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Unusual behavior of *o,o´*-dimethylene(tri-*p*-cresyl) bicyclophosphite as a novel organophosphorus ligand in olefin hydrogenation and hydroformylation*

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A representative of the new class of organophosphorus ligands, *viz.*, *o,o´*-dimethylene(tri-*p*-cresyl) bicyclophosphite (BCP), was studied as a promoter of $Rh(acac)(CO)_2$ in hydrogenation and hydroformylation. BCP enhances the activity and stability of the catalyst much more strongly than analogous organophosphorus ligands used previously (triphenylphosphine, triphenyl phosphite, and etriolphosphite). A reason for this behavior of BCP was studied using NMR spectroscopy, quantum-chemical calculations, and molecular simulation. The high sensitivity of the ^1H NMR signals of the methylene groups of BCP toward complexation appears due to the high density of the highest occupied and lowest unoccupied MO of protons of the CH_2 groups, especially those directed toward the P atom. The ^1H and ^{31}P NMR spectra indicate the formation of hydrides of two types ($HRh(BCP)_3$ and $HRh(BCP)_4$) directly upon the addition of BCP in amounts exceeding that corresponding to the BCP/Rh = 2 ratio to a solution of $Rh((acac)(CO)_2$. The most probable source of the hydride ion is the BCP molecule itself, namely, the bridging CH_2 groups. The molecular mechanics simulation showed that in the $[Rh(BCP)_3]^+$ complexes the aromatic rings of BCP formed two molecular cavities. These cavities can alternatively open and close, thus providing flexible screening of the catalytic site. This explains the unusual behavior of the Rh complexes with BCP in hydrogenation and hydroformylation.

Key words: bicyclophosphite, $Rh(acac)(CO)_2$, ^1H NMR spectroscopy, ^{31}P NMR spectroscopy, hydrides, catalysis, hydrogenation, hydroformylation.

Organophosphorus ligands (L), such as triphenyl phosphite and etriolphosphite $P(OCH_2)_3CEt$, are well known as promoters in hydrogenation and hydroformylation catalyzed by the rhodium catalysts.[1—3]

The catalytic activity of the $Rh(acac)(CO)_2$—*n*L systems correlates[4—7] with frequencies of CO vibrations, chemical shifts in the ^{31}P NMR spectra, and Rh—P spin-spin coupling constants (SSCC). The Tolman parameter (θ) that quantitatively estimates the steric state of substituents at the P atom is also used as a characteristic of the P ligands in these systems.[8—10]

The new organophosphorus promoter *o,o´*-dimethylene(tri-*p*-cresyl) bicyclophosphite (BCP, **1**) has recently been synthesized.[11,12]

According to the X-ray diffraction data,[11] the BCP molecule in crystal has an asymmetrical conformation, forming a conic molecular cavity. Some parameters of the organophosphorus compounds used in this work are presented below.

Comparative analysis of changes in the frequencies of CO vibrations, chemical shifts in the ^{31}P NMR spectra, and spin-spin coupling constants shows that the promoting ability of BCP in catalysis should be close to the ability of usually used neutral organic phosphites. How-

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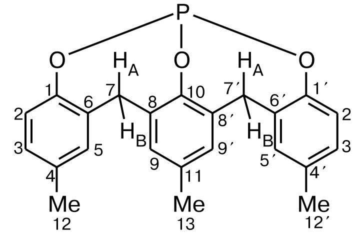

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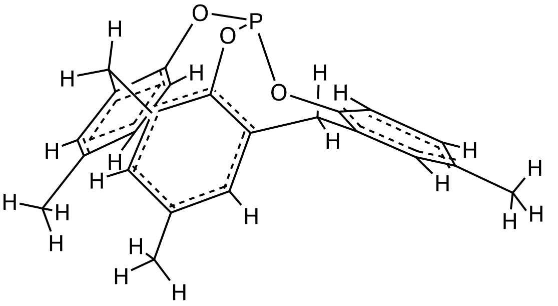


Parameter	PPh_3	$P(OPh)_3$	BCP	$P(OCH_2)_3CEt$
ν(CO)/cm^{-1} for Rh(acac)(CO)L	1975	1995	2000	2008
SSCC $^1J_{P,Rh}$/Hz for Rh(acac)(CO)L	175.3	297.4	294.2	287.7
Coordinational shift $\delta_{^{31}P}$	36.6	−7.8	7.9	22
Tolman parameter (θ)/deg	145	128	137	101

ever, the Tolman parameter for BCP is close to that for triphenyl phosphite. The molecular cavity is also known[10] to affect the catalytic activity.

