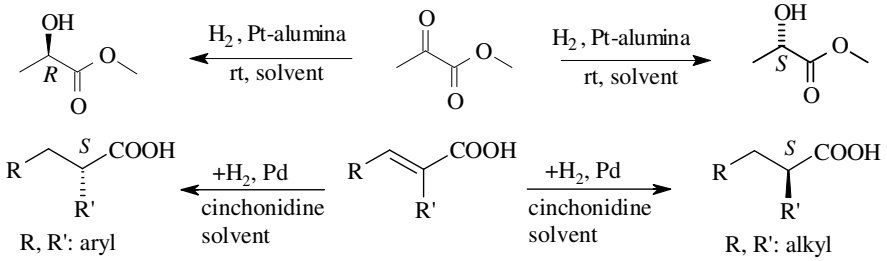
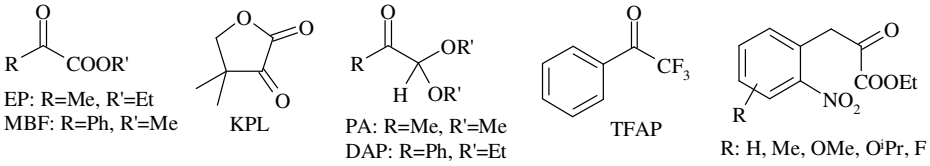
The synthesis of optically active compounds is an important field of organic chemical research. For well-known reasons (separation, recovery, recycling, stability, handling, environmental and safety considerations), heterogeneous enantioselective catalytic hydrogenations have gained an outstanding importance.

Probably the best-characterized such reactions are the hydrogenations of α-keto esters and prochiral activated olefins in which enantioselectivities exceeding 96–98% have been attained during our studies, too. The catalysts in these reactions are Pt- and Pd-modified by cinchona alkaloids.

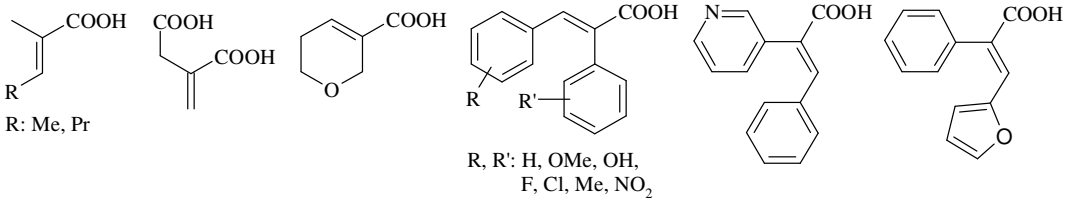


The main objectives of recent studies on these hydrogenations have been to expand its field of utilization, to elucidate the reaction mechanism, and to interpret chiral induction in this context. The results published in this field have been reviewed regularly (e.g. since 2007 [1-4]). It is generally accepted that the intermediate responsible for enantioselection is the 1:1 complex of the cinchona alkaloid as chiral modifier and the substrate in the hydrogenation of activated ketones. The structure of the intermediate complex however may differ in the hydrogenation of activated olefins depending on the structure of the acid, i.e. bearing aliphatic or aromatic substituents. No consensus has been reached, however, concerning the structure of this intermediate in both catalytic systems. The following substrates were examined in the enantioselective hydrogenations.

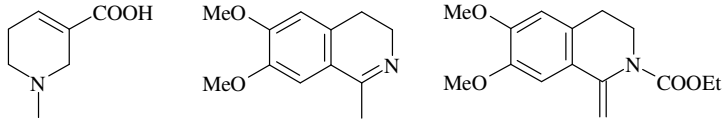
Activated ketones:



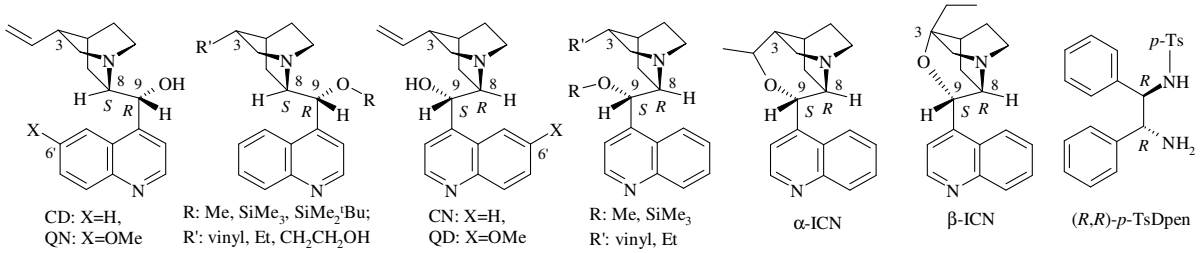
Prochiral unsaturated compounds:



Prochiral N-containing compounds:

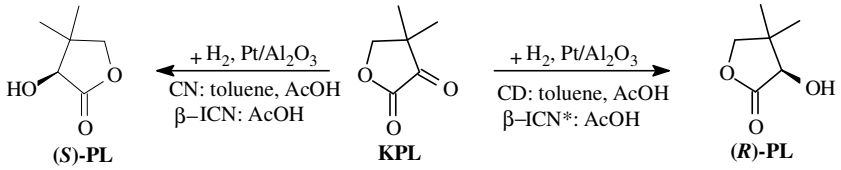


Compounds used as chiral modifiers: optically pure amino alcohols such as natural cinchona alkaloids and their derivatives and chiral diamine derivatives (*R*,*R*-*p*-TsDpen and *S*,*S*-*p*-TsDpen):



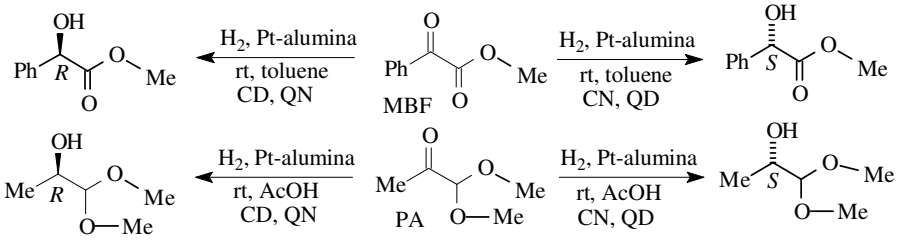
The results of these studies were published in 31 reports (∑IF 124.966), which were cited until now 289 times in independent papers. The most important results obtained in enantioselective hydrogenations of **activated ketones** over modified **Pt catalysts** and the conclusions reached based on these are summarized below.

Experimental evidence (measured by NMR) was given for the correlation between the solution-state concentration of the nucleophilic 1:1 modifier–substrate complex and the ee on enantioselective hydrogenation of KPL (ketopantolactone) using Pt–*β*-isocinchonine chiral catalyst (Pt/β-ICN). The relationship displays a saturation-type curve, which may indicate an underlying adsorption process involving the catalytically relevant nucleophilic complex [5]:



∗ inversion of enantioselectivity

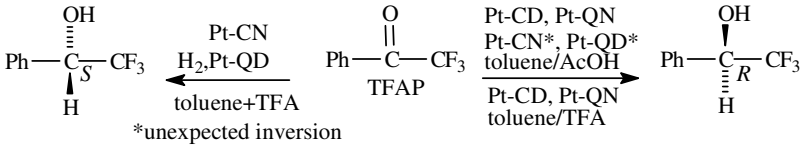
The nonlinear phenomenon (NLP) was studied for the first time in the enantioselective hydrogenation of EP (ethyl pyruvate), KPL, MBF (methyl benzoylformate) and PA (pyruvaldehyde dimethyl acetal) under identical conditions, on Pt catalyst modified by QN and CN, and for comparison with CD-CN modifiers pair. The order of the adsorption strengths of the parent cinchona alkaloids are: CD > CN > QN > QD and the results of the NLP measurements indirectly verify the so-called 1:1 model of enantioselection. The data obtained using the three methods allowed recognition of a new observation, namely that the NLP depends not only on the chiral modifier but also on the substrate to be hydrogenated. This observation can presumably be interpreted on the basis of differences in the structure of the substrate-modifier complexes formed and in the adsorption-desorption processes of the complexes, thus the NLP is not solely dependent on the adsorption of cinchona alkaloids, as suggested by earlier experimental data [6,7]:



The origin of rate enhancement in the enantioselective heterogeneous catalytic hydrogenation of EP on Pt modified with the parent cinchonas, as compared to the unmodified Pt, was studied in a solvent mixture toluene/acetic acid (AcOH) 9/1. Hydrogenation experiments were carried out in a continuous flow fixed-bed reactor (CFBR) and in several cases for comparison in batch reactors over E4759 Pt/Al_2O_3 catalyst. Our results obtained using a novel procedure, namely racemic hydrogenation followed by three changes of the chiral modifier (on the same catalyst) supported an intrinsic, kinetic character of the rate enhancement in EP hydrogenation [8].

The hydrogenations of MBF, KPL and PA were also carried out in CFBR system over Pt/Al_2O_3 catalyst. In our opinion, RE produced by the first modifier added after racemic hydrogenation and following further exchanges of modifiers (i.e. the regular dynamics of the changes in conversion) are indicative of the intrinsic-kinetic character of the phenomenon. This research suggested that the origin of enantiodifferentiation and RE is the same, namely, both may be traced back - probably in different ways - to the role of the intermediate complexes of the hydrogenation, to its formation and transformation, which in turn depends on numerous factors [9].

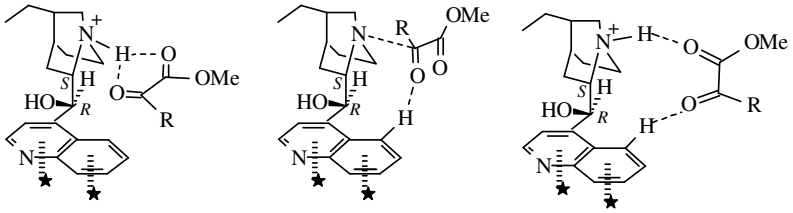
The enantioselective hydrogenation of TFAP (2,2,2-trifluoroacetophenone) was investigated for the first time using CFBR system in absence and presence of 0.1 v/v% trifluoroacetic acid (TFA). The enantioselective hydrogenations yielded the (*R*)-product in excess on Pt–CD, Pt–CN, Pt–QN and Pt–QD catalysts in toluene/AcOH mixture; consequently, unexpected inversion took place on the Pt–CN and Pt–QD catalysts [10,11]:



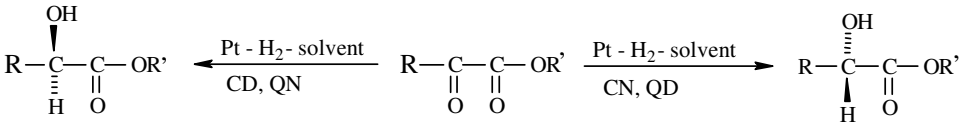
The experimental data of the racemic – cinchona 1 – cinchona 2 – cinchona 1 series in the hydrogenation of TFAP confirm the intrinsic-kinetic nature of rate enhancement, namely the so-called "ligand acceleration" phenomenon [12]. Hydrogenation in the presence of 0.1% (v/v) TFA follows the general rule of the Orito reaction, according to which the products formed in excess are (*R*)-alcohols on Pt-CD and Pt-QN and (*S*)-alcohols on Pt-CN and Pt-QD chiral catalysts. In toluene/AcOH mixture without TFA, unexpected inversion took place on the Pt-CN and Pt-QD catalysts since the (*R*)-product formed in excess instead of the (*S*)-product. The observed unexpected inversion can be interpreted on the basis of the nucleophilic intermediate complex. Based on these observations we propose that in the hydrogenation of TFAP the reaction route involves the equilibrium of electrophilic and nucleophilic intermediate complexes, which was found to be dependent on the acid strength and concentration [13].

