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Asymmetric hydroformylation and hydrogenation catalyzed by chiral rhodium and ruthenium complexes of phosphorylated 2,2′-bis(diphenyl-phosphino)-1,1′-binaphthyls

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Abstract

In situ-generated rhodium complexes of mono- and bisphosphorylated enantiopure BINAP ligands have been used for the asymmetric hydroformylation of styrene and vinylacetate. Corresponding Ru-complexes have been investigated in the homogeneous and biphasic asymmetric hydrogenation of dimethyl itaconate. An increase in the enantioselectivity of about 6–9% compared to BINAP was observed in the vinylacetate hydroformylation. For the aqueous biphasic hydroformylation of styrene the most enantioselective rhodium diphosphine catalyst (27% e.e.) up to now has been found. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Asymmetric hydroformylation; Asymmetric hydrogenation; Rhodium; Ruthenium; Aqueous biphasic catalysis

1. Introduction

The breakthrough in asymmetric hydroformylation was achieved by Takaya and co-workers with the C_2-symmetric phosphine–phosphite ligand BINAPHOS [1–6]. Rh/BINAPHOS catalysts showed conversions, regio- and enantioselectivities up to 98% with a broad range of substrates. Significant enantioselectivities were also induced by a number of other chiral phosphorus ligands almost bearing phosphite or phosphinite groups [7–12]. Nevertheless, none of them has been applied in a technical process probably due to insufficient catalyst stability, complicated ligand syntheses or difficult catalyst separation and recycling. Chiral diphosphines which are used in technical asymmetric hydrogenation processes can be obtained by convenient synthetic methods [13–17], but up to now these ligands showed only unsatisfactory enantioselectivities in asymmetric hydroformylation [18]. Little is known about this reaction in aqueous biphasic systems [19–21]. Compared to the homogeneous phase, lower or no enantiomeric excesses were observed. As yet, the technical and ecological advantages of the aqueous biphasic hydroformylation which are used in industrial processes of the hydroformylation of lower olefins [36,39] could not be utilized for enantioselective hydroformylation.

New theoretical approaches to hydroformylation with rhodium diphosphine catalysts and to the development of new and improved diphosphines were proposed in the last years [22–25,37]. Recently, Rampf and Herrmann reported on a ferrocenylethyldiphosphine/rhodium complex which showed the highest value of e.e. (76%) ever found in the hydroformylation of styrene with diphosphine catalysts [26]. Taking these advances into account it appears worthwhile to

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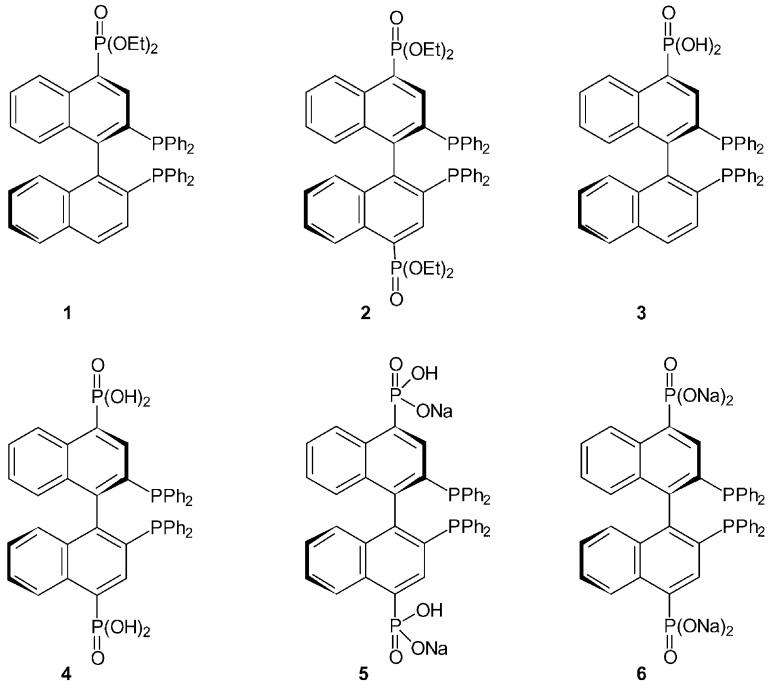


Fig. 1. Phosphorylated BINAP-type ligands used in catalysis.

put further efforts into the development of improved diphosphine-based catalysts.

