

# Active metal species assembled with heteropoly tungstate anion $\text{PW}_9\text{O}_{34}^{9-}$ for liquid phase hydrocarbon oxidation

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## Abstract

Monometallic  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$ ,  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2\text{Pd}_n\text{O}_x\text{H}_y]^{q-}$  (where on the average  $n = 3$ ), bimetallic  $[\text{Pd}_2\text{Cu}(\text{PW}_9\text{O}_{34})_2]^{12-}$ ,  $[\text{Pd}_2\text{Fe}(\text{PW}_9\text{O}_{34})_2]^{11-}$ ,  $[\text{PdFe}_2(\text{PW}_9\text{O}_{34})_2]^{10-}$  and a mixture of  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2\text{Pd}_n\text{O}_x\text{H}_y]^{q-}$  ( $n_{\text{av}} \approx 10$ ) +  $[(\text{VO})_3(\text{PW}_9\text{O}_{34})_2]^{9-}$  complexes were prepared and characterized by NMR  $^{31}\text{P}$ ,  $^{183}\text{W}$ ,  $^{51}\text{V}$  and IR spectroscopy. The complexes were tested in catalysis of  $\text{O}_2 + \text{H}_2$  reaction and benzene oxidation to phenol with  $\text{O}_2/\text{H}_2$ . Effectiveness of the catalytic performance depended on the composition of the complexes. Bimetallic Pd(II)–Fe(III) complexes were several times more active in phenol production than Pd(II) monometallic systems. © 2000 Elsevier Science B.V. All rights reserved.

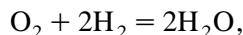
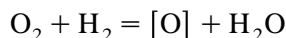
*Keywords:* Benzene oxidation;  $\text{O}_2/\text{H}_2$  mixture; Palladium(II); Heteropolycomplexes; NMR  $^{31}\text{P}$ ;  $^{183}\text{W}$ ;  $^{51}\text{V}$

## 1. Introduction

Selective oxidation of hydrocarbon substrates by dioxygen is achieved under mild conditions, when the process is participated by co-reductant, e.g., hydrogen. Appropriate catalytic systems are known for hydroxylation of aromatic compounds [1,2], oxidation of cyclohexane [3], and epoxidation of cyclohexene [4,5]. Typical catalysts consist of a noble metal modified by other transition metal ions, oxides or halide anions.

Active species of the catalysts combine different functions, which are (1) generation of a specific form of oxygen through partial reduction of dioxygen with hydrogen, (2) assistance of oxygen transfer to hydrocarbon substrate S,

and (3) catalyzing dioxygen complete reduction to water. Overall, process is determined by three reactions:



whose efficiency depends on a nature of active species of the catalyst.

We have made an attempt to find connection between the composition and properties of the system in catalysis of benzene oxidation using oxygen–hydrogen mixture. Choosing metal containing heteropoly tungstates, we have been able to vary the composition of the system.

In our previous study, catalytic systems based on Pd(II) complexes with heteropoly tungstate anion  $\text{PW}_{11}\text{O}_{39}^{7-}$  were studied in benzene oxidation [6]. Of the same type Pd(II) complexes were precursors of supported catalysts which

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operated in liquid-phase oxidation of benzene and cyclohexane [7]. In the course of this work, we have used  $\text{PW}_9\text{O}_{34}^{9-}$  anion as a matrix carrying three transition metal ions [8,9]. Pd(II) mono-, bimetallic Pd(II)–Fe(III), Pd(II)–Cu(II) and a mixture of Pd(II) and V(V) complexes with this heteropoly anion were prepared and applied to catalysis.

