Solvent free telomerization of butadiene with water into octadienols in the presence of nonionic surfactant: efficient micellar catalysis

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Telomerization of butadiene with water into 2,7-octadien-1-ol using a palladium–hydrosoluble phosphine system was investigated. The reaction was carried out without solvent in the presence of carbon dioxide and a nonionic surfactant. Promoted effect of neutral surfactant appeared above the critical micelle concentration and the conversion and the selectivity depended on the structure of nonionic surfactant hydrophilic part. The role of the nonionic surfactant is discussed.

Keywords: telomerization; octadienol; butadiene; nonionic surfactant, solubilization, micelle

1. Introduction

The telomerization of butadiene with water into 2,7-octadien-1-ol is a very attractive reaction. Indeed, 2,7-octadien-1-ol could be a key intermediate in the manufacture of the most important plasticizer raw material. So, this one-step synthesis of 2,7-octadien-1-ol from economical butadiene and water has been extensively studied by numerous industrial research groups [1,2] and Kuraray Company in Japan has even developed the reaction on an industral scale (5000 T/year). In contrast with telomerization with alcohols, phenols and amines [3,4], the efficient telomerization of butadiene requires the presence of carbon dioxide and a solvent

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such as sulfolane [1,2]. However, if the reaction is carried out in the presence of additives like Na_2CO_3, K_2CO_3 or $KHCO_3$, 2,7-octadien-1-ol can be obtained in good yields without solvent and carbon dioxide [5].

Recently, we have reported that this telomerization can also be carried out without solvent but in presence of cationic surfactant such as dodecyltrimethylammonium hydroxide or a precursor of this surfactant [6]. The role of this kind of surfactant was (i) to increase mass transfer between organic and aqueous layers, (ii) to increase the concentration in carbonate in the medium and (iii) to prevent decomposition of the catalyst [6].

Following these reactions conducted with cationic surfactants, we have shown an interest in the use of anionic and neutral surfactants.

The classical anionic surfactant (dodecylsulfate sodium, dodecylbenzenesulfonic acid sodium salt and oleic acid sodium salt) exhibited very low activities, and very poor chemoselectivities into the desired 2,7-octadien-1-ol. The inhibited effect of these surfactants has been interpreted from electrostatic repulsions between the surfactant and anionic species at the micelle–water interface [7].

In contrast with anionic surfactants, the neutral surfactants allowed to avoid, as in the case of cationic surfactants, the above electrostatic repulsions [7] and gave rise to 2,7-octadien-1-ol with high conversions and selectivities in the absence of solvent [8]. The catalytic system consists of a palladium salt and a hydrosoluble phosphine such as $Ph_2P(m\text{-}C_6H_4SO_3Na)$ (TPPMS) (scheme 1).

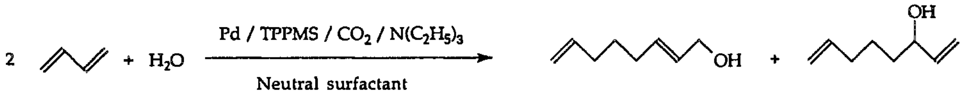
As advocated in the literature [1,2], the reaction was carried out in the presence of carbon dioxide (10 bar). Although some hypotheses have been suggested, the role of carbon dioxide or additives has not been totally elucidated yet [9]. The most likely hypothesis involves (i) the formation of carbonates from water and carbon dioxide and (ii) the reaction of these carbonates on a Pd complex. The latter reaction gives rise to a mono acid octadienylcarbonate which is unstable in the medium and which decomposes into octadienols and carbon dioxide. Scheme 2 presents the different steps invoked above.

The main by-products of the telomerization were butadiene dimers and dioctadienyl ethers (scheme 3). Minor by-products such as lactones, trimers and tetramers of butadiene have also been observed.

