Asymmetric Ir-catalyzed hydrogenation of 1,3-dihydro-2*H*-1,5-benzodiazepin-2-ones using phosphoramidites

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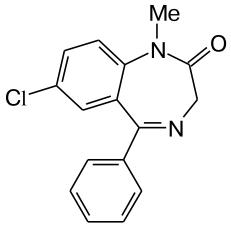
A series of phosphoramidite ligands was tested in the asymmetric hydrogenation of 4-aryl-substituted 1,3-dihydro-2*H*-benzodiazepine-2-ones and up to 52% *ee* was achieved. The effects of various factors (solvents, hydrogen pressure, and addition of phosphine ligands) on the hydrogenation were studied.

Key words: asymmetric hydrogenation, phosphoramidites, mixed ligands, 1,5-benzodiazepin-2-ones.

Widely used hypnotic agents, tranquilizers, and antidepressant drugs are based on benzodiazepine derivatives such as the mild tranquilizer diazepam (sibazon, seduxen (**1**)) and nitrazepam (**2**).[1,2] Grandaxin (tofisopam (**3**)) with anxiolytic action without a soporific effect also refers to benzodiazepine derivatives.[3] In addition, compounds of this class are applied as anesthetics, muscle relaxants, analgesics, and antiepileptic drugs.[4—7]



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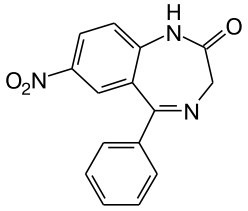

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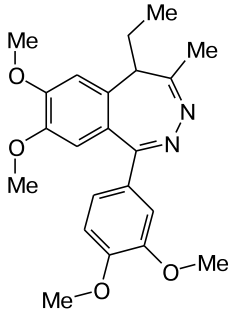

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Among benzodiazepinones there are anticancer agents (farnesyl protein transferase inhibitors);[8] HIV replication inhibitors;[9] caspase-3 inhibitors;[10] cholecystokinin antagonists[11] used, in particular, to treat the gastric ulcer; and antipyretics.[12]

Hydrogenated benzodiazepinone derivatives, tetrahydro-1*H*-benzodiazepinones, including chiral ones, represent a new, actively developed class of biologically active compounds exhibiting antiasthmatic, antitumor, and neuroprotective properties.[13—15] The existing approaches to the preparation of these chiral compounds are based on organocatalysis.[16,17] Also, the applicability of chiral diphosphine ligands in the asymmetric hydrogenation of 4-substituted 1,3-dihydro-2*H*-1,5-benzodiazepin-2-ones has been recently reported.[18] For attaining high enantioselectivity, it was necessary to use more than 5 mol.% of expensive NaBARF (BARF is tetrakis[3,5-bis(trifluoromethyl)phenyl]borate) and the (*R*)-DifluoroPhos chiral ligand, and the products were obrained in the *N*-acetyl form. Previously we briefly described the first examples of application of phosphite type ligands in asymmetric hydrogenation of 1,5-benzodiazepinones.[19,20] This study addresses the effect of the ligand, metal, nature of the solvent, and hydrogen pressure on the reactant conversion and enantioselectivity in 1,5-benzodiazepinone hydrogenation.

Results and Discussion

We prepared a small series of known phosphoramidite ligands (**L1—L3**) with cyclic and acyclic substituents for testing in the hydrogenation of benzodiazepinones.

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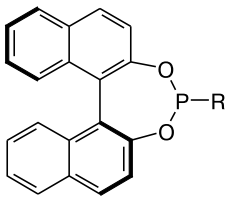
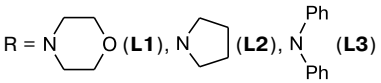
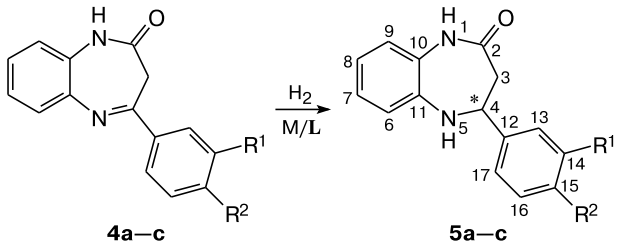


