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The Unexpected and the Unpredictable in Organic Synthesis

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1. Introduction

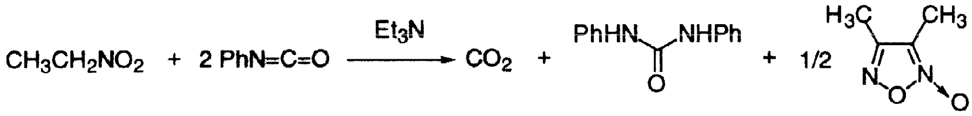
There are two kinds of approach to the research of synthetic organic chemistry: one is to study on fundamental subjects and the other on targeted subjects. The former is represented as new reaction chemistry and the latter is the orthodox total synthesis of complex molecules. Asymmetric organic reaction also belongs to the targeted subjects, which contributed a great deal to making optically active complex molecules in these 20 years.

In new reaction chemistry, *how* to get a valuable seed is the key issue. In most cases, such seed is sealed in one of the countless experiments and reveals itself only when every phenomenon is observed carefully lest the seed be missed inadvertently because of its unexpected appearance.

As we look back our research history, the first time when we faced the unexpected phenomenon was like this: a blast of gas, accompanied by a swift change in color of solution to brown with precipitates at the bottom of flask — all in one moment!

We never knew what happened. What we were trying to do was to prepare α-nitropropionanilide by addition reaction of phenyl isocyanate and the sodium salt of nitroethane after a similar procedure for the formation of α-nitroacetanilide. However, this approach was of no use. As an alternative choice, therefore, a catalytic amount of triethylamine was added to the benzene solution of nitroethane and phenyl isocyanate to see if it works. Then the above mentioned phenomenon had suddenly occurred. Although it was hard to follow what took place at first, every pathway could be explained easily once after we thoroughly learned the whole reaction. Such a turn of events was completely out of our expectation. However, that *was* the great incident which immensely affected my later career. Regardless of its appearances, therefore, to accept what you observed as it is will often provide you an unfathomable hint. Then, let that hint (seed) grow in continuous experimental work and develop it into a new idea. When this idea is led to a new concept, it will be a "leap" from 0 to 1.

Until then, I had been involved more or less in analytical study of organic reaction mechanism, but this single incident drove me to an utterly different research direction. After this experience and thus became aware of its magic, I switched my research interest from the reaction mechanism to the exploration of new synthetic reaction. What we observed in that flask that day confided great mystery of chemistry and it urged me to the exploration of new and useful dehydration reaction of nitroethane to generate acetonitrile oxide (1960).[1]



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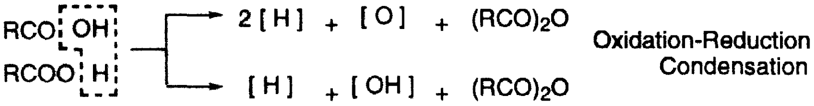
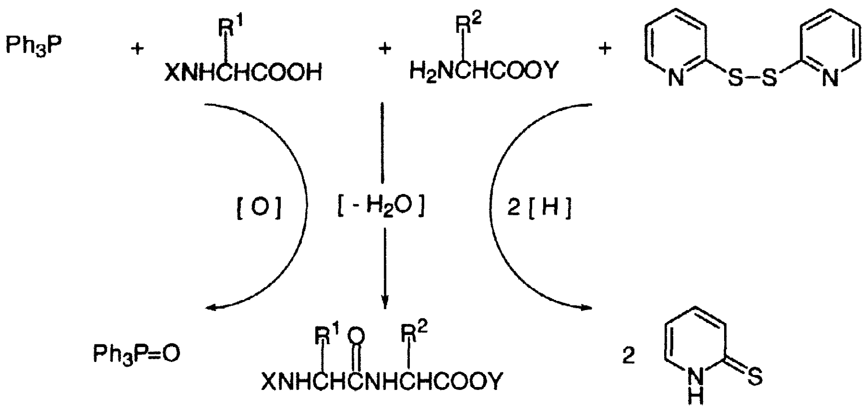
2. Deoxygenation Reactions: Oxidation-Reduction Condensation

With a cue we got from that dehydration reaction, we proceeded to deoxygenation reaction by using trivalent phosphorus compounds since we had already obtained a hint through the related experiments that the phosphorus compounds behaved as a strong deoxygenating reagent (1963).[2] The oxidation-reduction condensation takes place smoothly under neutral conditions by the combined use of characteristic properties of a reductant, P(III) compounds, and a week oxidant, such as S(II) compounds or dibenzoylethylene. Thus, a new concept concerning dehydration condensation reaction was established (1970).[3]

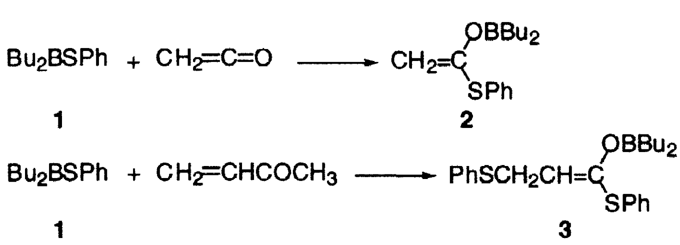




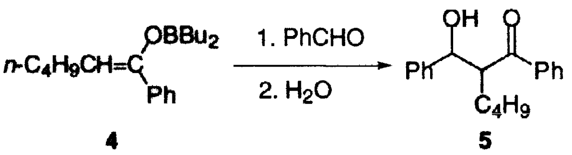
3. Crossed Aldol Reactions

3.1. *The Boron Enolate Mediated Aldol Reaction*

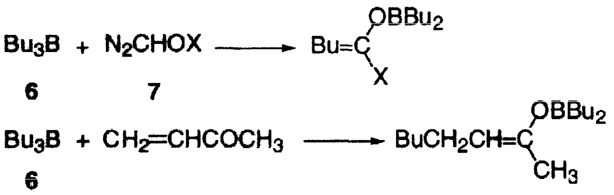
In 1971, it was found that the boron enolate derivative **2** is formed when phenyl di-*n*-butylthioboronite **1** is treated with a ketone after observation of an unexpected experimental result of our original study on the reaction of organothioboranes and carbonyl compounds. Similarly, boron enolate **3** was prepared by the reaction of methyl vinyl ketone with phenyl di-*n*-butylthioboranite.



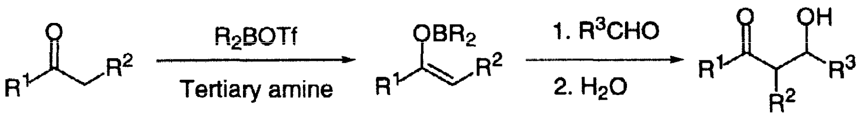
Then, it was pointed out as a first example that these boron enolates are quite useful synthetic intermediates to react with various aldehydes and acetone to afford β-hydroxycarbonyl compounds in excellent yields under essentially neutral conditions.[4] For example, treatment of boron enolate **4** with benzaldehyde at room temperature for 10 min gives the corresponding crossed aldol **5** in 98% yield. This method is a typical example of an aldol reaction carried out under neutral conditions.[5]



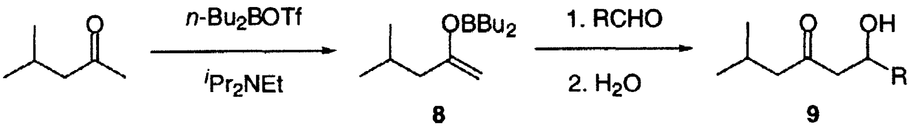
Alternatively, it had been known at that time that boron enolates could be prepared by reaction of a diazoketone **7** with a trialkylborane **6**,[6] or by the 1,4-addition of an alkylborane to an α,β-unsaturated ketone.[7].



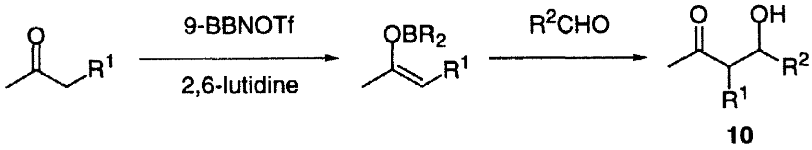
In addition, a convenient and an efficient method for generation of boron enolates from a wide variety of enolizable ketones was explored (1976). Dialkylboryl trifluoromethanesulfonate (R_2BOTf) reacts with ketones in the presence of a tertiary amine to produce boron enolates which in turn react with aldehydes to give crossed aldols in high yields.[8]



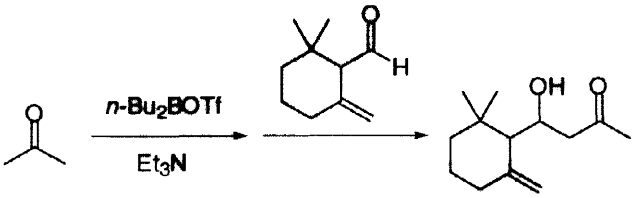
Regioselective generation of a boron enolate is readily controlled by a suitable combination of reagents. For example, the reaction of 4-methyl-2-pentanone with dibutylboryl trifluoromethanesulfonate and diisopropylethylamine produced the kinetically controlled boron enolate **8** which then reacts with an aldehyde to afford the β-hydroxyketone **9**.[8d]



In contrast, the thermodynamically stable boron enolate is generated by the reaction of the ketone and 9-borabicyclo[3.3.1]nonyl trifluoromethanesulfonate (9-BBN triflate) in the presence of 2,6-lutidine at -78 °C for 3 h. Subsequent reaction with an aldehyde gives the corresponding aldol **10** in a good yield.[8a]

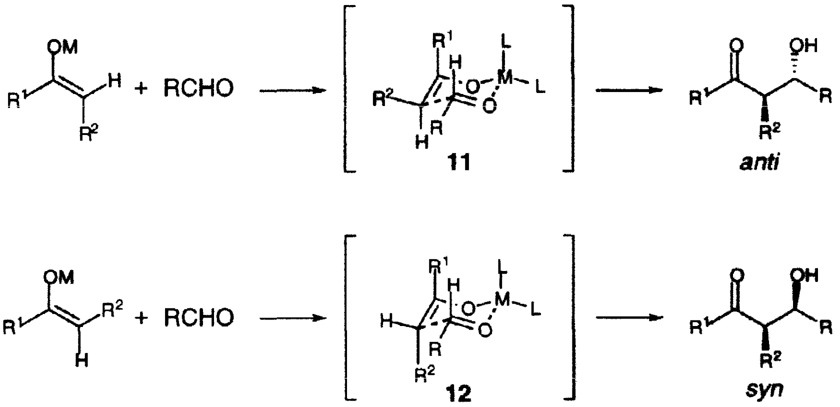


This reaction proceeds under very mild conditions without causing isomerization of the double bond of β,γ-unsaturated aldehydes. Thus γ-ionone is synthesized by a crossed aldol reaction of boron enolate with γ-citral without any contamination with isomeric α- and β-ionones.[9]

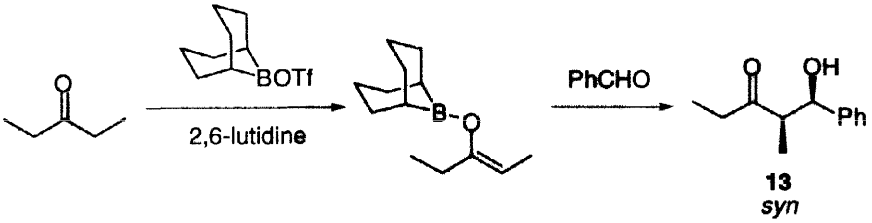


The most important stereochemical question in the directed aldol reaction concerns the formation of *anti-* and/or *syn*-isomers of aldols or ketols. Consequently, stereochemistry on the geometry of enolate species, the nature of the metal, kinetic vs. thermodynamic control, and steric effects was extensively studied.

The formation of stereoisomers is dependent on the geometry of the starting enolate under kinetically controlled conditions. The formation of stereoisomers is dependent on the geometry of the starting enolate. In general, the (*Z*)-enolate gives the *syn*-isomer and the (*E*)-enolate gives the *anti*-isomer, respectively.



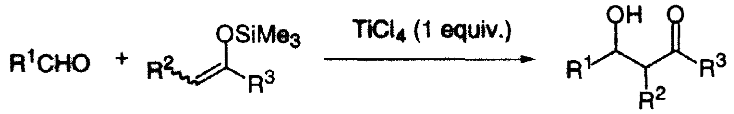
The carbonyl component approaches the enolate perpendicularly, and the reaction proceeds via a pericyclic process. Dialkylboron enolates have relatively short metal-ligand and metal-oxygen bonds, which are suited for maximizing 1,3-diaxial interactions in the transition states. Thus facilitating the formation of more stable transition states **11** and **12** where R occupies a pseudo-equatorial position, where the boron enolates undergo stereoselective aldol reaction.[6b] The boron enolate generated in situ from 3-pentanone and 9-BBN triflate by the action of 2,6-lutidine reacts with benzaldehyde in a completely stereoselective manner, giving *syn*-aldol **13** almost exclusively.

