solvent in vacuo the crude product was purified by flash chromatography (hexanes/ethyl acetate).

8: M.p. 78–79 °C (hexanes/diethyl ether); $[\alpha]^{25}_{D} = +3.5$ ($c = 2.6$, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$): $\delta = 1.21$ (s, 9H), 1.66 (d, $J = 4.7$ Hz, 3H), 3.59 (m, 1H), 4.74 (dd, $J = 8.6$, 8.6 Hz, 1H), 5.60–5.69 (m, 2H), 6.88 (d, $J = 8.6$ Hz, 1H), 7.19–7.34 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 17.54$, 27.36, 52.86, 56.75, 82.84, 115.52, 126.05, 127.18, 127.49, 128.20, 129.49, 138.83, 156.37, 168.80; elemental analysis calcd for $C_{18}H_{22}F_3NO_3$: C 60.50, H 6.20, N 3.92; found: C 60.42, H 6.44, N 3.99.

12: Colorless oil; $[\alpha]^{25}_{D} = +21.7$ ($c = 1.0$, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$): $\delta = 1.03$ (d, $J = 6.9$ Hz, 3H), 1.45 (s, 9H), 1.65 (d, $J = 6.3$ Hz, 3H), 2.75 (m, 1H), 4.42 (dd, $J = 8.7$, 4.7 Hz, 1H), 5.24 (dd, $J = 15.3$, 7.7 Hz, 1H), 5.52 (dqd, $J = 15.3$, 6.3, 1.0 Hz, 1H), 6.65 (brs, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 16.36$, 17.63, 27.75, 39.37, 56.96, 82.83, 115.56 (q, $J = 288$ Hz), 128.03, 129.30, 156.74 (q, $J = 37.2$ Hz), 168.83; elemental analysis calcd for $C_{13}H_{20}F_3NO_3$: C 52.88, H 6.83, N 4.74; found: C 52.85, H 6.66, N 4.71.

Received: June 28, 1999
Revised: August 25, 1999 [Z13635]

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[15] Unfortunately the chiral diphenyl derivative **9** could not be obtained by this protocol.

A Stable, Conspicuously Active, Water-Soluble Pd Catalyst for the Alternating Copolymerization of Ethene and CO in Water**

Göran Verspui, Frank Schanssema, and
Roger A. Sheldon*

Alternating copolymers of olefins and CO are relatively new, low-cost materials with interesting physical properties.[1] The discovery of active cationic palladium catalysts containing flexible *cis*-chelating diphosphanes has made the preparation of these copolymers commercially feasible.[2] Especially catalyst systems prepared in situ from a Pd precursor, a sterically demanding diphosphane such as 1,3-bis(di(2-methoxyphenyl)phosphino)propane (D-*o*-APPr), and an excess of a suitable Brønsted acid exhibit excellent catalyst performance in the ethene/CO copolymerization in organic solvents such as methanol.[3]

The current, increasing interest in sustainable technologies has intensified the research activity in organometallic catalysis in aqueous media over the past few years. Water is an environmentally friendly, economically attractive, and safe reaction medium for a variety of reactions catalyzed by homogeneous transition metal catalysts.[4]

We and others previously reported that the alternating copolymerization of ethene and CO can be performed efficiently in water by employing Pd complexes of water-soluble diphosphanes.[5] At 90 °C and 60 bar the aqueous Pd/DPPPr-S/HOTs (DPPPr-S = 1,3-$C_3H_6(P(C_6H_4\text{-}m\text{-}SO_3Na)_2)_2$, HOTs = *p*-toluenesulfonic acid) system exhibits catalyst ac-

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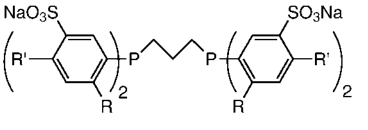
[**] Catalytic Conversions in Water, Part 16. Financial support by the Dutch Foundation for Chemical Research, NWO-CW, is gratefully acknowledged. Part 15: see ref. [4d].