L1—L3



First, the ligands were tested in the Ir-catalyzed hydrogenation of 4-phenyl-1,3-dihydro-2*H*-1,5-benzodiazepin-2-one (**4a**) (Scheme 1) in dichloromethane. The use of any of the ligands resulted in quantitative conversion,

Scheme 1



4a—c **5a—c**

4, **5**	R^1	R^2
a	H	H
b	H	OMe
c	OMe	H

but the enantioselectivity did not exceed 8%. The replacement of the solvent by ethanol markedly increased the reaction selectivity (Table 1, entries *1—3*), and **L1** proved to be the best ligand. The use of methanol produces some increase in the asymmetric induction, whereas trifluoroethanol (TFE) or propan-2-ol lead, conversely, to a pronounced decrease in the enantioselectivity (see Table 1, entries *4—6*).

We also checked the idea of combining chiral phosphoramidite ligands with achiral phosphines. This approach is known in the hydrogenation of heterocyclic compounds and, in some cases, it considerably increases the enantioselectivity.[19—22] By using **L1—L3** in the hydrogenation of **4a** in ethanol with addition of triphenylphosphine, full conversion was achieved, but the enantioselectivity was moderate (see Table 1, entries *7—9*). The replacement of ethanol by dichloromethane (see Table 1, entries *9* and *10*), as in the experiments without addition of phosphines, led to the formation of an almost racemic reaction product. The use of tricyclohexylphosphine resulted in a somewhat higher selectivity (see Table 1, entries *10* and *11*). The presence of methanol as the solvent slightly improved the result (see Table 1, entry *12*). The addition of tri-*o*-tolylphosphine in the reaction conducted in methanol gave 52% *ee* (see Table 1, entry *13*). A lower *ee* value was provided by ethanol (see Table 1, entry *14*), and with dichloromethane the process became non-selective (see Table 1, entry *15*). We also investigated the effect of the hydrogen pressure on the reaction rate and selectivity. A pressure decrease from 70 to 35 atm reduced both the

Table 1. Ir-Catalyzed hydrogenation of **4a—c**[a]

Entry	Catalyst	Substrate	Solvent	*C*[b] (%)	*ee* (%)[c]
1	$[Ir(COD)Cl]_2$/4**L2**	**4a**	EtOH	31	22 (–)
2	$[Ir(COD)Cl]_2$/4**L3**	**4a**	EtOH	100	0
3	$[Ir(COD)Cl]_2$/4**L1**	**4a**	EtOH	100	30 (–)
4	$[Ir(COD)Cl]_2$/4**L1**	**4a**	MeOH	100	36 (–)
5	$[Ir(COD)Cl]_2$/4**L1**	**4a**	Pr^iOH	100	4 (–)
6	$[Ir(COD)Cl]_2$/4**L1**	**4a**	TFE	100	20 (–)
7	$[Ir(COD)Cl]_2$/2**L2**/2PPh_3	**4a**	EtOH	100	18 (–)
8	$[Ir(COD)Cl]_2$/2**L3**/2PPh_3	**4a**	EtOH	100	0
9	$[Ir(COD)Cl]_2$/2**L1**/2PPh_3	**4a**	EtOH	100	13 (–)
10	$[Ir(COD)Cl]_2$/2**L1**/2PPh_3	**4a**	CH_2Cl_2	100	2 (–)
11	$[Ir(COD)Cl]_2$/2**L1**/2PCy_3	**4a**	CH_2Cl_2	100	4 (–)
12	$[Ir(COD)Cl]_2$/2**L1**/2PCy_3	**4a**	MeOH	100	14 (–)
13	$[Ir(COD)Cl]_2$/2**L1**/2P(*o*-tol)$_3$	**4a**	MeOH	100	52 (–)
14	$[Ir(COD)Cl]_2$/2**L1**/2P(*o*-tol)$_3$	**4a**	EtOH	100	42 (–)
15	$[Ir(COD)Cl]_2$/2**L1**/2P(*o*-tol)$_3$	**4a**	CH_2Cl_2	100	0
16	$[Ir(COD)Cl]_2$/2**L1**/2P(*o*-tol)$_3$[d]	**4a**	MeOH	78	30 (–)
17	$[Ir(COD)Cl]_2$/2**L1**/2P(*o*-tol)$_3$	**4b**	MeOH	73	32 (–)
18	$[Ir(COD)Cl]_2$/2**L1**/2P(*o*-tol)$_3$	**4c**	MeOH	68	42 (–)

[a] T = 20 °C, τ = 18 h, $P(H_2)$ = 70 atm, 1 mol.% $[Ir(COD)Cl]_2$.
[b] *C* is conversion.
[c] The specific rotation sign of the product is given in parentheses.
[d] $P(H_2)$ = 35 atm.

