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Account/Revue

Chiral diphosphines and diphosphinites derived from 2,2-biphosphole: Stereodynamic ligands for asymmetric catalysis

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ABSTRACT

In this account, the recent advances on chiral stereochemically dynamic 2,2'-biphosphole ligands for applications in asymmetric catalysis are reported. In the first part, the synthesis of stereodynamic diphosphines and diphosphinites derived from 2,2'-biphosphole is presented. The second part describes the kinetic dynamic resolution to give diastereo- and enantiopure complexes. Applications in asymmetric allylic substitution, hydroformylation and hydrogenation are presented in the last part.

1. Introduction

Advances in transition metal catalyzed asymmetric processes have been traditionally guided by the concept that stereochemically rigid enantiopure ligands are required to achieve high enantioselectivities [1]. Chiral diphosphines proved to be one of the most successful and widely used ligands for this purpose in particular the highly versatile BINAP which belongs to the atropos[1] class of diphosphine ligands since its axial chiral conformation can be resolved [1c]. A conceptually new approach has emerged where achiral and meso ligands can be used to convey asymmetry in enantioselective catalysis [2]. Therefore, conformationally flexible tropos[1] diphosphine ligands are intentionally used to either magnify the stereochemical induction of a chiral ligand or act as the only source of asymmetry for enantioselective transformation. In the latter case, coordination of the ligand to transition metal slows interconversion of its conformations and as a consequence a metastable enantiopure assembly can be resolved by a chiral controller and used for catalysis with or without it (Scheme 1). Enantiopure complexes with tropos diphosphines such as BIPHEP [3], NUPHOS [4], cyclo-NUPHOS or DPPF [5] demonstrate good to excellent enantioselectivities and can efficiently replace complexes bearing classical enantiopure ligands.

The advantage of this approach to asymmetric catalysis over the traditional one is that catalysts can be optimised by the synthesis of achiral or meso ligands instead of the synthesis of enantiopure ones which requires elaborate and expensive methods (e.g., asymmetric synthesis or resolution). Consequently, the design of new catalysts, based on dynamic chirality control (e.g., N-chirality control [6] or dual chirality control of axial and N-chirality [7]) by

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[1] The word atropos consists of "a" meaning "not" and "tropos" meaning "turn" in Greek.

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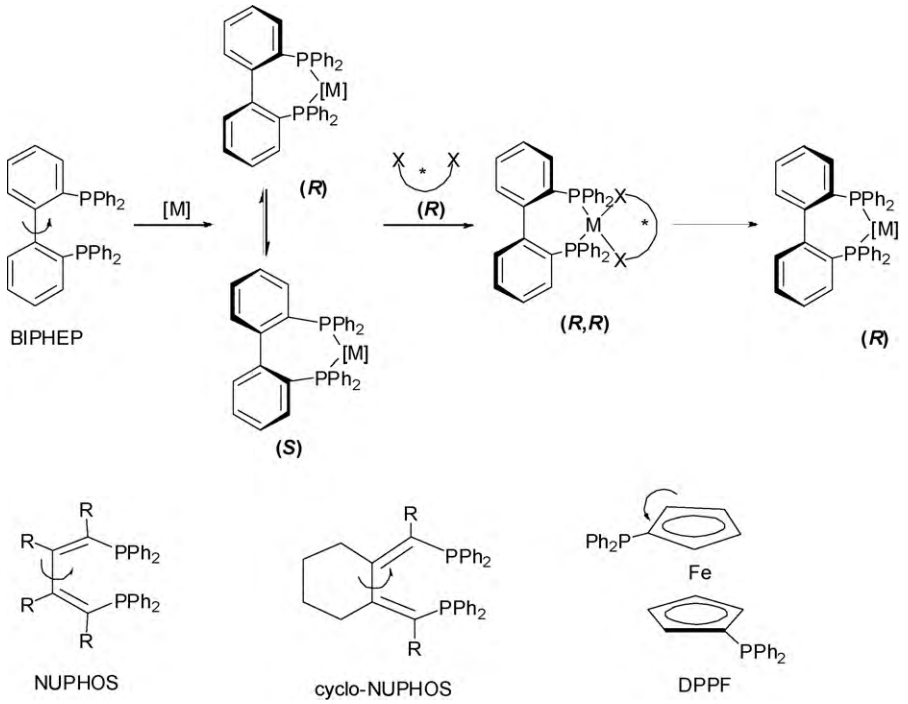


Scheme 1. BIPHEP, NUPHOS, cyclo-NUPHOS, DPPF.

chiral activator through complexation is actually an important field.