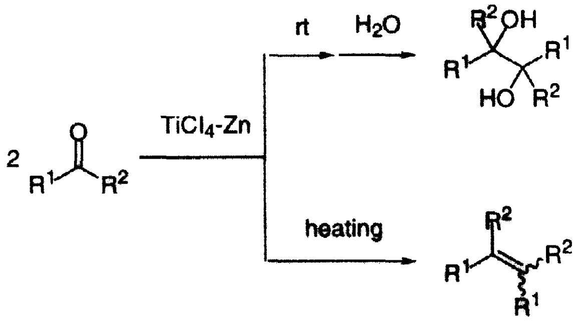


Further study on the aldol reactions of stereodefined boron enolate by D. A. Evans and S. Masamune revealed that the (*Z*)-isomers react with various aldehydes to yield predominantly the *syn*-aldols whereas the (*E*)-isomers react somewhat less stereoselectively to give *anti*-aldols as the major products. In some cases the preparation of either (*E*)-boron enolate or (*Z*)-boron enolate in a highly stereoselective manner can be achieved starting from the same ketone resulting in a stereoselective synthesis of either *syn*- or *anti*-aldol.[8b,c,f,10] Furthermore, Evans[11] and Meyers[12] showed diastereoselective aldol reactions using chiral boron enolates.

3.2. The Titanium Tetrachloride Promoted Aldol Reaction

During our research on new reactions based on organo-sulfur compounds (1967~ 1975),[13] we were using $AgClO_4$ as an activator of the reaction. However, $AgClO_4$ happened to be explosive and we were needed to find some other useful and safe metal salts. While screening various metal salts, we got the feeling of titanium chloride(IV)'s interesting character as an activator which is quite safe. The construction of carbon skeletons, such as the crossed aldol reaction and the Michael reaction by using $TiCl_4$ demonstrated that, in contrast to the common belief at that time, reactions under acidic conditions can provide highly selective synthetic schemes if one selects the proper combination of reagents and reaction conditions (1973~1976).[14] Further, several useful functional group interconversion by using $TiCl_4$ and low valent titanium compound were developed (1973).

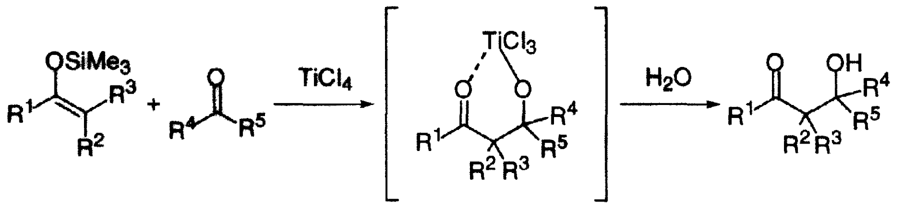




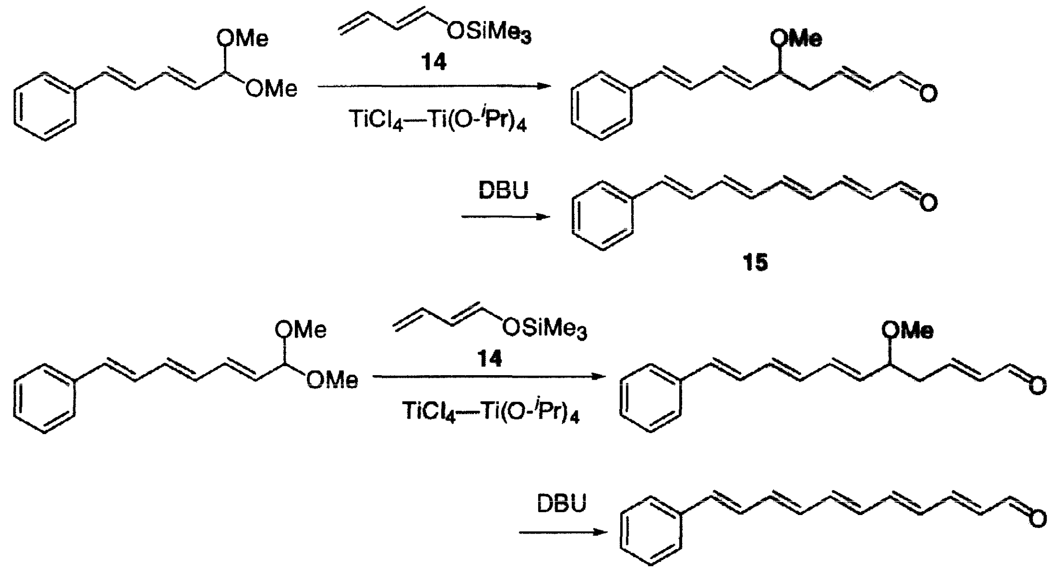
Those results stimulated wide interest in the Lewis acid nature of the titanium compounds, which are very commonly used in current organic synthesis. These studies also made silyl enol ethers popular as readily available and isolable enol derivatives, and established them as effective carbon nucleophiles indispensable for a variety of modern synthetic activities.

The aldol reaction has long been recognized as one of the most useful synthetic tools, and the reaction is usually carried out under basic conditions. However, under classical aldol reaction conditions in which basic media are employed, dimers, polymers, self-condensation products, or α,β-unsaturated carbonyl compounds are invariably formed as by-products. Useful synthetic methods have been developed to alleviate those difficulties, especially under basic conditions. The lithio derivative mediated method offers one of the solutions to those problems.[15] On the other hand, there appeared no practical procedure for a crossed aldol reaction carried out under acidic conditions. Enol ethers react with acetals or ketals by promotion of Lewis acids to give aldol-type adducts. However, these reactions are often accompanied by undesirable side reactions when stoichiometric amounts of carbonyl compounds and enol ethers, and acids are employed.[16]

The use of stoichiometric amounts of titanium tetrachloride, trimethylsilyl enol ether, and a carbonyl compound is a great advance in directed aldol reaction. Powerful activation of carbonyl groups by $TiCl_4$ enables the nucleophilic attack by trimethylsilyl enol ethers to form trimethylsilyl chloride and the titanium salt of the aldol-type product. In this case, undesirable dissociation of the adduct is inhibited by the formation of a stable titanium chelate, hydrolysis of which yields the desired β-hydroxy ketone.[17] This type of reaction is also promoted by various other Lewis acids such as $SnCl_4$, $BF_3{\cdot}OEt_2$, $AlCl_3$, and so on. Among these acids, titanium tetrachloride was found to be superior to the other Lewis acids with respect to yields. Starting enol ether compounds, silyl enol ethers, can be conveniently prepared regioselectively under either kinetically or thermodynamically controlled conditions. The reaction proceeds with retention of the regiochemical integrity of the starting silyl enol ethers to afford the corresponding aldol regiospecifically.

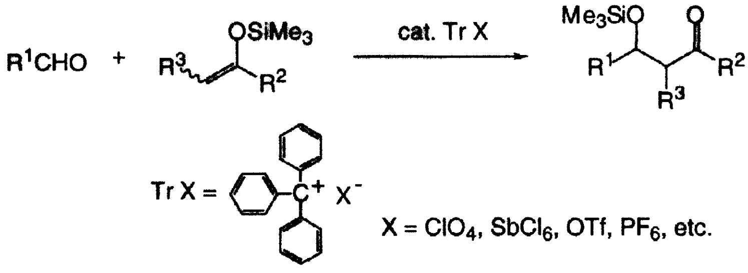


γ-Alkoxy-α,β-unsaturated aldehydes **15** are valuable synthetic intermediates because of their polyfunctionality. Reaction of acetals and the dienoxysilane **14** in the presence of $TiCl_4$ alone gives only polymers. However, the desired aldehyde **15** is obtained in good yield by treating acetal and silane **14** at -40 °C in the coexistence of $TiCl_4$ and $Ti(O\text{-}i\text{Pr})_4$.[18]



This method has been used successfully in the synthesis of natural products such as vitamin A, variotin and hypacrone.

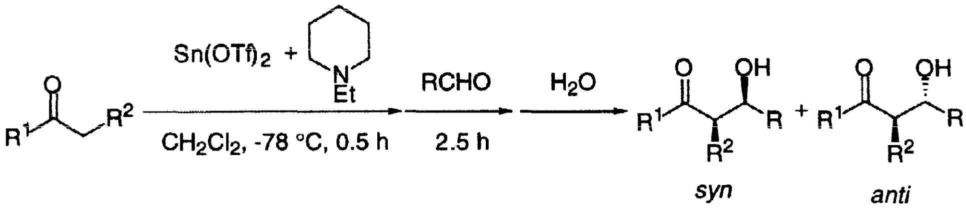
Thus, we initially reported on the $TiCl_4$-mediated aldol reaction of silyl enol ethers with aldehydes and showed subsequently that the trityl salt, represented by trityl perchlorate, can be used as an efficient catalyst in these types of reactions. While the original reaction required a stoichiometric amount of titanium tetrachloride, 5-10 mol% of trityl salt was sufficient to drive the reaction to completion (1984).[19]



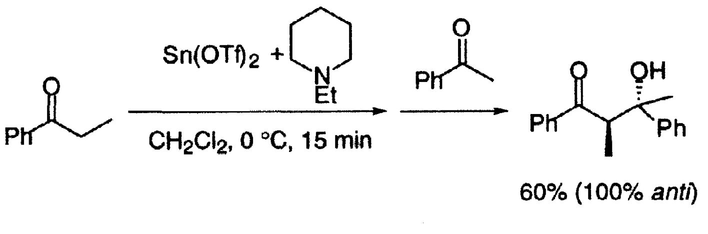
3.3. *The Tin Enolate Mediated Aldol Reaction*

Several interesting features of the tin(II) species were explored, for example, tin(II) enolates are prepared in situ by the oxidative addition of α-bromoketones to metallic tin.[20] The enolates are in turn react with aldehydes to yield aldols in highly regioselective manner with high *syn*-selectivity (1981). It was also found that the allylation reaction of carbonyl compound takes place smoothly by treating with tin(II) fluoride and allyl iodide (1980).[21]

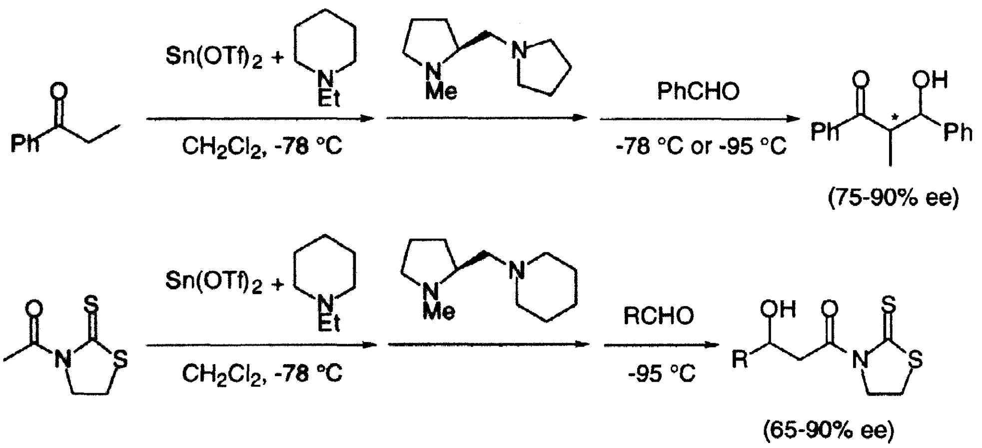
More conveniently, tin(II) enolates can be generated by reaction of ketones and $Sn(OTf)_2$ in the presence of a tertiary amine. In this reaction, the choice of the tertiary amine is crucial. For example, pyridine or DBU which can coordinate strongly to divalent tin failed to promote the reaction, while *N*-ethylpiperidine gave an excellent result. These divalent tin enolates undergo aldol reactions to give β-hydroxy ketones in good yields under extremely mild conditions with good to excellent *syn*-selectivities (1982).



