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Sandwich type tri-palladium substituted phosphotungstate, $[Pd_3(PW_9O_{34})_2]^{11-}$: Synthesis, structural characterization and catalytic evaluation

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ABSTRACT

The present work showcases, first time, the synthesis of tri-palladium substituted sandwich type phosphotungstate, $[Pd_3(PW_9O_{34})_2]^{11-}$, from its individual salts in super acidic medium as well as the single crystal structure of the same. Further, various spectral analysis carried out to support the crystal data. A preliminary study for the hydrogenation of **nitrobenzene** has been carried out to evaluate the catalytic activity of the complex.

1. Introduction

Polyoxometalates (POMs) are anionic metal oxygen clusters with interesting structural and chemical properties [1–6] that have led to a wide variety of applications in different fields such as electrochemical, magnetic, medicinal and catalysis [4,6–9]. They can also be rationally modified on the molecular level including shape, size, charge density, redox states and stability [6,10–12]. POMs containing noble metals, in particular palladium, have gained special interest over the last years mainly because the incorporation of noble metal ions into POM frameworks allows for keeping the noble metal ions soluble in aqueous or organic media and at the same time encapsulated in a fully inorganic, thermally stable and redox-stable metal-oxo matrix [13]. Looking into the importance of the same, Kortz and co-workers have started work in the same direction and contributed significantly over the past two decades, by synthesizing a large number of Pd-substituted polyoxometalates [13–19]. They successfully synthesized the Pd_{13}-nanocube $[Pd_{13}As_8O_{40}H_6]^{8-}$ in 2008 [20] and as a result, a new sub-class of metal substituted POMs, i.e., the polyoxopalladates (POPs) was born [21].

Literature survey presents a large number of mono- as well as di-substituted sandwich type Pd-phosphotungstates. For instance, in 2008, the group of Proust synthesized mono- as well as di-palladium substituted anions, $[Pd\{WO(H_2O)\}_2\{A,\alpha\text{-}PW_9O_{34}\}_2]^{8-}$ and $[Pd_2\{WO(H_2O)\}\{A,\alpha\text{-}PW_9O_{34}\}_2]^{10-}$ from the mono-vacant $[P_2W_{20}O_{70}(H_2O)_2]^{10-}$ and the bis-lacunary precursor $[P_2W_{19}O_{69}(H_2O)]^{14-}$ respectively, and thoroughly characterized by single crystal XRD as well as multinuclear NMR spectroscopy [22]. In another report, in 2011, Kortz et al described the synthesis of $Pd_2(PW_{11}O_{39})_2$ from mono-lacunary POM precursor, $K_9[PW_{11}O_{39}].13H_2O$ and $PdCl_2$ or $Pd(NO_3)_2$ as well as its detailed characterization by single crystal XRD and multinuclear NMR for confirmation of the dimer structure. Further, they proved that reaction of palladium salts with lacunary POM precursors did not lead to incorporation of metal ion into the lacuna, but formed a dimer instead [13].

On the other hand, the synthesis of tri-palladium substituted phosphotungstates has not been explored to a great extent. In 1986, Knoth et al reported the synthesis of $[Pd_3(A\text{-}PW_9O_{34})_2]^{12-}$ polyanion from trilacunary $K_9[A\text{-}\alpha\text{-}PW_9O_{33}]$ and palladium nitrate, along with its characterization by basic spectral techniques like NMR and FT-IR. Since the spectra were similar to the sandwich type cobalt complex synthesized earlier by the same group [23], the synthesized complex was presumed to be isostructural [24]. Almost a decade and a half later, Kuznetsova et al synthesized $[Pd_3(PW_9O_{34})_2)]^{12-}$ by adding solid $Na_8HPW_9O_{34}{\cdot}24H_2O$ to a solution of $[Pd(H_2O)_4]^{2+}$ at pH 1.6. This complex was characterized by NMR and FT-IR and as the results obtained were similar to previous reports [24], the structure was assumed to be similar. Further, catalytic activity was evaluated for two reactions; namely reduction of O_2 to water and hydroxylation of benzene to phenol [25].

