Although the regioselectivity of the cycloaddition could not be established solely from the ^1H NMR spectrum of the product, an off-resonance $^{13}C\{^1H\}$ NMR experiment[14] clearly established the structure of **15** to be as shown in Scheme II. This was deduced from the three nonaromatic, relatively low-field ^{13}C NMR resonances at δ 62.7, 67.0, and 83.9 assigned to (hobartine numbering) C(2), C(4), and C(6) (or C(5) had cycloaddition proceeded in the other sense). Thus, the resonances at δ 62.7 and 83.9 were found to arise from *quaternary* carbons, a result consistent only with structure **15**. The carbon α to oxygen in the other possible cycloadduct would not have been quaternary, and only one quaternary downfield resonance would have been observed.

The next task was the reductive cleavage of the isoxazolidine ring. The N–O bond of **15** was completely inert to all neutral and basic reducing systems investigated.[15] Upon treatment with buffered sodium amalgam,[16] **15** was smoothly converted[17] to isoxazolidine **5**[8] (91%), mp 210–210.5 °C, $[\alpha]^{28}_D$ −41 ± 2° (*c* 3.36, $CHCl_3$). The reluctance of similar isoxazolidines toward reduction has been noted by others.[18] However, **5** was cleanly reduced to the desired alcohol **4**[8,9] (96%), mp 145.5–146.5 °C, $[\alpha]^{28}_D$ +65 ± 2° (*c* 3.64, $CHCl_3$), by using zinc/aqueous acetic acid.[19] Treatment of **4** with *p*-toluenesulfonic acid (benzene, reflux, 30 min) afforded a mixture of (−)-hobartine (**2**) (50%) and (+)-aristoteline (**1**) (28%), separated by radial thick layer chromatography. Dehydration of **4** with neat trifluoroacetic acid (reflux, 12 h) produced (−)-hobartine (77%) exclusively. The (−)-hobartine obtained, mp 152.5–153.5 °C, $[\alpha]^{28}_D$ −27 ± 3 (*c* 1.69, $CHCl_3$) [lit.[1g] mp 149–150.5 °C, $[\alpha]^{20}_D$ −20 ± 3° (*c* 1.66, $CHCl_3$); lit.[3c] mp 151 °C, $[\alpha]^{25}_D$ −28° (*c* 1.2, $CHCl_3$)] gave spectra (IR, 300-MHz ^1H NMR, ^{13}C NMR, mass spectrum) identical with those of the natural product. In addition, our synthetic (+)-aristoteline (**1**)[8,20] was identical with a sample of the natural product (TLC, IR, ^1H NMR, ^{13}C NMR, mass spectrum).

Attempts to convert alcohol **4** to (+)-makomakine (**3**) were only partially successful. Thus, treatment of **4** with phosphorus oxychloride (pyridine, 3 h, 70 °C) gave a mixture (68%) of (−)-hobartine (**2**) and makomakine (**3**) in a ratio of ca. 88:12 (^1H NMR).

To summarize, we have described a convergent, stereocontrolled synthesis of (−)-hobartine in which a single chiral center (**7**) was elaborated into three through a highly regioselective and stereospecific intramolecular nitrone–olefin 1,3-dipolar cycloaddition. The synthetic sequence, while somewhat longer than the previous syntheses of hobartine, compares quite favorably in its overall efficiency.

Acknowledgment. This investigation was supported in part by PHS Grant GM-30761 awarded by the National Institutes of Health, the donors of the Petroleum Research Fund, administered by the American Chemical Society, and Merck Sharp and Dohme Research Laboratories. We also thank Professors I. R. C. Bick and M. Hesse for samples and spectra of natural hobartine, makomakine, and aristoteline and the National Science Foundation for funds to purchase a Varian XL-300 NMR spectrometer.

Supplementary Material Available: Complete experimental details (7 pages). Ordering information is given on any current masthead page.

