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Coordination Chemistry Reviews 251 (2007) 2072–2086
COORDINATION CHEMISTRY REVIEWS
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Review

Application of transition metals in hydroformylation
Annual survey covering the year 2005

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Received 6 March 2007; accepted 6 March 2007

Contents

Abstract

Hydroformylation in homogeneous and heterogeneous systems, and hydroformylation-related reactions of carbon monoxide reported in 2005 are reviewed.

Keywords: Hydroformylation; Silylformylation; Hydroxycarbonylation; Alkoxycarbonylation; Hydroaminomethylation; Water–gas shift reaction; Reduction of carbon dioxide; Transition metals

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doi:10.1016/j.ccr.2007.03.004

1. Hydroformylation

1.1. Homogeneous systems

1.1.1. Cobalt catalysts

Each of the previously proposed steps in cobalt-catalyzed hydroformylation were confirmed by detection of intermediates using *para*-hydrogen-induced polarization [1].

The $HCo(CO)_3$-catalyzed hydroformylation of 1,3-butadiene [2], allene and propyne [3] was investigated at the B3LYP density functional level of theory. It was found that 1,3-butadiene hydroformylation favors the formal 1,4-addition product in high regioselectivity. Allene favors the linear *anti*-Markovnikov product in high regioselectivity both kinetically and thermodynamically. On the other hand, propyne does not show any regioselectivity.

The hydroformylation of 1-pentene in the presence of $Co_2(CO)_6\{P(O\text{-}2,4\text{-}^tBu_2C_6H_3)_2\}_2$ as the catalyst precursor was studied at 120–180 °C and 20–80 bar CO/H_2. High-pressure IR and NMR spectroscopy was used to detect potential hydride intermediates [4].

Hydroformylation of vinyl acetate was studied as a key step in the alternative route for the synthesis of 1,2-propanediol and 1,3-propanediol using homogeneous cobalt and rhodium complex catalysts. The roles of the catalyst precursors, ligands, and solvents, and the effect of reaction conditions were investigated [5].

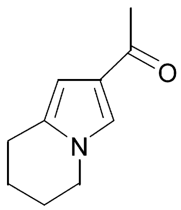
See also Refs. [21,22].

1.1.2. Rhodium catalysts

A theoretical investigation of the unmodified rhodium-catalyzed hydroformylation reaction was made. It was found that the alkyl-rhodium transition state stabilities can serve as a tool to predict regio- and stereoselectivity in the hydroformylation of chiral substrates [6].

The elementary steps in rhodium-catalyzed ethyne [7], acrolein [8], and vinyl formate [9] hydroformylation were calculated using the B3LYP method.

The isomeric 5,6,7,8-tetrahydroindazolines **1** and **2** were obtained in a domino hydroformylation/cyclization reaction of 3-acetyl-1-pyrrole with $Rh_4(CO)_{12}$ as catalyst precursor at 140 °C and 30 bar $CO:H_2 = 1:1$ [10].



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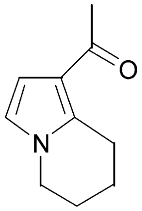

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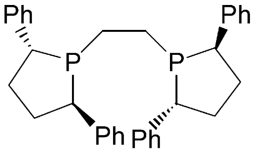
1-Hexene was hydroformylated in ethyl benzoate solution using a PPh_3-modified rhodium catalyst. The optimum reaction parameters were found to be 105–120 °C at 20 bar $H_2:CO = 1.13:1$ resulting in a linear/branched-heptanal product ratio of 7 [11]. The influence of temperature on the rate of phosphine-modified rhodium-catalyzed hydroformylation of propene was studied [12].

Four different rhodium–carbene complexes were tested as catalyst precursors in the hydroformylation reaction of 1-octene. Using 0.02 mol% [RhBr(1,3-dimesityl-3,4,5,6-tetrahydropyrimidin-2-ylidene)] (COD) in toluene at 100 °C and 50 bar $CO:H_2 = 1:1$, TOFs up to 1500 h^{-1} were observed [13].

Highly active, regioselective, and enantioselective hydroformylation of styrene, allyl cyanide, and vinyl acetate was reported using bis-3,4-diazaphospholanes-modified rhodium catalyst at 80 °C and 10 bar $CO:H_2 = 1:1$. Enantioselectivities of up to 82, 87, and 96% enantiomeric excesses were found for styrene, allyl cyanide, and vinyl acetate, respectively [14]. The diphosphine ligand (**3**) was found to be an excellent ligand for the rhodium-catalyzed asymmetric hydroformylation of styrene, allyl cyanide, and vinyl acetate. At 80 °C and 10 bar $CO:H_2 = 1:1$, enantioselectivities of 94, 90, and 82% enantiomeric excesses were found for styrene, allyl cyanide, and vinyl acetate, respectively [15].



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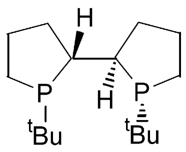

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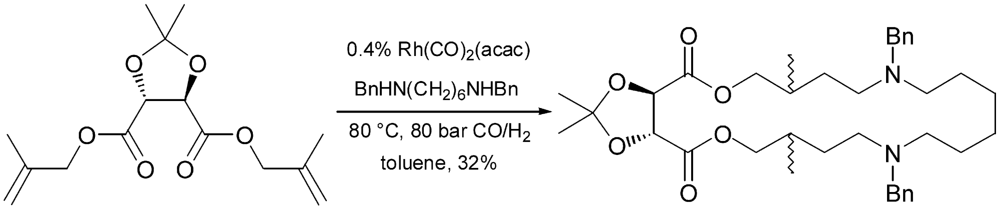
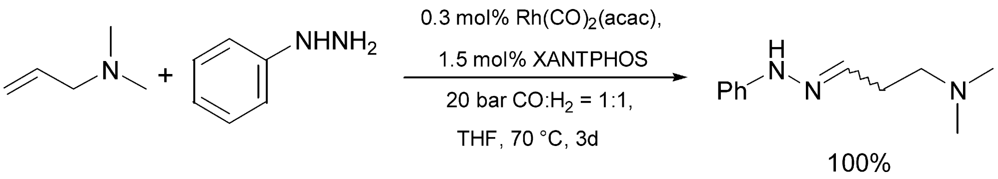
A rhodium catalyst composed of $Rh(CO)_2(acac)$ and **4** was found to be a highly enantioselective catalyst for asymmetric hydroformylation of norbornylene under mild reaction conditions (from r.t. to 70 °C, 8–41 bar $CO:H_2 = 1:1$). Enantioselectivities up to 92% were observed [16].

Rhodium(I) complexes composed of an anionic rhodium center containing chloride ligands, and a cationic rhodium center coordinated by a diamine ligand, were found to catalyze the hydroformylation of styrene and vinyl acetate under mild reaction conditions (e.g. 25 °C, 70 bar $CO:H_2 = 1:1$) in excellent activity and branched aldehyde selectivity [17].

A procedure for the efficient synthesis of azamacrocycles containing chiral (tartaric acid derivatives) and aryl units via tandem and stepwise hydroformylation/reductive amination reaction was reported [18]. For example:



Tandem hydroformylation/hydrazone formation from amino olefins and aryl hydrazines were described [19]. For example:



A novel one-pot synthesis of indole systems via tandem hydroformylation/hydrazone formation and Fischer indolization starting from allylic amides and aryl hydrazines was described [20].