In 2008, Proust et al synthesized $[Pd_3(A\text{-}PW_9O_{34})_2]^{12-}$ following the route by Knoth et al and described a detailed characterization

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0277-5387/

of the same [22]. The ^{31}P NMR of the mother solution of the sodium salt indicated that the tri-palladium, though a major product, always came with a mixture of unidentified by-products, and even precipitation with KCl did not yield pure compound. Preliminary single crystal studies revealed presence of an asymmetric unit composed of three independent thirds of an anion along with 11 potassium cations. Further determination of the crystal structure seemed not viable due to poor crystal quality and presence of unidentified mixtures.

In the present paper, we have synthesized tri-palladium substituted sandwich type phosphotungstate using individual salts, i.e., sodium tungstate and potassium hydrogen phosphate. For the first time, we were able to isolate the pure crystal and systematically deduce its crystal structure. Further characterizations by various spectral techniques were also carried out to support the data obtained from single crystal XRD, such as FT-IR, UV–Visible, Cyclic Voltammetry and NMR spectroscopy. A preliminary study on the catalytic activity was carried out for industrially important organic transformations such as hydrogenation of nitrobenzene, cyclohexene and crotonaldehyde.

2. Experimental

2.1. Materials

All chemicals used were of A.R. grade. $Na_2WO_4{\cdot}2H_2O$, $K_2HPO_4{\cdot}7H_2O$, $PdCl_2$, CsCl and diethyl ether, were obtained from Merck and used as received.

2.2. Synthesis

Tri-palladium substituted phosphotungstate, $[Pd_3(PW_9O_{34})_2]^{11-}$ **was synthesized by following method.** $PdCl_2$ (0.355 g, 2 mmol) was dissolved in minimum amount of water by heating (addition of 2–3 drops of conc. HCl is required for dissolution). To this, saturated aqueous solution of $K_2HPO_4{\cdot}7H_2O$ (0.071 g, 1 mmol) was added drop-wise, followed by addition of 1 mL conc. H_2SO_4 with continuous stirring. Finally, saturated solution of Na_2WO_4 (1.65 g, 10 mmol) prepared in water was added to above resultant mixture, followed by careful drop-wise addition of 5 mL conc. H_2SO_4 with stirring. The reaction mixture was allowed to cool at room temperature (due to exothermic nature of the resultant mixture), the obtained product was then extracted with diethyl ether (twice, using 25 mL). The etherate layer (lower layer) was bubbled out thoroughly with molecular oxygen to break the POM-etherate complex, finally the traces of ether were removed by gentle heating. To this, about 5 mL of distilled water was added, heated at 80 °C, filtered and the filtrate was kept aside for crystallization. After 10 days, long needle shaped dark brown crystals are obtained and designated Cs-K-$Pd_3(PW_9)_2$. (The crystals growth after 10 days and after 17 days are shown in Scheme 1) The visual appearance of crystals after 17 days showed no aggregates formation of Pd as well as no other isomeric product formation indicating the stability of the synthesized materials.

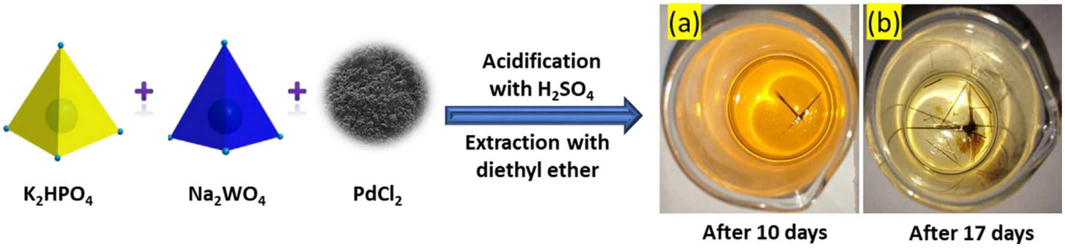
2.3. Characterization

The synthesized material was characterized by Single crystal XRD, elemental analysis, FT-IR Spectroscopy, solution phase ^{31}P NMR Spectroscopy, UV–Visible spectroscopy and Cyclic Voltammetry.

