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Synthesis, Characterization and Thermal Analysis of an Organic-Inorganic Hybrid Salt Involving *Trans*-Diaquabis(oxalato-$\kappa^2 O^1,O^2$)chromate(III) Complex Anion with Piperidinium as Counter Cation

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Abstract

A new organic-inorganic hybrid salt pipéridinium *trans*-diaquabis(oxalato)-chromate(III) tetrahydrate, $(C_5H_{10}NH_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$ (**1**), has been synthesized in water and characterized by FTIR and UV-Vis spectroscopies, elemental and thermal analyses and by single-crystal X-ray diffraction. **1** crystallizes in the orthorhombic non-centrosymmetric space group $Cmc2_1$ with the unit cell parameters $a = 7.4329(3)$, $b = 9.9356(5)$, $c = 23.6756(11)$ Å, $\alpha = \beta = \gamma = 90^\circ$, $V = 1748.45(14)$ Å^3 and $Z = 4$. The structure of **1** consists of $[Cr(C_2O_4)_2(H_2O)_2]^-$ mononuclear anions, piperidinium cations and uncoordinated water molecules. The Cr^{III} ion in the complex $[Cr(C_2O_4)_2(H_2O)_2]^-$ is coordinated in a slightly distorted octahedral environment by four O atoms from two chelating oxalate dianions in the equatorial plane, and two O atoms from *trans*-coordinated water molecules occupying the apical positions. In the crystal, N-H···O and O-H···O hydrogen bond interactions connect the components into a 3-D framework. The IR spectrum of **1** is consistent with the presence of the various molecular building constituents, namely oxalato and aqua ligands, piperidinium cations and solvent water molecules. The UV-Vis spectrum shows two absorption bands around 564 and 416 nm which are compatible with an anionic chromium(III) complex in an octahedral environment. Thermal analysis shows a three-step decomposition of **1**, leading to formation of a metal oxide residue.

Keywords

Hybrid Salt, Diaquabis(oxalato)chromate(III) Complex, Crystal Structure, Layered Structure, Spectroscopy, Thermal Analysis

1. Introduction

A great interest has been paid to the synthesis of novel organic-inorganic hybrid salt due to their structural diversity [1] and potential applications in gas storage [2], ion exchange and catalysis [3], photoluminescence [4] [5] and magnetism [6]. In this respect, the diaquabis(oxalato)metalate(III) complex anions, $[M^{III}(C_2O_4)_2(H_2O)_2]^-$, have been extensively used as building blocks for the generation of multifunctional materials formed by two distinct molecular networks, each furnishing a particular structural and physical property [7] [8] [9] [10]. Among the organic cations involved in these hybrid salts, the use of pyridinium cations and their derivatives to build supramolecular architectures has become an extremely active field of investigation around the world [7] [9] [11]-[16]. Furthermore, complexes involving piperidinium cations or their derivatives are known to be useful in the fields of medicine, bioinorganic chemistry and catalysis [17] [18] [19]. These cations obtained via protonation of the imine group of such organic molecules have the ability to self-assemble through the charge-assisted hydrogen bonds.

In continuation of the systematic search for other members of this family of materials, herein we report the synthesis, characterization and thermal analysis of a new organic-inorganic hybrid salt, piperidinium *trans*-diaquabis(oxalato-κ^2O^1,O^2)chromate(III) tetrahydrate, $(C_5H_{10}NH_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$ (**1**). Two aspects of focal relevance are associated with **1**: a) its framework is non-centrosymmetric; b) the number of solvent water molecules per formula unit seems to be the highest obtained so far for this family of bis(oxalato)metalate(III) salts.