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(14) Performed on a Varian XL-300 instrument with the proton decoupling frequency offset 2500 Hz.
(15) Aluminum amalgam,[16] sodium amalgam,[16] and $LiAlH_4$ in refluxing tetrahydrofuran (on **5**) were tried.
(16) Keck, G. L.; Fleming, S.; Nickell, D.; Weider, P. *Synth. Commun.* **1979**, *9*, 281–286.
(17) Sundberg, R. J.; Bloom, J. D. *J. Org. Chem.* **1981**, *46*, 4836–4842.
(18) See: DeShong, P.; Dicken, C. M.; Leginus, J. M.; Whittle, R. R. *J. Am. Chem. Soc.* **1984**, *106*, 5598–5602 and references cited therein.
(19) Iida, H.; Watanabe, Y.; Tanaka, M.; Kibayashi, C. *J. Org. Chem.* **1984**, *49*, 2412–2418.
(20) The sign of the $[\alpha]_D$ of **1** was found to be positive in agreement with ref 3c although an accurate value could not be obtained because of insufficient material.

The Synthesis and Relative Configuration of (±)-Lirionol

Summary: The synthesis of C-9 epimers **1** and **2** of the natural lignan lirionol is described. The relative configuration of the natural material is thereby established as **2** contrary to the previous assignment.

Sir: Lirionol, an unusual tetracyclic bridged lignan, was isolated from the bark of *Liriodendron tulipfera* and assigned[1] the structure and relative stereochemistry **1** from spectroscopic and biogenetic considerations. The bicyclo[3.3.1]nonadienone feature, constituting the central BC rings of lirionol, betrayed its probable biogenetic origin from a 1-aryl tetralin precursor and invited an attempt at synthesis by simple Friedel–Crafts cyclization to form the 3,4-bond of the molecule. Such a plan requires an efficient means for the stereocontrolled elaboration of an all-trans 1,2,3-substituted tetralin (e.g., **12**, Chart I) and we addressed this problem by application of the methods previously developed in our laboratory for the synthesis of (±) podophyllotoxin.[2,3] We now report the successful outcome of these efforts, the synthesis of both C-9 epimers **1** and **2** and the revision of the relative stereochemistry of lirionol.

Deprotonation of 2,3,4-trimethoxy-*N*,*N*-diethylbenzamide with *sec*-butyllithium–TMEDA and quenching the resultant 6-lithio species with 2,3,4-trimethoxybenzaldehyde was followed by lactonization of the amide–alcohol to provide[4] the phthalide **3** in 62% overall yield. Reduction with DIBAL-H in methylene chloride produced a mixture of diastereomeric lactols **4**, used after purification but without separation to generate the 1-arylisobenzofuran **5**, which was reacted[5] in situ with dimethyl acetylenedicarboxylate to yield (61% from **3**) the bicyclo adduct **6**. Thus all the carbon atoms of lirionol were assembled by this short sequence in 38% overall yield and in a convergent fashion.

Hydrogenolysis of **6** with 5% Pd/charcoal in ethyl acetate at 60 psi provided the all-cis tetralin **7** in 74% yield with no trace of any C-1 epimer. This was a surprising result, contrary to previous experience[2,6] with palladium-catalyzed hydrogenolysis. We are investigating the structural and stereochemical dependence of this reaction

(1) Chen, C.-L.; Chang, H.-M. *Phytochemistry* **1978**, *17*, 779. Neither a sample of natural lirionol nor spectra of it or any derivatives could be obtained.
(2) Rodrigo, R. *J. Org. Chem.* **1980**, *45*, 4538.
(3) Rajapaksa, D.; Rodrigo, R. *J. Am. Chem. Soc.* **1981**, *104*, 4725.
(4) de Silva, S. O.; Watanabe, M.; Snieckus, V. *J. Org. Chem.* **1979**, *44*, 4802.
(5) Keay, B. A.; Plaumann, H. P.; Rajapaksa, D.; Rodrigo, R. *Can. J. Chem.* **1983**, *61*, 1977.
(6) Rylander, P. N. "Catalytic Hydrogenation in Organic Synthesis"; Academic Press: New York, 1979.

