tassium carbonate. After the heterogenous mixture was stirred for 15 min, 1 mL of 25% ammonium acetate was added and diluted with 50 mL of dichloromethane.

After normal workup, purification and separation of the residue by standard-phase HPLC (Waters μ-Porasil column; 30% ethyl acetate in hexanes) gave 2 mg of 10,11-cis methyl ester and 2 mg of 10,11-trans methyl ester **52** (32%). For **52**: UV (MeOH) (max) 271 nm. ^{1}H NMR (250 MHz, acetone-d_6) δ 0.90 (t, 3 H, J = 7 Hz, CH_3), 1.32 (m, 6 H), 2.32 (m, 2 H, H-13), 2.79 (m, 2 H, CH_2S), 3.41 (s, 2 H, SCH_2CO), 3.71 (s, 3 H, CO_2Me), 4.18 (m, 1 H, H-12), 4.80 (m, 1 H, H-5), 5.47 (m, 3 H, H-6, H-14, H-15), 5.83 (dd, 1 H, J = 14.6 Hz, J = 6.3 Hz, H-11), 6.10 (t, 1 H, $J_{6,7} = J_{7,8}$ = 11 Hz, H-7), 6.33 (m, 2 H, H-9,10), 6.61 (t, 1 H, $J_{8,9}$ = 12.5 Hz, H-8).

5(*S*),12(*R*)-Dihydroxy-3-thia-6(*Z*),8(*E*),10(*E*),14(*Z*)-eicosatetraenoic Acid (3-Thia-LTB$_4$, 4). To a solution of methyl ester **52** (1 mg, 2.7 μmol) in 50 μL of methanol at 0 °C was added a solution of aqueous sodium hydroxide (1 N; 60 μL, excess). The resulting mixture was allowed to warm to room temperature and stirred for 4 h. The pH was adjusted to 3 by the addition of 1 N aqueous HCl and the mixture extracted with ether (20 mL). The combined organic phases were washed with saturated aqueous sodium chloride (5 mL) and dried over anhydrous sodium sulfate, and the solvent was removed at reduced pressure to give the desired acid **4** (700 μg, 72%): UV (MeOH) (max) 271 nm.

Acknowledgment. The Natural Sciences and Engineering Research Council of Canada is gratefully acknowledged for an Industrial Postdoctoral Fellowship to D.D. We thank Dr. J. Evans and S. Charleson for biological testing, Drs. A. O. King and D. K. Rupprecht from Merck Rahway Laboratories for the SF_4 reaction in the preparation of compound **54**.

Supplementary Material Available: Experimental procedures and characterization data for compounds **8**, **11**, **14–17**, **22**, **25–27**, **32α/β**, **35α/β**, **37**, **40–42**, **45**, **49**, **55**, and **57–60** and some unnumbered intermediates (15 pages). Ordering information is given on any current masthead page.

Organotin Phosphate Condensates as a Catalyst of Selective Ring-Opening of Oxiranes by Alcohols

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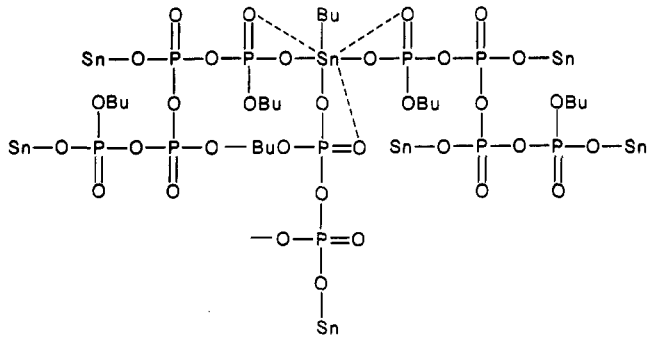
The highly regioselective alcoholysis of oxiranes is catalyzed by organotin phosphate condensates, providing a variety of β-alkoxy alcohols in good yields. The selectivity of the nucleophilic attack is dependent on the structures of epoxides. The *gem*-dialkyloxiranes are cleaved on the tertiary carbon, while β,γ-epoxy alcohols and their derivatives gave C-3 attack products. The anti stereoisomers are solely produced in the latter case. Thus the catalysis is both acidic and coordinative. Of practical importance is the recycled use of the catalyst without any appreciable decrease in the activity and the selectivities.

