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**Synthesis, Crystal Structure and Electrochemical Properties
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 $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}].(\text{CH}_5\text{N}).14\text{H}_2\text{O}$**

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Synthesis, Crystal Structure and Electrochemical Properties a new methylammonium sodium decavanate salt $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}].(\text{CH}_5\text{N}).14\text{H}_2\text{O}$

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abstract

A new hybrid decavanadate $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}].(\text{CH}_5\text{N}).14\text{H}_2\text{O}$ was obtained by a simple synthesis in solution and the crystal structure was determined by single-crystal X-ray diffraction. The compound consists of decavanadate anions $[\text{V}_{10}\text{O}_{28}]^{6-}$ isolated between sodium and methylammonium cations. The UV–Visible absorption, IR spectroscopy as well as electrochemical properties were characterized. The compound crystallizes in a triclinic system (space group $P-1$). The unit cell are: $a = 10.1584(11)$ Å, $b = 10.8123(5)$ Å, $c = 11.1582(7)$ Å, $\alpha = 107.723(4)^\circ$, $\beta = 106.811(5)^\circ$, $\gamma = 94.238(7)^\circ$. UV–Visible absorption spectroscopy shows a strong absorption band at 300nm. The main IR absorption bands appear at: 954 cm^{-1} , 800 cm^{-1} , 732 cm^{-1} and 580 cm^{-1} .

The electrochemical measurement of this compound shows two reversible one-electron redox process -105 mV ($\text{I-I}'$) and 335mV ($\text{II-II}'$), which was attributed to two consecutive one-electron $\text{V}^{\text{V}} \rightarrow \text{V}^{\text{IV}}$ reductions.

1. Introduction

Polyoxometalates (POMs) are inorganic oligomeric metal-oxo clusters, the simplest of which contain ions of a single transition metal in its highest oxidation state (e.g., Mo^{6+} , V^{5+} , W^{6+}) connected via bridging oxo ligands. In recent years, POMs have attracted increasing attention due to their potential applications as smart windows, cosmetics, multicolored photochromic pigments, UV sensors, optical switches, or for magnetism [1–9]. Among them, polyoxovanadates are of great interest for their electronic and optical properties [10]. However, the main challenge remains the rational design and preparation of novel organic–inorganic hybrid oxovanadate [11]. Within the large family of Lindqvist polyoxovanadates type anions, the decavanadate $[\text{V}_{10}\text{O}_{28}]^{6-}$ is one of the most common owing to the stability of the $\text{V}^{(\text{V})}$ oxo-clusters in the acidic solution [12]. This anion, discovered by Rosotti et al., [13] is recognized as a chemical specie of high biological interest [14–17].

In the past years, special attention has been paid in identifying the conditions for the assembling of precursors leading to decavanadate clusters. For example, some authors described of how the nature, size, and shape of the cation(s) and solvent(s), as well as the degree and site(s) of protonation(s) of decavanadate, are essential for forming a fascinating variety of supramolecular assemblies [18–21].

In this context, we investigated on oxovanadates POMs with mixed cations and successfully isolated $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}].(\text{CH}_5\text{N}).14\text{H}_2\text{O}$. As such compounds may have excellent electrocatalytic properties and high stability [22–24], we have investigated the electrochemical properties of this material. The investigation of electrochemical properties shows two consecutive one-electron $\text{V}^{\text{V}} \rightarrow \text{V}^{\text{IV}}$ reductions.

2. Experimental section

2.1. Materials

All chemicals (sodium metavanadate (85%), methylamine (40%) and hydrochloric acid (37%)) were purchased from Sigma-Aldrich and used without further purification. Distilled water was used as solvent.

2.2. Synthesis

$\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}].(\text{CH}_5\text{N}).14\text{H}_2\text{O}$ was obtained from a one pot synthesis. Sodium metavanadate (48mmol, 0.585g) was dissolved in a solution of methylamine (48mmol, 0.372g) and distilled water (30mL). Hydrochloric acid (48mmol, 0.397g) was added to acidify and adjust the pH solutions to 6. The solution was heated under reflux and agitation for two hours. Then, the solution was cooled to room temperature and filtered. After 2 weeks of slow evaporation at room temperature, pink color crystals suitable for single-crystal X-ray diffraction were obtained.

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2.3. Structure determination

Single-crystal X-ray diffraction data were collected with a Bruker-Nonius Kappa CCD diffractometer (MoK α radiation $\lambda = 0.71073$ Å). The crystal to detector distance was 60 mm. Absorption corrections were considered using SADABS [25]. The determination was carried out using direct methods [26]. The crystal structures including the anisotropic displacement parameters were refined with SHELXL-2013 [27]. PLATON [28] was used to check additional symmetry elements and Crystallographic Information Files were compiled with Olex2.12. Crystallographic data are summarized in Table 1.