A study on the hydrogenation of EP, MBF and TFAP over Pt–cinchona alkaloid chiral catalysts and over the "unmodified" catalysts resulted after a cleaning step at 323 K of the chirally modified surfaces in CFBR system is presented. According to these investigations the sense of the residual ee observed in the reactions carried out in the absence of modifiers over the catalysts rinsed after the chiral hydrogenations was influenced by the solvent and the structure of the activated ketone, pointing on the effect of these parameters on the structure of the adsorbed intermediate complexes and implicitly on the chiral induction [14].

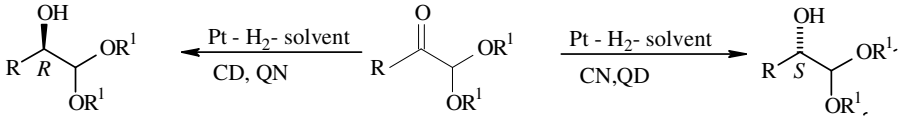
In competitive racemic hydrogenation of MBF + EP binary mixture over Pt/Al_2O_3: $k_{MBF} > k_{EP}$, but in competitive enantioselective hydrogenation $k_{MBF} < k_{EP}$; the phenomenon verified for the first time is dependent on the adsorption strength of the surface complexes of various compositions (MBF–Pt, EP–Pt, MBF–CD–Pt, EP–CD–Pt) [15]. Reaction rates of chiral and racemic hydrogenations were determined and relative adsorption coefficients were calculated in the competitive chiral hydrogenation of EP+MBF, EP+TFAP and KPL+MBF binary mixtures. A new phenomenon was observed: namely the EP and KPL are hydrogenated faster than MBF and TFAP, whereas in racemic one the MBF and TFAP are hydrogenated faster than EP or KPL. Effects of the activated ketones structure on their reactivity and the stability of the surface complexes were discussed. Differences in rate enhancement are caused by the differences both in the adsorptivity and in the reactivity of adsorbed substrates and adsorbed intermediate complexes [16].



In the competitive chiral hydrogenation of MBF + EP and DAP + PA binary mixtures (S1 + S2) over Pt-CD a new phenomenon was observed: namely the EP and PA are hydrogenated faster than MBF and DAP, whereas in racemic one the MBF and DAP are hydrogenated faster than the former ketones. The phenomenon verified for the first time in CFBR is dependent on the adsorption mode of the surface complexes of various compositions (S1–Pt, S2–Pt, S1–CD–Pt, S2–CD–Pt). In the chiral hydrogenation of DAP a rate decrease, i.e., ''ligand deceleration'' was observed instead of rate enhancement [17,18]:

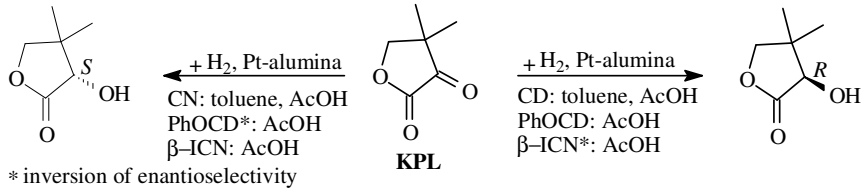


EP: R = Me, R' = Et MBF: R = Ph, R' = OMe

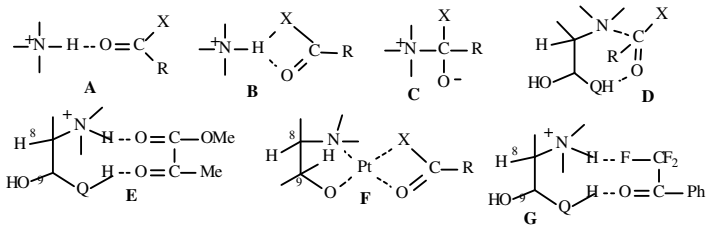


PA: R = R' = Me DAP: R = Ph, R' = Et

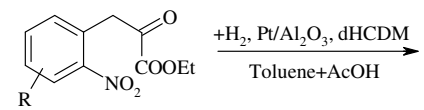
The enantioselective hydrogenation of KPL on Pt–alumina catalyst modified by β-ICN and PhOCD in toluene, acetic acid and their mixtures under otherwise identical experimental conditions was studied. Reversal of the enantioselection was obtained dependent on the concentration of acetic acid (eemax=17% (*S*) on Pt–PhOCD and 50% (*R*) on Pt–β-ICN, respectively). The possible role in enantioselection of adducts forming in the reaction mixture and the stability of PhOCD under the conditions of the hydrogenation was investigated by ESI-MS. The results of the NLP measurements on β-ICN+PhOCD mixtures suggest that the intermediate surface complexes β-ICN–KPL and PhOCD–KPL responsible for the opposite enantioselection include various types of interactions (e.g. A-E) and the enantioselection is directed by the competition between these interactions [19].



∗ inversion of enantioselectivity

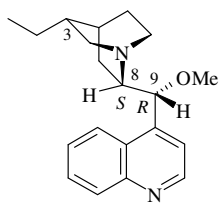


We have developed the first heterogeneous catalytic asymmetric cascade reaction for the synthesis of tetrahydroquinolines from 2-nitrophenylpyruvates. Optically enriched 3-hydroxy-3,4-dihydroquinolin-2(1H)-ones are prepared by enantioselective hydrogenation of the activated keto group over a cinchona alkaloid-modified Pt catalyst, reduction of the nitro group and spontaneous cyclization by intramolecular amidation cascade. Acceleration of the enantioselective hydrogenation of the activated keto group over the catalyst modified by cinchona alkaloids ensured high tetrahydroquinolinone selectivities. The scope of the reaction was checked using twelve substrates. Both yields and enantioselectivities were significantly influenced by the nature and position of the substituents on the phenyl ring. Substituents adjacent to the nitro group considerably increased the product yield (up to 98%), due to their effect on the nitro group's reduction rate [20].



R: H, Me, OMe, O^{i}Pr, F

	yield (%)	ee (%)
R: H,	65	90
R: 8-Me	97	90
R: 8-OMe	97	88
R: 8-O^{i}Pr	97	85
R: 6-Me	88	86
R: 5-Me	55	89
R: 5-F	35	90

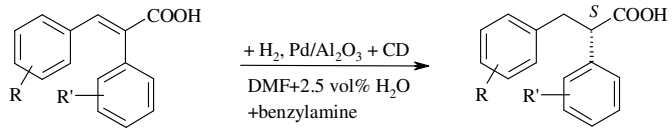


dHCDM

Our studies on the enantioselective hydrogenations of **activated olefins**, i.e. *prochiral unsaturated carboxylic acids* over **Pd catalysts** in presence of chiral modifiers led to the following results and conclusions on the mechanism of these reactions.

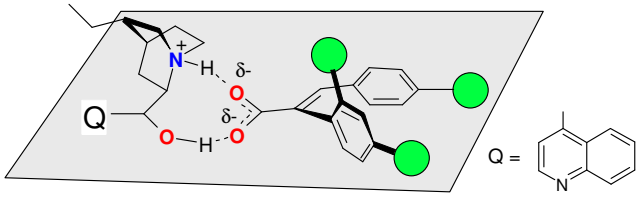
By the time our studies were initiated it was already known that the best enantioselectivities are obtained in the hydrogenation of (*E*)-2,3-diphenylpropenoic acid derivatives over Pd catalysts modified by CD and the methoxy substituents in para positions of both phenyl ring increase the optical purity of the products.

