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THE FIRST CHROMATE CONTAINING BOTH Cr_2O_7 AND Cr_3O_{10} GROUPS, bis(1,3-bis(4-PIPERIDIUM)PROPANE) DICHROMATE TRICHROMATE MONOHYDRATE: SYNTHESIS AND CRYSTAL STRUCTURE**R. Ben Smail^{1,2}, M.F. Zid², A. Driss²**¹University of Carthage, Nabeul Preparatory Engineering Institute, University Campus, Nabeul, Tunisia
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A new organic-inorganic hybrid material ($\text{C}_{13}\text{H}_{28}\text{N}_2$)₂[Cr_2O_7][Cr_3O_{10}]· H_2O (**1**) was synthesized by slow solvent evaporation at room temperature, and its crystal structure was determined by single crystal X-ray diffraction. This compound crystallizes in the monoclinic space group $P2_1/c$. The asymmetric unit contains two crystallographically independent 1,3-bis(4-piperidinium)propane, (H_2bppp)²⁺ cations (A and B), one trichromate $\text{Cr}_3\text{O}_{10}^{2-}$ anion, one dichromate $\text{Cr}_2\text{O}_7^{2-}$ anion, and one water molecule. All these entities are interconnected into a complicated two-dimensional hydrogen bonded network *via* N—H···O and O—H···O hydrogen bonds. Furthermore, this structure is stabilized by a large number of C—H···O interactions, thus establishing a three-dimensional network structure. This compound appears to be the first example of chromates containing both Cr_2O_7 and Cr_3O_{10} groups.

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Keywords: X-ray diffraction, crystal structure, dichromate trichromate, 1,3-bis(4-piperidinium)propane.**INTRODUCTION**

A large number of dichromates of nitrogen organic bases have been prepared and used as reagents in mild selective oxidation processes of organic substrates [1]. It has been reported that the type of the nitrogen organic cation associated with the dichromate anions determines the oxidizing property of the dichromate ion and this is inversely related to the donor strength of the associated amine ligand [2, 3]. During the last two and half decades, many studies have been reported on crystal structure of organic dichromates [1], as well as a few cases of trichromates [4—9].

For dichromates, these crystallographic studies can help to illustrate the relationship between molecular structure and selectivity in oxidation reactions.

In continuation of our search for new nitrogen organic dichromates, we have reported the synthesis and the crystal structure of tri(*N,N,N',N'*-tetramethylethylenediammonium) bis(dichromate) oxalate tetrahydrate ((CH_3)₂NH(CH₂)₂NH(CH₃)₂)₃(C₂O₄)[Cr_2O_7]₂·4H₂O [10], *N,N,N',N'*-tetramethylethylenediammonium dichromate monohydrate ((CH_3)₂NH(CH₂)₂NH(CH₃)₂)[Cr_2O_7]·H₂O [11], bis(ethylenediammonium) dichromate oxalate (H₃N(CH₂)₂NH₃)₂[Cr_2O_7](C₂O₄) [12], bis(guaninium) dichromate dihydrate (C₅H₆N₅O)₂[Cr_2O_7]·2H₂O (Crystallographic data for the structure have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 1010792) [13], 1-cyclohexyl-

piperazine-1,4-dium dichromate ($C_{10}H_{22}N_2$)[Cr_2O_7] [14] and room-temperature structure of bis(4-aminopyridinium) dichromate ($C_5H_7N_2$) $_2$ [Cr_2O_7] [1].

In this paper, the synthesis and the crystal structure of bis(1,3-bis(4-piperidinium)propane) dichromate trichromate monohydrate, ($H_2NC_5H_9(CH_2)_3C_5H_9NH_2$) $_2$ [Cr_2O_7][Cr_3O_{10}]· H_2O (**1**) are reported. This compound represents, to the best of our knowledge, the first chromate containing both Cr_2O_7 and Cr_3O_{10} groups and the first organic chromate containing the protonated 1,3-bis(4-piperidyl)propane molecule.

EXPERIMENTAL

Preparation of 1. CrO_3 (1.8 g; 18 mmol) was dissolved in distilled water (20 ml). 1,3-bis(4-piperidyl)propane (Bppp), also called 4,4'-trimethylenedipiperidine (1.0 g; 4.75 mmol) was added under stirring. The mixture was stirred for 30 min at room temperature. The resulting solution was allowed to stand for a week at room temperature. Orange-red single crystals suitable for X-ray analysis were obtained by slow evaporation of water.