Organotin phosphate condensates (Sn-P Cat.) are thermolysis products of organotin oxides or chlorides in the presence of di- or trialkyl phosphates at 200–250 °C and catalyze polymerization of various epoxides, providing high polymers with high crystallinity.[1] The fact suggests that the polymerization proceeds in a stereospecific manner. We have revealed the actual active species to be associated with the unique seven-coordinate monoalkyltin tris(diphosphate) as shown below.[2] The stereospecificity of the polymerization is ascribable to the three-dimensional rigid network involving alternating tin diphosphate units on which the highly orientated coordination of epoxide monomers occurs.



Ring-opening of epoxides by alcohols is useful for preparing protected *vic*-diols if the reaction proceeds with high selectivities. To this end various efforts have been devoted with limited satisfaction.[3] Most promising and versatile among them is the alumina-catalyzed reaction disclosed by Psner et al.[4] Despite its wide applicability, this method occasionally suffers from the poor regioselectivity with unsymmetrical epoxides: the ratio of the two regioisomers varies between 6/1 and 1.5/1. In addition, no application to functionalized epoxides has been reported. More recently, Caron and Sharpless have reported the titanium tetraisopropoxide mediated reaction of β,γ-epoxy alcohols with up to 100/1 preference of nucleophilic attack at C-3 over C-2.[5] Furthermore, the exclusive C-3 attack also has been achieved for β,γ-epoxy esters with $AlPO_4$–Al_2O_3.[6]

We report here that Sn-P Cat. is quite effective for the selective cleavage of oxiranes by alcohols.[7]

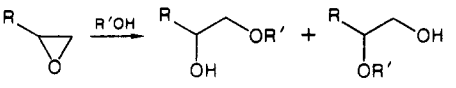
Results

In a typical run, a mixture of an epoxide (5 mmol) and an alcohol (5–15 mmol) except methanol (vide infra) in an

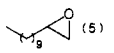
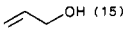
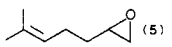
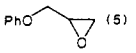
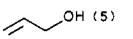
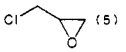
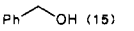
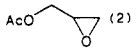
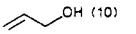
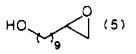
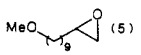
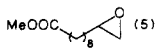
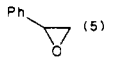
(1) Nakata, T. *Coordination Polymerization*; Price, C. C., Vandenberg, E. J., Eds.; Plenum Press: New York, 1983; p 55.
(2) Otera, J.; Yano, T.; Kunimoto, E.; Nakata, T. *Organometallics* **1984**, *3*, 426.
(3) Smith, J. *Synthesis* **1984**, 629.
(4) Posner, G. H.; Rogers, D. Z.; Kinzig, C. M.; Gurria, G. M. *Tetrahedron Lett.* **1975**, 3597. Posner, G. H.; Rogers, D. Z. *J. Am. Chem. Soc.* **1977**, *99*, 8028 and 8214. Posner, G. H.; Hulce, M.; Rose, R. K. *Synth. Commun.* **1981**, *11*, 737.
(5) Caron, M.; Sharpless, K. B. *J. Org. Chem.* **1985**, *50*, 1557.
(6) Riego, J.; Costa, A.; Saa, J. M. *Chem. Lett.* **1986**, 1565.
(7) For a preliminary report: Otera, J.; Yoshinaga, Y.; Hirakawa, K. *Tetrahedron Lett.* **1985**, *26*, 3219.

