ELSEVIER

Journal of Molecular Catalysis A: Chemical 120 (1997) 101–108

JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL

Enantioselective catalytic epoxidation of nonfunctionalized prochiral olefins by dissymmetric chiral Schiff base complexes of Mn(III) and Ru(III) metal ions II

R.I. Kureshy *, N.H. Khan, S.H.R. Abdi, P. Iyer, A.K. Bhatt

Central Salt and Marine Chemicals Research Institute, Bhavnagar 364 002, India

Received 24 June 1996; accepted 22 October 1996

Abstract

A series of new dissymmetric chiral Schiff base complexes has been obtained by a systematic condensation of (1*S*,2*S*)(+)-diaminocyclohexane and 3-acetyl-4-hydroxy-6-methyl-2-pyrone with salicylaldehyde, 5-chloro-, 5-methoxy- and 5-nitrosalicylaldehyde and by subsequent metallation with manganese and ruthenium. The characterization of the complexes **1–8** was accomplished by physico chemical studies viz. microanalysis, IR-, UV/VIS-, and CD spectral studies, optical rotation, molar conductance measurements and cyclic voltammetry. Enantioselective epoxidation of non functionalised olefins, viz. *cis*-stilbene, *trans*-3-nonene and *trans*-4-octene with iodosyl benzene as oxidant was demonstrated in the presence of catalytic amounts of chiral Mn(III) and Ru(III) dissymmetric Schiff base complexes. Good optical yields of epoxides were obtained for the catalyst **4** with the substrates *trans*-3-nonene and *cis*-stilbene.

Keywords: Enantioselective epoxidation; Dissymmetric complexes; Schiff bases; *cis*-stilbene; *trans*-3 nonene; *trans*-4 octene; Manganese; Ruthenium

1. Introduction

Enantioselective epoxidation of olefins is one of the most important and challenging areas in organic synthesis as their chiral epoxides play an eminent role as drug intermediates and chiral building blocks in the synthesis of optically active complex molecules [1–4]. Much attention has been paid to enantioselective epoxidation of allylic alcohol by Sharpless [5,6] and this method led to the synthesis of a variety of different allylic epoxides of which many of them have been used for the synthesis of valuable compounds [7]. However, enantioselective epoxidation of prochiral non-functionalised olefins and several enzymatic reactions with terminal alkenes were achieved with great success [8–11]. Several examples have been reported using artificial metal porphyrin designed as cytochrome P-450 peroxidase [10,11], a synthetic metalloporphyrin [12,13] model system, some macrocyclic ligands [14,15] and Mn(III) salen [16–20] complexes together with terminal oxidants such as iodosyl benzene, sodium hypochlorite and hydrogen peroxide for asymmetric epoxidation of non-functionalised olefins. Jacobsen et al. [21–23] have reported the catalytic enantioselective epoxidation of non-functionalised alkenes,

* Corresponding author. Fax: +91-278-426970.

PII S1381-1169(96)00432-3

viz. *cis*-stilbene and trisubstituted alkenes using chiral Mn(III) salen complexes as catalyst, and this procedure led to *trans* epoxide preferentially.

In continuation of our earlier work on enantioselective epoxidation of prochiral non-functionalised olefins, viz. styrene, substituted styrenes and *cis*-stilbene, by Ru(II), Ru(III) and Mn(III) chiral Schiff base complexes [24–27], we are reporting here the synthesis and characterization of a series of Ru(III) and Mn(III) dissymmetric chiral Schiff base complexes derived from (1*S*,2*S*)(+)-cyclohexane diamine and 3-acetyl-4-hydroxy-6-methyl-2-pyrone with salicylaldehyde, 5-chloro-, 5-methoxy- and 5-nitrosalicylaldehyde in order to make efforts towards elucidation of the factors controlling *cis–trans* selectivity in epoxide formation, for an improvement in enantioselectivity by varying different electron withdrawing or releasing groups at the 5th position of the salicylaldehyde moiety. Such substituents are able to produce different electronic and steric effects, thereby tuning the catalytic efficiency.