In a related approach, we have examined the dual chirality control of axial and central phosphorus chiralities of 2,2'-biphosphole ligands [8]. Some years ago, we reported the first application of the chiral stereochemically dynamic 2,2'-biphophole ligand (BIPHOS) to asymmetric allylic substitution involving crystallization-induced spontaneous resolution and kinetic stabilization by coordination to Pd centre[2] [9]. In a more convenient procedure, we have discovered that a dual chirality control can be achieved by introducing a chiral linker between the two phosphorus atoms that favours a single enantiomeric form upon coordination to a metal [10]. In this article, we report our approach to stereodynamic diphosphines and diphosphinites derived from 2'2-biphospholes for asymmetric catalysis.

2. Synthesis of stereodynamic diphosphines and diphosphinites

This approach requires, in the first step, the synthesis of new 2,2'-biphosphole ligands with restricted configurations. The axial and central chiralities of the 2,2'-biphosphole framework could be controlled and limited by introducing a chiral linker between the two phosphorus atoms. Thus, we have investigated the introduction of enantiomerically pure carbon and alkoxy linkers in order to obtain, respectively, diphosphines and diphosphinites derived from 2,2'-biphosphole.

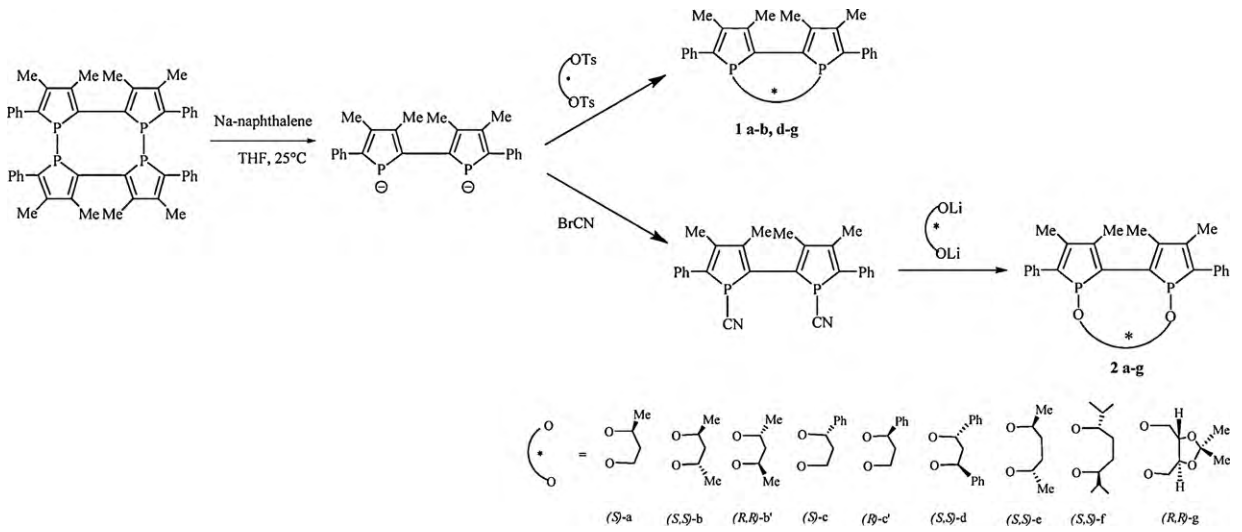
2.1. Diphosphines derived from 2,2'-biphosphole

Chiral stereochemically dynamic diphosphines [11] derived from 2,2'-biphosphole are prepared in two steps starting from the tetraphosphole (Scheme 2). In the first step, cleavage of the two phosphorus–phosphorus bonds using sodium naphtalene lead to the 2,2'-biphospholyl anion [13]. Then, the asymmetric alkylation in high dilution conditions using various enantiomerically pure diol ditosylates or dimesylates affords the expected diphosphines **1** (Scheme 2) in good yields (35 to 78%) as a mixture of three diastereoisomers among the six expected. The three diastereoisomers of each diphosphines **1** are fully characterized as disulfide derivatives since these diphosphines become stereorigid as disulfide derivatives, allowing structural and stereochemistry studies by X-ray diffraction analysis.

The stereochemical analysis of the different possibilities of combining axial and central chiralities in 2,2'-biphosphole shows the occurrence of six really inequivalent diastereoisomers (Fig. 1)[3].

[2] These diphosphanes are chirally flexible ligands because of the configurational instability of the axial chirality generated by the 2,2'-biphosphole framework and of the central chiralities at the phosphorus atoms.

[3] The different possibilities of combining axial and central chiralities in 2,2'-biphosphole lead to 8 diastereosiomers. Among these eight diastereosimers, six are really inequivalents because the four diastereoisomers with opposite configurations of the two phosphorus atoms are equivalent two by two. For more details on the stereochemical analysis [8].



Scheme 2. Synthesis of the diphosphines **1** and diphosphinites **2** derived from 2,2'-biphosphole.

