Table I. 1:1 Metal Complexation Constants (log K)

metal ion (ionic diamtr, Å)	1[a,c]	2[a,d]	15-crown-5[b,e]	18-crown-6[b,f]
Mg²⁺ (1.30)	3.3	3.1	noncomplxtn	unreported
Ca²⁺ (1.98)	2.9	2.9	2.1	3.9
Sr²⁺ (2.26)	2.3	2.6	2.6	>5.5
Ba²⁺ (2.70)	1.6	2.4	unreported	7.0

[a] $K = $ [M²⁺-complex]/[M²⁺][H₋₁L] (M⁻¹) in EtOH at 25 °C. Standard deviation is ±0.1. [b] $K = $ [M²⁺-L]/[M²⁺][L] (M⁻¹) in MeOH at 25 °C (Izatt, R. M.; Bradshaw, J. S.; Nielsen, S. A.; Lamb, J. D.; Christensen, J. J. *Chem. Rev.* **1985**, *85*, 271–339). [c] Cavity size (1.4–2.0 Å). [d] Cavity size (1.8–2.2 Å). [e] Cavity size (1.7–2.2 Å). [f] Cavity size (2.6–3.2 Å).

trophotometrically), <2, and <1.[14] The phenol pK_a value of 4.8 is much lower than 8.8 of *p*-bromophenol alone, presumably under the strong influence of +3 charge of the N₅ macrocycle. We could isolate crystalline triprotonated (on N₅) phenolate species **7** (as 2ClO₄⁻ salt) out of pH 7 aqueous solution containing excess NaClO₄. Its UV spectrum (in H₂O) of λ_{max} 309 nm (ϵ 3900) supports the phenolate form of **7**. On the other hand, the ratio of the phenolate species is minor (1:0.25) with tetraamine homologue **1** in EtOH. Its pK_a values in 0.1 M Et₄NClO₄ are 10.7, 10.5, 8.3 (phenol), 4.2, and 3.7. The phenol pK_a of 8.3 is not as low as 4.8 of **2**, under the lesser influence of +2 charge of the N₄ macrocycle. For the sake of comparison, pK_a values of the relevant phenol macrocycles are 11.8, 10.5, 8.9 (phenol), <2, and <2 for **5a**,[10] 11.8, 10.4, 6.4 (phenol), <2, and <2 for **5b**,[11] 10.8 for **3a**, and 10.6 for **4a**.[5] Apparently, self dissociation of the phenol protons should be negligible with **3** and **4** in EtOH[5] and with **5a** in CHCl₃ and EtOH.[11] Thus, an enormous influence of the azacrowns on reactivity of the intraannular phenol protons is revealed.

The phenol ionization in EtOH is further promoted by addition of *neutral alkaline earth metal salts* MgCl₂, CaCl₂, SrCl₂, and Ba(SCN)₂, as illustrated by the increase in the phenolate form or accompanying decrease in the phenol form in the UV absorptions (see Figure 1b,c).[15] In varying the [ligand]/[M²⁺] ratio ([ligand] + [M²⁺] = 1 mM), the total phenolate concentration reaches maximum at [ligand]/[M²⁺] = 1 with all the metals. The degree of 1:1 association uniformly varies with the size of metal ions, i.e., Mg > Ca > Sr > Ba, for **1** and **2**. Monovalent alkaline metal salts (LiCl, NaCl, KCl, CsCl), on the other hand, do not dispel the phenolate protons of **1** and **2**. With crown homologues **3** and **4** basic conditions are needed to displace the phenol protons for M⁺ and M²⁺.[5–9] The phenol-pendant N₄ homologues **5a** and **5b** do not interact with alkaline earth metal salts in EtOH solution.[11] Evidently, *synergism between the macrocyclic N donors and intraannular phenolate donor is essential for uptake of divalent metal ions in the nonbasic conditions*. The pH metric titration indicates the complexation of **2** with Mg²⁺ to occur above pH 9 in aqueous solution (at 25 °C, I = 0.1 M Et₄NClO₄).