Single Crystal X-Ray diffraction studies were carried out in a Bruker D8 Venture SC-X-Ray diffractometer using Cu(α) source (λ = 1.5406 Å), KAPPA goniometer and a PHOTON 100 CMOS detector. Data collection was carried out with APEX III software [26]. Reflections were merged and corrected for Lorenz and polarization effects, scan speed, and background using SAINT [2]. Absorption corrections, including odd and even ordered spherical harmonics were performed using SADABS [27]. Space group assignments were based upon systematic absences, E-statistics, and successful refinement of the structures. Structures were solved by direct methods with the aid of successive difference Fourier maps, and were refined against all data using the APEX III software [26,28] in conjunction with SHELXL-2014 [29]. H atoms of the phenyl rings were placed in calculated positions and refined using a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Refinements were conducted by full-matrix least squares against $|F|$ using all data. Images of the crystal structures were generated by Diamond, version 3.2 (software copyright, Crystal Impact GbR) and PLATON [30]. Some of the disagreeable reflections were removed by the SQUEEZE command in PLATON [30]. However, it is well known that due to presence of heavy tungsten atoms, artefacts may appear close to the lighter oxygen atoms. Further, in the presence of heavy elements, such as W's, it is almost impossible to locate or fix hydrogens corresponding to water molecules. Hence, the crystal data shows high Rint value as well as discrepancy in the formula weight. The data is provided free of charge by The Cambridge Crystallographic Data Centre (CSD 1979233).

2.4. Catalytic study

The synthesized material was used as a catalyst for the hydrogenation of nitrobenzene, cyclohexene and crotonaldehyde. The reaction was carried out in a parr reactor instrument with the following components: SS-316 batch reactor (100 mL), H_2 reservoir and electric temperature and pressure controller. Known amount of the catalyst was taken along with a mixture of 20 mL acetonitrile and 30 mL water. To this 1.23 mL (1 mmol) of nitrobenzene was added. Preliminary reaction was carried out at **8 bar H_2 pressure**, 60 °C for 5 h. The reaction mixture was then extracted multiple times using dichloromethane and analyzed in Shimadzu GC-2014 gas chromatograph instrument with RTx-5 capillary column. Hydrogenation reaction of cyclohexene and crotonaldehyde were also carried out under similar conditions.



Scheme 1. Synthesis of Cs-K-$Pd_3(PW_9)_2$ and crystal growth after (a) 10 days and (b) 17 days.

3. Results and discussion

3.1. Crystal structure determination

The title compound Cs-K-$Pd_3(PW_9)_2$ crystallized in the monoclinic space group $P2_1/n$ and is composed of two trilacunary $[\alpha\text{-}PW_9O_{34}]^{9-}$ anions sandwiching three crystallographically distinct square-planar Pd^{2+} ions that have two Pd-O(W) bonds to each Keggin unit (See Fig. 1) leading to a sandwich-type structure with idealized C_{2h} point symmetry. The trilacunary Keggin units hence act as bidentate ligands, resulting in the square-planar coordination geometry around the Pd^{2+} ions with Pd-O bond lengths in the range of 1.98(2)–2.08(2) Å (av. 2.015 Å).(Table 1). This basic building block, $[\alpha\text{-}PW_9O_{34}]^{9-}$, in the Cs-K-$(PW_9)_2Pd_3$ is similar to the well-defined trivacant Keggin-type polyoxoanions, which comprise a tetrahedral PO_4 group surrounded by three edge sharing W_3O_{13} triads [22,24,25]. In all the mentioned reports, the structure proposed is similar to the present work, on the bases of physico-chemical characterizations like IR and NMR studies. However, this could not be confirmed by single crystal studies due to lack of good quality crystals as well as formation of multiple products. The present work is the first instance where the single crystal structure has been solved and described in detail. The important bond distances are presented in Table S1 (Supporting information).

3.2. Analytical and spectral analysis

The observed and the theoretical values for the elemental analysis were obtained from ICP analysis and are in good agreement with each other. Theoretical: P, 0.99; Pd, 5.13; W, 53.12; K, 2.01; Cs, 16.14. Observed: P, 0.98; Pd, 5.09; W, 52.92; K, 1.94; Cs, 16.89. The elemental analysis of the compound also confirms 8 Cs ions. The total number of water molecules was calculated based on the weight loss from TGA which corresponds to 16 water molecules. Based on the crystal structure, elemental and thermal analysis, the molecular formula was proposed to be $Cs_8K_3[(PW_9O_{34})_2Pd_3].16H_2O$ [Cs-K-$Pd_3(PW_9)_2$].