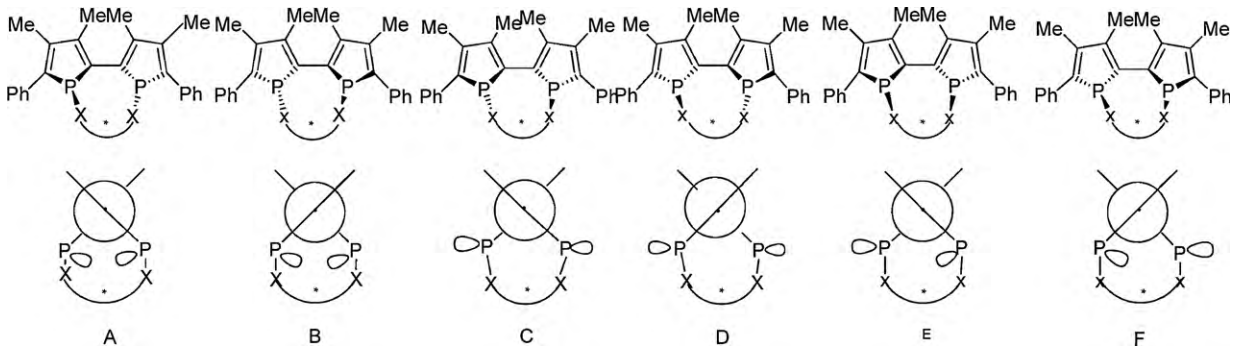


Fig. 1. The 6 diastereoisomers of diphosphines **1** or diphosphinites **2** corresponding to the different possibilities of combining axial and central chiralities of 2,2'-biphosphole framework. Each diastereoisomer is also represented in a Newman projection along the axis of the C–C linking the phosphole rings.

The introduction of a short chain between the two phosphorus atoms prevents the formation of the A and B diastereoisomers and favours the C, D, E and F forms. Indeed, the three diastereoisomers (C, D and E [or F] forms) are observed in solution as an equilibrium mixture at room temperature. The isomerization process, studied by desulfurization of diphosphine sulfides using a fully stereoselective method [14] that affords, at low temperature, chiral phosphines with retention of configuration, occurs at temperature below −60 °C. Since the chiral bridge prevents the formation of A and B forms, the only possible pathway for this isomerisation process requires a phosphorus-inversion[4] inducing atropo-inversion, the driving force being the pyramidal inversion barrier of the phosphorus atom as illustrated in Fig. 2.

2.2. Diphosphinites derived from 2,2'-biphosphole

The strategy to obtain chiral stereochemically dynamic diphosphinites **2**[5] derived from 2,2'-biphosphole is based on the introduction of an enantiomerically pure alkoxy linker between the two phosphorus atoms which involves a nucleophilic substitution reaction on 1,1'-dicyano-3,3',4,4'-tetramethyl-5,5'-diphenyl-2,2'-biphosphole **2** [13] by using enantiomerically pure diols. The synthetic pathway involves three steps starting from tetraphosphole **1** [12] as shown in Scheme 2. The first step leads to the 2,2'-biphospholyl anion which reacts in the next step with BrCN[6] to give the dicyano compound **2**. Treatment of this intermediate with the lithium derivative of diols **a–g** in high dilution conditions affords in the last step the air-stable diphosphinite compounds **2a–g** in high

[4] In phospholes, the inversion barrier for the pyramidal phosphorus is reduced, relative to that in phosphines, as a result of the increase in aromatic character of the phosphole in the transition state. The activation barrier to phosphorus inversion in 2,2'-biphosphole is measured to be only 16.5 kcal/mole leading to phosphorus inversion at −60 °C [8,10].

[5] The refinement of the Flack's parameter clearly indicates that these compounds are enantiomerically pure in solid state.

[6] Cyanogen bromide is toxic by inhalation of its vapors or by the hydrogen cyanide from decomposition.

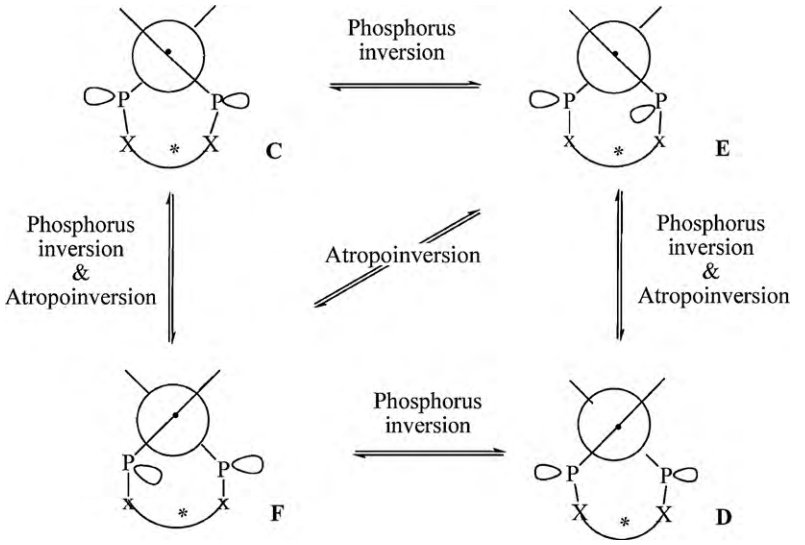


Fig. 2. Isomerization process of diphosphines **1** or diphosphinites **2**.