Table 1: Crystallographic data of the compound

Empirical formula	Na ₃ (CH ₆ N) ₃ [V ₁₀ O ₂₈].14H ₂ O
Formula weight	1406.82
Crystal system	Triclinic
a(Å)	10.1584 (11)
b(Å)	10.8123 (5)
c(Å)	11.1582 (7)
α (°)	107.723 (4)
β (°)	106.811 (5)
γ (°)	94.238 (7)
V (Å ³)	1099.75 (15)
Space group	P1
Z	1
D _x (Mg.m ⁻³)	2.045
F(000)	490
λ (Å)	0.71073
θ (°) range	6,4 ≤ θ ≤ 25
T(K)	293
Absorption coefficient (mm ⁻¹)	2.17
hkl range	-12 ≤ h ≤ 12, -12 ≤ k ≤ 12, -13 ≤ l ≤ 13
Final R indices	R1=0,065 ; wR2=0,166
Reflections collected	24582
Independent reflections	3799[R(int) = 0.088]
Largest diff. peak/hole (e Å ⁻³)	-0,58/2,54

2.4. IR and UV Spectroscopy

The UV-Visible absorption spectra were recorded using a Thermo Scientific Genesis 10S UV-Vis spectrophotometer at room temperature in aqueous solution. 15 mg was solubilized in 10ml of distilled water. A scan between 200 nm and 700 nm was carried out and distilled water was used as blank.

IR spectroscopy measurements were carried out for the decavanadate based compound using ATR (Attenuated Total Reflectance) method from 4000 to 400 cm⁻¹ [29].

2.5 Cyclic voltammetry measurements

The electrochemical properties were investigated in aqueous acidic medium (2 M H₂SO₄) with a scan rate of 20 mV/s. The electrochemical measurements with different scanning rate from 5mV/s to 100 mV/s were performed in the same conditions.

3. Results and discussion

3.1. Description of the crystal structure

The structure determination from single-crystal X-ray diffraction reveals that the material crystallizes in the space group *P*-1. The asymmetric unit

contains, V₅O₁₄N(H₂O)₃ group, two methylammonium cations and one Na⁺ cation. The new crystal structure consists in [V₁₀O₂₈]⁶⁻ decavanadate anions (Figure 1a) stabilized by methylammonium and sodium cations. The [V₁₀O₂₈]⁶⁻ anions are interconnected through two Na(H₂O)₄⁺ (Figure 1b). The ion [V₁₀O₂₈]⁶⁻ is counterbalanced by three CH₃NH₃⁺ and three Na⁺. Interestingly, both protonated and non-protonated amines (CH₃NH₃⁺ and CH₃NH₂) are present in the network. This phenomenon was also observed in our previously reported phosphomolybdate based compound [30] and in decavanadate/chitosan complex reported by Zhetcheva *et al.*, where both protonated and non protonated amine groups are present [31]. The polyanion [V₁₀O₂₈]⁶⁻ is formed of ten distorted, edge-sharing octahedral VO₆ units arranged in a 2x3 rectangular array [32, 33].

Five categories of V-O bonds can be distinguished: (a) V-O_t, with O_t = terminal oxygen atoms (bond lengths range 1.597(6) – 1.601 (6)Å); (b) V-Ob1, with Ob1 = oxygen atoms shared by two vanadium atoms (bond lengths range = 1.672(6) – 2.084(6)Å) ; (c) V-Ob2, with Ob2 = oxygen atoms shared by three vanadium atoms, (bond lengths range = 1.909(5) – 2.020(6)Å) ,(d) V-Ob3, with Ob3 = oxygen atoms shared by six vanadium atoms (bond lengths range = 2.078(5) – 2.334(5)Å).

The bond lengths and angles associated with the Na ions are in the ranges of 2.401 Å – 2.520Å (Na–O) and 75.8(2)° – 170.0(3)° (O–Na–O) in agreement with the ones observed for other Na-containing POMs [34].

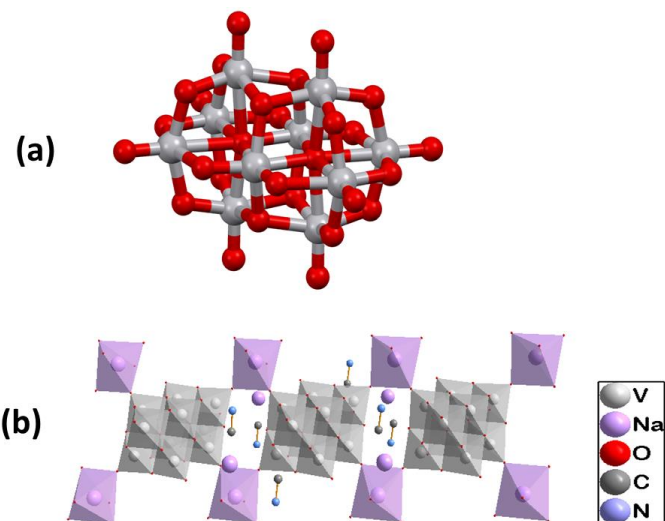


Figure 1. (a) View of decavanadate anion; (b) View of the extended crystal structure. All the hydrogen atoms are omitted for clarity.