In our studies we have showed using methoxy derivatives of (*E*)-2,3-diphenylpropenoic acid that the position of the substituent has a decisive influence on the reaction rate and the enantioselectivity. High enantioselectivities, 86–90%, were obtained in the hydrogenation of derivatives with a favorable substituent position. The results were rationalized in terms of either the electronic or the steric effects of the methoxy substituent. These suggestions were also applicable in interpreting the results of the hydrogenation of dimethoxy (*E*)-2,3-diphenylpropenoic acids. The combined steric and electronic effects of the substituents on both phenyl rings ensured up to 92% ee in the hydrogenation of (*E*)-2-(2-methoxyphenyl)-3-(4-methoxyphenyl)propenoic acid [21]. The above conclusions were confirmed using fluorine and methyl substituted compounds. The fluorine substituent in the appropriate position was even more efficient than the methoxy group in increasing the optical purity. High enantioselectivities, up to 96%, were obtained in the hydrogenation of some disubstituted derivatives, unprecedented in the hydrogenation of unsaturated carboxylic acids over heterogeneous catalyst. The influence of the substituent on the β phenyl ring was attributed to the increase in the efficiency of the modifier-substrate interaction by electronic effects or to a decrease in the adsorption strength of the acid over modified surface sites. The beneficial effect of the ortho-substituent on the α phenyl ring was assumed to be due to the additional interaction of this substituent with the modifier on the surface [22]. The hydrogenation of two 2,3-diarylpropenoic acids bearing heteroaromatic substituents, i.e. (*E*)-2-phenyl-3-(2-furyl)propenoic acid and (*E*)-2-(3-pyridyl)-3-phenylpropenoic acid was also studied. These compounds could be selectively hydrogenated to saturated acids in the presence of CD [23].



R=H, R'=H: ee 80%;
R=H, R'=2-OMe: ee 86%; R=3-OMe, R'=H: ee 90%;
R=4-OMe, R'=H: ee 89%; R=4-OMe, R'=4-OMe: ee 90%;
R=3-OMe, R'=4-OMe: ee 88%; R=4-OMe, R'=2-OMe: ee 92%;
R=4-OMe, R'=4-F: ee 90%; R=4-OMe, R'=2-F: ee 93%;
R=4-F, R'=2-OMe: ee 96%; R=3-F, R'=2-OMe: ee 93%;
R=3-F, R'=4-F: ee 89%; R=4-F, R'=2-F: ee 96%;
R=3-F, R'=2-F: ee 90%; R=4-Me, R'=2-OMe: ee 89%.

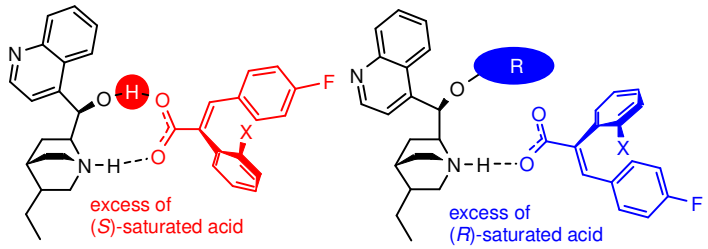
Our studies were extended to (*E*)-2,3-diphenylpropenoic acid derivatives bearing substituents which may interact or may be transformed on the Pd surface. The effect of the chlorine position on the C–Cl bond hydrogenolysis and the enantioselective hydrogenation of Cl substituted (*E*)-2,3-diphenylpropenoic acid derivatives has been studied. In contrast to the fast hydrodechlorination of the *para*-Cl-3-phenyl- substituted acids the Cl on the 2-phenyl ring was barely hydrogenolized. These observations were interpreted by the different arrangements of the two phenyl rings on the surface, with the 2- and 3-phenyl rings adsorbed tilted and parallel, respectively. The 2,3-diphenylpropionic acids substituted by Cl on the 2-phenyl ring could be prepared in good yields and optical purities (up to ee 92%). The conclusions were used for the rational design of an acid, i.e. (*E*)-2-(2-methoxyphenyl)-3-(3,4-difluorophenyl)propenoic acid, which afforded the best optical purity (ee up to 95% at 295 K) in this system [24].