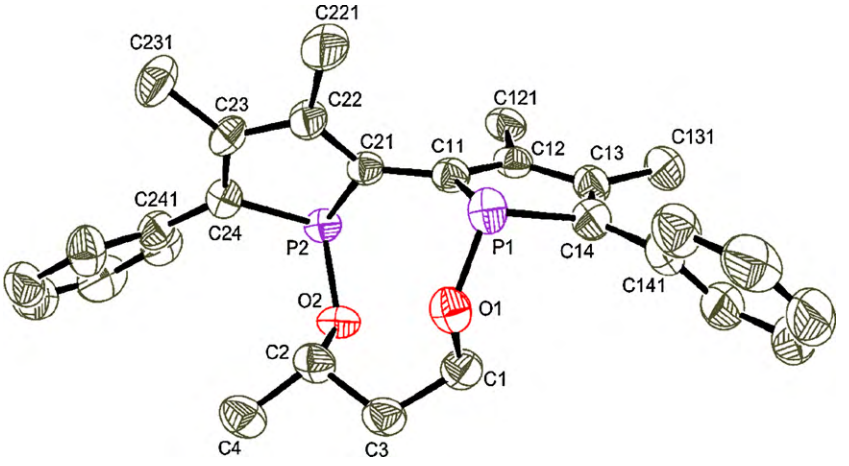


Fig. 3. Molecular view of *R[Sp,Sp,Sc]*- **2a** space group: $P2_12_12_1$, Flack's parameter: 0.06(14).

yields (71–86%). These compounds have been fully characterized and the molecular structures of **2a** and **2b'** have been established by X-ray diffraction. Compounds **2a** and **2b'** are enantiomerically pure in solid state according to X-ray diffraction analysis[7] [15] (Figs. 3 and 4). The absolute configuration is *R[Sp,Sp,Sc]* for compound **2a** and *S[Rp,Rp,Rc,Rc]* for compound **2b'** (axial configuration [phosphorus configuration, carbon configuration]).

It is worth mentioning the influence of the configuration of the starting diol on the axial and central configuration of the skeleton 2,2'–biphosphole in solid state as the *(S)* configuration provides the *R[Sp,Sp]* configuration of **2a** whereas the *(R,R)* configuration leads to the *S[Rp,Rp]* configuration of **2b'**.

Contrary to the diphosphines **1**, the diphosphinites **2** have been observed in solution by NMR as a single compound even within the temperature range −60 °C to +85 °C, corresponding probably to an equilibrium mixture of several diastereoisomers interconverting rapidly on the NMR time scale. Indeed, as the pyramidal inversion barrier of the phosphorus atom is not affected by π-donor substituents on the phosphorus [16], a similar isomerization process occurs rapidly in solution between the most four favoured diastereoisomers (C, D, E or F) of the diphosphinites **2** (Fig. 2).

These studies show that a partial chirality control of the axial and the central chiralities of 2,2'-biphosphole ligands can be achieved by introducing an enantiomerically pure linker between the two phosphorus atoms. In addition, this

[7] π-donor substituents on the phosphorus of phospholes do not affect the activation barrier according to experimental measurements and calculations.

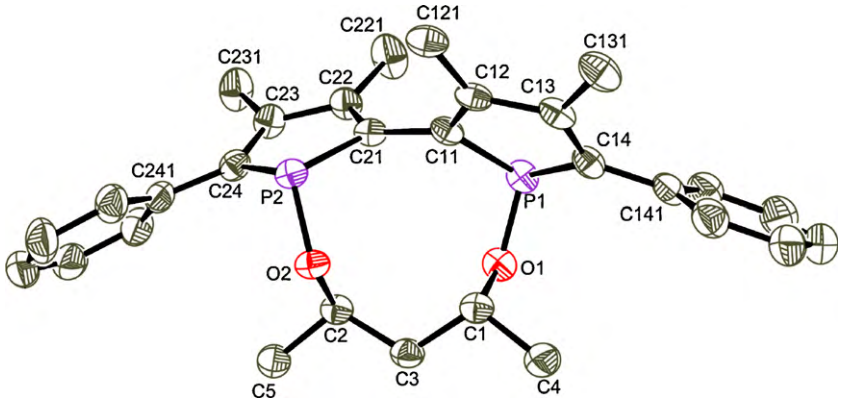
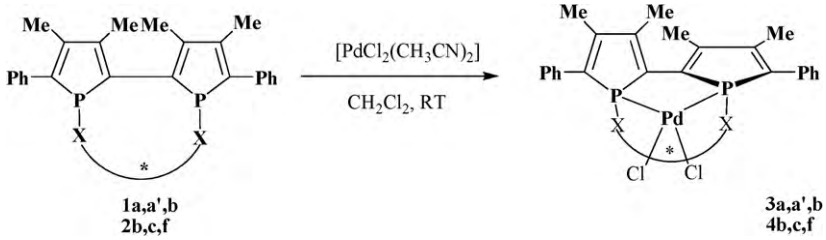