0022-3263/88/1953-0275$01.50/0

Table I. Reaction of Type I Epoxides with Alcohols[a]



1 2

entry	epoxide (mmol)	R'OH[b] (mmol)	reactn time (h)	yield[c] (%)	1/2[d]
1		MeOH	24	62	83/17
2			22	66	82/18
3		MeOH	15	86	77/23
4		MeOH	5	71	95/5
5			5	60	98/2
6		MeOH	5	83	100/0
7			48[e]	86	100/0
8		MeOH	10	65	100/0
9			10	62	100/0
10		MeOH	24	91	53/47
11		MeOH	24	90	55/45
12		MeOH	24	93	65/35
13		MeOH	5	77	2/98

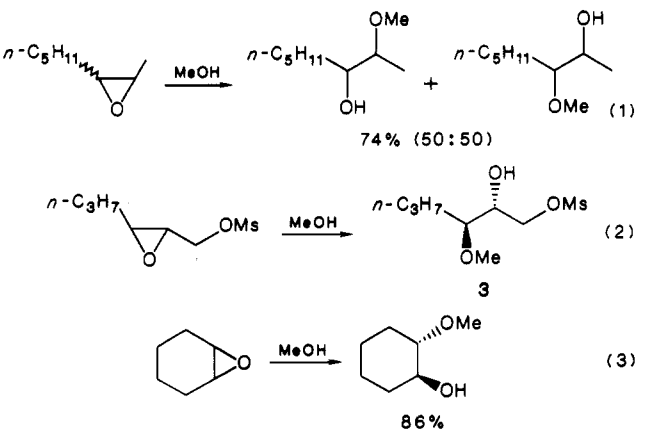
[a] In all reactions, 1 g of the catalyst was employed in refluxing hexane or methanol. [b] Excess methanol (10 mL) was used as a solvent and as a reaction component. [c] Isolated by distillation or column chromatography. [d] Determined on the basis of GLC analysis. [e] Reaction was conducted at room temperature.

appropriate solvent was heated under reflux in the presence of Sn-P Cat. (1 g). The catalyst is soluble in halocarbons, slightly in aromatic hydrocarbons, and sparingly in aliphatic hydrocarbons and methanol. Although the reaction proceeded smoothly in any of these solvents, hexane was our choice since the heterogeneous reaction enabled easy separation of the catalyst. Excess methanol was utilized when it was a reaction component. Care must be given for the reaction of highly polymerizable glycidyl acetate. An increased amount (2–3 g) of Sn-P Cat. supressed the polymer formation.

The epoxides discussed in this paper are classified as follows: type I involves 2-monoalkylated oxiranes, type II 2,3-dialkylated, type III 2,2,3-trialkylated, and type IV 2,2-dialkylated ones.

Table I exhibits the results with type I epoxides. GLC analysis readily gave the ratio of the regioisomers, which also were separated by means of column chromatography. Acetylation of the isolated isomers allowed us to unambiguously assign the regiochemistry on the basis of ^{1}H NMR spectra.[8] In all cases, the nucleophilic attack occurred on the unsubstituted methylene at C-3. A wide variety of functional groups on the alkyl substituents were tolerated under the present reaction conditions. Of special interest is the improvement of the regioselectivity with the epoxides bearing a functional group on the α-carbon of the alkyl substituents (entries 4–9), whose reaction proceeded much faster as compared with other cases. However, functional groups at remote sites rather reduced the selectivity (entries 10–12). Styrene oxide gave rise to the C-2 attack with a complete reversal in the regioselectivity (entry 13).

The eq 1–3 represent the reaction of type II epoxides. While 2-octene oxide led to no regioselectivity, remarkable

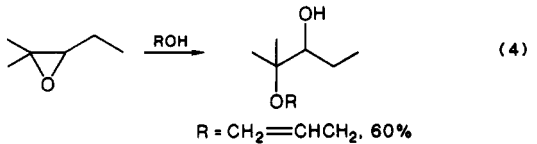


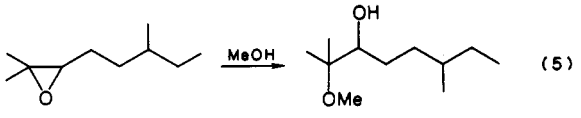
improvement was achieved with the epoxides derived from *trans*-2-hexen-1-ol. The mesylate has been proven to result in the exclusive C-3 attack, which can be related to the results for functionalized type I epoxides such as epichlorohydrin and glycidyl derivatives. GLC analyses indicated **3** thus formed to be a single isomer. The anti stereochemistry of the isolated compounds was confirmed from the vicinal coupling constant between α-protons of the methoxy and hydroxy groups (5–6 Hz). These high regio- and stereoselectivities for the derivatives of β,γ-epoxy alcohols are synthetically promising in view of the