Materials such as (NH₄)₆[V₁₀O₂₈].6H₂O [35], Cs₄[H₂V₁₀O₂₈].4H₂O [36], Na₆[V₁₀O₂₈].18H₂O [37], Na₆[V₁₀O₂₈].12H₂O [37] and Li₆(H₂O)₁₆[V₁₀O₂₈] [38] containing the decavanadate anion [V₁₀O₂₈]⁶⁻ with monovalent counterions or with mixed monovalent counterions such as (NH₄)₄Na₂[V₁₀O₂₈].10H₂O [39], or K₄Na₂[V₁₀O₂₈].10H₂O [40] have also been previously reported. In those materials, the decavanadate anion acts as an excellent acceptor of protons of organic molecules such as amines [41-44].

3.2. UV

The UV–Visible absorption of the $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}](\text{CH}_5\text{N})\cdot 14\text{H}_2\text{O}$ compound was carried out in the range of 200–700 nm. One absorption band at 300 nm is observed and can be attributed to the Ligand-to-Metal Charge Transfer (LMCT) $\text{O} \rightarrow \text{V}$ transition (Figure 2) [45, 46].

3.3. IR spectroscopy

In IR-spectra of the compound displayed in Figure S1, the spectra can be divided into two regions. The first region between 4000–1500 cm^{-1} is attributed to the vibration of water molecules and organic ligands and the second region between 1000–400 cm^{-1} contain bands attributed to the decavanadate cluster vibration. Thus, the characteristic bands at 954 cm^{-1} , 800 cm^{-1} , 732 cm^{-1} and 580 cm^{-1} can be attributed to the $\text{V}=\text{O}_t$ stretch or bridging and $\text{V}-\text{O}-\text{V}$ stretch [45, 47]. The broad band observed around 3350 cm^{-1} can be assigned to characteristic stretching peaks vibrations $\nu(\text{O}-\text{H})$ of water molecules [48]. The bands between 3133 cm^{-1} and 2457 cm^{-1} are respectively assigned to characteristic peaks vibrations $\text{N}-\text{H}$ and $\text{C}-\text{H}$ of CH_3NH_3^+ [33, 49]. Vibrations bands between 1650–1350 cm^{-1} are associated to $\text{O}-\text{H}$ bending of water molecules, $\text{N}-\text{H}$ and $\text{C}-\text{H}$ bending and $\text{C}-\text{N}$ stretching vibrations of the organic part [33, 50].

3.4. Electrochemical properties

In order to investigate the electrochemical response of our compound, the cyclic voltammetry behavior was measured in 2 M H_2SO_4 solution containing 1 mmol of the compound (Figure 3a). In the potential range from -1.0 V to $+1.0$ V, two reversible redox peaks are observed and the mean peak potentials (1) are -105 mV (I-I') and 335 mV (II-II').

$$E_{1/2} = (E_{pa} + E_{pc})/2 \quad (1)$$

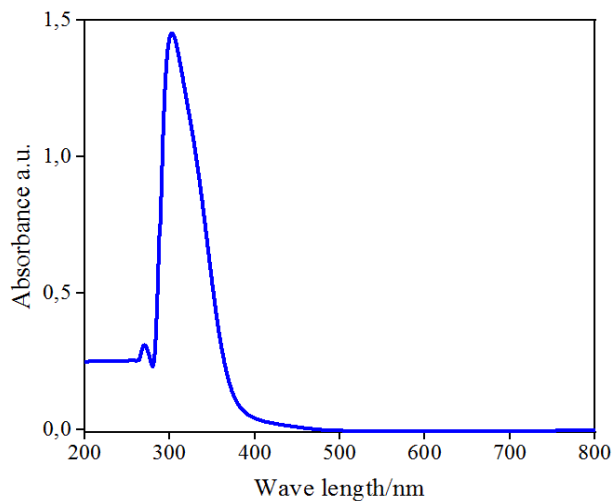


Figure 2. UV–Visible absorption of the $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}](\text{CH}_5\text{N})\cdot 14\text{H}_2\text{O}$ compound.