Fig. 4. Molecular view of *S[Rp,Rp,Rc,Rc]*-**2b'** space group: $P2_12_12_1$, Flack's parameter:-0.03(6).



Scheme 3. Synthesis of the palladium complexes.

partial control maintains some degree of freedom to accomplish a metal dynamic resolution.

3. Dynamic resolution by coordination to a transition metal

We have examined the dynamic chirality control of the axial and central chiralities upon coordination since the use of these stereodynamic ligands in asymmetric catalysis relies on obtaining enantiopure complexes.

3.1. Palladium complexes

Complexation of the diastereoisomeric mixture of equilibrating diphosphines **1a, 1a'** and **1b** with $[PdCl_2(CH_3CN)_2]$ in dichloromethane at room temperature produces $[PdCl_2$(diphosphine)] **3a, 3a'** and **3b** [12] respectively in excellent yield (90%) and an unidentified complex (10%)[8] in each case (Scheme 3).

Palladium complexes **3** are obtained in a pure diastereoisomeric form in solution according to ^{31}P NMR and no dynamic configuration change of the 2,2'-biphosphole chelated to palladium can be observed within the temperature range −60 °C to +85 °C.[9] According to these results, stabilization of the 2,2'-biphosphole framework chiralities has occurred by complexation as the result of the hindered inversion of the phosphorus configurations as well as axial configuration of 2,2'-biphosphole on metal center.

In addition, complexes **3a, 3a'** and **3b** are also enantiomerically pure in the solid state according to X-ray diffraction analysis [15]. Complexes **3a** and **3a'** [17], which differ in the configuration of atom C1 within the backbone linking the two P atoms, are enantiomers to each other. The molecular views of the these two enantiomers, *S[Sp,Rp,Rc]*-**3a** and *R[Rp,Sp,Sc]*-**3a'** (axial configuration [phosphorus configuration, carbon configuration]) are presented in Figs. 5 and 6.

According to these results, the chiral stereodynamic diphosphines **1** derived from 2,2'-biphosphole afford diastereo- and enantiopure complexes through a dual chirality control locked by palladium coordination. Structural studies by X-ray analysis reveal that the 2,2'-biphosphole adopts a single configuration in these palladium complexes in which the two phosphorus have opposite configurations, [*Sp,Rp*] or [*Rp,Sp*]. In addition, these analyses prove unambiguously the influence of the configuration of the carbon backbone on the axial and central configuration of the 2,2'-biphosphole skeleton in complexes **3a** and **3a'**, as the R configuration provides the *S[Sp,Rp]* configuration of **3a**, whereas the S configuration leads to the *R[Rp,Sp]* configuration of **3a'**.

In contrast, palladium-complexes of diphosphinites **2** cannot be as well structurally characterized. Treatment of diphosphinites **2b,c,f** with $[PdCl_2(PhCN)_2]$ leads to the complete consumption of the ligand to probably afford the corresponding $[PdCl_2$(diphosphinite)] **4b,c,f** (Scheme 3). But in each case, the Pd-complex gives a broad signal in ^{31}P and ^{1}H NMR even within the temperature range −70 °C to

[8] These unidentified products are palladium complexes with four diastereotopic phosphorus having opposite phosphorus configurations according to ^{31}P NMR.

[9] This lack of changes shows that the energy barrier for the stereochemical interconversion is higher than ~18 kcal/mol at 85 °C.

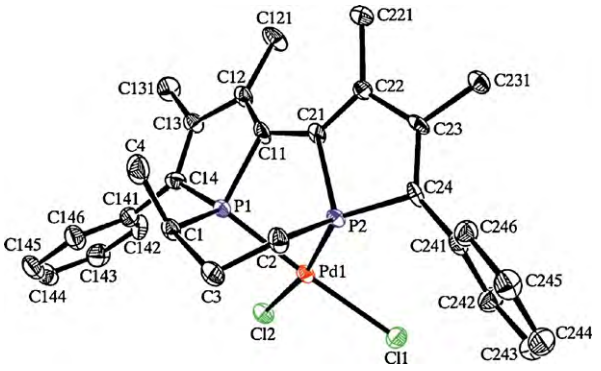


Fig. 5. Molecular view of *S[Sp,Rp,Rc]*-**3a** space group $P2_1$, Flack's parameter 0.03(5).