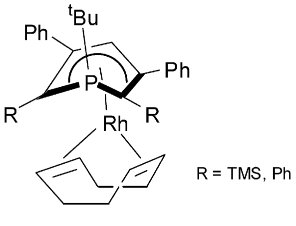
The catalytic activities of the complexes of Pd, Ru, Co, and Rh with PPh_3, $AsPh_3$, and $SbPh_3$ ligands were investigated for the hydroformylation of ethene in benzene as the solvent. The Pd, Ru, and Co complexes with $AsPh_3$ ligand were found to show better hydroformylation catalytic activity than those of their corresponding complexes with PPh_3 and $SbPh_3$ ligands [21]. A comparative study was made of transition metal-catalyzed hydroformylation of 1-hexene using rhodium, cobalt, and ruthenium complexes of PPh_3, $AsPh_3$, and $SbPh_3$ as the catalysts [22]. A detailed kinetic study of 1-hexene hydroformylation using $RhCl(AsPh_3)_3$ as a homogeneous catalyst was published [23].

Evidence was presented for the formation and for the different evaluation of tertiary rhodium alkyl intermediates in the PPh_3-modified rhodium-catalyzed deutero(hydro)formylation of 1-(*n*-pyridyl)-1-phenylethenes (80 °C, 100 bar $CO{:}D_2 = 100$ bar) at partial and complete substrate conversion [24].

Hydroformylation of ethyl ricinoleate and castor oil using $HRh(CO)(PPh_3)_3$ as a catalyst precursor was performed under mild reaction conditions. 2-Hydroxy-furans were obtained as the major product. Aldehydes were selectively obtained if the hydroxyl group of ethyl ricinoleate or castor oil was silylated before the hydroformylation [25]. Hydroformylation of technical-grade methyl oleate and soybean oil using $HRh(CO)(PPh_3)_3$ and $RhCl_3{\cdot}3H_2O$ as the catalyst precursors modified by PPh_3, was investigated. Conversions of 100% and selectivities to aldehydes (80–91%) were achieved at 100 °C and 40 bar $CO{:}H_2 = 1{:}1$ [26].

Rhodium(III) complexes of the type RhCl(Cp*)(L) (L = P-heterocyclic or cyclopropane-based P-ligand) were tested in styrene hydroformylation. High activity at 40–100 °C and 100 bar $CO{:}H_2 = 1{:}1$ coupled with excellent chemoselectivity towards aldehydes were observed [27]. The influence of 1:1 mixtures of 14 different monodentate achiral ligands on the regioselectivity of rhodium-catalyzed hydroformylation of *tert*-butyl methacrylate (at 50 °C, 60 bar $CO{:}H_2 = 1{:}1$) was investigated using combinatorial methods [28].

The catalytic activity of **5** was tested in the hydroformylation of olefins. Good conversion yields and turnover frequencies were obtained in the hydroformylation of styrene and cyclohexene under mild conditions with low catalyst loading. The hydroformylation of styrene occurs with a high regioselectivity (93/7) in favor of the branched aldehyde isomer [29].



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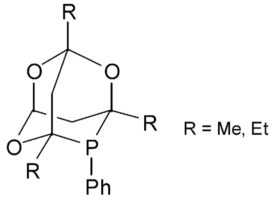


6

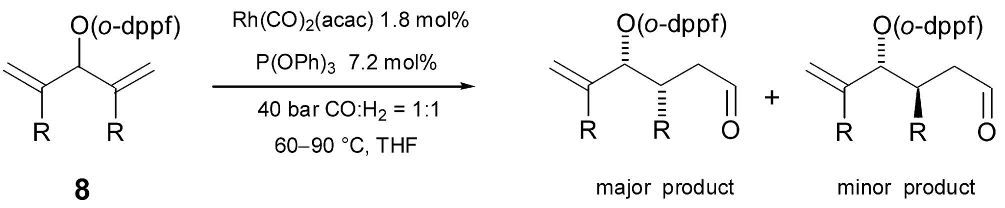
Rhodium complexes of ligand **6** were found to be similarly active catalysts in hydroformylation as catalysts derived from phosphites. The catalysts derived from **6** gave unusual low linear selectivity in the hydroformylation of hexenes. Using ligand **6** in rhodium-catalyzed hydroformylation of unsaturated esters a quaternary-selective synthetic method was developed [30].

Fluorophilic phosphines incorporating at least one aromatic ring containing two directly attached perfluoroalkyl groups were used as modifying ligands for the rhodium-catalyzed hydroformylation of 1-octene in perfluorocarbon solvents. Catalyst activity, regioselectivity and the levels of rhodium leaching to the product phase were studied [31]. The effects of fluoroaryl phosphines on the activity and selectivity of modified rhodium catalysts in rhodium-catalyzed hydroformylation of 1-hexene and 4-methoxystyrene were tested. The results were compared to those obtained with PPh_3 and $P(OPh)_3$ [32].

The hydroformylation of 1-hexene catalyzed by rhodium–TPPTS complexes in the ionic liquid $[bmim]BF_4$ was studied [33]. Functionalized calix[4]arenes with bis-phosphites and bis-phoshinites attached at the distal phenolic oxygen atoms were used to modify rhodium catalysts for hydroformylation of 1-octene and styrene [34].

The catalytic hydroformylation of alkenes and of allyl alcohol was achieved with paraformaldehyde in dioxane solution using phosphine-modified rhodium catalysts. The highest activities were obtained for an *in situ* derived catalyst from $Rh(CO)_2(acac)$ with 2 equiv. of $Ph_2PCH_2CH_2PPh_2$ [35].

The simultaneous construction of two new, vicinal stereogenic centers in dialkenylcarbinols (**8**) [39] and in diallylcarbinols (**9**) [40] with high levels of stereo control was achieved in rhodium-catalyzed hydroformylation using the substrate bound catalyst directing group *o*-dppf (**10**).



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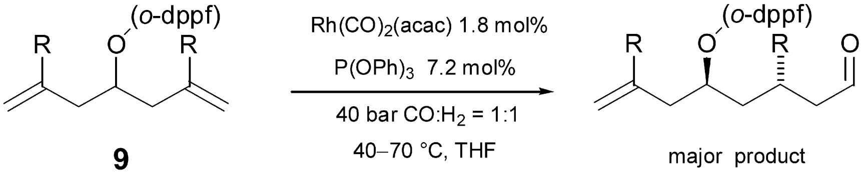

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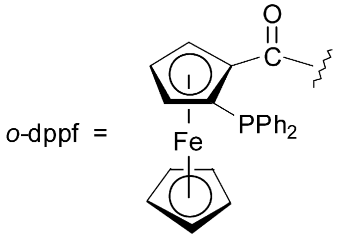
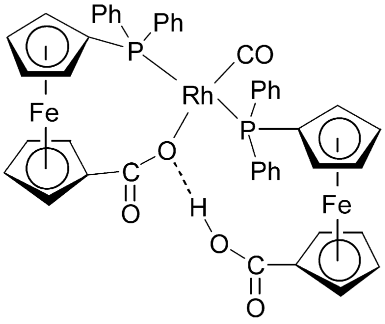


10

Dendritic ligands with triphenylphosphines at the periphery were applied to rhodium-catalyzed hydroformylation of 1-octene and styrene at 80 °C and 20 bar $CO:H_2 = 1:1$ [36].

The hydroformylation reaction of 1-hexene catalyzed by 1,1′-bis(diarylphosphino)metallocene rhodium(I) complexes was investigated by both high-pressure NMR and high-pressure IR spectroscopy under catalytic conditions [37]. The rhodium complex **7** was used as an efficient and recyclable catalyst for 1-hexene hydroformylation at 80 °C and 10 bar $CO:H_2 = 1:1$. The effect of modifying ligands, phosphines and phosphites, on the catalytic activity of **7** was investigated [38].



7

The rhodium-catalyzed hydroformylations of allylic double bond using perbenzylated C-glucosides as chiral directors were reported as a novel case of 1,3-asymmetric induction [41].