In this report, we present the data on the unusual behavior of BCP in styrene hydrogenation and but-2-ene hydroformylation, which are explained by the results of the NMR study of the complexation of BCP with $Rh(acac)(CO)_2$ and molecular simulation.

Experimental

The organic phosphites and Rh complexes were synthesized as described previously.[11,13–15] Styrene was hydrogenated according to a known procedure.[16] The catalyst precursor was prepared in air by the addition of the corresponding organophosphorus ligand (in a necessary ratio) to a solution of $Rh(acac)(CO)_2$ ($4.3 \cdot 10^{-5}$ mol) in DMF (5 mL). The resulting solution was activated by $NaBH_4$ (1 mg) for 5 min. The catalyst precursor was placed in a glass reactor, through which a fivefold volume of H_2 was passed, and then styrene was added (with a syringe through a septum) at the styrene/Rh ratio equal to 100. Hydrogenation was carried out at an atmospheric pressure and 323 K. The reaction rate was measured from the volume of absorbed hydrogen. The reaction products were identified by GLC.

But-2-ene was hydroformylated in a stainless steel autoclave equipped with a stirrer. An equimolar mixture of CO and H_2 at 6 MPa and 363 K was used. The concentration of Rh in the liquid phase was $3 \cdot 10^{-4}$ mol L^{-1}. The reaction rate was measured by a decrease in the synthesis-gas pressure in a calibrated vessel. The reaction products were identified by GLC.[13–15]

NMR spectroscopy. NMR spectra were recorded on Bruker CXP-200 and Bruker AC-250 spectrometers at 298 K, unless otherwise specified. Chemical shifts were determined from the ratio of signals of deuterons of the solvent and recalculated to the scale relatively to Me_4Si (^1H) or 85% H_3PO_4 (^{31}P). Solutions for studies by NMR spectroscopy were prepared in air. Reference experiments with solutions obtained in a nitrogen atmosphere revealed no difference.

Molecular simulation. Calculations were performed using the HyperChem 5$^{(TM)}$ program. The optimization of the BCP molecule by semiempirical quantum-chemical calculations[17] (AM1) gave the structure well consistent with the X-ray diffraction data. This structure (with the unchanged distribution of atomic charges) was used in the simulation of the Rh complexes with BCP by the molecular mechanics method (MM$^+$ force field). The charge distribution in the Rh—H fragment was found by the *ab initio* calculation using the Hartree—Fock method in the STO-4G* basis set.

Results and Discussion

Catalysis. The rhodium system (under the conditions described) is inefficient in the hydrogenation of styrene to ethylbenzene when triphenylphosphine is added. Among organic phosphites, triphenyl phosphite turned out to be most efficient (at the ratio P/Rh = 1, Fig. 1), whereas etriolphosphite slightly decreases the activity of unmodi-

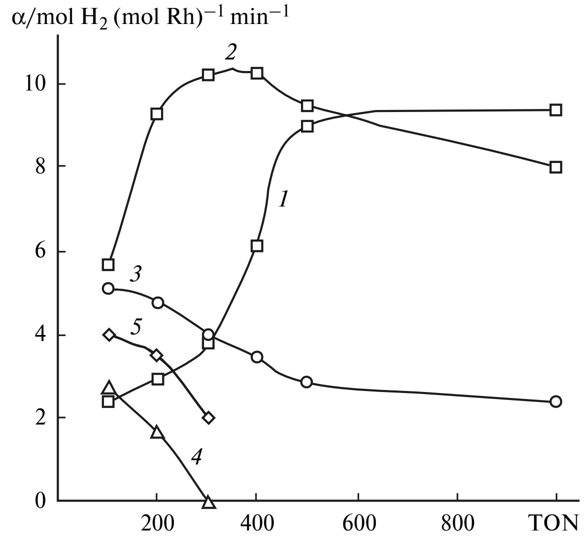


Fig. 1. Plots of the catalytic activity of $Rh(acac)(CO)_2$—nL in styrene hydrogenation *vs.* turnover number of the catalyst (TON, number of moles of the hydrogenated substrate per mole of the catalyst): BCP at P/Rh = 1 (*1*) and 5 (*2*), triphenyl phosphite (*3*), etriolphosphite (*4*), and without a P promoter (*5*).

fied Rh(acac)$(CO)_2$. The activities of the catalysts modified by triphenyl phosphite and etriolphosphite decrease in time, which is usually explained by the oxidation of phosphites to phosphates[18] and formation of clusters[19] (in fact, organic phosphates were found among the reaction products).