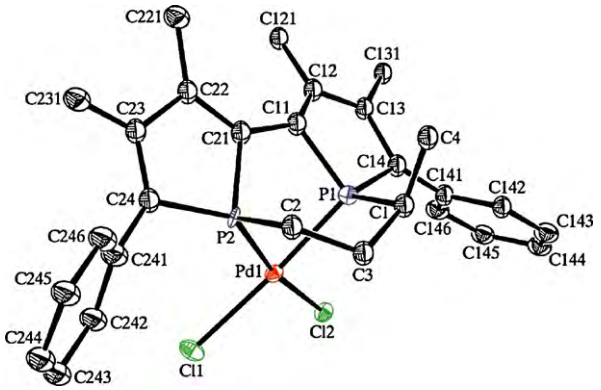


Fig. 6. Molecular view of *R[Rp,Sp,Sc]*-**3a'** space group $P2_1$, Flack's parameter -0.02(11).

+85 °C which can be explained by an aggregation phenomena in solution.[10] Unfortunately, no structural determinations by X-ray analysis can be performed.

3.2. Platinum complexes

Enantiopure platinum (II) complexes were also synthetized with chiral stereodynamic diphosphines **1** and diphosphinites **2** [18].

Diphosphines **1b,e,g** react with $[PtCl_2(CH_3CN)_2]$ in 24 h in dichloromethane at room temperature to quantitatively afford a diastereomerically pure complex **5b,e,g** (Scheme 4) as evidenced by ^{31}P NMR spectra. Complexes **5b,e,g**, are also enantiomerically pure in solid state as confirmed by X-ray analysis performed on two of these three complexes [18]. The molecular view of complex **4b'** is presented in Fig. 7. The configuration of the ligand **1b'** in the platinum complex, described as *S[Rp,Sp,Sc,Sc],* reveals again the opposite configuration for the two phosphorus atoms.

Diphosphinites **2a–g** are more reactive towards $[PtCl_2(CH_3CN)_2]$ than diphosphines as the complete consumption of the ligand is obtained in 3 h at room temperature in dichloromethane (Scheme 4). Contrary to diphosphine-complexes **5**, these platinum complexes give broad signals in ^{31}P and ^{1}H NMR, except for complexes **6d** and **6f**, even within the temperature range −70 °C to +85 °C. The well-resolved ^{31}P NMR spectra of complexes **6d** and **6f** clearly indicate the formation of a pure diastereoisomer as in the case of diphosphine complexes **5**. In addition, X-ray structural determinations of **6f** [18] reveal the formation of an enantiopure complex (Fig. 8).

As already observed in others complexes, the two phosphorus atoms have opposite configuration [*Sp,Rp*] in complex **4f**.

3.3. Rhodium complexes

Dynamic chirality control of diphosphines **1** and diphosphinites **2** can be also obtained by coordination to the rhodium centre. For example, reaction of diphosphine **1b** and diphosphinites **2a,d** with $[Rh(COD)_2]BF_4$ or $[Rh(COD)_2]CF_3SO_3$ (Scheme 5) in dichloromethane quantitatively leads, in each case, to the formation of a diastereomerically pure complex, **7b** and **8a,d**, respectively, as evidenced by $^{31}P\{^{1}H\}$ NMR. Unfortunately, these rhodium complexes give a red hygroscopic and water sensitive powder after evaporating the solvent and all attempts to purify this complex failed. No single crystals could be obtained.

However, as complexing to a metal centre locks both the central phosphorus configuration and the axial configurations of 2,2'-biphosphole, the rhodium complexes **7** and **8** are obtained as a diastereo- and enantiopure complex.

4. Enantioselective catalytic reactions

To demonstrate the potential of our stereodynamic ligands for enantioselective catalysis, we studied Pd-catalyzed allylic substitution, Pt-catalyzed hydroformylation and Rh-catalyzed hydrogenation reactions.

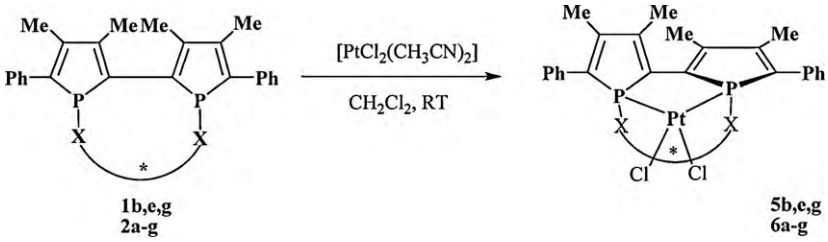
4.1. Asymmetric allylic substitution

We screened various palladium catalysts in the allylic substitution of 1,3-diphenylprop-2-enyle acetate, which is widely used as a model substrate, by the dimethylmalonate anion (Scheme 6). The reaction is carried out in dichloromethane at room temperature in the presence of a catalyst generated *in situ* from 1 mol% of the corresponding ligand, 0.5 mol% of π-allyl-palladium chloride dimer $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$.