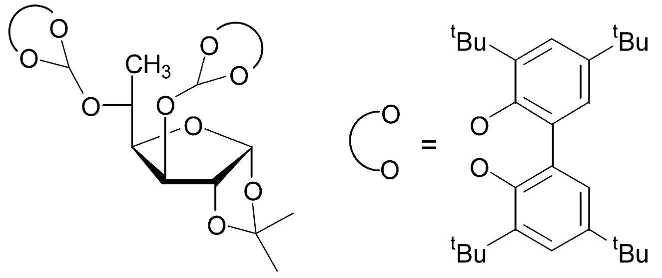
The catalytic activity of two novel rhodium–NHC complexes, Rh(CO)(acac)(L) (L = 1,3-bis-(2,4,6-trimethylphenyl) imidazolinylidene, and 1,3-bis-(2,6-diisopropylphenyl)imidazolinylidene towards hydroformylation of 1-hexene was tested with and without added PPh_3 and $P(O\text{-}2,4\text{-}^t Bu_2C_6H_3)_3$ as auxiliary ligands. No catalyst activity was observed in the absence of an additional ligand. The catalytic activity observed in the presence of the auxiliary ligands corresponds that of using $Rh(CO)_2(acac)$ as the catalyst precursor with the respective ligand [42]. Eight different rhodium-*N*-heterocyclic carbene complexes were used as catalyst precursors in hydroformylation of 1-octene [43]. The effect of the auxiliary phosphine ligands on the hydroformylation activity of rhodium-*N*-heterocyclic carbene complexes of the type $Rh(NHC)(PR_3)(CO)Cl$ was investigated. Very high selectivities for the branched isomer (>95:5) were obtained in the hydroformylation of vinylarenes [44].

The asymmetric hydroformylation of variously substituted 4-vinyl β-lactams catalyzed by rhodium aminophosphonite–phosphinite and rhodium aminophosphine–phosphite complexes was studied. The stereoselectivity was found to be related to the presence of a substituent at the nitrogen atom of the β-lactam ring. The regioselectivity (branched/linear) but not the stereoselectivity (β/α) was found to depend on the substrate to catalyst ratio [45].

The effects of the amounts and the type of heteropoly-acids on the rhodium-catalyzed hydroformylation of styrene and 1-octene were studied. The rhodium cluster $Rh_6(CO)_{12}$ associated with the heteropolyacid $H_3PW_{12}O_4{\cdot}25H_2O$ was found to improve the conversion of styrene and the selectivity towards the branched aldehyde [46].

The one-pot acetal-formation in the rhodium-catalyzed styrene hydroformylation in alcohols as solvents was investigated. The effects of the addition of different types and amounts of phosphines and phosphites were studied in order to improve the regioselectivity of the reaction [47].

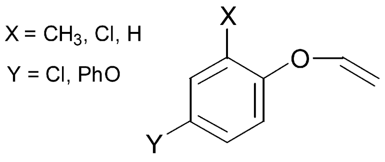
Diphosphinite ligands prepared from D(+)-xylose were tested in the rhodium-catalyzed asymmetric hydroformylation of several vinyl arenes. High regioselectivities in branched aldehyde (up to 99%) and moderate enantioselectivities (up to 63% ee) were found at 20–40 °C and 30–60 bar $CO:H_2 = 1:1$ [48]. Enantioselectivities up to 75% ee and excellent regioselectivities were achieved in the rhodium-catalyzed asymmetric hydroformylation of 2,5- and 2,3-dihydrofuran at 45 °C and 18 bar $CO:H_2 = 1:1$ using diphosphite ligands such as **11** [49].



11

New chiral phosphite ligands, based on incompletely condensed silsesquioxane frameworks were applied in the rhodium-catalyzed asymmetric hydroformylation of vinyl acetate. High activities and moderate enantioselectivities of up to 38% ee were obtained at 60 °C and 20 bar $CO:H_2 = 1:1$ [50].

The behavior of the rhodium(I)/(*S*,*R*)-BINAPHOS complex in asymmetric hydroformylation of styrene and aryloxy substituted ethenes (**12**) was studied. Under optimized conditions, the hydroformylation of **12** provides the relevant branched aldehyde in 70% regioselectivity and in up to 80% ee [51].



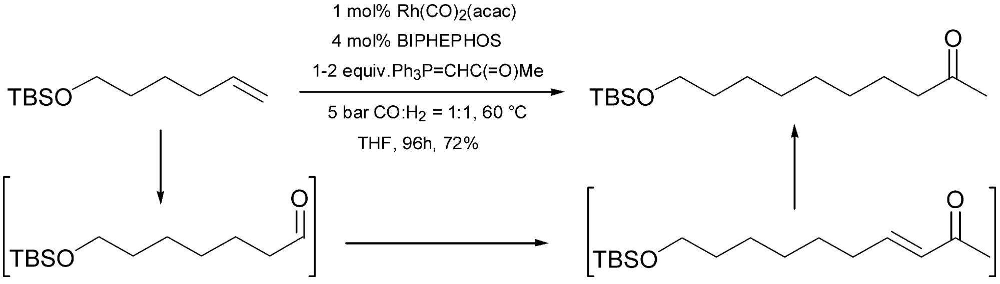
12

Several new chiral *P*,*N*-ligands derived from 1,2:5,6-di-*O*-cyclohexylidene-D-mannitol, 1,1′-binaphthol and phenylisocyanate were applied as modifiers in the rhodium-catalyzed asymmetric hydroformylation of vinylarenes. A synergic effect between the sterogenic centers of D-mannitol and the chiral binaphthol substituents was observed [52].

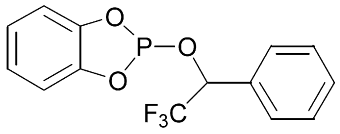
New cationic rhodium(I) complexes with a chiral nitrogen-containing BINOL-based diphosphite ligand were tested in hydroformylation of styrene. High activity, chemo- and regioselectivity, but no asymmetric induction was found [53].

The hydroformylation of terminal and internal olefins to terminal aldehydes using a catalyst system composed of $Rh(CO)_2(acac)$ and BIPHEPHOS was investigated. Conversions up to 99% and yields up to 86% of the aldehydes (linear:branched ratio 99:1) were found. A very high TOF of 44,000 h^{-1} was achieved at 140 °C and 30 bar $CO:H_2 = 1:1$ using a rhodium to 1-dodecene ratio of 1:100,000 [54]. An efficient product and catalyst separation was achieved by the application of a temperature-dependent multi-component solvent system in rhodium–BIPHEPHOS catalyzed hydroformylation of *trans*-4-octene to *n*-nonanal [55]. The rhodium–BIPHEPHOS catalyzed isomerizing hydroformylation of oleic acid ester and linoleic acid ester was found to give the corresponding linear aldehyde at 115 °C and 20 bar $CO:H_2 = 1:1$ in up to 26 and 34% yield, respectively [56].

Rhodium-catalyzed hydroformylation of terminal alkenes in the presence of stabilized phosphorus ylides was found to initiate a domino hydroformylation–Wittig olefination process. When monosubstituted acceptor-stabilized phosphorus ylides were employed, a hydrogenation step follows the Wittig olefination to give a domino hydroformylation–Wittig olefination hydrogenation process [57]. For example:



Preformed rhodium complexes or *in situ* systems with asymmetrical cyclic phosphite (**13–16**) and phosphinite (**17**) ligands bearing either trifluoromethyl or pentafluorophenyl group(s) were tested in the hydroformylation of styrene. Both systems were found to provide excellent hydroformylation activities at 100 °C [58].



