






Research Article

# Synthesis, Structural Characterization, and Photochromic Behavior of a New Hybrid Diphosphomolybdate Compound

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## Abstract

The integration of organic species with polyoxometalates leads to the formation of hybrid materials that benefit from the combined functionalities of both components. These organo-inorganic systems are of great interest due to their potential synergistic behavior. Developing straightforward and efficient synthetic approaches to design such materials combining the rigidity of the inorganic backbone and the tunability of organic units remains a key objective in the field of materials chemistry. In this work, we present a simple "one-pot" synthesis route for the compound  $(C_6H_{20}N_3)_2[P_2Mo_5O_{23}] \cdot 1.5H_2O$ . Structural characterization was performed using single-crystal X-ray diffraction alongside infrared and UV-Visible spectroscopy. The compound crystallizes in the monoclinic system, space group  $P2_1/c$ , with the following unit cell parameters:  $a = 18.9626(2) \text{ \AA}$ ,  $b = 10.8514(1) \text{ \AA}$ ,  $c = 16.4933(1) \text{ \AA}$ ,  $\beta = 107.37(1)^\circ$ , and  $Z = 4$ . Its structure is based on a diphosphomolybdate anion  $[P_2Mo_5O_{23}]^{6-}$ , neutralized by two organic cations  $(C_6H_{20}N_3)^{3+}$ , and accompanied by 1.5 lattice water molecules. The three-dimensional arrangement is characterized by layered assemblies oriented along the  $a$ -axis, stabilized through extensive hydrogen bonding. These layers alternate between polyanionic clusters, organic moieties, and water molecules, forming a robust supramolecular network. Notably, this material displays photochromic behavior, suggesting its potential for applications in responsive optical systems.

## Keywords

Diphosphomolybdate, Polyoxometalate, Strandberg-type, Hybrid, Hydrogen Bonds, Organic Counterions, Photochromic, Single X-ray Diffraction

## 1. Introduction

Supramolecular assemblies based on polyoxometalates (POMs) particularly on phosphomolybdates (POMos) cluster anions are captivating organic-inorganic hybrid solids on

account of their multitudinous structures and fascinating properties applied in various fields such as catalysis, magnetism, ion-exchange, electrochemistry and nano-science

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[1-11].

Among the POMos cluster anions, Strandberg-type with general formula  $[H_nP_2Mo_5O_{23}]^{(6-n)-}$  ( $n=0, 1, 2$ ), (abbreviated as  $\{P_2Mo_5\}$  henceforth) is the most stable cluster anion that can be crystallized under ambient conditions [12-14].

Among the various types of building blocks, Strandberg-type fragments offer several intrinsic advantages, including their nanometric dimensions, oxygen-rich nucleophilic surfaces, and multiple coordination sites. These features make them suitable candidates for functionalization with organic moieties.

via a controllable synthesis, leading to crystallized hybrid architectures gaining much attention because of the combination of organic and inorganic components which provide new multi-functional hybrid materials. [15-27].

Furthermore, hitherto, the synthetic methods of Strandberg-type POMos and hybrids are mostly relied on hydrothermal synthesis method [15-24]. The challenge is to exploit a simpler, less energy-intensive synthesis method assembling  $\{P_2Mo_5\}$  precursor with organoamine groups leading to crystallized hybrid architectures in which the organic-inorganic interface is established via hydrogen bonding networks, this synergistic effect may lead to unique properties of the resulting compound.

In this paper, we focus on structural description and electrochemical properties new organic-inorganic hybrid diphosphopentamolybdates  $(C_6H_{20}N_3)_2[P_2Mo_5O_{23}].1.5 H_2O$  building block.

This research work offers a unique opportunity to optimize electrochemical performance, thus opening exciting new perspectives in key sectors such as electrochemistry, catalysis, and energy storage. We report a convenient synthesis method used to obtain this hybrid phosphomolybdate, as well as the characterization techniques used to thoroughly explore its structure and properties.

## 2. Materials and Methods

Ammonium molybdate (98%) and phosphoric acid (98%), 3,3'-Diaminodipropylamine (99%) were purchased from Sigma-Aldrich and used without purification with distilled water.

IR spectroscopy measurements were carried out for the compounds. IR measurements were performed using ATR (Attenuated Total Reflectance) method from 4000 to  $400\text{cm}^{-1}$  [28].