Single crystal X-ray diffraction. The single crystal X-ray diffraction data for structure determination were collected at room temperature on Enraf-Nonious CAD-4 diffractometer equipped with graphite monochromated MoK_α radiation ($\lambda = 0.71073 \text{ \AA}$) using the $\omega/2\theta$ scan mode. Lattice parameters and orientation matrix were obtained by the least squares refinement of 25 reflections in the range $10.3\text{--}13.6^\circ$ using CAD4-EXPRESS program [15, 16]. The intensities of two standard reflections were measured every 400 reflections throughout the course of data collection and no significant decay was detected.

The crystallographic data of **1**: ($C_{13}H_{28}N_2$) $_2$ [Cr_2O_7][Cr_3O_{10}]· H_2O , $FW = 974.76$, $T = 298 \text{ K}$. Crystal size $0.31 \times 0.25 \times 0.18 \text{ mm}$, monoclinic, $P2_1/c$ (No. 14), $a = 12.486(3)$, $b = 13.118(3)$, $c = 25.408(5) \text{ \AA}$, $\beta = 91.65(3)^\circ$, $V = 4160(2) \text{ \AA}^3$, $Z = 4$, $\rho_{\text{calc}} = 1.556 \text{ g/cm}^3$, $\mu(MoK_\alpha) = 1.34 \text{ mm}^{-1}$, a total of 9067 reflections ($\theta_{\text{min/max}} = 2.2/27^\circ$), 3651 observed reflections ($I \geq 2\sigma(I)$), 484 parameters, min/max transmission 0.790/0.875, $GOOF = 0.99$, $R_1 = 0.077$, $wR_2 = 0.225$ ($I \geq 2\sigma(I)$), max/min diff. peak $0.71\text{--}0.45 \text{ e/\AA}^3$.

The data reduction was processed with XCAD4 program [17] included in the WinGX software package [18]. The reflections were corrected for Lorentz and polarization effects; absorption correction was obtained *via* a psi-scan [19] and secondary extinction correction was applied too [19].

The structure was solved and refined using the SHELX-97 package [20]. Non-hydrogen atoms were directly located from difference Fourier maps and refined with anisotropic displacement parameters.

Hydrogen atoms on the organic species were clearly observed in the difference electron density maps. However, for the final refinement cycles these atoms were placed in geometrically calculated positions and included with positional and displacement parameters riding on their parent atoms ($U_{\text{iso}}(H) = 1.2 U_{\text{eq}}(C,N)$) using appropriate HFIX instructions in SHELXL-97 [20]. Water hydrogen atoms were refined with distances restraints $Ow\text{--}H = 0.82(1) \text{ \AA}$ with $U_{\text{iso}}(H) = 1.5 U_{\text{eq}}(Ow)$ using DFIX instruction. SHELXL-97 shows that it might be possible to interpret the anisotropic displacement of O8, O10 and Ow atoms in terms of two discrete sites. Several cycles of refinement of this disorder indicate that the single-site anisotropic description is still eminently suitable. This is due to the fact that the crystal quality was not good. After several attempt of data collection, the reported one is found to be the best one. So we note here that the reliability factor is relatively high. Graphics were prepared using Diamond program [21]. Hydrogen bond scheme is described in Table 1. CIF file containing complete information about the structure of **1** was deposited with the Cambridge Crystallographic Data Center (No. 1043331); the file is freely available upon request from the following web site: http://www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

The molecular structure of **1** contains two crystallographically independent 1,3-bis(4-piperidinium)propane (H_2Bppp) $^{2+}$ cations (**A** and **B**), one trichromate $Cr_3O_{10}^{2-}$ anion, one dichromate $Cr_2O_7^{2-}$ anion, and one water molecule (Fig. 1). These entities are linked by $N\text{--}H\cdots O$ and $O\text{--}H\cdots O$ hydro-

