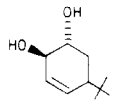
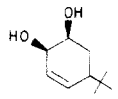
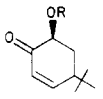
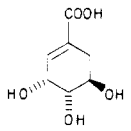
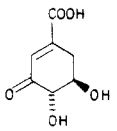
Table I. DDQ Oxidations of Enediols

reactant	DDQ equiv (solv)	time, h	temp, °C	product (yield,[a] %)
14	2.7 (C_6H_6)	12	60	**16** (100)
15	3.1 (C_6H_6)	26.5	60	**17** (64)
 18[b]	1.4 (C_6H_6)	16	70	**16** (70)
 19[c]	3 (C_6H_6)	 20, R = H (82)	70	#c
 21	3 (THF)	 22 (60)	65	#e

[a] All new compounds were completely characterized by IR, NMR, and MS. [b] Prepared from **7** in five steps: (i) TBDMS–Cl, imidazole; (ii) MCPBA–CH_2Cl_2, (iii) PhSeNa; (iv) MCPBA, –PhSeOH; (v) Bu_4NF. [c] Prepared from **8** in three steps: (i) MCPBA–CH_2Cl_2, (ii) PhSeNa; (iii) MCPBA, –PhSeOH.

acid (**21**) to 3-dehydroshikimic acid (**22**) in 60% yield, representing a major improvement over the rather capricious published procedure using Pt–O_2.[12]

To complete the model A-ring construction, enone **16** underwent one-pot cuprate addition [2.2 equiv of Li(C_3H_3)$_2$Cu, 5 equiv of Me_3SiCl, ether)] and oxidation [Pd(OAc)$_2$, CH_3CN] using the method of Saegusa[13] and furnished **23** (46%), which could be desilylated to the desired hydroxymethyl enone **24**. The same sequence on protected enones **17** and **20** (R = TBDMS) produced **24** and **25** in yields of 85% and 54%, respectively, without epimerization at the acidic α'-carbon. This short and efficient approach to highly functionalized A-rings should find application in several quassinoid total synthesis endeavors.

Acknowledgment. We thank the National Institutes of Health (GM 30050) for generous financial assistance. Support of the Cornell Nuclear Magnetic Resonance Facility by NSF (CHE 7904825, PCM 8018643) and NIH (RR02002) is gratefully acknowledged.

Registry No. **7**, 17502-34-0; **8**, 26819-49-8; **9**, 26828-76-2; **10**, 99417-69-3; **11**, 99417-68-2; **12**, 99417-70-6; **13**, 99417-67-1; **13** mono–TBDMS ether, 99417-71-7; **14**, 99417-61-5; **15**, 99417-62-6; **16**, 99417-64-8; **17**, 99417-65-9; **18**, 99439-58-4; **19**, 99417-63-7; **20**, 99417-66-0; **20** (R = TBDMS), 99457-35-9; **21**, 138-59-0; **22**, 2922-42-1; **23**, 99417-72-8; **24**, 99417-73-9; **24** (R′ = TBDMS), 99417-74-0; **25** (R = TBDMS), 99417-75-1; **25** (R = H), 99417-76-2; DIBPS, 85028-55-3.

Supplementary Material Available: Representative experimental procedures for the major transformations shown and characterization data for all new substances (7 pages). Ordering information is given on any current masthead page.

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Stereochemistry of Intramolecular Cyclopropanation of an Organoiron Reagent[1]

Summary: Intramolecular cyclopropanation using an iron carbene precursor results in formation of a trans-fused methanodecalin system.