The UV-visible absorption measurements of the compound were recorded using a Thermo Scientific GENESYS 10S UV-Vis spectrophotometer at room temperature in acidic aqueous solution ( $H_2SO_4$ ). Thus 15 mg of the compound was solubilized in 10ml of sulfuric acid aqueous solution (2M) and a scan between 200 nm and 1100 nm was carried. Sulfuric acid was used as blank.

Photochromic properties test of the compounds was evaluated at room temperature with a UV-lamp (254 nm /365nm;

2X6W UVGL-58).

A single-crystal X-ray diffraction data for the compound were measured on a Rigaku Synergy-S diffractometer (Cu K $\alpha$  radiation,  $\lambda = 1.54184 \text{ \AA}$ ). Data collection reduction and multi-scan ABSPACK correction were performed with CrysAlisPro (Rigaku). The crystal including the anisotropic displacement parameters were refined with CRYSTALS. [29-32]. Crystallographic Information Files were compiled Crystals 15.4.1. Crystallographic data are summarized in Table 1.

## 3. Synthesis

The compound  $(C_6H_{20}N_3)_2[P_2Mo_5O_{23}].1.5 H_2O$  was obtained using one pot synthesis process. In 40 ml of distilled water, were mixing 5 mmol of ammonium molybdate and 5 mmol of 3,3'-Diaminodipropylamine. After stirring for thirty minutes, the pH of the solution was adjusted to 6.4 by addition of the phosphoric acid. The solution was stirred under room temperature for two hours to obtain colorless solution. After a few days of slow evaporation of the final solution, colorless crystals suitable for X-ray diffraction were obtained without any purification.

## 4. Results and Discussion

### 4.1. Spectroscopic Analysis

The UV-Visible absorption of the compound was analyzed in the range of 200–1100 nm in acidic solution. In this region, the UV spectra revealed a strong absorption band centered at 221nm and a shoulder at 264nm (Figure 1). These absorptions bands can be ascribed to Ligand-Metal Charge Transfer (LMCT) transition of the  $Ot \rightarrow Mo$  and  $(\mu-O) \rightarrow Mo$  bands respectively [33, 34].

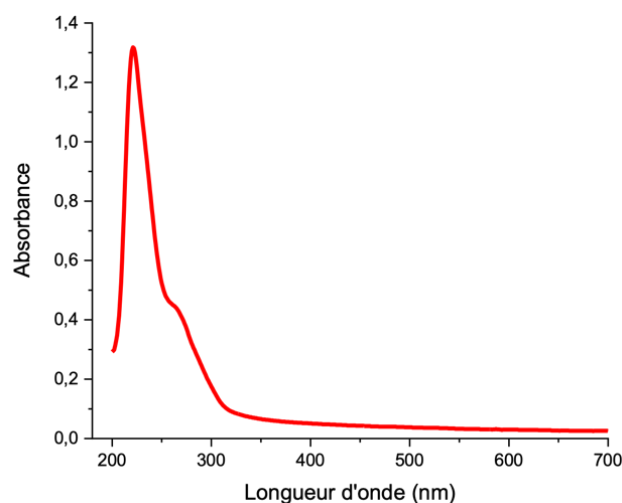


Figure 1. UV visible absorption spectrum of the compound.

Indeed, during the charge transfer process, electrons are promoted from low-energy electronic states, mainly consisting of 2p orbitals of oxygen, to high-energy states, which mainly consist of d metal orbitals of molybdenum. Then, both compounds absorb in the ultraviolet region [35].

The IR-spectrum of the compound shown in Figure 2 can be divided into two regions. The first region ranging from 4000 to 1100  $\text{cm}^{-1}$  is attributed to the vibrations of water molecules and organic ammoniums groups. The second region from 1100 to 400  $\text{cm}^{-1}$  contains bands attributed to the polyanions vibrations.

Thus, the characteristic bands centered at 972, 807, 648, 530 and 1031  $\text{cm}^{-1}$  correspond to the  $\nu(\text{Mo-Ot})$ ,  $\nu(\text{Mo-O-Mo})$  and  $\nu(\text{P-O})$  characteristic vibration bands of compound [36, 37].

The range 3600–2500  $\text{cm}^{-1}$  shows many bands ascribed to O–H stretching vibration of water molecules, as well as N–H and C–H stretching vibrations of organic groups.

On the IR spectra, bands between 1650 and 1300  $\text{cm}^{-1}$  are associated to O–H bending of water molecules, and N–H and C–H bending vibrations of the organic moiety.