The FT–IR spectrum of Cs-K-$Pd_3(PW_9)_2$ (Fig. 2) shows P—O stretching bands at 1180, 1141 and 1057 cm^{-1}, indicating the formation of tri-lacunary species, while bands observed at 979 cm^{-1}, 856 and 810 cm^{-1} correspond to W=O and W—O—W stretching vibrations respectively. Further, the peak at 686 cm^{-1} is attributed to the Pd—O stretching vibrations.

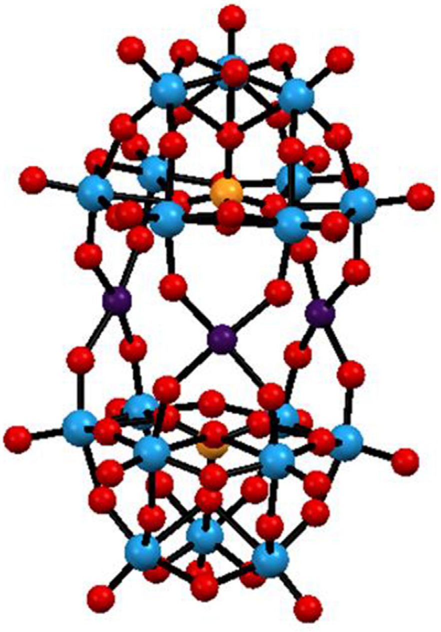


Fig. 1. Ball and stick model of the anion $[Pd_3(PW_9O_{34})]^{11-}$.

Table 1
Single crystal data and structure refinement parameters for Cs-K-$Pd_3(PW_9)_2$.

Compound	Cs-K-$Pd_3(PW_9)_2$
Empirical Formula	$Cs_8Pd_3P_2W_{18}O_{82}$ $(H_{32}Cs_8K_3Pd_3P_2W_{18}O_{84})$*
Formula Weight (gm/mol)	6065.72 (6247.07)*
Crystal System	Monoclinic
Space Group	$P2_1/n$
a (Å)	16.9441(13)
b (Å)	19.9567(13)
c (Å)	26.9788(17)
α (°)	90
ß (°)	98.975(2)
γ (°)	90
Volume (Å^3)	9011.1(11)
Z	4
$D_{calc.}$ (gm/cm^3)	4.471
Absorption Coefficient (mm^{-1})	26.776
F(0 0 0)	10,384
θ range for data collection	1.275 to 25.828
Completeness to θ_{max}	99.9%
Index Ranges	$-20\leq h\leq 20$, $-24\leq k\leq 24$, $-33\leq l\leq 33$
Reflections Collected	106,159
Unique Reflections	17,340
R_{int}	0.1166
Absorption Correction	SADABS
Data/Restraints/Parameters	17,340/18/616
Goodness of Fit on F^2	1.044
R_1[a] ($I > 2\sigma(I)$)	0.1364
wR_2[b] (all data)	0.2120
Largest difference peak and hole (e/Å^3)	5.596 and −5.365

*Values in brackets indicate the actual formula unit and molar mass as obtained from elemental analysis for a bulk sample.
[a] $R_1 = \Sigma|F_o| - |F_c|/\Sigma|F_o|$, [b] $wR_2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