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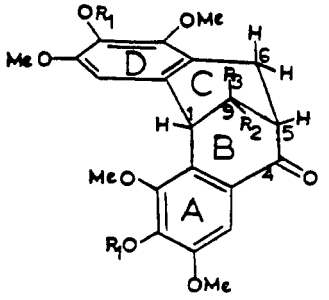
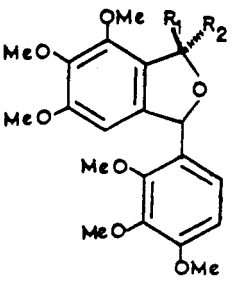
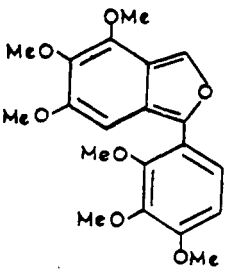
Chart I



1 R_1=R_2=H, R_3=CH_2OH
2 R_1=R_3=H, R_2=CH_2OH
11 R_1=Me, R_2=CO_2Me, R_3=H
14 R_1=Me, R_2=H, R_3=CO_2Me
16 R_1=Ac, R_2=CH_2OH, R_3=H
17 R_1=Ac, R_2=CH_2OAc, R_3=H
18 R_1=Ac, R_2=H, R_3=CH_2OAc



3 R_1+R_2=O
4 R_1,R_2=H, OH



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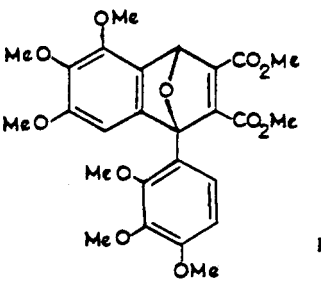
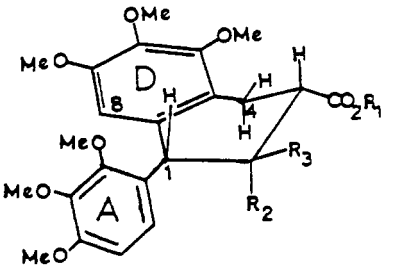
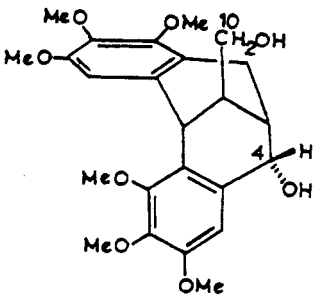


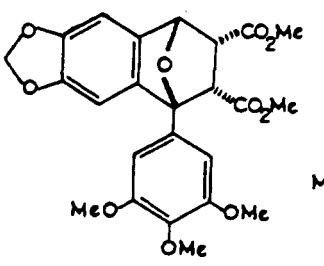
6



7 R_1=Me, R_2=CO_2Me, R_3=H
10 R_1=R_3=H, R_2=CO_2Me
12 R_1=R_2=H, R_3=CO_2Me



15



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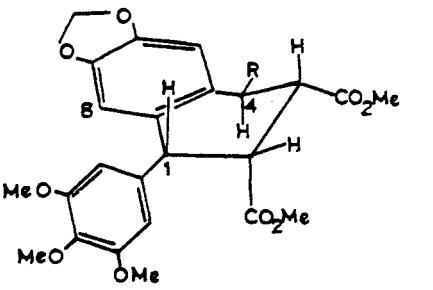