2. Experimental

2.1. Materials and Physical Measurements

Reagents were obtained from Prolabo (oxalic acid and piperidine) and Riedel-de Haën (chromium(III) chloride hexahydrate) and used as such without further purification. Elemental analyses were performed using a Thermo Scientific FLASH 2000 Analyzer. The FTIR spectrum was perfomed with an Alpha-P spectrophotometer in the range 4000 - 400 cm^{-1} using KBr pallets. The UV-Vis spectrum was recorded on an Aqualytic spectrophotometer in water solution in the range 200 - 800 nm. Thermogravimetric (TG) analysis was investigated on a Mettler-Toledo TGA/DSC Thermogravimetric Analyser with a heating rate of 10˚C/min in flowing air. Single-crystal X-ray measurements were performed using the Agilent SuperNova diffractometer (λ = 0.71073 Å).

2.2. Synthesis of $(C_5H_{10}NH_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$ (1)

Chromium(III) trichloride hexahydrate, $CrCl_3\cdot 6H_2O$ (6 mmol ; 1.6 g) was dissolved in 40 mL of water : solution A. Commercial piperidine $C_5H_{11}N$ (6 mmol ; 0.43 g) and oxalic acid $H_2C_2O_4\cdot 2H_2O$ (12 mmol ; 1.52 g) were dissolved in 30 mL of water (solution B) and added dropwise in solution A. The mixture was stirred at room temperature for 5 h, then filtered and the filtrate was left to stand in the hood at room temperature. After two weeks, violet crystals suitable for X-ray structure determination were harvested. Yield: 71% based on $CrCl_3\cdot 6H_2O$. Anal. Calcd. for $C_9H_{24}NCrO_{14}$ (422.29 g·mol^{-1}): C, 25.60; H, 5.73; N, 3.32%. Found: C, 25.23; H, 5.68; N, 3.32%. FTIR (KBr disk, cm^{-1}): 3502, 3393, 3135, 2960, 1705, 1400, 1261, 903, 622, 482. UV-Vis (H_2O solution, nm): 416, 564.

2.3. Crystal Structure Determination and Refinement

A suitable single crystal of the material was selected and mounted on a glass fiber. Diffraction data were obtained at 100 K on a Rigaku Oxford Diffraction SuperNova diffractometer with Mo-Kα radiation (λ = 0.71073 Å). The X-ray intensities were corrected using numerical absorption correction based on Gaussian integration over a multifaceted crystal model [20]. The crystal structure was solved by direct method of SHELXT-2014 [21] and refined by full-matrix least-square techniques on F^2 using the SHELXL-2018 program package [22]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were added in idealized geometrical positions for the organic cations. The positions of hydrogen atoms from the water molecules were assigned from the electron density map generated by Fourier difference and they were refined freely apart from the hydrogen atoms of O5W for which a restraint (O-H bond length of (0.9 ± 0.020) Å was used. They were included as riding atoms with isotopic displacement parameters ADPs ($U_{iso}H = 1.2U_{eq}C = 1.2U_{eq}N = 1.2U_{eq}O_w$). DIAMOND program [23] was used to deal with the processed crystallographic data and artwork representations. Details of the structure determination and final refinements are summarized in **Table 1** and selected bond lengths (Å) and angles (˚) around the central chromium (III) ion are listed in **Table 2**.

3. Results and Discussion

3.1. Formation of $(C_5H_{10}NH_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$ (1)

The combination of piperidine ($C_5H_{10}NH$) with oxalic acid in aqueous medium generates in situ $(C_5H_{10}NH_2)_2C_2O_4$ which reacts with an aqueous solution of $CrCl_3\cdot 6H_2O$. This reaction affords, by slow evaporation of the resulting solution at room temperature after two weeks, the title compound $(C_5H_{10}NH_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$ **(1)** as violet prismatic crystals. **1** is thermally stable up to 90˚C.

Table 1. Crystal data and structure refinement details for **1**.

