Table II. Energies of MM2 (MODEL) Minimized Conformations of Calix[4]resorcinarenes

conformation	E (kcal)	conformation	E (kcal)
flattened cone (C_{2v})	86.7	flattened partial cone 2 (C_{2h})	89.1
flattened partial cone 1 (C_s)	87.6	1,2 alternate (C_{2h})	90.0
1,3 alternate (D_{2d})	87.8		

Table III. Energies of MM2 (MODEL) Minimized Conformations of *C*-(Carbomethoxymethyl)calix[4]resorcinarenes

conformation	E (kcal)	conformation	E (kcal)
flattened cone (C_{2v})	109.4	flattened partial cone 1 (C_s)	118.2
1,2 alternate (C_s)	111.6	flattened partial cone 2 (C_2h)	121.3
1,3 alternate (D_{2d})	116.1		

Since it was not possible to obtain suitable crystals of **3** and **4** for X-ray analysis, a molecular modeling study was undertaken in order to compare the 3D-structures with those suggested by the NMR results. A calix[4]resorcinarene with methylene bridges was manually input by the SKETCH mode of the program SYBIL[6] and minimized with the SYBIL MAXIMIN2 routine (TRIPOS force field) on a Silicon Graphics Personal Iris workstation. A random conformational search with a 10° stepwise increment of the eight nonaromatic rotatable C–C bonds was carried out on this molecule using the SEARCH module within SYBIL with an energy cutoff of 70 kcal/mol (default value). This calculation yielded 33 geometries, which converged after a MAXIMIN2 energy minimization to five conformations, resembling with some distortion the flattened cone, two kinds of flattened partial cone, the 1,3 alternate, and the 1,2 alternate conformations. Further minimization of these structures with the program MODEL[7] (MM2 force field) led to the first above-reported four conformations, with the symmetries C_{2v}, C_{2h}, C_s, and D_{2d}, respectively. The 1,2-alternate conformation was built up from the fifth structure, which showed a very high strain energy, by changing manually the coordinates of half of the molecules as to obtain a C_{2h} symmetry. The input structure was then minimized with the program MODEL. The energies of the final five MM2 minimized conformations are reported in Table II. The four aliphatic chain CH_2COOCH_3 were then added to each conformation; in the case of the 1,2 alternate conformation the substituents were drawn in cis–trans–cis configuration relative to C-2, in accordance with the ^1H NMR data, while in the remaining conformations the R groups were added in all-cis relative configuration. The five structures so obtained were minimized until convergence with MODEL and then submitted to a further random search on the side chains to find the preferred spatial orientation. The results of the molecular mechanics calculations on the five conformations of the calix[4]resorcinarene **2a** (n = 4) are summarized in Table III. The calculations predict the flattened cone conformation to be at the lowest energy in agreement with our results and the literature data.[4,8] The 1,2 alternate conformation with the cis–trans–cis configuration was also shown to have a low conformational energy, in agreement with the experimental results.

An all-cis configuration of the minimized 1,2 alternate conformation was built up by epimerization of C-14 atom by EPIMR command within MODEL. Notably, this stereoisomer minimized to a higher steric energy of 116.5 kcal/mol. In summary, the molecular modeling study confirmed the conformations and the configurations assigned on the basis of the NMR experiments.

Treatment with $BF_3 \cdot Et_2O$ of other cinnamates (**1b** and **1c**), to be presented in a future detailed paper, gave compounds with general structures **2b** ($n = 4$) and **2c** ($n = 4$), respectively, thus affording evidence for the general versatility of the reaction.[9] The nonphenolic substrate, the low (room) temperature, and the good yields are the outstanding features of this approach to calixarenes.

Acknowledgment. We thank Professor James P. Kutney, University of British Columbia, Vancouver, for a discussion of the paper. This work was supported by a grant from the Consiglio Nazionale delle Ricerche of Italy and the Hungarian Academy of Sciences, as well as from Progetto Speciale C.N.R. "Meccanismi molecolari della trasduzione del segnale".