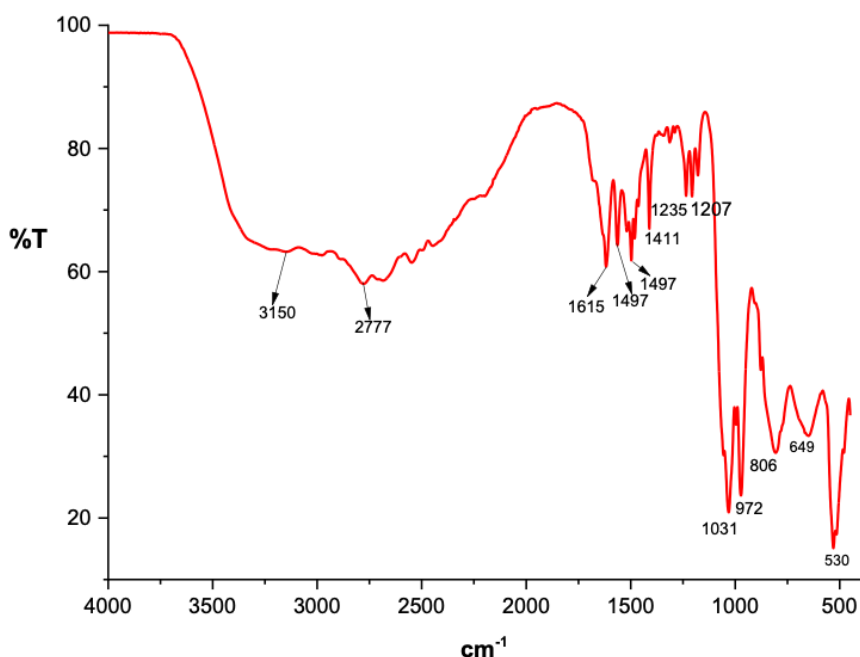


Figure 2. IR spectrum of the compound.

## 4.2. Crystallographic Study

The crystallographic data was collected using X-ray diffraction analysis on a single crystal. The compound crystallizes in monoclinic system with the space group  $P2_1/c$ .

Table 1. Crystal data and structure refinement for the compound.

Column 1	Column 2
Molecular formula	$C_{12}H_{43}N_6Mo_5N_6O_{24.50}P_2$
Formula weight ( $\text{g}\cdot\text{mol}^{-1}$ )	1205.15
Crystal color, Shape	Colorless, Block
Crystal system	Monoclinic
$a(\text{\AA})$	18.9626 (2)

Column 1	Column 2
$b(\text{\AA})$	10.8514 (1)
$c(\text{\AA})$	16.4933 (1)
$\alpha(\text{deg})$	90
$\beta(\text{deg})$	107.37 (1)
$\gamma(\text{deg})$	90
$V(\text{\AA}^3)$	3239.07 (18)
Space group	$P2_1/c$
Radiation type	Cu $K\alpha$
Crystal size (mm)	$0.03 \times 0.05 \times 0.07$
Tmin, Tmax	0.40/0.59
Z	4
F(000)	2372

Column 1	Column 2
$\theta$ range, deg	5–80
Absorption coefficient, mm <sup>-1</sup>	17.333
T(K)	100
$\lambda$ (Å)	1.54180
h	-23 → 24
k	-13 → 13
l	-19 → 21
Reflections collected	66180
Independent reflections	7035 [Rint = 0.0317]
Final R indices, $I > 2\sigma(I)$	R1 = 0.017, wR2 = 0.044
GOF on F2	1.049
Peak, hole/eÅ <sup>-3</sup>	$\Delta\rho_{\min} = -0.55$ , $\Delta\rho_{\max} = 0.50$

The asymmetric unit is composed of one diphosphomolybdate polyanion, two 3,3'-Diaminodipropylammonium cations and two water molecules (Figure 3).

The polyanion of the compound is built with non-protonated diphosphopentamolybdate the Strandberg type anion  $[P_2Mo_5O_{23}]^{6-}$ . The polyhedral description of the polyanion  $[P_2Mo_5O_{23}]^{6-}$  consists of a ring of five distorted  $MoO_6$  octahedra with two  $PO_4$  tetrahedra capping on each side. The octahedra form a pentagonal ring by sharing four edges and one corner. Each phosphate subunit shares three oxo-groups with the molybdate ring (Figure 4).