8 R = OH
13 R = H

and will report our results later. The observed coupling constants [250-MHz ^{1}H NMR, aided by decoupling], $J_{1,2}$ = 6.4 Hz, $J_{2,3}$ = 3.4 Hz, $J_{3,4_e}$ = 5.7 Hz, and $J_{3,4_a}$ = 12.3 Hz define the all-cis configuration and 1,3-diequatorial and 2-axial conformation shown for **7**. In addition H-8 is found at δ 6.22 shielded by the equatorial 1-aryl substituent, and the methyl resonance of the 2-axial carbomethoxy group appears at unusually high field [δ 3.27 (3 H, s)] shielded by aromatic rings A and/or D. Very similar chemical shifts and coupling constants were observed for **8**, obtained in our laboratory[7] from the Raney nickel hydrogenolysis of the bicyclo system **9**. Since such hydrogenolyses usually proceed with retention of stereochemistry,[2,6] the configurations and conformations of **7** and **8** appear to be secure. Selective hydrolysis of the 3-equatorial ester (2 N HCl, aqueous THF, 18 h at reflux) provided the acid **10** (67%) whose ^{1}H NMR spectrum lacked a 3-proton singlet at δ 3.74 but was otherwise identical with that of the diester **7**. Cyclization with TFA–TFAA proceeded in 65% yield to the bicyclononadienone **11**, whose structure and stereochemistry were established by X-ray analysis,[8] thus providing unequivocal confirmation of the structure and configuration of **7** and **10**. Epimerization of this compound at C-9 with methoxide in methanol was not successful, but C-9 deuteration could be effected under the same conditions. We therefore returned to the precursor **10** and achieved the desired epimerization of the C-2 axial ester

(7) Forsey, S. P.; Rodrigo, R., unpublished data. **8**: ^{1}H NMR ($CDCl_3$, 250 MHz) δ [$J_{1,2}$ = 5.8 Hz, $J_{2,3}$ = 3.9 Hz, $J_{3,4_a}$ = 9.7 Hz] 6.38 (s, 1 H, H-8) and 3.36 (s, 3 H, 2 axial CO_2Me).

(8) This X-ray structure was determined by Dr. N. J. Taylor; details will be published later.

in this compound by use of the same reagent to obtain **12** whose ^{1}H NMR spectrum reflected the all-trans all-equatorial stereochemistry. The high-field ester signal at δ 3.28 in **10** was now found downfield among the singlets for the seven methoxy groups and could not be identified, and the H-8 aromatic proton produced a singlet at δ 6.03 and H-1 a doublet at δ 4.3 ($J_{1,2}$ = 10.6 Hz, indicative of a diaxial coupling); H-2 and H-4$_e$ formed an overlapping multiplet comprised of a triplet (H-2) at δ 3.31 and a quartet (H-4$_e$) centered at δ 3.37; H-3 appears as split triplet at δ 3.15 and H-4$_a$ as a clean quartet at δ 2.83. Approximate coupling constants could be obtained by first order analysis aided by decoupling as follows: $J_{2,3}$ = 11 Hz, $J_{3,4_a}$ = 12 Hz, $J_{3,4_e}$ = 4.8 Hz, and J_{gem} = 16.1 Hz. The synthesis and stereochemistry of 1-aryl tetralins have attracted some recent attention,[9,10] and our results are in excellent agreement with data for some all-trans trisubstituted cases[10] but differ from the ^{1}H NMR properties reported[9] for the all-cis tetralin **13**.