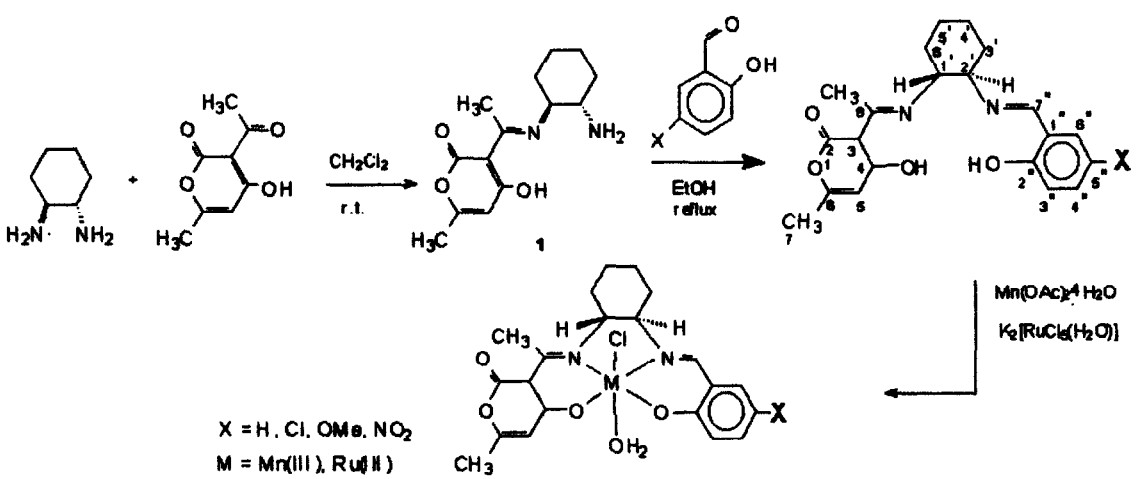
2. Experimental

$Mn(CH_3COO)_2 \cdot 4H_2O$ (Sisco), $RuCl_3 \cdot 3H_2O$ (Johnson and Mathey), salicylaldehyde, 4-chloro-, 5-methoxy-, and 5-nitrosalicylaldehyde, dehydroacetic acid, *cis*-stilbene, *trans*-3-nonene, *trans*-4-octene, (1*S*,2*S*)(+)-cyclohexane diamine and $Eu(hfc)_3$ (Aldrich) were used as such. The metal complex $K_2[RuCl_3(H_2O)]$ was prepared by the known method [28].

2.1. Synthesis of dissymmetric chiral Schiff bases

Synthesis of dissymmetric chiral Schiff bases was carried out with a little modification to the reported method. (1*S*,2*S*)(+)-cyclohexane diamine (0.01 mol) dissolved in dichloro methane was stirred at room temperature with 3-acetyl-4-hydroxy-6-methyl-2-pyrone (0.01 mol) in dichloromethane for 10 h (TLC checked). After the completion of reaction the solvent was removed on a rotaevaporator to get a light cream compound referred as to as **1** in Scheme 1. It was recrystallised twice by dichloromethane/methanol (1:1). Compound *1* (0.002 mol) and salicylaldehyde, 5-chloro-, 5-methoxy- and 5-nitrosalicylaldehyde (0.002 mol) in ethanol were refluxed for 6–8 h on a water bath (TLC checked). The solution was concentrated on a rotaevaporator and the desired ligands were precipitated by 40–60 petroleum ether.

The dissymmetric chiral Schiff bases (*S*,*S*(−)-DISSAL), (*S*,*S*(−)-DISSAL Cl),



Scheme 1.

(*S,S*(−)-DISSAL MeO) and (*S,S*(−)-DISSAL NO_2) were characterised by microanalysis, IR-, $^1H-$, $^{13}C^1H$-NMR spectroscopy as previously reported [27].

2.2. Preparation of Mn(III) dissymmetric chiral Schiff base complexes 1–4

Ethanolic solution of the chiral Schiff bases (0.001 mol) containing KOH 0.5M (4 ml) was allowed to reflux in inert atmosphere with Mn $(CH_3COO)_2 \cdot 4H_2O$ (0.002 mol) for 8 to 10 h. The reaction mixture was cooled to room temperature and 0.127 g of LiCl in minimum quantity of water was added. The resulting solution was filtered and concentrated in rotaevaporator. The residue was redissolved in dried dichloromethane filtered again and water was removed by a separating funnel and the solution was again concentrated till dryness. Recrystallization of the complexes was done in acetonitrile. The overall yield for all the complexes lies in the range 56–63%. The analytical data for the complexes is given below.