The initial effect of modification by minor amounts of BCP (P/Rh = 1, see Fig. 1) is negative analogously to the effect of etriolphosphite. However, the promoting activity of BCP increases during the reaction. It turned out to be equal to the activity of triphenyl phosphite at the turnover number (number of moles of the hydrogenated substrate per mole of the catalyst, TON) equal to 300 and continues to increase, despite the partial oxidation of BCP to phosphate (the corresponding product was identified by ^{1}H and ^{31}P NMR spectroscopy). These data indicate indirectly that cluster formation processes are absent for this catalyst.

Cluster formation can be prevented by an excess of the promoting P ligand. However, its effect usually results in blocking of the reaction sites, so that the activity of the catalysts promoted by triphenyl phosphite and etriolphosphite decreases at high P/Rh ratios.

Surprisingly, the behavior of BCP is opposite again: the addition of a fivefold BCP excess increased sharply the catalytic activity, which increases in time and exceeds the activity of the catalyst promoted by triphenyl phosphite.

These facts indicate that BCP reacts slowly with Rh(acac)$(CO)_2$ to form a catalytic complex with the unusually high activity and stability. In this complex, the active sites are not blocked by ligand excess.

Blocking of the reaction sites is very important in hydroformylation when the high selectivity can be achieved only at high P/Rh ratios. For example, for the hydroformylation of but-2-ene, the selectivity with respect to 2-methylbutanal achieves ~100% at P/Rh ≈ 9 (for all ligands studied including PPh_3).[13]

However, an increase in the P/Rh ratio, as a rule, decreases simultaneously the reaction rate, resulting in the appearance of a maximum in the plot of the rate *vs.* P/Rh ratio (Fig. 2). The system with triphenyl phosphite is characterized by a maximum at the comparatively low ratio P/Rh = 2. The reaction rate decreases considerably at higher ratios. This optimum is usually explained by cluster formation at low P/Rh ratios and blocking of the reactions sites at a high concentration of the P promoter (resulting in the CO removal from the coordination sphere of Rh).[13,19]

The behavior of BCP differs again from that of other ligands: the reaction rate remains constant regardless of the P/Rh value, at least unless P/Rh = 9 is achieved, which corresponds to the ~100% selectivity with respect to 2-methylbutanal. The considerable catalytic effect of BCP even at low P/Rh ratios indicates that cluster formation is efficiently hindered. The reaction rate independent of the P/Rh ratio indicates that the reaction sites are not blocked even at high excess of the promoting ligand. Thus, the catalytic complex involving BCP differs from complexes formed by other P promoters.

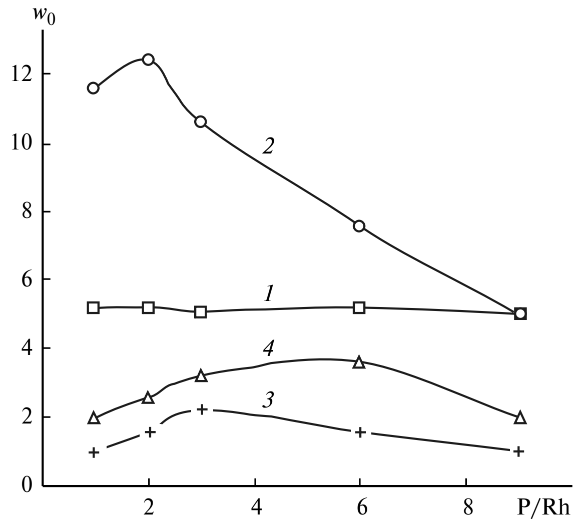


Fig. 2. Plots of the catalytic activity of Rh(acac)$(CO)_2$—*x*L in the hydroformylation of but-2-ene *vs.* P/Rh molar ratio: BCP (*1*), triphenyl phosphite (*2*), triphenylphosphine (*3*), and etriolphosphite (*4*); w_0 is the initial hydroformylation rate in mmoles of absorbed synthesis-gas (CO/H_2) for 1 min.