(II-II') which are ascribed to two consecutive one-electron vanadium-centered $\text{V}^V \rightarrow \text{V}^{IV}$ redox process [51,52].

The cyclic voltammetry (CV) curves of the glassy carbon electrodes of the compound, obtained at different scan rates ranging from 5 to 100 mV/s is represented on Figure 3b. The peak currents densities and the peak potential difference values of these oxidation/reduction processes on the electrode, increase with the scan rate, indicating these electrode processes are irreversible. Furthermore, the plot of the peak current density versus the square root of scan rate shows good linearity (Figure 4). When the square root of the scan varied from 0.1 to 1 (V/s)^{1/2}, the peak potentials change gradually. Therefore, the cathodic peak potentials shift toward negative values and the corresponding anodic peak potentials to positive values with increasing scan rates. This behaviour indicates that the redox reaction on the surface of glassy carbon electrode is a process controlled by diffusion.

The regression equations of the anodic (J_a) and cathodic (J_c) peak currents with respect to the square root of the scan rate are expressed as:

$$J_I = (0.053 \pm 0.0004) v^{1/2} - (0.045 \pm 0.003) \quad \text{with } R^2 = 0.999 \quad (2)$$

$$J_{II} = (0.072 \pm 0.001) v^{1/2} - (0.052 \pm 0.007) \quad \text{with } R^2 = 0.998 \quad (3)$$

$$J_{I'} = (-0.173 \pm 0.007) v^{1/2} + (0.067 \pm 0.044) \quad \text{with } R^2 = 0.989 \quad (4)$$

$$J_{II'} = (-0.087 \pm 0.004) v^{1/2} + (0.084 \pm 0.026) \quad \text{with } R^2 = 0.985 \quad (5)$$

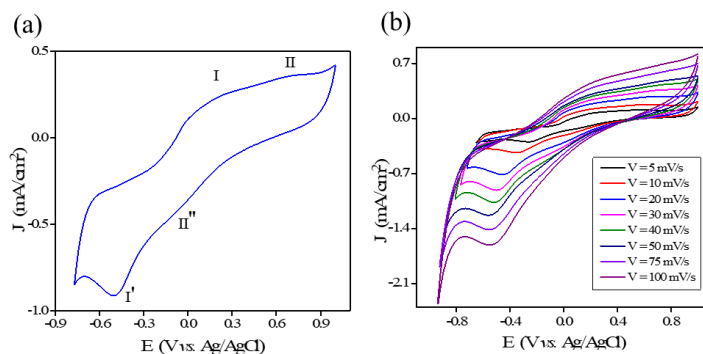


Figure 3. Electrochemical measurements of $\text{Na}_3(\text{CH}_6\text{N})_3[\text{V}_{10}\text{O}_{28}](\text{CH}_5\text{N})\cdot 14\text{H}_2\text{O}$: (a) Cyclic voltammogram in 2 M H_2SO_4 at scan rate = 20 mV/s, (b) Cyclic voltammograms in 2 M H_2SO_4 with different scan rates.

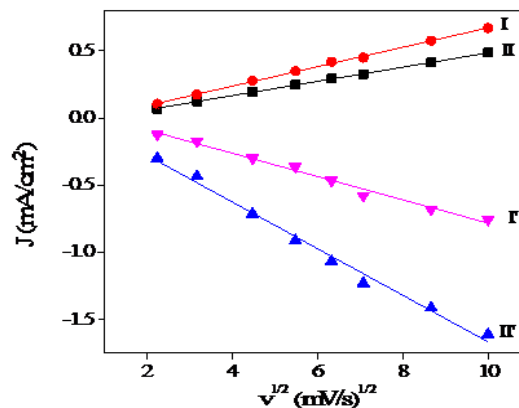


Figure 4. Calibration curve for peak currents with respect to the square root of the scanning speed.

4. Conclusion

In summary, a new decavanadate hybrid compound has been synthesized and structurally characterized. In this crystal structure, the $[V_{10}O_{28}]^{6-}$ decavanadate anions are connected through $[Na(H_2O)_4]^+$ cations. The electrochemical properties of compounds were successfully investigated and show two reversible consecutive one-electron redox process. The square root of scan rate indicates a process controlled by diffusion. We believe this material will inspire the introduction of mixed organic-inorganic counterions for the synthesis of new functional decavanadates.

5. Abbreviation

UV = Ultraviolet; IR = Infrared; CCD = Charge Coupled Device; POMs = Polyoxometalates; ATR = Attenuated Total Reflectance; Epa = Anodic peak potential; Epc = Cathodic peak potential; CV = cyclic voltammetry; LMCT = Ligand-to-Metal Charge Transfer;

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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Appendix A. Supplementary data

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