## 2. Experimental

Monometallic Pd(II) complexes with the  $\text{PW}_9\text{O}_{34}^{9-}$  anion were prepared by adding solid  $\text{Na}_8\text{HPW}_9\text{O}_{34} \cdot 24\text{H}_2\text{O}$  to the  $[\text{Pd}(\text{H}_2\text{O})_4]^{2+}$  solution in  $\text{H}_2\text{SO}_4$  immediately after adding of  $\text{Na}_2\text{CO}_3$  solution until pH 1.6. The mixture was stirred to dissolving the solid and neutralized to pH 3.7–4.0 with  $\text{Na}_2\text{CO}_3$  solution.

To prepare bimetallic complexes,  $[\text{Pd}(\text{H}_2\text{O})_4]^{2+}$  and  $\text{Fe}_2(\text{SO}_4)_3$  (or  $\text{CuSO}_4$ , or  $\text{NaVO}_3$ ) solutions were mixed before adding heteropoly tungstate.

In the resulting solutions the concentration of heteropoly tungstate anion was 0.02 M, the total concentration of Pd(II) plus Fe(III) (or Cu(II), or V(V)) was 0.03 to 0.06 M.

Preparation of  $[\text{Pd}(\text{PW}_{11}\text{O}_{39})]^{5-}$  complexes was previously described in Ref. [6].

$[\text{Pd}_2\text{WO}(\text{AsW}_9\text{O}_{33})_2]^{10-}$  was prepared as described in Ref. [10] by mixing  $[\text{As}_2\text{W}_{19}\text{O}_{67}]^{14-}$  and  $[\text{Pd}(\text{H}_2\text{O})_4]^{2+}$  solutions and neutralizing to pH 6.5 ( $\text{Na}_2\text{CO}_3$ ). Acidity of the resulting solution was adjusted to pH 4 by adding  $\text{H}_2\text{SO}_4$  just before catalytic experiments.

The oxidation reactions were performed in thermostatic reactor supplied with magnetic stirrer and connected to burette for measuring gas consumption. NMR spectra were registered on a Bruker MSL-400 spectrometer at room temperature. The resonance and accumulation frequencies were 161.98 MHz and 0.05 Hz for  $^{31}\text{P}$ , 16.67 MHz and 0.05 Hz for  $^{183}\text{W}$ , 105.15 MHz and 20 Hz for  $^{51}\text{V}$ . IR spectra of Cs-salts in KBr were registered on a Specord IR-75 instrument.

## 3. Results and discussion

### 3.1. Characterization of the complexes

We selected heteropoly tungstate anion which could serve as a ligand for palladium and other metal ions, and at the same time, participate in the catalytic process. A number of complexes of the composition  $[\text{M}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$  are known for metal ions  $\text{M} = \text{Co}(\text{II}), \text{Cu}(\text{II}), \text{Sn}(\text{II})$  [8,9,11]. Analogous Pd(II) complex was also assumed [9]. We have characterized Pd(II) complex which was obtained in solution at  $\text{PW}_9$  concentration 0.02 M, a Pd(II) to  $\text{PW}_9$  molar ratio of 3:2 and  $\text{pH} \approx 4$ . According to NMR  $^{31}\text{P}$  data, a signal of the complex ( $-12.45$  ppm) comprised 80% of total phosphorus. Admixtures identified in solution were phosphate anions ( $\approx 0$  ppm),  $[\text{Pd}(\text{PW}_{11}\text{O}_{39})]^{5-}$  ( $-12.8$  ppm),  $\text{PW}_{12}\text{O}_{40}^{3-}$  ( $-15.3$  ppm). In NMR  $^{183}\text{W}$  spectrum Pd(II) complex (concentrated to 0.1 M  $\text{PW}_9$ ) exhibited two signals  $-69.6$  and  $-85.5$  ppm with intensities ratio of 2:1, that confirmed the structural type of  $[\text{M}_3(\text{A}-\alpha\text{-PW}_9\text{O}_{34})_2]^{12-}$  [9,11]. Of this type, complexes have three metal–oxygen polyhedron located between two  $\text{A}-\alpha\text{-PW}_9\text{O}_{34}^{9-}$  fragments: each M atom is bonded to four oxygen atoms of both heteropoly anions; water molecule or another particles may occur as additional ligands of M.