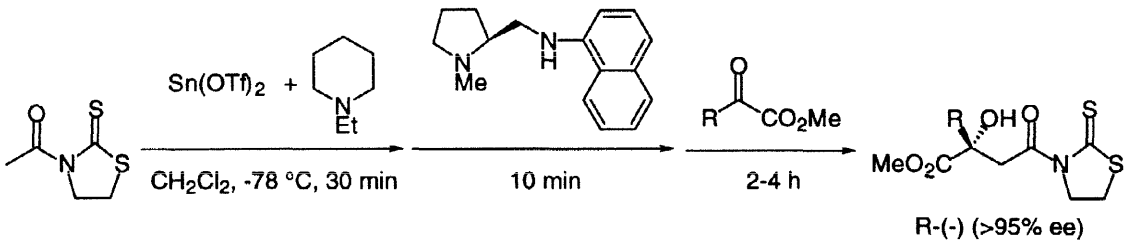
Moreover, the tin(II) enolates generated by the above procedure are highly reactive and can react even with various ketones to give ketone-ketone cross coupling products in good yields. While, boron enolates, which are very versatile metal enolates, display poor reactivity toward ketones and even more nucleophilic lithium enolates react with less-hindered ketones in moderate yield.



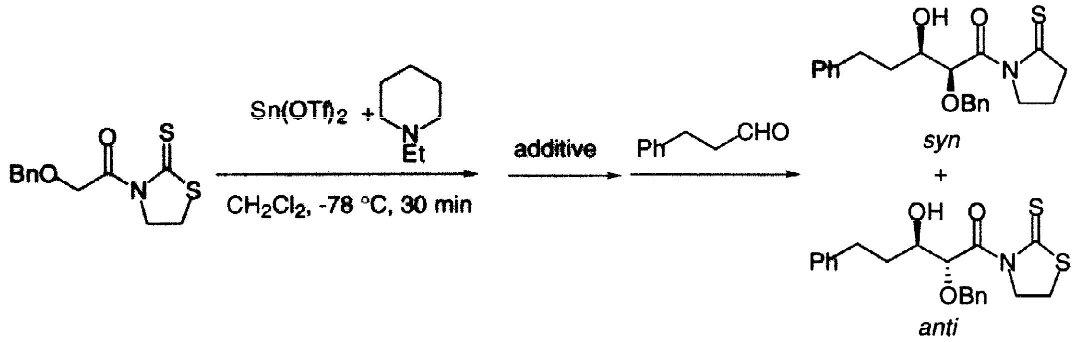
Although several asymmetric aldol reactions have been reported, chiral auxiliary groups are usually attached to the ketone equivalent molecules in these reactions. No example has existed so far in aldol-type reaction where two achiral carbonyl compounds are used for constructing a chiral molecule with the aid of a ligand (1982). Since the divalent tin having vacant d orbitals is capable of accepting a bidentate ligand, chiral diamines derived from (*S*)-proline was successfully employed in certain asymmetric reactions. Enantioselective aldol reaction via divalent tin enolates with chiral diamines was explored, for example, a highly enantioselective crossed aldol reaction between aromatic ketones or 3-acetylthiazolidine-2-thione and various aldehydes was carried out using the above chiral diamines. This is a first example for the formation of crossed aldol products in high optical purity starting from two achiral carbonyl compounds employing chiral diamines as chelating agents.[22]

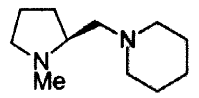


These compounds derived from 3-acetylthiazolidine-2-thione are very versatile chiral materials, capable of being transformed into various synthetic intermediates.[22b]



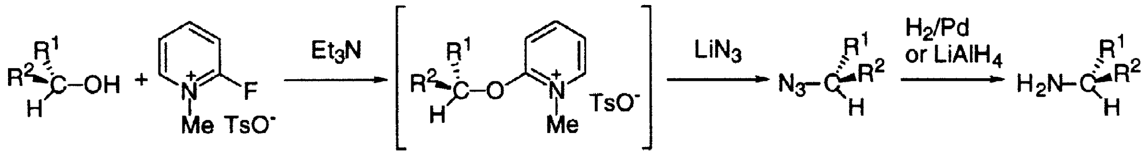
Furthermore, in the tin enolate mediated aldol-type reactions of 3-(2-benzyloxyacetyl)thiazolidine-2-thione, the stereochemical course of the reaction is dramatically altered by the addition of TMEDA as a ligand. High asymmetric induction is also achieved by the addition of the chiral diamine derived from (*S*)-proline.[23] Thus the characteristic features of tin(II) enolates enable the stereoselective synthesis of aldol products even from two different ketones. Combination of $Sn(OTf)_2$ and *N*-ethylpiperidine provides an easy approach to tin(II) enolates, whereas tin(IV) enolates have been prepared through relatively laborious multi-step procedures. Enantioselective aldol reactions effected by chiral diamines also enhances the utility of tin(II) enolates which will find useful applications in organic synthesis.



additive	yield (%)	syn/anti	ee (%)
—	62	75/25	
TMEDA	70	14/86	
	81	13/87	94

4. Synthetic Reactions with the Onium Salts of Aza-arenes

The synthetic procedures based on the onium salts of azaaromatics were successfully brought about in efficient inter- and intra-molecular dehydration reactions (1975~1977).[24] For example, carboxylic acids and alcohols were converted into various acylated and alkylated compounds on treating with several nucleophiles under mild conditions very smoothly. Furthermore, the success in converting chiral secondary alcohols into various chiral compounds prompted us to seek new possibilities for the synthesis of optically active alcohols. Thus, we embarked on a new area that previously we had left untouched as it had looked so difficult — the field of asymmetric synthesis (1977).[25]



The new concepts of "synthetic control" was our next major concern and we were absorbed in the studies of stereoselective synthesis of monosaccharides from chiral polyoxy aldehydes and in controlled aldol and Michael reactions. The use of chiral Lewis acid composed of tin(II) triflate and chiral diamine in asymmetric synthesis further urged our interests in enantioselective crossed aldol reactions.

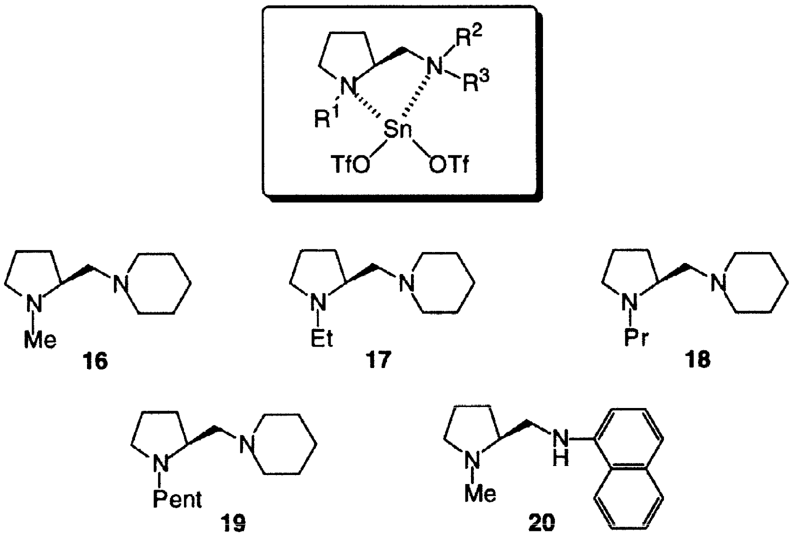
5. Aldol Reaction Using Tin(II) Complex as a Lewis Acid

For the asymmetric aldol reaction, chiral tin(II) Lewis acids prepared in situ by the coordination of chiral pyrrolidine derivatives to tin(II) triflate was chosen. Divalent tin has several vacant orbitals. Therefore, it easily forms complexes with chiral diamines, the chiral auxiliaries. After the coordination of two nitrogen atoms to tin(II), it can accept an aldehyde as a Lewis acid without losing the favorable asymmetric environment created by the chiral ligand.[26]

The asymmetric aldol reaction is one of the most powerful tools for the construction of new carbon-carbon bonds with the control of absolute configurations of new chiral centers,[27] and the utility of this reaction has been demonstrated by a number of applications to the synthesis of natural products such as macrolide and polyether antibiotics, carbohydrates, etc.[28]

Conventional asymmetric aldol reactions have been mostly performed in a diastereoselective manner by using chiral enolates and achiral carbonyl compounds as starting materials.[29-34]

The quite important key for the asymmetric aldol reaction is the choice of the chiral Lewis acid. In 1989 when this project started, some chiral Lewis acids were already reported and fruitful results were observed especially in the field of the Diels-Alder and related reactions. The chiral Lewis acids employed were consisting of rather strong Lewis acidic and hard metals such as aluminum and titanium. Since these metals strongly coordinate to oxygen, the smooth metal exchange from the metal to silicon would hardly take place. Then, chiral tin(II) Lewis acids, which were prepared in situ by the coordination of chiral pyrrolidine derivatives to tin(II) triflate was chosen (Scheme 1). Divalent tin has vacant d orbitals and easily forms a complex with an amine.[35] One feature of this chiral tin(II) Lewis acid is that the tin(II) metal is coordinately bonded with the chiral auxiliary. After the coordination of two nitrogen atoms to tin(II) metal, one vacant orbital still remains and tin(II) can coordinate to aldehyde as a Lewis acid without loosing the favorable asymmetric environment. Several efficient asymmetric aldol reactions between achiral enolates and achiral carbonyl compounds were developed based on this consideration.



Scheme 1. Chiral Tin(II) Lewis Acids

5.1. Asymmetric Synthesis of *syn*- and *anti*-1,2-diol Derivatives

Optically active 1,2-diol units are often observed in nature such as carbohydrates, macrolides or polyethers, etc. Several excellent asymmetric oxidation reactions of olefins using osmium tetraoxide with a chiral ligand has been developed to attain high enantiomeric excesses.[36,37] However, there still remains some problems such as preparation of optically active *anti*-1,2-diols. Then, development of the asymmetric aldol reaction of the silyl enol ether derived from α-benzyloxy thioester (**21**) with aldehydes was tried in order to introduce two hydroxyl groups simultaneously with stereoselective carbon-carbon bond formation.

First, the reaction of **21** with benzaldehyde was carried out in dichloromethane by using the chiral promoter consisting of tin(II) triflate, (*S*)-1-methyl-2-[(*N*-1-naphthylamino)methyl]-pyrrolidine (**20**) and tributyltin fluoride. The reaction proceeded smoothly at -78 °C to afford the corresponding aldol adduct in 69% yield with *anti*-preference. The enantiomeric excesses of *syn*- and *anti*-aldols proved to be 30% and 97%, respectively. Several chiral diamines were examined in order to improve the diastereoselectivity (Table 1). When (*S*)-1-ethyl-2-[(piperidin-1-yl)methyl]pyrrolidine (**17**) was employed, the aldol adduct was obtained in 54% yield with excellent diastereo- and enantioselectivities. Furthermore, the yield was improved without any loss of the stereoselectivity by the combination of tin(II) triflate, **17**, and dibutyltin diacetate.

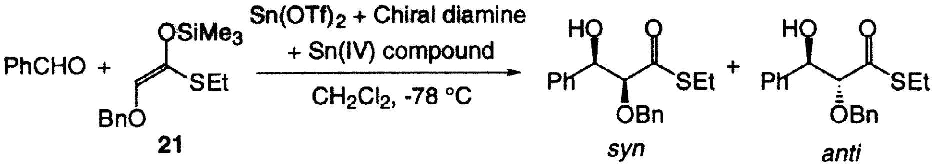


Table 1. Effect of Chiral Diamine in the reaction of **21** with benzaldehyde

Entry	Chiral diamine	Sn(IV) compound	Yield/%	syn/anti	ee/% (anti)
1	**16**	*n*-Bu_3SnF	70	1 /99	97
2	**17**	*n*-Bu_3SnF	54	1 /99	99
3	**18**	*n*-Bu_3SnF	54	1 /99	99
4	**19**	*n*-Bu_3SnF	38	1 /99	97
5	**20**	*n*-Bu_3SnF	69	26/74	97
6	**16**	*n*-$Bu_2Sn(OAc)_2$	74	1 /99	96
7	**17**	*n*-$Bu_2Sn(OAc)_2$	83	1 /99	96

The result of this asymmetric aldol reaction by employing several kinds of aldehydes such as aromatic, aliphatic, α,β-unsaturated aldehydes and a dienal are shown in Table 2.[38] In every case, *anti*-α,β-dihydroxy thioesters are obtained in high yields with excellent diastereo- and enantioselectivities. The aldol adducts thus obtained, optically active *anti*-α,β-dihydroxy ester derivatives, are generally difficult to prepare by the conventional asymmetric oxidative procedure because starting materials, *cis*-α,β-unsaturated ester equivalents, are not easily available. Moreover, a consideration of the mechanistic model of the asymmetric dioxyosmylation was reported that preparation of *anti*-1,2-diols in high enantiomeric excesses is hardly achieved.[39] According to the present aldol methodology, two hydroxyl groups can be introduced in 1,2-*trans*-position stereoselectively during new carbon-carbon bond formation.