Supporting information for this article is available on the WWW under http://www.wiley-vch.de/home/angewandte/ or from the author.

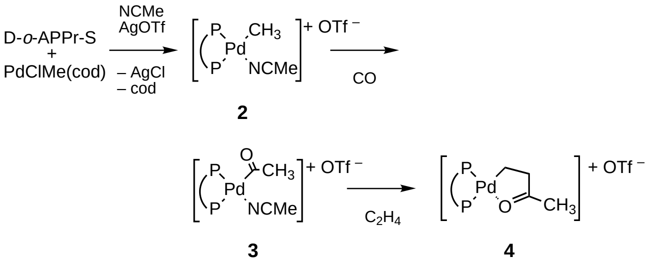
tivities up to 7.7 kg copolymer per g Pd per hour.[6] Despite the excellent catalyst performance, the catalyst precursor is rather unstable and decomposition of the catalyst during the copolymerization is frequently observed.

To develop a more stable catalyst system, we turned to the synthesis of a water-soluble analogue of D-*o*-APPr. Initial attempts to introduce sodium sulfonate groups to the aryl rings under conventional sulfonation conditions (30 % oleum) were unsuccessful due to the decomposition of the ligand. We developed a much milder sulfonation procedure which afforded the tetra-sulfonated diphosphane 1,3-$C_3H_6[P(C_6H_3\text{-}2\text{-}OMe\text{-}5\text{-}SO_3Na)_2]_2$ (D-*o*-APPr-S) **1** in high purity and moderate yield (see supporting information).



DPPPr-S: R = H, R' = H
1 D-*o*-APPr-S: R = OCH_3, R' = H
5 D-*p*-APPr-S: R = H, R' = OCH_3
6 D-*o*-TPPr-S: R = CH_3, R' = H

Stirring **1** with $[PdCl(CH_3)(cod)]$ (cod = cyclooctadiene) in methanol and abstraction of the chloro ligand with AgOTf and NCMe afforded $[Pd(CH_3)$(D-*o*-APPr-S)(NCMe)]OTf (**2**), which was isolated as an air-stable white solid. At lower temperatures, this compound reacts readily with CO and ethene to afford the intermediates **3** and **4** (Scheme 1); the ^1H and ^{31}P NMR spectra are consistent with those of related complexes in organic media.[7] In contrast with **2**, **3** is unstable in solution and decomposes to Pd-black. Compound **4** is more stable in aqueous solution but at room temperature it undergoes a slow hydrolysis reaction to afford 2-butanone. NMR experiments with ^{13}CO revealed that the oxygen atom of the carbonyl group in **4** is coordinated to the palladium center, resulting in a five-membered chelate ring.



Scheme 1. The synthesis of **2**–**4**.

Compound **2** was tested in the aqueous alternating copolymerization of ethene and CO, and indeed, the reaction proceeded smoothly in neat water. At 90 °C and 60 bar of an ethene/CO mixture (1:1) an activity of 32.2 kg copolymer per gram Pd per hour was reached (Table 1)—four times faster than the previously reported Pd/DPPPr-S/HOTs system. This corresponds to an average turnover frequency (TOF = mol ethene per mol Pd per hour) of $>61\,000\ h^{-1}$, the highest activity reported for a water-soluble Pd catalyst so far. When the catalyst was prepared in situ by adding an aqueous solution of **1** to $[Pd(OTs)_2(NCMe)_2]$ somewhat lower catalyst activities (25.0 $kg\,g\,Pd^{-1}\,h^{-1}$) were observed.

Table 1. Alternating copolymerization of ethene and CO in water catalyzed by Pd/D-*o*-APPr-S.