Table 1

Hydrogen bond geometry (Å, deg.) in the structure of **1**

D—H···A	D—H	H···A	D···A	D—H···A	D—H···A	D—H	H···A	D···A	D—H···A
Ow—HwA···O17	0.82(1)	2.2(2)	2.753(12)	123(21)	N3—H3A···O3	0.90	2.37	2.958(9)	123
N1—H1A···Ow ⁱ	0.90	1.85	2.748(12)	173	N3—H3A···O12	0.90	2.47	3.253(10)	146
N1—H1B···O1	0.90	2.01	2.886(10)	163	N3—H3A···O14	0.90	2.39	3.056(9)	131
N2—H2A···O3 ⁱⁱ	0.90	2.47	3.053(11)	123	N3—H3B···O5 ^{iv}	0.90	1.93	2.817(10)	166
N2—H2A···O4 ⁱⁱ	0.90	2.36	3.223(11)	160	N4—H4A···O7 ⁱⁱⁱ	0.90	1.91	2.810(10)	175
N2—H2B···O16 ⁱⁱ	0.90	2.40	3.096(11)	134	N4—H4B···O9 ^v	0.90	2.00	2.857(10)	158
N2—H2B···O5 ⁱⁱⁱ	0.90	2.48	3.040(10)	120					

Symmetry operations: ⁱ $x-1, y, z$; ⁱⁱ $x, y+1, z$; ⁱⁱⁱ $-x+1, -y+2, -z+1$; ^{iv} $-x+1, -y+1, -z+1$; ^v $x+1, y+1, z$.

gen bonds (Table 1). The cation **B** is linked to $\text{Cr}_3\text{O}_{10}^{2-}$ and $\text{Cr}_2\text{O}_7^{2-}$ by $D(3)$ and $R_1^2(4)$ motifs via trifurcated $\text{N3—H3A}\cdots\text{O3(O12,O14)}$, and cation **A** is linked to $\text{Cr}_2\text{O}_7^{2-}$ anion by $D'(3)$ motif via $\text{N1—H1B}\cdots\text{O1}$. Water molecule is linked to $\text{Cr}_3\text{O}_{10}^{2-}$ anion with $D''(3)$ motif through $\text{Ow—HwA}\cdots\text{O17}$ hydrogen bonds (Fig. 1) [22, 23]. Each of the components occupies a general position.

Discrete inversely oriented $(\text{H}_2\text{Bppp})^{2+}$ cations are arranged in double (X, X') columns running along the a axis (Fig. 2). Trichromate anions and water molecules are linked through $D''(3)$ motifs via $\text{Ow—HwA}\cdots\text{O17}$ hydrogen bond into $\{\text{Cr}_3\text{O}_{10}^{2-}\cdots\text{H}_2\text{O}\}_n$ columns running along the a axis (Figs. 2 and 3), which are arranged approximately in $z = 1/4 + n/2$ (n is integer) planes. The dichromate anions are stacked in columns along the same direction (Fig. 2), which are arranged approximately in $z = n/2$ (n is integer) planes. Fig. 2 shows that every such column is surrounded by four cationic columns, whereas each anionic $\{\text{Cr}_3\text{O}_{10}^{2-}\cdots\text{H}_2\text{O}\}_n$ column is surrounded only by three cationic columns.