Complexation studied by ^{1}H and ^{31}P spectroscopy. In order to elucidate the role of the molecular cavity and other properties of BCP, we studied the complexation of BCP with Rh(acac)$(CO)_2$.

The ^{1}H NMR spectrum of BCP (Fig. 3) exhibits two doublets of the CH_2 protons split by the geminal coupling constant (*J* = 13.8 Hz). They belong to nonequivalent protons A and B (structure **1**). However, the CH_2 groups themselves are equivalent, *i.e.*, conformational transformations in this nonsymmetrical structure are rather fast. The spectra recorded specially at low temperatures in acetone-d_6 (down to 203 K, 250 MHz) manifest no properties of signal separation. Thus, the structure of the BCP molecule is strained (the H—C—H angle in the CH_2

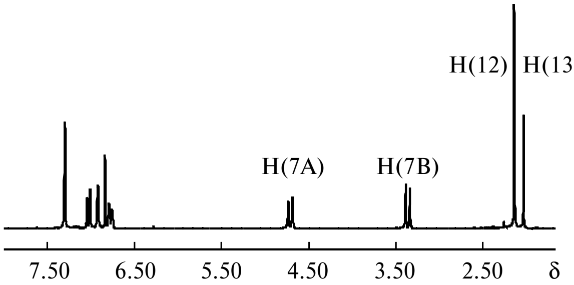


Fig. 3. ^{1}H NMR spectrum (250 MHz) of a solution of BCP in benzene-d_6.

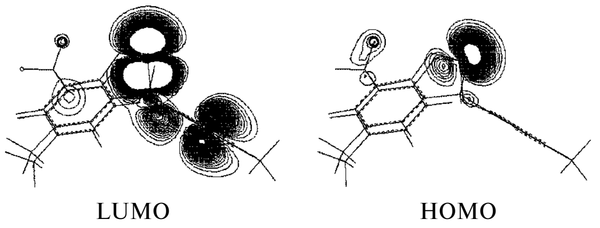


Fig. 4. Electron densities (AM1 calculation) of the lowest unoccupied and highest occupied MO in the BCP molecule (cross section by the HCH plane of one of the bridging CH_2 groups).

groups is ~106° according to the results of AM1 calculations) and simultaneously mobile.

The signals of protons of the CH_2 and CH_3 groups in the BCP molecule are very sensitive to complexation. Note that the signal of the H(7A) atoms (see Fig. 3) is selectively broadened. This broadening seems to be related to the unresolved interaction with the ^{31}P nucleus.

The quantum-chemical calculations (AM1) show that the highest occupied and lowest unoccupied MO are mainly concentrated on the P atom. However, a significant density of these orbitals is also observed on the CH_2 protons and mainly on the proton directed toward the P atom (H(7A), Fig. 4). Any complexation involving these orbitals should affect the electronic density on the CH_2 protons. This explains the considerable broadenings and shifts of the corresponding signals in the ^1H NMR spectra observed during complexation with $Rh(acac)(CO)_2$.

The ^{31}P NMR spectrum of BCP is a singlet (δ 103.6). On mixing of equimolar amounts of solutions of BCP and $Rh(acac)(CO)_2$, CO is evolved. The ^{31}P NMR spectrum (recorded after the completion of this reaction) contains a doublet corresponding to Rh(acac)(CO)BCP ($\delta = 111.5$, $^1J_{Rh,P} = 295$ Hz).

The further addition of BCP results in the spectrum containing doublet signals of Rh(acac)(CO)BCP and $Rh(acac)(BCP)_2$ ($\delta = 114.9$, $^1J_{Rh,P} = 307$ Hz) and multiplets at δ 125—135 (Fig. 5, *a*). The latter are doublets of doublets at δ 130.5 and 132.8 ($^1J_{Rh,P} = 229$ and 238 Hz and $J_{P,H} = 44$ and 14.6 Hz, respectively) (Fig. 5, *b*). These protons (responsible for additional splitting of the ^{31}P signals at 125—135 ppm) give two multiplets in the ^1H NMR spectrum (Fig. 6) in the region of ~−10 ppm typical[20] of Rh hydrides.