Cyclization of **12** (TFA–TFAA) produced the bicyclononadienone **14**, whose structure and stereochemistry was confirmed by C-9 epimerization to **11** with methoxide.[11] Reduction of **14** with DIBAL-H in methylene chloride proceeded to a single diol **15**, which was selectively oxidized at the C-4 benzylic alcohol moiety with bis(trinitrocerium) chromate (BTNCC)[12] in refluxing benzene, and the synthesis of **1** was completed by selective demethylation of the central methoxyl groups of rings A and D with aluminum chloride in methylene chloride. We found, to our dismay, that the ^{1}H NMR spectra of synthetic **1** and its triacetate **18** were considerably different from the published data[1] for lirionol and its triacetate. The protons of the CH_2O group (C-10) in particular did not display geminal coupling and appeared merely as a doublet equally coupled to H-9 (Table I). Acting on the belief that the C-9 stereochemistry of natural lirionol had been incorrectly assigned we carried out the same sequence of reactions on the bicyclononadienone **11** to obtain the C-9 epimer **2** whose 250-MHz ^{1}H NMR spectrum showed large but consistent chemical shift differences from published data[1] but this time the C-10 protons were geminally coupled and did appear as two quartets with coupling constants similar to those obtained for the natural lignan (Table I). The diacetate **16** and triacetate **17** of **2** were also prepared and their spectra in both $CDCl_3$ and C_6D_6 were a near-perfect match of the data reported[1] for the natural counterparts. The small long-range coupling ($^4J_{1,5}$ = 1.6 Hz) reported for natural lirionol and its acetates could not be accurately obtained from our spectra of the synthetic compounds. A small coupling (W-pathway) undoubtedly exists but it is ≤0.5 Hz and could not be confidently measured in **1**, **2**, or **16–18** although we did find $^4J_{1,5} \approx 2$ and 1.7 Hz in **11** and **14**, respectively. Our synthesis thus provides[13] (±)-lirionol in ten steps from 2,3,4-trimethoxybenzamide in 4.5% overall yield and establishes its structure and stereochemistry as **2**.

Table I. Chemical Shifts (δ) and Coupling Constants (Hz) for Lirionol and Derivatives

compound	Ar H	H-1	H-5	H6-α	H-6β	H-9	10-CH$_2$O	OMe	OAc
2, synthetic [Me_2SO, D_2O]	6.74, 7.11	4.35 ($J_{1,9}$ = 2.6)	2.86 ($J_{5,6\beta}$ = 7.3)	2.73 (J_{gem} = 17.3)	3.0 ($J_{5,6\beta}$ = 7.3, J_{gem} = 17.3)	2.42 ($J_{1,9}$ = 2.6)	3.28 (J_{gem} = 10.5, $J_{9,10A}$ = 8.7, 3.38, J_{gem} = 10.5, $J_{9,10A}$ = 8.7)	3.63, 3.72, 3.75, 3.89	
natural lirionol[a] [Me_2SO, D_2O]	7.11, 7.5	4.7 ($J_{1,9}$ = 3.0, $J_{1,5}$ = 1.6)		3.1–3.2		2.75 (m)	3.16 (J_{gem} = 11.6, $J_{9,10A}$ = 8.0, 3.34, J_{gem} = 11.6, $J_{9,10B}$ = 7)	3.96, 4.04, 4.09, 4.23	
1, synthetic [Me_2SO, D_2O]	6.72, 7.15	4.27		2.75–3.0		obscured by DMSO at 2.52	3.37 (d, $J_{9,10}$ = 7.2)	3.61, 3.72, 3.77, 3.93	
16, synthetic [$CDCl_3$]	6.85, 7.34	4.54 ($J_{1,9}$ = 2.9)		2.93–3.18		2.64 (m)	3.53 (J_{gem} = 10.2, $J_{9,10A}$ = 9, 3.69, J_{gem} = 10.2, $J_{9,10B}$ = 7)	3.72, 3.76, 3.81, 3.97	2.31, 2.37
diacetate, natural[a]	6.85, 7.34	4.53 ($J_{1,9}$ = 3.0, $J_{1,5}$ = 1.6)		3.03		2.76	3.53 (J_{gem} = 11.6, $J_{9,10A}$ = 8, 3.66 (obscured))	3.69, 3.73, 3.79, 3.96	2.26, 2.32
17, synthetic [$CDCl_3$]	6.84, 7.35	4.43 ($J_{1,9}$ = 2.9)		2.95–3.16		2.76	3.98 (J_{gem} = 11.3, $J_{9,10A}$ = 8.3, 4.20, J_{gem} = 11.3, $J_{9,10B}$ = 6.8)	3.72, 3.77, 3.82, 3.96	2.03, 2.31, 2.37
triacetate, natural[a]	6.82, 7.34	4.43 ($J_{1,9}$ = 3.0, $J_{1,5}$ = 1.6)		3.05		2.77	4.0 (m), 4.2 (J_{gem} = 11.6, $J_{9,10}$ = 7)	3.68, 3.72, 3.78, 3.92	1.99, 2.26, 2.32
18, synthetic [$CDCl_3$]	6.80, 7.36	4.34		2.8–3.2			4.10 (d, $J_{9,10}$ = 7.6)	3.72, 3.78, 3.83, 4.0	2.07, 2.30, 2.37