Sir: Cyclopropanation of alkenes is an important operation in synthetic organic chemistry.[3] Intramolecular versions of these reactions have also proven to be quite useful, especially in the construction of complex polycyclic, cyclopropane-containing systems.[4] In some cases, cyclopropanations are followed by cleavage of the three-membered rings leaving carbon skeletons bearing stereoselectively introduced alkyl substituents.[5]

In recent years, carbene complexes of several transition metals have been shown to have many applications,[6] and cationic carbene complexes of the general structure $[(\eta^5\text{-}C_5H_5)(CO)_2Fe{=}CR^1R^2]^+X^-$ have proven to be excellent reagents for alkene cyclopropanations.[7] However, there have been very few reports of carbene complexes undergoing intramolecular cyclopropanation reactions.[8]

(1) Taken in part from the Ph.D. Dissertations of R. S. Iyer and G.-H. Kuo, State University of New York at Stony Brook, 1985.
(2) Address correspondence to this author at the Department of Chemistry, University of Notre Dame, Notre Dame, IN 46556.
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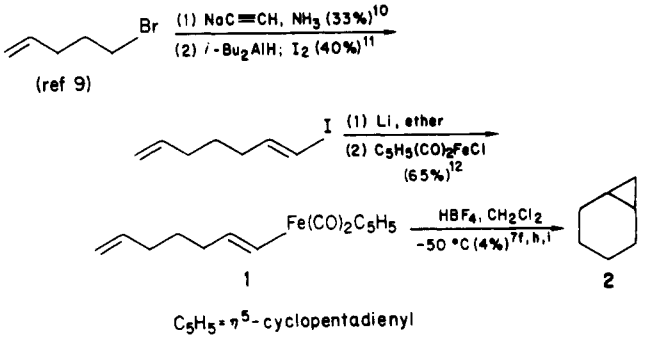
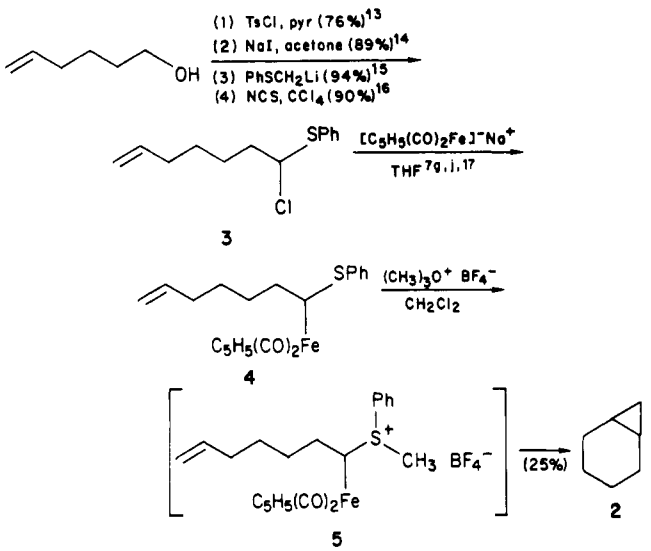
0022-3263/85/1950-5898$01.50/0

