Russian Journal of Organic Chemistry, Vol. 39, No. 7, 2003, pp. 926–932. Translated from Zhurnal Organicheskoi Khimii, Vol. 39, No. 7, 2003, pp. 987–993.

Dedicated to Full Member of the Russian Academy of Sciences I.P. Beletskaya on Her Jubilee

Double Stereoselection in the Hydrogenation over Cationic Rh(I) Complexes with Two Different Chiral Ligands

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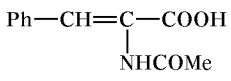
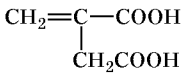
Received July 5, 2002

Abstract—The catalytic activity and stereoselectivity in the hydrogenation of itaconic and α-(acetylamino)-cinnamic acids were studied in the presence of the complex $[Rh(COD)(L^1)_2]^+ TfO^-$ (where COD is cyclooctadiene and L^1 is (1*S*,2*S*,5*R*)-(+)-neomenthyldiphenylphosphine] which was generated *in situ*. The optical yield of the hydrogenation of itaconic acid increases both on addition of chiral (4*S*,5*S*)-(+)-2,2-dimethyl-4,5-bis-(dimethylaminomethyl)-1,3-dioxolane (L^2) as an auxiliary ligand to the complex $[Rh(COD)(L^1)_2]^+ TfO^-$ and on addition of achiral and chiral tertiary phosphines to the complex $[Rh(L^2)_2]^+ TfO^-$. The result of joint action of two ligands can be regarded as "matched effect." Transformations of the complexes in a hydrogen atmosphere were examined by ^1H and ^{31}P NMR spectroscopy. It was found that at least three complexes: diamine complex $[Rh(L^2)_2]^+ TfO^-$, solvate complex $[Rh(L^1)_2(solv)_2]^+ TfO^-$, and diamine–bis-phosphine complex $[Rh(L^1)_2L^2]^+ TfO^-$ may be catalytic precursors.

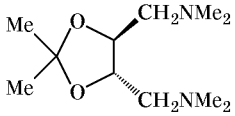
We previously showed that hydrogenation of α-(acetylamino)cinnamic (**I**) and itaconic acids (**II**) over rhodium catalyst with a chiral diamine ligand, (4*S*,5*S*)-(+)-2,2-dimethyl-4,5-bis(dimethylaminomethyl)-1,3-dioxolane (**III**, L^2) is characterized by moderate enantioselectivity: the optical yields do not exceed 32 and 15%, respectively. Addition of phosphine ligands, such as triphenylphosphine or 2,3-*O*-isopropylidene-2,3-dihydroxy-1,4-bis(diphenylphosphino)butane, strongly affect both the substrate conversion and the optical yield of the hydrogenation products; here, addition of an optically active bis-phosphine changes the sign of asymmetric induction [1]. ^{31}P NMR study of the transformations occurring in the catalytic system in the presence of PPh_3 showed that bis-phosphino–diamine complexes are formed therein. In continuation of our studies on enantioselective hydrogenation over rhodium complexes with chiral bis-phosphine and diamine ligands [1–3], in the present work we examined the hydrogenation of unsaturated acids **I** and **II** over trifluoromethanesulfonate rhodium(I) complex with optically active (1*S*,2*S*,5*R*)-(+)-neomenthyldiphenylphosphine (**IV**, L^1), $[Rh(COD)(L^1)_2]^+ TfO^-$ (**V**), which was generated *in situ*. Also, complex **V** with addition of diamine **III** and trifluoromethanesulfonate rhodium(I) complex with diamine **III** (L^2), $[Rh(L^2)_2]^+ TfO^-$ (**VI**), in the presence of phosphine **IV** were tested.



