regard to optical purity, the % ee of the ring-opened products in entries 13–20 are higher than the literature[3] % ee value of the starting epoxide **3**. In our hands, however, the specific rotation of **3** is higher[8] than the reported value, which may explain the unexpectedly high optical purities of the ring-opened products we obtained. On the other hand, the low apparent % ee values of the opening products shown in entries 7–12 reflect the relatively low optical purity of commercially available (−)-**2a** and (+)-**2b** compared with twice-recrystallized (+)-**2b**.[9] We also note that the ee of the crystalline product **4** from reaction of (−)-**1a** with 1-hexadecanol, as estimated by chiral HPLC, was improved by three recrystallizations from 94 to ~98% (entry 5).

Entries 1 and 3 represent a more efficient route to 3-*O*-benzyl- and 3-phenylthio-2-hydroxy-1-tosyloxypropane than have been reported previously.[10] Both of these are important chiral synthons;



(6) The nucleophilic species liberated during Ti-, Al-, and Zn-mediated ring opening of glycidyl derivatives may compete with the nucleophile; however, BF_3 etherate is used in catalytic amount, and the non-nucleophilic molecule ether is displaced. Furthermore, workup is much easier with catalytic BF_3 etherate than with stoichiometric $Ti(OPr\text{-}i)_4$. Workup of Ti-mediated reactions requires use of sulfuric acid or sodium hydroxide with prolonged stirring to obtain phase separation.[4g,i] In small-scale BF_3·Et_2O-mediated reactions, solvent is removed and the residue is purified by flash chromatography, whereas in large-scale reactions 10% aqueous $NaHCO_3$ is added, followed by extraction with dichloromethane.

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(9) Klunder, J. M.; Onami, T.; Sharpless, K. B. *J. Org. Chem.* **1989**, *54*, 1295. These investigators found (+)-**2b** had, after two recrystallizations, $[\alpha]^{25}_D$ +23.0° (*c* 2.14, $CHCl_3$), corresponding to 99% ee. We used (−)-**2a** and (+)-**2b** from Aldrich; $[\alpha]^{25}_D$ −21.42° and +20.82° (*c* 2.14, $CHCl_3$), respectively.

for example, **4a** (Nu = OBn) is a precursor in carbohydrate, terpene, and alkaloid chemistry,[10c] and **4a** (Nu = SPh) has been used to prepare insect pheromones.[10b]

In summary, opening of (*R*)- and (*S*)-glycidyl derivatives catalyzed by BF_3 etherate is highly efficient and proceeds with excellent regio- and stereoselectivity. The results summarized in Table I with hexadecanol, oleyl alcohol, and petroselinyl alcohol as nucleophiles show that glycidyl derivatives **1–3** are precursors to optically active ether-linked lipids, an important class of biologically active compounds.[11] Many previous syntheses of ether-linked lipids involved D-mannitol or its derivatives as starting material and were thus lengthy.[12] Optically active glycidol has been used as a lipid precursor but gives *unprotected* monoglycerides in low yield on ring opening.[4h,13] In contrast, the route reported in this communication is a practical, short synthesis of the enantiomers of alkyl lipids and other chiral 1,2-monoprotected diols.

Acknowledgment. This work was supported by National Institutes of Health Grant HL 16660.

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Synthesis and Reactivity of $(\eta^5\text{-}C_5Me_5)(PMe_3)Ir(CH_2)$: A Monomeric (Pentamethylcyclopentadienyl)iridium Methylene Complex