For several years, we worked on the synthesis and catalytic properties of phosphine ligands for aqueous biphasic [27,28] and for asymmetric hydroformylation [29]. The excellent water-solubility of the phosphines which is absolutely essential for the aqueous biphasic method was achieved by the introduction of sodium phosphonate groups. Rhodium complexes formed with phosphorylated alkyldiarylphosphines showed excellent activities and selectivities particularly in the biphasic hydroformylation of higher 1-olefins.

These encouraging results prompted us to apply the biphasic protocol to asymmetric hydroformylations using phosphorylated enantiopure ligands. BINAP, which is conveniently available in both enantiopure forms [13], was used as chiral model ligand because it was already known to induce enantioselectivity in several hydroformylations [24,33]. Therefore, the binaphthyl backbone appeared suitable for phosphorylation. We prepared mono- and bisphosphorylated BINAP derivatives [30] (Fig. 1) and investigated them in both homogeneous and aqueous biphasic hydroformylation of styrene and vinylacetate. Because BINAP is known as an excellent ligand for enantioselective hydrogenations we tested the ligands **2**, **4** and **5** in the hydrogenation of dimethyl itaconate, too.

2. Experimental

All operations were carried out using Schlenk techniques with argon as inert gas. Synthesis gas ($CO:H_2$ 1:1, 99.98%) and hydrogen (99.999%) were purchased from Messer Griesheim. Tetrahydrofuran, toluene and styrene were distilled in an argon stream before use. Water, vinylacetate and dimethyl itaconate were deoxygenated by repeated evacuation and argon purging.

2.1. Materials

The preparation of the chiral ligands has been described elsewhere [30]. $Rh(CO)_2acac$ and $RuCl_2(p\text{-cymene})_2$ were used as purchased (Aldrich). $[Rh(OMe)(cod)]_2$ was prepared according to a procedure cited in literature [31]. The rhodium and

Table 1
Hydroformylation of vinylacetate

Run	Ligand	Rh precursor	Ligand/Rh	Solvent	TOF (h)	Temperature (°C)	Pressure (bar)	Time (h)	Conversion (%)	Aldehyde selectivity (%)	*iso*:*n* (%)	e.e. (%)
1	BINAP	$Rh(CO)_2acac$	3:1	THF	1.3	60	20	20	4.3	86.6	88:12	50.0
2	**1**	$Rh(CO)_2acac$	3:1	THF	0.7	60	20	20	2.3	96.4	85:15	57.5
3	**2**	$Rh(CO)_2acac$	3:1	THF	1.3	60	20	20	4.2	72.9	88:12	59.6
4	**3**	$Rh(CO)_2acac$	3:1	THF	0	60	20	70	0	–	–	–
5	–	$Rh(CO)_2acac$		Toluene	81.5	70	20	4	54.3	95.3	99:1	2.0
6	BINAP	$Rh(CO)_2acac$	3:1	Toluene	3.3	60	10	24	32.0	93.5	98:2	37.8
7	BINAP	$Rh(CO)_2acac$	3:1	Toluene	3.3	60	10	44	58.8	91.4	99:1	26.7
8	**2**	$Rh(CO)_2acac$	3:1	Toluene	9.1	60	10	24	87.7	91.3	99:1	28.6
9	**2**	$Rh(CO)_2acac$	3:1	Toluene	5.0	60	10	48	95.1	83.2	98:2	30.5
10	**2**	$[Rh(OMe)cod]_2$	3:1	THF	1.5	60	20	20	5.1	92.0	83:17	56.5
11	BINAP	$[Rh(OMe)cod]_2$	3:1	Toluene	3.3	60	10	20	26.4	88.8	95:5	55.3
12	**2**	$[Rh(OMe)cod]_2$	3:1	Toluene	4.4	60	10	20	35.3	88.6	93:7	61.3
13	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1	3.5	60	20	20	5.4	84.0	90:10	25.6
14	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1	4.6	60	50	20	7.0	64.0	83:17	23.5
15	**5**	$[Rh(OMe)cod]_2$	2:1	H_2O/EtOH 2:1	11.0	60	20	20	17.2	41.0	93:7	38.8