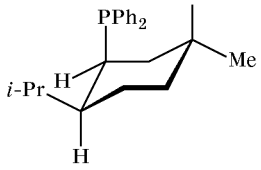
Z-**I**



II



III



IV

In the two latter cases, joint action of two chiral ligands could lead to different optical yields of the hydrogenation products than those obtained over complexes **V** and **VI** (double stereoselection). The term *double stereoselection* or *double stereodifferen-*

tiation means a selection between diastereoisomeric combinations of two independent chiral systems which form intermediate at the stage determining the stereoselectivity of the process. Here, two independent chiral systems may be a ligand and a substrate [4, 5], two different ligands [6, 7], or parts of the same ligand [8]. Diastereoisomeric combination of such chiral systems, leading to increased enantioselectivity as compared to each particular chiral system, is referred to as *matched*, and that giving rise to reduced enantioselectivity, as *mismatched* [8, 9]. A multiplicativity rule has been postulated, according to which the stereoselectivity of a process is equal to the product of stereoselectivities for each chiral pair in the case of their matched combination and to the quotient in the case of mismatched combination [9]. A combination of two chiral ligands **III** and **IV** in the system under study allows us to examine the effect of double stereodifferentiation in homogeneous catalytic hydrogenation, which was studied previously with a combination of a chiral ligand and a chiral substrate as an example [4, 5].

Compound **IV** was synthesized from naturally occurring (–)-menthol [10]; it was used in the hydrogenation of prochiral α-phenylacrylic (atropic) acid, (*E*)-α-methylcinnamic acid, and (*E*)-β-methylcinnamic acid over the complex $Rh(L^1)_3Cl$ (60°C, hydrogen pressure 20 atm) which was generated *in situ* [11, 12]. Optical yields of 28, 60, and 61% were achieved for (*S*)-2-phenylpropionic, (*R*)-2-methyl-3-phenylpropionic, and (*S*)-3-phenylbutanoc acids, respectively. Later on, Valentine *et al.* [13] succeeded in effecting hydrogenation of 3,7-dimethyl-2,6-octadienoic (geranic) acid to obtain optically active (*R*)-3,7-dimethyl-6-octenoic (citronellic) acid under mild conditions (23°C, hydrogen pressure 3 atm) over a dimeric covalent complex, $[Rh(COD)Cl]_2$ + **IV** in the presence of sodium methoxide (optical yield 65–70%; substrate-to-Rh ratio 75 : 100, reaction time 3 days). A fairly moderate catalytic activity was also observed in the hydrogenation of α-(acetylamino)-6-methylindole-3-acrylic acid in the presence of triethylamine: the conversion was as low as 29% in 7 days at a substrate-to-Rh ratio of 75 : 100; the optical yield and configuration of the hydrogenation product were not determined.

The results of hydrogenation of acids **I** and **II** in the catalytic system $[Rh(COD)_2]^+ TfO^-$–**IV** under mild conditions are presented in Table 1. As follows from these data, the cationic rhodium catalyst is more active in the hydrogenation of acid **II** under mild conditions (p_{H_2} = 1 atm, 20–60°C). With acid **I** in which the double bond is less accessible to coordination, lower chemical yields were obtained (cf. run nos. 1, 2, and 5; Table 1). The dependence of the rate of hydrogenation on the phosphine-to-[Rh] ratio and the formation of one enantiomer of the hydrogenation products in excess unambiguously indicate generation of phosphine rhodium complexes *in situ* and the presence of a chiral ligand in the rhodium coordination sphere at the stage determining the rate and enantioselectivity of the process. The hydrogenation of acid **II** gives preferentially the (*R*)-(+)-enantiomer of α-methylsuccinic acid, while in the hydrogenation of acid **I**, (*S*)-(+)-*N*-acetylphenylalanine is the major product.

Rhodium complexes with diamine **III** [1] and phosphine **IV** ensure similar signs of asymmetric induction in the hydrogenation of itaconic acid (**II**) (Table 1). Therefore, it was important to elucidate whether ligands **III** and **IV** are simultaneously incorporated into the catalytic rhodium–substrate complex and, if so, whether their effect is matched or mismatched (see above). Table 2 contains the results of hydrogenation of acids **I** and **II** over complex **VI** on addition of one or two equivalents of phosphine **IV**.