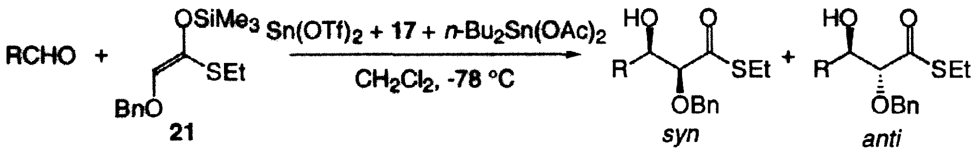


Table 2. Asymmetric Aldol Reaction of **21**

Entry	R	Yield/%	syn/anti	ee/%(anti)
1	Ph	83	1 / 99	96
2	CH_3CH_2	72	2 / 98	97
3	c-C_6H_{11}	59	9 / 91	96
4	(*E*)-PhCH=CH	88	2 / 98	98
5	(*E*)-CH_3CH=CH	85	2 / 98	97
6	(*E*,*E*)-CH_3CH=CHCH=CH	83	2 / 98	95

Although the excellent aldol reaction of **21** with aldehydes was developed, the high *anti*-selectivities attained in this reaction were unexpected and interesting results, because the aldol reaction of the silyl enol ether derived from *S*-ethyl propanethioate (**22Z**) with aldehydes using the above chiral promoter proceeded with *syn*-preference in excellent diastereo- and enantioselectivities (Table 3).

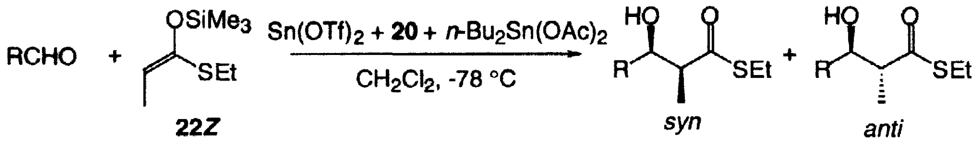
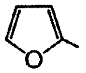
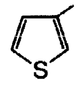


Table 3. Asymmetric Aldol Reaction of **22Z**

Entry	R	Yield/%	syn/anti	ee/%
1	Ph	85	100/ 0	>98
2	*p*-Cl Ph	96	100/ 0	>98
3	*p*-CH_3 Ph	92	100/ 0	>98
4	*p*-OCH_3 Ph	95	100/ 0	>98
5	$CH_3(CH_2)_6$	90	100/ 0	>98
6	*c*-C_6H_{11}	90	100/ 0	>98
7	*i*-Pr	70	100/ 0	>98
8	*t*-Bu	86	100/ 0	>98
9	(*E*)-CH_3CH=CH	92	100/ 0	>98
10	(*E*)-PhCH=CH	91	100/ 0	>98
11	(*E*)-*n*-PrCH=CH	91	100/ 0	>98
12		93	100/ 0	>98
13		92	100/ 0	>98

The consideration on the transition states of these aldol reactions led us to assume that i) coordination of the oxygen atom of the α-benzyloxy group of the silyl enol ether **21** to tin(II) atom of tin(II) triflate was essential in the *anti*-selective transition state, leading to the different course in the diastereofacial selectivity from that of the *syn*-selective reaction of **22Z**, ii) thus *syn*-α,β-dihydroxy thioesters would be formed when this coordination was restrained.

According to this hypothesis, *t*-butyldimethylsilyl group was chosen as a sterically hindered functional group, which would forbid the coordination of the oxygen atom to tin(II) atom, and such silyl enol ether **23** was prepared. As expected, the *syn*-aldol adduct was obtained under the same reaction conditions; namely in the presence of tin(II) triflate, chiral diamine **20** and dibutyltin diacetate, the reaction of the silyl enol ether **23** with benzaldehyde proceeded smoothly to give the corresponding aldol adduct in high yield with *syn*-preference, and enantiomeric excess of the *syn*-isomer was also very high. After examination of several reaction conditions, it was found that the corresponding aldol adduct was obtained in 79% chemical yield with *syn*/*anti* ratio = 85/15, and the enantiomeric excess of the *syn*-isomer was 92% ee when (*S*)-1-*n*-propyl-2-[(piperidin-1-yl)methyl]pyrrolidine (**18**) was employed (Table 4).

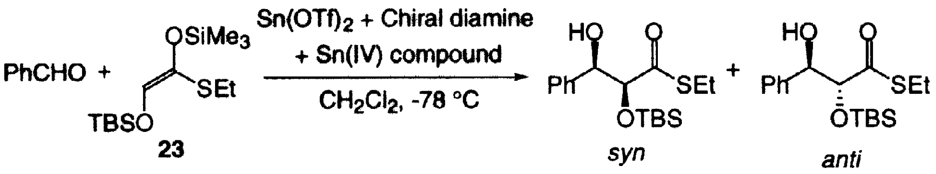
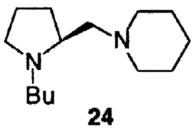


Table 4. Effect of Chiral Diamine in the reaction of **23** with benzaldehyde

Entry	Chiral diamine	Sn(IV) compound	Yield/%	syn/anti	ee/% (syn)
1	**16**	n-Bu_3SnF	81	86/14	49
2	**16**	n-$Bu_2Sn(OAc)_2$	83	91/ 9	49
3	**17**	n-$Bu_2Sn(OAc)_2$	83	86/14	90
4	**18**	n-$Bu_2Sn(OAc)_2$	86	88/12	90
5	**24**	n-$Bu_2Sn(OAc)_2$	75	87/13	89
6	**19**	n-$Bu_2Sn(OAc)_2$	63	83/17	89
7	**20**	n-$Bu_2Sn(OAc)_2$	73	73/27	94



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Several examples of this *syn*-selective aldol reaction are shown in Table 5. In every case, the reactions proceeded smoothly to afford the aldol adducts in good yields with very high *syn*-selectivities, and the enantiomeric excesses of these *syn*-isomers were proven to be more than 90% in most cases.[40]

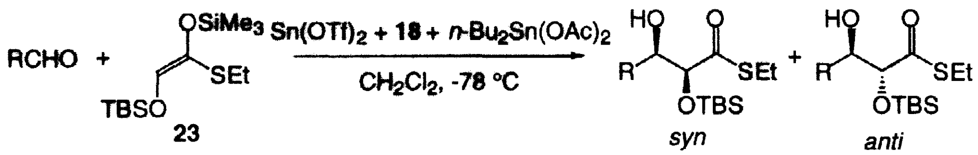
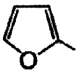
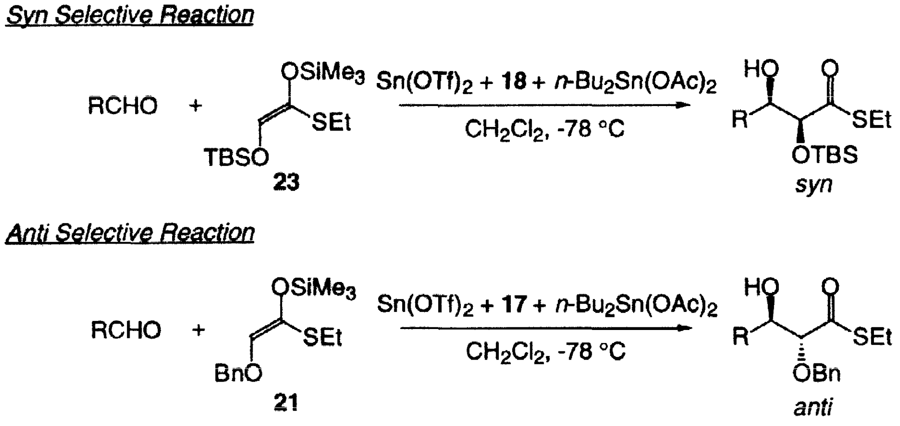


Table 5. Asymmetric Aldol Reaction of **23**

Entry	R	Yield/%	syn/anti	ee/%(syn)
1	Ph	86	88/12	90
2	CH_3CH_2	46	92/ 8	82
3		93	94/ 6	93
4	(*E*)-PhCH=CH	76	90/10	92
5	(*E*)-$CH_3CH=CH$	75	97/ 3	94
6	(*E*,*E*)-$CH_3CH=CHCH=CH$	83	93/ 7	94

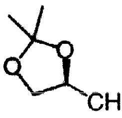
Now it becomes possible to control the enantiofacial selectivity of the silyl enol ethers derived from α-alkoxy thioesters **21** and **23** by just choosing the appropriate protective groups of the alkoxy parts of the silyl enol ethers, and both diastereomers of optically active α,β-dihydroxy thioesters can be synthesized.



Scheme 2. Synthesis of Optically Active *syn*- and *anti*-1,2-Diol Derivatives

5.2. Synthesis of Monosaccharides

In the last decade, chemical synthesis of monosaccharides made a great advance based on the stereoselective addition reactions of 2,3-*O*-isopropylidene-D or L-glyceraldehyde (**25**)^{41} or 4-*O*-benzyl-2,3-*O*-isopropylidene-L-threose (**26**)^{42} with enolate components or allyl nucleophiles, and many examples of the effective synthesis of sugars including natural and unnatural forms were demonstrated. In these syntheses, one of the starting materials, glyceraldehyde or threose derivative, is prepared from a natural chiral pool, mannitol and tartaric acid, respectively. Also, Masamune and Sharpless reported the synthesis of L-hexoses starting from an achiral allylic alcohol **27** using the Sharpless epoxidation as a key step.^{43}



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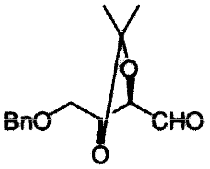

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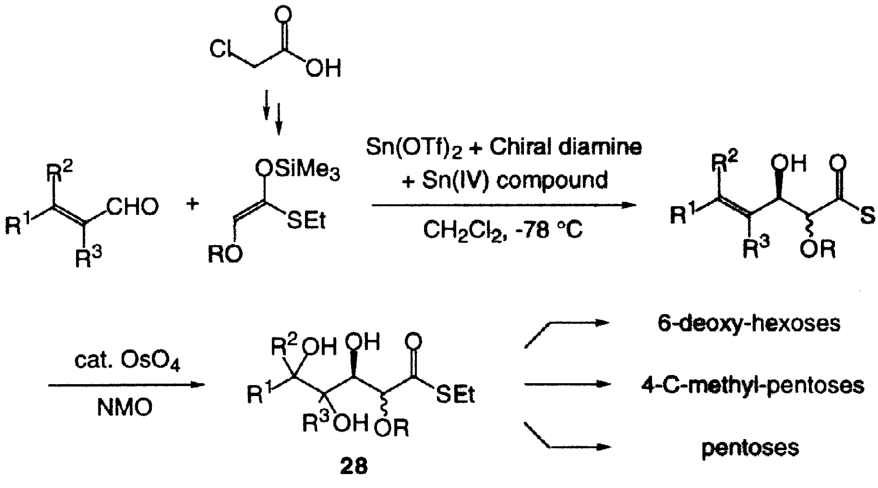
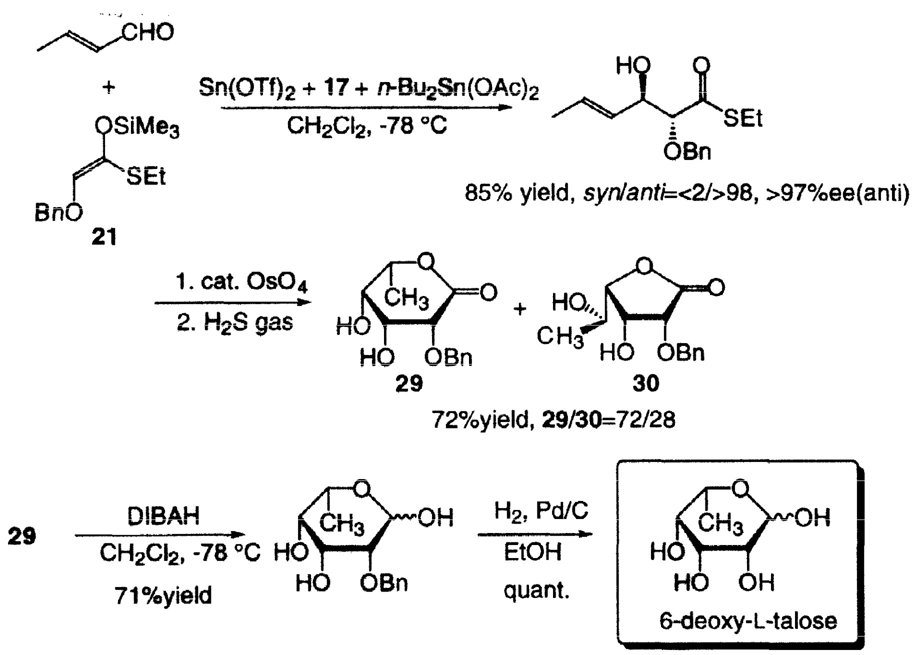
The synthetic plan according to the present asymmetric reaction is shown in Scheme 3. The starting materials are both achiral α,β-unsaturated aldehydes and the silyl enol ethers of α-alkoxy thioesters, which are readily prepared from chloroacetic acid. The chiral induction is accomplished by the asymmetric aldol reaction between the α,β-unsaturated aldehydes and the silyl enol ethers by using the chiral promoter consisting of tin(II) triflate, a chiral diamine, and a tin(IV) compound. The successive oxidation of olefinic part of the aldol adduct forms tetrahydroxy thioester derivatives **28**, which are useful precursors for the synthesis of various monosaccharides including rare sugars.