Table 2. Full assignment of the ^{1}H NMR signals fot compounds **4b**,**s** and **5b**,**c** ($CDCl_3$)

Atom, gtoup	δ, *J*/Hz			
	4b	**5b**	**4c**	**5c**
1	8.55 (br.s, 1 H)	7.80 (br.s, 1 H)	8.62 (br.s, 1 H)	8.05 (br.s, 1 H)
3	3.57 (br.s, 2 H)	2.88 (dd, 1 H, *J* = 9.6, *J* = 13.8); 2.75 (br.dd, 1 H, *J* = 4.2, *J* = 13.8)	3.59 (s, 2 H)	2.91 (dd, 1 H, *J* = 10.2, *J* = 13.2); 2.76 (dd, 1 H, *J* = 2.4, *J* = 13.2)
4	—	4.99 (br.dd, 1 H, *J* = 3.6, *J* = 9.6)	—	5.01 (br.dd, 1 H, *J* = 2.4, *J* = 9.6)
5	—	3.80 (br.s, 1 H)	—	3.86 (br.s, 1 H)
6	7.50 (d, 1 H, *J* = 7.8)	6.83 (d, 1 H, *J* = 7.8)	7.53 (dd, 1 H, *J* = 7.8, *J* = 1.2)	6.87 (d, 1 H, *J* = 7.8)
7	7.28 (br. t, 1 H, *J* = 7.8)	7.05—7.09 (m, 1 H)	7.27—7.31 (m, 1 H)	7.08 (t, 1 H, *J* = 7.2)
8	7.23 (dt, 1 H, *J* = 7.8, *J* = 0.6)	6.93—6.96 (m, 1 H)	7.24—7.28 (m, 1 H)	6.93—6.97 (m, 1 H)
9	7.10 (d, 1 H, *J* = 7.8)	6.94—6.97 (m, 1 H)	7.12 (d, 1 H, *J* = 7.8)	6.98 (d, 1 H, *J* = 7.2)
13	8.10 (d, 1 H, *J* = 9.0)	7.32 (d, 1 H, *J* = 8.4)	7.71 (t, 1 H, *J* = 1.8)	6.95 (s, 1 H)
14	7.00 (d, 1 H, *J* = 9.0)	6.91 (d, 1 H, *J* = 9.0)	—	—
15	—	—	7.07 (dd, 1 H, *J* = 8.4, *J* = 2.4)	6.87 (d, 1 H, *J* = 7.8)
16	7.00 (d, 1 H, *J* = 9.0)	6.91 (d, 2 H, *J* = 9.0)	7.41 (t, 1 H, *J* = 7.8)	7.30 (t, 1 H, *J* = 7.8)
17	8.10 (d, 1 H, *J* = 9.0)	7.32 (d, 1 H, *J* = 8.4)	7.68 (d, 1 H, *J* = 7.8)	6.98 (d, 1 H, *J* = 7.2)
OMe	3.89 (s, 3 H)	3.83 (s, 3 H)	3.91 (s, 3 H)	3.82 (s, 3 H)

conversion and the enantioselectivity (see Table 1, entries *13* and *16*).

We attempted using $[Rh(COD)Cl]_2$ as a precatalyst. Unlike the use of $[Ir(COD)Cl]_2$, the application of $[Rh(COD)Cl]_2$ and **L1** (or **L1** combined with P(*o*-tol)$_3$) in CH_2Cl_2 leads to very low (below 12%) conversion and virtually no enantioselectivity, while the same reaction in methanol does not occur at all.

According to the study of the Ir-catalyzed hydrogenation of methoxy-substituted substrates **4b,c** (see Scheme 1, Table 1, entries *17* and *18*) using **L1** and P(*o*-tol)$_3$ as an additive, the catalysis is fairly sensitive to the nature of substituent. Indeed, the introduction of electron-donating methoxy groups leads to a slight decrease in the conversion as compared with the reaction of **4a** under the same reaction conditions. In addition, more pronounced electron-donating effect of the 4-methoxy substituent towards the C=N bond being hydrogenated results in lower selectivity in comparison with that for 3-methoxy-substituted substrate **4c**.