The apparent constants for the 1:1 complexation between **1** (or **2**) and M²⁺ in EtOH are calculable in terms of $K = $ [M²⁺-complex]/[M²⁺][H₋₁L] (M⁻¹) (where H₋₁L is a free ligand in phenolate anion form, and [M²⁺-complex] is the [total phenolate anion] – [H₋₁L]) with use of the UV spectral data. The results

1

8

(for M=Sr,Ba)
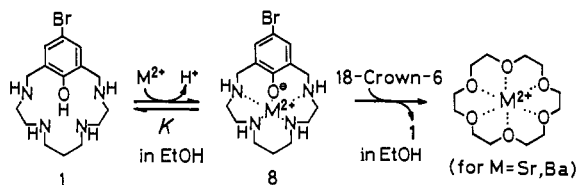
(14) The standard deviation for pK_a values of **1** and **2** is ±0.05. The titration curves of **1** and **2** in 0.1 M Et₄NClO₄ completely overlap those in 0.1 M NaClO₄. These facts indicate that Na⁺ has no interaction with **1** and **2**.

(15) With **3** and **4**, M⁺ and M²⁺ ion size correlate linearly with wave numbers of the phenolate absorption maxima, suggesting some degree of ion pairing in nonaqueous solvents.[5–9] On the other hand, the absorption maxima (300 nm) remain practically the same regardless of M²⁺ with **1** and **2**.

are summarized in Table I, in comparison with reported K values for crown ether complexes.[16] Very interestingly, Mg²⁺ uptake is more favored with smaller macrocyclic **1** than with larger **2**, despite the fact that the self dissociation of the former phenol hydrogen is less feasible. As the M²⁺ size increases, complexation becomes more favorable with larger macro ligand **2**. These size effects suggest that the metal inclusion into the size-fitted polyamine hole is a determining factor for the selective uptake. It is to be emphasized that the macrocyclic polyamine ligands are most appropriate for the smallest Mg²⁺, while crown ethers favor larger M²⁺.

Furthermore, in EtOH the complexation of Mg²⁺ and Ca²⁺ with **1** or **2** is proved stronger than with 15-crown-5 or 18-crown-6,[17] while that of Sr²⁺ and Ba²⁺ with **1** or **2** proved to be weaker than with 18-crown-6. This is revealed by the change of the phenolate UV absorptions upon addition of crown ethers (1 equiv) to a 1:1 mixture of **1** (or **2**) and MX₂ in EtOH.

Finally, the 1:1 complexation of **1** with Mg²⁺ was proven by isolation of its monoperchlorate salt, Mg²⁺-H₋₁L·ClO₄·H₂O (as light yellow powder), out of aqueous EtOH solution. The structure was characterized by elemental analysis (C, H, N), UV spectra (λ_{max} 301 nm, ϵ 3800 in EtOH at 25 °C), and analysis of Mg by atomic absorption technique.

The present results have thus revealed new potentials of macrocyclic polyamines for selective receptors of hard metal ions.

(16) The 1:2 complexation is reported for M²⁺ and **3b**.[8,9] However, the stoichiometry with **4**, and the complexation constants are unknown.

(17) Despite the fact that the reported log K value (3.9) for 18-crown-6 (in MeOH) is greater than 2.9 for **1** and **2** in EtOH, see Table I.

Enantioface Differentiation in Cis Dihydroxylation of C–C Double Bonds by Osmium Tetroxide with Use of a Chiral Diamine with D_2 Symmetry

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Molecules with a high symmetry have been shown to be effective auxiliary ligands in asymmetric reactions.[1,2] Here we describe extremely efficient enantioface differentiation in the dihydroxylation of C–C double bonds by osmium tetroxide using a chiral diamine with D_2 symmetry.[2] Virtually complete asymmetric induction was observed in the reaction of *trans*-1-phenylpropene.