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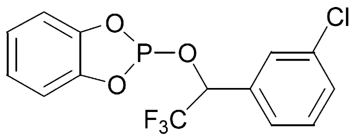

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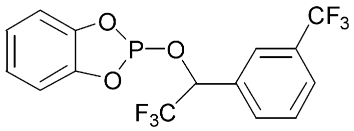

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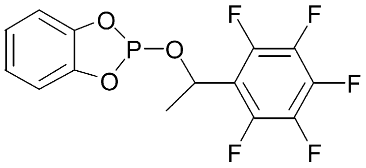

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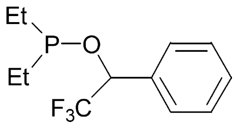

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The hydroformylation reaction of protoporphyrin-IX dimethyl ester using a PPh_3-modified rhodium catalyst at 30 bar and 31 °C was found to yield 98% of branched aldehyde. Using XANTPHOS as the catalyst modifier gave 74% of the linear aldehyde at 20 bar and 80 °C [59].

Encapsulated phosphines, constructed through coordinative interactions between Zn(II)–salphen complexes and pyridylphosphine templates, were used as ligands in the rhodium-catalyzed hydroformylation of 1-octene in toluene solvent at 25 °C and 20 bar $CO:H_2 = 1:1$ [60].

The effectivity and activity of rhodium–thione and rhodium–NHC complexes were compared as precatalysts in the hydroformylation of 1-hexene under mild (80 °C, 80 bar) reaction conditions [61].

A room temperature/ambient pressure regioselective hydroformylation of terminal alkenes was reported [62].

See also Refs. [5,67,94,99,100,113,114,156].

1.1.3. Other metals as catalysts and bimetallic catalysts

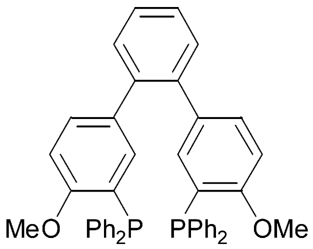
The hydrocarbonylation of ethene with CO/H_2O to give propanal and CO was found to be highly selective in diglyme containing 7 wt% H_2O in the presence of propionic acid/propionate. A TOF = 20 h^{-1} [mol of propanal/(mol Ru h)] and a selectivity >90% were obtained at 140 °C and 50 bar CO:ethene = 3:2 [63]. Hydroformylation of ethene with carbon monoxide and H_2O using a ruthenium catalyst in ionic-liquid media was reported [64].

Ruthenium carbonyl complexes with 2-substituted pyrazine ligands were found to show activity in 1-hexene hydroformylation at 120 °C and 20 bar pressure [65].

The reactivity of a series of ruthenium *ortho*-substituted triphenylphosphine complexes was studied as catalysts for the hydroformylation of 1-hexene. The activities were found to depend on the binding mode of the phosphine and on the strength of the ruthenium–phosphine interaction [66].

The hydroformylation reaction of vinylsilanes catalyzed by siloxide complexes of rhodium(I) and iridium(I) was investigated at 80 °C and 10 bar $CO:H_2 = 1:1$ [67].

The new diphosphines **18** and **19** were applied in the platinum/tin-catalyzed hydroformylation of 1-octene. Moderate activities and high regioselectivities (linear:branched ratio up to 45) were found [68].



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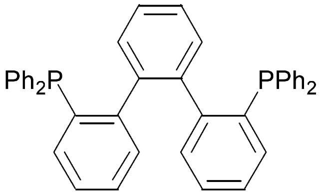

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See also Refs. [21,22,137].

1.2. Heterogeneous systems

1.2.1. Supported metal catalysts

Supported Co/SiO_2 and Co/active carbon catalysts were studied as heterogeneous catalysts in the liquid-phase hydroformylation of 1-hexene. Alcoholic solvents were found to promote the oxygenate formation significantly [69]. Rhodium/Al_2O_3 catalysts with low metal loading were found to be very active in the hydroformylation of styrene, but leaching of rhodium was observed [70]. Vanadia-modified rhodium/SiO_2 catalyst was found to exhibit high selectivity of 1-propanol formation in ethene hydroformylation [71]. The addition of molybdenum to rhodium/SiO_2 catalyst enhanced the hydroformylation activity [72].

Hydroformylation of ethene using PPh_3-modified rhodium/SiO_2 catalysts was studied. Two types of active species were found by *in situ* FTIR techniques [73].

The catalytic behavior of the two kinds of palladium/SiO_2 catalysts prepared from dinitroamminepalladium and palladium nitrate were compared for ethene hydroformylation and carbon monoxide hydrogenation. The Pd/SiO_2 catalyst having very fine particles of Pd on the support was found to show high activity for ethene hydroformylation [74].

See also Refs. [87,91].

1.2.2. Supported complexes

Hydroformylation of 1-hexene, 1-decene, 1-tetradecene, 2-hexene, and 2,3-dimethyl-2-butene was studied using a catalyst system composed of carbonylhydrido-tris-(*m*-sulfotriphenylphosphine)-rhodium immobilized on activated carbon [75].

The kinetics of the hydroformylation reaction of 1-octene in the presence of a silica-supported water-soluble rhodium complex was studied [76]. Exchange of the Rh/TPPTS complex on to an anion exchange resin was found to result in a stable heterogenized catalyst for the hydroformylation of alkenes. The kinetics of hydroformylation of 1-hexene using this catalyst was investigated. The rate was found to be first-order-dependent on catalyst, 1-hexene concentration and H_2 partial pressure. A maximum in the rate with increasing partial pressure of carbon monoxide was observed [77].

A “heterogenized” version of homogeneous ionic-liquid catalyst system in which the catalyst remains active, highly selective, and stable over extended periods in a continuous gas-phase hydroformylation process, was described. Thus using a rhodium–sulfoXANTPHOS complex in [bmim][*n*-$C_8H_{17}OSO_3$] on a partly dehydrogenated silica support gave a >20 normal/*iso*-butyraldehyde ratio, TON = 2600, TOF = 44 h^{-1} over at least 60 h [78]. Supported ionic-liquid-phase catalysts were studied with regard to their long-term stability in the continuous gas-phase hydroformylation of propene [79]. Mesoporous silica-supported water-soluble rhodium/TPPTS complex in the ionic liquid 1,1,3,3-tetramethylguanidinium lactate was found to exhibit high catalytic performance and stability for the hydroformylation of 1-hexene [80]. A similar catalyst was applied for the hydroformylation of higher olefins [81].

Rhodium-supported catalysts were prepared by impregnating rhodium(I) and rhodium(III) complexes with and without heteropolyacids for the hydroformylation of styrene derivatives. A clear effect of the heteropolyacid $H_3PW_{12}O_{40}\cdot yH_2O$ in increasing the catalytic activity of the rhodium-supported catalyst was found [82]. Immobilized catalyst system based on $Rh(CO)_2(acac)/Ph_2P(CH_2)_2S(CH_2)_3Si(OMe)_3$ was found to be active in the hydroformylation of 1-hexene and 1-octadecene without any rhodium leaching [83].

An organometallic rhodium complex entrapped within a partially hydrophobicized silica sol–gel matrix was tested as catalyst in olefin hydroformylation using a three-phase microemulsion transport methodology [84].

Zirconia-based materials complexed with the $Rh(CO)_2^+$ fragment were found to be active catalysts in internal olefin hydroformylation. Investigation of the mechanism of this reaction showed that the isomerization step is catalyzed by the Lewis acidic support, whereas the hydroformylation step is driven by the rhodium catalyst [85].

Rhodium catalysts anchored on phosphinated silica-based supports were evaluated for their performance in the hydroformylation of 1-hexene in supercritical carbon dioxide. The surface mechanism was probed using high-pressure diffuse reflectance infrared spectroscopy. The results suggest that the homogeneous mechanism is effectively transferred to the supported material [86].

A fibre-supported rhodium–phosphine catalyst was found to be an active and selective catalyst with 95% selectivity for propanal at 100 °C and 5 bar CO:H_2 = 1:1 in the hydroformylation reaction of ethene [87].

A heterogenized rhodium carbonyl complex catalyst for hydroformylation of olefins was prepared by copolymerization of functionalized 3-aminopropyltriethoxysilane with tetraethoxysilane via the sol–gel method. The activity, selectivity, and stability of this catalyst for 1-hexene hydroformylation were examined [88].