One of these examples, synthesis of 6-deoxy-L-talose, is shown in Scheme 4.[44] The asymmetric aldol reaction between crotonaldehyde and the silyl enol ether of α-benzyloxy thioester **21** was carried out in the presence of tin(II) triflate, chiral diamine **17**, and dibutyltin diacetate, and the corresponding aldol adduct was obtained in 85% yield with >97% enantiomeric excess. The successive oxidation of this chiral synthon in acetone-water (8/1) at room temperature in the presence of a catalytic amount of osmium tetraoxide and a stoichiometric amount of *N*-methylmorpholineoxide resulted in the formation of the corresponding lactones **29**, **30** in 72% yield. The major lactone **29** was isolated and reduced with diisobutylaluminum hydride (DIBAL) in dichloromethane at -78 °C. Deprotection of the benzyl group under Pd/C in ethanol gave desired 6-deoxy-L-talose in quantitative yield.

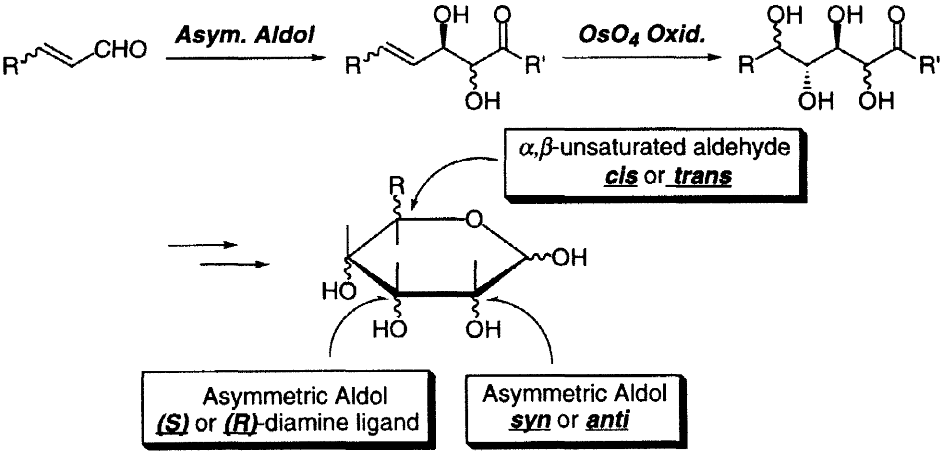


Scheme 3. Asymmetric Aldol Synthetic Route to Monosaccharides



Scheme 4. Synthesis of 6-Deoxy-L-talose

According to this method, several monosaccharides including branched, deoxy, and amino sugars were synthesized.[45] Since the key asymmetric reactions, asymmetric aldol reaction and successive dihydroxylation, have some flexibilities in controlling newly created chiral centers, the present methods are expected to provide useful routes to the synthesis of various monosaccharides from achiral unsaturated aldehydes and chloroacetic acid without using any starting materials containing natural chiral pools (Scheme 5).

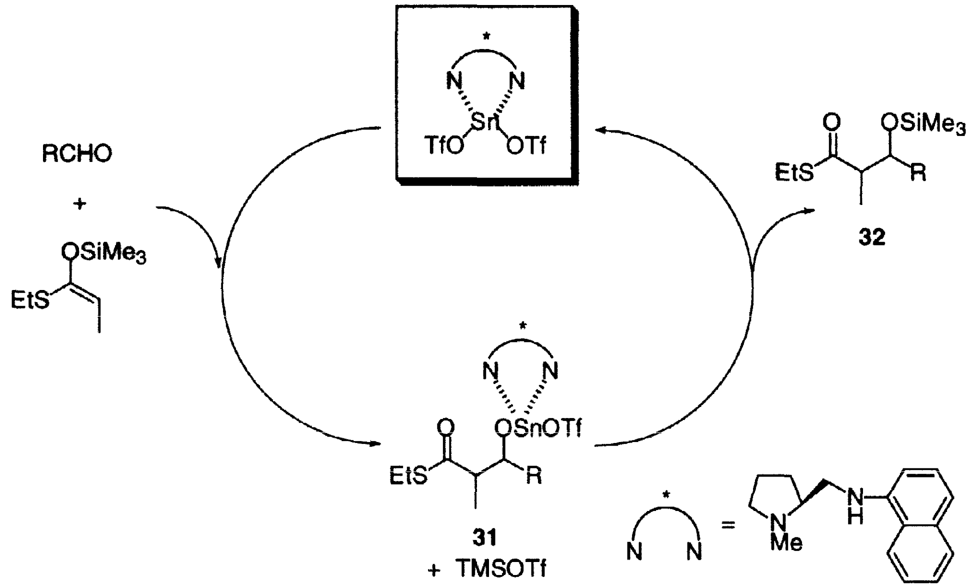


Scheme 5. General Synthetic Route to Monosaccharides.

5.3. *The Catalytic Asymmetric Aldol Reaction*

As shown in the previous sections, highly diastereo- and enantioselective aldol reactions of silyl enol ethers with aldehydes by using the novel promoter system, combined use of stoichiometric amounts of tin(II) triflate, a chiral diamine, and a tin(IV) compound (tributyltin fluoride or dibutyltin diacetate), were developed. According to these reactions, optically active aldol adducts were easily prepared from both achiral aldehydes and silyl enol ethers, while stoichiometric use of chiral source still remained as a problem in terms of practical use. In the course of the investigations to characterize the above promoter system as well as to clarify the mechanism of these reactions toward a truly catalytic aldol process, the following catalytic cycle was postulated (Scheme 6).[46]

Tin(II) triflate coordinated with a chiral diamine (a chiral tin(II) Lewis acid) interacts with an aldehyde, and tin(II) alkoxide **31** and trimethylsilyl triflate (TMSOTf) are initially produced by the attack of silyl enol ether **22Z** onto the activated aldehyde. When the metal exchange between tin(II) and silicon of the above product **31** takes place smoothly, the corresponding aldol adduct can be obtained as its trimethylsilyl ether **32** along with the regeneration of the catalyst. If the above mentioned metal exchange step is slow, undesirable TMSOTf-promoted reaction of affording the achiral aldol adduct proceeds at the same time and results in lowering the selectivity.[47]



Scheme 6. The Catalytic cycle of the Asymmetric Aldol Reaction.

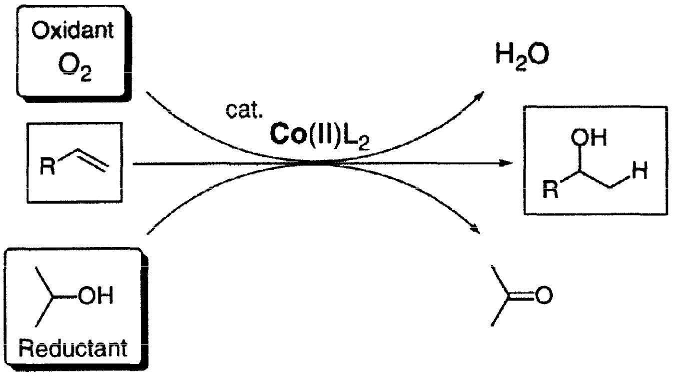
It is necessary to complete the above catalytic cycle, especially the metal exchange reaction of the initially formed aldol adduct **31** with trimethylsilyl triflate (metal exchange between tin(II) and silicon). In order to accelerate this metal exchange step, various polar solvents with low melting points (under -78 °C) were carefully examined by taking the reaction of **22Z** with benzaldehyde as a model, and finally **propionitrile** (C_2H_5CN) was found to be an excellent solvent (1990).[48] The examination of the addition time (addition of the reactants to the solution of the catalyst) revealed that the rate of the metal exchange in propionitrile is faster than that in dichloromethane. Several aldehydes including aromatic, aliphatic and α,β-unsaturated aldehydes were applicable to this reaction, and the desired products were obtained in good yields with high selectivities (>90% ee, Table 6). In particular, the lower yields or selectivities observed in the reaction of *p*-tolualdehyde, (*E*)-crotonaldehyde, (*E*)-2-hexenal, and cyclohexanecarboxaldehyde, were remarkably improved by using propionitrile as a solvent. High selectivities were also attained even when 10 mol% of the catalyst was employed.[49]

Table 6. Synthesis of *syn*-α-Methyl-β-hydroxythioesters (solvent: C_2H_5CN)

Entry	R	Addition Time/h	Yield/%	syn/anti	ee/% (syn)
1	Ph	3	77	92/ 8	90
2	*p*-Cl Ph	4.5	83	87/13	90
3	*p*-CH_3 Ph	3	75	89/11	91
4	$CH_3(CH_2)_6$	4.5	80	100/ 0	>98
5	*c*-C_6H_{11}	3	71	100/ 0	>98
6	(*E*)-$CH_3CH{=}CH$	3	76	96/ 4	93
7	(*E*)-$CH_3(CH_2)_2CH{=}CH$	3	73	97/ 3	93

6. Catalytic Oxygenations of Olefins with Molecular Oxygen

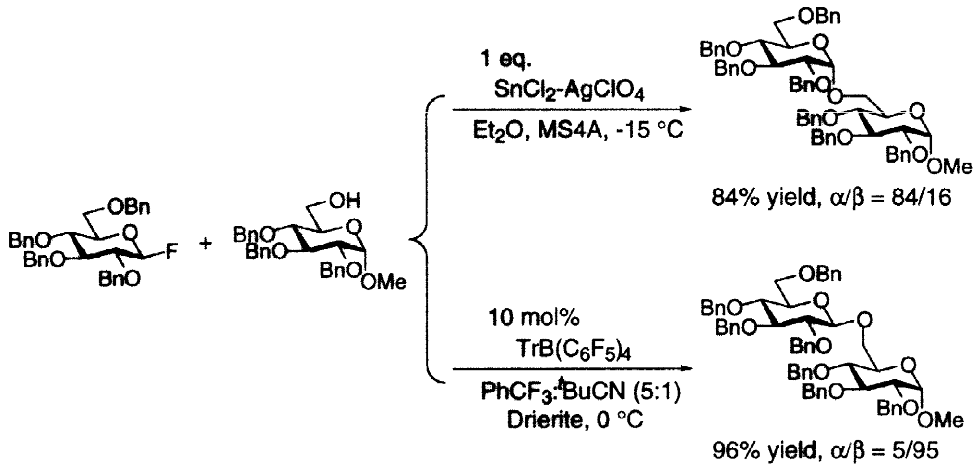
The study of metal complex catalysts that allow one to control reactivity and regioselectivity by the judicious choice of their ligands would be of particular interest. Therefore, a study on the oxygenation of olefins with molecular oxygen using a catalytic amount of metal complex containing 1,3-diketone ligand was initiated (1989). Then, cobalt(II)-catalyzed oxidation-reduction hydration,[50] catalytic peroxygenation[51] and nickel(II)-catalyzed epoxidation were developed.[52]



Further, a practical enantioselective borohydride reduction of aromatic ketones were successfully carried out by using sodium borohydride in the presence of chiral Co(II) complex catalysts (1995).[53]