All of the diphosphines **1** and diphosphinites **2** [11,15] ligands give palladium catalytic systems that are active in allylic substitution producing the corresponding allylic substitution product with enantiomeric excess in the range of 4 to 66%. The best stereoselectivity is achieved with the palladium catalyst derived from diphosphine **1d**.

The stereoselectivities are, however, moderate but these results demonstrate the direct use of the stereodynamic diphosphines and diphosphinites to convey enantioselectivity in metal-catalyzed reaction through an *in situ* dual chirality control.

[10] The broadness of the line can be explain by a short T2 relaxation associated to an aggregation phenomena in solution as no dynamic effect have been observed by variable temperature experiment.



Scheme 4. Synthesis of the platinum complexes.

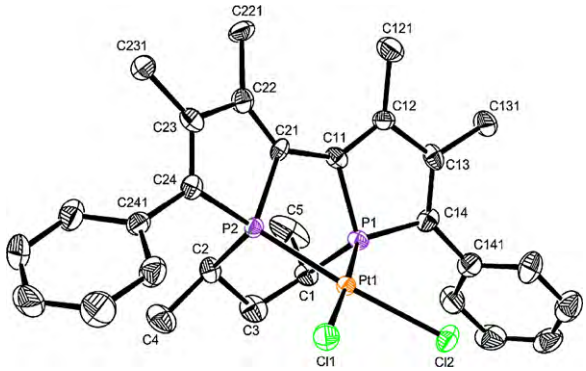


Fig. 7. Molecular view of *S[Rp,Sp,Sc,Sc]*-**3b'** Space group : $P2_1$, Flack's parameter : 0.04(2).

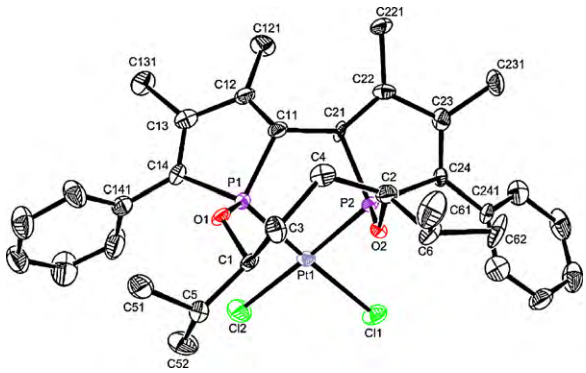
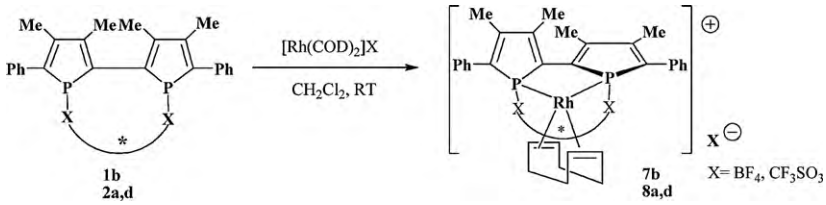


Fig. 8. Molecular view of complex *S[Sp,Rp,Sc,Sc]*-**4f** Space group : $P2_1$, Flack's parameter : 0.013(8).

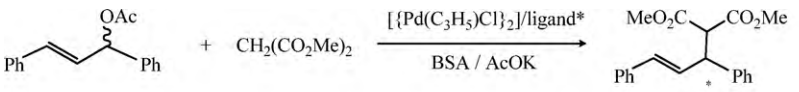
4.2. Asymmetric hydroformylation

The enantiopure platinum complexes of diphosphines and diphosphinites are examined in the asymmetric hydroformylation of styrene [18], which is an attractive catalytic approach to the synthesis of the 2-arylpropionic acid type of anti-inflammatories (Scheme 7).

In this case, experiments are performed using the preformed complexes [$PtCl_2$(**L***)] **5** and **6** with anhydrous $SnCl_2$ as co-catalyst at room temperature in dichloromethane under 100 atm of CO:H_2 (1:1). Excellent chemoselectivities are observed with both diphosphine and diphosphinite complexes. In addition, high regioselectivities are recorded in each case giving predominantly

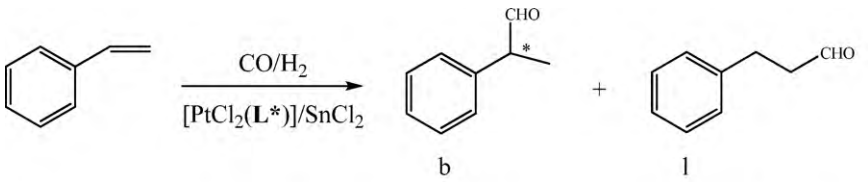


Scheme 5. Synthesis of the rhodium complexes.