The ^1H NMR spectra of these hydrides (see Fig. 6) are interpreted as follows: (1) a quartet of doublets at δ −9.6 with quartet splitting $J_{P,H} = 14.6$ Hz and doublet splitting $J_{Rh,H} = 4.9$ Hz, (2) a quintet of doublets at δ −10.4 with quintet splitting $J_{P,H} = 44$ Hz and doublet splitting $J_{Rh,H} = 7.5$ Hz. The splittings remain unchanged at resonance frequencies of 200 and 250 MHz.

The quartet and quintet splittings indicate the formation of two types of rhodium hydrides with three and four BCP molecules bonded to Rh, *i.e.*, $[HRh(BCP)_3]$ and $[HRh(BCP)_4]$.

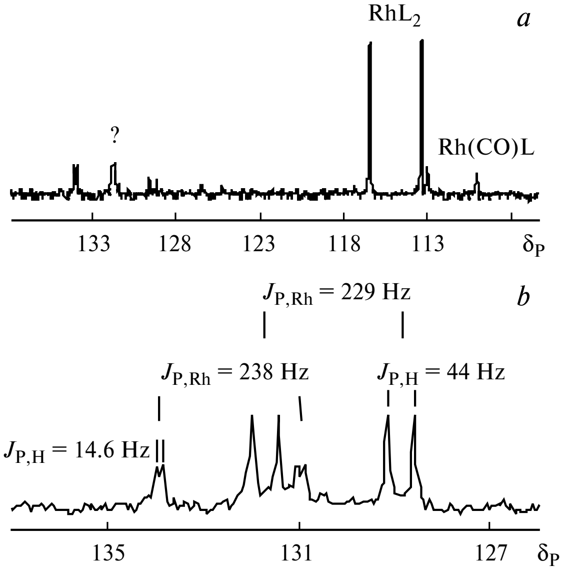


Fig. 5. ^{31}P NMR spectra (without ^1H decoupling) of a mixture of solutions of BCP and $Rh(acac)(CO)_2$ at the ratios P/Rh = 2.2 (*a*, 101 MHz, benzene-d_6) and P/Rh = 3 (*b*, 81 MHz, toluene-d_8).

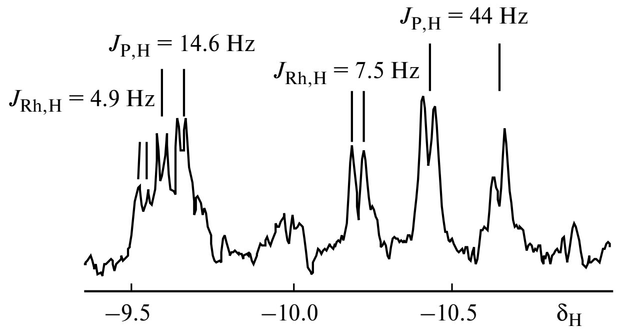


Fig. 6. ^1H NMR spectrum (200 MHz) of a mixture of solutions of BCP and $Rh(acac)(CO)_2$ at the ratio P/Rh = 3 (81 MHz) in the region of signals from hydrides (toluene-d_8 as a solvent).

The ^1H and ^{31}P NMR spectra of these complexes are well resolved, while other signals are often broadened due to the ligand exchange. Thus, the hydrides obtained are kinetically stable. The $HRh(BCP)_4$ complex contains 18 electrons in the valent shell and, hence, an enhanced stability of this compound should be expected. The $HRh(BCP)_3$ hydride has a 16-electron structure and, therefore, should be less stable.