The effect of the acidic hydroxyl substituents was compared with that of the methoxy group in the same position. The 4-OH substituent on the 3-phenyl ring had similar effect on the enantioselectivity as the methoxy group, whereas the 3-OH decreased the optical purity of the saturated acid. This was explained by different origin of the effect of these substituents. It was also

demonstrated that the CD modified Pd catalyst is appropriate for the preparation of hydroxy-substituted 2,3-diphenylpropionic acids in good optical purities [25]. The hydrogenation of nitro-substituted (*E*)-2,3-diphenylpropenoic acids over Pd catalyst modified by CD showed that the reaction route is determined by the position of the substituent, which was indicated by the H_2 consumption rate and the enantioselectivity of the final amino acid or 3-phenyl-1,2,3,4-tetrahydro-2-quinolone formed. This study was the first to report the enantioselective heterogeneous catalytic preparation of 1,2,3,4-tetrahydro-2-quinolones and presents an attractive method for obtaining optically enriched 3-(4-aminophenyl)-2-phenylpropionic acids [26].

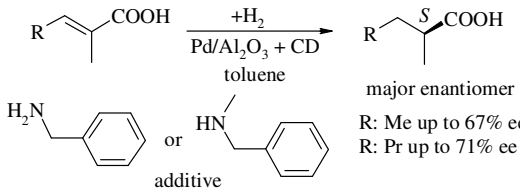
The effect of the structure of the cinchona alkaloid used as modifier was also studied. The use of CN and QD ethers resulted in the inversion of the sense of the enantioselectivity in the hydrogenation of (*E*)-2,3-diphenylpropenoic acids. The investigations indicated that the interaction of the ether derivatives with the unsaturated acids is more flexible and the presence of the ether group reshapes the chiral surface sites. This may lead to inversion of the docking preference of the substrates in the altered chiral pocket of the adsorbed modifier [27]. In the CD series larger substituents were needed to obtain inversion, such as the tert-butyl-dimethylsilyl group. To find an explanation of the phenomenon the modifiers' relative adsorption strengths were studied using mixtures of cinchona alkaloids. Decrease in the interaction strength of the cinchona ether derivatives with the acid and the catalyst surface explained the observed decrease in the ee or the inversion of its sense. These studies suggested the gradual alteration of the shape of the chiral sites by increasing the size of the substituent. The presence of benzylamine always increased the amount of the enantiomer formed in excess using the parent cinchona alkaloids and accelerates desorption of the modifier, suggesting the participation of the additive in the surface intermediate [28].

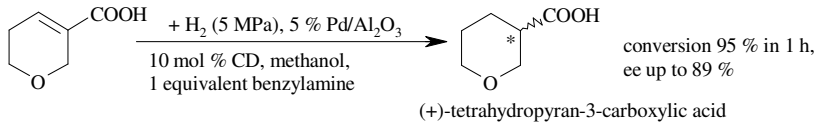


Due to its high practical importance the enantioselective hydrogenation of aliphatic unsaturated acids is also a frequently studied reaction. Similarly with the cinnamic acid derivatives, achiral amines are efficient additives for increasing the optical purity of the aliphatic acids, too.

The effect of the structure of the achiral primary amine on the enantioselective heterogeneous catalytic hydrogenation of (*E*)-2-methyl-2-butenoic acid over CD modified Pd/Al_2O_3 was studied. It was found that a variety of amines increase the enantioselectivity accompanied by decrease in the initial rate of the hydrogenations. Based on these results participation of the additive in the formation of the intermediate complex responsible for enantioselection was suggested [29]. We have also studied the effect of the achiral amine additive structure on the enantioselective hydrogenation of (*E*)-2-methyl-2-butenoic acid and (*E*)-2-methyl-2-hexenoic acid. Secondary amines were similarly or even more efficient in increasing the enantioselectivity as primary amines. The basicity of the amine may vary in a rather wide range, but appropriate steric properties are also needed for increasing the enantioselectivity. The best performing amines were the benzylamine and *N*-methylbenzylamine. The effect of the amine amount on the hydrogenation was also investigated. The involvement of the amine additive in the formation of surface intermediates responsible for enantioselection was proposed. Decrease of the reaction temperature increased the ee (up to 71%) in the presence of amines, the highest value obtained until now in the enantioselective hydrogenation of aliphatic unsaturated carboxylic acids over chirally modified heterogeneous catalyst [30].

