Research Article

A New Decavanadate with Organic Cation: Synthesis, Crystal Structure, and Hirshfeld Surface Analysis

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A new 2,6-bis aminomethyl piperidine decavanadate hydrate, $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$, was synthesized by slow evaporation of a solution at room temperature. The molecular structure was investigated by single-crystal X-ray diffraction. In the crystal structure, the layers of decavanadate groups, organic cations, and water molecules are arranged parallel to the (010) plane. Also, the prepared compound has been analysed by FTIR spectroscopy and scanning electron microscopy (SEM). The crystal structure of the title compound is stabilized by hydrogen bonds and van der Waals interactions. The cohesion of the structure is ensured by O-H...O and N-H...O hydrogen bonds. The three-dimensional Hirshfeld surface (3D-HS) and the relative two-dimensional fingerprint plots (2D-FPs) of $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ compound revealed that the structure is dominated by O...H/H...O (70.8%) and H...H (18.5%) contacts.

1. Introduction

Recently, the influence of noncovalent interactions has been widely studied due to their importance for the self-assembly and supramolecular stabilization [1]. However, hydrogen bonding has a significant function in crystal structure construction because of highly directive and relatively strong specific interactions [2]. These interactions exist in polyoxometalate (POM) groups, and the cohesion of these compounds is ensured by hydrogen bonds and van der Waals interactions, which encourage researchers to study this family especially in the field of biology [3]. Polyoxometalates (POMs) are a kind of inorganic clusters, which can be combined with organic ligands and metal ions to form supramolecular compounds. These groups have been extensively studied due to their potential applications in various fields such as catalysis [4], materials science [5], and antibacterial and antitumor activities [6]. Among the various types of

POMs, polyoxovanadates (POVs) are an important part of the group and invariably contain highly symmetrical core assemblies of VO_n units, which assemble into either discrete molecular clusters or link together to form one-dimensional chains, two-dimensional layers, or three-dimensional frameworks [7]. Several previous studies have shown that the decavanadate group has a significant role in some biological reactions [8, 9]. This role is derived mainly from interactions between anions and biological molecules such as proteins [10, 11]. These molecules have a nanomolar affinity for casein kinase 2 (CK2), making them the most potent inhibitors of CK2 to date [12]. Decavanadate compounds are known for their low toxicity and very potent antitumor activities. These results are verified by several authors such as Aissa et al. [13], Liu et al. [7], and Louati et al. [14].

In this paper, we report the synthesis, crystal structure, and Hirshfeld surface analysis of the novel decavanadate $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$.

TABLE 1: Crystallographic characteristics, X-ray data collection, and structure-refinement parameters for $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$.

System, sp. gr., Z	Monoclinic, $P2_1/c$, 2
a, b, c (Å)	10.488(3), 15.441(2), 25.841(6)
β (°)	90.14(2)
V (Å ³)	4184.98(1)
ρ_{calc} (g cm ⁻³)	2.08
Radiation, λ (Å)	MoK α , 0.71073
μ (mm ⁻¹)	2.219
T (K)	298
Sample size (mm ³)	0.25 $\times$ 0.19 $\times$ 0.15
Scan mode	$\omega/2\theta$
Absorption correction	Psi-scan
$T_{\text{min}}, T_{\text{max}}$	0.9052, 0.9996
θ_{max} (°)	22.00
h, k, l ranges	$0 \leq h \leq 11, 0 \leq k \leq 16, -27 \leq l \leq 27$
Number of reflections: measured/unique ($N1$)	5454/5113 ($N1$)
R_{int}	0.05
Number of refined parameters	616
$R(F^2)/wR(F^2)$ /relative to $N1$	0.091/0.222
Number of reflections ($I > 2\sigma(I)$) ($N2$)	3881 ($N2$)
$R(F^2)/wR(F^2)$ relative to $N2$	0.074/0.207
S	1.074
$\Delta\rho_{\text{max}}/\Delta\rho_{\text{min}}$, e/Å ³	1.63/-0.94

FIGURE 1: A view of the formula unit of $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$. Displacement ellipsoids are drawn at the 50% probability level.