Run[a]	Catalyst	Additive [mmol]	Activity	M_n $[kg\,g\,Pd^{-1}\,h^{-1}]$	M_n/M_w $[kg\,mol^{-1}]$
1[b]	**2**	–	32.2	69.2	1.9
2	Pd/D-*o*-APPr-S	–	24.4	64.4	2.1
3	Pd/D-*o*-APPr-S	HOTs (1.0)	18.6	59.7	2.0
4	Pd/D-*o*-APPr-S	NaOH (3.2)	0.0	–	–
5[c]	Pd/D-*o*-APPr-S	D-*o*-APPr-S	24.4	n.d.[d]	n.d.[d]

[a] Reaction conditions: $[Pd] = 17.6 \times 10^{-6}\,mol\,L^{-1}$, 142 mL water, D-*o*-APPr-S/Pd = 1.0, 90 °C, 60 bar ethene/CO (1/1), 1000 rpm, reaction time: 60 min. [b] 0.00125 mmol of **2**; $[Pd] = 8.8 \times 10^{-6}\,mol\,l^{-1}$. [c] D-*o*-APPr-S/Pd = 2.0, reaction time: 65 min. [d] n.d. = not determined.

In contrast with Pd/DPPPr-S, the Pd/D-*o*-APPr-S catalyst is air stable in solution; only a minor decrease in catalyst activity was observed (from 16.0 to 15.6 $kg\,g\,Pd^{-1}\,h^{-1}$, at 70 °C and 40 bar) when an aqueous solution of **2** was stirred in air for 16 h. The addition of a Brønsted acid cocatalyst is not required for a higher catalyst stability and activity. In fact, the addition of some HOTs led to a lower copolymer production. When a small amount of NaOH was added no reaction was observed, analogous to our previously studied Pd/DPPPr-S/HOTs system. We suggest that in the case of in situ prepared catalysts, the copolymerization starts with the formation of a palladium–hydrido complex,[8] which is presumably not formed in basic media.

Quantitative ^{13}C NMR spectroscopy confirmed the perfectly alternating ethene/CO distribution in the copolymer, and the absence of end group signals indicated a very high molecular weight. Size-exclusion chromatography confirmed the high average molecular weight; $M_n = 124.9\ kg\,mol^{-1}$ for the copolymer sample obtained with Pd/D-*o*-APPr-S/HOTs at 70 °C and 40 bar, a weight six times higher than that of the copolymer obtained with Pd/DPPPr-S/HOTs under the same reaction conditions (Table 2). Even higher molecular weights were obtained when the Brønsted acid cocatalyst was omitted.

We also synthesized the *para*-methoxy isomer of **1**, D-*p*-APPr-S (**5**), and D-*o*-TPPr-S (**6**) containing *ortho*-CH_3 groups and tested them in the aqueous alternating copolymerization. Compared to the Pd/DPPPr-S/HOTs system the catalysts prepared in situ from **5** or **6** are considerably less active. The sharp contrast between ligands **1** and **5** suggests that the rate-

Table 2. The alternating copolymerization of ethene and CO with various water-soluble catalysts.

Run[a]	Catalyst	Additive [mmol]	Activity	M_n $[kg\,g\,Pd^{-1}\,h^{-1}]$	M_n/M_w $[kg\,mol^{-1}]$
1	Pd/DPPPr-S	HOTs (1.0)	2.61	21.8	2.0
2[b]	Pd/D-*o*-APPr-S	HOTs (1.0)	8.22	124.9	1.9
3	Pd/D-*p*-APPr-S	HOTs (1.0)	1.67	55.9	2.0
4	Pd/D-*o*-TPPr-S	HOTs (1.0)	1.71	3.9	1.4
5[c]	Pd/T-*o*-APTS	HOTs (1.0)	0.00	–	–

[a] Reaction conditions: $[Pd] = 70.4 \times 10^{-6}\,mol\,L^{-1}$, 142 mL water, ligand/Pd = 1.0, 70 °C, 40 bar ethene/CO (1/1), 1000 rpm, reaction time: 120 min. [b] $[Pd] = 14.1 \times 10^{-6}\,mol\,l^{-1}$, reaction time: 60 min. [c] Catalyst decomposition, T-*o*-APTS = $P(C_6H_3\text{-}2\text{-}OCH_3\text{-}5\text{-}SO_3Na)_3$.

enhancing effect of the *ortho*-methoxy substituents is steric in nature. This is confirmed by the fact that no decrease in catalyst activity was observed when an excess of **1** was used, in contrast with the Pd/DPPPr-S/HOTs system, where the reaction is almost completely inhibited when two equivalents of ligand are added.[5b] Apparently, catalytically inactive $[Pd^{II}(D\text{-}o\text{-}APPr\text{-}S)_2]$ complexes cannot be formed with **1**.