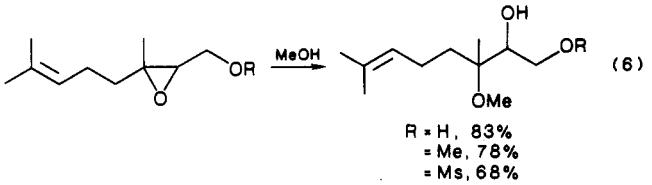
(8) Characteristic signals of α-protons of the terminal acetyl group appears at around δ 4 while those of the internal acetyl group at around δ 5. The spin-decoupling technique lends additional support.

established asymmetric epoxidation of allylic alcohols.[9] Cyclohexene oxide was employable as well but less reactive cyclooctene and cyclododecene oxides afforded the desired products only in lower yields (20–30%).

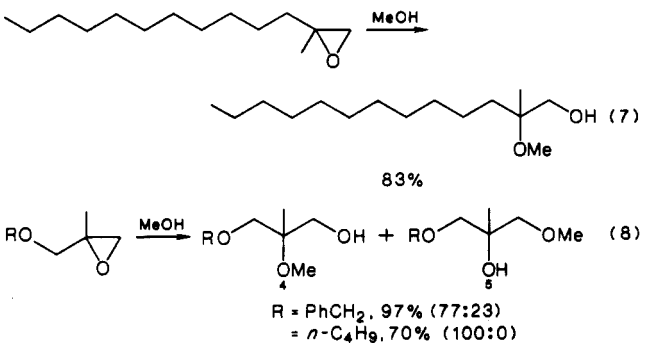
For the type III epoxides, our reaction exhibited a marked difference from the alumina-promoted cleavage in which an alkoxide group was incorporated into the less substituted carbon with low regioselectivity.[4] By contrast, an exclusive attack of the alkoxide group on the tertiary carbon occurred with Sn-P Cat. irrespective of the presence of functional groups (eq 4–6).







Analogous preference of a tertiary carbon was observed with the type IV epoxides (eq 7 and 8). The regiochemical



outcome of 2-(alkoxymethyl)-2-methylyoxiranes (eq 8) seems of special interest since the preference for the tertiary C-2 would provide the primary alcohol **4**, whereas the tertiary alcohol **5** should be formed upon the attack on C-3. Actually, the benzyl ether afforded both isomers in a 77:23 ratio while **4** was the sole product in the case of the butyl analogue. Evidently the attack on the tertiary carbon predominates.

Discussion

In general, a variety of oxiranes showed satisfactory regiochemical outcome, although type I and II compounds without a functional group exhibited somewhat lower selectivity. The most decisive factor for the regiochemistry is the preference to the tertiary epoxide carbon. In this sense, the catalyst is acidic. Consistently, styrene oxide is attacked by alcohols at the benzylic position.

The functional group on the α-carbon of the alkyl substituents also improves the regioselectivity to a great extent. The polar group may assist the oriented epoxide coordination on the organotin template through the interaction with the polar site of the catalyst. In this sense, the catalysis is coordinative. This accounts for the acceleration of the reaction rate: the polar interaction facilitates the approach of the epoxide oxygen to the catalytically effective tin atom. The anti stereochemistry resulted from type II epoxides also is consistent with this character. We suppose that the interacting sites are located regularly nearby the tin atoms responsible for the accommodation of epoxides in the three-dimensional network since the remote functional group is not effective for the regioselectivity. Presumably, the arrangement of the active tin atoms and the diphosphate units in a highly regular fashion is crucial for the stereospecific polymerization of epoxides. Sharpless et al. proposed a bidentate β,γ-epoxy alkoxytitanium intermediate in their own reaction.[5] In the present system, such alkoxide formation is not plausible since functional groups other than a hydroxy group are effective as well. Obviously the polar interaction is weaker than the covalent metal–oxygen bond so that the C-3 attack is not so preferred in our reaction. This explains why the attack on the tertiary carbon predominates over the C-3 attack for type IV epoxides.