We used the D_2 symmetric chiral diamine **1**, which consists of two *trans*-3,4-diphenylpyrrolidine units with C_2 symmetry at the both ends of the ethylene chain.[3] A typical experimental pro-

(1) C_2 symmetric ligands follow. Noyori, R.; Tomino, I.; Tanimoto, Y. *J. Am. Chem. Soc.* **1979**, *101*, 3129. Katsuki, T.; Sharpless, K. B. *J. Am. Chem. Soc.* **1980**, *102*, 5974. Fujiwara, J.; Fukutani, Y.; Hasegawa, M.; Maruoka, K.; Yamamoto, H. *J. Am. Chem. Soc.* **1984**, *106*, 5004. Choi, V. M. F.; Elliot, J. D.; Johnson, W. S. *Tetrahedron Lett.* **1984**, *25*, 591. Ito, Y.; Katsuki, T.; Yamaguchi, M. *Tetrahedron Lett.* **1985**, *26*, 4643. Masamune, S.; Kim, B.-M.; Petersen, J. S.; Sato, T.; Veenstra, S. J.; Imai, T. *J. Am. Chem. Soc.* **1985**, *107*, 4549. Noyori, R.; Ohta, M.; Hsiao, Yi.; Kimura, M.; Ohta, T.; Takaya, H. *J. Am. Chem. Soc.* **1986**, *108*, 7117. Kelly, T. R.; Whiting, A.; Chandrakumar, N. S. *J. Am. Chem. Soc.* **1986**, *108*, 3510. Narasaka, K.; Inoue, M.; Yamada, T. *Chem. Lett.* **1986**, 1967.

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Communications to the Editor

Table I. Enantioselective Cis Dihydroxylation of Olefins with OsO$_4$ Using (-)- and (+)-1[a]

entry	1	olefin	diol product			
			yield[b] (%)	[α]$_D$ ° (solvent)	ee[c] (%)	confgn
1	(-)	Ph⌒⫽	71	+57.3 (CDCl$_3$)[d]	90	*S*
2	(-)	Ph⌒⫽Me	73	+31.1 (EtOH)[e]	99	*SS*
3	(-)	Ph⌒⫽Ph	85	-88.5 (EtOH)[f]	97	*SS*
4	(+)	Ph⌒⫽Ph	71	+87.4 (EtOH)[f]	96	*RR*
5	(-)	Et⌒⫽Et	80	-20.4 (H$_2$O)[g]	90	*SS*
6	(-)	MeO$_2$C⌒⫽CO$_2$Me	67	+17.3 (H$_2$O)[h]	93	*RR*
7	(-)	cyclohexenyl-Ph	83	-16.1 (PhH)[i]	83	*SS*

[a] The reaction was carried out according to the typical reaction procedure described in the text, except entry 6 (treated with aqueous NaHSO$_3$, instead of LiAlH$_4$ reduction). [b] Isolated yield. [c] Determined by comparison of the magnitude of optical rotation with the highest reported value and confirmed by NMR analysis of the corresponding MTPA ester. [d] (*R*) [α]$^{25.5}_D$ -63.7° (CDCl$_3$): Dale, J. A.; Mosher, H. S. *J. Org. Chem.* **1970**, *35*, 4002. [e] See ref 5. [f] (1*R*,2*R*) [α]$^{21}_D$ +91.0° (EtOH): Berti, G.; Bottari, F. *J. Org. Chem.* **1960**, *25*, 1286. [g] (3*R*,4*R*) [α]$^{25}_D$ +22.7° (H$_2$O): Cope, A. C.; Shen, T. Y. *J. Am. Chem. Soc.* **1956**, *78*, 5916. [h] (2*R*,3*R*) [α]$^{20}_D$ +18.65° (H$_2$O): Buchaman, C. *J. Chem. Soc.* **1937**, 581. [i] (1*S*,2*S*) [α]$^{25}_D$ -19.4° (PhH): Berti, G.; Macchia, B.; Macchia, F.; Monti, L. *J. Chem. Soc. C* **1971**, 3371.