2. Experimental

2.1. Materials and Physical Measurements. The infrared spectrum was measured at room temperature, using a Perkin Elmer Spectrum™ 100 FTIR spectrometer (France, Perkin Elmer) in the 4000–500 cm⁻¹ region, in an ATR cell.

The SEM-EDX (energy dispersive X-ray) was made of a single crystal chosen by a polarizing microscope. Scanning electron microscopy (SEM) coupled to energy dispersive X-ray analysis spectrometry is performed using an FEI Quanta 200 environmental apparatus (LEUVEN Belgium, PHILIPS/FEI).

2.2. Synthesis of 2,6-Bis Aminomethyl Piperidinium Decavanadate Hydrate $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$. The crystals are obtained from a mixture of vanadium oxide (V_2O_5) (Fluka, 99.9%) and 2,6-bis (ammoniomethyl) piperidinium (Sigma–Aldrich, 96%) in water with the respective molar proportion of 1 : 4. The mixture obtained is stirred with magnetic stirring, along with heating for about 3 hours. The solution thus obtained is transferred to a Petri dish. After a few days, the solution allows to deposit orange-coloured crystals suitable for X-ray diffraction analysis.

FIGURE 2: Projection of the $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ structure along the $[100]$ direction.TABLE 2: Distortion, indexes (ID), and BVS calculations for $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$.

Octahedra	V1O ₆	V2O ₆	V3O ₆	V4O ₆	V5O ₆
BVS	4.955	4.925	4.978	5.000	4.889
ID	0.081	0.083	0.086	0.0813	0.0814
Octahedra	V6O ₆	V7O ₆	V8O ₆	V9O ₆	V10O ₆
BVS	4.910	4.934	5.000	5.051	4.986
ID	0.099	0.100	0.167	0.088	0.009

2.3. X-Ray Diffraction Study. A single orange crystal of $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ was selected and used for X-ray diffraction experiment. Intensity data were collected using an Enraf-Nonius CAD-4 automatic diffractometer equipped with graphite monochromatic MoK α ($\lambda = 0.7107$ Å). The data reduction was processed with XCAD4 [15] included in the WINGX software package [16]. The structure was solved by the direct method using the program SHELXL-97 [17] and refined against F^2 data using the program SHELXL-2014 [18]. The reflections were corrected for Lorentz and polarization effects followed by psi-scan absorption correction [19]. All nonhydrogen atoms were refined with anisotropic atomic displacement parameters, whereas all hydrogen atoms were fixed using the HFIX instruction. The structure representation was prepared using DIAMOND [20]. The crystal data of $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ are summarized in Table 1. The CIF file containing complete information about the structure of the obtained structure was deposited in the Cambridge Crystallographic Data Center (CCDC no. 1968413); the file is freely available upon request to the following website: http://www.ccdc.cam.ac.uk/data_request/cif.

2.4. Hirshfeld Surface Study. The molecular Hirshfeld surface is created based on the electron distribution of a molecule and has been calculated as the sum of electron densities of spherical

atoms. The Hirshfeld surface was produced from the asymmetric unit of the $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ molecule. The Hirshfeld surfaces are mapped with d_{norm} and 2D fingerprint plots presented in this paper were generated using Crystal Explorer 3.1 [21]. The mapping of d_{norm} on the Hirshfeld surface highlights the directional intermolecular interactions. The value of the d_{norm} is negative or positive when intermolecular contacts are shorter or longer than van der Waals separations.