Registry No. **1a**, 66417-42-3; **1b**, 24393-63-3; **1c**, 140111-45-1; **3** (R = $COOCH_3$), 140111-46-2; **3** (R = $COOCH_2CH_3$), 140111-47-3; **3** (R = $COOCH(CH_3)_2$), 140111-48-4; **4** (R = $COOCH_3$), 140223-16-1; **4** (R = $COOCH_2CH_3$), 140223-17-2; **4** (R= $COOCH(CH_3)_2$), 140223-18-3; **5**, 140111-49-5.

Supplementary Material Available: Experimental details and data of **3**–**5**, including ^1H and ^{13}C NMR spectra (8 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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[6] SYBIL (Version 5.4); Tripos Associates, Inc.: St. Louis, MO 63144.

[7] Steliou, K. MODEL (Version K.S.2.96); University of Montreal, Canada.

[8] Hogberg, A. G. S. *J. Org. Chem.* **1980**, *45*, 4498.

[9] On the other hand, 2,6-dimethoxycinnamic acid ethyl ester undergoes initial rearrangement to 2,4-dimethoxycinnamate (**1b**) and subsequent tetramerization to afford calixarenes with general structure **2b** ($n = 4$).

Stereocontrolled Synthesis of a C_1–C_{15} Segment for the Marine Macrolides Swinholide A and Scytophycin C: Use of a Vinylogous Mukaiyama Aldol Reaction

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Received February 25, 1992

Summary: The C_1–C_{15} segment (±)-**8** of swinholide A/scytophycin C was prepared in eight steps from (*E*)-4-chlorobut-3-en-2-one (**10**) in 19% overall yield with 87% diastereoselectivity. The C_7 stereocenter was controlled by the novel, vinylogous Mukaiyama aldol reaction, **16** + **18** → **19**, mediated by $BF_3 \cdot OEt_2$. The related C_1–C_{13} segment (±)-**9** for misakinolide A was also prepared.

Swinholide A, a novel cytotoxic macrolide isolated from marine sponges of the genus *Theonella swinhoei*, was first

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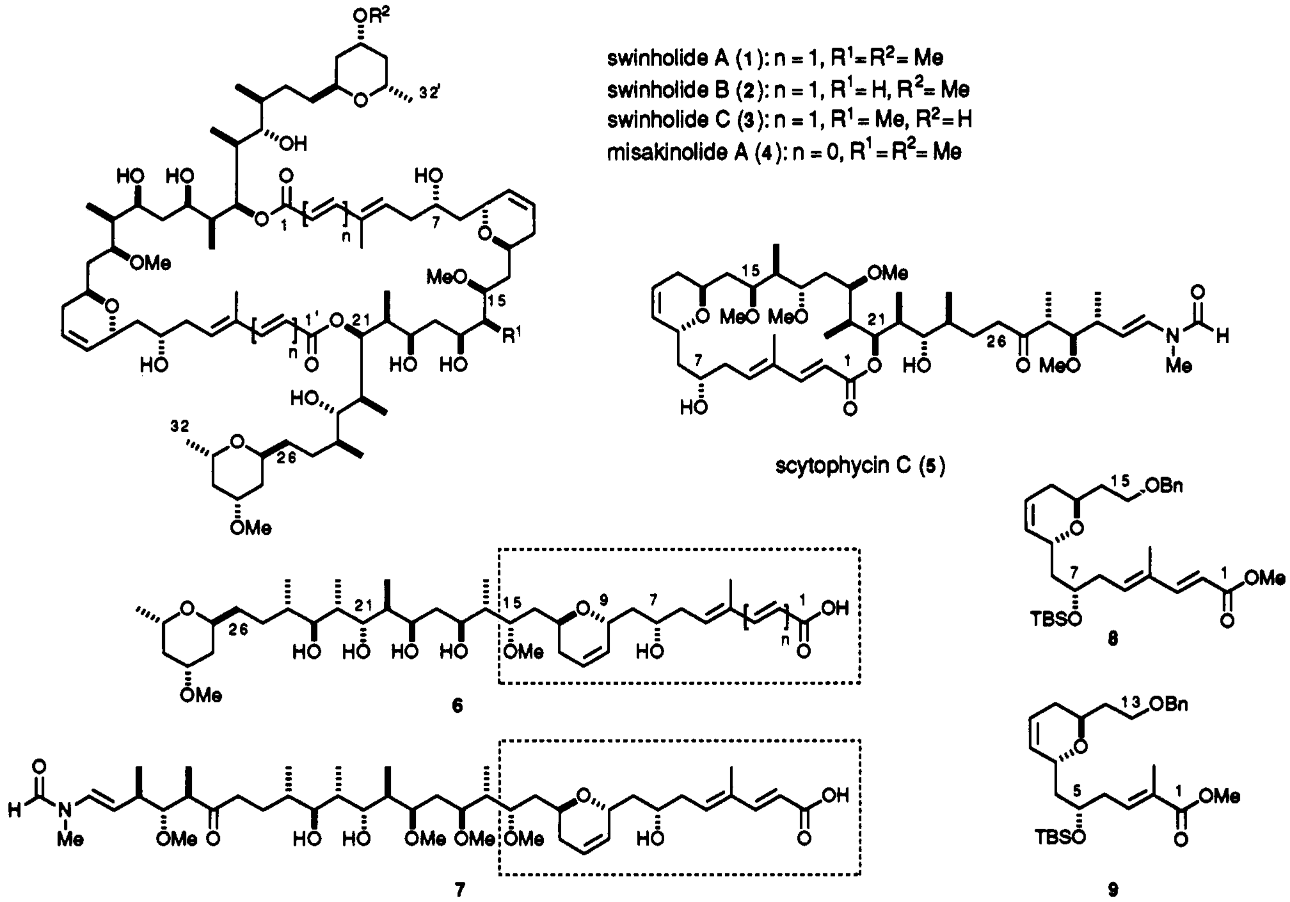
Chart I



