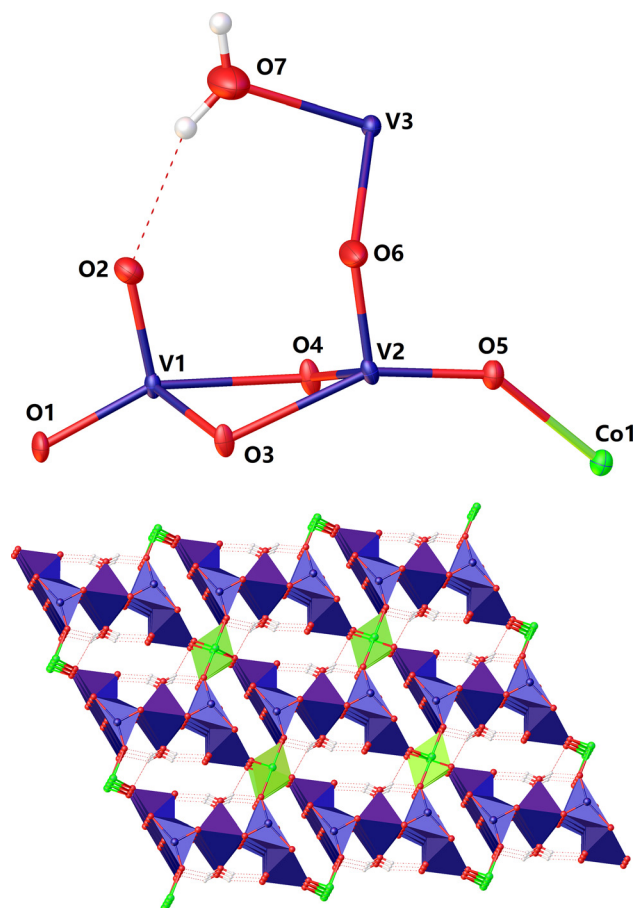


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The crystal structure of a cobalt-vanadium-oxido hydrate



Abstract

$\text{H}_4\text{O}_{14}\text{CoV}_{4.4}$, monolinic, $P2_1/m$ (no. 10), $a = 8.1091(8) \text{ \AA}$, $b = 3.5128(2) \text{ \AA}$, $c = 9.7590(9) \text{ \AA}$, $\beta = 111.553(11)^\circ$, $V = 258.55(4) \text{ \AA}^3$, $Z = 1$, $R_{\text{gt}}(F) = 0.0401$, $wR_{\text{ref}}(F^2) = 0.0922$, $T = 293(2) \text{ K}$.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

A mixture containing $(\text{NH}_4)\text{VO}_3$ (3.850 g), $\text{CoSO}_4 \cdot 6\text{H}_2\text{O}$ (2.750 g), 1,10-phenanthroline ($\text{C}_{12}\text{H}_8\text{N}_2$), (2.650 g), and deionized water (H_2O) (18.0 mL), with pH value ~ 8.0 , was prepared by mixing these components and sealed in a 30 mL Teflon-lined stainless steel autoclave (75 % of the total volume of the autoclave). Then, the resulting slurry was heated to 433 K in an oven and maintained at the temperature for five days. Then, the dark brown purity-phase crystals of the title compound (about 42 % yield based on Co) were obtained. The results of elemental analyses provided the chemical compositions: (wt%) for the title compound: H, 1.06; O, 44.24; Co, 10.67; V, 44.03 (in contrast, anal. calcd. (wt%): H, 0.78; O, 43.83; Co, 11.53; V, 43.86).

2 Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints.

3 Comment

Inorganic porous materials with high surface area and diverse pore structures as well its unique physicochemical properties, such as adsorption performance, chemical stability and thermostability, are endowed with wide range of industrial applications [4–7]. With the rapid development of

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Table 1: Data collection and handling.

Crystal:	Brown needle
Size:	$0.36 \times 0.03 \times 0.02$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	5.47 mm ⁻¹
Diffractometer, scan mode:	New Xcalibur, ω
θ_{max} , completeness:	29.1°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	1232, 708, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 621
$N(\text{param})_{\text{refined}}$:	67
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	1.0000	1.0000	1.0000	0.0117 (3)
V1	0.24584 (13)	0.5000	0.83517 (12)	0.0085 (3)
V2	0.54861 (14)	1.0000	0.81158 (12)	0.0091 (3)
V3 ^a	0.5000	0.5000	0.5000	0.0092 (7)
O1	0.5177 (6)	0.5000	0.8597 (5)	0.0148 (8)
O2	0.3278 (5)	1.0000	0.8811 (5)	0.0125 (8)
O3	0.7663 (6)	1.0000	0.8447 (5)	0.0144 (8)
O4	0.0792 (5)	0.5000	0.9009 (5)	0.0133 (8)
O5	0.1469 (6)	0.5000	0.6596 (5)	0.0176 (9)
O6	0.4616 (6)	1.0000	0.6332 (5)	0.0230 (9)
O7	0.2285 (8)	0.5000	0.4023 (6)	0.0387 (14)
H7A	0.168 (11)	0.5000	0.455 (9)	0.058*
H7B	0.187 (10)	0.5000	0.312 (2)	0.058*

^aOccupancy: 0.4.

science and technology, the application of inorganic porous materials in many fields has also shown a more extensive and in-depth trend, including but not limited to catalysis, separation, gas storage, sensing, electrochemistry, energy conversion and so on [8, 9]. Therefore, the development of new structural inorganic porous materials and the exploitation of its applications will be of a great significance work. At present, the research of inorganic porous materials mainly focus on the improvement of preparation methods exploring the relationship between structure and performances, and the exploration of new application fields [10–12].

Single-crystal X-ray diffraction structure study reveals that the title compound has open 3D-network structure. The asymmetric unit of the compound contains dimeric V–O polyanion, $[\text{V}_2\text{O}_6]^{2-}$, bridging Co^{2+} cation and bridging $[\text{V}(\text{H}_2\text{O})]$ unit. Dimeric $[\text{V}_2\text{O}_6]^{2-}$ polyanion is made up of two similar triangle pyramidal building units, $[\text{VO}_3]$, via two-bridging oxygen atoms. The V atoms in $[\text{V}_2\text{O}_6]^{2-}$ are in five-coordinated state and show tetragonal-pyramidal configuration, $[\text{VO}_5]$, with the bond distances (V–O) of 1.603(5) – 2.125(4) Å for V(1) and 1.624(5) – 2.134(4) Å for V(2). The O–V–O bond angles in $[\text{V}_2\text{O}_6]^{2-}$ are in the range of

73.22(13) – 153.3(2)°. The other bridging vanadium atom, V(3) is in six-coordinated state with bond distances (V–O) of 2.053(6) – 2.274(3) Å and the O–V–O bond angles in the range of 78.85(16) – 180.0°. The Co atoms in the title compound are also in six-coordinated state with the $[\text{CoO}_6]$ unit showing a deformed octahedral configuration, the bond lengths (Co–O) vary in the range of 1.945(5) – 2.211(3) Å, and the O–Co–O band angles in the range of 74.81(17) – 180.0°. The 3D title compound displays a ordered mesoporous structure with the aperture about 20 nm, viewed along *b*-axis. Bond valence calculations (BVS) [13] on V(1), V(2), V(3) and Co(1) sites afford values of 5.131, 5.185, 5.345 and 1.996, respectively.

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