No Pd(II) hydroxide and, from the other hand, the same NMR  $^{31}\text{P}$  signal  $-12.45$  ppm were observed at a Pd(II) to  $\text{PW}_9$  ratio as high as 3:1. That indicated formation of  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2\text{Pd}_3\text{O}_x\text{H}_y]^{q-}$  species, which have been precipitated with  $\text{Cs}^+$  cations, and P:W:Pd ratio was conformed by elemental analysis. Additional palladium ions are, probably, combined into oligomeric hydroxo fragments, as previously determined for Pd(II) complexes with  $[\text{PW}_{11}\text{O}_{39}]^{7-}$  anion [6].

When starting from solutions containing Pd(II) and the second metal ion, we obtained complexes  $[\text{Pd}_x\text{M}_{3-x}(\text{PW}_9\text{O}_{34})_2]^{10-, 11- \text{ or } 12-}$  for  $\text{M} = \text{Fe}(\text{III}), \text{Cu}(\text{II})$  and  $x = 1$  or 2. Insertion

of the paramagnetic ions changed the width of the NMR  $^{31}\text{P}$  signal from sharp for the  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$  complex to broad peak.

According to NMR  $^{31}\text{P}$  spectra, the most part of heteropoly tungstate occurred in bimetallic Pd(II)–Fe(III) complexes. Issuing from intensities of the signals  $-66$  and  $-45$  ppm in spectra for Pd(II):Fe(III):  $\text{PW}_9 = 1:2:2$  ratio and for Pd(II): Fe(III):  $\text{PW}_9 = 2:1:2$  ratio, the signals can be assigned to  $[\text{PdFe}_2(\text{PW}_9\text{O}_{34})_2]^{10-}$  and  $[\text{Pd}_2\text{Fe}(\text{PW}_9\text{O}_{34})_2]^{11-}$ , respectively. Fe(III) +  $\text{PW}_9\text{O}_{34}^{9-}$  solution in the absence of Pd(II) gave a broad signal in positive region of chemical shifts.

Bimetallic  $[\text{Pd}_2\text{Cu}(\text{PW}_9\text{O}_{34})_2]^{12-}$  complex formed when Pd(II), Cu(II) and  $\text{PW}_9$  ions were taken in ratio of 2:1:2. In the NMR  $^{31}\text{P}$  spectrum predominated signal at  $-22$  ppm. Minor signals at approximately  $-32$ – $39$  ppm arised from Cu(II) monometallic complexes.

The structural type  $[\text{M}_3(\text{PW}_9\text{O}_{34})_2]$  of the prepared bimetallic complexes, as well as overstoichiometric Pd(II)-species was supported by IR spectra. The data of IR spectroscopy for the Pd(II), Pd(II)–Fe(III) and Pd(II)–Cu(II) complexes isolated from solution as Cs-salts are presented in Table 1. Vibration frequencies of P–O and W=O bands are identical for the complexes obtained here and known in the literature Co(II) and Sn(II) compounds. Specific interaction of different metal ions with the heteropoly tungstate was reflected in relative intensities of W–O–W and W–O–M bands.

When mixing  $\text{NaVO}_3$ ,  $[\text{Pd}(\text{H}_2\text{O})_4]^{2+}$  and  $\text{PW}_9\text{O}_{34}^{9-}$  in a ratio of 4:2:2, the NMR  $^{31}\text{P}$  spectrum indicated formation of a new complex giving a signal at  $-12.3$  ppm, that also predom-