Table 2
Hydroformylation of styrene

Run	Ligand	Rh precursor	Ligand/Rh	Solvent	TOF (h)	Temperature (°C)	Pressure (bar)	Time (h)	Conversion (%)	Aldehyde selectivity (%)	*iso*:*n*	e.e. (%)
1	–	$Rh(CO)_2acac$		THF	112.5	70	40	4	100	99	71:29	2.8
2	BINAP	$Rh(CO)_2acac$	3:1	THF	1.6	70	40	20	7.0	99	92:8	22.5
3	BINAP[a]	$Rh(CO)_2acac$	3:1	THF	1.7	70	40	20	7.5	99	93:7	17.8
4	**1**	$Rh(CO)_2acac$	3:1	THF	2.5	70	40	20	11.0	99	92.8	18.3
5	**2**	$Rh(CO)_2acac$	3:1	THF	2.8	70	40	20	12.6	99	92:8	21.4
6	**3**	$Rh(CO)_2acac$	3:1	THF	0	70	40	20	0	–	–	–
7	**5**	$[Rh(OMe)cod]_2$	2:1	H_2O/EtOH 2:1[b]	4.0	60	10	20	9.0	97	94:6	16.1
8	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1[b]	0.4	40	10	20	1.0	100	95:4	26.6
9	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1[b]	4.0	60	10	20	8.8	96	95:5	23.5
10	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1[b]	4.6	60	20	20	10.3	93	94:6	13.6
11	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1[b]	4.8	60	30	20	10.7	89	95:5	18.6
12	**5**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1[b]	1.0	60	50	20	2.2	96	96:4	23.2
13	**6**	$Rh(CO)_2acac$	2:1	H_2O/EtOH 2:1[b]	7.1	60	10	20	15.8	89	94:6	12.2

[a] Performed at 100°C, 50 bar, 2 h.
[b] An excess of styrene (5 ml) was used as organic phase.

ruthenium diphosphine complexes were prepared as follows: hydroformylation: 5.0 mg $Rh(CO)_2$acac or 10.50 mg $[Rh(OMe)(cod)]_2$ and the ligand (see Tables 1 and 2) were put together in 10 ml of the solvent. Thereafter, the solution was stirred for 1 h. The water soluble complexes were obtained by adding an ethanolic solution (5 ml) of 12.5 mg (4.86×10^{-5} mol) $Rh(CO)_2$acac to an aqueous solution (10 ml) of 9.71×10^{-5} mol of **5** or **6** (formed from **4** and a two-fold or four-fold excess of NaOH). Hydrogenation: 1.65 mg (2.69×10^{-6} mol) $[RuCl_2(p\text{-cymene})]_2$ and 2.69×10^{-6} mol of the ligand **2** or **4** were dissolved each in 5 ml dry ethanol. Both solutions were mixed and stirred for 1 h. To obtain a water–soluble complex 10.76×10^{-6} mol NaOH (2.0 equivalents) were added to the mixture of 5.38×10^{-6} mol of **4** and $[RuCl_2(p\text{-cymene})]_2$ in 10 ml H_2O/2 ml EtOH.

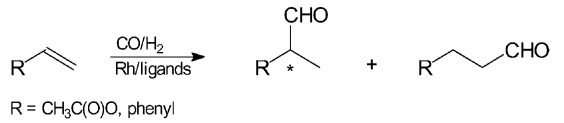
2.2. Catalysis

To exclude air, a 100 ml stainless steel autoclave equipped with a magnetic stirrer was filled using Schlenk techniques with solutions containing the substrates and the catalyst precursors (for homogeneous runs 1 ml styrene or 1 ml vinylacetate were used as substrates while 5 ml styrene or 5 ml vinylacetate were employed in the biphasic hydroformylations. An amount of 255 mg dimethyl itaconate were used both in homogeneous and biphasic asymmetric hydrogenations.). After filling in the liquids the autoclave was purged with synthesis gas or with hydrogen for three times and heated up to the desired temperature. Synthesis gas or hydrogen were added to adjust the pressure. Samples were taken to follow the reaction using GC analysis (HP 5890, FID, helium carrier gas). A 30 m × 0.25 mm × 0.25 μm capillary with a β-cyclodextrine/DV-101 phase (BGB 174, Seitz) was used for the separation and quantitative analysis of the sample components including the chiral products.

3. Results and discussion

3.1. Asymmetric hydroformylation of vinylacetate

Generally, the hydroformylation of vinylacetate yielded 2- and 3-acetoxypropanals with high selectivity. Ethyl acetate and acetic acid were found as by-products. The results obtained with the novel rhodium diphosphine complex catalysts are reported in Tables 1 and 2. Generally, the solvent and the metal precursors influenced the activity and selectivity significantly. Such effects have recently been published by Hoegaerts and Jacobs [24] who showed that the enantioselectivity can be improved by combining the optimal metal precursor, solvent, pressure and reaction temperature. Also Bayón and co-workers discussed this dependence for catalysts with Chiraphos and BDPP [37] (Scheme 1).



Scheme 1. Hydroformylation of vinylacetate and styrene.