3. Results and Discussion

3.1. Crystal Structure. The single-crystal X-ray diffraction analysis shows that the formula unit of $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ consists of one $[V_{10}O_{28}]^{6-}$ decavanadate group, two organic cations of 2,6-bis (ammoniomethyl) piperidinium, and 4.21 water molecules (Figure 1). The $[V_{10}O_{28}]^{6-}$ anions consist of ten sharing edges $[VO_6]$ which is similar to those reported in the literature [22]. The decavanadate group in this structure is noncentrosymmetric. The V-O distances in this group depend on the type of oxygen atoms involved: they are between 1.575(4) and 1.620(4) Å for terminal oxygen atoms, between 1.680(4) and 2.080(4) Å for doubly coordinated oxygen atoms, between 1.923(4) and 2.046(3) Å for triply coordinated oxygen atoms, and between 2.090(3) Å and 2.355(4) Å for hexacoordinated oxygen atoms. V-V distances range

FIGURE 3: Projection of the $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ structure along the $[010]$ direction.

TABLE 3: Hydrogen bonds in $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ compound.

D-H...A	D-H (Å)	H...A (Å)	D...A (Å)	D-H...A (°)
N11-H11A...O14 ⁱ	0.890	2.437	2.933	115.57
N11-H11A...O27 ⁱⁱ	0.890	2.271	3.075	150.21
N11-H11B...O4	0.890	2.431	2.942	116.83
N11-H11B...O26 ⁱ	0.890	2.502	3.355	160.88
N11-H11C...O22 ⁱⁱ	0.890	2.253	2.846	123.85
N13-H13A...OW1 ⁱⁱⁱ	0.890	2.371	3.091	138.08
N13-H13B...O9 ⁱⁱ	0.890	2.055	2.925	165.13
N13-H13C...O13	0.890	2.095	2.917	153.19
N13-H13C...O25	0.890	2.564	3.248	134.17
C11-H11E...O6 ⁱ	0.970	2.619	3.508	152.36
N21-H21A...O15	0.890	2.403	2.899	149.63
N21-H21A...O26	0.890	2.289	3.090	164.32
N21-H21B...O19	0.890	2.229	2.828	124.34
N21-H21C...O3 ⁱⁱ	0.890	2.412	2.934	117.73
N21-H21C...O27 ⁱ	0.890	2.427	3.282	161.14
N23-H23A...O18 ⁱ	0.890	2.490	3.232	141.20
N23-H23A...O25 ⁱ	0.890	2.584	3.199	126.99
N23-H23A...OW6	0.890	2.636	3.304	132.60
N23-H23B...O16 ⁱⁱ	0.890	2.125	2.916	147.53
N23-H23B...O28 ⁱⁱ	0.890	2.445	3.164	138.15
N23-H23C...O12	0.890	2.077	2.938	162.55

Symmetry codes are (i) $-x+1, y+1/2, -z+3/2$, (ii) $x-1, y, z$, and (iii) $x, y-1, z$.

FIGURE 4: SEM-EDX of the $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ compound. FIGURE 5: Hirshfeld surface mapped over d_{norm} of the $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ compound.

FIGURE 6: Continued.

FIGURE 6: 2D fingerprint plots of the studied compound resolved into all contacts (a) and O...H/H...O (b), H...H (c), and O...O (d) contacts.

from 3.092(1) to 3.121(2) Å. These distances are consistent with those found in the compounds studied in the bibliography [23, 24].

The projection of the structure along the [100] direction shows that the decavanadate groups and organic cations form a three-dimensional framework that leaves free tunnels in which the water molecules are lodged (Figure 2).

The distortion indexes of VO_6 octahedra ranged between 8.1% and 16.7% [11] (Table 2). The vanadium is in the +5 oxidation state. This result was confirmed by the bond valence sum calculations (Table 2) according to Brown [25] ($S = \sum s_i = \sum \exp[(R_0 - R_i)/B]$).

The structure of $(\text{C}_7\text{N}_3\text{H}_{20})_2\text{V}_{10}\text{O}_{28} \cdot 4.21\text{H}_2\text{O}$ compound can be described as a layered structure parallel to the (001) plane (Figure 3).