The ^1H NMR spectra of solutions with the ratio P/Rh ≈ 2 contain well resolved sets of doublets of the CH_2 groups, which were assigned[17] to the following complexes: Rh(acac)(CO)L (**2**), $Rh(acac)L_2$ (**3**), $HRhL_3$ (**4**), and $HRhL_4$ (**5**). This assignment makes it possible to monitor the change in the parameters of the ^1H and ^{31}P spectra with successive filling of the coordination sphere of Rh with the BCP molecules and formation of hydrides (Fig. 7). The ^{31}P chemical shifts increase consecutively

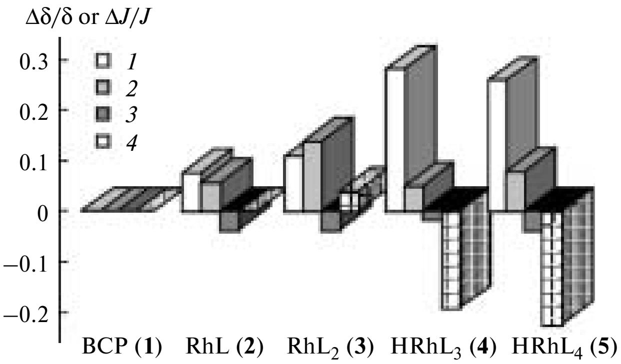


Fig. 7. Relative changes (with respect to BCP) in the NMR parameters observed by successive filling of the coordination sphere of Rh and formation of hydrides: δ_P (*1*), δ_H (H(7A)) (*2*), J_{gem} (*3*), and $J_{P,Rh}$ (*4*).

upon coordination with the Rh atom and hydride formation (a slight decrease is observed when $HRh(BCP)_3$ is transformed into the 18-electron $HRh(BCP)_4$ complex). The chemical shift of the H(7A) atoms increases significantly (by ~1 ppm) during coordination but decreases to low values for hydride formation. The geminal interaction constant changes upon coordination and, probably, upon the formation of the $HRh(BCP)_3$ hydride. The $^1J_{Rh,P}$ spin-spin coupling constant changes slightly on going from Rh(acac)(CO)BCP to $Rh(acac)(BCP)_2$ but changes substantially upon the formation of hydrides.

These results make it possible to explain qualitatively the described behavior of BCP during catalysis. According to the NMR experiments in a nitrogen atmosphere, the hydrides are formed only at high BCP excess (P/Rh > 2). The low initial activity of BCP at P/Rh = 1 can be explained by the absence of hydrides. An increase in the activity in time indicates that the reaction of hydride formation is slow and the P/Rh ratio can increase in time (for example, due to the removal of Rh from the system upon cluster formation). In fact, when BCP is added in great amounts and the appearance of hydrides becomes possible (P/Rh = 5, see Fig. 1), the initial catalytic activity is high.

Reasons for the existence of the P/Rh ratio critical for hydride formation and the absence of reaction site blocking by BCP excess remain unclear. The rule of 18 electrons explains only the relative stability of hydride with four BCP molecules. The accessibility of Rh for substrates in these complexes (especially for hydroformylation) should be clarified by the determination of the structures of these hydrides. Our attempts to isolate crystals of hydrides, which were found *in situ*, were unsuccessful up to now. However, some information can be obtained by molecular simulation.

Molecular simulation. Two BCP molecules can easily substitute two carbonyl groups, remaining acetylacetonate bonded to Rh. However, when the third BCP molecule adds to Rh and the corresponding hydride is formed, the $acac^-$ anions should leave the first coordination sphere of Rh. When the $[Rh(BCP)_3]^+$ cation is considered as the precursor of the corresponding hydride, the latter can be formed only at BCP excess.

The optimization of the $[Rh(BCP)_3]^+$ cation by the molecular mechanics methods gives the highly symmetrical structure (Fig. 8). This structure contains two cavities near the Rh atom. Three H atoms of the CH_2 groups are sufficiently close to the Rh atom (at a distance of ~3.88 Å), facilitating the transfer of the hydride ion from BCP to Rh.

According to the AM1 calculations, the removal of H^- from the BCP molecule requires an energy expense of