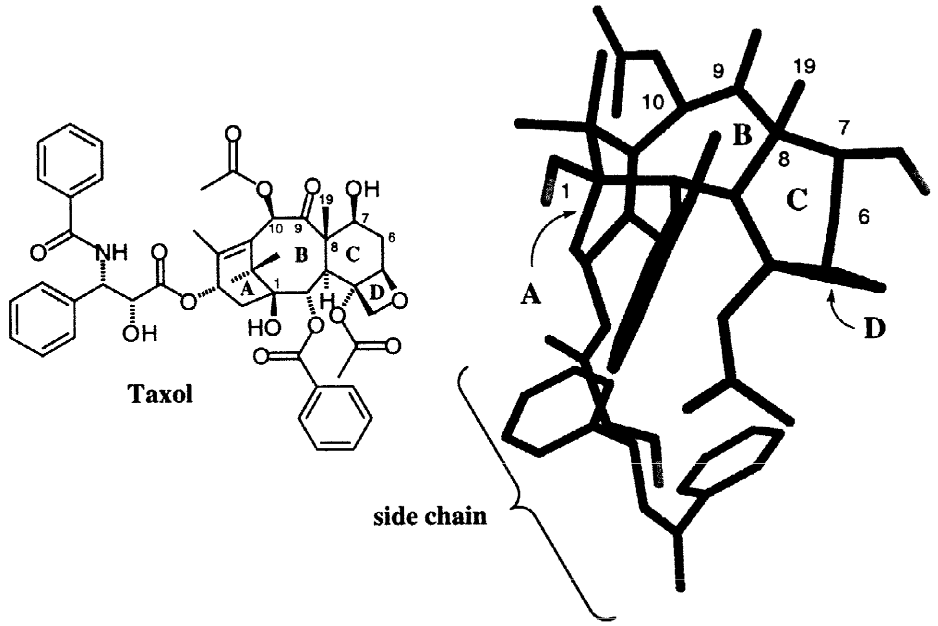
7. Stereoselective Glycosylation Reactions

The use of glycosyl fluoride proved to be quite effective in stereoselective glycosylation and has widely been used in the synthesis of many complex oligosaccharide chains after our publication in 1981.[54] Recently, the above reaction was found to be successfully performed by using a catalytic amount of trityl salt. In addition, several useful methods for the *catalytic and stereoselective* preparation of various saccharides were also developed (1992~1999).[55]

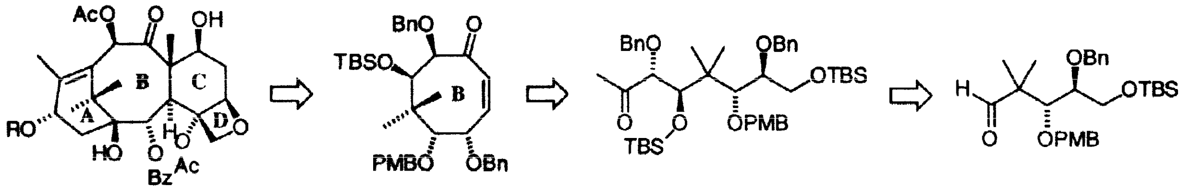


8. Asymmetric Total Synthesis of Taxol

In 1992, we turned our eyes on a targeted subject for the first time in over 40 years of our research on the exploration of new synthetic methodologies. We took up an asymmetric total synthesis of Taxol as our target motivated by a telephone call from my son, an oncologist, requesting its total synthesis.



As always, a working hypothesis is put forward by the help of wisdom from experience. We therefore planned the following strategy after we got to know the basic skeleton of Taxol: that is, from optically active polyoxy unit of the basic skeleton which should be prepared by using our highly controlled enantioselective aldol reaction, to synthesize 8-membered B ring system first. After that to fuse the A and C ring systems onto this framework.[56]

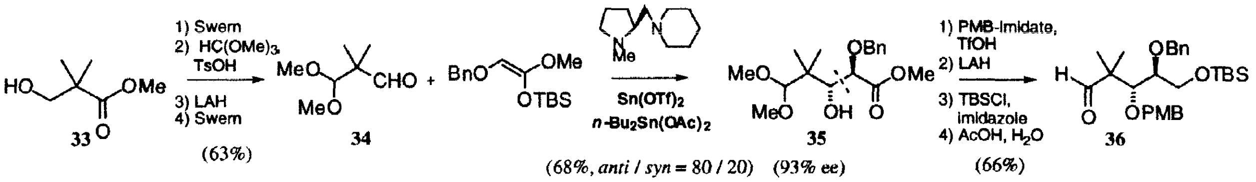


Taxol

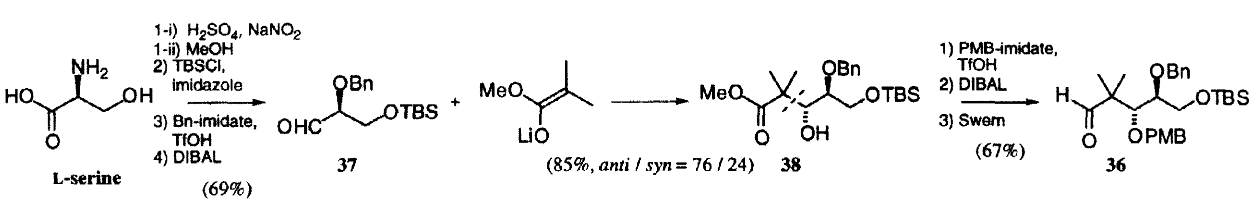
Scheme 7. Retrosynthesis of Taxol

There happens many "unpredictable phenomena" we never expect of while doing research on targeted subjects even if we planned careful strategies toward a definite goal. Repeated discussions and experiments along with their feedback to experimental observations are absolutely necessary before the goal: that is, from 1 to 5 and 5 to 10.

Optically active aldehyde **36** was prepared from commercially available methyl 3-hydroxy-2,2-dimethylpropionate (**33**) by the asymmetric aldol reaction (Scheme 8) or by the following alternative route starting from L-serine (Scheme 9).

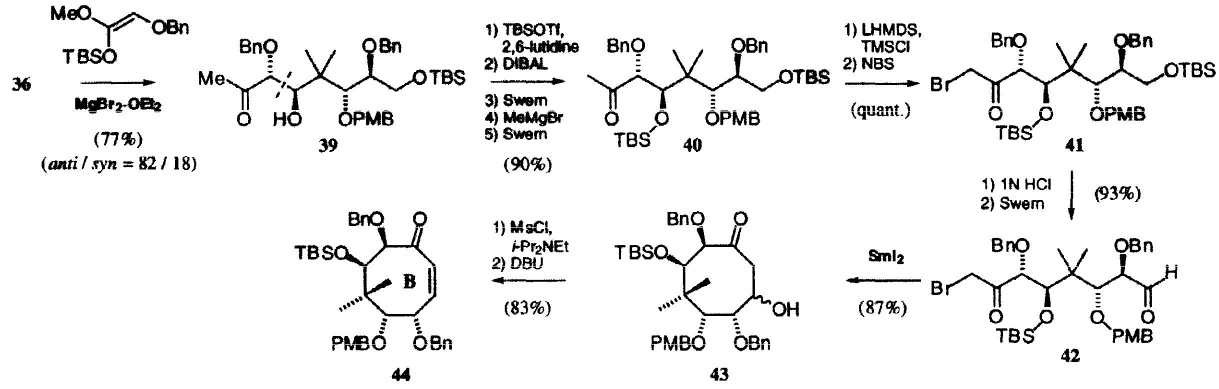


Scheme 8. Synthesis of optically acvtive trialkoxy aldehyde **36** (Route a)



Scheme 9. Synthesis of optically acvtive trialkoxy aldehyde **36** (Route b)

The aldol reaction between **36** and the appropriate ketene silyl acetal took place rapidly in the presence of $MgBr_2 \cdot OEt_2$ to yield the desired ester **39** in good stereoselectivity which in turn was led to methyl ketone **40** *via* methylation and Swern oxidation. α-Bromoketoaldehyde **42**, a precursor of 8-membered ring compound, was prepared by successive bromination and methylation of the α-position of the ketone **40**, followed by deprotection of the *t*-butyldimethylsilyl group and Swern oxidation. Synthesis of 8-membered ring compound **43** from the optically active polyoxy-unit **42** containing all the functionalities necessary for the construction of Taxol was performed by the intramolecular aldol reaction using SmI_2. Successive mesylation of this mixture of isomeric alcohols **43** and treatment with DBU gave the desired 8-membered enone **44** in good yield.[57]

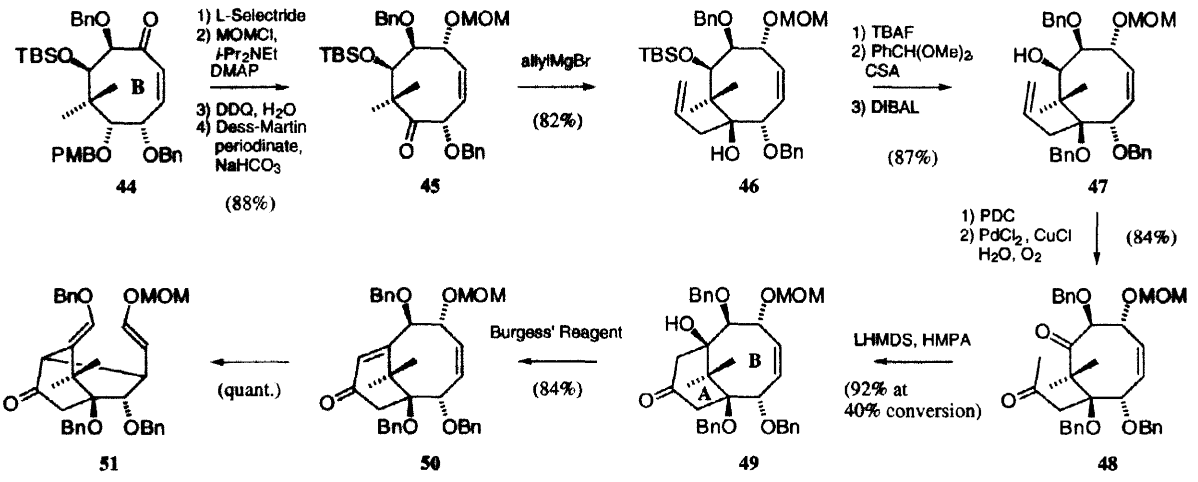


Scheme 10. Synthesis of 8-membered ring enone **44**

It had been generally considered difficult to synthesize 8-membered ring from a linear precursor. Fortunate enough, however, we were able to obtain 8-membered ring compound **43** in the case of this polyoxy compound with the yield as high as 90% because of its conformational control. Now, there are two alternative routes for constructing ABC ring system starting from B ring: that is, a route of B to AB to ABC, or a route of B to BC to ABC. Our synthetic approach was of the former route which we started working in 1993.[58,59]

A highly stereoselective reduction of **44** with L-Selectride afforded the corresponding α-alcohol, which was converted to MOM ether. β,γ-Unsaturated ketone **45** was obtained by DDQ oxidation followed by oxidation using Dess-Martin periodinane. Allylation of **45** with allylmagnesium bromide afforded homoallyl alcohol **46** in high yield with good diastereoselectivity. Deprotection of silyl ether **46** gave the *cis*-diol in quantitative yield, further treatment of thus formed *cis*-diol with benzaldehyde dimethylacetal in the presence of a catalytic amount of camphorsulfonic acid afforded a single benzylidene derivative in quantitative yield. Reductive cleavage of the benzylidene derivative proceeded with almost perfect chemoselectivity to give secondary alcohol **47** in 87% yield. γ,δ-Unsaturated ketone was obtained in high yield by oxidation of the alcohol with PDC, and diketone **48**, a precursor of the AB ring system of 12-demethyltaxoid, was prepared by successive Wacker oxidation. Next, intramolecular aldol condensation of the diketone **48** was tried in order to produce the desired α,β-unsaturated ketone **50** directly from **48**. However, in the case of substrate **48**, the corresponding aldol **49** was unexpectedly isolated on treatment with LHMDS and HMPA combined system. The aldol **49** was stable at room temperature and dehydration of **49** to form the desired AB ring system **50** did not proceed at all under the above reaction conditions. On the other hand, facile dehydration of **49** was achieved by using Burgess' reagent to afford **50** in high yield. It is noteworthy that optically active **50** was synthesized in high yield from 8-membered ring enone **44** *via* successive highly stereoselective allylation and intramolecular aldol reactions.