CCDC N°	1,988,875
Empirical formula	$C_9H_{24}NCrO_{14}$
Formula weight	422.29
Temperature (K)	100 (1)
Wavelength (Å)	0.71073
Crystal system	Orthorhombic
Space group	$Cmc2_1$
a (Å)	7.4329 (3)
b (Å)	9.9356 (5)
c (Å)	23.6756 (1)
α (°)	90.0
β (°)	90.0
γ (°)	90.0
Volume (Å^3)	1748.5 (1)
Z, Z'	4, 1/2
Absorption coefficient (mm^{-1})	0.726
F(000)	884
Crystal size (mm)	0.21 × 0.17 × 0.15
Theta range for data collection (°)	3.4 - 37.7
Index ranges	$-12 < h < 12$, $-16 < k < 17$, $-39 < l < 40$
Total reflections	25774
Unique reflections (R_{int})	4831(0.025)
Refinement method	Full-matrix least squares on F^2
Data/restraints/parameters	4831/2/153
Goodness-of-fit (GOF) on F^2	1.06
Final R indices [$I > 2$ sigma (I)]	$R_1 = 0.0220$, w$R_2 = 0.0571$
R indices (all data)	$R_1 = 0.0228$, w$R_2 = 0.0578$
$\Delta\rho_{max}$ and $\Delta\rho_{min}$ (e/Å^3)	0.31 and −0.81
Flack parameter	0.00 (4)

Table 2. Selected bond lengths (Å) and bond angles (°) of **1**.

Selected bond lengths (Å)			
Cr1-O1	1.9754 (8)	Cr1-O3i	1.9701 (8)
Cr1-O1i	1.9755 (8)	Cr1-O5W	1.9589 (1)
Cr1-O3	1.9701 (8)	Cr1-O6W	1.9653 (1)
Selected bond angles (°)			
O1-Cr-O1^i	82.32 (5)	O5W-Cr-O3	90.07 (4)

Continued

O3^{i}-Cr1-O1	97.56 (3)	O5W-Cr1-O3^{i}	90.07 (4)
O3-Cr1-O1^{i}	97.56 (3)	O6W-Cr1-O1	90.12 (4)
O3-Cr1-O1	179.87 (4)	O6W-Cr1-O1^{i}	90.12 (4)
O3^{i}-Cr1-O1^{i}	179.87 (4)	O6W-Cr1-O3	89.92 (4)
O3-Cr1-O3^{i}	82.57 (5)	O6W-Cr1-O3^{i}	89.92 (4)
O5W-Cr1-O1	89.89 (4)	O5W-Cr-O6W	179.99 (7)
O5W-Cr1-O1^{i}	89.89 (4)		

Symmetry code: (i) −x + 1, y, z.

3.2. Infrared Spectrum of 1

The FTIR spectrum of **1** (**Figure 1**) exhibits a weak absorption band centered at 3502 cm^{-1} attributed to ν_{N-H} of piperidinium cations. The sharp bands observed at 3393 cm^{-1} and 3135 cm^{-1} can be assigned to the well-known ν_{O-H} vibrations of the H_2O molecules of crystallization involved in hydrogen bonding and the H_2O ligands that are coordinated to the Cr^{III} sites respectively [24] [25]. The stretching vibration band of C-H (ν_{C-H}) is situated at 2960 cm^{-1}. The strong band appearing at 1705 cm^{-1} corresponds to $\nu_{C=O}$ [26] and the medium-size band at 1400 cm^{-1} can be assigned to the symmetric stretching absorption of the carboxylate groups of the oxalato ligand [26]. Strong to medium well-resolved bands appear at 1261 cm^{-1} (ν_{C-N}), 1181 cm^{-1} (ν_{C-O}), 1081 cm^{-1} (ν_{C-C}) [27] [28]. The pattern of the $\delta_{O-C=O}$ vibrations range of 944 - 875 cm^{-1} supports the presence of chelating oxalate dianions in the structure of **1**. Medium to weak bands observed in the region 622 - 482 cm^{-1} may be attributed to vibrations ν_{Cr-O} within the coordination spheres around the metallic centers ions. These results are consistent with the presence of $C_5H_{10}NH_2^+$ cation, $[Cr(C_2O_4)_2(H_2O)_2]^-$ complex anion, and H_2O molecules of crystallization in **1**.

