Preparation and Characterization of $Cs_{2.8}H_{1.2}PMo_{11}Fe(H_2O)O_{39} \cdot 6H_2O$ and Investigation of Effects of Iron-substitution on Heterogeneous Oxidative Dehydrogenation of 2-Propanol

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Table S1. Crystallographic data and results of refinement for $[C_6H_5(CH_3)_3N]_5[PMo_{11}\{Fe(Cl)\}O_{39}] \cdot CH_3CN \cdot H_2O$

Empirical formula	$C_{47}H_{75}N_6O_{40}ClFeMo_{11}P$
Formula weight	2539.72
Temperature	20 (2) °C
Wavelength	0.71069 Å
Crystal shape	prismatic
Crystal color	yellow
Crystal size	$0.35 \times 0.30 \times 0.30$ mm
Crystal system, space group	triclinic, $P\bar{1}$
Unit cell dimensions	$a = 15.103(5)$ Å $\alpha = 100.20(3)°$
	$b = 20.708(5)$ Å $\beta = 116.55(2)°$
	$c = 14.834(6)$ Å $\gamma = 81.07(3)°$
Volume	$4069(2)$ Å^3
Absorption coefficient	1.945 mm^{-1}
$F(000)$	2474
θ range for data collection	2.59 to 30.02°
Data collection method	ω-2θ
Transmission factors	0.9384 – 1.0000
Final R values ($F_0 > 4.0\sigma(F_0)$)	$R1 = 0.0675$, $wR2 = 0.1848$
Final R values (all data)[a]	$R1 = 0.1853$, $wR2 = 0.2286$
Goodness of fit on F^2	1.036
Extinction coefficient[b]	0.0004(1)
Index ranges	$-18 \leq h \leq 21$, $-28 \leq k \leq 29$, $-20 \leq l \leq 0$

[a] $R1 = \Sigma||Fo|-|Fc||/\Sigma|Fo|$, $wR2 = \{\Sigma[w(Fo^2-Fc^2)^2]/\Sigma[w(Fo^2)^2]\}^{1/2}$; $w = 1/[\sigma^2(Fo^2)+(0.1071P)^2+1.4837P]$ ($P=[0.3333(0,Fo^2)max+0.6667Fc^2]$).

[b] Fc is multiplied by $k[1+0.0003590eFc^2/\sin(2\theta)]^{-1/4}$ where k is overall scale.

Table S2. Acidity of catalysts[a]

Catalyst	Acidity	
	Amount[b]	Temp. range[c]
I	170	100 - 560
Fe^{3+}(1.3wt%)/$Cs_{3.0}PMo_{12}O_{40}$	90	100 - 510
Fe^{3+}(2.5wt%)/$Cs_{3.0}PMo_{12}O_{40}$	150	100 - 520
$Cs_{3.0}PMo_{12}O_{40}$	trace	-

[a] Acidic properties of the catalysts were measured by temperature programmed desorption (TPD) of NH_3 by a TPD system equipped with a quadrupole mass spectrometer (Multitask TPD, BEL Japan Inc.). Catalysts (80 mg) were treated in a He flow at 200 ^{o}C for 2 h, and NH_3 was adsorbed at the partial pressure of 1.33 kPa and 100 ^{o}C. After the excess NH_3 was flushed in a He flow at 100 ^{o}C for 1 h, the sample was heated up to 800 ^{o}C at a rate of 10 ^{o}C min^{-1}. Mass numbers (*m/z*) of 16, 17, and 28 were used for the detection of amounts of NH_3 and N_2.

[b] Amount of ammonia desorbed / μmol g^{-1}.

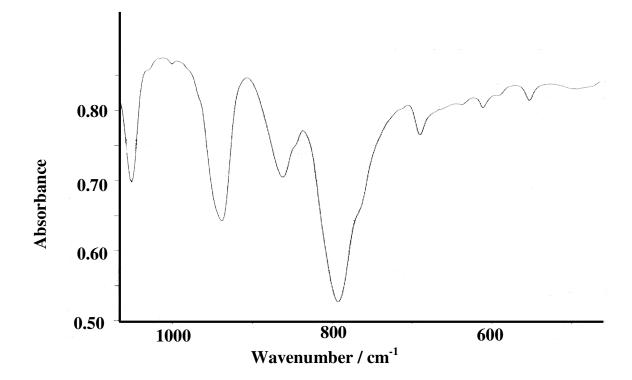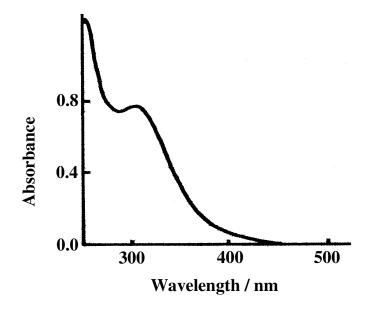[c] Temperature range of ammonia desorbed / ^{o}C.

Figure S1. IR spectrum of $[C_6H_5(CH_3)_3N]_5[PMo_{11}\{Fe(Cl)\}O_{39}]\cdot CH_3CN\cdot H_2O(KBr)$.

Figure S2. UV-*vis* spectrum of $[C_6H_5(CH_3)_3N]_5[PMo_{11}\{Fe(Cl)\}O_{39}]\cdot CH_3CN\cdot H_2O$ in acetonitrile/dimethylsulfoxide (*v*/*v* = 7/3).
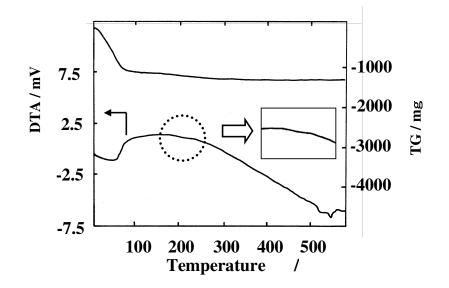
Figure S3. TG/DTA profile of $Cs_{2.8}H_{1.2}[PMo_{11}\{Fe(H_2O)\}O_{39}]\cdot6H_2O$.