Finally, recycled use of the catalyst is discussed. As mentioned already, the catalyst is readily filtered off from the reaction mixture. The catalyst thus recovered exhibited no depression of the activity and selectivities as compared with the fresh one. For example, the repeated use of the catalyst in the successive reactions of epichlorohydrin with methanol afforded the 1-methoxy-2-alkanol **1** in 83%, 80%, and 82% yields, respectively. Moreover, the catalyst recovered from the reaction between glycidyl acetate and allyl alcohol provided **1** in 71% yield in the reaction of phenyl glycidyl ether with methanol. Thus employment of a large amount of catalyst is not a significant problem. The increase of the catalyst amount is useful when an epoxide is polymerizable since the dispersed coordination of epoxides on the catalyst surface prevents mutual interactions. Employment of a large amount of the catalyst is also effective for shortening of the reaction time if necessary.

Experimental Section

All reactions were conducted under a nitrogen atmosphere. Solvents were purified by standard methods but reagent grade methanol was employed as supplied. Preparation of Sn-P Cat. has been described in the previous paper.[2] Column chromatography was performed on silica gel (Wako gel C-200). ^{1}H NMR spectra were recorded with Hitachi R-24B (60 MHz) and JEOLCO JNM FX-100 (100 MHz) spectrometers. GLC analyses were carried out on a Hitachi 163 gas chromatograph with a 3 mm × 3 m column packed with Silicone OV-17. In addition to the spectroscopic characterization, the reaction products were confirmed by comparison with authentic samples prepared according to the literature methods[4,5] or by alkoxylation of the corresponding diols.

Reaction of Epoxides with Alcohols (General Procedure). A reaction flask containing Sn-P Cat. (1 g) was heated at 150 °C for 1 h, charged with nitrogen, and cooled to room temperature. In this flask were added methanol (10 mL) and 1-[(methylsulfonyl)oxy]-2-hexene oxide (975 mg, 5 mmol). The reaction mixture was heated at reflux for 24 h while being stirred. After removal of the catalyst by filtration, the filtrate was concentrated. Column chromatography on silica gel (3:1 hexane–ethyl acetate) provided 2-hydroxy-1-[(methylsulfonyl)oxy]-3-methoxyhexane (**3**): 1.25 g (91%); ^{1}H NMR ($CDCl_3$) δ 0.83–1.06 (m, 3 H), 1.34–1.60 (m, 2 H), 2.77 (br s, 1 H), 3.09 (s, 3 H), 3.28 (dt, 1 H, J = 6 and 6 Hz), 3.43 (s, 3 H), 3.83–4.06 (m, 1 H), 4.29–4.40 (m, 2 H).

Acetylation of **3** (Ac_2O–pyridine) afforded the corresponding acetate: ^{1}H NMR ($CDCl_3$) δ 0.80–1.03 (m, 3 H), 1.34–1.57 (m, 2 H), 2.14 (s, 3 H), 3.06 (s, 3 H), 3.31–3.51 (m, 1 H), 3.43 (s, 3 H), 4.37–4.46 (m, 2 H), 5.09 (dt, 1 H, J = 5.4 and 4.2 Hz).

The ^{1}H NMR and mass spectroscopic data of other products are compiled in the supplementary material.

(9) Katsuki, T.; Sharpless, K. B. *J. Am. Chem. Soc.* **1980**, *102*, 5974.

Supplementary Material Available: ^1H NMR and mass spectroscopic data of remaining products (6 pages). Ordering information is given on any current masthead page.