3.3. UV-Vis Spectrum of 1

The electronic absorption spectrum of **1** (**Figure 2**) reveals two absorption bands at 416 nm (24038 cm^{-1}) and 564 nm (17730 cm^{-1}) corresponding respectively to $^4A_{2g} \rightarrow {}^4T_{1g}(F)$ and $^4A_{2g} \rightarrow {}^4T_{2g}$ (*d*–*d*) transitions within the octahedral $[Cr(C_2O_4)_2(H_2O)_2]^-$ anionic complex of **1** [8] [24] [29]. Obviously, the present electronic absorption spectrum is virtually superimposable with that reported since the spectral information thus obtained solely relates to $[Cr(C_2O_4)_2(H_2O)_2]^-$ species.

3.4. Thermal Analysis of 1

The thermogravimetric (TG) and differential scanning calorimetry (DSC) curves of **1** depicted in **Figure 3** evidence three distinct weight losses in the temperature range 90°C - 389°C with endothermic processes. The possible decomposition reactions, the experimental and calculated percentage weight losses are summarized in

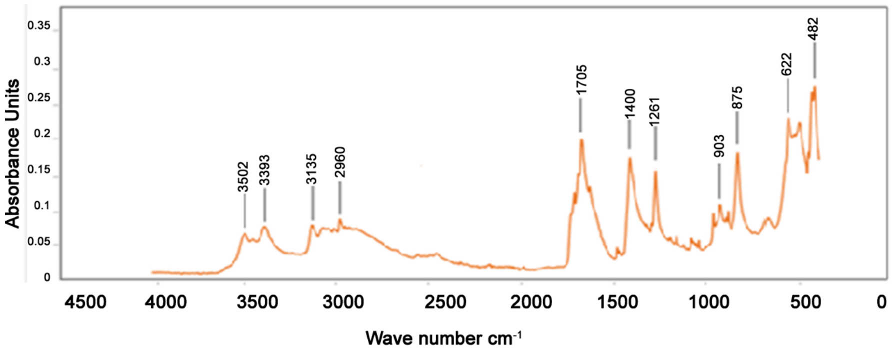

Figure 1. Infrared absorption spectrum of **1**.

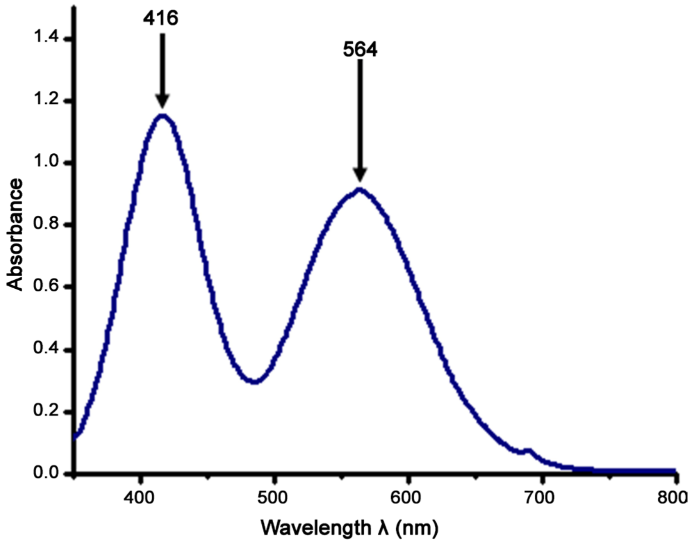

Figure 2. Electronic absorption spectrum of **1**.

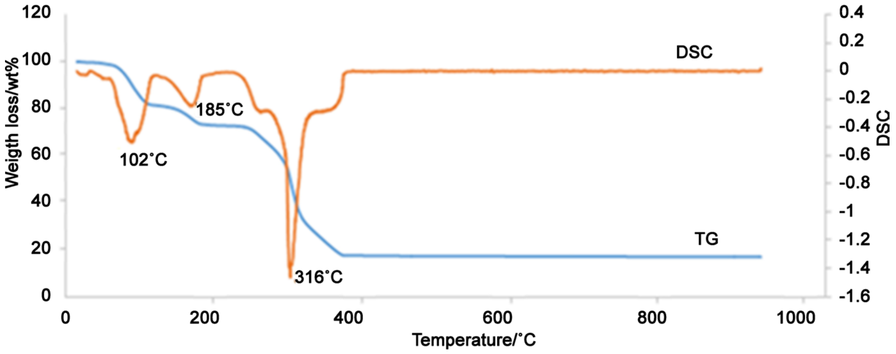

Figure 3. TG curve (blue) and DSC curve (orange) of **1**.