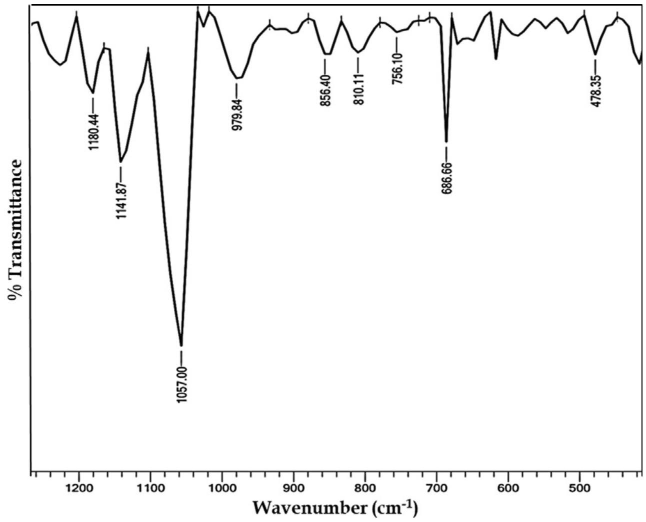


Fig. 2. FT-IR spectrum of Cs-K-$Pd_3(PW_9)_2$.

The UV–Vis spectra of Cs-K-$Pd_3(PW_9)_2$ is shown in Fig. 3. Distinct peaks for W–O charge transfer at about 300 nm are seen clearly, along with distinctive peak for the Pd(II)–O charge transfer at 392 nm, confirming presence of Pd.

The cyclic voltammetry of 1 mM solutions of Cs-K-$Pd_3(PW_9)_2$ was performed in an acetate buffer of pH–5. A glassy carbon electrode was used as the working electrode, while Ag/AgCl electrode

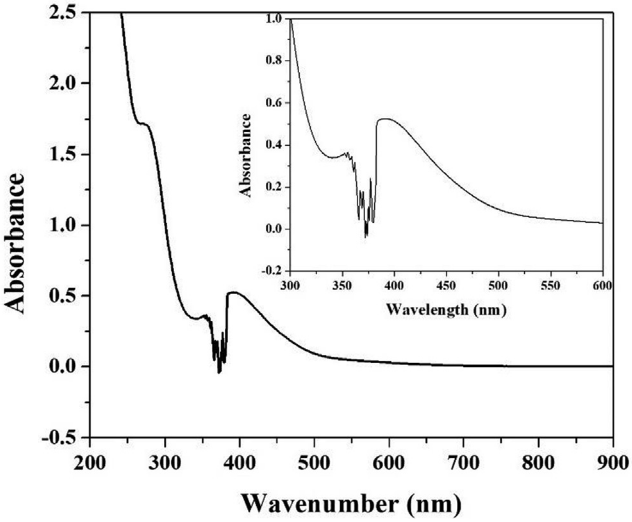


Fig. 3. UV–Vis spectra of Cs-K-$Pd_3(PW_9)_2$.

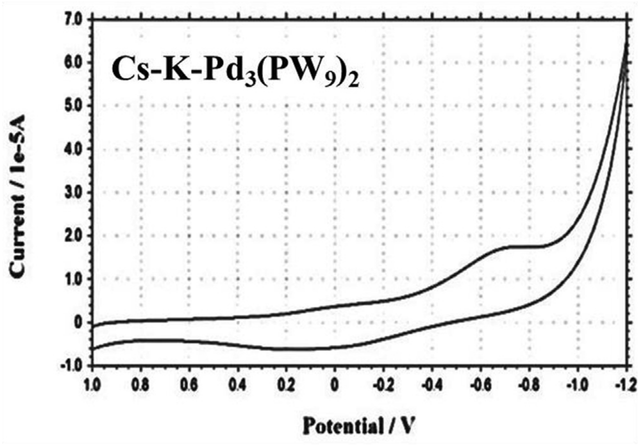


Fig. 4. Cyclic Voltammogram of Cs-K-$Pd_3(PW_9)_2$.

was used as the reference electrode and Pt-wire was used as the counter electrode and the cyclic voltammogram is shown in the Fig. 4. An irreversible peak at 0.21 V indicates the oxidation of Pd (II) → Pd(IV). This value is considerably lower compared to other Pd^{2+} species in water, but the decrease in the potential difference is generally seen in case of polyoxometalates [31].

The ^{31}P solution NMR of Cs-K-$Pd_3(PW_9)_2$ (Fig. 5) shows two peaks at −10.80 and −12.21 ppm, consistent with the A-type structure. The peak at −12.21 ppm is in agreement with that obtained by Proust et al, and also indicates that the two PW_9 units are equivalent [22]. On the other hand, a more intense peak at −10.80 ppm may be attributed to thermolytic dehydration of the anion where it slowly oxidises in air in the aqueous medium [23].