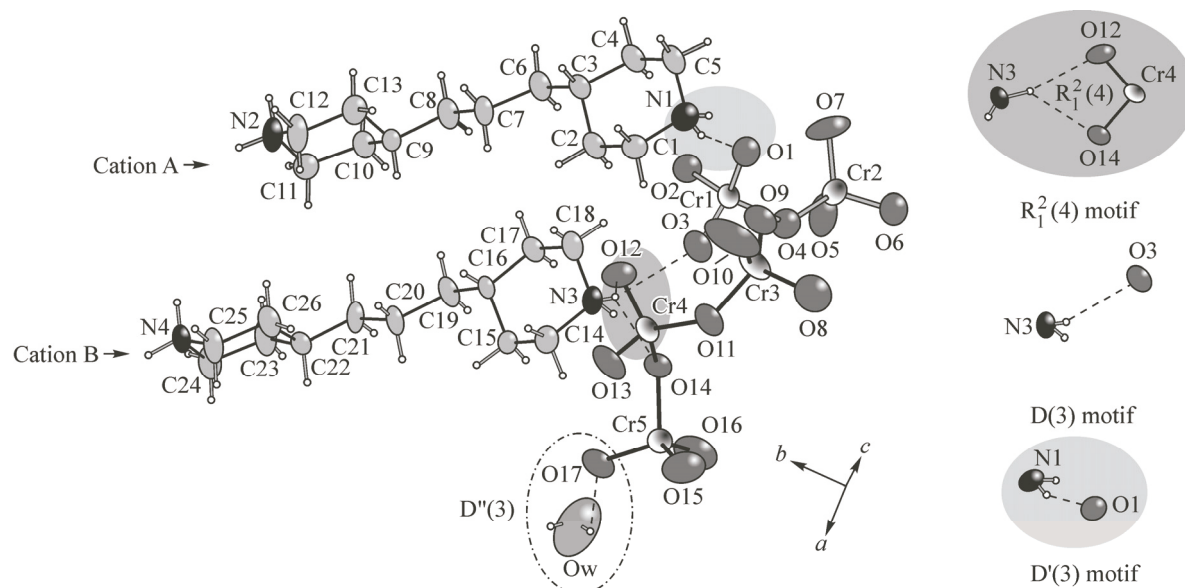


Fig. 1. The molecular structure of **1** showing the formation of $R_1^2(4)$, $D(3)$, $D'(3)$ and $D''(3)$ hydrogen-bonding motifs.

Displacement ellipsoids are shown at the 50% probability level. The hydrogen bonds are indicated by a dashed line

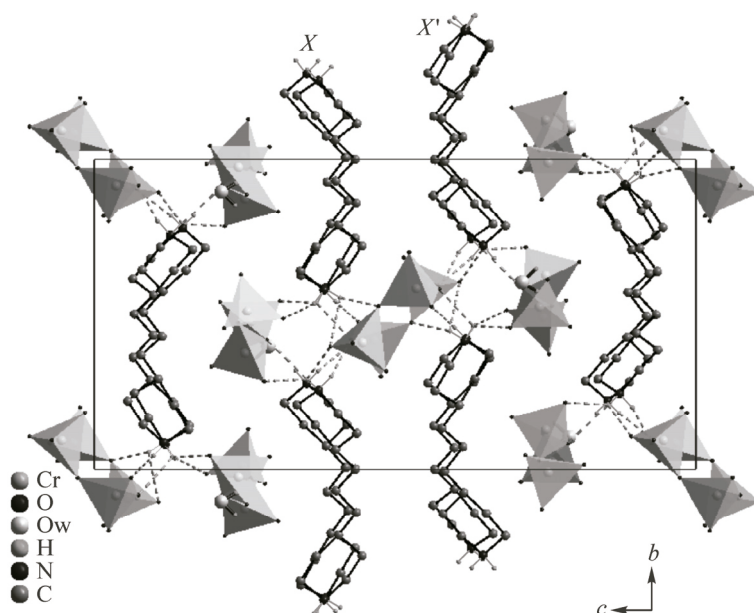


Fig. 2. Projection of the structure of **1** along *a* axis showing N—H···O hydrogen bonds (dashed lines). Hydrogen atoms attached to carbon atoms have been omitted for clarity

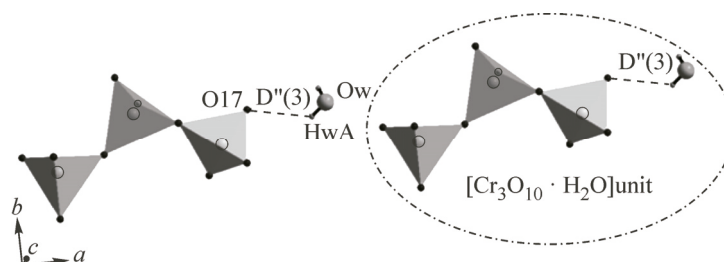


Fig. 3. Part of the crystal structure of **1**, showing the formation of $D''(3)$ hydrogen-bonding motif

These organic and inorganic columns are interconnected into a complicated two-dimensional hydrogen-bonded network *via* normal, bifurcated and trifurcated N—H···O moderate hydrogen bond (Table 1, Fig. 4) [24].