A rhodium–phosphine complex was encapsulated within the surface alkylated poly(propyleneimine) dendrimers through ionic interactions. The resulting dendrimer complexes functioned as nanoreactors for hydroformylation of higher olefins [89]. Polyamidoamine dendrimers up to the third generation, grown on the surface of a large-pore (18 nm) silica support and complexed with rhodium, were found to be very active for the hydroformylation of olefins. The hydroformylation of 1-octene was accomplished with a turnover frequency of 1700 h^{-1} at 70 °C [90].

See also Ref. [115].

1.2.3. Biphasic systems

The gas-phase hydroformylation of ethene to propanal was studied over solid $RhCl(PPh_3)_3$ and $RhCl(CO)(PPh_3)_2$ at 3 bar and 185 °C. The selectivity of $RhCl(PPh_3)_3$ to propanal was found to be much better than that of supported rhodium metal [91].

The water-soluble rhodium complex $[Rh(\mu\text{-pz})(CO)(TPPTS)]_2$ (pz = pyrazolate) was used as catalyst precursor during the two-phase catalytic hydroformylation of different olefins at 100 °C, 50 bar (CO:H_2 = 1:1), 600 rpm, and substrate/catalyst ratio of 100:1. A reaction order: 1-hexene > styrene > allylbenzene > 2,3-dimethyl-1-butene > cyclohexene was found [92].

The influence of the concentration of gemini surfactants and the stirring rate on the biphasic 1-dodecene hydroformylation catalyzed by $RhCl(CO)(TPPTS)_2$ were investigated. In the presence of gemini surfactants higher turn over frequencies and higher regioselectivities toward the linear aldehyde, than those obtained with cetyltrimethylammonium bromide, were observed [93].

The hydroformylation of styrene, cyclohexene, and 1,2-diacetoxy-2-butene were studied using alkylpolyglycol ether-type non-ionic surfactant in the presence of phosphine-modified rhodium catalyst [94]. The rhodium/TPPTS-catalyzed hydroformylation of 1-octene and other higher olefins in an aqueous biphasic system was studied at pressures from 40 to 90 bar and temperatures of up to 120 °C. Non-ionic amphiphiles of the alkoxyethylene type were applied to promote the contact between the reacting species by enlargement of the interfacial area. The highest reaction rates were obtained at a surfactant concentration of about 1 wt% [95].

The two-phase hydroformylation of higher olefins with the rhodium/TPPTS catalytic system in the presence of various chemically modified α-cyclodexrins was investigated [96]. Rhodium/TPPTS complexes non-covalently bound to cyclodextrins were tested as water-soluble supramolecular catalysts for the biphasic hydroformylation of higher olefins. The cyclodextrins greatly increased the reaction rate, the chemoselectivity, and the linear-to-branched aldehyde ratio [97]. The potential of sulfonated XANTPHOS as ligand for a cyclodextrin-based rhodium-catalyzed 1-octene and 1-decene hydroformylation process was investigated. Activity enhancement and an increase of the linear-to-branched aldehyde ratio (up to 33) was observed [98].

Synthesis of *n*-hexanal from 2-pentene in water using $Rh(CO)_2(acac)$ and sulfonated NAPHOS as catalyst precursors was investigated. It was found that by lowering the partial pressure of carbon monoxide in a buffered system at pH 7 and 8, a significant increase of the aldehyde yield to 72–73% and excellent regioselectivity (normal:iso = 99:1) was observed. Similar results for the hydroformylation of 2-pentene were also obtained in the presence of tertiary amines such as triethylamine [99].

The rhodium/BIPHEPHOS-catalyzed isomerizing hydroformylation of *trans*-4-octene to *n*-nonanal in a temperature-depending multi-component solvent system was investigated. A solvent composition of propylene carbonate, dodecane, and *p*-xylene gave 99% conversion of the *trans*-4-octene with a selectivity of 90% in *n*-nonanal. During the phase separation, however, 47% rhodium leaching was observed [100].

A thermoregulated phase-transfer rhodium(I) complex catalyst prepared from $Rh(CO)_2(acac)$ and $CH_3(OCH_2CH_2)_mPPh_2$ (molecular weight = 918) was applied to the biphasic hydroformylation of 1-octene. High catalyst activity with an aldehyde yield of 97.5% was observed [101].

See also Refs. [55,81].

1.3. Coordination chemistry related to hydroformylation

The mechanism of the migratory carbonyl insertion in $CpFe(CO)_2Me$ was studied by NMR, IR, and UV–vis spectroscopies [102]. A DFT study on CO insertion into the rhodium–carbon bond of rhodium–ethyl, –ethenyl, –2-propenyl, –*trans*-propenyl, –*cis*-propenyl, and –allyl complexes was carried out [103].

Hydrogenation of the water-soluble $Rh(OAc)(CO)(TPPMS)_2$ containing monosulfonated triphenylphosphine ligands was studied in aqueous solution. In the presence of additional TPPMS, the reaction yielded $RhH(CO)(TPPMS)_3$. The extent of the $Rh(OAc)(CO)(TPPMS)_2 \rightarrow RhH(CO)(TPPMS)_3$ transformation was found to be strongly depend on the solution pH [104].

The formation of rhodium hydride species in the bulky monophosphite (tris(2,4-di-*tert*-butylphenyl)phosphite) ligand-modified rhodium catalyst was investigated under hydroformylation conditions using *in situ* spectroscopic methods [105].

The kinetics of the formation of the active species *cis*-$[Pt(PPh_3)_2Cl(SnCl_3)]$ and *cis*-$[Pt(PPh_3)_2(SnCl_3)_2]$ from the hydroformylation catalyst precursor *cis*-$[Pt(PPh_3)_2Cl_2]$ in the presence of $SnCl_2$, was studied in two different imidazolium-based ionic liquids. DFT calculations were employed to model the reaction progress and to resolve the role of the ionic liquid in the activation of the catalyst [106].

See also Refs. [2,63].

1.4. Engineering aspects of hydroformylation

Results of solubility measurements of a phosphine-modified carbonylcobalt hydroformylation precatalyst, *trans*-$Co_2(CO)_6[3,5\text{-bis}(CF_3)C_6H_3P^iPr_2]_2$ in dense carbon dioxide were described [107].

A continuous flow process of hydroformylation of long chain alkenes was elaborated using an *in situ* made catalyst from $Rh(CO)_2(acac)$ and [1-propyl-3-methylimidazolium]$[Ph_2P(3\text{-}C_6H_4SO_3)]$ dissolved in 1-octyl-3-methylimidazolium bis(trifluoromethylsulfonamide). The substrates are introduced into and the products are removed from the reactor dissolved in continuously flowing supercritical carbon dioxide. In long-term operations rates up to 500 catalyst turnovers h^{-1} and rhodium leaching into the product as low as 0.012 ppm were achieved [108].

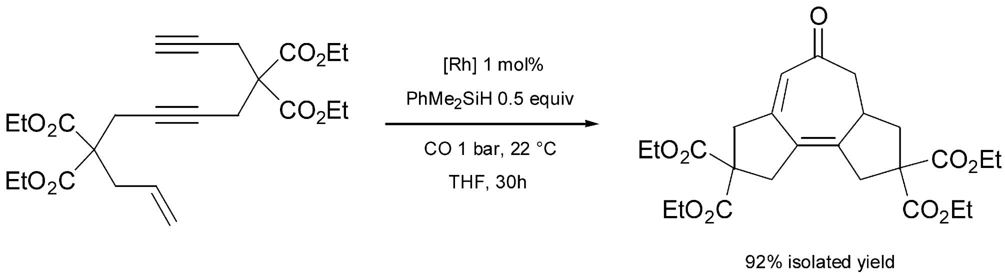
See also Refs. [15,78,79].