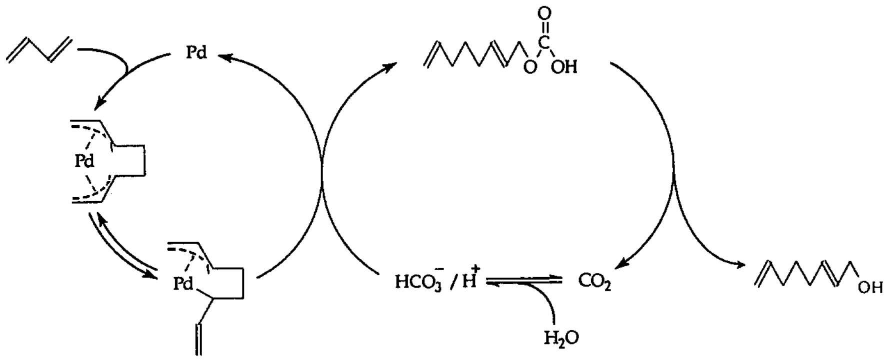
2. Experimental

2.1. MATERIALS

Nonionic surfactants were purchased from Aldrich and Tokyo Kasei Organic



Scheme 1. Telomerization of butadiene with water in the presence of neutral surfactant.

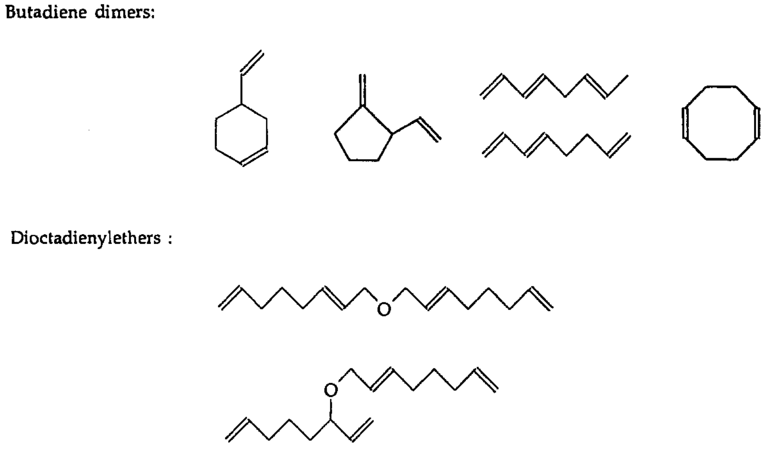


Scheme 2. Catalytic cycle proposed for the telomerization of butadiene into octadienols in the presence of carbon dioxide.

Chemicals and were used as received without further purification. TPPMS was synthesized according to published procedures [10]. Distilled deionized water and palladium acetate obtained from Aldrich (98% pure) were used in all the experiments. Carbon dioxide (>99.9% pure – Air Liquide) and 1,3-butadiene (>99.9% pure – Union Carbide) were used directly from cylinders.

2.2. APPARATUS

All experiments were performed under an nitrogen atmosphere using standard Schlenk techniques. All the telomerization experiments were carried out in a 100 ml stainless steel autoclave reactor. The autoclave supplied by Autoclave Engineer was equipped with arrangements for automatic temperature control and variable stirrer speeds. A safety rupture disk was also fitted to the reactor. The optical



Scheme 3. Main by-products of telomerization of butadiene with water.

birefringence of the reaction crude has been examinated with a polarizing microscope (Leitz, magnification 320X). Mass spectra of the organic compounds were obtained on a MS-50 RF Kratos apparatus using the fast atom bombardment technique (FAB, Ar, 8 keV); data were collected on a MCA (multichannel analysis) station with a resolution of 1500.

2.3. EXPERIMENTAL PROCEDURE

In a typical catalytic reaction, $Pd(OAc)_2$ (3.25×10^{-4} mol), TPPMS (10^{-3} mol), triethylamine (5×10^{-3} mol), nonionic surfactant (5×10^{-3} mol), 1,3-butadiene (0.166 mol) and H_2O (0.33 mol) were introduced, under a nitrogen atmosphere, into the autoclave. The autoclave was pressurized to 10 bar with carbon dioxide and heated at 85°C during 1h45 (stirring speed: 800 rpm). The autoclave was then cooled and degassed. The reaction crude was analyzed by gas–liquid chromatography (packed glass column: 5% XE 60 + 10% OV 210; Chromosorb WHP 80/100; 10 m × 2 mm), using *n*-octanol as internal standard. Products were identified by comparison of GLC retention times and spectral characteristics with authentic samples.