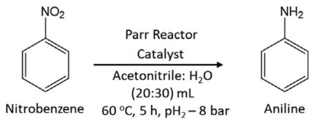
3.3. Catalytic study (Preliminary)

To evaluate the catalytic efficiency of the catalyst for the hydrogenation, nitrobenzene was selected as model substrate as shown in Scheme 2. Effect of different reaction parameters such catalyst amount and pressure were studied for maximum % conversion.

Control experinment were performed using $K_9PW_9O_{34}$ (PW_9), $PdCl_2$, Cs-K-$(PW_9)_2Pd_3$ and without catalyst to prob the active species for the reaction as shown in Table 2. Obtained resutls shows that hydrogenation did not proceed without catalyst, whereas PW_9 was inactive towards the reaction. However, almost identical conversion was achieved in case of $PdCl_2$ and Cs-K-$(PW_9)_2Pd_3$, confirmig the presence of Pd, as well as indicating that Pd is the only active species responsible for the hydrogenation reaction.

The effect of catalyst amount was evaluated by varying the amount from 5 to 15 mg. From the obtained results shown in Fig. 6, 10 mg catalyst was considered for further study.

Influence of pressure was studied by screening applied pressure from 4 to 10 bar (Fig. 7). Achieved results showed the linear increase of the % conversion with increase in pressure from 4 to 8 bar. Further increase in pressure had no influence on the % conversion, 8 bar was optimized as optimised pressure.



Scheme 2. Nitrobenzene hydrogenation.

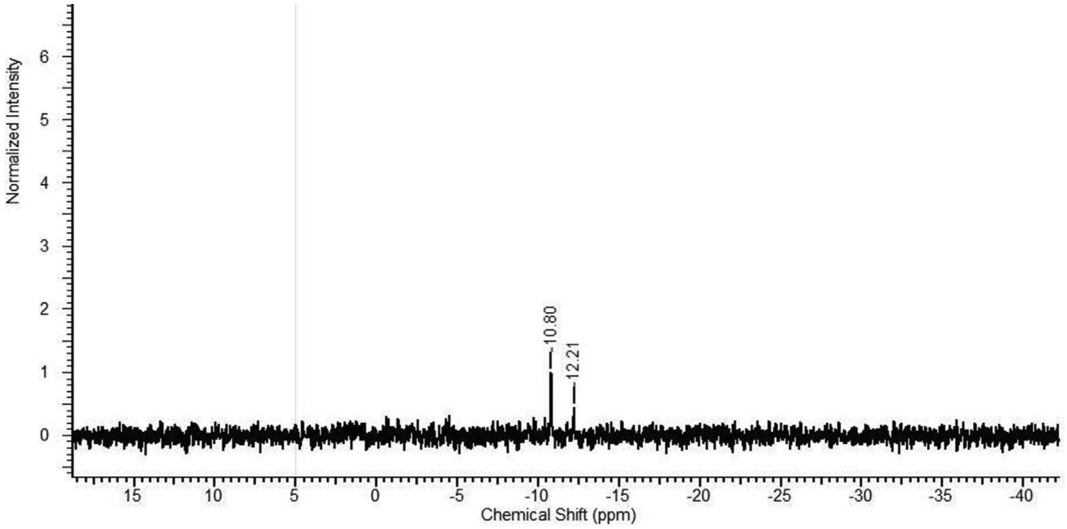


Fig. 5. ^{31}P solution NMR of Cs-K-$Pd_3(PW_9)_2$.

Table 2
Control experiments.

Catalyst	% Conversion
Without catalyst	NA
PW_9 (8.29 mg)	NA
$^aPdCl_2$ (0.86 mg)	98
^{a}Cs-K-$Pd_3(PW_9)_2$ (10 mg)	99

Reaction condition: Nitrobenzene (9.87 mM), ^{a}-Active amount of Pd (0.049 mol%), Acetonitrile: H_2O (20:30) mL, Temperature (60 °C), pH_2 (8 bar), (Time (5 h). NA = Not active.