The optical yields achieved in the reaction over $[Rh(L^2)_2]^+ TfO^-$ (**VI**) are small. Presumably, this is explained not only by the nature of the diamine ligand (in which two σ-donor dimethylamino groups are linked through a chiral backbone) but also by gradual liberation of metallic rhodium during the hydrogenation (run no. 1; Table 2). In order to avoid this process, triphenylphosphine was added to the catalytic system (PPh_3-to-Rh ratio 1) (run nos. 2 and 3; Table 2), and the optical yield increased. Achiral phosphine ligand is likely to stabilize the rhodium complex with diamine **III** during the hydrogenation process via formation of a new complex containing both donor ligands in the metal coordination sphere. Another reason may be increased steric hindrance in the rhodium coordination sphere due to coordination of triphenylphosphine, which gives rise to a more rigorous selection of the *re* or *si* side of the double bond upon formation of a substrate complex, i.e., to increased difference in the free energies of diastereoisomeric olefin–rhodium complexes. The formation of a bisphosphine–diamine rhodium complex is confirmed by our ^1H and ^{31}P NMR studies [1]. They showed that $[Rh(COD)_2]^+ TfO^-$ reacts with molecular hydrogen in the presence of 2 equiv of triphenylphosphine and 1 equiv of diamine **III** to give a cationic bis-triphenylphosphine–diamine rhodium complex.

Addition of chiral phosphine **IV** to complex **VI** increases the optical yield of α-methylsuccinic acid to

Table 1. Hydrogenation of acids **I** and **II** in the catalytic system $[Rh(COD)_2]^+ TfO^-$–(1*S*,2*S*,5*R*)-(+)-neomenthyldiphenylphosphine (**IV**) (C_6H_6–MeOH, 1:2; c_{Rh} = 2 mM; reaction time 24 h; p_{H_2} = 1 atm)

Run no.	Phosphine-to-Rh ratio	Temperature, °C	Substrate (substrate-to-Rh ratio)	Yield, % %	Optical yield, %
1	1	20	**II** (25)	100	28 (*R*)
2	2	20	**II** (25)	25	45 (*R*)
3	2	40	**II** (50)	28	41 (*R*)
4	1	20	**I** (50)	23	52 (*S*)
5	2	60	**I** (50)	7.0[a]	–
6[b]	*in situ* RhL_3Cl or RhL_2ClS[c]	60	Atropic acid (*E*)-α-Methylcinnamic acid (*E*)-β-Methylcinnamic acid	Not given 100	28 (*S*) 60 (*R*) 61 (*S*)

[a] 84% of acid **I** and 9% of its methyl ester.
[b] Data of [11, 12].
[c] Hydrogen pressure 20 atm.

Table 2. Hydrogenation of α-(acetylamino)cinnamic acid (**I**) and itaconic acid (**II**) over $Rh(L^2)_2^+ TfO^-$ (**VI**) in the presence of phosphine **IV** (MeOH–benzene, 7:3; c_{Rh} = 2 mM; reaction time 5–48 h)

Run no.	Phosphine-to-Rh ratio	Reaction conditions		Substrate (substrat-to-Rh ratio)	Yield, %	Optical yield, %
		time, h	temperature, °C			
1[a]	0	24	40	**II** (25)	100	15 (*R*)
2	1 (PPh_3)	48	40	**II** (40)	73	17 (*R*)
3	1 (PPh_3)	12	20	**II** (25)	22	30 (*R*)
4	1	24	20	**II** (25)	35	62 (*R*)
5	1.9	24	20	**II** (25)	8	74 (*R*)
6	0.5	12	40	**II** (50)	20	36 (*R*)
7	1.0	24	40	**II** (50)	63	51 (*R*)
8	2.0	24	40	**II** (50)	18	72 (*R*)
9	1.2	24	60	**II** (25)	100	48 (*R*)
10	2	24	60	**II** (25)	21	65 (*R*)
11	1	24	60	**I** (50)	7.0[b]	
12	1	8 (20 atm)	20	**I** (50)	25.0	33 (*S*)
13	0	8 (35 atm)	20	**I** (40)	100	20 (*S*)

[a] The reaction was accompanied by gradual liberation of metallic rhodium.
[b] 73% of acid **I** and 20% of its methyl ester.

74% (run no. 5, Table 2), the stereoselection direction remaining unchanged. In this case, the optical yield exceeds those obtained with complex **VI** in the presence of achiral phosphine (PPh_3; 17% [1]) and with phosphine–rhodium catalyst in the presence of phosphine **IV** (run nos. 2 and 3; Table 1). This means that we observed a concerted stereochemical effect of two chiral ligands in the rhodium coordination sphere, i.e., matched effect.