We have developed a novel application of the cinchona-modified supported Pd catalytic system. The key step in the asymmetric synthesis of the cockroach attractant methyl (+)-tetrahydro-2H-pyran-3-carboxylate was the enantioselective hydrogenation of 5,6-dihydro-2H-pyran-3-carboxylic acid in up to 89% optical purity [31].

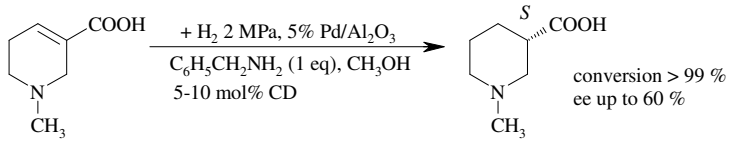




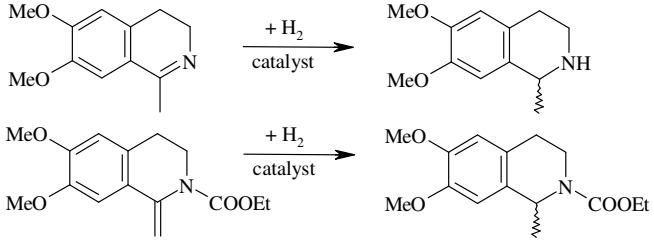
(+)-tetrahydropyran-3-carboxylic acid

We have reported for the first time the application of nanotube-supported Pd and Pd nanoparticle-graphene catalysts in the enantioselective hydrogenation of α,β-unsaturated carboxylic acids using CD as chiral modifier. The Pd particles were prepared by deposition– precipitation from the aqueous phase and subsequent reduction in flow of H_2 or using $NaBH_4$. The novel materials were characterized by thermal analysis, X-ray diffraction spectroscopy, transmission and scanning electron microscopy, ICP optical emission spectroscopy, Raman spectroscopy, and X-ray photoelectron spectroscopy. Both types of materials were found efficient in catalyzing the enantioselective hydrogenation of several α,β-unsaturated carboxylic acids.

Our studies included the development of novel methods for the enantioselective preparation of chiral ***N*-heterocyclic compounds**, which are known to be of high pharmaceutical importance. Accordingly, we have extended the applicability of the Pd/Al_2O_3 catalyst modified by CD on the preparation of a *N*-heterocyclic carboxylic acid. Thus, the hydrogenation of *N*-methyl-3,4-dehydronipecotic acid (arecaidine) resulted in the quantitative formation of *N*-methylnipecotic acid in good (up to 60%) optical purity [34].



The preparation and application of novel heterogeneous Pd, Ir and Ru chiral catalysts were also attempted, which were tested in the enantioselective hydrogenations of *N*-heterocyclic compounds, namely 6,7-dimethoxy-3,4-dihydroisoquinoline and 1-methylene-2-ethoxycarbonyl-6,7-dimetoxy-3,4-dihydroisoquinoline in the presence of CD, (*S,S*)- and (*R,R*)-Ts-Dpen ligands. CD-modified metal catalysts exhibited low ee, whereas catalysts stabilized by triphenylphosphane and modified by (*S,S*)-Ts-Dpen afforded promising ee values (70–80%). Immobilized Ru(II)-aminophosphane complexes were found to be active in the hydrogenations producing the corresponding tetrahydroisoquinoline derivatives in high optical purities (up to 97%). Recycling of these catalysts showed constant or increasing activities in racemic hydrogenation, whereas the presence of the chiral ligands led to leaching of the active species in the liquid phase [35].



Finally, based on the experimental data published in the literature it seems justified to assume that the mechanism of enantioselective hydrogenations cannot be interpreted via a single generalizable surface intermediate complex; it may rather be the simultaneous presence of different types of interactions dependent on a number of factors (modifier, substrate, and solvent) that determines the sense of enantioselection [36-43].

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