reported by Carmely and Kashman in 1985.[1] Recently, mass spectroscopic[2a] and X-ray crystallographic[2b-d] studies have shown it to be the symmetrical dimer 1 with an unusual 44-membered ring. Several other dimeric macrolides have also been obtained from *Theonella*, including swinholides B (2) and C (3)[2e] and the analogous 40-membered dilactone, misakinolide A (4)[3a-c] (≡ bistheonellide A[3b,d]). Scytophycin C (5), a related monomeric 22-membered macrolide, has been isolated by Moore et al.[4] from the blue-green alga *Scytonema pseudohofmanni*, and this also exhibits cytotoxicity and antifungal activity. As can be seen from the respective seco acid structures 6 (n = 0 for misakinolide A, *n* = 1 for swinholide A[2f]) and 7 (for scytophycin C), these marine macrolides have identical stereostructures spanning C$_1$–C$_{24}$ (C$_1$–C$_{22}$ for misakinolide A) and differ mainly in the ring size and the nature of the side-chain terminus attached to C$_{26}$. To date, no synthetic

Scheme I

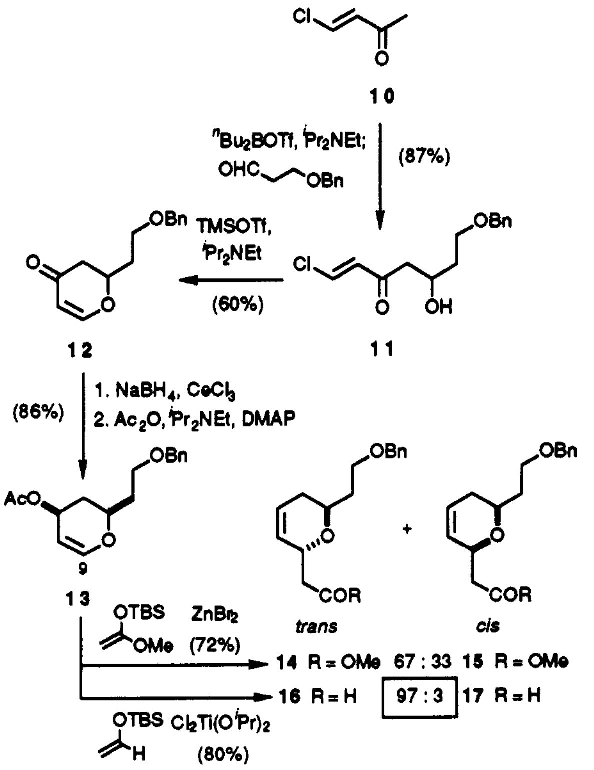


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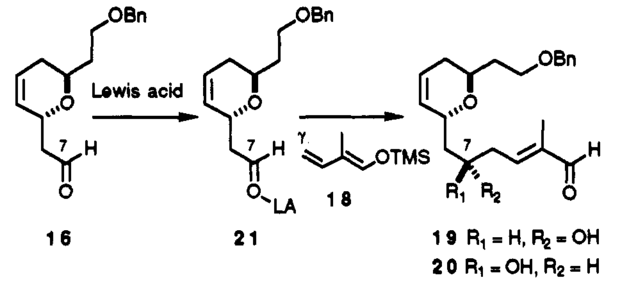
Table I. Lewis Acid-Promoted Additions of Silyl Dienol Ether 18 to Aldehyde 16