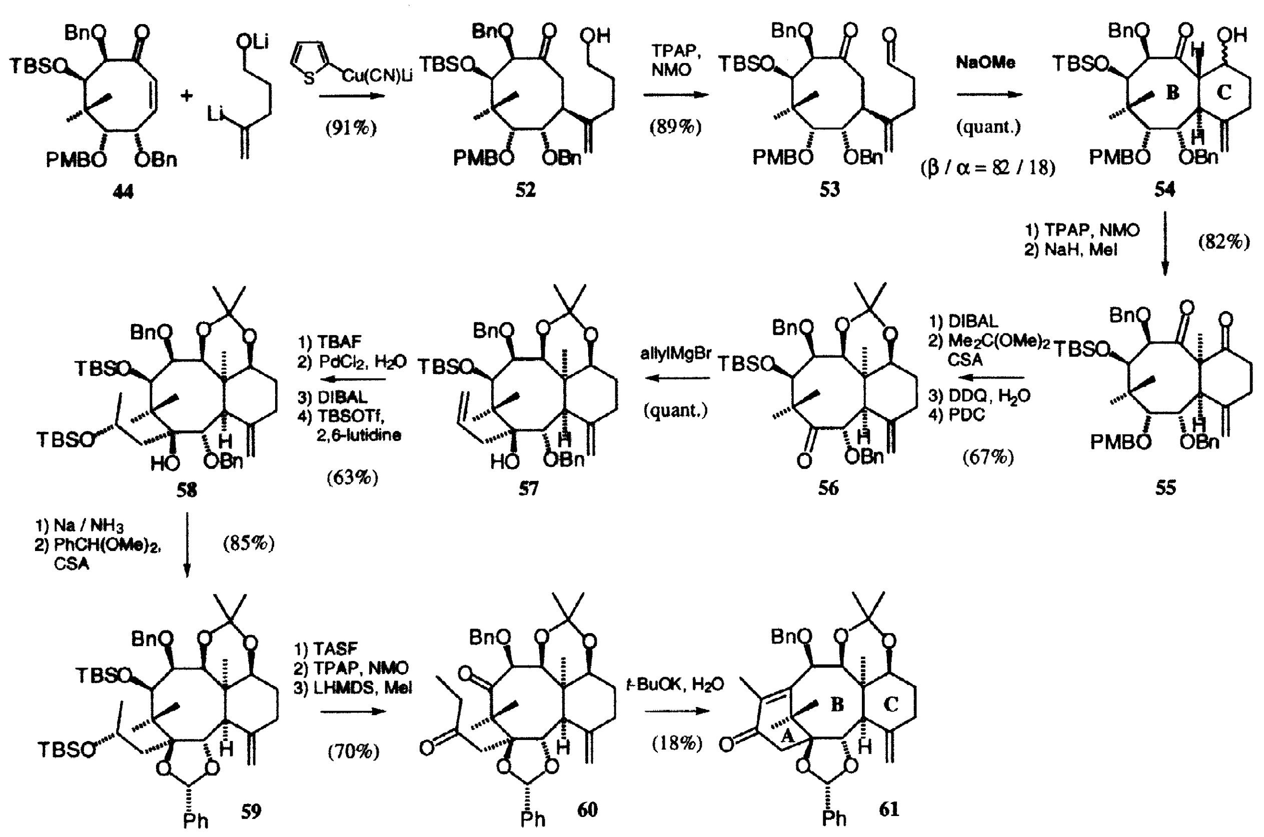
However, thus obtained AB ring system **50** including 1,5-diene structure is an unstable compound, and intramolecular Cope rearrangement occurs spontaneously at room temperature to give [2.2.2] bicyclic compound **51** because C-3 and C-12 of this compound locate closely each other and the overlapping of molecular orbitals causes facile rearrangement. Therefore, we decided to change our synthetic plan of the ABC ring system to that of B to BC to ABC ring route in 1994.



Scheme 11. Synthesis of the AB ring system **50**

The synthetic strategy was next designed toward the BC ring system of Taxol *via* intramolecular aldol cyclization and following stereoselective methylation with methyl iodide.^{60} The Michael addition of cuprate reagent, generated in situ from 2 mol equivalent of 4-bromo-4-pentene-1-ol with 6 mol equivalent of t-BuLi and 1 mol equivalent of CuCN, to the 8-membered ring enone **44** proceeded smoothly and the desired β-monosubstituted 8-membered ring hydroxyketone **52** was obtained in high yield with perfect diastereoselectivity. Ketoaldehyde **53**, a precursor of BC ring system of 8-demethyltaxoid, was prepared directly by oxidation of **52** with TPAP and NMO combined system. When intramolecular aldol reaction of the precursor **53** was tried in the presence of NaOMe at room temperature, the desired reaction proceeded smoothly to afford a mixture of bicyclic compounds **54** in nearly quantitative yield with good diastereoselectivity (87 / 13 / 0 / 0). Oxidation of the mixture afforded the corresponding diketone in high yield. Methylation of the diketone took place by using 1 mol of NaH and excess amount of methyl iodide where the desired methylated compound **55**, corresponding to BC ring system of Taxol, was obtained in high yield. Though the bicyclic compound **55** had an opposite configuration to Taxol at C-8 position, α-isomer concerning methyl group at C-8 position was supposed to be epimerized to more favorable β-isomer by successive retro-aldol and aldol reactions after constructing ABC ring system.

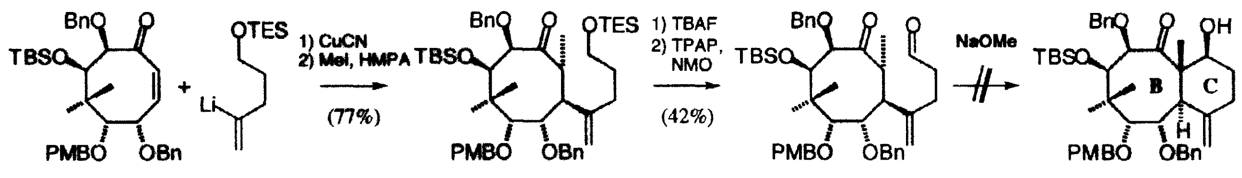
A highly diastereoselective reduction of the diketone **55** with DIBAL, followed by protection thus formed diol with isopropylidene acetal provided tricyclic compound. 8-Membered ring ketone **56** was obtained in good yield by DDQ oxidation of the above tricyclic compound, followed by oxidation with PDC. Allylation of **56** with allylmagnesium bromide afforded homoallyl alcohol **57** as a single stereoisomer in quantitative yield. Wacker oxygenation of C-13 position in the above homoallyl alcohol **57** was carried out under forced conditions and the corresponding hydroxyketone was obtained in good yield. Reduction of the carbonyl group and successive protection of hydroxyl group gave disilyl ether **58**. Regioselective deprotection of benzyl group in **58** and following protection thus formed 1,2-diol gave benzylidene acetal **59**. The acetal was converted to the desired diketone **60**, a precursor of the ABC ring system of 8-epitaxoid, by deprotection of TBS groups, oxidation with TPAP and NMO, and successive methylation. Then, synthesis of the ABC ring system was tried in order to construct the basic skeleton of 8-epitaxoid by intramolecular aldol condensation reaction. When aldol reaction of **60** was carried out in the presence of an excess amount of KO^{t}Bu in THF with a trace amount of water, the ABC ring system **61** which possesses C-11, C-12 bridgehead double bond was obtained in low yield.



Scheme 12. Synthesis of the ABC ring system of 8-epitaxoid **61**

However, we concluded that this route was not appropriate because it needed too many steps to come out and the yield of the desired enone **61** was low. In addition, it was considered that this route directed to the ABC ring system of 8-epitaxoid included additional deprotection-protection steps for preparing the precursor, and the stereochemistry at C-8 had to be inverted in a later stage of the synthesis. Then in 1995, we decided to make an alternative plan once again for the synthesis of the desired BC ring system.

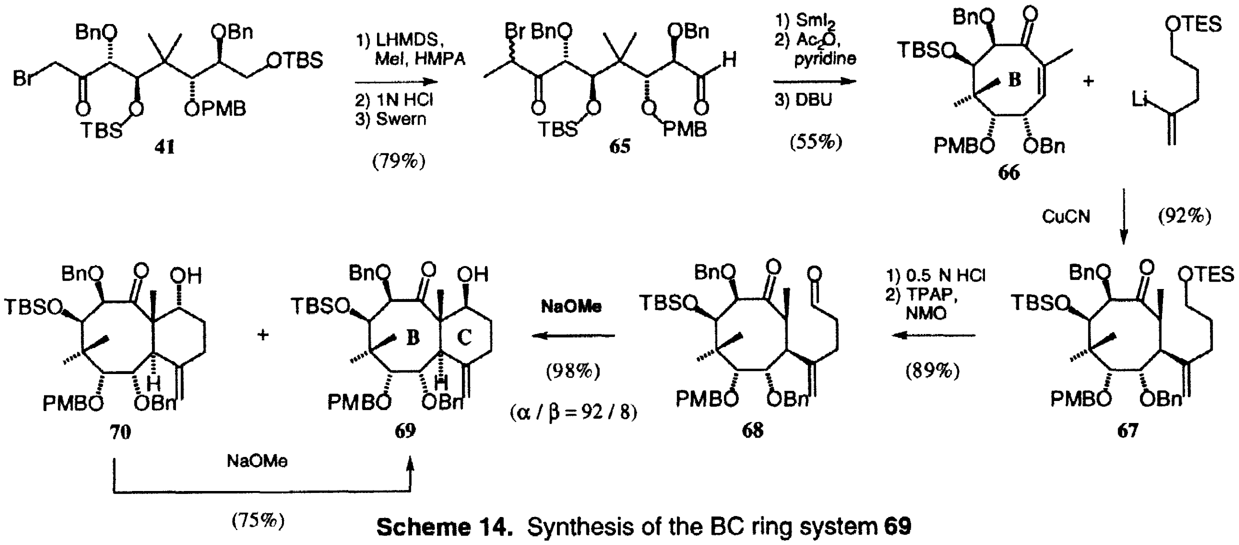
In order to construct the BC ring system having methyl group at C-8 position, a three-component coupling reaction of **44** with a cuprate reagent, followed by trapping with methyl iodide was tried under several reaction conditions. The three-component coupling reaction was carried out smoothly by employing the cuprate reagent generated in situ from 2 mol equivalent of 4-bromo-1-triethylsiloxy-4-pentene with 4 mol equivalent of *t*-BuLi and 1 mol equivalent of CuCN, and successive treatment with excess amounts of methyl iodide and HMPA. Ketoaldehyde **63**, a precursor of the BC ring system of Taxol, was obtained in high yield by deprotecting **62** with TBAF, followed by Swern oxidation. Next, synthesis of bicyclic compound **64** from the precursor **63** which contains all the required functionalities for constructing Taxol was tried under several reaction conditions. Contrary to our expectation, intramolecular aldol reaction did not proceed at all in any case, and ketoaldehyde **63** was recovered almost quantitatively.



Scheme 13. Attempted synthesis of the BC ring system 64

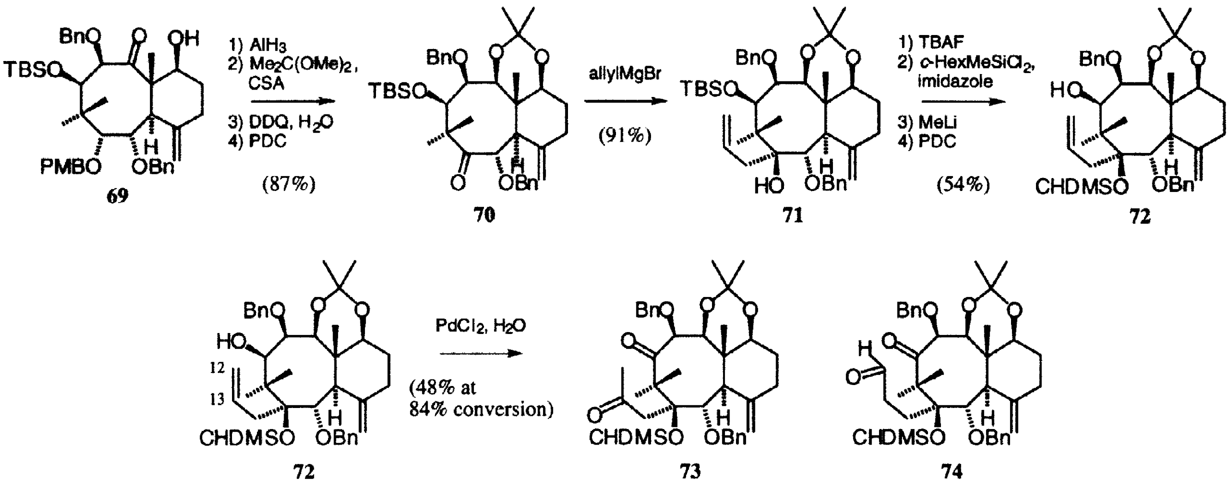
By the conformational analysis of ketoaldehyde 63, it was assumed that generation of enolate anion by deprotonation from 63 having a C-3,8 *trans*-configuration is hardly possible under the standard conditions because the dihedral angle between the H-C8-C9=O bonds is nearly antiperiplanar. This gave us an important recognition that a ketoaldehyde 68 having the C-3,8 *cis*-configuration is able to generate the key enolate anion (Scheme 14). The enolate anion thus formed should easily cyclize with the aldehyde at C-7 to form the BC ring system of Taxol by intramolecular aldol reaction. Then, it was planned to synthesize the desired ketoaldehyde 68 having the C-3,8 *cis*-configuration by α-face selective hydrolysis of the intermediate Michael adduct, formed from 8-membered ring enone 66 and the cuprate reagent.[61,62]

Methylated 8-membered ring enone 66 was prepared after synthesis of 8-demethyl compound via methylation of an enolate anion derived from the polyoxy linear intermediate 41. Michael addition of the cuprate reagent generated in situ from 7 mol equivalent of 2-bromo-5-triethylsiloxypentene, 14 mol equivalent of *t*-BuLi and 3.6 mol equivalent of copper cyanide to the enone 67 gave the 8-membered ring ketone having the C-3,8 *cis*-configuration in high yield with almost perfect diastereoselectivity by way of α-face selective hydrolysis of the enolate anion. Ketoaldehyde 68, a precursor of the BC ring system of Taxol, was obtained in good yield by the deprotection of the above Michael adduct with 0.5 N HCl, followed by oxidation with TPAP and NMO. As expected, the cyclization proceeded smoothly to afford a mixture of bicyclic compounds in nearly quantitative yield with high diastereoselectivity when intramolecular aldol reaction of the precursor 68 was carried out in the presence of NaOMe. The diastereomer 70 that has an α-hydroxyl group at the C-7 position could be epimerized to the desired β-alcohol 69 in good yield on treatment with NaOMe.