Scheme I



Scheme II

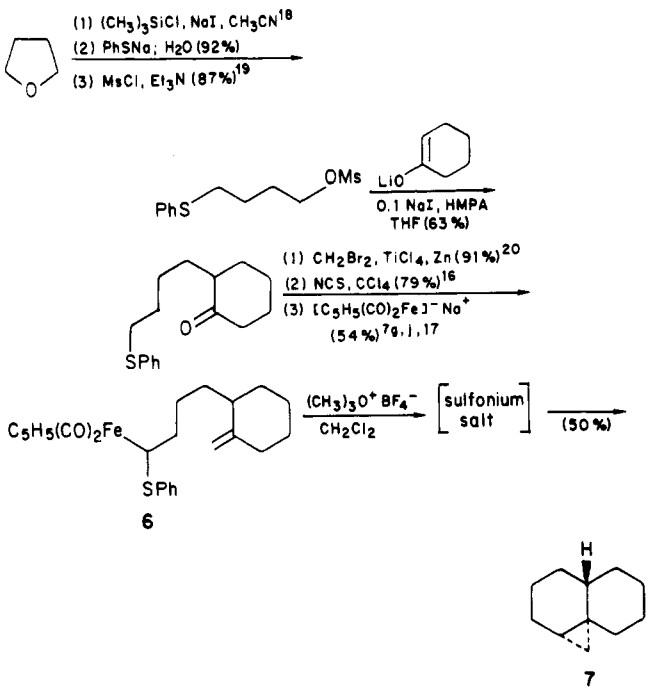


Herein we report an important case of an iron-based intramolecular cyclopropanation that proceeds with a very useful stereochemical outcome opposite to that obtained with other methods.

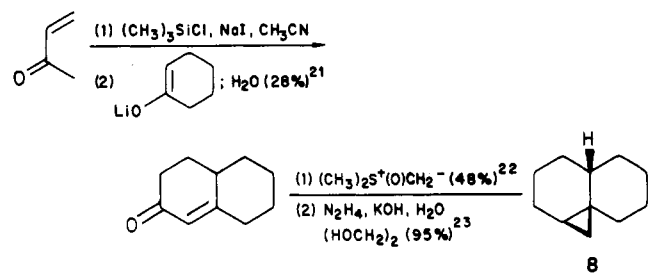
We had previously developed general methods for *intermolecular* cyclopropanations employing either iron-substituted sulfonium salts[7g,j] or alkenyliron complexes.[7f,h,i] For the sole purpose of determining which of these two methods would be most amenable to intramolecular cyclopropanations, we first prepared the simple model compounds 1 and 4 (Schemes I and II) as precursors of norcarane (2). In these sequences, no attempts were made to optimize the yields for any of the reactions, including the final cyclopropanations. The sulfur-containing derivative 4 was not fully purified before conversion into 2, but rather it was used in crude form, and no attempt was made to isolate the sulfonium salt 5. Therefore, the indicated yield of 2 in the second route is for the overall three-step conversion from the chloro sulfide 3.

Based upon the greater promise shown by the sulfur-based strategy, we chose to prepare the sulfide 6 (Scheme III) in order to examine a key stereochemical issue; cyclization could conceivably occur to give the trans-fused decalin derivative 7 or the cis-isomer 8. In actual practice, 7 was obtained with at least 10:1 selectivity, this lower limit being based upon limits of detection by ^{1}H NMR. In order to obtain material for comparison, 8 was prepared by two independent routes (Schemes IV and V). We note that the Simmons–Smith reaction[3a] of 9 fails, as is also the case for some related substrates.[23c]

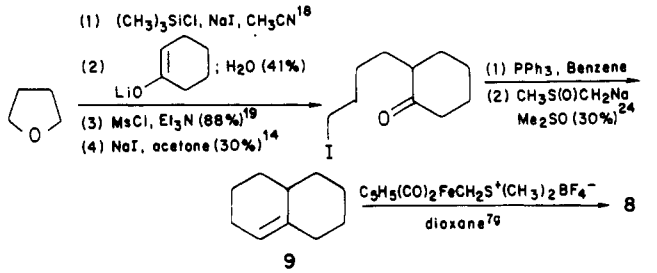
Scheme III



Scheme IV



Scheme V



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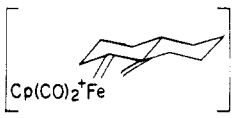
The isomeric cyclopropanes **7** and **8** exhibit quite different ^1H and ^{13}C NMR spectra.[25] Compared to **7**, **8** shows much more fully resolved and interpretable signals for the cyclopropyl protons; thorough decoupling experiments permit us to make peak assignments that are in accord with earlier data for related cyclopropanes.[23c,26]

Of significance is that the stereochemistry of the intramolecular cyclopropanation to give **7** is not only the opposite of the alternative *inter*molecular cyclopropanation routes shown above, but it is also the opposite of closely related cases of *intra*molecular cyclopropanations of diazo compounds.[4a,23b,27] We tentatively rationalize the stereochemical outcome of our reaction by hypothesizing a chair-like transition state **10** leading to **7**.[28]



10

The trans stereochemistry of **7** coincides with formerly proposed structures of the natural product cycloeudesmol (the structural assignment of which has been revised recently)[23,29] and with key intermediates in syntheses of other compounds.[30] Of potential importance is that our reaction may provide an approach to angularly alkylated, fused ring systems of defined stereochemistry,[28,31] a point which we are continuing to pursue in general.