entry	Lewis acid	solvent	temp (°C)	19:20[a]	% yield[b]
1	$Cl_2Ti(O^iPr)_2$	CH_2Cl_2	−78	72:28	24 (48)
2	$Cl_2Ti(O^iPr)_2$	CH_2Cl_2	−40	78:22	40 (72)
3	$BF_3{\cdot}OEt_2$	Et_2O	−78 → −40	83:17	61 (76)
4	$BF_3{\cdot}OEt_2$	THF	−78 → 20	58:42	50 (62)
5	$BF_3{\cdot}OEt_2$	CH_2Cl_2	−78	86:14	62
6	$BF_3{\cdot}OEt_2$	9:1 CH_2Cl_2/Et_2O	−78	90:10	70 (78)

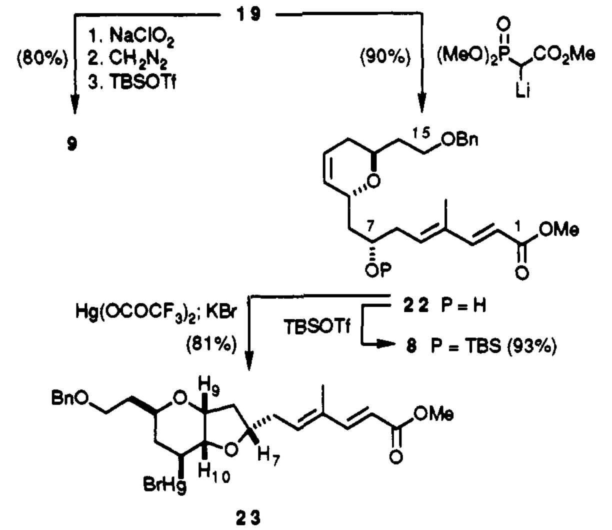
[a] Determined by weighing after chromatographic separation. [b] Number in parentheses is yield based on recovered 16.

work has been described for any of these structurally-complex, marine macrolides.[5] Herein we report the stereocontrolled synthesis of the C_1–C_{15} segment 8 for swinholide A and scytophycin C and the related C_1–C_{13} segment 9 for misakinolide A.

A boron aldol reaction between (*E*)-4-chlorobut-3-en-2-one (10)[6] and 3-(benzyloxy)propanal, mediated by $^nBu_2BOTf/^iPr_2NEt$ (CH_2Cl_2, −78 °C),[7] gave the β-hydroxy ketone 11 in 87% yield (Scheme I). Cyclization to the dihydropyrone, 11 → 12 (60%), was then achieved using Me_3SiOTf (1.05 equiv) and $^iPr_2NEt$ (0.80 equiv) in CH_2Cl_2, under our previously reported conditions.[7] Reduction of ketone 12 by $NaBH_4/CeCl_3$[8] (MeOH/EtOH, −78 °C), followed by acetylation with Ac_2O, gave the acid-sensitive dihydropyran 13 (86%), in readiness for the stereoselective introduction of the C_9 side chain.

Initial investigations of the carbon[9] Ferrier rearrangement[10] of 13 used 1-(*tert*-butyldimethylsiloxy)-1-methoxyethene, in the presence of various Lewis acids ($TiCl_4$, $BF_3{\cdot}OEt_2$, Me_3SiOTf, $ZnBr_2$). These reactions all exhibited low stereoselectivity, e.g., $ZnBr_2$ catalysis in CH_2Cl_2 (20 °C, 2.5 h) gave a 2:1 mixture of the trans and cis dihydropyrans 14 and 15 in 72% yield. DIBAL reduction of each of these esters then gave the corresponding aldehydes 16 and 17. In contrast, the use of (*tert*-butyldimethylsiloxy)ethene[11] (1.3 equiv), in conjunction with $Cl_2Ti(O^iPr)_2$ (1.1 equiv) in PhMe at −42 °C, resulted in the direct formation of the desired aldehyde 16 in 80% yield—now with essentially complete trans selectivity (16:17 = 97:3 by capillary GC analysis; stereochemistry determined by ^1H NMR NOE difference experiments). Similar stereochemical results have been reported by Danishefsky for carbon Ferrier rearrangements using allylsilanes on related systems,[9b,c] although the use of silyl enol ethers usually gives much lower levels of stereocontrol.[9a]