Table 3. In the temperature range 90˚C - 130˚C, **1** suffers a first weight loss of 18.48% (calc. 18.47%) corresponding to the release of the four water molecules of crystallization and three molecules of hydrogen from a partial degradation of organic moieties. A second weight loss of 8.56% (calc. 8.57%) occurs between 156˚C - 196˚C, corresponding to the partial decomposition of the $[Cr(C_2O_4)_2(H_2O)_2]^-$ complex anion framework with release of the coordinating water molecules. The

third weight loss of 55.83% (calc. 54.94%) in the temperature range 250˚C - 389˚C, attributed to the total decomposition of **1**, leads to a final Cr_2O_3 residue [28] [30].

3.5. Crystal Structure of 1

The asymmetric unit of the title compound is shown in **Figure 4**. The molecular structure is formed by the piperidinium $(C_5H_{10}NH_2)^+$ cation in chair conformation, the complex anion, $[Cr(C_2O_4)_2(H_2O)_2]^-$ in *trans*-geometry and four water molecules of crystallization. In the $[Cr(C_2O_4)_2(H_2O)_2]^-$ complex anion, the Cr^{III} ion adopts a slightly distorted octahedral coordination environment defined by two chelating bidentate oxalate ligands in the equatorial plane and by two water O atoms in the axial sites. The expected ideal values 90˚ and 180˚ in bond angles O-Cr-O in the above complex anion, vary within a range from 82.32 (5)˚ to 97.56 (3)˚ and from 179.87 (4)˚ to 179.99 (7)˚ respectively (see **Table 2**). The two

Table 3. Thermal decomposition data of **1**.

Reaction	TGA (˚C)	DSC (˚C)	Mass loss (%)	
			W_{exp}	$W_{calcd.}$
$(C_5H_{10}NH_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$ $\downarrow$ $-4H_2O$, $3H_2$ $(C_5H_5NH)[Cr(C_2O_4)_2(H_2O)_2]$	90 - 130	104 (endo)	18.48	18.47
$\downarrow$ $-2H_2O$ $(C_5H_5NH)[Cr(C_2O_4)_2]$	156 - 196	184 (endo)	8.56	8.57
$\downarrow$ $-(C_5H_5NH)$, $2CO_2$, $2CO$, $\frac{1}{4}O_2$ $1/2Cr_2O_3$	250 - 389	316 (endo)	55.83	54.94