inated in the absence of Pd(II). The rest of phosphorus was found in the Pd(II) complex with  $\text{PW}_9\text{O}_{34}^{9-}$  anion ( $\approx 10\%$ ,  $\delta = -12.45$  ppm). Only small amounts of  $\text{PW}_9\text{V}_3\text{O}_{40}^{6-}$  isomers and other  $[\text{PW}_{12-n}\text{V}_n\text{O}_{40}]^{-3-n}$  anions [12] were presented ( $< 10\%$ ,  $\delta = -13.2$ – $13.5$  ppm). NMR  $^{51}\text{V}$  spectrum of the solution consisted mainly of one signal at  $-535$  ppm. NMR  $^{183}\text{V}$  spectrum consists of four signals at  $-72.1$ ,  $-87.2$ ,  $-102$ ,  $-104$  ppm with intensities 2:1:4:8, the first two of them belong to Pd(II) complex and the last two can be assigned to a new complex. There is a little doubt that the solution contained two monometallic complexes  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2\text{Pd}_n\text{O}_x\text{H}_y]^{q-}$  ( $n_{\text{av}} \approx 10$ ) and, probably, according to NMR data,  $[(\text{VO})_3(\text{PW}_9\text{O}_{34})_2]^{9-}$  in a ratio of approximately 1:10. Structural type of V(V)-complex was confirmed by IR spectrum that was found to be typical to  $[\text{M}_3(\text{PW}_9\text{O}_{34})_2]$  anions (Table 1).

### 3.2. Catalysis

The catalytic performance of the Pd(II) complexes was tested for two oxidation reactions: complete reduction of oxygen to water and hydroxylation of benzene to phenol in two-phase water–benzene medium (Table 2). These data should be considered, taking into account the reducibility of the complexes with hydrogen.

It was determined that the complexes  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$ ,  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2\text{Pd}_3\text{O}_x\text{H}_y]^{q-}$ ,  $[\text{PdFe}_2(\text{PW}_9\text{O}_{34})_2]^{10-}$  as well as  $[\text{Pd}(\text{PW}_{11}\text{O}_{39})]^{5-}$ , and the mixture of two complexes  $[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2\text{Pd}_n\text{O}_x\text{H}_y]^{q-}$  and  $[(\text{VO})_3(\text{PW}_9\text{O}_{34})_2]^{9-}$  react with  $\text{H}_2$  in aqueous solution at room temperature to produce metal-

Table 1  
IR spectra of Cs (1–4)- and K (5–6)-salts of the complex anions

No.	Composition	$\nu(\text{P-O}), \text{cm}^{-1}$	$\nu(\text{W=O}), \text{cm}^{-1}$	$\nu(\text{W-O-W and W-O-M})$	Ref.
1	$[\text{Pd}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$	1075(s), 1013(m)	935(s)	880(w), 785(vs), 695(m)	Present work
2	$[\text{Pd}_2\text{Fe}(\text{PW}_9\text{O}_{34})_2]^{11-}$	1080(s), 1020(m)	945(s)	885(m), 790(vs), 700(sh)	
3	$[\text{Pd}_2\text{Cu}(\text{PW}_9\text{O}_{34})_2]^{12-}$	1073(s), 1015(m)	943(s)	890(w), 790(vs), 750(sh), 705(sh)	
4	$[(\text{VO})_3(\text{PW}_9\text{O}_{34})_2]^{9-}$	1070(s), 1045(m)	950(s)	790(vs), 700(sh)	
5	$[\text{Co}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$	1060(s), 1020(m)	930(s)	800(vs), 715(s)	[8]
6	$[\text{Sn}_3(\text{PW}_9\text{O}_{34})_2]^{12-}$	1074(s), 1025(m)	940(s)	890(m), 807(s), 758(s), 712(s)	[11]

Table 2

Data on H<sub>2</sub> to H<sub>2</sub>O (I) and C<sub>6</sub>H<sub>6</sub> to phenol (II) oxidation in the presence of Pd(II)-containing complexes. Conditions: O<sub>2</sub>/H<sub>2</sub> = 1/2, pH 4 (except for 3), reaction time 1 h; (I) 5 ml of 0.01 M solution of complex, T 20°C; (II) 0.3 ml of 0.01 M solution of complex, 1 ml of benzene, T 25°C (T 20°C for 3, 4); turnover numbers (TN) of catalysts were calculated per 1 g-atom of palladium