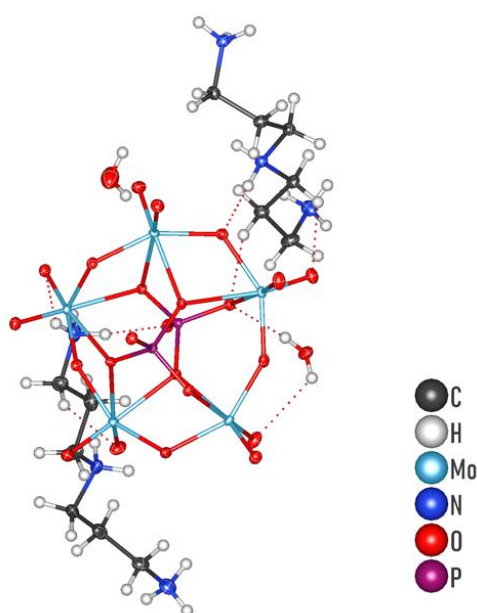


Figure 3. Asymmetric units of the compound.

In the polyanion, bond-valence sum calculations yield average for all Mo atoms to (+6) and all O atoms are in the (-2) oxidation state. Structural cohesion in the polyanion  $[P_2Mo_5O_{23}]^{6-}$  is ensured by strong interactions between the metal, phosphorus and oxygen atoms. Its result from these interactions four categories of Mo–O bonds in the polyanion: (a) ten Mo–O<sub>t</sub> bonds (O<sub>t</sub> = terminal oxygen) with bond lengths range of 1.7046 (16)–1.7401 (16) Å; (b) five Mo–(μ<sub>1</sub>-O) (μ<sub>1</sub>-O = oxygen atoms bridging two molybdenum atoms) with bond lengths range of 1.8874 (16)–1.9587 (16) Å; (c) Mo–(μ<sub>2</sub>-O), (μ<sub>2</sub>-O = oxygen atoms shared by two molybdenum and one phosphorus atoms), with bond lengths between 2.2028 (15) to 2.2293 (16) Å; (d) Mo–(μ<sub>3</sub>-O), (μ<sub>3</sub>-O = oxygen atoms bridging one molybdenum and one phosphorus atoms) with bond lengths range of 2.1835 (16)–2.4053 (15) Å (Figure 4). [38-42] For the phosphorus atoms, P–O<sub>t</sub> bond lengths involving a phosphorus and a terminal oxygen vary between 1.5174 (16) and 1.5299 (16) Å, P–(μ<sub>2</sub>-O) bond lengths vary between 1.5237 (16) and 1.5307 (16) Å and P–(μ<sub>3</sub>-O), bond lengths vary between 1.5554 (16) Å and 1.5628 (16) Å.

In the polyanion, the bonds around molybdenum vary between 1.7046 (17)Å (Mo<sub>1</sub>–O<sub>29</sub>) and 2.4053 (15) Å (Mo<sub>15</sub>–O<sub>14</sub>) and the O–Mo–O angles vary between 68.99 (6)° (O<sub>9</sub>–Mo<sub>1</sub>–O<sub>22</sub>) and 172.85 (7)° (O<sub>2</sub>–Mo<sub>1</sub>–O<sub>30</sub>) whereas for phosphorus atoms, bond lengths vary between 1.5174 (16)Å (P<sub>8</sub>–O<sub>25</sub>) and 1.5628 (16)Å (P<sub>8</sub>–O<sub>9</sub>) and the O–P–O angles between 105.83 (9)° (O<sub>7</sub>–P<sub>8</sub>–O<sub>9</sub>) and 111.44 (9)° (O<sub>9</sub>–P<sub>8</sub>–O<sub>25</sub>).

These variations lead to irregularity and deformation of the  $[MoO_6]$  octahedra and  $[PO_4]$  tetrahedra in the polyanion which is in perfect agreement with the literature of Strandberg type POMs [33].

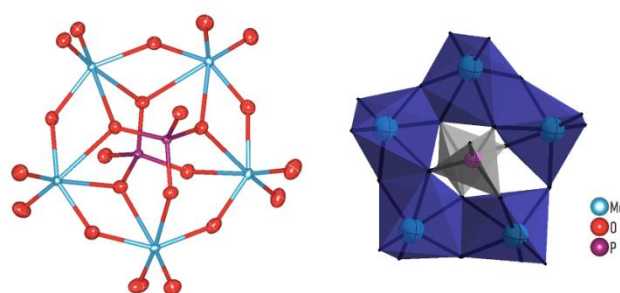


Figure 4. Structures of polyanion. (a): sticks representation and (b) polyhedral representation.