The hydrogenation of acid **I** in the catalytic system $Rh(L^2)_2$–(+)-**IV** under elevated hydrogen pressure is characterized by increased chemical yield (run nos. 11 and 12; Table 2). The optical yield also increases in the presence of two chiral ligands (run nos. 12 and 13, Table 2). Systems containing phosphine **IV** act as Lewis acids toward acid **I** and thus catalyze its esterification with methyl alcohol (run no. 5, Table 1; run no. 11, Table 2).

Table 3. ^{1}H NMR spectral parameters of diamine ligand **III** and 1,5-cyclooctadiene in rhodium(I) complexes (δ, ppm; acetone-d_6)

Compound	CH	CH_A in CH_2	CH_B in CH_2	NCH_3	CCH_3
			Diamine **III**		
Diamine **III**	3.80	2.52	2.37	2.21	1.29
Complex **VI**	4.06	2.97	2.89	2.61	1.36
Complex **V** + diamine **III** (complex **VII**)	3.94	2.74	2.63	2.41	1.32
Complex **V** + diamine **III** + H_2	4.05	2.94	2.86	2.60	1.35
$[RhL^2–L^3]^+$ TfO^- [a] (complex **X**)	4.21	3.37	3.02	2.79	1.38
			1,5-Cyclooctadiene		
$Rh(COD)_2^+$ TfO^-	4.15	2.50	1.77		
$Rh(COD)(PPh_3)_2^+$ TfO^-	4.70	2.58	2.28		
Complex **V**	5.12 br	2.50 m [b]	2.50 m [b]		
Complex **V** + diamine **III** (complex **VII**)	4.18 br				

[a] L^3 is itaconic acid (**II**).

[b] Overlapped by the signals of diamine **III**.

Rhodium complexes with (+)-**IV** were reported as hydrogenation catalysts [10–13]; however, their spectral parameters were not obtained. We examined the reaction between components of the catalytic system by ^{1}H and ^{31}P NMR spectroscopy. Addition of 2 equiv of phosphine **IV** to a brick red solution of $[Rh(COD)_2]^+$ TfO^- in acetone-d_6 changes the color to yellow–orange, and in the ^{31}P NMR spectrum of the mixture a doublet appears at δ_P 26.42 ppm with a coupling constant $^1J_{P,Rh}$ of 141.3 Hz (in addition to the signal at δ_P –14.10 ppm from initial phosphine **IV**). The ^{1}H NMR spectrum contains signals from free cyclooctadiene at δ 5.5 (CH) and 2.32 ppm (CH_2). These data indicate coordination of phosphine **IV** to rhodium with formation of complex **V**.

After addition of 1 equiv of diamine **III** (with respect to Rh), the color changes insignificantly, but in the ^{31}P NMR spectrum the signal intensity ratio between the coordinated and free phosphine **IV** changes in favor of the latter. In the ^{1}H NMR spectrum, the signals from diamine **III** shift downfield: $\Delta\delta(CH) = 0.14$, $\Delta\delta(CH_A) = 0.22$, $\Delta\delta(CH_B) = 0.27$, $\Delta\delta(CH_3N) = 0.20$, $\Delta\delta(CH_3C) = 0.03$ ppm. This means that diamine **III** coordinates to rhodium to give new complex **VII**. Subsequent treatment of the solution with molecular hydrogen over a period of 20 min leads to appearance in the ^{31}P NMR spectrum of two new doublets at δ_P 55.15 ppm ($^1J_{P,Rh}$ = 204.2 Hz; complex **VIII**) and 78.45 ppm ($^1J_{P,Rh}$ = 190.4 Hz; complex **IX**) which are characterized by approximately equal intensities; the signal from complex **V** disappears. In the ^{1}H NMR spectrum, signals of diamine **III** shift even more downfield, approaching the positions typical of the same ligand in complex **V**. The ^{1}H NMR parameters are given in Table 3.