2. Hydroformylation-related reactions of CO

2.1. Silylformylation and silylcarbocyclization

Silylcarbonylation of vinylsilanes catalyzed by iridium(I) siloxide complexes was investigated [109].

The reaction of dodec-11-ene-1,6-diynes or their heteroatom congeners with a hydrosilane catalyzed by $Rh(CO)_2(acac)$ at ambient temperature and pressure of carbon monoxide was found to give the corresponding fused tricyclic products, 5-oxo-1,3*a*,4,5,7,9-hexahydro-3*H*-cyclopenta[*e*]azulenes or their heteroatom congeners in excellent yields [110]. For example:



2.2. Hydroxycarbonylation, hydroalkoxycarbonylation and alkoxycarbonylation

Solutions formed by activation of [(*p*-cymene)Ru(diphosphine)Cl][SbF_6] (diphosphine = BIPHEP, MeO–BIPHEP) with $AgSbF_6$ were found to catalyze the regio- and stereoselective anti-Markovnikov hydrocarboxylation of phenylacetylene [111].

Supported rhodium catalysts (Rh on charcoal, silica, alumina and zeolite) were found to be active and selective catalysts for the vapor phase carbonylation of alcohols and hydrocarboxylation of alkenes to carboxilic acids [112].

The hydroesterification and hydroformylation–acetalization reaction of 1-hexene was investigated in ethanol, 80% aqueous ethanol, or methanol using [$Rh(CO)_2(amine)_2$][PF_6] complexes (amine = pyridine, 2-picoline, 3-picoline, 4-picoline, 3,5-lutidine or 2,6-lutidine) as the catalyst precursors at 100 °C and *P*(CO) = 0.9 bar. The composition of the reaction product was found to depend on the nature of the amine [113,114]. Similar catalyst precursors but immobilized on poly(4-vinylpyridine) were used for the reaction of 1-hexene with carbon monoxide in methanol solution [115,116].

Palladium(II) complexes with a *P–N*-chelating ligand were used as initiators for the formation of 4-oxo-hexanoic acid methyl ester from CO/ethene at 38 bar and 90 °C in methanol solution [117]. Water-soluble palladium complexes with guanidinumphosphine ligands were found to be active catalyst precursors for the hydrocarboxylation of styrene in water with conversions up to 96% [118].

The palladium-catalyzed asymmetric methoxycarbonylation of styrene was investigated using phosphetanes as chiral ligands. Good catalytic activity, excellent regioselectivity to the branched product, and enantioselectivities up to 29% were observed [119].

Highly active, selective and recyclable palladium catalyst systems for the hydroesterification of styrene (at room temperature and 6 bar CO pressure) and vinyl acetate (at 40–60 °C and 6–10 bar CO pressure) were found by using 1,2-bis(di-*tert*-butylphosphinomethyl)benzene as ligand and polymeric sulfonic acids of limited SO_3H loadings as promoter [120]. High selectivities to methyl acetoxypropanoate esters (branched:linear ratio up to 3.6:1) were obtained at 25–80 °C and 3–30 bar CO pressure for the methoxycarbonylation of vinyl acetate catalyzed by palladium complexes of 1,2-bis(di-*tert*-butylphosphinomethyl)benzene in the presence of methanesulfonic acid, provided that the acid concentration does not exceed that of the free phosphine [121]. The methoxycarbonylation of unsaturated acids or esters catalyzed by palladium complexes of 1,2-bis(di-*tert*-butylphosphinomethyl)benzene was found to produce α,ω-diesters with selectivities >95%, even if the double bond is deep in the chain or conjugated to the carbonyl group [122].

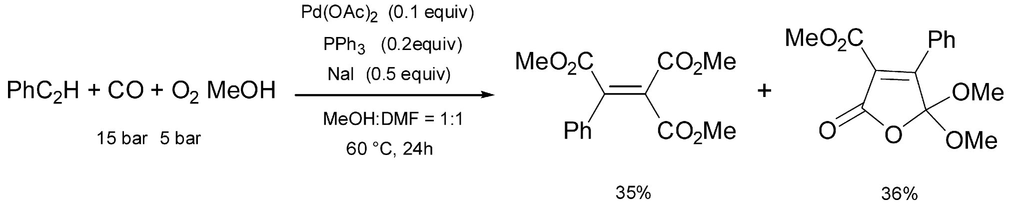
A catalyst composed of $Pd_2(dba)_3$ and XANTPHOS was found to catalyze the alkoxycarbonylation of 4-penten-1-ol to form ε-caprolactone and 2-methylvalerolactone, as well as oligocaprolactone [123]. Ionic-liquid media were tested in the palladium-catalyzed hydroesterification of olefins. Selectivities ranging from 5 to 7:1 for the linear ester were obtained with styrene [124]. The effect of various alcohols on the regioselectivity of the palladium-catalyzed hydroalkoxycarbonylation of styrene in [bmim][BF_4] and [bmim][PF_6] ionic liquids was investigated [125]. The palladium-catalyzed carboxylation of styrene using a $N(C_4H_9)_4Br$–heptane system was studied [126].

The active species of the hydride palladium-catalyzed alkoxycarbonylation of terminal alkenes were studied by multinuclear NMR spectroscopy [127].

Palladium(0)-alkene bis(triarylphoshane) complexes as catalyst precursors were evaluated in the methoxycarbonylation reaction of styrene [128].

Palladium complexes with a variety of *P*,*N*-bidentate ligands were tested as catalyst precursors in methoxycarbonylation of phenylacetylene in organic solvents and in supercritical CO_2. High activity (TOF up to 8000 in methanol) and high selectivity (>99%) toward the branched ester product was reported [129]. Heterocyclic derivative syntheses by palladium-catalyzed oxidative cyclization–alkoxycarbonylation of substituted γ-oxoalkynes were described [130].

A palladium-catalyzed alkoxycarbonylation process, in which three CO units are introduced to the alkynes, was developed [131]. For example:



A catalytic system based on palladium-1,2-bis(di-*tert*-butylphosphinomethyl)benzene was found to show good activity for the methoxycarbonylation of strongly activated aryl chlorides, such as methyl 4-chlorobenzoate or 4-chlorobenzonitrile [132].

High-yielding syntheses of 3-alkoxycarbonyl- and 3-carboxamido-3-tropone derivatives were reported in homogeneous palladium-catalyzed reactions of 3-iodo-2-tropene [133]. New phosphinite and phosphonite complexes were found to be efficient catalysts for the methoxycarbonylation of iodobenzene [134].

The results of a theoretical DFT (B3LYP) investigation on the $PdCl_2(PH_3)_2$-catalyzed carbonylation reaction of allyl chloride to 3-butenoylchloride were described [135].

See also [63].

2.3. Cyclocarbonylation

Molybdenum-mediated cyclocarbonylation of 1-ethynyl-2-allenylbenzenes to 1*H*-cyclopenta[*a*]inden-2-ones was studied. The molybdenum complex, $Mo(CO)_3(CH_3CN)_3$ was found to be the most efficient in the syntheses [136].

Microwave-accelerated protocols were described for palladium(0)-catalyzed carbonylative cyclization of unsaturated aryl bromides and chlorides to obtain various indan-1-one products in high yields [137].