[a] Obtained from ref 1. These spectra were run at 100 MHz, all others at 250 MHz.

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(10) Charlton, J. L.; Durst, T. *Tetrahedron Lett.* **1984**, 5287.

(11) The epimeric compounds **11** and **14** can be distinguished in their 250-MHz ^{1}H NMR spectra by the anisotropic effect of the C-9 carbomethoxy group on the benzylic H-6 protons. In **11** both H-6 protons are coincident at δ 3.07, but only H-6β is coupled to H-5 ($J_{5,6\beta}$ = 4.2 Hz). In **14** where the 9-carbomethoxy group is syn to C-6, H-6α is a doublet at δ 2.8 with J_{gem} = 18.6 Hz and H-6β a quartet at δ 3.1 ($J_{5,6\beta}$ = 7.7 Hz).

(12) Firouzabadi, H.; Iranpoor, N.; Parham, H.; Tootan, J. *Synthetic Commun.* **1984**, *14*, 631.

(13) All intermediates provided spectroscopic and/or analytical data consistent with their respective structures. Full details will be published later.

Acknowledgment. We thank the Natural Sciences and Engineering Research Council of Canada for support of this work.

Supplementary Material Available: Complete experimental details (7 pages). Ordering information is given on any current masthead page.

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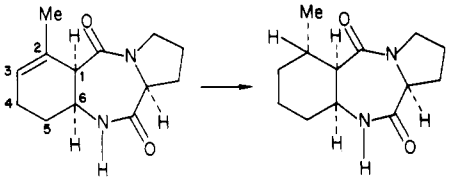
Carboxamide and Carbalkoxy Group Directed Stereoselective Iridium-Catalyzed Homogeneous Olefin Hydrogenations

Summary: Carboxamide and carbalkoxy substituents are capable of directing the stereochemical course of homogeneous $[Ir(cod)py(PCy_3)]PF_6/CH_2Cl_2$-catalyzed hydrogenation (1 atm) of cyclohexenes.

Sir: In studies directed at total syntheses of the pumiliotoxins,[1] we desired a stereoselective method for effecting the conversion of **1** into **2**. Hydrogenation of **1** under



1 **2**, >99:1(1:9)

heterogeneous conditions with 5% palladium on carbon gave an unfavorable 1:9 ratio of **2** and its diastereoisomer, presumably as a result of steric approach control. Indeed, molecular models of **1** show that the tertiary amide carbonyl group very effectively shields the β-face of the C(2)–C(3) double bond.

We then considered the possibility of directing the course of the hydrogenation of **1** by catalyst coordination with the amide carbonyl group. Support for this proposition came from the work of Halpern and co-workers concerning the mechanism of homogeneous rhodium-catalyzed hydrogenations of α-(acylamino)acrylic acid derivatives.[2] Furthermore, several research groups have demonstrated impressive stereochemical control by hydroxyl group coordination with rhodium and iridium catalyst systems.[3] We now report that excellent stereochemical control can be obtained by hydrogenation of **1**, and related olefins (Table I), with the catalyst system $[Ir(cod)py(PCy_3)]PF_6/CH_2Cl_2$ described by Crabtree and co-workers.[4]