Acknowledgment. We are grateful for the financial support provided by the National Science Foundation (Grant CHE 8120466) and the donors of the Petroleum Research Fund, administered by the American Chemical Society. The 300-MHz NMR spectrometer used in this work was purchased in part with funds provided by the NSF Instrumentation Program (Grant 8114412).

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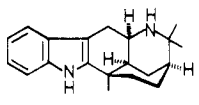
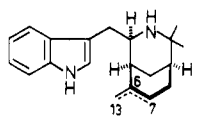
Stereocontrolled Total Syntheses of (-)-Hobartine and (+)-Aristoteline via an Intramolecular Nitrone-Olefin Cycloaddition

Summary: The *Aristotelia* alkaloids (-)-hobartine and (+)-aristoteline have been synthesized from indole and (*S*)-1-*p*-menthen-8-ylamine (**7**) in 11 steps (19%) via an intramolecular nitrone-olefin 1,3-dipolar cycloaddition.

Sir: Several species of plants from the genus *Aristotelia* contain a series of novel, structurally similar $C_{20}N_2$ indole alkaloids.[1] Distinguishing characteristics of these bases include tryptamine and nonrearranged geranyl subunits that have been functionalized and cyclized to varying degrees. (+)-Aristoteline (**1**), the major component in many species,[1a,2] (-)-hobartine (**2**),[1g] and (+)-makomakine (**3**)[1f] are representative examples.



(+)-1



(-)-2 Δ6,7

(+)-3 Δ6,13

Of the previous synthetic entries into this class of alkaloids,[1e,3] two reports describe syntheses of hobartine and makomakine and the conversion of each to aristoteline by treatment with concentrated hydrochloric acid.[3a,b] Although these routes to makomakine were both direct and enantioselective, the hobartine produced by each approach was necessarily racemic even when optically active starting materials were used. More recently, an asymmetric synthesis of (-)-hobartine was achieved via a biomimetic iminium ion cyclization.[3c]

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(25) **7**: ^1H NMR (300 MHz, $CDCl_3$) δ 0.96-2.08 (several m, 15 noncyclopropyl H), 0.73 (unresolved m, 2 cyclopropyl H), 0.17 (unresolved m, 1 cyclopropyl H); ^{13}C NMR (75 MHz, $CDCl_3$, apparent coincidence of 2 C) δ 39.32, 38.40, 33.23, 27.33, 27.25, 25.80, 23.94, 23.24, 17.35, 15.44. **8**: ^1H NMR (300 MHz, $CDCl_3$) δ 0.75-2.04 (several m, 15 non-cyclopropyl H), 0.66 (m, cyclopropyl methine H), 0.28 (apparent t, J = 4 Hz, endo cyclopropyl H), 0.14 (dd, J = 9, 4 Hz, exo cyclopropyl H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 38.75, 33.45, 30.85, 26.51, 26.42, 25.64, 23.15, 22.23, 17.95, 17.46, 15.93.
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(27) (a) McMurry, J. E.; Blaszczak, L. C. *J. Org. Chem.* **1974**, *39*, 2217. (b) Mori, K.; Ohki, M.; Kobayashi, A.; Matsui, M. *Tetrahedron* **1970**, *26*, 2815. A key factor in governing the stereochemistry in our case may be the absence of other ring substituents in substrate **6** compared to earlier examples (see especially ref 4a).
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