In the hydroformylation of vinyl acetate with the phosphine ligands **1**, **2** and **5** high chemo- and regioselectivities have been found (runs 2, 3, 8–10, 12, 13). An increase in enantioselectivity by 6–9% compared to the unsubstituted BINAP was observed. Hemilabile coordination of the phosphonate oxygen to the rhodium center, as discussed by Rampf and Herrmann to explain the enhanced selectivity of a methoxy-substituted diphosphine [26], appears unlikely for the phosphorylated BINAP ligands because of the high energy barrier of about 50 kcal/mol estimated by MM^+ calculations. A plausible explanation for the enhanced selectivity is an interaction between the polar phosphonate group and the polar substrate vinylacetate leading to the preference of one of the diastereomeric transition states. Such interactions are unlikely for the non-polar styrene where no improvement of the enantioselection was found. Accordingly, this effect did not occur in the hydroformylation of the non-polar styrene.

The phosphonic acid **3** inhibited both the hydroformylations of vinylacetate and of styrene completely while in the hydroformylation of vinylacetate the phosphorylated complex formed by **2** and $[Rh(OMe)cod]_2$ was by one third more active in toluene than the unsubstituted rhodium BINAP complex (run 12 versus run 11). This complex showed the best enantioselectivities, whereas the analogous complex with the $Rh(CO)_2$acac precursor was more active

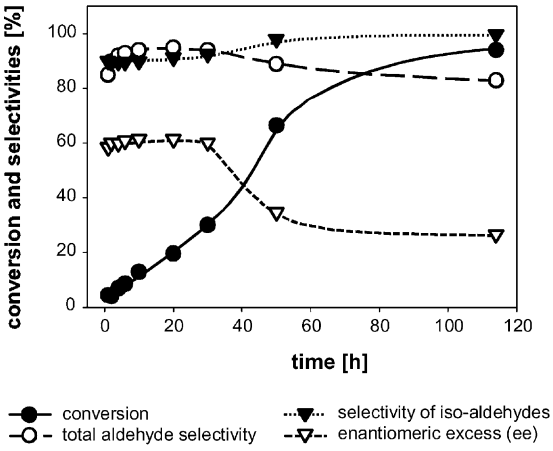


Fig. 2. Long-term behavior of a 2/$[Rh(OMe)]_2$ catalyst in the hydroformylation of vinylacetate (solvent: toluene, 60°C, 10 bar, ligand/Rh 3:1, substrate/Rh 250:1.

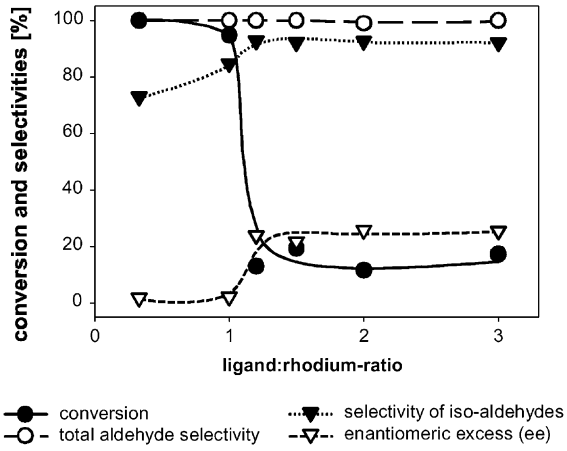


Fig. 3. Dependence of conversion and selectivity on the ligand:rhodium ratio (substrate: styrene, catalyst: S-BINAP/$[Rh(OMe)]_2$, toluene, 70°C, 40 bar, 20 h; (for BINAP/Rh 1:1 and 1:3 6 h)).

but less stereoselective (runs 8 and 9). Compared to homogeneous hydroformylation the enantioselectivity decreased markedly in aqueous biphasic systems (runs 13–15), as known from other asymmetric reactions [32]. But also at these conditions the complex formed from $[Rh(OMe)cod]_2$ and **5** showed the best enantioselectivity (run 15).

The stability of the catalyst has been checked in a separate experiment (Fig. 2). Over a period of 30 h no changes in the catalyst performance were found. Thereafter, the enantioselectivity dropped significantly and an activity increase was observed. Both, changes in the catalyst structure or razemization of the branched aldehydes via enolization could explain this effect. However, taking into account the opposite trends in activity and enantioselectivity, the formation of unmodified Rh carbonyls appears more likely since these non-chiral species are known to be orders of magnitudes more active than Rh/phosphine catalysts. Therefore, even small amounts of unmodified rhodium catalyst in the reaction mixture can bring about the observed decline in enantioselectivity while the activity increases.