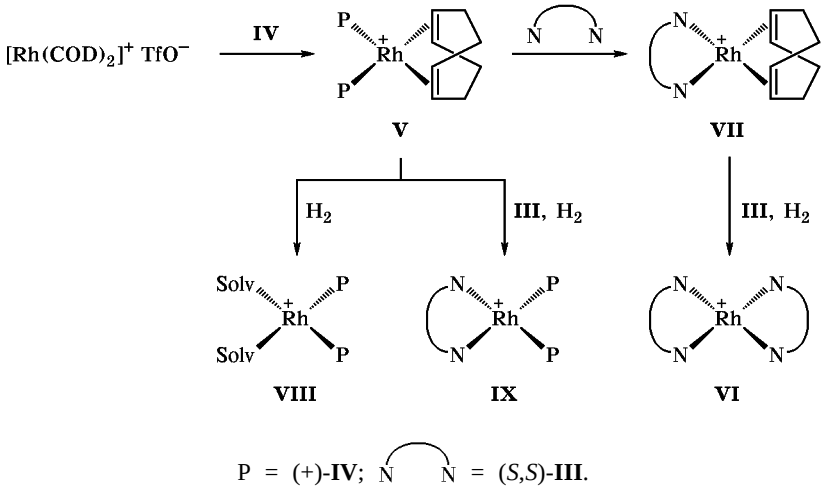
On addition of 1 equiv of phosphine **IV** to a solution of complex **VI** in acetone, only signals of the initial compounds were observed in the ^{1}H and ^{31}P NMR spectra. Introduction of itaconic acid and subsequent treatment with hydrogen over a period of 20 min leads to downfield shift of the ^{1}H signals of diamine **III** in complex **VI**, indicating formation of olefin complex **X** [1].

Thus, the following conclusions can be drawn from the results of NMR study of the catalytic system:

(1) (+)-Phosphine **IV** as ligand does not replace diamine ligands from complex **VI**, whereas the substrate is capable of replacing one of the two ligands with formation of olefin–diamine Rh(I) complex **X**;

(2) In the reaction of diamine **III** with bis-neomenthyldiphenylphosphine–cyclooctadiene trifluorosulfonate complex **V**, the latter is partially converted into diamine–diene rhodium complex **VII** which is transformed into complex **VI** by hydrogenation of the diene ligand under hydrogen;

Scheme 1.



(3) Complex **V** under hydrogen also loses the diene ligand to afford complexes **VIII** and **IX** with chiral phosphine (+)-**IV**.

On the basis of the above results, Scheme 1 illustrates the transformations of rhodium complexes (trifluorosulfonate ion is omitted for clarity). Thus, at least three complexes having chiral ligands in different combinations are formed in the catalytic system. Most probably, complex **VIII** containing sterically hindered phosphine ligands undergoes isomerization into *trans*-bis-phosphine Rh(I) complex. The reaction with the latter involves initial coordination of molecular hydrogen rather than of substrate, so that it follows a hydride mechanism [14]. Complex **VI** having bidentate amine ligands reacts with itaconic acid to give a fairly stable diamine–olefin rhodium(I) complex **X** which slowly reacts with hydrogen, affording the hydrogenation product [1].

It is known that the hydrogenation process involves octahedral dihydride olefin rhodium complex [15] which implies the presence of six coordination sites for the diamine, two hydrogen atoms, and acid **II** or **I** (the latter acts as a bidentate ligand). As a result, there are no free coordination sites for an additional ligand (in our case, phosphine **IV**). Nevertheless, the data given in Table 2 suggest that phosphine-containing complexes do participate in the catalytic process. This is seen most clearly in the hydrogenation of acid **II**, where addition of 2 equiv of phosphine sharply reduces the hydrogenating activity of the catalyst and increases the optical yield (cf. run nos. 1, 7, and 8; Table 2). Probably, the coordination of chiral phosphine (+)-**IV** is accompanied by change of the coordination mode of the diamine ligand (L^2) to the metal from bidentate to monodentate at the stage responsible for stereodifferentiation.

Taking into account that the formation of complex **IX** having two kinds of chiral ligands was detected by spectral methods just in the catalytic system (complex **V** + diamine **III**), we performed hydrogenation of itaconic acid in this system. Table 4 compares the results of hydrogenation over complex **V** with those obtained in the presence of 1 equiv of diamine **III**. It is seen that the optical yield of (*R*)-(+)-methylsuccinic acid also increases when the order of catalyst generation is the reverse, i.e., matching effect of chiral ligands is observed.