In the work of Yaffa et al, it was demonstrated that bulky amine groups such as diamines favor the formation of non-protonated polyanions, while smaller groups such as monoamines promote the formation of protonated polyanions. [43]. Indeed, the stabilization of a non-protonated polyanion of the Strandberg type, requires three organic diammonium or six organic monoammonium molecules to counterbalance

the charge. Six organic monoammonium molecules around the anion would lead to strong steric effects. To prevent such steric effects, the non-protonated anion must be stabilized with diammonium cations. On the other hand, monoammonium cations can stabilize protonated anions [43].

Accordingly, the 3,3'-diaminodipropylammonium cations, being a triamine, promotes the formation of a non-protonated anion, thus supporting the findings reported by Yaffa et al.

In compound  $(C_6H_{20}N_3)_2[P_2Mo_5O_{23}] \cdot 1.5 H_2O$ , the constituents are involved in two types of hydrogen bonds. Each polyanion  $[P_2Mo_5O_{23}]^{6-}$  is connected to its neighbors through  $O_w-H \cdots (O)$  and  $N-H \cdots (O)$  interactions.

Water molecules act as hydrogen donors, interacting with the  $[P_2Mo_5O_{23}]^{6-}$  anions via  $O_w-H \cdots O$  bonds and with organic counterions through  $N-H \cdots O$  interactions.

All species such as the polyanion  $[P_2Mo_5O_{23}]^{6-}$ , the  $(C_6H_{20}N_3)^{3+}$  cation and water molecule ( $H_2O$ ) participate in these interactions, leading to the formation of one-dimensional chains.

These chains are further interconnected through the same hydrogen-bonding motifs among the cations, water molecules and polyanion units, resulting in a three-dimensional network (Figure 5) [40, 44-46].

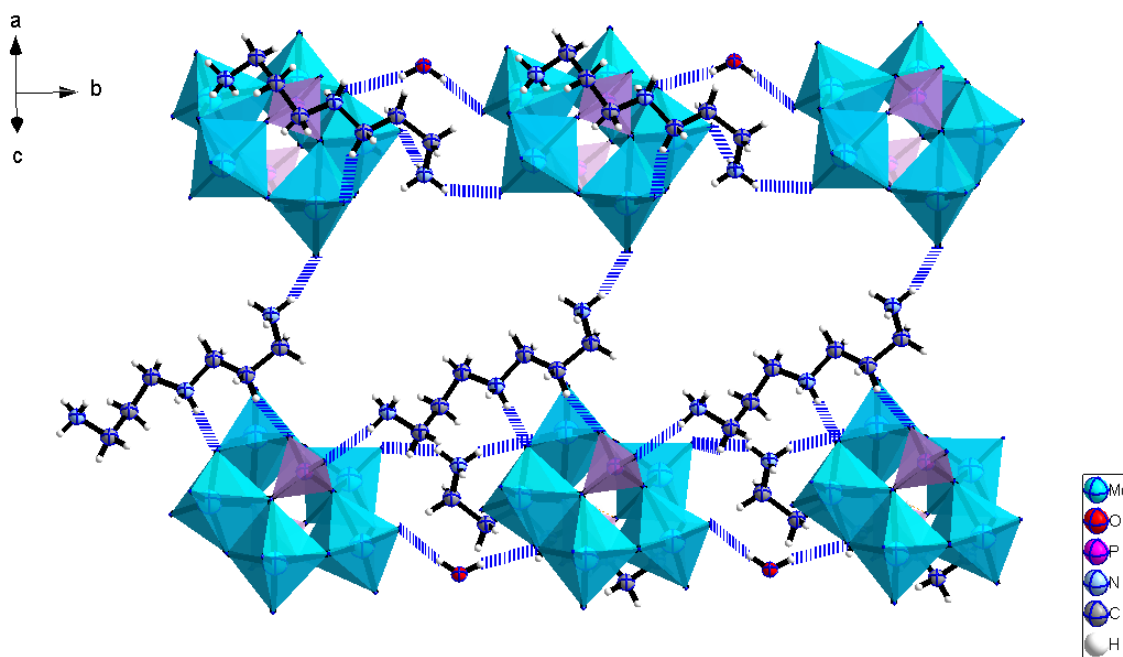


Figure 5. Three-dimensional representation of the compound.