Scheme II



The Lewis acid promoted addition of the silyl dienol ether 18[12] to aldehyde 16 was next investigated (Table I). Substrate-controlled introduction of the C_7 stereocenter in a 1,3-anti sense might be possible by chelation[13] of the aldehyde and the dihydropyran ether oxygens with a dicoordinating Lewis acid (e.g., $TiCl_4$). However, use of $TiCl_4$ led to decomposition, and the milder Lewis acid $Cl_2Ti(O^iPr)_2$, while giving some of the desired products 19 and 20 with moderate selectivity, suffered from poor conversion (entries 1–2). $BF_3{\cdot}OEt_2$ circumvented this problem and—when used in a solvent mixture of 9:1 CH_2Cl_2/Et_2O at −78 °C for 80 min (entry 6)—promoted the addition to give a 90:10 ratio of 19 and 20 (assigned stereochemistry later verified) in 78% yield. This novel, vinylogous Mukaiyama aldol reaction[14] only gave products of γ-attack[15] on 18, while the trisubstituted double bond in the enal products was exclusively the *E* isomer (NOE). Moreover, the sense of diastereoselectivity in the reaction was the same with both the boron and titanium Lewis acids. Thus, chelation was not required to obtain useful levels of diastereoface selectivity in the chiral aldehyde 16. Notably, the monocoordinating Lewis acid, $BF_3{\cdot}OEt_2$, gave highest diastereoselectivity, presumably due to conformationally-controlled attack of the nucleophile on the 1:1 aldehyde·BF_3 complex 21.[16,17]

The major aldol adduct 19 was converted (Scheme II) into the corresponding *E,E* unsaturated ester 22 in 90% yield by a Horner–Emmons reaction with methyl dimethylphosphonoacetate (^nBuLi, THF, 0 °C). At this stage, detailed comparisons of the ^1H and ^{13}C NMR spectra with that reported[2a,c] for the C_1–C_{15} section of the monomeric seco acid (methyl ester) of swinholide A indicated

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(17) The conformational preferences of this aldehyde–Lewis acid complex are currently under investigation by NMR and molecular modeling.

an almost exact match.[18] The assigned stereochemistry was subsequently verified by cyclization of the C_7 hydroxyl group on to the $C_{10,11}$ alkene in **22** by an intramolecular oxymercuration using $Hg(OCOCF_3)_2$ in THF, followed by treatment with aqueous KBr (81%).[19] The resulting cis-fused bicyclic mercurial **23** was sufficiently stable to isolate and characterize, and ^1H NMR NOE difference experiments in CD_3CN showed that the methine protons H_7, H_9, and H_{10} were all on the same face. In contrast, the corresponding C_7 epimeric mercurial, prepared in a similar fashion from the minor aldol adduct **20**, did not show these enhancements. This proved that the major aldol isomer had the correct C_7 stereochemistry for the target macrolides. Finally, protection of the hydroxyl group in **22** as the TBS ether ($^tBuMe_2SiOTf$, 2,6-lutidine, CH_2Cl_2, −78 °C; 93%) then gave the fully protected C_1–C_{15} segment **8** of swinholide A and scytophycin C. Additionally, the aldehyde **19** was oxidized with sodium chlorite[20] (NaH_2PO_4, $Me_2C{=}CHMe$, ^tBuOH) to the corresponding *E* unsaturated acid which, after esterification with CH_2N_2 and TBS protection, gave **9**, a fully protected C_1–C_{13} segment of misakinolide A.