Detailed investigation of $CDCl_3$ solutions of hydrogenation products **5b,c** (as well as substrates **4b,c**) by 1D and 2D NMR techniques, namely, ^{1}H, $^{13}C\{^1H\}$, ^{13}C DEPT, ^{1}H—^{1}H COSY, ^{1}H—^{1}H NOESY, ^{1}H—^{13}C HSQC, and ^{1}H—^{13}C HMBC experiments, allowed full assignment of all resonance signals in the ^{1}H and ^{13}C NMR spectra (see Tables 2 and 3).

In the comparison of the spectral parameters of compounds **4b**,**c** and **5b**,**c**, attention is attracted by the pronounced (more than 95 ppm) upfield shift of the C(4)

Table 3. Full assignment of the ^{13}C NMR signals for compounds **4b**,**c** and **5b**,**c** ($CDCl_3$)

Atom, group	δ			
	4b	**5b**	**4c**	**5c**
2	167.63	171.89	167.57	172.07 (br)
3	39.48	41.99	39.92	41.92 (br)
4	158.19	62.59	158.64	63.21
6	128.26	121.13	128.37	121.15
7	125.12	126.06	125.12	126.11
8	126.04	121.41	126.50	121.57
9	121.73	122.35	121.75	122.47
10	129.02	127.70	129.09	127.81
11	140.27	138.39	139.98	138.38
12	130.22	136.57	139.11	146.05
13	129.66	127.16	112.36	111.68
14	114.08	114.32	159.97	160.07
15	162.14	159.44	117.60	113.48
16	114.08	114.32	129.70	130.08
17	129.66	127.16	120.40	118.18
OMe	55.46	55.33	55.46	55.30

signal in the ^{13}C NMR spectra, which proves the reduction of the C=N bond. Correspondingly, the ^{1}H NMR spectra of **5b**,**c** reasonably exhibit a broad singlet for the secondary amino group at 3.80—3.86 ppm and a doublet of doublets at 4.99—5.01 ppm for protons at C(4) as a result of spin—spin coupling with two non-equivalent protons at C(3). The spectral parameters for the peripheral part of molecules **5b**,**c** (phenyl ring) change insignificantly with respect to those of **4b**,**c**. Conversely, despite the different positions of the methoxy substituent in the peripheral benzene ring in the hydrogenation products **5b** and **5c**, the ^{1}H и ^{13}C NMR resonance signals for the 4,5-dihydrobenzodiazepinone core atoms are largely similar.

Thus, a series of phosphoramidite ligands was tested in asymmetric hydrogenation of 4-substituted 1,3-dihydro-2*H*-1,5-benzodiazepin-2-ones. The addition of tri-*ortho*-tolylphosphine markedly increases the enantioselectivity. Methanol was found to be the solvent of choice for hydrogenation, while in dichloromethane, virtually no enantioselectivity is observed in all cases. A combination of $[Ir(COD)Cl]_2$, chiral phosphoramidite **L1**, and tri-*ortho*-tolylphosphine shows the best values of both conversion and enantioselectivity for the same reaction conditions.

Experimental

^{31}P, ^{1}H, and ^{13}C NMR spectra were recorded on Bruker Avance 400 (161.98, 400.13, and 100.61 MHz) and Bruker Avance III 600 (242.94, 600.13, 150.90 MHz) instruments, with 85% H_3PO_4 in D_2O and Me_4Si, respectively, as the standards.