Registry No. 1 (R = $Me(CH_2)_9$, R^1 = Me), 67217-00-9; 1 (R = $Me(CH_2)_9$, R^1 = CH_2=$CHCH_2$), 28783-83-7; 1 (R = Me_2C=$CH(CH_2)_2$, R^1 = Me), 100011-01-6; 1 (R = $PhOCH_2$, R^1 = Me), 32017-84-8; 1 (R = $PhOCH_2$, R^1 = CH_2=$CHCH_2$), 20040-20-4; 1 (R = $ClCH_2$, R^1 = Me), 4151-97-7; 1 (R = $ClCH_2$, R^1 = $PhCH_2$), 13991-52-1; 1 (R = $AcOCH_2$, R^1 = Me), 89534-59-8; 1 (R = $AcOCH_2$, R^1 = CH_2=$CHCH_2$), 100011-03-8; 1 (R = $HO(CH_2)_9$, R^1 = Me), 111903-73-2; 1 (R = $MeO(CH_2)_9$, R^1 = Me), 111903-75-4; 1 (R = $MeOCO(CH_2)_8$, R^1 = Me), 100011-04-9; 1 (R = Ph, R^1 = Me), 3587-84-6; 2 (R = $Me(CH_2)_9$, R^1 = Me), 56256-81-6; 2 (R = $Me(CH_2)_9$, R^1 = CH_2=$CHCH_2$), 111903-71-0; 2 (R = Me_2C=$CH(CH_2)_2$, R^1 = Me), 100011-02-7; 2 (R = $PhOCH_2$, R^1 = Me), 40453-79-0; 2 (R = $PhOCH_2$, R^1 = CH_2=$CHCH_2$), 111903-72-1; 2 (R = $HO(CH_2)_9$, R^1 = Me), 111903-74-3; 2 (R = $MeO(CH_2)_9$, R^1 = Me), 111903-76-5; 2 (R = $MeOCO(CH_2)_8$, R^1 = Me), 111903-77-6; 2 (R = Ph, R^1 = Me), 2979-22-8; 3, 111933-41-6; 3 (acetate), 111903-79-8; 4 (R = $PhCH_2$), 111903-91-4; 4 (R = Bu), 111903-92-5; 5 (R = $PhCH_2$), 58021-03-7; $Me(CH_2)_9\overline{CHCH_2O}$, 2404-44-6; Me_2C=$CH(CH_2)_2\overline{CHCH_2O}$, 98322-92-0; $PhOCH_2\overline{CHCH_2O}$, 122-60-1; $ClCH_2\overline{CHCH_2O}$, 106-89-8; $AcOCH_2\overline{CHCH_2O}$, 6387-89-9; $HO(CH_2)_9\overline{CHCH_2O}$, 15764-66-6; $MeO(CH_2)_9\overline{CHCH_2O}$, 111903-70-9; $MeOCO(CH_2)_8\overline{CHCH_2O}$, 22663-09-8; $Ph\overline{CHCH_2O}$, 96-09-3; MeOH, 67-56-1; CH_2=$CHCH_2OH$, 107-18-6; $PhCH_2OH$, 100-51-6; Sn, 7440-31-5; $Me(CH_2)_4\overline{CHCH(Me)O}$, 3234-26-2; $Me(CH_2)_4CH(OH)CH(OMe)Me$, 111903-80-1; $Me(CH_2)_4CH(OMe)CH(OH)Me$, 111903-81-2; $Me_2\overline{CCH(Et)O}$, 1192-22-9; $Me_2\overline{CCH(CH_2CH_2CH(Me)Et)O}$, 60814-44-0; Me_2C=$CH(CH_2)_2\overline{C(Me)CH(CH_2OMe)O}$, 111903-82-3; Me_2C=$CH(CH_2)_2\overline{C(Me)CH(SO_3Me)O}$, 111903-83-4; CH_2=$CHCH_2OC(Me_2)CH(OH)Et$, 111903-84-5; $PhCH_2OC(Me_2)CH(OH)Et$, 111933-42-7; $MeOC(Me_2)CH(OH)(CH_2)_2CH(Me)Et$, 60814-45-1; Me_2C=$CH(CH_2)_2C(Me)(OMe)CH(OH)CH_2OH$, 111903-85-6; Me_2C=$CH(CH_2)_2C(Me)(OMe)CH(OH)CH_2OMe$, 111903-86-7; Me_2C=$CH(CH_2)_2C(Me)OMe)CH(OH)CH_2SO_3Me$, 111903-87-8; $Me(CH_2)_{10}\overline{C(Me)CH_2O}$, 111903-88-9; $PhCH_2OCH_2\overline{C(Me)CH_2O}$, 97389-48-5; $BuOCH_2\overline{C(Me)CH_2O}$, 111903-89-0; $Me(CH_2)_{10}C(Me)(OMe)CH_2OH$, 111903-90-3; 1-[(methylsulfonyl)oxy]-2-hexene oxide, 111903-78-7; cyclohexene oxide, 286-20-4; 2-methylcyclohexanol, 7429-40-5; 2,3-oxidogeraniol, 50727-94-1.