No.	Catalyst composition	(I)	(II)	
		TN (H <sub>2</sub> O)	Phenol, 10 <sup>6</sup> mol	TN (phenol)
1	[Pd <sub>3</sub> (PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> ] <sup>12-</sup>	11	3.1	0.35
2	[Pd <sub>3</sub> (PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> Pd <sub>3</sub> O <sub>x</sub> H <sub>y</sub> ] <sup>q-</sup>	16	5.2	0.30
3	[Pd(PW <sub>11</sub> O <sub>39</sub> )] <sup>5-</sup> (pH 2)	34	4.0	1.30
4	[Pd(PW <sub>11</sub> O <sub>39</sub> )] <sup>5-</sup>	16	1.0	0.30
5	[Pd <sub>2</sub> Cu(PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> ] <sup>12-</sup>	0	0	0
6	[Pd <sub>3</sub> (PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> Pd <sub>n</sub> O <sub>x</sub> H <sub>y</sub> ] <sup>q-</sup> + [(VO) <sub>3</sub> (PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> ] <sup>9-</sup>	32	5.3	0.90
7	[Pd <sub>2</sub> Fe(PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> ] <sup>11-</sup>		21	3.50
8	[PdFe <sub>2</sub> (PW <sub>9</sub> O <sub>34</sub> ) <sub>2</sub> ] <sup>10-</sup>	30	15	5.0
9	[Pd <sub>2</sub> WO(AsW <sub>9</sub> O <sub>33</sub> ) <sub>2</sub> ] <sup>10-</sup>	0	1.0	0.15

lic palladium. The rate of reduction was controlled by H<sub>2</sub> diffusion (curves 1, 2 in Fig. 1). Based on the amount of the gas consumed, complete reduction of Pd(II) was achieved in Pd(II) and Pd(II)–Fe(III) complexes, whereas in Pd(II)–V(V) system not only complete reduction of Pd(II), but also partial reduction of V(V) was observed. [Pd<sub>2</sub>Cu(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>12-</sup> and complex of tentative composition [Pd<sub>2</sub>WO(AsW<sub>9</sub>O<sub>33</sub>)<sub>2</sub>]<sup>10-</sup> reacted very slowly, but the rate increased with time, probably, because of appearance of metallic palladium catalyzing following reduction (curves 3, 4 in Fig. 1).

As revealed, the rate of H<sub>2</sub> + O<sub>2</sub> reaction for different complexes (Table 2) correlated basically with effectiveness of the reduction with hydrogen. Slowly reduced complexes showed zero activity in O<sub>2</sub> + H<sub>2</sub> consumption. Equimolar solutions of the [Pd<sub>3</sub>(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>12-</sup> (1) and [Pd(PW<sub>11</sub>O<sub>39</sub>)]<sup>5-</sup> (4) complexes showed close activity referred to the amount of palladium, however, [Pd(PW<sub>11</sub>O<sub>39</sub>)]<sup>5-</sup> prepared in more acidic solution (3) was more active.

The presence of readily reducible polyhydroxo fragments in the complex was a significant factor for the H<sub>2</sub> + O<sub>2</sub> reaction that was responsible for the high activity of [Pd<sub>3</sub>(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>Pd<sub>3</sub>O<sub>x</sub>H<sub>y</sub>]<sup>q-</sup> (2) and Pd(II)–V(V) system (6).

In the course of the reaction, all Pd(II)-containing complexes were kept in solution. After 1 h of reaction, according to the spectrophotomet-

ric data, only 10% to 30% of complexes decomposed to produce palladium metal.