The telomerization products of butadiene with ethers or esters of glucosides or sorbitans were analyzed as follows. The crude reaction mixture was evaporated under vacuum. 50 mg of this solid product were acetylated with a mixture of acetic anhydride (0.1 mol) and pyridine (0.03 mol). The solution was stirred overnight at room temperature. After evaporation of the unreacted compounds, the peracetylated telomerization products were dissolved in 0.5 ml of ethanol and separated by gas chromatography on a high temperature SGE HT5 0.1 column (length: 25 m, internal diameter: 0.22 mm). Products were identified by comparison of GLC retention times with authentic samples. These telomerization products were also characterized by Positive FAB (matrix: thioglycerol/sodium acetate). For example, FAB spectra of the peracetylated telomerization products obtained with the dodecyl-β-glucopyranoside surfactant displayed mainly two signals (*m/z*; relative intensity): 605 (peracetylated dodecyloctadienyl-β-glucopyranoside + Na; 30%) and 713 (peracetylated dodecyloctadienyl-β-glucopyranoside + thioglycerol + Na; 20%).

3. Results

The most significant results obtained with various nonionic surfactants are collected in tables 1 and 2. Nonionic surfactants, also called neutral surfactant, are amphiphilic molecules containing both a hydrophobic part (an alkyl chain) and a hydrophilic part bearing no electric charge. The common hydrophilic groups are polyoxyethylene chains or polyol groups (sucrose, sorbitan) [12].

Table 1 summarizes some data for the telomerization in the presence of various

Table 1
Telomerization of butadiene with water in the presence of various surfactants[a]

Entry	Surfactant	HLB[b]	Butadiene conversion (mol%)	Yield of octadienols (mol%)	Selectivity[b] octadienols (mol%)	Selectivity (1-ol/ols)[d] (mol%)	Yield[e] of dimers (mol%)	Yield[f] of ethers (mol%)
1	(–)	(-)	33	20	60	85	10	3
2[g]	$CH_3(OCH_2CH_2)_7OH$	(-)	25	16	64	88	8	1
3[g]	$CH_3(OCH_2CH_2)_{18}OH$	(-)	34	24	71	92	9	1
4[g]	$CH_3(OCH_2CH_2)_8OCH_3$	(-)	16	8	50	88	8	0
5	$C_{12}H_{25}(OCH_2CH_2)_4OH$	10.6	48	29	60	90	9	10
6	$C_{12}H_{25}(OCH_2CH_2)_{23}OH$	17.1	67	54	80	92	8	5
7	$C_{18}H_{37}(OCH_2CH_2)_nOH$ $n = 2$	5.8	42	25	60	90	10	7
8	$n = 10$	12.8	59	39	66	90	13	7
9	$n = 20$	15.6	69	49	71	91	12	10
10	$n = 50$	17.9	72	54	77	91	11	7
11	$C_{17}H_{35}CO(OCH_2CH_2)_nOH$ $n = 2$	7.6	29	20	69	90	9	–
12	$n = 10$	13.5	48	25	52	92	18	5
13	$n = 25$	16.5	60	43	72	91	12	5
14	$n = 40$	17.6	76	56	74	91	14	6
15	$n = 55$	18.2	77	60	78	92	13	4

[a] Reaction conditions: $Pd(OAc)_2 = 0.073$ g (3.25×10^{-4} mol); $N(C_2H_5)_3$ = surfactant = 5×10^{-3} mol; TPPMS = 1×10^{-3} mol; $C_4H_6 = 9$ g (0.166 mol); $H_2O = 6$ g (0.33 mol); $P_{CO_2} = 10$ bar; $T = 85°C$; t = 1h45.

[b] Hydrophile–lipophile balance calculated from the general formula: $20\, M_H/(M_H + M_L)$. M_H and M_L are the molecular weights of hydrophilic and lipophilic parts of the surfactant [12,13].

[c] (moles of octadienols ×2/moles of converted butadiene) × 100.

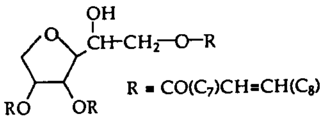
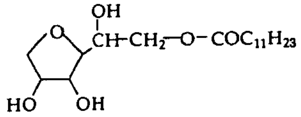
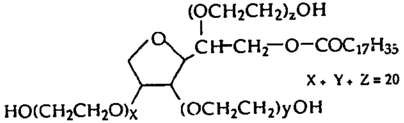
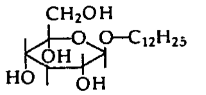
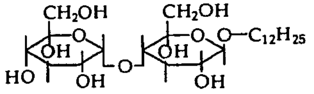
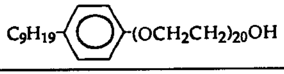
[d] (moles of 1-octadienol/moles of 1- and 3-octadienol) × 100.