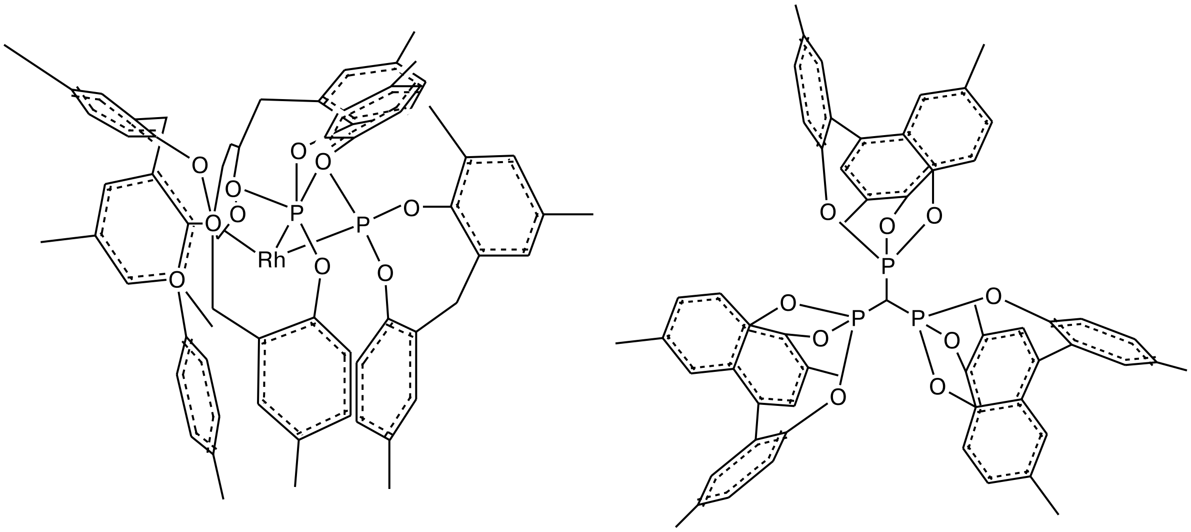
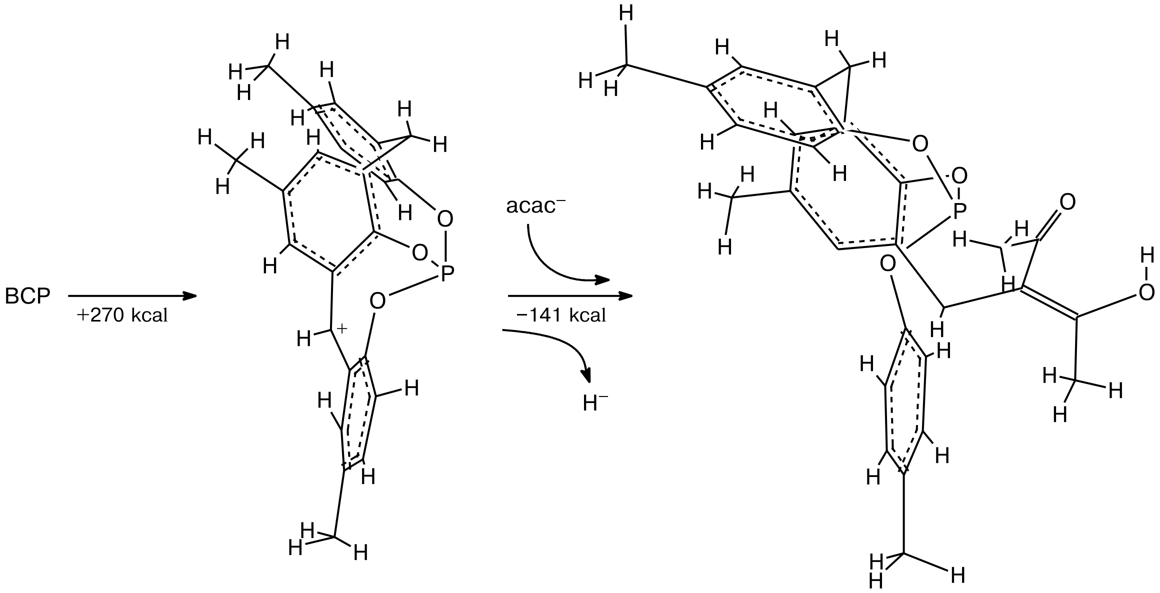


Fig. 8. Arrangement of the BCP molecules in the $[Rh(BCP)_3]^+$ complex obtained by geometry optimization using the molecular mechanics method.

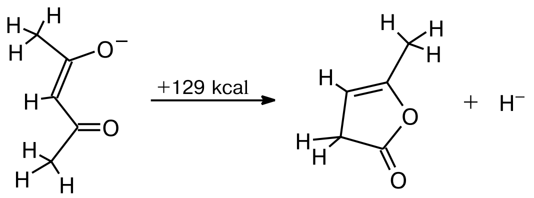
Scheme 1



~270 kcal mol^{-1}. However, an energy of 141 kcal mol^{-1} is compensated by the recombination of BCP$^+$ and acac$^-$ (Scheme 1).