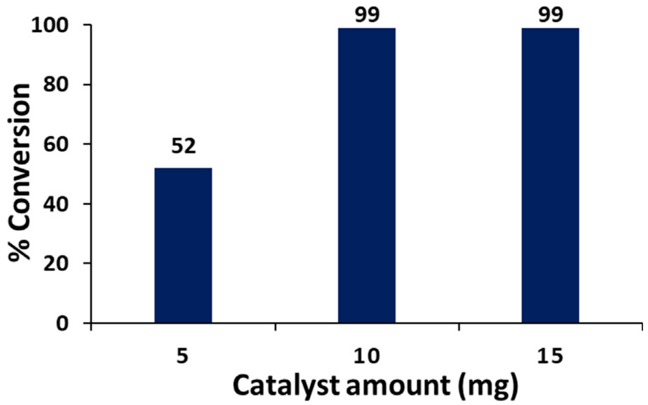


Fig. 6. Effect of Catalyst amount. Reaction conditions: Nitrobenzene (9.87 mM), Acetonitrile:H_2O (20:30) mL, Temperature (60 °C), pH2 (8 bar), Time (5 h).

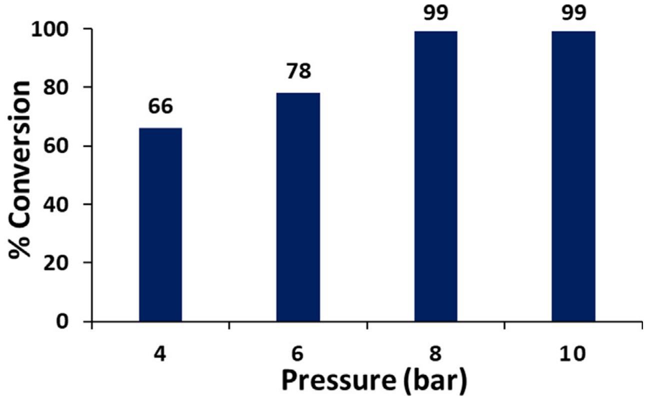
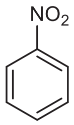
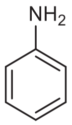
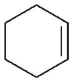
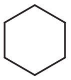
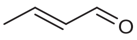
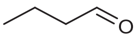


Fig. 7. Effect of pressure. Reaction conditions: Nitrobenzene (9.87 mM), Catalyst (10 mg = 0.049 mol%), Acetonitrile:H_2O (20:30) mL, Temperature (60 °C), Time (5 h), Substrate/catalyst ratio (2056/1).

Table 3
Preliminary substrate study.

Substrate	% conversion	Product/% selectivity	TON
	99	 100	2028
	85	 100	1742
	83	 99	1701

Reaction condition: Substrate (9.87 mM), Catalyst (0.049 mol%), Acetonitrile: H_2O (20:30) mL, Temperature (60 °C), pH_2 (8 bar), (Time (5 h).

Preliminary study was also carried out for hydrogenation of cyclohexene and crotonaldehyde under experimental conditions (as shown in Scheme 2). Obtained results (Table 3) show that the catalyst is viable for the substrates having different functionality, moreover in case of crotonaldehyde it shows selective C═C hydrogenation.

4. Conclusion

We have successfully isolated the tri-palladium sandwich type phosphotungstate as a single product starting from individual salts through slight modification in reported technique. The single crystal was obtained in aqueous media, from which, the crystal structure was elucidated. Further spectral analysis confirmed the results of single crystal analysis. This complex showed excellent catalytic activity for the hydrogenation of various alkenes, with very high conversion and single selectivity. Though further optimization for the reactions are in progress, this material opens new avenues towards selective hydrogenation of different functional groups.

CRediT authorship contribution statement

Rajesh Sadasivan: Visualization, Investigation, Data curation, Validation, Writing - original draft. **Anjali Patel:** Conceptualization, Methodology, Writing - review & editing, Supervision. **Anish Patel:** Visualization, Investigation, Data curation, Validation, Writing - original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Associated content

Crystal data is available and is provided free of charge by The Cambridge Crystallographic Data Centre (CSD 1979233).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.poly.2020.114896.

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