[e] The main dimers were 1,3,6-, 1,3,7- and 2,4,6-octatrienes and 4-vinylcyclohexene.

[f] Dioctadienyl ethers.

[g] Comparative examples. These compounds do not present surfactant properties.

Table 2
Telomerization of butadiene with water in the presence of various surfactants [a]

Entry	Surfactant	HLB [b]	Butadiene conversion (mol%)	Yield of octadienols (mol%)	Selectivity [b] octadienols (mol%)	Selectivity (1-ol/ols) [d] (mol%)	Yield [e] of dimers (mol%)	Yield [f] of ethers (mol%)
1	 R = CO(C$_7$)CH=CH(C$_8$)	1.8	38	24	63	92	10	4
2		8.6	55	37	67	92	8	9
3	 X + Y + Z = 20	15.6	35	27	77	93	5	3
4		10.3 [g]	66	42	56	88	10	13
5		13.4 [g]	52	36	70	92	8	7
6		16	58	38	89	65	11	9

[a] Reaction conditions: see table 1.
[b] Experimental hydrophile–lipophile balance.
[c] (moles of octadienols ×2/moles of converted butadiene) ×100.
[d] (moles of 1-octadienol/moles of 1- and 3-octadienol) ×100.
[e] The main dimers were 1,3,6-, 1,3,7- and 2,4,6-octatrienes and 4-vinylcyclohexene.
[f] Dioctadienyl ethers.
[g] Calculated hydrophile–lipophile balance (see table 1).

polyoxyethylenated long-straight chain alcohols (POEA) and polyoxyethylenated long-straight chain carboxylic acid esters (POEC). Entries 1–4 compared with entries 5–15 indicate clearly that the presence of a neutral surfactant is essential to produce 2,7-octadien-1-ol in good yields. Indeed, telomerization without surfactant afforded only octadienols in poor yield (entry 1) and poly(ethylene glycol) methyl ether and poly(ethylene glycol) dimethyl ethers did not promote the telomerization (entries 2–4). These latter molecules possess the structure of the neutral surfactant hydrophilic part but do not exhibit amphiphilic characteristics.

In contrast, POEA and POEC containing 20 to 30 units of oxyethylene can be recommended as suitable surfactants. Indeed, conversions, selectivities and turnover frequency (moles of converted butadiene/moles of palladium h) reached 70%, 80% and 230 h^{-1}, respectively (entries 6, 9, 13 in table 1). The effects of such surfactants start to occur at every low concentration (ca. 10^{-4} mol/ℓ, see figs. 1 and 2). Although the conversions remained almost identical when the surfactant concentration increased, high concentrations allowed to obtain octadienols with higher selectivities (see figs. 1 and 2). It is worth mentioning that, with these compounds, the conversion and the yield increase no more when the oxyethylene unit number is higher than 30 (compare entries 9, 10 and 14, 15 in table 1).

Other neutral surfactants such as polyoxyethylenated alkylphenols, ethers or esters of glucosides or sorbitans can also be used but conversions and selectivities were slightly lower (table 2). Furthermore, a side reaction has been observed when ethers or esters of glucosides or sorbitans have been used like surfactants. Indeed,

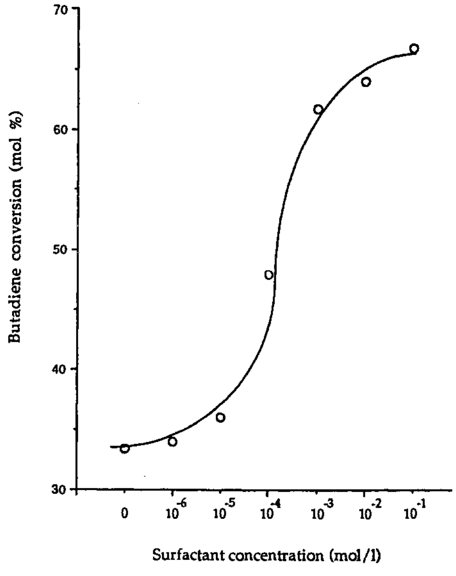


Fig. 1. Effect of $C_{12}H_{25}(OCH_2CH_2)_{23}OH$ concentration on butadiene conversion. $Pd(OAc)_2$: 0.325 mmol; TPPMS: 1 mmol; $N(C_2H_5)_3$: 5 mmol; C_2H_6: 0.166 mol; H_2O: 0.33 mol; P_{CO_2}: 10 bar; T: 85°C; 1h45.

