Cd(VO₂)₄(SeO₃)₃·H₂O: A New Bimetallic Vanadium Selenite Compound with Heptacoordinated Cadmium Ion

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A new bimetallic selenite compound, **Cd(VO₂)₄(SeO₃)₃·H₂O**, with vanadium and cadmium metals was synthesized from hydrothermal reactions of V₂O₅, Cd(NO₃)₂·2H₂O, and SeO₂ in water at 230°C. This compound crystallizes in a triclinic unit cell with the space group *P*$\bar{1}$ (No. 2) and the lattice parameters $a = 6.2999(4)$ Å, $b = 7.4078(4)$ Å, $c = 15.7525(9)$ Å, $\alpha = 100.881(1)°$, $\beta = 98.796(1)°$, $\gamma = 93.690(1)°$, $V = 710.14(7)$ Å³, $Z = 2$. The crystal structure was determined by X-ray single-crystal diffraction techniques and was refined with $R = 0.0336$ and $wR_2 = 0.0819$ for all 3175 reflection data. The structure can be described as layers with pillars between the layers. The layer is composed of dimers of VO₅ square pyramids and dimmers of CdO₇ decahedra connected to one another by sharing corner and/or edge oxygen atoms, and the pillars are composed of VO₆ distorted octahedra connected to one another with capping SeO₃²⁻ selenite groups. The coordination number of Cd atom in the structure shows a rare example of hepta-coordination with only oxygen donor atoms   © 2001 Academic Press

Key Words: hydrothermal synthesis; bimetallic selenite; vanadium cadmium selenite; crystal structure; heptacoordinated cadmium.

INTRODUCTION

Metal selenites form a large group of compounds with diverse structures (1, 2). Parts of the reasons for the interest in selenites are their possible structural resemblance to the larger group of metal phosphonates and phosphates and the desire to understand the nature of the lone pair of the Se atom in the selenite group. While the majority of the sele-

nites are with mono-, di-, and trivalent metal atoms, new selenite compounds with metal atoms of groups 5 and 6 have been reported mostly during the past decade (3–8), notably with vanadium (9–18). Our group also has synthesized some vanadium selenites such as AV₂SeO₇ ($A = $ K (14) and Rb (15)), AVSeO₅ ($A = $ Rb, Cs), AV₃Se₂O₁₂ ($A = $ K, Rb, Cs, NH₄) (16), K(VO)(SeO₃)₂H (17), and V₂Se₂O₉ (18). A general feature of these vanadium selenites is that the vanadium and the selenite group form frameworks with alkali or alkaline earth metal cations to compensate the negative charges of the frameworks. A logical way of extending the exploratory syntheses of the vanadium selenites would be bimetallic selenite compounds with vanadium and another metal atoms both in the frameworks. Toward this goal a combination of Cd and V appears to be promising because there are a number of cadmium selenite compounds reported in the literature (19). There are a few bimetallic selenites with a combination of mono-, di-, and trivalent metal atoms reported in the literature (20, 21), but none with vanadium.

In this paper, we report the synthesis and crystal structure of a new bimetallic selenite compound, Cd(VO₂)₄(SeO₃)₃·H₂O, with both Cd and V in the 3D framework. The details of the synthesis and crystal structure are given in the following.

EXPERIMENTAL

Synthesis

In a typical high-yield reaction for the titled compound, V₂O₅, SeO₂, and Cd(NO₃)₂·H₂O, in a molar ratio 2:20:5, were placed in a Teflon container of 20 mL volume. Distilled water (10 mL) was added to make the degree of fill about 80%. The solution pH was 0.42 immediately after preparing the V₂O₅ suspended solution. The container was placed in