Scheme 14. Synthesis of the BC ring system 69

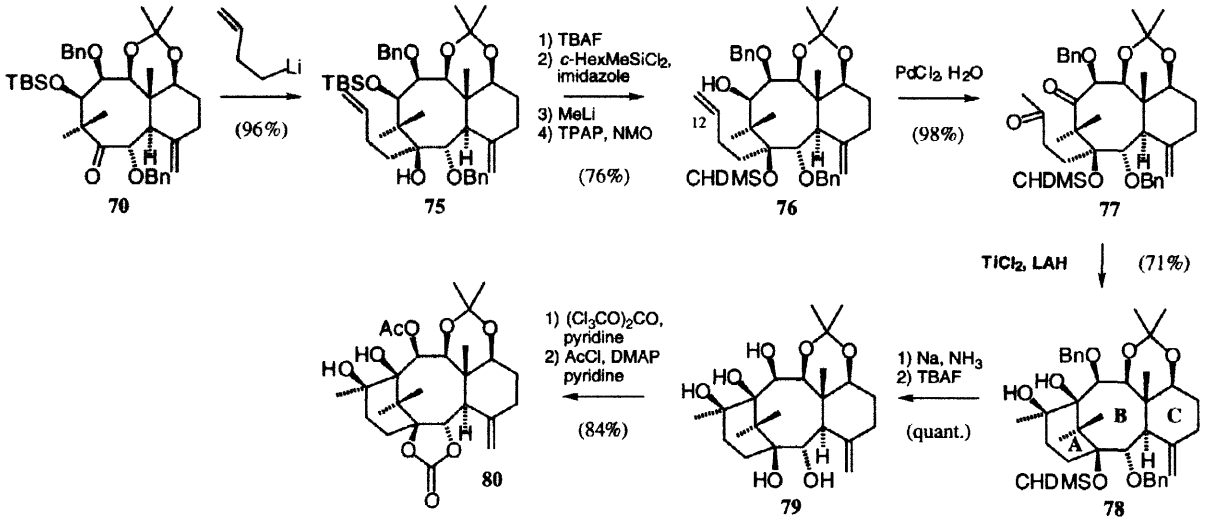
Then, we focused our attention on the synthesis of ABC ring system of Taxol by intramolecular aldol reaction using C-11, C-13 diketone **73** as a precursor (Scheme 15). Diastereoselective reduction of aldol **69** with AlH_3 and followed by treatment with isopropylidene acetal provided tricyclic compound, which was converted to 8-membered ring ketone **70** by successive DDQ and PDC oxidations. When **70** was treated with allyllithium reagent, the desired homoallyl β-alcohol **71** was obtained as a single stereoisomer in quantitative yield. Removal of the TBS group gave *cis*-diol and subsequent treatment of the diol with dichlorocyclohexylmethylsilane yielded a silylene compound, which was then converted to the trialkylsilylether at the C-1 position by way of alkylation of the bridged silicon atom with alkyllithium reagent. Oxidation of thus formed secondary alcohol with PDC gave 8-membered ring ketone **72** in good yield. In order to prepare C-13 oxygenated compound **73**, a precursor of the ABC ring system of Taxol, several methods of oxygenation at C-13 position were examined. However, in no case did the desired reaction take place and, against our expectation, the C-12 position was regioselectively oxygenated to form ketoaldehyde **74** under forced conditions.



Scheme 15. Attempted synthesis of a precursor of the ABC ring system **73**

Again, we were on the verge of another turning point. Although it was impossible to think of the exact strategy right away, we at once proceeded to reconsider every unsolved questions all over again, for our motto is "to catch the interesting while running." After a while, we succeeded at last to construct the A ring, our immediate target, by the pinacol coupling according to the hint obtained through experiments which is to be described later.

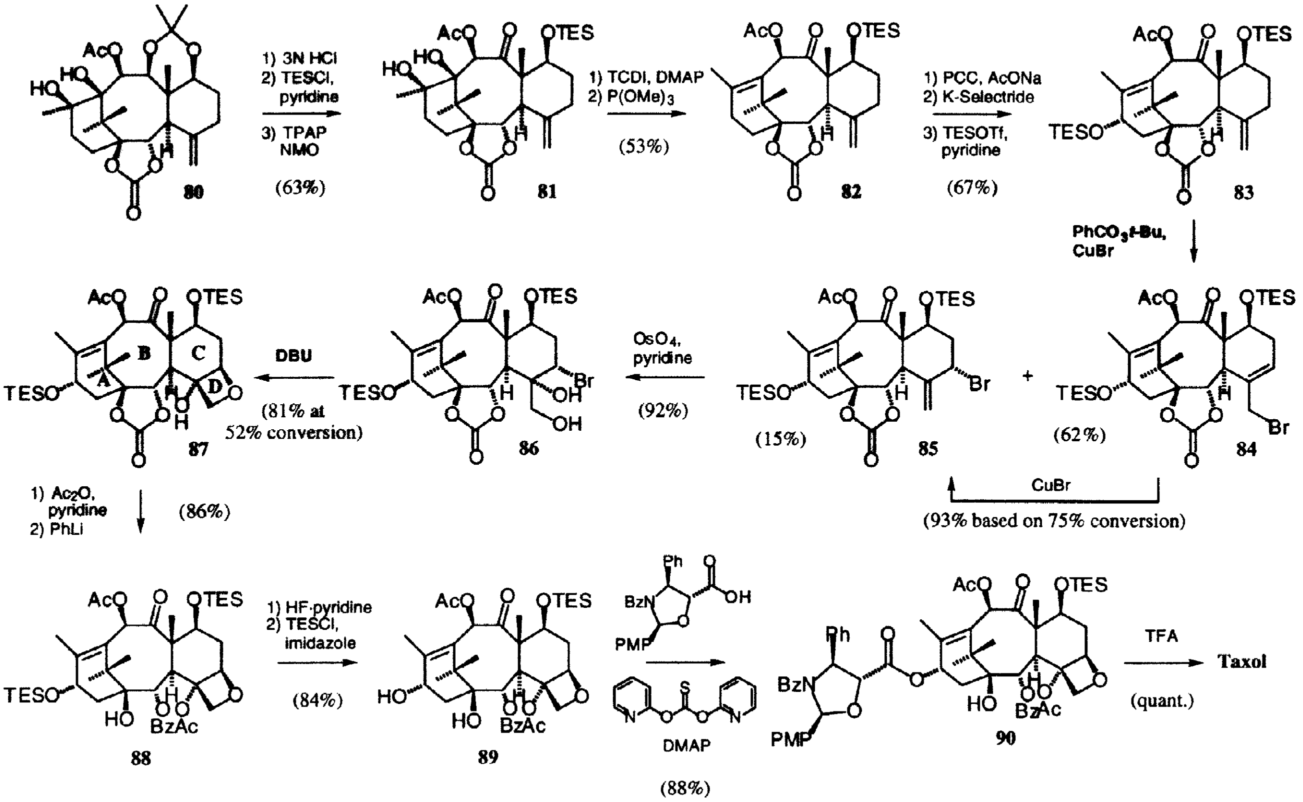
The ^1H NMR of **72** and MM2 calculation of its conformation indicate that **72** has a rigid tricyclic structure. Comparison of the environments of the C-12 and C-13 positions in the model suggested that the C-12 position was more easily attacked by a water molecule during Wacker oxygenation because the C-13 position is considerably shielded by both the trialkylsilyl group and the exo olefin on the C ring of **72**. These results led to a new synthetic strategy of forming the ABC ring system of Taxol from intermediate **77** (Scheme 16). This **77** was prepared by the preferential oxygenation of the C-12 position of **76** as shown in the Scheme. Therefore, alkylation of the C-1 position of **70** with homoallyllithium reagent was tried in order to produce bishomoallyl β-alcohol which was in turn directly converted to the ABC ring system by way of an intramolecular pinacol coupling reaction by using a low valent metal reagent. Treatment of *cis*-diol derived from **75** with dichlorocyclohexylmethylsilane yielded the silylene derivative in high yield. Alkylation with methyllithium furnished secondary alcohol having the desired C-1 protected hydroxyl group. The desired diketone **77**, precursor of ABC ring system of Taxol, was obtained from the alcohol by successive oxidation with PDC and Wacker oxygenation. Next, the intramolecular pinacol coupling reaction of **77** using the low valent titanium reagent, prepared from $TiCl_2$ and $LiAlH_4$, gave the novel taxoid **78** containing the ABC ring system of Taxol.[63] Successive deprotections of **78** with Na/NH_3 and TBAF gave the desired pentaol **79** in high yields. Regioselective protection of the pentaol **79** with bis(trichloromethyl)carbonate and with acetic anhydride afforded the corresponding C-10 acetoxy, C-1, C-2 carbonate **80** in good yield.



Scheme 16. Synthesis of the ABC ring system **80**

Deprotection of the acetonide group and regioselective protection of thus formed tetraol, followed by oxidation of triol with a combination of TPAP and NMO yielded C-9 ketone **81**. A novel taxoid **82** was formed by the desulfurization of thionocarbonate derived from the above ketone **81**. Regioselective oxygenation at the C-13 position of **82** with PCC and NaOAc gave an enone, which in turn was reduced to the desired α-alcohol stereoselectively on treatment with K-Selectride. Protection of thus formed α-alcohol afforded tetracyclic compound **83** possessing all the functionalities of Taxol. Allylic bromination at the C-5 position of **83** by using excess amounts of CuBr and $PhCO_3t$-Bu (1:1 molar ratio) gave two separable allylic bromides **84** and **85** in 62% and 15% yields, respectively. Further treatment of the allylic bromide **84** with CuBr in CH_3CN, 25% of **84** and 64% of **85** were obtained. Dihydroxylation of this allylic bromide **85** with OsO_4 in pyridine gave a dihydroxy bromide **86** in 92% yield as a single stereoisomer. The desired oxetanol **87** was obtained in good yield when this dihydroxy bromide was treated with DBU. The corresponding acetate was prepared by acetylation of the tertiary alcohol **87** with acetic anhydride in pyridine. The carbonate was benzoylated at the C-2 position by treating with phenyllithium to give 7,13-bistriethylsilylbaccatin III (**88**) in high yield. Desilylation of **88** followed by monosilylation of the intermediate triol afforded 7-triethylsilylbaccatin III (**89**) in good yield.[63,64]

The so-called β–lactam method has been the most popular method for the introduction of side chains to 7-*O*-protected baccatin III, and was utilized in all the reported total syntheses of Taxol. By making good use of what we have built up in reaction chemistry, we tried at this stage to develop a new method for the direct condensation reaction between **89** and the side chain as its free carboxylic acid form under rather mild conditions because the β–lactam method was generally carried out under strongly basic conditions. The dehydration condensation between **89** and a derivative of *N*-benzoylphenylisoserine, prepared from achiral starting materials by our enantioselective aldol reactions, was studied and it was found that the condensation reaction proceeded smoothly when DPTC (di(2-pyridyl)thionocarbonate) was used as a novel coupling reagent in the presence of DMAP affording the corresponding ester **90** in 95% yield at 93% conversion.[65,66] Finally, deprotection of the protected Taxol **90** thus formed gave the final target molecule Taxol in nearly quantitative yield.[67,68]



Scheme 17. Completion of the asymmetric total synthesis of Taxol

It took us about 5 years to complete this total synthesis and there were many times of standstill. Yet we never ceased to challenge to new possibilities because of the dream of reaching goal. We are proud that we never lost our enthusiasm all through and that we have done this with the members of only graduate students, none with doctorates except myself.

I would like to express my hearty thanks to many coworkers who worked very hard to complete all of the topics described herein.

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