A Facile Synthesis of (+)- and (-)-Shikimic Acid with Asymmetric Deuterium Labeling, Using Tricarbonyliron as a Lateral Control Group

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(-)-Methyl shikimate has been prepared from tricarbonyliron complexes of methyl dihydrobenzoate. Reaction of optically pure (+)-tricarbonyl(1-carbomethoxycyclohexa-1,3-dienyl)iron hexafluorophosphate with hydroxide ion and then *tert*-butyldimethylsilyl chloride (TBDMSCl) followed by metal removal with Me_3NO gave (+)-1-carbomethoxy-5-hydroxycyclohexa-1,3-diene as its TBDMS ether. Osmium tetraoxide oxidation and then desilylation gave optically pure (-)-methyl shikimate. Also, fully resolved (-)-tricarbonyl(1-carbomethoxycyclohexa-1,3-dienyl)iron hexafluorophosphate was hydroxylated and then treated with CrO_3 followed successively by $ZnBH_4$ and TBDMS triflate. Demetalation gave the same TBDMS-protected (+)-1-carbomethoxy-5-hydroxycyclohexa-1,3-diene as that obtained above by direct reaction. Thus, although resolution is necessary, both enantiomers of tricarbonyl(1-carbomethoxycyclohexa-1,3-dienyl)iron hexafluorophosphate are convertible into natural (-)-methyl shikimate. Deuterium was incorporated enantiospecifically to give (6*R*)- or (6*S*)-methyl 6-deuterioshikimate.

The lateral control of synthesis exercised by a complexed transition-metal atom can often mimic the control exercised by enzymes but with a wider range of reaction mechanisms and substrates.[1,2] In particular, optically active complexes provide unique synthetic opportunities for asymmetric bond formation[3] at new chiral centers of known absolute configuration, if that of the complex is known. This capability results from the normal complete stereospecificity of the bond formations, and the resulting asymmetry is equivalent to that of the complex. We have provided one example[1] in the total enantiospecific synthesis of the enzyme-inhibitor gabaculine and its derivative containing also a chiral center of known absolute configuration in which asymmetry is due to ^2H vs H.

The key intermediate in that enantiospecific synthesis was the resolved complex **2b** (R = H or ^2H) obtained[1] from benzoic acid via 1,4-dihydrobenzoic acid. The derived cation **3** (R = H or ^2H) had previously been shown to react with nucleophiles solely at the 5-exo position.[4] We have now employed these complexes in the form of *both antipodes* to synthesize the one natural enantiomer (-)-shikimic acid as its Me ester (**1a**).

Shikimic acid has been synthesized in racemic and optically active forms.[5] The synthesis of Campbell et al. attracted our attention because we envisaged enantiospecific synthesis of their key precursor **6** (R = R′ = H) by a method analogous to the processes used for gabaculine.[1] The related deuterio labeled derivatives **1b** (R = ^2H, R′ = H) and **1c** (R = H, R′ = ^2H) can be also prepared from the resolved cation salts **3a** (R = ^2H) and **3b** (R = ^2H), already described,[1] as shown for salt **3a** (R = H or ^2H) in

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