2.2.1. (S,S)(−)-DISSAL Mn (1)

Calcd. for $C_{21}H_{24}N_2O_5ClMn$: C, 53.12: H, 5.10: N, 5.90. Found: C, 54.12; H, 5.20; N, 5.96. IR (KBr) (cm^{-1}) 1575 ν(H–C=N), 3400 ν(OH) 1100, 1170 δ(OH); UV/VIS (nm) MeOH λ_{max} (ε), 330 (2400), 400 (1986); CD λ_{max} ($\Delta\varepsilon$) (MeOH) 360 (+5.4), 401 (+2), 470 (+1.4), 555 (−1.4); $[\alpha]_D^t = -9.64$; configuration (*S*); Λ_M (MeOH), 3 mho cm^{-1} mol^{-1}: μ_{eff} (BM) 4.80; $\Delta E_{pc} = -0.28$ V.

2.2.2. (S,S)(−)-DISSAL Cl Mn (2)

Calcd. for $C_{21}H_{23}N_2O_5Cl_2Mn$: C, 49.53; H, 4.55; N, 5.50. Found: C, 50.00; H, 4.60; N, 5.53. IR(KBr) cm^{-1} 1580 ν(H–C=N), 3400 ν(OH), 1100, 1170 (OH); UV/VIS (nm) (MeOH) λ_{max} (ε), 346 (2500), 380 (1533); CD: λ_{max} (MeOH) 340(−2), 440(+2.5), 550(−1.8); $[\alpha]_D^t = -13.60$; configuration (*S*); Λ_M (McOH) 3 mho cm^{-1} mol^{-1}; μ_{eff} (BM) 4.80; $\Delta E_{pc} = -0.19$ V.

2.3. (S,S)(−)-DISSAL MeO Mn (3)

Calcd. for $C_{22}H_{26}N_2O_6ClMn$: C, 52.34; H, 5.19; N, 5.54. Found: C, 52.30; H, 5.12; N, 5.50. IR(KBr) (cm^{-1}) 1575 ν(H–C=N), 3400 ν(OH), 1100, 1170 δ(OH); UV/VIS (nm) (MeOH) λ_{max} (ε), 367 (1012), 315 (2221), sh 439 (645); CD λ_{max} ($\Delta\varepsilon$) (MeOH) 370 (−1), 430 (+2.8), 600 (−1.3); $[\alpha]_D^t = -18.85$; configuration (*S*); Λ_M (MeOH) 4 mho cm^{-1} mol^{-1}, μ_{eff} (BM) 4.9; $\Delta E_{pc} = -0.50$ V.

2.3.1. (S,S)(−)-DISSAL NO_2 Mn (4)

Calcd. for $C_{21}H_{23}N_3O_7ClMn$: C, 48.53; H, 4.46, N, 8.08. Found: C, 48.50; H, 4.43; N, 8.03. IR(KBr) (cm^{-1}) 1585 ν(H–C=N), 3400 ν(OH) 1100, 1170 δ(OH); UV/VIS (nm) (MeOH) λ_{max} (ε), 335 (2400), 448 (2500); CD λ_{max} ($\Delta\varepsilon$) (MeOH) 415 (−2), 455 (+3), 540 (−4); $[\alpha]_D^t = -12.59$; configuration (*S*); Λ_M (MeOH) 4 mho cm^{-1} $nmol^{-1}$; μ_{eff} (BM) 4.85; $\Delta E_{pc} = -0.15$ V.

2.4. Methods

Microanalysis of the complexes was done in a Carlo Erba Analyzer Model 1106. Molar conductance was measured at room temperature on a Digisun Electronic Conductivity Bridge DI-909. The IR spectra were recorded on a Carl Zeiss Specord M-80 spectrophotometer in a KBr/nujol mull. Electronic spectra were recorded on Shimadzu a UV/visible recording spectrophotometer Model 160. ^1H NMR (99.55 MHz) and $^{13}C\{^1H\}$ NMR (24.99 MHz) were carried out on a Jeol FX100 NMR spectrophotometer in $CDCl_3$ and CH_2Cl_2. The magnetic moment measurements were done at 298°C by the Gouy method using $Hg[Co(SCN)_4]$ as calibrant and experimental susceptibilities were corrected for diamagnetism. Cyclic voltammetry, differential pulse voltammograms were recorded with a Princeton Applied Research (PAR) instrument using tetrabutyl ammonium fluoroborate as supporting electrolyte in acetonitrile. The optical rotation of the complexes in