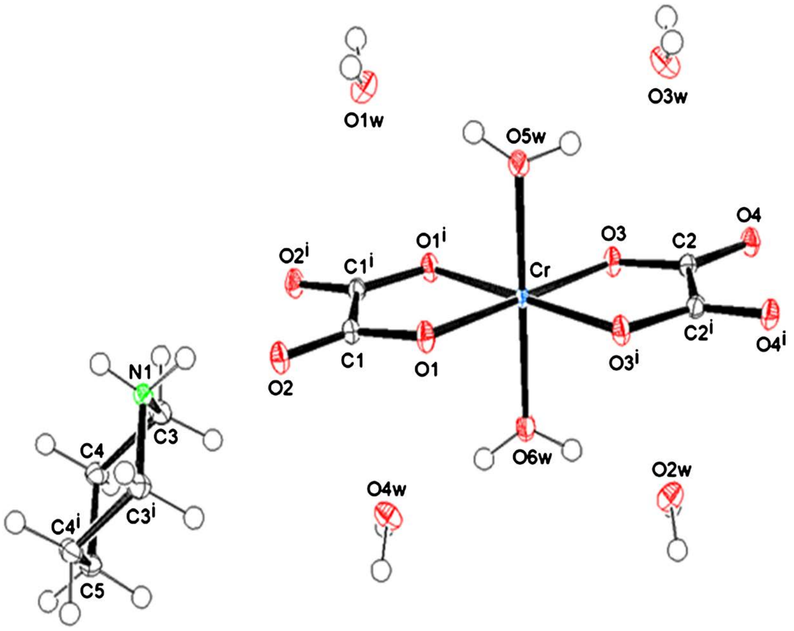

Figure 4. Molecular structure of **1** with the atom numbering scheme. (i = x + 1, y, z).

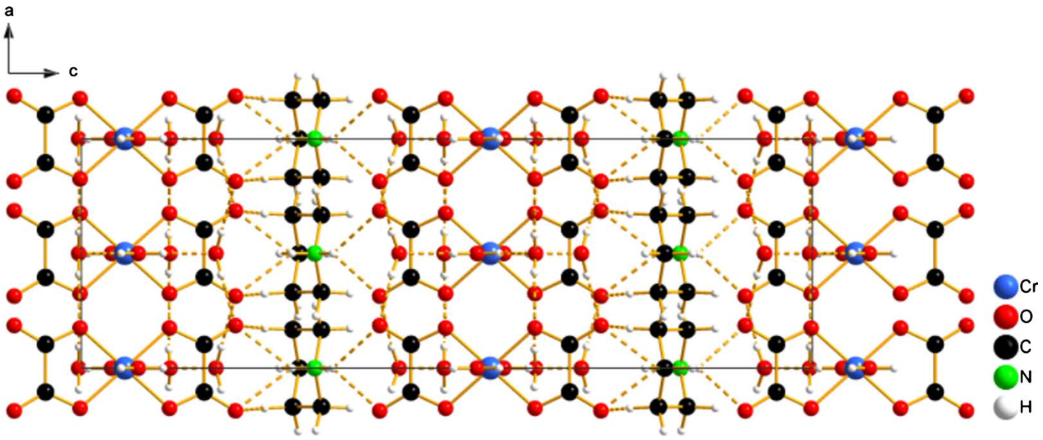

Figure 5. Packing diagram of **1**, viewed along the *b* axis, showing pillars of alternating complex anions and piperidinium cations and highlighting the interconnection of the components into a tridimensional network by hydrogen bonds (dashed lines).

Table 4. Hydrogen bond lengths (Å) and bond angles (˚) of **1**.

D-H···*A*	*D*-H	H···*A*	*D*···*A*	*D*-H···*A*
O6W-H7W···O2W	0.83 (3)	1.76 (3)	2.593 (2)	178 (3)
O5W-H6W···O1W	0.83 (3)	1.78 (3)	2.603 (2)	167 (3)
O2W-H2W···O4$^{\text{ii}}$	0.86 (2)	1.94 (2)	2.799 (1)	175 (2)
O4W-H4W···O1$^{\text{iii}}$	0.88 (2)	2.01 (2)	2.878 (1)	173 (2)
O3W-H3W···O3$^{\text{iv}}$	0.76 (2)	2.12 (2)	2.879 (1)	174 (2)
O1W-H1W···O2$^{\text{v}}$	0.85 (2)	1.95 (2)	2.797 (1)	175 (2)
N1-H1···O4$^{\text{vi}}$	0.82 (3)	2.32 (2)	3.002 (1)	141 (9)
N1-H1···O4$^{\text{i}}$	0.82 (3)	2.32 (2)	3.002 (1)	141 (9)
N1-H2···O2	0.91 (3)	2.24 (2)	2.973 (1)	137 (1)
N1-H2···O2$^{\text{vii}}$	0.91 (3)	2.24 (2)	2.973 (1)	137 (1)
O5W-H5W···O3W	0.84 (2)	1.81 (2)	2.627 (2)	162 (3)
O6W-H8W···4W	0.84 (3)	1.82 (3)	2.648 (2)	173 (3)