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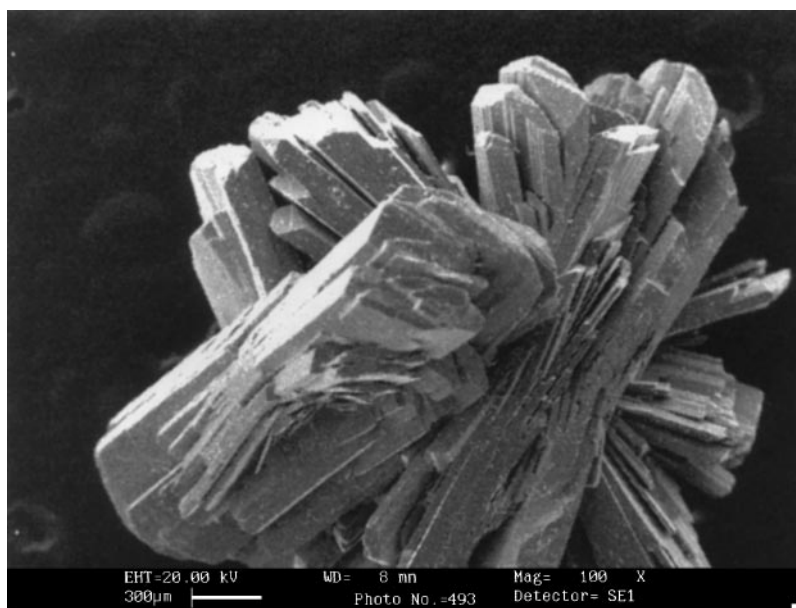
FIG. 1. Scanning electron microscope image of $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals.

a home-made autoclave, which was placed in a 230°C oven. After the hydrothermal reaction for 3 days, the autoclave was cooled to room temperature, and the brown crystalline products were recovered by filtration, washing with distilled water, and air-drying. The scanning electron microscope (SEM) image in Fig. 1 of these crystals shows plate-like morphology. Typical yield of the reaction was over 85% based on V. The results of other attempted syntheses in this system with different solution compositions and temperatures are summarized in Table 1.

Single Crystal X-ray Structure Determination

A crystal (approximate dimensions $0.15 \times 0.10 \times 0.10 \text{ mm}^3$) was placed onto the tip of a 0.1-mm diameter glass capillary and mounted on a Bruker SMART system for a data collection at 173(2) K. A preliminary set of cell constants indicating a triclinic unit cell was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 120 reflections. The data collection was carried out using MoK_α radiation (graphite monochromator) with a frame time of 20 s and a detector distance of 4.92 cm. A randomly oriented region of reciprocal space was surveyed to the extent of 1.5 hemispheres and to a resolution of 0.77 Å. Five major sections of frames were collected with 0.30° steps in ω at four different ϕ settings and a detector position of $-28°$ in 2θ. The

TABLE 1
Hydrothermal Reactions in the Cd^{2+}–V_2O_5–SeO_3^{2-} System

Reaction No.	Reagents (mmol)				Temperature (°C)	Results
	$Cd(NO_3)_2 \cdot 2H_2O$	V_2O_5	SeO_2	H_2O (mL)		
1	5	2	20	10	230	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals
2	10	2	20	10	230	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals
3	5	1	20	10	230	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals
4	10	2	20	10	200	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals
5	5	1.5	20	10	215	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals
6	5	1	20	10	215	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals and $CdSeO_3$ microcrystals
7	10	1	20	10	230	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ and $CdSeO_3$ single crystals
8	10	1	20	10	215	$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ and $CdSeO_3$ single crystals
9	5	2	10	10	230	V_2O_5 powder
10	10	2	10	10	230	V_2O_5 powder

TABLE 2
Crystal Data and Structure Refinement for Cd(VO₂)₄(SeO₃)₃·H₂O

Empirical formula	$H_2CdO_{18}Se_3V_4$
Formula weight	843.06
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	$a = 6.2999(4)$ Å $\alpha = 100.881(1)°$
	$b = 7.4078(4)$ Å $\beta = 98.796(1)°$
	$c = 15.7525(9)$ Å $\gamma = 93.690(1)°$
Volume	710.14(7) Å³
Z	2
Density (calculated)	3.943 Mg/m³
Absorption coefficient	11.799 mm⁻¹
F(000)	776
Crystal size	$0.15 \times 0.10 \times 0.10$ mm
Theta range for data collection	2.67° to 27.52°
Index ranges	$-8 \leq h \leq 8, -9 \leq k \leq 9,$
	$0 \leq l \leq 20$
Reflections collected	3175
Independent reflections	3175 [R(int) = 0.0285]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3175/0/235
Goodness-of-fit on F^2	1.096
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0315, wR₂ = 0.0808
R indices (all data)	R1 = 0.0336, wR₂ = 0.0819
Largest diff. peak and hole	2.555 and -1.392 e Å⁻³

TABLE 3
Atomic Coordinates and Equivalent Isotropic Displacement Parameters for Cd(VO₂)₄(SeO₃)₃·H₂O