cedure is exemplified in the reaction of *trans*-1-phenylpropene. To a cooled (-78 °C) solution of the chiral diamine ((-)-1, [α]$^{20}_D$ -143° (chloroform)) (180 mg, 0.38 mmol) in THF[4] (10 mL) was added a solution of osmium tetroxide (88 mg, 0.35 mmol) in THF (2 mL). The resulting bright wine-red solution was cooled to -110 °C. A solution of *trans*-1-phenylpropene (38 mg, 0.32 mmol) in THF (1 mL) was added to the solution above, and the whole was stirred for 6 h at -110 °C. Lithium aluminum hydride (100 mg, 2.6 mmol) was added, and the mixture was stirred overnight at room temperature. After the addition of water, the mixture was filtered. After concentration of the filtrate, ether (10 mL) and 10% aqueous HCl (10 mL) were added to the residue. Filtration produced the hydrochloride of (-)-1 in 90% yield for reuse without

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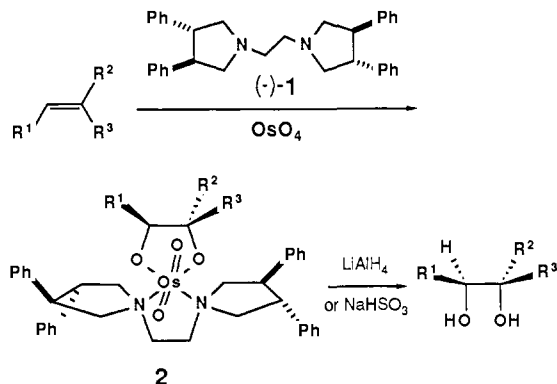
any loss of optical purity. The filtrate was diluted with ethyl acetate and washed with brine. Concentration followed by silica gel chromatography afforded the essentially optically pure (1*S*,2*S*)-1-phenylpropane-1,2-diol (36 mg, 73% yield). The enantiomeric excess (ee) of the diol produced was determined to be greater than 99% by optical rotation ([α]$^{20}_D$ +31.1° (*c* 1.79, EtOH))[5] and further determined by ^{1}H and ^{19}F NMR of the corresponding MTPA[6] diester.[7]

Additional examples are given in Table I. Thus the hydroxylation employing 1 as a chiral ligand gave exceptionally high

optical yields in the production of diols from mono-, *trans*-di-, and trisubstituted olefins.[8] It is also noteworthy that the enantioface selection in the present reaction is shown by the general presentation **3**. Since both (+)- and (-)-1 are readily accessible in

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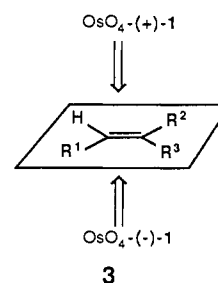
optically pure form,[3] this method allows the synthesis of both enantiomers of diols with predictable absolute configuration from olefins. The oxidation of *cis* olefins did not give satisfactory optical yields.[9]

The present procedure is extremely effective in the enantioselective synthesis of biologically active compounds. Thus the oxidation of ester **4**[10] followed by triethylsilane ionic reduction

4   **5**

6
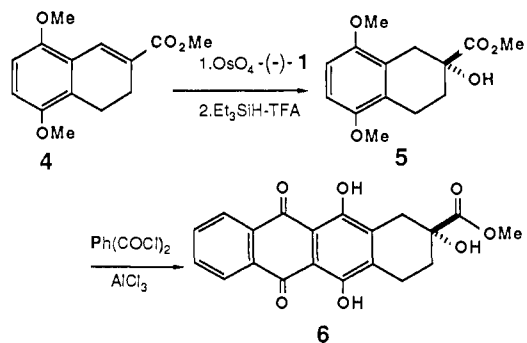
of the corresponding diol afforded **5**, a key intermediate for an-

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(4) THF was the solvent of choice in criteria of ee and yield at the reaction temperature of -78 °C. Solvents (% ee, yield): THF (94, 77); ether (95, 16); toluene (90, 86); methylene chloride (77, 53); DME (-, 0).