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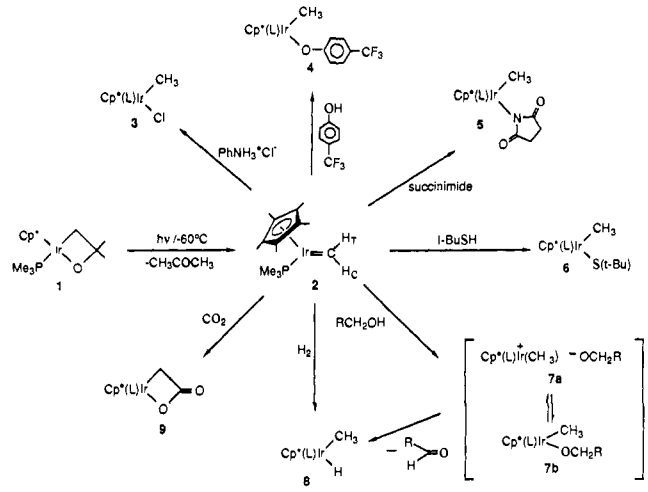
Since their discovery in 1964 by Fischer, transition-metal carbene complexes have been studied extensively.[1] Although a large number of dinuclear cyclopentadienyl and pentamethylcyclopentadienyl cobalt, rhodium, and iridium bridging alkylidene complexes are known,[2] no *monomeric* alkylidene complex in this well-studied series has ever been made.[3] We now report the generation of such a complex in an experimentally simple photoextrusion reaction. This species is remarkable for two reasons: (a) it is indefinitely stable in solution at −40 °C, showing no tendency to give a stable dimer before undergoing slow decomposition upon concentration or warming to higher temperatures; and (b) preliminary investigations of its chemistry indicate that the methylene carbon is exceptionally basic.

Scheme I



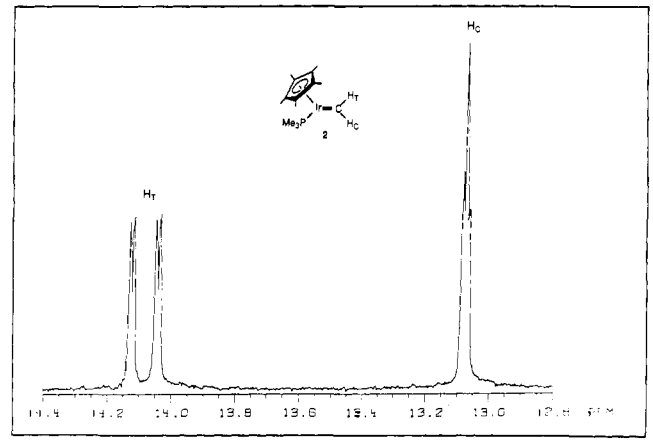


Figure 1. Low field region of the ^1H NMR spectrum of **2** at −45 °C in toluene-d_8.

Irradiation of a toluene solution of 2-oxametallacycle **1**[4] at −70 °C for 8 h leads to the disappearance of starting material and the generation of 1 equiv of acetone (δ 1.54 ppm) and a single, new organometallic species as determined by NMR spectrometry. The new species exhibits typical absorptions in both the ^1H and ^{13}C NMR spectra due to simple PMe_3 and η^5-coordinated pentamethylcyclopentadienyl ligands. The only other resonances observable confirm the identity of this species as the methylidene complex **2** (Scheme I).[5] As shown in Figure 1, the signals for two protons are evident in the ^1H NMR spectrum at very low field (14.08 and 13.08 ppm). These hydrogens are coupled weakly to one another. One exhibits a large trans coupling to phosphorus ($J(H_TP)$ = 24 Hz) and the other a smaller cis coupling ($J(H_CP)$ = 3 Hz). The $^{13}C\{^1H\}$ NMR spectrum shows a single low field resonance at 189.9 ppm; a proton-coupled spectrum establishes this as a carbon to which two hydrogens are attached (J_{CH} = 146, 134 Hz). The chemical shifts of these hydrogen and carbon resonances (as well as the reactivity of the species; see below) confirm the existence of **2** as a monomer; bridging CH_2, CHR, and CR_2 groups in dinuclear transition-metal complexes uniformly resonate at substantially higher field.[6] GC analysis of the volatile

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(5) NMR data for **2**: ^1H NMR (toluene-d_8, −45 °C) d 14.08 (dd, J = 23.8, 3.3, 1 H), 13.08 (dd, J = 3.3, 2.8, 1 H), 2.03 (d, J = 1.3 Hz, 15 H), 1.34 (d, J = 9.5 Hz, 9 H); $^{13}C\{^1H\}$ NMR (toluene-d_8, 45 °C) d 189.9 (d, J = 15.6 Hz), 92.1 (d, J = 1.4 Hz), 20.1 (d, J = 35.8), 11.0 (s).

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