Enantiomeric analysis of the products of catalytic reactions was performed by HPLC using an Agilent HP-1100 chromatograph. (S_a)-2-(4-Tetrahydrooxazin-1-yl)-dinaphtho-[2,1-*d*:1′,2′-*f*][1,3,2]dioxaphosphepine (**L1**),[23] (S_a)-2-(pyrrolidin-1-yl)-dinaphtho[2,1-*d*:1′,2′-*f*][1,3,2]dioxaphosphepine (**L2**),[24] (S_a)-2-(diphenylamino)-dinaphtho[2,1-*d*:1′,2′-*f*][1,3,2]dioxaphosphepine (**L3**),[25] [Ir(COD)Cl]2,[26] $[Rh(COD)Cl]_2$,[27] and 4-phenyl-1*H*-benzo[*b*][1,4]diazepin-2(3*H*)-one (**4a**)[28] were prepared by reported procedures. The spectral characteristics of 4-phenyl-1,3-dihydro-2*H*-1,5-benzodiazepin-2-one (**4a**) correspond to literature data.[28] The enantiomeric excess for reaction product **5a** was determined using a Kromasil 3-AmyCoat column and a reported procedure.[19]

Synthesis of benzodiazepines 4b,c (general procedure). A solution of ethyl 3-(4-methoxyphenyl)-3-oxopropanoate or ethyl 3-(3-methoxyphenyl)-3-oxopropanoate (1.422 g, 0.0064 mol) in xylene (2 mL) was added dropwise over a period of 20 min to a boiling solution of *o*-phenylenediamine (0.54 g, 0.005 mol) in *o*-xylene (1.5 mL). The mixture was refluxed for 2 h, cooled to room temperature, and kept for 2 h. The crystals of the product that precipitated on storage were collected on a filter, washed with cold *o*-xylene, and recrystallized from ethyl acetate.

4-(4-Methoxyphenyl)-1*H*-benzo[*b*][1,4]diazepin-2(3*H*)-one (4b). Yield 0.666 g (52%). White crystals, m.p. 218 °C. Found (%): C, 72.21; H, 5.35; N, 10.46. $C_{16}H_{14}N_2O_2$. Calculated (%): C, 72.16; H, 5.30; N, 10.52.

4-(3-Methoxyphenyl)-1*H*-benzo[*b*][1,4]diazepin-2(3*H*)-one (4c). Yield 0.746 g (56%). White crystals, m.p. 206 °C. Found (%): C, 72.22; H, 5.37; N, 10.45. $C_{16}H_{14}N_2O_2$. Calculated (%): C, 72.16; H, 5.30; N, 10.52.

Asymmetric hydrogenation of 4a—c. A 10 mL autoclave was charged with a solution of $[Ir(COD)Cl]_2$ or $[Rh(COD)Cl]_2$ (0.005 mmol) in a solvent (0.2 mL) together with ligand **L1**—**L3** (0.02 mmol) or with a mixture of a chiral ligand (0.01 mmol) and appropriate phosphine (0.01 mmol) (see Table 1) in CH_2Cl_2 (0.2 mL). The reaction mixture was stirred for 2 min, the solvent was removed *in vacuo*, and the specified solvent was added without degassing (2 mL, see Table 1). Then the substrate **4a**—**c** (0.5 mmol) was added. The autoclave was filled with hydrogen (70 or 35 atm) and the reaction was conducted with magnetic stirring. After the end of the reaction, hydrogen was released and the solvent was removed *in vacuo*. The conversion was determined by ^{1}H NMR spectroscopy.

4-(4-Methoxyphenyl)-4,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2(3*H*)-one (5b). White crystals, m.p. 150—152 °C. Found (%): C, 71.91; H, 6.29; N, 10.27. $C_{16}H_{16}N_2O_2$. Calculated (%): C, 71.62; H, 6.01; N, 10.44. The enantiomeric excess of **5b** was determined by HPLC on a Kromasil 5-TBB column, 1 mL min^{-1}, 219 nm, hexane—propan-2-ol (95 : 5); the retention time of the (+)-isomer of **5b** was 18 min, that of the (−)-isomer of **5b** was 19 min.

4-(3-Methoxyphenyl)-4,5-dihydro-1*H*-benzo[*b*][1,4]diazepin-2(3*H*)-one (5c). White crystals, m.p. 80—81 °C. Found (%): C, 71.90; H, 6.27; N, 10.18. $C_{16}H_{16}N_2O_2$. Calculated (%): C, 71.62; H, 6.01; N, 10.44. The enantiomeric excess for **5c** was determined by HPLC on a Chiralcel AD-H column, 1 mL min^{-1}, 219 nm, hexane—propan-2-ol (7 : 3), the retention time of the (−)-isomer of **5c** was 14 min, that of the (+)-isomer of **5c** was 19 min.

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