In addition to the motifs $R_1^2(4)$, $D(3)$, $D'(3)$, $D''(3)$ and the rings $R_1'^2(4)$ formed between $\text{Cr}_2\text{O}_7^{2-}$ anion and cation **A** *via* bifurcated $\text{N}2\text{—H}2\text{A}\cdots\text{O}3^{\text{ii}}(\text{O}4^{\text{ii}})$ hydrogen bonds (Fig. 4), a supramolecular rings, which can be described by the graph-set motif $R_4^4(32)$, are built *via* $\text{N}1\text{—H}1\text{B}\cdots\text{O}1$, $\text{N}2\text{—H}2\text{B}\cdots\text{O}5^{\text{iii}}$, $\text{N}3\text{—H}3\text{A}\cdots\text{O}3$ and $\text{N}4\text{—H}4\text{A}\cdots\text{O}7^{\text{iii}}$ hydrogen bonds interactions plays an important roles in forming of the structure (Fig. 4). Moreover, these motifs are reinforced by $\text{N}2\text{—H}2\text{B}\cdots\text{O}16^{\text{iv}}$, $\text{N}4\text{—H}4\text{B}\cdots\text{O}9^{\text{iv}}$, $\text{N}3\text{—H}3\text{B}\cdots\text{O}5^{\text{iv}}$ and $\text{N}1\text{—H}1\text{A}\cdots\text{Ow}^{\text{i}}$ hydrogen bonds giving rise to different graph-set motifs and generates a complicated two-dimensional network (Fig. 2). Furthermore, this structure is stabilized by a large number of C—H···O interactions, thus establishing a three-dimensional network structure.

The $(\text{H}_2\text{bppp})^{2+}$ cations **A** and **B** adopt the anti-anti (*T-T*) conformation, which is the most stable [25], with dihedral angles of $\{-175.9(7)^\circ$ ($\text{C}3\text{—C}6\text{—C}7\text{—C}8$) and $176.0(7)^\circ$ ($\text{C}9\text{—C}8\text{—C}7\text{—C}6$) and $\{171.7(7)^\circ$ ($\text{C}16\text{—C}19\text{—C}20\text{—C}21$) and $-178.0(7)^\circ$ ($\text{C}22\text{—C}21\text{—C}20\text{—C}19$) and N-to-N distances of $10.674(17)$ Å ($d_{\text{N}1\cdots\text{N}2}$) and $10.58(1)$ Å ($d_{\text{N}3\cdots\text{N}4}$), respectively. These results are in good agreement with those found for same species in other structures [25—27].

In both cations **A** and **B**, the cyclic six-membered piperidinium rings exist in 4C_1 chair conformation [28] with the central $\text{—}(\text{CH}_2)_3\text{—}$ spacer unit occupying equatorial sites, where the letter *C* stands

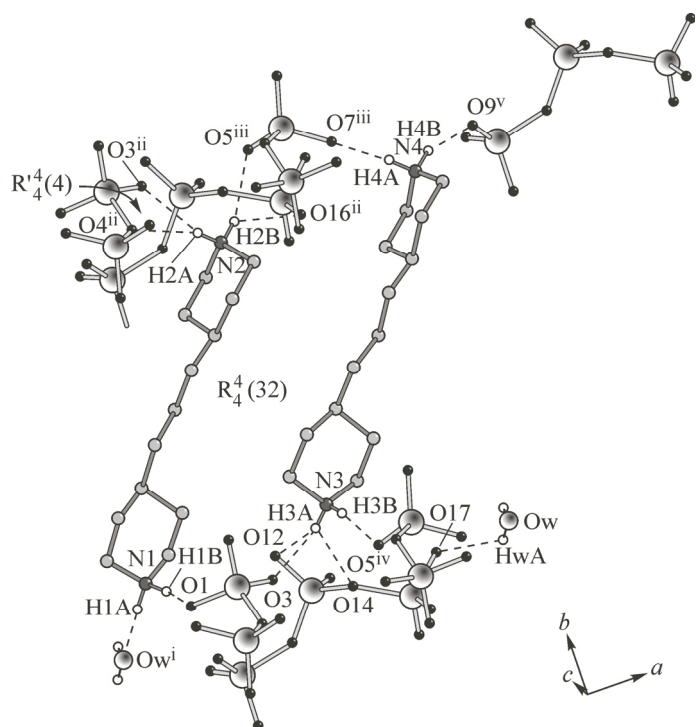


Fig. 4. Part of the crystal structure of **1**, showing N—H...O hydrogen bonds (dashed lines)

for 'Chair' and the numbers indicate the carbon atoms located above or below the reference plane of the chair, made up 2nd C, 3rd C, 5th C and the ring nitrogen (Fig. 5).