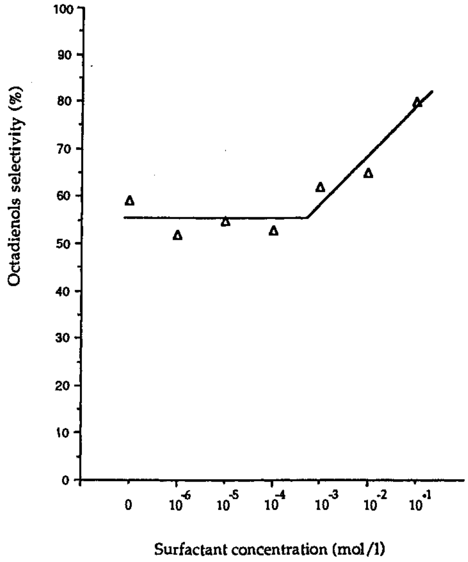


Fig. 2. Effect of $C_{12}H_{25}(OCH_2CH_2)_{23}OH$ concentration on selectivity to octadienols. $Pd(OAc)_2$: 0.325 mmol; TPPMS: 1 mmol; $N(C_2H_5)_3$: 5 mmol; C_2H_6: 0.166 mol; H_2O: 0.33 mol; P_{CO_2}: 10 bar; T: 85°C; 1h45.

analyses of reaction mixtures have shown that the alcohol functions of the hydrophilic part of these surfactants reacted with butadiene and led to a mixture of octadienyl ethers of the corresponding surfactants. For example, during the telomerization of butadiene into octadienols, the surfactant dodecyl-β-glocupyranoside was converted into dodecyloctadienyl-β-glucopyranoside with ca. 30% yield. Traces of dodecyldioctadienyl-β-glucopyranoside have been observed. Such a result is in agreement with our recent patent which described the telomerization of butadiene with sugar derivatives in a two-phase system [11]. It must be noticed that this side reaction was not observed with the POEA and POEC surfactants.

4. Discussion

4.1. INFLUENCE OF HLB NUMBER OF THE SURFACTANT ON THE BUTADIENE CONVERSION

In tables 1 and 2, the hydrophile–lipophile balance (HLB) number of each surfactant was indicated. Each surfactant can be characterized by a HLB number comprised between 0 and 20 [12,13]. At the higher values of the scale (8–18) lie hydrophilic surfactants which possess high water solubility and generally act as good aqueous solubilizing agents and stabilizers for oil/water emulsions. Lower HLB values (3–6) concern surfactants with low water solubility which act as solubilizers of water in oil [12,13].

Except entry 3 in table 2, neutral surfactants with high HLB lead to high conversions. Furthermore, in each surfactant class (POEA, POEC, ethers or esters of glucosides or sorbitans), the tables 1 and 2 indicate also that the higher the HLB, the higher the octadienols selectivity. In other words, the surfactant efficiency for the telomerization seems to be correlated to its high ability to solubilize butadiene in water. This suggests that the hydrodimerization is promoted by increasing of the interfacial area between the organic substrate phase and the aqueous phase.

This solubilization can proceed theoretically via the formation of liquid–crystalline phases, association colloids or microemulsions [12,13].

As liquid–crystalline phases (also called mesophases) are typically formed at high surfactant concentration, the reaction crudes with a high surfactant concentration (30–40 wt%; entries 6, 9, 14 and 15 in table 1) have been analyzed by polarizing microscopy. Under orthoscopic observation, the samples observed between crossed polarizers were dark, whatever the orientation of the revolving sample support. As a result, the optical birefringence of these solutions is lower than 0.005. Furthermore, no usual uniaxial black cross has been observed under conoscopic observation. These measurements indicate clearly that lamellar or hexagonal liquid–crystalline phases are not formed in our experimental conditions [14]. As a result, the solubilization does not proceed hereby via liquid–crystalline phases.