methanol was measured by an Atago (Japan) polarimeter. The CD spectra were recorded in methanol by a Jasco Machine Model J-20, Japan. The purity of the solvent, the substrate and analysis of the product was determined by GLC using a Shimadzu GC 14B coupled with a PC using a 2 m long, 3 mm ID, 4 mm OD stainless steel column packed with SE30, 5% mesh size 60 to 80, with an FID detector. The column temperature was programmed between 70 to 170°C and an injection temperature of 200°C. Nitrogen served as carrier gas with a flow of 30 ml/min. Synthetic standards of the product were used to determine yields by comparison of peak height and area. The optical yield of the product was determined by ^{1}H NMR using Eu(hfc)$_3$ as chiral shift reagent.

2.5. Epoxidation of prochiral non functionalised alkenes by catalysts 1–8

Enantioselective epoxidation of *trans*-4-octene, *trans*-3-nonene and *cis*-stilbene by catalysts **1–8** was attempted in a homogenous system with iodosyl benzene by the following procedure: The chiral catalyst (0.02 mmol), *cis*-stilbene, *trans*-4-octene and *trans*-3-nonene (1 mmol), pyridine-*N*-oxide (0.24 mmol) and *n*-tridecane (0.1 mmol) as GLC internal standard were dissolved in 5 ml dichloromethane. The reaction was initiated by the addition of iodosyl benzene (1 mmol) and stirred at a constant speed in an inert atmosphere at 4°C. After each interval of 60 min an aliquot was taken from the reaction mixture quenched with PPh_3 and analysed by OLC. After completion of the reaction the solvent was removed and the product was separated by a short silica gel column (60–120 mesh) using hexane:dichloromethane as eluent. Evaluation of enantiomeric excess was done by ^{1}H NMR using NMR shift reagent Eu(hfc)$_3$.

3. Results and discussion

The new dissymmetric chiral Schiff base complexes **1–8** were isolated as neutral solids (Scheme 1) with the stoichiometry MLX(H_2O) where X = chloride, L = chiral dissymmetric tetradendate Schiff base derived from (1*S*,2*S*)(+)-diamino cyclohexane and 3-acetyl-4-hydroxy-6-methyl-2-pyrone with salicylaldehyde (DISSAL), 5-chloro-(DISSAL Cl), 5-methoxy-(DISSAL MeO), and 5-nitro-salicylaldehyde (DISSAL NO_2) and M = Mn(III) and Ru(III) metal ions. The magnetic moment of the complexes **1–4** lies in the range 4.80–4.90BM, which is consistent with a high spin d^4 system, while μ_{eff} values for complexes **5–8** are close to 1.90–2.04BM, indicating the presence of a Ru(III) ion with a spin paired $4d^5$ electronic configuration.

In the infrared region a strong band near 1620–1630 cm^{-1} of the chiral dissymmetric Schiff bases undergoes a modest decrease to lower wave numbers after complexation, inferring the involvement of an azomethine group in the coordination to the metal ions. A broad band centred at ca. 3400 cm^{-1} assigned to ν(OH) along with two deformation bands at 1100 and 1170 cm^{-1} to coordinated water [29].

The electronic spectra in methanol show the high intensity charge transfer band in the range 241 ($\varepsilon = 93840$) and 346 ($\varepsilon = 2500$) nm while the MLCT bands fall between 400 ($\varepsilon = 1986$) and 448 ($\varepsilon = 2264$) nm. The position of MLCT bands depend on the substituent [30] attached at the 5th position of the salicylaldehyde in the chiral Schiff base complexes and the energy of the band decreases in the order (*S*,*S*)(−)-DISSAL NO_2 > (*S*,*S*)(−)-DISSAL > (*S*,*S*)(−)-DISSAL Cl > (*S*,*S*)(−)-DISSAL MeO. In the case of complexes **5** to **8** one more band lies near 612 ($\varepsilon = 133$) and 689 ($\varepsilon = 171$) nm, assigned to the forbidden ligand field transition of Ru(III).

