Research Summary and Presentation I: Encapsulation of Two Potassium Cations in Preyssler-Type Phosphotungstates : Preparation, Structural Characterization, Thermal Stability, Activity as Acid Catalyst, and HAADF-STEM Images

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In all cases, only one cation is encapsulated in one Preyssler type molecule

In this research, new Preyssler type phosphotungstate was isolated with two encapsulated K^+

Preparation of $\text{K}_{13}[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]\cdot 26\text{H}_2\text{O}$

- $\text{K}_{12}[\text{P}_5\text{W}_{30}\text{O}_{110}\text{Bi}(\text{H}_2\text{O})]\cdot 24\text{H}_2\text{O}$ (2.46 g)
- KCl (1.34 g)
- CH_3COOK Buffer (5 mL, pH= 4.7)

Preparation of $\text{H}_{13}[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]\cdot 39\text{H}_2\text{O}$

Preparation of $\text{H}_{13}[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]\cdot 20\text{DMSO}\cdot 1\text{H}_2\text{O}$

Heating 70°C

Oil Bath

Solution

Cooled down

$\text{H}_{13}[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]\cdot 39\text{H}_2\text{O}$
(0.40 g) + 0.1 mL DMSO

$\text{H}_{13}[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]\cdot 20\text{DMSO}\cdot 1\text{H}_2\text{O}$

Preparation and Isolation of $[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]^{13-}$

Preparation and Isolation of $[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]^{13-}$

$$P-O_a = 2.692-2.727 \text{ \AA}$$

$$W-O_b-W = 2.834-2.941 \text{ \AA}$$

Preparation and Isolation of $[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]^{13-}$

Redox Potential

Thermal Stability

a) Potassium salts

b) Acidic form

Acid Catalyst Activity

Catalyst	Conv. [%]	Rate	
		Per weight [mmol g ⁻¹ min ⁻¹]	Per acid amount [mmol (acid·mol) ⁻¹ min ⁻¹]
H ₁₄ [P ₅ W ₃₀ O ₁₁₀ Na] ^b	46.2	275.7	164.0
H ₁₃ [P ₅ W ₃₀ O ₁₁₀ Ca] ^b	48.2	257.0	171.3
H ₁₂ [P ₅ W ₃₀ O ₁₁₀ Eu] ^b	50.2	258.5	178.5
H ₁₂ [P ₅ W ₃₀ O ₁₁₀ Bi] ^b	51.2	254.9	182.1
H ₁₃ [P ₅ W ₃₀ O ₁₁₀ K ₂]	55.1	304.8	195.9
H ₃ PW ₁₂ O ₄₀ ^b	49.1	175.2	174.5
Blank ^b	1.4		

HAADF STEM Image

Preyssler molecule
arrangement under
HAADF STEM
observation

Side Product Analysis

Preyssler Type

Degradation

Keggin Type

- The first Preyssler type phosphotungstate with two encapsulated cations, $[\text{P}_5\text{W}_{30}\text{O}_{110}\text{K}_2]^{13-}$ was prepared as a potassium salt and acid form and was characterized.
- The potassium salt shows higher thermal stability than that of other Preyssler type phosphotungstate
- The acid form shows catalytic activity similar to other phosphotungstates for hydration of ethyl acetate.