Thus, the total energy required for the removal of the hydride ion *via* this mechanism (compensated by the formation of Rh hydride) is ~129 kcal mol^{-1}. An alternative source of the hydride ion is the acac$^-$ anion. The removal of the hydride ion from the acac$^-$ anion results, *e.g.*, in lactone (Scheme 2).

Scheme 2



In this case, the required energy is also 129 kcal mol^{-1} (AM1 calculation). However, hydride would also be removed from the acac$^-$ anion in the case of such P ligands as triphenyl phosphite. We never observed signals from hydrides in the spectra of $P(OPh)_3$ and PPh_3. Therefore, the most probable source of hydride is the CH_2 group in the BCP molecule.

The processes occurred upon addition of BCP to $Rh(acac)(CO)_2$ can be presented by Scheme 3.

The substitution of the carbonyl groups is likely a reversible reaction that occurs *via* the associative mechanism. We observed signals from several intermediate forms when the reaction was carried out in a tightly closed tube. Two doublets (δ 124.7, $^1J_{\mathrm{Rh,P}}$ = 266 Hz and δ 121.8, $^1J_{\mathrm{Rh,P}}$ = 244 Hz) in the ^{31}P NMR spectrum can likely be attributed to $Rh(acac)(CO)_2(BCP)_n$ (n = 1 or 2), and the BCP molecules are always in axial positions. These signals disappear when the tube is open, giving the doublet of Rh(acac)(CO)(BCP). The further addition of BCP affords hydrides (Scheme 4).

Scheme 3

$$\mathrm{acacRh(CO)_2} \xrightleftharpoons[-\mathrm{CO}]{+\mathrm{BCP}} \mathrm{acacRh(CO)BCP} \xrightleftharpoons[-\mathrm{CO}]{+\mathrm{BCP}}$$

$$\rightleftharpoons \mathrm{acacRh(BCP)_2}$$

Scheme 4

$$\mathrm{Rh(acac)(BCP)_2} \xrightleftharpoons{+\mathrm{BCP}} \mathrm{Rh(acac)(BCP)_3} \xrightarrow[-[\mathrm{BCP^+acac^-}]]{+\mathrm{BCP}}$$

$$\rightleftharpoons \mathrm{HRh(BCP)_3}$$

The fact that signals from hydrides are not observed at P/Rh < 2 indicates the formation of the $[Rh(BCP)_3]^+$ cation as an intermediate product. The BCP molecule itself is the source of hydride. The hydride containing four BCP molecules is likely formed by the addition of BCP to the $[HRh(BCP)_3]$ complex (Scheme 5).

The initial formation of the 16-electron $[Rh(BCP)_4]^+$ cation seems improbable.

Scheme 5

$$\mathrm{HRh(BCP)_3} \xrightleftharpoons{+\mathrm{BCP}} \mathrm{HRh(BCP)_4}$$

Both $HRh(BCP)_3$ and $HRh(BCP)_4$ can be active in hydrogenation, because the catalytic site is accessible for small substrate molecules. The optimization of the geometry (by the molecular mechanics method) of the $HRh(BCP)_3$—styrene system indicates that the electric field of the metal complex directs olefin to the cavity containing hydride. In this cavity, olefin is protected from external factors and can undergo several transformations.[21] Similar processes occur during hydroformylation: the olefin—Rh—hydride system is transformed into the AlkRh complex, which then coordinates CO, being transformed into the acyl derivative and regenerated Rh hydride.

Thus, the unusual behavior of the new P ligand (BCP) belonging to the class of neutral organic phosphites during styrene hydrogenation and but-2-ene hydroformylation is explained by the results of studying the complexation of BCP with Rh by NMR spectroscopy. The direct formation of the $HRh(BCP)_3$ and $HRh(BCP)_4$ hydrides was observed.

Molecular simulation of these complexes shows that the high stability of the hydrides is related to the formation of molecular cavities near the Rh atom by the benzene rings of three BCP molecules.

The property distinguishing BCP from traditional organophosphorus promoters is the presence of the diarylmethane fragments with strained bonds, which transforms this molecule into a donor of hydride ions.

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