The presence of the *ortho*-methyl groups in **6** did not induce a similar rate-enhancing effect, which suggests that the effect of the *ortho*-methoxy substituents in **1** is not only steric. The oxygen atoms might coordinate to the palladium atom at some stages during the reaction to stabilize energetically less favorable configurations.[9] Nevertheless, a permanent coordinative bond with the oxygen atom was not observed in the NMR spectra of **2**–**4**. When the monodentate $P(C_6H_3\text{-}2\text{-}OCH_3\text{-}5\text{-}SO_3Na)_3$ was used as ligand, catalyst decomposition took place, showing that the copolymerization does not proceed in the presence of a $P \sim O$ chelate alone, but requires a $P \sim P$ ligand.

Although the alternating copolymerization of ethene and CO is initially homogeneous, in a later stage the growing polymer precipitates and the reaction might proceed heterogeneously. Indeed, when we recovered the Pd/D-*o*-APPr-S catalyst by filtering off the product, only traces of copolymer were formed in a second run. This indicates that the catalyst was still attached to the polymer chain, which is precipitated on the rest of the product. Nonetheless, by introducing a “termination procedure” (see supporting information) 61 % of the catalyst activity could be restored.[10]

The Pd/D-*o*-APPr-S catalyst was also active in a supported aqueous phase (SAP) system, in which the copolymer itself is used as a support for an aqueous catalyst solution, which is distributed as a thin film over the surface. Such a “gas-phase process” minimizes the amount of solvent needed in the process and avoids fouling of the reaction vessel. In this SAP system, under nonoptimized conditions (90 °C, 60 bar), catalyst activities of 21.3 $kg\,g\,Pd^{-1}\,h^{-1}$ were reached.

In conclusion, we have developed a simple, highly active, air-stable, and environmentally friendly catalyst for the alternating copolymerization of ethene and CO.

Received: September 2, 1999 [Z13953]

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Generation-Dependent Intramolecular CT Complexation in a Dendrimer Electron Sponge Consisting of a Viologen Skeleton

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In the last two decades efficient methodologies for the cascade synthesis of dendrimers have been developed. Nowadays the main research activities are focused on dendritic effects resulting from the topology and the interaction of built-in molecular functionalities.[1] Redox-active subunits play an important role in this respect. They may be located: a) in the core,[2, 3] b) in the periphery,[4–7] or c) in the core, the branches, and the periphery of the dendrimer.[8–11] The resulting dendritic effects concern redox protein mimetics,[3] redox gradients,[11] charge transfer (CT) complex-based conductivity,[7] electron sponges,[4] and redox sensors.[12]

We present here a new homologous series of electroactive dendrimers belonging to class (c). The dendrimers consist of a viologen skeleton that contains up to 45 electrochemically accessible 4,4′-bipyridinium subunits which cause electron sponge properties. Electrochemical measurements revealed an excellent correlation between the hydrodynamic radius and the generation number of the dendrimers. Furthermore, an electrochemically switchable and generation-dependent CT complex was observed for the first time in the cation radical oxidation state.

The synthesis of the dendrimers of generations zero to three follows the “divergent method with preformed branching units” (Scheme 1).[13] The well-known trifunctional initiator core $\mathbf{P0} \cdot 3\,PF_6^-$ consists of a mesityl derivative linked to three 4,4′-bipyridinium units.[14] The three peripheral nitrogens react quantitatively with primary benzyl or alkyl bromides or iodides. 1,3-Di-(hydroxymethyl)-benzyl bromide (**V1**)

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