The trichromate ion is composed of a chain of three CrO₄ tetrahedra, joined by shared corners. The coordination modes of Cr^{VI} in the central Cr(4)O₄ and the terminal Cr(3)O₄ and Cr(5)O₄ tetrahedra are 2+2 and 3+1, respectively. Indeed, in the terminal tetrahedra, the Cr—O_t bond lengths are shorter than the Cr—O_b bonds, the mean lengths being 1.601(8) and 1.834(6) Å, respectively, with a low dispersion around these mean values: the Cr—O_t distances range from 1.565(8) to 1.604(8) Å, and the Cr—O_b distances range from 1.825(6) to 1.843(6) Å. In the

inner tetrahedron the Cr—O_t range is comparable to those of the terminal tetrahedra while the Cr—O_b distances are longer. For these three tetrahedra, the O—Cr—O angles range from 104.6(4)° to 112.7(5)°, and thus deviate from the ideal value (109.5°). These results are in good agreement with those reported in the literature for organic trichromates.

On the other hand, the trichromate anion exhibits dissimilar Cr—O—Cr angles (141.5(4)° and 126.0(3)°), like related compounds, e.g., tetramethylammonium trichromate (C₄H₁₂N)₂Cr₃O₁₀ (133.8° and 149.1°) [29] and imidazolium linked cyclophane tichromate C₂₀H₂₀N₆Cr₃O₁₀ (137.6(5)° and 127.2(5)°) [8] and thus differs from other organic trichromates, e.g. guanidinium trichromate (CH₆N₃)₂Cr₃O₁₀ (132.7(11)°) [5], dipyrazinium trichromate (C₄H₅N₂)₂Cr₃O₁₀ (137.3(1)° and 137.5(2)°) [6], and 1,4-diazabicyclo[2.2.2]octane-1,4-dium trichromate (C₆H₁₄N₂)[Cr₃O₁₀] (127.34(12)° and 132.80(12)°) [9].

The Cr₂O₇ group, which is built up from Cr(1)O₄ and Cr(2)O₄ tetrahedra, shows a staggered conformation with O2—Cr1—Cr2—O7 torsion angles of 62.7(4)°. The Cr—O—Cr angle equal to 130.6(4)° is comparable with Cr—O—Cr angles in other organic dichromates [30–37].

Nevertheless, the calculated values of the distortion indices (DI) [38] corresponding to the different angles and distances in each CrO₄ tetrahedron of Cr₂O₇ and Cr₃O₁₀ units show a slight distortion

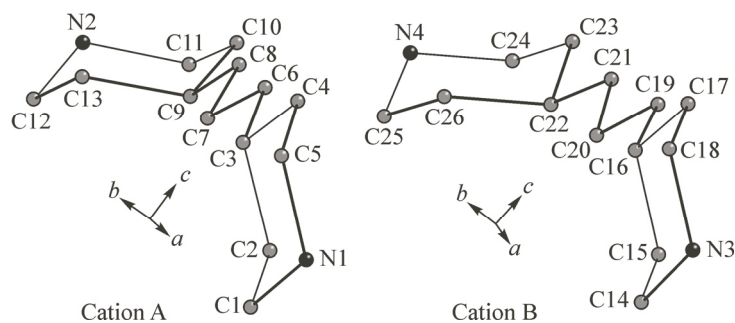


Fig. 5. ⁴C₁ conformation of piperidinium rings in the structure of **1**

Table 2

DI for the coordination polyhedral around Cr in the structure of 1

Tetrahedron	(Cr—O) _m , Å	(O...O) _m , Å	(O—Cr—O) _m , deg.	DI(Cr—O)	DI(O...O)	DI(O—Cr—O)
Cr(1)O ₄	1.650(7)	2.696(9)	109.5(4)	0.0367	0.0241	0.0123
Cr(2)O ₄	1.651(7)	2.697(10)	109.5(4)	0.0465	0.0284	0.0084
Cr(3)O ₄	1.640(7)	2.681(10)	109.5(5)	0.0563	0.0340	0.0105
Cr(4)O ₄	1.652(7)	2.698(9)	109.5(4)	0.0468	0.0256	0.0146
Cr(5)O ₄	1.662(7)	2.714(10)	109.4(4)	0.0546	0.0278	0.0155