CD spectra of these complexes were measured in methanol. Interestingly, complexes **3** and **7**, although prepared from the same ligand, namely DISSAL MeO, showed mirror image CD spectra (Fig. 1). Complex **7** ((*S*,*S*)(−)-DISSAL MeORu) was stereospecifically coordinated to the metal so that the gauche ring was

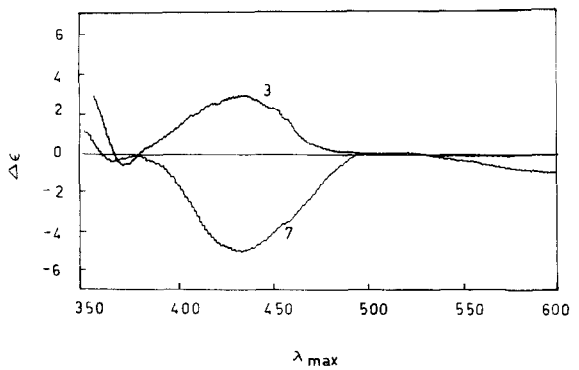


Fig. 1.

almost exclusively in the δ form whereas complex **3** ((*S*,*S*)(−)-DISSAL MeOMn) is located in the λ form with a little contribution from the δ form. A similar trend was also reported elsewhere [28,30] and it depends on the steric interaction between the substituents at the asymmetric centre and the size of the metal ions. In the ligand field region the CD bands near 550 (−1.4) and 670 (−0.6) nm are assigned to dd bands and spin forbidden ligand bands while $d \rightarrow \pi^*$ bands fall between 401 (+2) and 440 (−4.0) nm. The high intensity $\pi \rightarrow \pi^*$ transition lies between 333 (+2.8) and 370 (−1) nm.

The cyclic voltammogram of the complexes in acetronitrile is a one electron reduction process. For the complexes **1–4** the reduction potential of Mn(III)/Mn(II) falls in the range of −0.15 to −0.50 V while the Ru(III)/Ru(II) couple lies near −0.20 to −0.55 V for **5–8**. A positive shift is seen in the reduction potential when an electron withdrawing group is attached at the 5th position of the salicylaldehyde moiety of the ligand and the reverse is true for electron donating groups.

4. Enantioselective epoxidation

Mn(III) and Ru(III) dissymmetric chiral Schiff base complexes have been used as catalysts for enantioselective epoxidation of *cis*-stilbene, *trans*-4-octene and *trans*-3-nonene Table 1. Evaluation of the optical yield for the resulting epoxide separated by a short silica gel column (mesh 60–120) was carried out by ^{1}H NMR using chiral shift reagent Tris-[heptafluoropropyl hydroxy methylene] camphorato-(+)-Eu(III).

Table 1
Data for enantioselective epoxidation of *trans*-4-octene, *trans*-3-nonene and *cis*-stilbene by disymmetric chiral Schiff base complexes of Mn(III) and Ru(III)

Catalyst	Substrate	Reaction time (h)	% conversion to epoxide	ee (%) [a]
1/(**5**)	*t*-4-octene	24/(48)	36/(27)	12.6/(27.4)
	t-3-nonene	24/(48)	68/(35)	21.2/(33.3)
	cis-stilbene	24/(24)	30/(10)	
			cis/*trans* 30/70/(*cis*/*trans* 46/54)	*cis* 36, *trans* 1/(*cis* 33, *trans* 9)
2/(**6**)	*t*-4-octene	24/(48)	30/(13)	9.0/(23.07)
	t-3-nonene	24/(48)	70/(25)	16.6/(16.13)
	cis-stilbene	24/(24)	20/(20)	
			cis/*trans* 26/74/(*cis*/*trans* 44/56)	*cis* 22, *trans* 3/(*cis* 25, *trans* 1)
3/(**7**)	*t*-4-octene	24/(48)	35/(11)	23.0/(30)
	t-3-nonene	24/(48)	60/(18)	5.8/(18.18)
	cis-stilbene	24/(24)	30/(22)	
			cis/*trans* 16/84/(*cis*/*trans* 45/55)	*cis* 28.6, *trans* 5/(*cis* 20, *trans* 1)
4/(**8**)	*t*-4-octene	24/(48)	60/(28)	40.7/(33.3)
	t-3-nonene	24/(48)	84/(40)	54.0/(33.3)
	cis-stilbene	24/(24)	26/(25)	
			cis/*trans* 48/52/(*cis*/*trans* 48/52)	*cis* 50, *trans* 0/(*cis* 23, *trans* 1)

[a] Configuration not determined; figures within parentheses are for Ru(III) catalysts.