### 4.3. Photochromic Properties Test

The Photochromic property characterizes the ability of a material to reversibly change color under electromagnetic irradiation effect in the ultraviolet and visible areas.

Thus, the photochromic behavior of the compound was demonstrated under a UV-Lamp with excitation at a wavelength of 254 nm (4.89 eV).

All the crystals material shows a colorless coloration in their ground state. Under UV excitation at 365 nm (3.4 eV),

the compound has no photochromic response after thirty-minutes of irradiation. There is no change in color of the compound. However, the compound has photochromic response after twenty minutes of irradiation under UV excitation at 254 nm (4.89 eV).

After thirty-minutes of UV excitation under the lamp, the compound's color changed from orange to green color and the material showed strong photochromic response with high coloration, as contrast and the photoinduced color become increasingly intense with time (Figure 6).

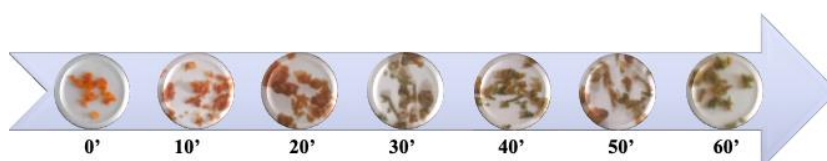


Figure 6. Photochromic behavior.

The photo-induced color did not change after thirty minutes of irradiation.

However, the return to the original color requires a long time at room temperature (i.e. 2 days) or 5min at 100 °C.

Based on the literature, the coloration is due to the photo-reduction of central metals. This reduction is generated via this process during the absorption of a photon, the homolytic breaking of the N-H bond of the organic groups, leading to the hydrogen atoms the transfer to the terminal oxygen ligand and of the polyanion [38, 48].

The oxygen atoms transfer their electrons to the central metal, the  $\text{Mo}^{\text{VI}}$  which are reduced to  $\text{Mo}^{\text{V}}$ . The  $\text{Mo}^{\text{V}}$  cations with a  $d^1$  electronic configuration may then be involved in d-d transitions or intervalence  $\text{Mo}^{\text{V}} + \text{Mo}^{\text{VI}} \rightarrow \text{Mo}^{\text{VI}} + \text{Mo}^{\text{V}}$  charge transfer inducing new colors [46-48].

## 5. Conclusion

In summary, the novel Strandberg-type POM complex functionalized with 3,3'-diaminodipropylammonium groups was successfully synthesized under mild condition using a one-step procedure in distilled water.

This POM was characterized in the solid state by single crystal X-ray diffraction, and in solution by UV-Visible and IR spectroscopies.

The compound exhibits a 3D supramolecular structure, with hydrogen-bonding networks involving the  $[\text{P}_2\text{Mo}_5\text{O}_{23}]^{6-}$  anions, organoamines groups and water molecules. These networks are quite similar and significantly influence the compound's photochromic behavior. The photochromic properties tests show that the compound exhibits a remarkable and rapid color change from orange to green under UV irradiation at 4.89 eV.

To date, these are among the most efficient photochromic organoammonium/POM systems known.

The outstanding photochromic performances in the solid state under ambient conditions is correlated to the numerous of  $\text{Ow}-\text{H}\cdots(\text{O})$  and  $\text{N}-\text{H}\cdots(\text{O})$  hydrogen-bonding interactions involving the oxygen atoms of the diphosphomolybdate units, the protonated amino groups, and water molecules present in the structure.

## Abbreviations

UV	Ultraviolet
POMs	Polyoxometalates
IR	Infrared
XRD	X-ray Diffraction
Ow	Oxygen of Water Molecule

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## Author Contributions

**Aboubacar Soumano:** Methodology, Resources, Writing – original draft

**Lamine Yaffa:** Conceptualization, Supervision, Writing – review & editing

**Dame Seye:** Validation, Writing – review & editing

**Assane Toure:** Validation, Writing – review & editing

**Carine Duhayon:** Formal Analysis, Resources

**Cheikh Abdoul Khadir Diop:** Project administration, Resources

**Mamadou Sidibe:** Project administration, Resources

## Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

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## Conflicts of Interest

The authors declare no conflicts of interest.