$$DI(\text{Cr—O}) = \sum_{i=4}^4 \frac{|(\text{Cr—O})_i - (\text{Cr—O})_m|}{4(\text{Cr—O})_m}, DI(\text{O—O}) = \sum_{i=4}^6 \frac{|(\text{O—O})_i - (\text{O—O})_m|}{6(\text{O—O})_m}, DI(\text{O—Cr—O}) = \sum_{i=4}^6 \frac{|(\text{O—Cr—O})_i - (\text{O—Cr—O})_m|}{6(\text{O—Cr—O})_m}. \text{ The subscripts m refer to the mean and i to individual values.}$$

of the O—Cr—O angles if compared to O...O and Cr—O distances with an above distortion of the Cr—O distances compared to the O...O distances (Table 2). Thus, tetrahedra in the dichromate and trichromate anions exhibit a compact assembly of oxygen atoms in which the chromium atom shows a slight displacement from the gravity center. In spite of this apparent large distortion inside these anions, the average Cr—O distance never departs significantly from 1.651(7) Å; similarly the O—Cr—O average angle is always very close to the ideal value of 109.5°.

Bond valence sum (BVS) calculation, using the empirical formula of Brown and Altermat [39], resulted in values 6.01, 6.00, 6.29, 6.01, and 5.93 for Cr1, Cr2, Cr3, Cr4, and Cr5, respectively. These BVS values are in agreement with the oxidation state of Cr atoms.

Among organic phosphates, vanadates, arsenates or silicates, no representatives of mixed (M₂O₇) and (M₃O₁₀) units are so far known. A search in the Inorganic Crystal Structure Database (ICSD) reveals that only five inorganic compounds containing (M₂O₇) and (M₃O₁₀) groups with the ratio 1:1 have been structurally characterized [40]. These compounds show no structural similarities to our new chromate. Furthermore, only one salt combining (H₂bppp)²⁺ organic cation and an inorganic oxyanion, (H₂Bppp)SO₄·H₂O [26] was found in literature. This compound is structurally different to the title chromate.

However, very few similarities can be noticed with (H₂Bppp)₂[(Bi₂I₉)(BiCl₂I₂)] [27]. In both compounds, discrete inorganic and organic species are stacked in columns running along the same direction. In addition, (H₂bppp)²⁺ organic cations adopt the *T-T* conformation with comparable dihedral angles and N...N distances. In spite of this similarity of organic cations conformation, these species present different arrangement in these structures. In the title compound inversely oriented cations are arranged in double (X, X') columns while in the 'homologous compound' they are orthogonal and arranged in simple columns. This is the result of the great difference of chemical nature of anionic networks which generate differently hydrogen bonds scheme.

CONCLUSIONS

Crystals of (C₁₃H₂₈N₂)₂[Cr₂O₇][Cr₃O₁₀]·H₂O have been grown by slow solvent evaporation method at room temperature from chromium trioxide and 1,3-bis(4-piperidyl)propane (Bppp) in aqueous solution. This compound has a monoclinic centrosymmetric structure built up from a succession of double organic columns formed of discrete inversely oriented (H₂Bppp)²⁺ cations alternated with two type of inorganic columns made of discrete dichromate anions and [Cr₃O₁₀·H₂O] units, respectively, running along the [100] direction. These organic and inorganic columns are interconnected into a complicated two-dimensional hydrogen-bonded network *via* N—H...O and O—H...O hydrogen bonds. Furthermore, this structure is stabilized by a large number of C—H...O interactions, thus

establishing a three-dimensional network structure. Thus, to the best of our knowledge, $(C_{13}H_{28}N_2)_2[Cr_2O_7][Cr_3O_{10}] \cdot H_2O$ is the first representative of a new type of mixed-anion chromates including dichromate and trichromate anions.

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