In summary, we have achieved a short and highly diastereoselective synthesis (8 steps, 87% overall ds) of two related racemic intermediates for the cytotoxic macrolides **1**–**5**. Key steps are (i) the construction of the dihydropyrone **12** using our recently developed boron aldol-cyclization sequence,[7] (ii) the highly diastereoselective, carbon Ferrier rearrangement of **13** to give the aldehyde **16** directly, followed by (iii) a novel, vinylogous Mukaiyama aldol reaction with the silyl dienol ether **18** selectively giving the enal **19**, with stereocontrol at C_7 arising from a *nonchelation* pathway. Studies towards their asymmetric synthesis, using chiral boron reagents in step (i),[7] and further elaboration into swinholide A, misakinolide A, and scytophycin C are underway.

Acknowledgment. We thank the SERC (GR/H01922), Rhône-Poulenc Rorer, and Merck Sharp and Dohme (Terlings Park) for their support, Dr. P. Pitchen and Dr. C. G. Newton (RPR, Dagenham) for helpful discussions, and Dr. R. D. Tillyer (Cambridge) for his NMR expertise.

Registry No. **1**, 95927-67-6; **3**, 105694-30-2; **4**, 105304-96-9; **8**, 140902-85-8; **9**, 140902-87-0; **10**, 4643-20-3; **11**, 140902-76-7; **12**, 140902-77-8; **13**, 140902-79-0; **13** (alcohol), 140902-78-9; **14**, 140902-81-4; **15**, 140902-82-5; **16**, 140902-80-3; **18**, 98670-68-9; **19**, 140902-83-6; **20**, 141041-72-7; **22**, 140902-84-7; 7-epi-**22**, 141042-59-3; **23**, 140902-86-9; 7-epi-**23**, 141041-73-8; $PhCH_2O(CH_2)_2CHO$, 19790-60-4; $TBSOCH{=}CH_2$, 66031-93-4; $TBSOC(OMe){=}CH_2$, 77086-38-5; trimethyl phosphonoacetate, 5927-18-4; swinholide A, 95927-67-6; swinholide C, 105694-30-2; misakinolide A, 105304-96-9.

Supplementary Material Available: Experimental procedures and characterization data for all new compounds (5 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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[18] For the major isomer **22**, the ^{13}C chemical shifts in $CDCl_3$ agreed within ±1.1 ppm, while the ^1H NMR chemical shifts and multiplicities gave a close fit. In contrast, the C_7 epimer showed significant differences, particularly in the ^{13}C NMR spectrum, e.g., the carbon resonances for C_7 and C_9 differed by ca. 5 ppm relative to those in the monomeric seco acid (methyl ester) of swinholide A.[2c]

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Asymmetric Aza-Diels–Alder Reaction Mediated by Chiral Boron Reagent

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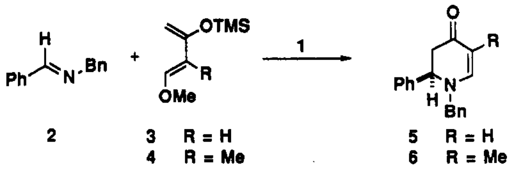
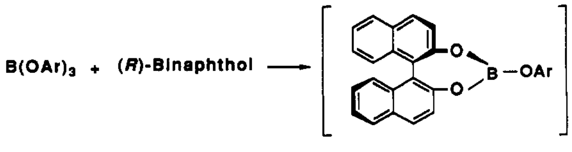
Received February 24, 1992

Summary: An asymmetric aza-Diels–Alder reaction of an imines mediated by an in situ generated chiral boron complex is described. The method is successful with several aldimines and affords products of up to 90% ee.

The development of chiral Lewis acid catalysts for carbon–carbon bond forming reactions is one of the most challenging and formidable goals in organic synthesis.[1] Unfortunately, however, the catalytic asymmetric reaction with *imine*, which can open up a wide variety of possibilities for the synthesis of natural products of the alkaloid family,[2] has never been developed to a useful level. In this paper, we wish to describe an asymmetric aza-Diels–Alder reaction[3] of an imine (eq 1) mediated by an in situ generated chiral boron complex of type **1**.[4] The method is successful with several aldimines and affords products of high enantiomeric purity.



(1)



(2)

1

The chiral boron complex was conveniently prepared in situ simply by mixing a 1:1 molar ratio of optically active binaphthol and triphenyl borate[5] in CH_2Cl_2 at ambient

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