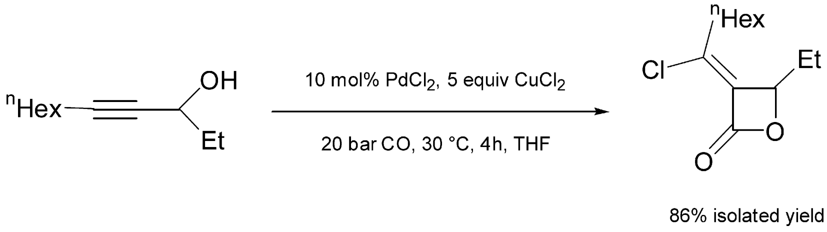
Cobalt(I) complexes modified with (*R*)-(6,6′-dimethoxybiphenyl-2,2′-diyl)-bis-(diphenylphosphine) were tested as catalyst precursors for the enantioselective cyclocarbonylation of 4,4′-bis(carboethoxy)hept-5-en-1-yne. Enantioselectivities up to 78.5% were obtained [138]. The use of immobilized Co–Rh catalysts in cyclohydrocarbonylation of substituted alkynes and tandem cyclohydrocarbonylation–CO insertion of α-keto alkynes to obtain 2(3*H*)- or 2(5*H*)-furanones were described [139].

A DFT study on the mechanism of [Rh(*R*,*R*)-Me-DuPHOS)]$^+$-catalyzed asymmetric carbonylative [4 + 1] cycloaddition of vinylallenes with CO was published [140].

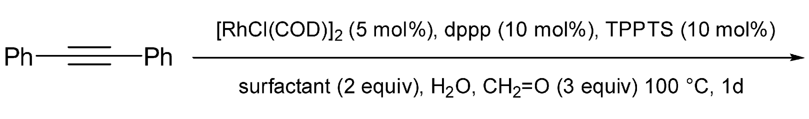
The synthesis of highly substituted chiral butenolides from 1,2-disubstituted alkynes promoted by a chiral bioxazoline Pd(II) complex was studied [143]. The asymmetric cyclocarbonylation reaction of allyl naphthol to a lactone derivative catalyzed by palladium complexes and chiral phosphine at 80–100 °C and 20–40 bar in dichloromethane as the solvent was reported [144].

Palladium complexes immobilized onto silica-supported dendrimers in the presence of 1,4-bis(diphenylphosphino)butane were used as catalysts for the cyclocarbonylation of 2-allylphenols, 2-allylaniline, 2-vinylphenol and 2-vinylaniline to afford five-, six- or seven-membered ring lactones and lactams [145]. The mechanism of monoterpene cyclocarbonylation reaction catalyzed by a palladium complex was studied by multinuclear NMR spectroscopy [146]. An efficient catalytic system for cyclocarbonylation of terpenes into lactones using $[HPd(SnCl_3)L_2]$ (L_2 = dppb) as the active catalyst species was described [147].

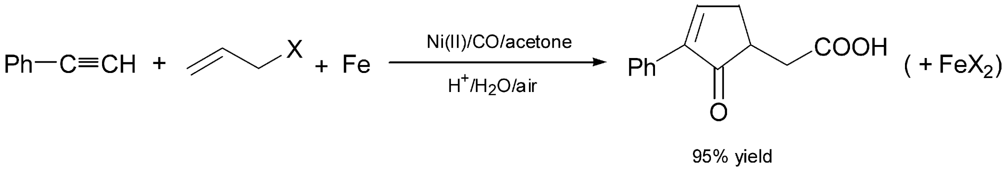
Good regio- and stereoselectivities were observed for the palladium(II) chloride-catalyzed cyclocarbonylation of 2-alkynols in the presence of excess $CuCl_2$ affording (*Z*)-α-chloroalkylidene-β-lactams [148]. For example:



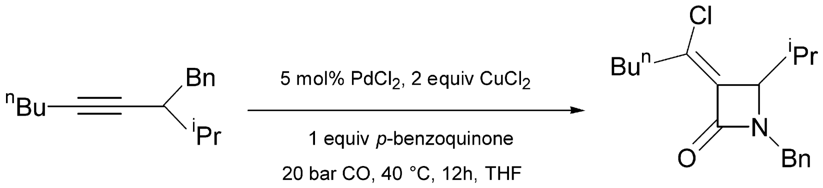
A rhodium(I)-catalyzed CO gas-free cyclohydrocarbonylation of alkynes with formaldehyde leading to α,β-butenolides was reported [141]. For example:



An efficient method to synthesize the cyclopentane skeleton under mild reaction conditions (room temperature, 1 bar CO) by means of a stoichiometric amount of iron and a catalytic amount of nickel halide was found [142]. For example:



The palladium(II) chloride-catalyzed cyclocarbonylation of propargylic amines with $CuCl_2$ and benzoquinone afforded (*E*)-α-chloroalkylidene-β-lactams in moderate to good yields [149]. For example:



80% isolated yield

2.4. Aminocarbonylation, amidocarbonylation, and hydroaminomethylation

Palladium-catalyzed aminocarbonylation of iodoferrocene with amino acid esters as nucleophiles was found to result in the selective formation of *N*-ferrocenyl amino acid esters in the presence of Et_3N as the base. The use of DBU leads to the formation of new *N*-ferrocenylglyoxyl amino acid derivatives [150]. Palladium-catalyzed aminocarbonylation proved to be an efficient method for the functionalization of iodobenzene and iodoalkenes (1-iodo-cyclohexene and 17-iodo-androst-16-ene) using amino acid methyl esters as amine nucleophiles. 2-Oxo-carboxamide type derivatives were isolated in up to 82% isolated yields as a result of double carbonylation of iodobenzene at 50 °C, 40 bar CO, in DMF solution. On the other hand carboxamides were obtained selectively in up to 88% isolated yield from iodoalkenes under 1 bar CO [151].

Aromatic acyl sulfonamides were prepared using palladium-catalyzed amidocarbonylation. Molybdenum hexacarbonyl was found to be a convenient CO-releasing reagent in the preparation of acyl sulfonamides from both aryl iodides and aryl bromides [152].

Hydroaminomethylation of arylethylenes with anilines was found to proceed under mild conditions in the presence of $[Rh(COD)_2]BF_4$ and dppf as catalyst system [153]. The rhodium-catalyzed hydroaminomethylation of 1-octene with morpholine was studied using temperature-dependent solvent systems consisting of propylene carbonate, an alkane and a semi-polar mediator such as *N*-octylpyrrolidone. The conversion of 1-octene and the selectivity to the corresponding amine is 92%. After the reaction the catalyst can be easily recovered by a simple phase separation with only a negligible loss of rhodium [154].

The application of a highly regioselective rhodium-bisphosphite catalyst in the synthesis of biologically active tertiary amines was reported [155].

Selective rhodium-catalysts were used for sequential hydroformylation/reductive amination of dendritic perallylated polyglycerols with various amines in a one-pot procedure to give dendritic polyamines in high yields. The dendritic polyamines were obtained in high to excellent isolated yields after simple purification by dialysis [156].

A new synthesis of 4-dialkylamino-1,5-dihydropyrrol-2-ones by PdI_2-catalyzed oxidative aminocarbonylation of 2-ynylamines was described [157].

See also [133,145].

2.5. Water–gas shift reaction and reduction with CO or $CO + H_2O$

Poly(4-vinylpyridine)-immobilized copper catalysts under CO in basic (NaOH, Et_3N or NaOAc) aqueous 2-ethoxyethanol medium was found to catalyze the water–gas shift reaction [158].

The immobilized catalyst obtained from dichlorocopper(II) and a poly(4-vinylpyridine)/divinylbenzene (2%) polymeric matrix was found to catalyze the water–gas shift reaction as well as the reduction of nitrobenzene to aniline and azobenzene [159].

See also Refs. [63,64,114–116].

2.6. Reduction of CO_2

Highly active ruthenium catalysts for the production of formamide from amines, hydrogen and carbon dioxide were prepared *in situ* by adding 1,2-bis(diphenylphosphino)ethane or triphenylphosphine to the reaction mixture containing $RuCl_3$ as the precatalyst [160].