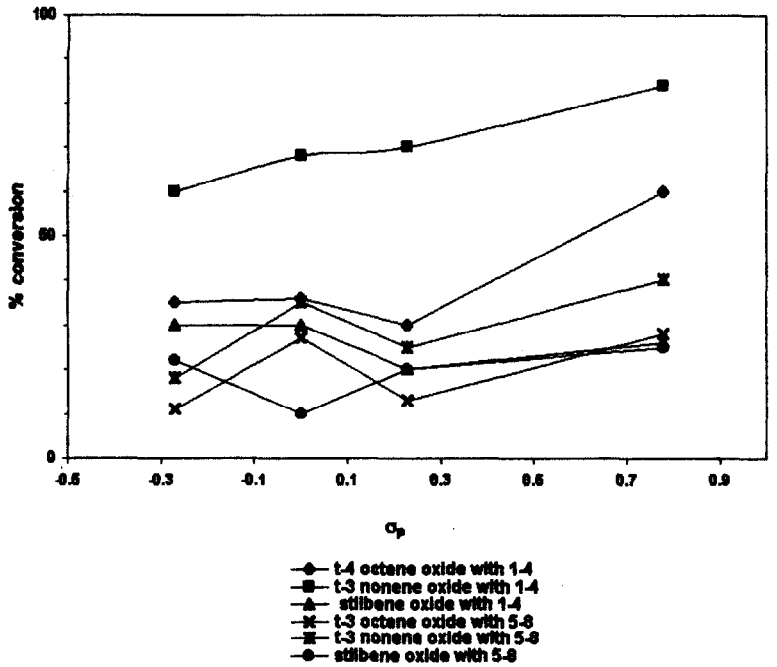


Fig. 2.

In the case of stilbene oxide, on addition of several equivalents of $Eu(hfc)_3$ the peaks for the *trans* isomer at 3.87 ppm and the *cis* isomer at 4.36 pm shifted downfield and splitted into two singlets at 4.22 and 4.25 ppm and 6.10 and 6.25 ppm, respectively. Relative intensities of the splitted peaks were used for calculating enantiomeric excesses. A broad triplet at approxi-

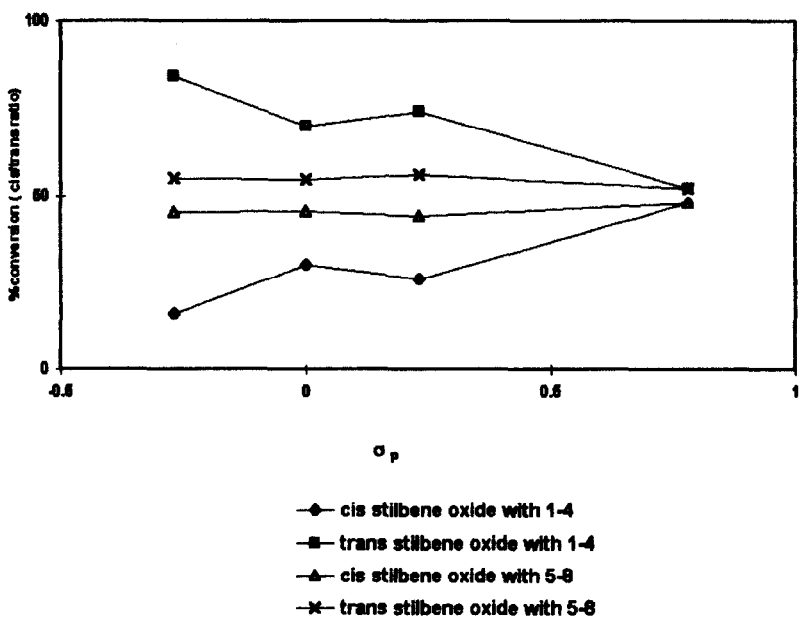


Fig. 3.

mately δ 2.6 ppm for *trans*-4-octene oxide and *trans*-3-nonene oxide shifted to 3.6–3.9 ppm and 4.78–5.03 as two sets of triplets on addition of several equivalents of $Eu(hfc)_3$, respectively, were used to evaluate enantiomeric excesses.