Symmetry codes: (i) *x*, −*y* + 1, *z*–1/2; (ii) *x*–1/2, *y*–1/2, *z*; (iii) *x* + 1/2, *y*–1/2, *z*; (iv) *x*–1/2, *y*+1/2, *z*; (v) −*x* + 1/2, *y*+1/2, *z*; (vi) −*x* + 1, −*y* + 1, *z*–1/2; (vii) −*x* + 1, *y*, *z*.

pairs of equatorial Cr-O(ox) distances 1.9754 (8) and 1.9701 (8) Å and the two axial Cr-Ow distances 1.9589 (13) and 1.9653 (14) (Å) are comparable to those reported in similar compounds [8] [9] [10] [11] [12] [30] [31] and in agreement with the Cr-O(ox) (2.189 ± 0.293) Å and Cr-Ow (2.226 ± 0.411) Å mean values found in the CSD [32]. By contrast, the Cr-Ow bond lengths are slightly shorter than the Cr-O(ox) ones. This situation was not previously observed in homologous salts involving $[Cr(C_2O_4)_2(H_2O)_2]^-$ complex anion [7]-[12]. Packing diagram of **1**, viewed along the *b* axis, showing its layered structure formed of pillars of alternating $[Cr(C_2O_4)_2(H_2O)_2]^-$ complex anions and piperidinium $(C_5H_{10}NH_2)^+$ cations plus H_2O molecules of crystallization (**Figure 5**). In fact, the layered framework is exclusively the result of 3D interconnectivity between Cr^{III} ions, oxalato and aqua ligands. The framework formed by $[Cr(C_2O_4)_2(H_2O)_2]^-$ carries an overall excess negative charge and delineates unoccupied spaces parallel to the *a* axis, encapsulating water molecules of hydration and charge-balancing piperidinium cations, $(C_5H_{10}NH_2)^+$. The components are linked by N-H···O [2.24 (2) to 2.32 (2) Å] and O-H···O [1.76 (3) to 2.12 (2) Å] hydrogen bonds (dashed lines), generating a non-centrosymmetric 3D framework. The values of hydrogen bond lengths (Å) and angles (˚) are summarized in Table 4. To the best of our knowledge, **1** and 4-aminopyridinium *trans*-diaquabis(oxalato-κ^2O^1,O^2)chromate(III) monohydrate [14] seem to be the only compounds having a non-centrosymmetric structure built with the $[Cr(C_2O_4)_2(H_2O)_2]^-$ complex anions so far. In the other hand, it is worth noting that the number of solvent water molecules per formula unit in **1** is higher, compared to the homologous bis(oxalato)metalate(III) salts known hitherto [7] [10] [11] [14]. This fact of matter reinforces the crystal packing framework through extended hydrogen bridgings.

4. Conclusion

In summary, compound **1**, a novel organic-inorganic hybrid salt comprising the trans-diaquabis(oxalato)chromate(III) complex anion and piperidinium cation has been synthesized and characterized spectroscopically, thermally and structurally. Thermal studies revealed that compound **1** is stable to heat up to 90˚C. It crystallizes in the chiral orthorhombic $Cmc2_1$ space group. This work confirms the great flexibility of synthetic manoeuvres for the self-assembly of bis(oxalato)chromate(III) complex anions with various organic cations. One could consider with serenity the fabrication of a compound in which the organic cation compensating the charge of the anionic complex would be solely replaced by small charged species such as hydronium ions. Such a system with protons balancing the negative charge of the anionic framework could be a good candidate for the exploration of the concept of one-dimensional proton conducting solids [33] [34] [35]. It could be also worth studying the magnetic properties of **1** as well as its antibacterial activities as with related compounds [10] [13]. Work in this direction is in progress in our group.

Supplementary Material

Detailed crystallographic data in CIF format for this paper were deposited with the Cambridge Crystallographic Data Centre (CCDC-1988875). The data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html [or from Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 IEZ, UK; fax: +44 (0) 1223-336033; e-mail: deposit@ccdc.cam.ac.uk].

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Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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