3.2. Asymmetric hydroformylation of styrene

The results obtained for the homogeneous hydroformylation of styrene are given in Table 2. Rh–BINAP and Rh–**2** complexes induced enantiomeric excesses of 22.5 and 21.4% and showed high chemo- and regioselectivities as also reported for BINAP by Claver and co-workers [33] Compared to the parent BINAP complex the catalyst with the bisphosphorylated ligand **2** was about two times more active (run 5).

Fig. 3 shows the influence of the BINAP/Rh ratio on the activity and the selectivity in the hydroformylation of styrene. As expected, at the low ratios below one, the results match to those obtained with the unmodified Rh catalyst. Although much higher ligand excesses have usually been applied, the catalyst reaches its typical enantioselectivity already at a ligand/Rh ratio as low as 1.2. Further increases in this ratio did not improve the enantioselectivity and hardly affected the rate of the reaction.

In the aqueous biphasic system the disodium salt **5** was the best ligand (runs 8 and 9). Surprisingly, and in contrast to previously published results with chiral water-soluble catalysts [19–21], no decrease in enantioselectivity compared with the homogeneous system was observed with this ligand. Even a higher activity has been found in the aqueous milieu. The more basic tetrasodium salt **6** afforded an enhanced activity at the cost of enantioselectivity. This indicates a dependence of the activity on the pH value, as reported by Chaudhari and co-workers [35]. Runs 9–12 were carried out to determine the optimal syngas pressure for the

Table 3
Hydrogenation of dimethyl itaconate[a]

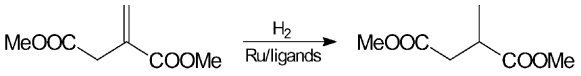
Run	Ligand	Solvent	Yield (%)	e.e. (%)
1	BINAP	EtOH	99.8	91.5
2	**2**	EtOH	99.1	88.7
3	**4**	EtOH	99.3	93.2
4	**5**	H_2O/EtOH/hexane 5:1:5	99.4	79.4

[a] Reaction conditions: 60 °C, 20 bar, 2 h, ligand/Ru 1:1, substrate/Ru 300:1, precursor $[RuCl_2(p\text{-cymene})]_2$.

reaction. The pressure of 10 bar has been found as optimum (run 9), whereas with rising pressure the enantioselectivity passed through a minimum and reached the same level at 50 bar as at 10 bar. This contrasts with the view that higher syngas pressures support the formation of the achiral $RhH(CO)_4$ complex, which reduces enantioselectivity [24]. Although the styrene hydroformylation was observed to be linearly dependent with respect to the H_2 pressure [41], the inhibition of the reaction at 50 bar total syngas pressure could be explained by the formation of inactive di- and tricarbonyl rhodium complexes [42].

3.3. Asymmetric hydrogenation of dimethyl itaconate

Ruthenium complexes of BINAP are excellent catalysts for asymmetric hydrogenation [14]. Therefore, we tested the potential of the phosphorylated BINAP ligands in the hydrogenation of dimethyl itaconate (Table 3), whereas the ruthenium catalysts were formed in situ. [1] Compared to BINAP, the complex of the bisphosphonic acid **4** showed a slight increase of the enantiomeric excess, while the bisphosphonate ligand **2** was less enantioselective. Obviously, the substitution in fourth position of the binaphthyl skeleton does not disturb significantly the enantioselection step during the catalytic cycle. In an aqueous biphasic system the enantiomeric excess was slightly diminished, as observed in many cases for asymmetric hydrogenations in water [32,40]. To our knowledge, only one example for the asymmetric hydrogenation of dimethyl itaconate in an aqueous two-phase system has been published [38] and only low enantiomeric excesses (maximum 28%) were achieved with sulfonated BDPP/Rh complexes (Scheme 2).



Scheme 2. Hydrogenation of dimethyl itaconate.

4. Conclusions

The phosphorylated BINAP derivatives are suitable ligands both for homogeneous and aqueous biphasic hydroformylation. Their rhodium complexes showed high chemo- and regioselectivities in the hydroformylations of styrene and vinylacetate. Compared with catalysts prepared by the parent BINAP ligand the enantioselectivity in the hydroformylation of vinylacetate was slightly increased using the phosphorylated ligands **1** and **2**. The enantiomeric excess obtained with the catalyst made of **5** and $Rh(CO)_2acac$ is the highest one obtained in the aqueous biphasic hydroformylation of styrene [20].

The BINAP-derivatives **2** and **4** performed like BINAP in the asymmetric hydrogenation of dimethyl itaconate. Moreover, phosphorylated BINAP ligands can be employed in aqueous biphasic systems and thus offer the advantage of simple catalyst recycling.

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