The results of testing Pd(II) containing complexes in benzene hydroxylation with O<sub>2</sub>/H<sub>2</sub> are also given in Table 2. Inactive in water production [Pd<sub>2</sub>Cu(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>12-</sup> and [Pd<sub>2</sub>WO(AsW<sub>9</sub>O<sub>33</sub>)<sub>2</sub>]<sup>10-</sup> complexes possessed very low if any activity in hydroxylation. On this basis, it can be assumed that O<sub>2</sub>/H<sub>2</sub> hydroxylation, as well as formation of water, needs the presence of Pd<sup>0</sup> species. As we believe, the considerable condition for effective hydroxyl-

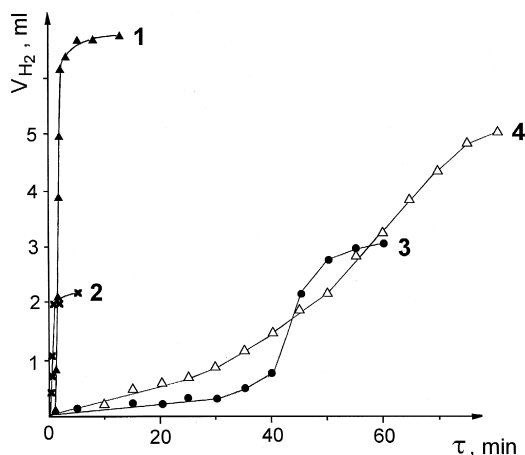
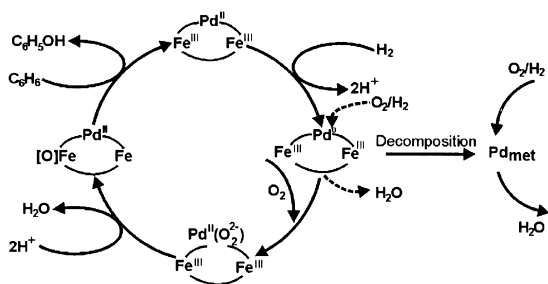


Fig. 1. Dependence on time of H<sub>2</sub> consumption with aqueous solutions of heteropolycomplexes. 1 — [Pd<sub>3</sub>(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>Pd<sub>n</sub>O<sub>x</sub>H<sub>y</sub>]<sup>q-</sup> + [(VO)<sub>3</sub>(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>9-</sup>, 2 — [PdFe<sub>2</sub>(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>10-</sup>, 3 — [Pd<sub>2</sub>Cu(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>12-</sup>, 4 — [Pd<sub>2</sub>WO(AsW<sub>9</sub>O<sub>33</sub>)<sub>2</sub>]<sup>10-</sup>, pH 4, 0.02 M P (1–3), 0.04 M As (4), 20°C, V = 5 cm<sup>3</sup>.

ation is stabilizing the Pd<sup>0</sup> species in solution by means of heteropoly tungstate [6].

On the other hand, it is well known that hydroxylation of benzene is participated by an appropriate oxygen transfer agent. This function was realized by the [(VO)<sub>3</sub>(PW<sub>9</sub>O<sub>34</sub>)<sub>2</sub>]<sup>9-</sup> complex in the system (6) that was more active than Pd(II) monometallic complexes. As far as the dioxygen activator and the oxygen transfer are located in different compounds, it can be suggested that active oxygen is transported through solution, probably, as hydrogen peroxide.

Bimetallic Pd(II)–Fe(III) complexes were found to possess the most substantial activity in hydroxylation of benzene. Catalytic action of Fe(III)-containing heteropoly tungstates in hydrocarbon oxidations with H<sub>2</sub>O<sub>2</sub> has been reported [13,14]. The oxidation process is facilitated owing to location of oxygen transferring Fe(III) ions in a close connection to palladium. It should be noted that the presence of Fe(III) in the complexes increased by one order a fraction of the O<sub>2</sub>/H<sub>2</sub> gases consumed for benzene hydroxylation (compare 1 and 8 in Table 2). The overall scheme of the process participated by Pd(II)–Fe(III) complexes can be represented hypothetically as follows.



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