Overall chemical yields for respective epoxides obtained with Mn(III) catalysts **1–4** were better than those obtained by catalysts **5–8** with all the olefins studied here. Further, in case of *trans*-3-nonene there is a linear increase in % conversion to its epoxide when the catalyst bears more electron withdrawing groups on the ligand (Fig. 2). However, no such trend was obtained with other substrates but the nitro substituted catalyst gave better conversions. The *cis/trans* ratio for stilbene oxide was again better in case of the nitro catalyst and lowest in case of methoxy bearing complexes (Fig. 3). Therefore, one can conclude that any substituent at position 5 of the salicylaldehyde moiety of the catalyst does plays a distinctive role for these groups. Besides playing a crucial role in tuning electron density around the metal ion they can also determine the path of the incoming substrate. Thus, a nitro group facilitates the sidewise approach of electron-rich olefins [3,17]. These trends are also reflected in enantiomeric excesses of product oxides (Fig. 4). Moderate to good enantiomeric excesses in case of *cis*-stil-

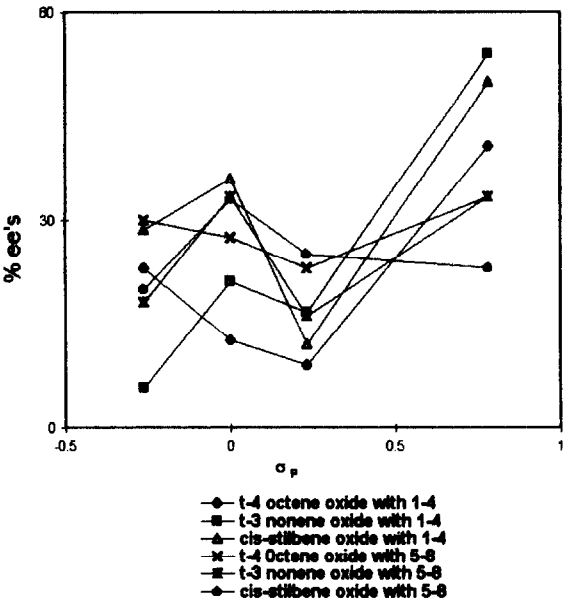
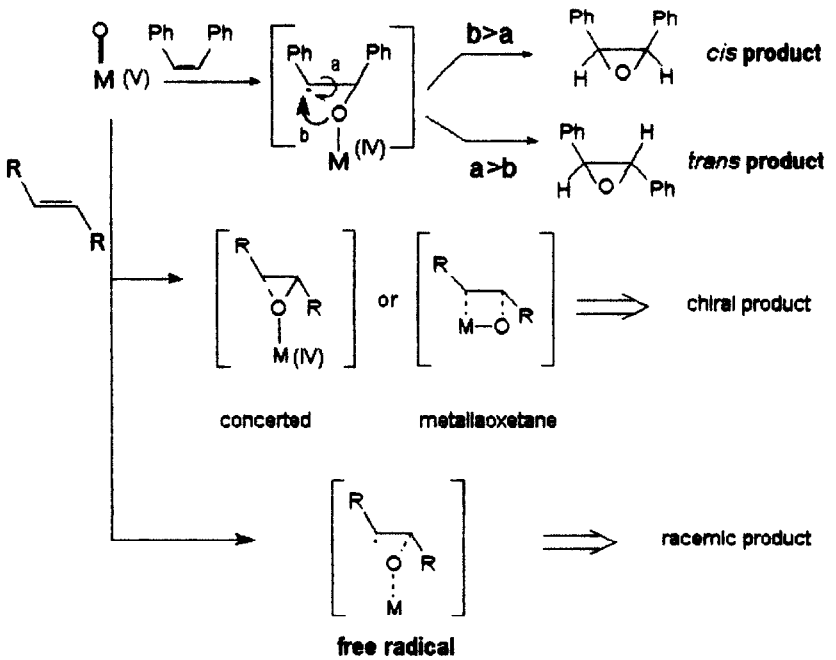


Fig. 4.



Scheme 2.

bene oxide were obtained, while in *trans*-stilbene oxide enantiomeric excesses were poor, which suggests the racemisation of *trans* product at the oxygen atom transfer stage where rotation (a) is faster than collaps (b) (Scheme 2). In the case of *trans*-3-nonene and *trans*-4-octene no *cis* products were obtained showing the oxygen atom transfer (Scheme 2) mostly through concerted addition/oxametallacyclic intermediates with a contribution of a radical mechanism which accounts for the loss in enantiomeric excess.

Acknowledgements

Two of us (R.I.K. and N.H.K.) are grateful for financial assistance from the CSIR Young Scientist Project, the DST Young Scientist Project and the Third World Academy of Sciences.