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## Biography



**Aboubacar Soumano** is a physics and chemistry teacher in secondary education at high school Mame Yelli Badiane at Pikine Guédiawaye district of Dakar. He is also a PhD student at the Inorganic and Analytical Chemistry Laboratory in the Chemistry Department of the Faculty of Sciences and Techniques at Cheikh Anta Diop University in Dakar, where he works on polyoxomolybdates. In 2017, he defended his master's thesis on the study of some phosphorated ligand complex.



**Lamine Yaffa** earned his Ph. D. in Molecular Chemistry from Cheikh Anta Diop University in Dakar in 2022. His research focuses primarily on polyoxometalates and polynuclear complexes, with several peer-reviewed publications contributing to advancements in these fields. He first

gained recognition in 2017 for his master's thesis on an antimalarial iron complex. Currently a researcher at the Laboratory of Inorganic and Analytical Chemistry (Laboratoire de Chimie Minérale et Analytique), Dr. Yaffa is also engaged in the physicochemical study of clay sands, aiming to develop novel applications and deepen the understanding of complex inorganic systems.



**Dame Seye**, is a teacher-researcher, at the Iba Der Thiam University of Thiès, Department of Chemistry and Physical, UFR Science and Technology since 2021. He successfully obtained his PhD in inorganic chemistry at the Cheikh Anta Diop University of Dakar in 2019, as well as a Master's

degree in molecular chemistry from the same institution in 2013. Dr. Seye is known for his unwavering commitment to research and teaching, as evidenced by his CAES diploma obtained at the faculty of sciences and education technologies at the Cheikh Anta Diop University of Dakar. His research, particularly his numerous articles on oxoanions and their complexes, makes him a key figure in the field. Currently, Dr. Seye is the head of the Licence and Master of Chemistry course at the Department of Chemistry and Physical, UFR Science and Technology, he continues to stand out for his expertise and passion for chemistry.



**Assane Toure** is a professor of molecular chemistry with specialization in structural chemistry and organometallic chemistry. He is affiliated with the Department of Physics and Chemistry at the Faculty of Science and Technology of Education and Training (FASTEF), Cheikh Anta Diop University

(UCAD), Dakar, Senegal. His research activities are primarily centered on inorganic chemistry, with a particular focus on the structural analysis and reactivity of organometallic compounds. Over the years, Dr. Touré has published numerous peer-reviewed articles in recognized scientific journals, contributing significantly to the understanding of molecular interactions and the development of new applications for organometallic systems.



**Carine Duhayon** obtained her PhD degree in 1999 in Toulouse, France. She was appointed an X-Ray engineer in 1999 at Sorbonne University in Paris. She entered the CNRS in 2003 at the Laboratory of Coordination Chemistry in Toulouse. She works as a Research Engineer. Her interests are focused on structure determination using X-ray diffraction on single crystals.



**Cheikh Abdoul Khadir Diop** is a specialist in inorganic chemistry at Cheikh Anta Diop University of Dakar. He is widely recognized for his impactful contributions to scientific research and has played an active role in numerous national projects. In 2012, he was promoted to the

rank of full professor, a distinction that reflects his academic excellence and dedication to higher education. Currently, Professor Diop serves as an assessor at the Faculty of Science and Technology at Cheikh Anta Diop University, where he continues to demonstrate his commitment to both education and advanced research in the field of chemistry.



**Mamadou Sidibe** is a distinguished researcher and educator, currently serving as a full professor in the Department of Chemistry at Cheikh Anta Diop University in Dakar. Renowned for his significant contributions to scientific research, he was appointed Dean of the Faculty of Technical Sciences in 2018 in recognition of his expertise and

longstanding commitment to higher education. Over the years, Professor Sidibe has played a pivotal role in numerous national and international collaborative projects, establishing himself as a key figure in the scientific community. He is currently the Rector of Souleymane Niang University of Matam, where he continues to advance academic excellence and research leadership.

## Research Field

**Aboubacar Soumano:** research on polyoxometalate materials chemistry and hybrid materials.

**Lamine Yaffa:** research on polyoxometalate chemistry, hybrid materials and Clay materials sciences.

**Dame Seye:** research on inorganic chemistry.

**Assane Toure:** research on inorganic chemistry.

**Cheikh Abdoul Khadir Diop:** research on inorganic chemistry.

**Mamadou Sidibe:** research on inorganic chemistry.

**Carine Duhayon:** structure determinations using X-ray diffraction on single crystals.