(5) Reported maximum value ([α]$^{20}_D$ +24.65° (EtOH) for (1*R*,2*R*)-enantiomer: Fisher, F. *Chem. Ber.* **1961**, *94*, 893) was too low. The optical purity was therefore determined by NMR analysis of the corresponding MTPA ester.

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(9) Dihydronaphthalene gave the diol in 26% ee. 3,5,5-Trimethyl- and 2-methylcyclohexenones were converted to the corresponding diols in 50 and 41% ee, respectively (see ref 8c).

(10) Treated the osmate ester with aqueous NaHSO$_3$ instead of LiAlH$_4$.

ticancer antibiotic anthracyclinones, in 89% yield and in 85% ee.[11] Friedel–Crafts reaction[12] of **5** with phthaloyl chloride followed by recrystallization of the product afforded nearly optically pure **6** ($[\alpha]^{20}_D$ -57.2° (CHCl$_3$))[11] in 68% yield.

The observed enantioface differentiation is kinetically controlled by the relative stabilities of the diastereomeric transition states and is not a result of thermodynamic control governed by the stability of the resulting osmate ester–diamine complex **2**.[13] Further studies are in progress to establish the scope and limitation and to elucidate mechanism of the enantioselective dihydroxylation of olefins by osmium tetroxide employing D_2 symmetric chiral diamine.[14]

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(13) The structure of the osmate ester (**2**, R^1, R^2 = Ph; R^3 = H) was firmly established by ^{1}H and ^{13}C NMR and further established by X-ray crystallography (We are grateful to Prof. Y. Iitaka for X-ray crystallography.). Treatment of excess *dl*-osmate ester (pyridine ligand) (4 equiv), prepared in pyridine, with (−)-**1** gave a nearly 1:1 mixture of two diastereomers. This ligand exchange reaction revealed the nearly equal thermodynamic stability of the diastereomeric osmate esters.

(14) The mechanism of osmium tetroxide oxidations of olefins has been the subject of focused discussion. Schröder, M. *Chem. Rev.* **1980**, *80*, 187. Sharpless, K. B.; Teranishi, A. Y.; Bäckvall, J.-E. *J. Am. Chem. Soc.* **1977**, *99*, 3120. Jørgensen, K. A.; Hoffmann, R. *J. Am. Chem. Soc.* **1986**, *108*, 1867.
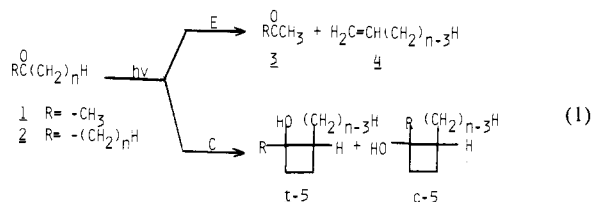
Control of Norrish II Reactions of 2- and *sym*-Alkanones by the Ordered Solid Phases of Heneicosane[1]

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Previously, we exploited the Norrish II reactions of ketones (eq 1) to assess the limitations of the influence of solvent order on solute dynamics.[2] We find that photolyses of 2- and *sym*-alka-

nones (**1** and **2**) provide sensitive monitors of local solvent order in the smectic B and solid phases of *n*-butyl stearate (BS)[3] and in the aqueous gel phases of potassium stearate, potassium palmitate, and 1/1 potassium stearate/1-octadecanol.[1,4] Each lipophilic solvent molecule consists of a polymethylene chain and a head group.