References

[1] T. Hamada, K. Daikai, R. Irie and T. Katsuki, Tetrahedron Asymm. 6 (1995) 2441.
[2] B.B. De, B.B. Lohray, S. Sivaraman and P.K. Dhal, Tetrahedron Asymm. 6 (1995) 2105.
[3] K. Bernardo, S. Leppard, A. Robert, G. Commenges, F. Dahan and B. Meunier, Inorg. Chem. 35 (1996) 387.
[4] H. Sasaki, R. Irie, T. Hamada, K. Susuki and T. Katsuki, Tetrahedron 50 (1994) 11827.
[5] R.A. Johnson and K.B. Sharpless, in: Comprehensive organic synthesis, Vol. 7, Oxidation (Pergamon Press, Oxford, 1992) ch. 3.2.
[6] M.G. Finn and K.B. Sharpless, J. Am. Chem. Soc. 113 (1991) 106–113.
[7] B.E. Rossitier, in: J.D. Morrison (Ed.), Asymmetric synthesis, Vol. 5 (Academic press, London, 1986) p. 194.
[8] Y. Naruta, N. Ishihara, F. Tani and K. Maruyama, Bull. Chem. Soc. Jpn. 66 (1993) 158.
[9] E.J. Corey and C.J. Helal, Tetrahedron Lett. 34 (1993) 5227.
[10] D. Wistuba, H.P. Nowotny, O. Trager and V. Schurig, Chirality (1989) L127–L136.
[11] H. Fu, M. Newcomb and C.H. Wong, J. Am. Chem. Soc. 113 (1991) 5878.
[12] T.J. McMurry and J.T. Groves, in: P.R. Ortiz de Montellano (Ed.), Cytochrome P-450. Mechanism and Biochemistry (Plenum Press, New York, 1986) pp. 1–29.
[13] B. Meunier, Chem. Rev. 92 (1992) 1411–1456.
[14] J.T. Groves, W.J.Jr. Kruper and R.C. Haushalter, J. Am. Chem. Soc. 102 (1980) 6375.
[15] J.T. Groves, W.J.Jr. Kruper, T.E. Nemo and R.S. Myers, J. Mol. Catal. 7 (1980) 169.
[16] M. Paulucki, P. Hauson and E.N. Jacobsen, Tetrahedron Lett. 33 (1992) 7111.
[17] K. Srinivasan, S. Perier and J.K. Kochi, J. Am. Chem. Soc. 108 (1986) 2309.
[18] H. Sabaki, I. Irie and T. Katsuki, Synlett (1994) 356–356.
[19] T. Furuda, R. Irie and T. Katsuki, Synlett (1995) 197.
[20] K. Irie, K. Noda, Y. Ito, N. Matsumoto and T. Katsuki, Tetrahedron Asymm. 2 (1991) 481.
[21] S. Chang, J.M. Galvin and E.N. Jacobsen, J. Am. Chem. Soc. 59 (1994) 6937.
[22] J.F. Larrow, E.N. Jacobsen, Y. Gao, Y. Hong, X. Nie and C.M. Zepp, J. Org. Chem. 59 (1994) 1939.
[23] E.N. Jacobsen, L. Deng, Y. Furukawa and L.E. Martinez, Tetrahedron 50 (1994) 4323.
[24] R.I. Kureshy, N.H. Khan, S.H.R. Abdi and K.N. Bhatt, Tetrahedron Asymm. 1 (1993) 1693.
[25] R.I. Kureshy, N.H. Khan and S.H.R. Abdi, J. Mol. Catal. 96 (1995) 117.
[26] R.I. Kureshy, N.H. Khan, S.H.R. Abdi and A K. Bhatt, J. Mol. Catal. 110 (1996) 33.
[27] R.I. Kureshy, N.H. Khan, S.H.R. Abdi and P. Iyer, J. Chem. Soc. Perkin Trans. 1, communicated.
[28] M.A. Bangres, M. Gonzales, M.E. Peres and R.J. Ruano, Polyhedron 5 (1986) 1371.
[29] K. Nakajima, K. Kojima, M. Kojima and J. Fujita, Bull. Chem. Soc. Jpn. 63 (1990) 2620.
[30] L.J. Boucher and C.G. Coe, Inorg. Chem. 15 (1976) 1334.