L*	ee (%)	L*	ee (%)
1a	14 (*S*)	**2a**	20 (*S*)
1b'	50 (*S*)	**2b'**	35(*R*)
1d	66 (*R*)	**2d**	11 (*S*)
1e	5 (*R*)	**2e**	10 (*S*)
1f	16 (*S*)	**2f**	10 (*R*)
1g	4 (*R*)		

Scheme 6. Allylic substitution of 1,3-diphenylprop-2-enyle acetate by the dimethylmalonate anion.



[PtCl$_2$(L*)]	b/l	ee (%)	[PtCl$_2$(L*)]	b/l	ee (%)
5b	83/17	18 (*R*)	**6c'**	79/21	17(*S*)
5e	83/17	/	**6e**	87/13	/
5g	91/9	12 (*R*)	**6g**	81/19	/

Scheme 7. Hydroformylation of styrene.

the branched aldehydes (76–91%). However, low activities and enantioselectivities are obtained with both diphosphine and diphosphinite complexes at room temperature (up to 18%).

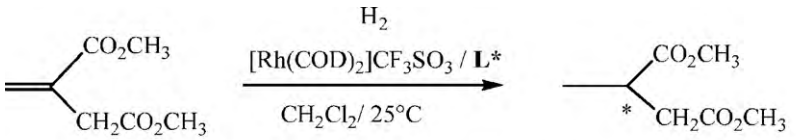
4.3. Asymmetric hydrogenation

Finally, we have evaluated the catalytic performance of the diphosphines **1** and diphosphinites **2** in the asymmetric hydrogenation of dimethyl itaconate [17] (Scheme 8).

We used catalysts prepared *in situ*[11] by adding the ligand **1** or **2** to [Rh(COD)$_2$]CF$_3$SO$_3$.[12] All of the ligands give rhodium catalytic systems that are active in hydrogenation of dimethyl itaconate producing enantiomeric excess in the range of 31 to 85%. Diphosphines **1** give catalytic systems that are more active as complete conversion is achieved in 3 hours at room temperature under an hydrogen pressure of 20 bar whereas diphosphinites **2** require an hydrogen pressure of 40 bar during 6 hours to reach a complete conversion. The best result in terms of enantioselectivities is obtained with diphosphinites **2c** and **2d**. The enantiomeric excess values are strongly influenced by the nature of the backbone whereas the sense of the enantioselection is determined by the configuration of the stereocentres as observed in asymmetric allylic substitution. The *(R)* enantiomer of the methylsuccinate is obtained with ligands **1–2** (**a**, **b**, **c**, **d**, **e)** derived from the *(S)* or *(S,S)* diols whereas the *(S)* enantiomer is obtained with the ligand **1–2 (b'**, **2c')** derived from the *(R)* or *(R,R)* diols. The enantioselectivity observed in the hydrogenation of dimethyl itaconate is likely to be governed by the electronic properties rather than the steric factors of the ligand.

[11] Experiments carried out by catalytic systems formed in situ provide essentially the same enantioselectivitiy values as those obtained from preformed complexes.

[12] In situ catalyst were prepared by adding 1.4 eq. of ligand to [Rh(COD)$_2$]CF$_3$SO$_3$. In the absence of free ligand, demetallation was observed in each case.



L*	ee (%)	L*	ee (%)
1a	15 (*R*)	**2a**	40 (*R*)
1b	29 (*R*)	**2b**	58 (*R*)
1b'	30 (*S*)	**2b'**	60 (*S*)
		2c	85 (*R*)
		2c'	83 (*S*)
1d	32 (*R*)	**2d**	82 (*R*)
1e	30 (*R*)	**2e**	60 (R)

Scheme 8. Asymmetric hydrogenation of dimethyl itaconate.

5. Conclusion

Stereodynamic chiral diphosphines and diphosphinites derived from 2,2'-biphosphole have been synthetised by a versatile methodology. By the introduction of a relatively simple backbone between the two phosphorus, the axial and P-chiralities can be controlled and locked upon coordination to a metal centre to afford diastereo- and enantiopure palladium, platinum or rhodium complexes.

These stereodynamic ligands have been evaluated in asymmetric Pd-catalyzed allylic substitution, Pt-catalyzed hydroformylation and Rh-catalyzed hydrogenation.

Notable features are the direct use of these stereodynamic diphosphinites to convey enantioselectivity in Rh-catalyzed hydrogenation through an *in situ* dual chirality control.

The modular construction of these stereodynamic ligands by combination of the 2,2'-biphosphole framework and chiral linker offers immense scope for structural variations and catalysts tuning. The application of the stereodynamic chiral diphosphines and diphosphinites derived from 2,2'-biphosphole in a broad range of rhodium group metal-catalyzed asymmetric transformations are currently in progress.

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