We can conclude that the hydrogenation of itaconic acid (**II**) both over the phosphine rhodium complex $[Rh(COD)(L^1)_2]^+$ TfO^- in the presence of diamine **III** and over the diamine complex $[Rh(L^2)_2]^+$ TfO^- in the presence of phosphine (+)-**IV** is characterized by matching ligand effect leading to increased stereoselectivity of the process. This is explained by formation of rhodium complexes containing two kinds of chiral ligands in the coordination sphere, together with the phosphine and diamine complexes. The concentration ratio of these complexes is likely to depend on the ratio of the components.

EXPERIMENTAL

The NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400 (^{1}H) or 162 MHz (^{31}P) from solutions in $CDCl_3$; hexamethyldisiloxane was used as internal reference for ^{1}H; the ^{31}P chemical shifts were measured relative to H_3PO_4. The optical rotations were measured on a Polamat A polarimeter at

Table 4. Hydrogenation of itaconic acid (**II**) over complex **V** in the presence of diamine **III** (methanol–benzene, 7 : 3; c_{Rh} = 2 mM; hydrogen pressure 1 atm)

Run no.	Catalytic system	Reaction conditions		**II**-to-Rh ratio	Yield, %	Optical yield, %
		time, h	temperature, °C			
1	$[Rh(COD)_2]^+ TfO^-$ + 2(+)-**IV**	24	20	25	25	45 (*R*)
2	$[Rh(COD)_2]^+ TfO^-$ + 2(+)-**IV** + **III**	24	20	50	33	61 (*R*)
3	$[Rh(COD)_2]^+ TfO^-$ + 2(+)-**IV**	24	40	50	28	41 (*R*)
4	$[Rh(COD)_2]^+ TfO^-$ + 2(+)-**IV** + **III**	32	40	50	47	61 (*R*)

λ 546 nm; the specific rotations were recalculated to $[\alpha]_D$ using a coefficient of 1.17543. The solvents were thoroughly purified and degassed. All syntheses were performed under argon in a system ensuring supply of a purified gas and evacuation. Phosphine (+)-**IV** [10] and (*Z*)-(α)-(acetylamino)cinnamic acid [16] were synthesized by known methods. The procedure for preparation of complex **VI** was described previously [1].

(+)-Neomenthyldiphenylphosphine (IV). mp 98–99°C; $[\alpha]_D^{20}$ = 91.8° (*c* = 0.89, CH_2Cl_2); relative optical purity 97% [10].

Hydrogenation of α-(acetylamino)cinnamic acid (I) and itaconic acid (II). *a*. The hydrogenation was carried out under vigorous shaking in a glass reactor maintained at a constant temperature. The reactor was connected with a pressure gauge and hydrogen supply system. Hydrogen was purified and dried by standard procedures. The reactor was charged under a stream of hydrogen with 5 ml of methanol and about 1 mmol of substrate **I** or **II**; hydrogen pressure was raised to 1.2–1.4 atm, a solution of appropriate catalyst (c_{Rh} = 2 mM) was added through a syringe (the catalyst was prepared *in situ* in a separate vessel under argon, using 5 ml of a 2 : 3 methanol–benzene mixture as solvent), and shaking was activated.

b. Complex **VI**, 0.02 mmol, was dissolved in a mixture of 3 ml of benzene and 7 ml of MeOH or 1–2 equiv of phosphine (+)-**IV** was added to 0.02 mmol of $[(COD)_2Rh]^+ CF_3SO_3^-$ in a vessel purged with dry argon. The mixture was stirred for 10 min, 0.13–0.22 g of the substrate was added, and the mixture was transferred into a high-pressure reactor which was preliminarily evacuated. Hydrogen was supplied to the reactor under pressure, and the reactor was placed in a shaker. The mixture was then treated as described in [1].

The chemical yield of the hydrogenation product was determined from the intensity ratio of the acetyl proton signals in the ^1H NMR spectra of initial acid **I** (δ 2.08 ppm) and *N*-acetylphenylalanine (δ 1.88 ppm) or $=CH_2$ signals of itaconic acid (**II**) (δ 6.27 and 5.78 ppm) and CH_3 signal of α-methylsuccinic acid (δ 1.19 ppm, d). The optical yields were determined relative to enantiomerically pure samples of (*S*)-(+)-*N*-acetylphenylalanine, $[\alpha]_D$ = +46.5° (*c* = 1.0, EtOH) [17], and (*R*)-(+)-2-methylsuccinic acid, $[\alpha]_D$ = +17.09° (*c* = 4.41, EtOH) [18].

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