To discern how ordered polymethylene chains in the *absence* of a head group influence the Norrish II reactions, we have irradiated **1** and **2** in the isotropic and the two solid phases of

Table I. Norrish II Product Ratios from **1** and **2** in the Isotropic (45 °C), the Hexagonal Solid (35 °C; Phase II), and Orthorhombic Solid (25 °C; Phase I) Phases of Heneicosane

C chain length of ketones	T (°C)	1 E/C	1 t/c	2 E/C	2 t/c
15	45	3.3 ± 0.1	1.9 ± 0.2	1.5 ± 0.1	2.1 ± 0.1
	35	3.7 ± 0.3	2.4 ± 0.2	1.8 ± 0.4	1.8 ± 0.1
	25	16.2 ± 0.4	3.0 ± 0.1	2.6 ± 0.1	1.5 ± 0.1
17	45	3.3 ± 0.2	1.7 ± 0.4	1.4 ± 0.1	2.4 ± 0.1
	35	4.6 ± 0.7	3.4 ± 0.2	2.4 ± 0.1	5.9 ± 0.1
	25	15.0 ± 4.0	2.4 ± 0.4	5.0 ± 0.9	1.5 ± 0.7
21	45	2.4 ± 0.6	1.0 ± 0.1	1.8 ± 0.1	2.5 ± 0.1
	35	5.7 ± 1.3	2.5 ± 0.2	3.5 ± 0.1	24.9 ± 3.0
	25	46.0 ± 16.0	3.6 ± 0.9	69.0 ± 7.0	0.9 ± 0.4
23	45	3.1 ± 0.7	1.9 ± 0.2	2.0 ± 0.1	2.0 ± 0.2
	35	1.6 ± 0.3	2.4 ± 0.2	2.9 ± 0.1	10.8 ± 0.5
	25	3.6 ± 0.1	3.0 ± 0.1	8.1 ± 0.3	9.0 ± 0.8
25	45			2.3 ± 0.4	2.9 ± 0.3
	35			2.9 ± 0.5	18.0 ± 1.0
	25			4.3 ± 0.6	15.0 ± 0.7

heneicosane (C$_{21}$H$_{44}$). We find that these ordered *n*-alkane phases are more sensitive and selective toward size and shape changes of the ketones than are the analogous phases of BS and the aqueous gels. Above 40 °C, heneicosane is an isotropic liquid in which the alkane chains are weakly correlated with each other and with cylindrically shaped solutes.[5] From 40.2[6] to 32.5 °C, a solid phase (phase II) obtains.[7] The molecules are elongated (approaching *all-trans*) and are stacked hexagonally in layers. Gauche conformational defects are much more prevalent near the ends than in the middle of the chain.[8] Individual molecules rotate rapidly about their long axes, which are perpendicular to the layer planes. This phase bears a close resemblance to the liquid–crystalline smectic B phase of BS[9a] and the lipophilic layer organization of the aqueous gels.[9b]

Below 32.5 °C, another solid phase (phase I) forms in which the molecular rotational motions are quenched and the hexagonal arrangement within layers collapses to orthorhombic.[8] Very little deviation from the *all-trans* conformation is observed. The organizations of phase I and of a smectic E phase or the solid phases of BS[9a] are very similar.

In previous publications we have discussed in detail the mechanistic basis for solvent order induced product selectivity in Norrish II reactions.[2–4] In essence, the immediate precursors to the elimination products (**3** and **4**) and diastereomeric cyclization products (*t*-**5** and *c*-**5**) are different conformers of the same hydroxy 1,4-biradical. If solvent order enhances the population of one conformer over the others, its product will be formed preferentially. The basis for that preference may be related to the ease with which a particular intermediate may be incorporated within a solvent matrix (size and shape considerations) and/or to specific attractive interactions between functional groups on an intermediate molecule and neighboring solvent molecules (e.g., hydrogen bonding). Since the latter is not available to hydroxy 1,4-biradicals in heneicosane, size and shape considerations can be investigated without complications.

The resemblance of **1** and **2** to the shape of heneicosane leads us to conclude[1–4] that the ketones should be incorporated into the solvent layers with their long axes perpendicular to the layer plane.

(1) Part 27 in our series: Liquid-Crystalline Solvents as Mechanistic Probes. For part 26, see: Treanor, R. L.; Weiss, R. G. *J. Am. Chem. Soc.*, submitted for publication.

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