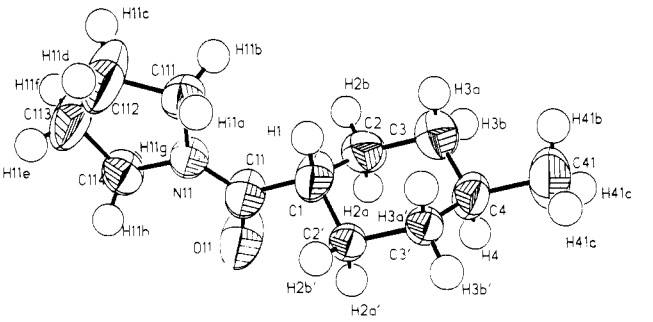


Figure 1. Molecular structure of **4**.

Hydrogenation of **1**[5] in CH_2Cl_2 with ~5 mol % of the iridium catalyst at atmospheric pressure gives **2** with better than 99:1 diastereoselectivity in quantitative yield.[6] Stereochemical configuration of the cyclohexane ring in **2** was determined by conversion to a derivative of an intermediate in the Overman synthesis of *dl*-pumiliotoxin C[7] and to the enantiomer of natural pumiliotoxin C.[8]

Table I shows that the carboxamide group is a superior stereocontrol agent for the iridium-catalyzed hydrogenation of cyclohexene rings. Also included in the table are product ratios for the hydrogenation of each substrate with palladium on carbon. Conversion of **3** to **4** is highly stereoselective with the iridium catalyst (130:1) but is nearly stereorandom with palladium on carbon. Stereochemistry in **4** has been established by single-crystal X-ray structure determination.[9] The molecular structure of **4** is shown in Figure 1. This X-ray diffraction analysis coupled with chemical interconversions and spectroscopic comparisons provides unambiguous stereochemical assignments within the product series **4**, **6**, **8**, and **10** (vide infra).

Iridium-catalyzed hydrogenation of the methyl ester analogue of **3** occurs with decreased diastereoselectivity (**5** → **6**; 41:1).[10] Extending the distance of the amide carbonyl group from the olefinic center by one methylene unit results in negligible erosion of the stereoselectivity of hydrogenation (e.g., **7** → **8**; >100:1), but with the methyl ester analogue **9a** conversion to **10a** is stereorandom.

The absence of stereoselectivity in hydrogenations of olefinic ester **9a** is consistent with Stork's observation[3c] that hydrogenation of acetate derivatives of homoallylic alcohols with structures similar to **9a** (e.g., **9b**) proceeds with essentially no selectivity under the homogeneous iridium conditions. These reactivity patterns must be a result of more effective coordination between the amide carbonyl group and iridium than is obtainable with the ester carbonyl group. Interestingly, the nitrile analogue **9c** failed to undergo hydrogenation with the iridium catalyst.[11]

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(5) Heterocycle **1** is obtained in enantiomerically pure form by a modification of the Birch reduction–alkylation method described by: Schultz, A. G.; McCloskey, P. J.; Sundararaman, P. *Tetrahedron Lett.* **1985**, *26*, 1619.
(6) After our work was nearly completed, a report concerning the stereochemistry of rhodium- and iridium-catalyzed hydrogenation of several cyclohexenecarboxylic acids and their esters appeared; see: Brown, J. M.; Hall, S. A. *J. Organomet. Chem.* **1985**, *285*, 333.
(7) Overman, L. E.; Jessup, P. J. *J. Am. Chem. Soc.* **1978**, *100*, 5179.
(8) Schultz, A. G.; McCloskey, P. J., manuscript in preparation.
(9) Suitable crystals of **4** (mp 97–98 °C) for X-ray diffraction studies were obtained from ethyl acetate solution.
(10) Brown and Hall report[6] that iridium-catalyzed hydrogenation of **5** gives **6** "in excess of 90%". These workers do not indicate how product stereochemistry was determined. Our assignment rests on chemical interconversions between **4** and **6**.

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