Association colloids arise from combination of surfactants. These surfactants associate in a dynamic and thermodynamically driven process leading to a system that may be simultaneously a molecular solution and a true colloidal system [13]. In our experimental conditions, we think that these aggregates of colloidal dimensions are principally spherical, cylindral or swollen micelles. Figs. 1 and 2 show that promoted effects of surfactant $C_{12}H_{25}(OCH_2CH_2)_{23}OH$ on conversion and selectivity occur at a concentration of 10^{-4} mol/ℓ. As this concentration coincides with the critical micelle concentration of this surfactant [12], it is likely that micellar aggregates are formed. Such a behaviour has recently been reported by Oehme et al. during rhodium catalyzed asymmetric hydrogenation in the presence of micelle-forming nonionic surfactants [15]. The increase in selectivity with the surfactant concentration (fig. 2) could be due to preferential orientation of the palladium complex in the more and more extended micellar aggregates. A similar hypothesis has already been reported by Quinn et al. for rhodium catalyzed hydrogenation in liposome systems [16]. The formation of larger and more extended aggregates such as vesicles or bilayers cannot probably be invoked hereby. Indeed, it is known that the latter aggregates are preferentially formed from phosphatidylcholines, quaternary ammonium salts having two long alkyl chains or dialkyl compounds with anionic head groups [12,17]. It is worth noticing that the solubilization by the micellar aggregate must be improved by the octadienol produced during the telomerization. Indeed, it is known that long-chain alcohols increase the solubility of nonpolar molecules in surfactant solutions by insertion between adjacent surfactant molecules in the association colloids [12,13].

It must also be pointed out that a solubilization by the temporary formation of a microemulsion during the reaction cannot be excluded. A microemulsion is composed of two mutually immiscible liquid phases, one spontaneously dispersed in the other with the assistance of one surfactant and a cosurfactant [12,13]. Cosurfactant is a polar compound of intermediate chain length. Therefore, octadienol which is produced during the telomerization could be considered as a cosurfactant and, then, able to stabilize a microemulsion.

4.2. INFLUENCE OF THE SHAPE OF THE HYDROPHILIC PART OF THE SURFACTANT ON THE BUTADIENE CONVERSION

Tables 1 and 2 also indicate that the HLB number is not the only factor which governs the conversion and the yield. Indeed, a notable variation of conversion was observed with various hydrophilic groups under similar reaction conditions (constant HLB number and identical lipophilic group – compare entry 13 (table 1) and entry 3 (table 2)). Moreover, a modification of the hydrophilic part of surfactant can induce a decrease in the conversion in spite of a HLB number increase (compare entries 2 and 3 in table 2). These observations clearly indicate that the conversion depends also strongly on the size and the geometric shape of the hydrophilic part of the neutral surfactant.

The reasons for this phenomenon are not yet clear. However, although no conclusive evidence has been obtained, we think that these results could be explained by geometric and packing constraints of the palisade layer of association colloids. the so-called palisade layer is the region comprised between the hydrophilic group and the first few carbon atoms of the hydrophobic groups of the neutral surfactant [12]. With the nonionic surfactant, this region is a relatively large fraction of the micelle volume and its size and geometric shape are due to the hydrophilic part of the surfactant [12,13]. It is known that TPPMS phosphine has an amphiphilic character owing to sulfonated and nonsulfonated phenyl groups and, so, is preferentially localized in this palisade layer [16]. So, on the above bases, we suggest that a geometric shape modification of the palisade layer by the use of various neutral surfactants can affect the chemical environment of the palladium–TPPMS catalytic system and leads to a less efficient catalytic system.

5. Conclusion

We have demonstrated that telomerization of butadiene into 2,7-octadien-1-ol can be achieved in high yield and conversion in the presence of neutral surfactant in an associated colloidal system. However, a high HLB number and a suitable shape of the hydrophilic part of the surfactant are required. A more detailed investigation with the aim to determine the nature of association colloids is now under way. The few examples of transition-metal-complex catalysis in micellar solution described in the literature [15,16,18] and this work support, so far, the idea that metal-complex catalysis in the presence of amphiphilic molecules is a phenomenon of general importance.

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