Organic cations are linked only by van der Waals interactions. The cohesion of the structure is ensured by hydrogen bonds, van der Waals interactions, and N-H...O and O-H...O hydrogen bonds.

In this structure, the hydrogen bonds are weak with the D-A bond lengths varying from 2.846 to 3.508 Å according to Brown [26] (Table 3). Mean values of distances C-C and C-N are, respectively, 1.512(2) and 1.509(2) Å. These bond lengths are in agreement with those reported in the literature [27, 28].

3.2. IR Spectroscopic Characterisation. The swept frequency range of the IR spectrum extends from 4000 to 500 cm^{-1} (Figure SI. 1). The bands at 825, 750, 676, 600, 546, and 398 cm^{-1} are assigned to the internal vibration modes of the VO_6 octahedra [14]. The strong band at 966 cm^{-1} is assigned to the vibration modes $\nu(\text{V}=\text{O})$. The broad band located in the 2800–3430 cm^{-1} domain is attributed to -OH groups involved in hydrogen bonds [29]. The vibration modes $\nu(\text{C-H})$, $\nu(\text{C-N})$, $\nu(\text{C-C})$, and $\nu(\text{N-H})$ are located around 1164, 1199, 1384, 1487, 1624, 1940, and 2098 cm^{-1} [30].

3.3. SEM-EDX Analysis. The scanning electron microscopy of single crystals of $(\text{C}_7\text{N}_3\text{H}_{20})_2\text{V}_{10}\text{O}_{28} \cdot 4.21\text{H}_2\text{O}$ phase revealed the presence of vanadium (V), nitrogen (N), carbon (C), and oxygen (O) in the sample studied. The morphology of crystals and the spectrum obtained are presented in Figure 4.

3.4. Hirshfeld Surface Analysis. The Hirshfeld surfaces of $(\text{C}_7\text{N}_3\text{H}_{20})_2\text{V}_{10}\text{O}_{28} \cdot 4.21\text{H}_2\text{O}$, over a d_{norm} , are shown in Figure 5. In the crystal, molecules are linked into a three-dimensional network by N-H...O and O-H...O hydrogen bonds. In order to visualize the intermolecular interactions in the crystal of the title compound, the Hirshfeld surface (HS) analysis was carried out by using Crystal Explorer. Figure 5 shows the presence of O-H...O and N-H...O bonds and H...H interactions.

The total contacts are given in Figure 6(a). The structure is dominated by O...H/H...O (70.8%) (Figure 6(b)) and H...H (18.5%) (Figure 6(c)) contacts. The O...O-type interactions represent 10.7% (Figure 6(d)).

4. Conclusion

A novel organic-inorganic framework of $(\text{C}_7\text{N}_3\text{H}_{20})_2\text{V}_{10}\text{O}_{28} \cdot 4.21\text{H}_2\text{O}$ has been successfully synthesized by evaporation of an aqueous solution at room temperature. Its single structure was determined using single-crystal X-ray diffraction, and it was characterized by FTIR and SEM analysis. The structure of the obtained compound is formed by the decavanadate groups and the organic cations. It can be described as a three-dimensional framework that contains tunnels in which the water molecules are located. The crystal structure was stabilized by O-H...O and N-H...O hydrogen bonds. The Hirshfeld surface analysis reveals that O...H/H...O (70.8%) and H...H (18.5%) intermolecular interactions are the most dominant in the crystal structure. In this perspective, we plan to perform

further characterizations to explore the anticancer activity and therapeutic intervention of the title compound.

Data Availability

The CIF file containing complete information about the structure of the obtained structure was deposited with the Cambridge Crystallographic Data Center (CCDC no.1968413). The file is freely available upon request from the following web site: http://www.ccdc.cam.ac.uk/data_request/cif.

Conflicts of Interest

The authors declare no conflicts of interest.

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Supplementary Materials

Figure S1: infrared spectrum of the $(C_7N_3H_{20})_2V_{10}O_{28} \cdot 4.21H_2O$ compound. (Supplementary Materials)

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