Reductions of CO_2 on platinum electrodes modified with RuO_2/TiO_2 nanotubes and nanoparticles were investigated in aqueous solution by cyclic voltammetry, steady state polarization and electrolysis. The prolonged potentiostatic electrolysis of CO_2 led to the formation of methanol even under low overpotential. The current efficiency for conversion of CO_2 to methanol was found to be up to 65% on the RuO_2/TiO_2 nanotube-modified platinum electrode [161]. The electrochemical reduction of carbon dioxide was studied on a glassy carbon electrode modified with either polymeric M-tetrakis aminophthalocyanines (M = Co, Ni and Fe) or with the polymeric free ligand in aqueous electrolyte. The reaction products were found to depend on the central ion: for cobalt-polymer the only reaction product is formic acid, for nickel-polymer formic acid and formaldehyde, whereas formaldehyde and hydrogen are detected for iron-polymer [162].

The nickel complexes, $[Ni_2(\mu\text{-dppa})_2((\mu\text{-CNR})(CNR)_2)]$ (dppa = bis(diphenylphosphino)amine, R = Me, ^{n}Bu, 2,6-$Me_2C_6H_3$) were found to be one-electron electro-catalysts for the reduction of carbon dioxide. The products of carbon dioxide electro-reduction in the presence of these complexes are mainly CO and CO_3^{2-}, with small amounts of formate formed when residual water is present [163].

The catalytic reduction of supercritical carbon dioxide by hydrogen to carbon onion particles using thiaplatinacycles as the catalysts at 40 °C was investigated [164].

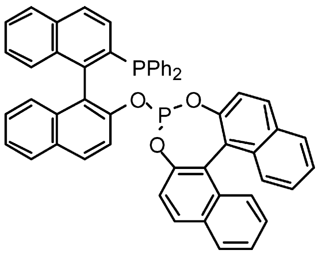
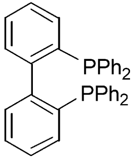
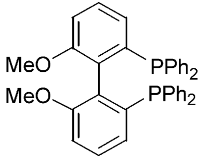
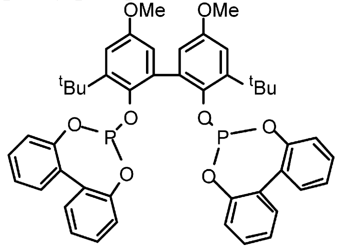
3. Reviews

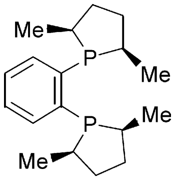
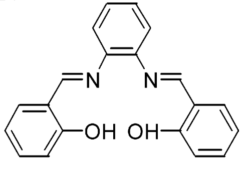
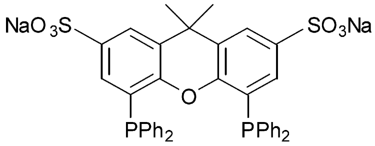
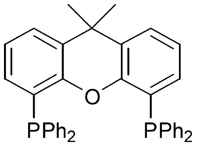
(1) The multi-substrate screening of asymmetric catalysts. A review of the principles and application of the one-pot multi-substrate screening in various types of catalyzed enantioselective reactions, such as hydroformylation of olefins with 39 references [165].
(2) Application of hybrid DFT/molecular mechanics to homogeneous catalysis. A review with 129 references [166].
(3) Branched selective hydroformylation. A useful tool for organic synthesis. A review with 90 references [167].
(4) Polymeric rhodium-containing catalysts in olefin hydroformylation. A review compiling the main results obtained by studying hydroformylation of olefins on polymeric rhodium-containing catalysts with 22 references [168].
(5) New directions in supramolecular transition metal catalysis. A review with 192 references [169].
(6) Supramolecular approaches to generate libraries of chelating bidentate ligands for homogeneous catalysis. A review of self-assembly of chelating ligands using a combinatorial approach with 59 references [170].
(7) Dendrons and dendritic catalysts immobilized on solid support: synthesis and dendritic effects in catalysis. A review of synthesis and catalytic application of dendritic catalysts among others in hydroformylation with 114 references [171].
(8) *P*-Modular bis(phosphines) based on the 1,2-*trans*-disubstituted cyclopentane framework in synthesis, coordination chemistry, and catalysis. A review of applications of versatile bidentate phosphines having a homochiral 1,2-disubstituted cyclopentane backbone among others in transition metal-catalyzed hydroformylation with 122 references [172].
(9) Supported ionic-liquid phases. Recent advances made in the area of supported ionic-liquid catalysis were reviewed with 63 references [173].
(10) Studies of hydroformylation of higher olefins. Recent progress in the development on homogeneous and biphasic systems was reviewed with 61 references [174].
(11) The development in hydroformylation of higher olefin in liquid/liquid biphasic systems. A review with 69 references [175].
(12) Recent advances in cyclohydrocarbonylation reactions. A review of recent advances in the cyclohydrocarbonylation, amidocarbonylation, and aminocarbonylation reactions covering the relevant literature up to the middle of 2004 with 47 references [176].
(13) Progress in hydroaminomethylation of olefins. A review with 35 references [177].

Acknowledgment

The author thanks the Hungarian Science Fund for support under Grant No. OTKA T037817.

Appendix A. List of abbreviations

Ac	acetyl
acac	acetylacetonate
Ar	aryl
BINAPHOS	
BIPHEP	2,2′-bis(diphenylphosphino)-1,1′-biphenyl 
MeO-BIPHEP	(6,6′-dimethoxybiphenyl-2,2′-diyl)-  bis(diphenylphosphine) 
BIPHEPHOS	
[bmim]$^+$	1-butyl-3-methylimidazolium cation
Bn	benzyl
bpy	2,2′-bipyridine
Bu	butyl
^{i}Bu	isobutyl
COD	1,5-cyclooctadiene
Cp	cyclopentadienyl
Cp*	pentametylcyclopentadienyl
dba	dibenzylideneacetone
DBU	1,8-diazabicyclo[5.4.0]undec-7-ene
DMF	*N*,*N*-dimethylformamide
DFT	density functional theory
dppa	$\mathrm{Ph_2PN(H)PPh_2}$
dppb	$\mathrm{Ph_2PCH_2CH_2CH_2CH_2PPh_2}$
o-DPPB	*ortho*-diphenylphosphinylbenzoate
dppe	$\mathrm{Ph_2PCH_2CH_2PPh_2}$
o-dppf	*ortho*-(diphenylphosphinyl)ferrocenylcarbonyl
ee	enantiomeric excess
Et	ethyl
FTIR	Fourier transform infrared
^{c}Hex	cyclohexyl
Me	methyl

Me-DuPHOS	
NBD	norbornadiene
NHC	nitrogen-heterocyclic carbene
Ph	phenyl
^{i}Pr	isopropyl
py	pyridine
	
salphen	supercritical
sc	supercritical
	
sulfoXANTPHOS	
TBS	*tert*-butyldimethylsilyl
THF	tetrahydrofuran
TMS	trimethylsilyl
TOF	turnover frequency (mol product/mol catalyst time)
TPPDS	disodium salt of the disulfonated triphenylphosphine
TPPMS	sodium salt of the monosulfonated triphenylphosphine
TPPTS	trisodium salt of the trisulfonated triphenylphosphine
Ts	tosyl (*p*-toluenesulfonyl)
TsOH	*p*-toluenesulfonic acid
XANTPHOS	

Appendix B. The metals and their associated references

Metal	References
Ti	[161]
Zr	[85]
V	[71]
Mo	[72,136,137,152]
W	[46,82]
Fe	[39,40,102,142,150]
Ru	[21,22,63–66,111,160,161]
Co	[1–5,21,22,69,107,138,139,162]
Rh	[5–62,67,70,73,75–101,103–105,108,110,112–116,139–141,153–157,166–168,171,176]
Ir	[67,109]
Ni	[59,142,162,163]
Pd	[21,117–135,137,143–152,157]
Pt	[68,106,146,164]
Cu	[148,149,158,159]
Zn	[59]
Sn	[68,106,127,146,147]

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