	Atomic coordinates ($\times 10^4$)			
	x	y	z	$U(eq)$ (Å² $\times 10^3$)
Cd(1)	3179(1)	7988(1)	4230(1)	10(1)
Se(1)	1802(1)	7241(1)	571(1)	8(1)
Se(2)	7944(1)	9682(1)	3714(1)	7(1)
Se(3)	10039(1)	4649(1)	2283(1)	8(1)
V(1)	8395(1)	6199(1)	4814(1)	10(1)
V(2)	5413(1)	4561(1)	2918(1)	7(1)
V(3)	4352(1)	3540(1)	612(1)	7(1)
V(4)	6731(1)	7708(1)	1659(1)	7(1)
O(1)	5831(6)	5842(5)	4083(2)	12(1)
O(2)	1463(6)	6108(5)	4979(2)	14(1)
O(3)	3880(6)	9913(5)	5627(2)	10(1)
O(4)	$-522(5)$	8248(5)	4226(2)	11(1)
O(5)	2506(5)	5453(5)	2914(2)	11(1)
O(6)	2955(6)	7318(5)	$-338(2)$	10(1)
O(7)	3723(6)	8268(5)	1447(2)	10(1)
O(8)	1886(5)	4984(5)	681(2)	10(1)
O(9)	6366(5)	8045(5)	2873(2)	8(1)
O(10)	8567(6)	4594(5)	3108(2)	12(1)
O(11)	9440(5)	6664(5)	1972(2)	9(1)
O(12)	5430(5)	4683(5)	1788(2)	10(1)
O(13)	3774(5)	3652(5)	$-567(2)$	9(1)
O(14)	7879(6)	9636(5)	1596(3)	14(1)
O(15)	7857(6)	7407(5)	5678(2)	17(1)
O(16)	4832(6)	2441(5)	2900(2)	14(1)
O(17)	3107(6)	1669(5)	692(2)	14(1)
OW	2061(6)	9495(5)	3092(2)	13(1)

intensity data were corrected for absorption and decay (SADABS) (22).

Final cell constants ($a = 6.2999(4)$ Å, $b = 7.4078(4)$ Å, $c = 15.7525(9)$ Å, $\alpha = 100.881(1)°$, $\beta = 98.796(1)°$, $\gamma = 93.690(1)°$, $V = 710.14(7)$ Å³) were calculated from 5054 strong reflections from the actual data collection after integration (SAINT 6.01) (23). The structure was solved using SHELXS-86 (24) and refined using SHELXL-97 (25). The space group $P\bar{1}$ was determined based on the lattice parameters and intensity statistics and was confirmed by the final structure refinement results. A direct-methods solution was calculated which provided most of the atomic positions from the E-map. Full-matrix least squares/difference Fourier cycles were performed which located the remaining atoms except for the two hydrogen atoms of the water molecule. All atoms in the atom list were refined with anisotropic displacement parameters. The final full matrix least squares refinement converged to $R = 0.0336$ and wR₂ = 0.0819 (F_o^2, all data). The crystallographic data are summarized in Table 2. Important atomic parameters, important bond distances with bond valence sum calculations are given in Tables 3 and 4, respectively. Bond angles around Cd ion are summarized in Table 5.

Characterization

Thermogravimetric analysis was performed with a Perkin-Elmer TGA-7 under a N₂ flow from room temper-

ature up to 700°C. Weight losses of 2.78 and 40.68% were observed below 200°C by one step and in the 320–520°C range by two steps, corresponding to the theoretical values of 2.14 and 39.48% for water and SeO₂ losses based in the stoichiometry determined from the X-ray single-crystal structure.

Infrared absorption spectrum was recorded on a Nicolet 205 FT-IR spectrometer using the KBr pellet method. Scanning electron microscopic images were obtained with a Hitachi S-4200 scanning electron microscope. The atomic ratio from the EDX data was Cd : V : Se = 1 : 3.8 : 3.2 in good agreement with the single crystal structure data 1 : 4 : 3.

RESULTS AND DISCUSSION

Synthesis

The attempted syntheses and their results in the present system are summarized in Table 1. The synthesis results depend on the composition of the solutions and reaction temperature. The optimal conditions for the synthesis of Cd(VO₂)₄(SeO₃)₃·H₂O single crystals we have found (reactions 1–3 in Table 1) are described order Experimental. Large excess of Cd(NO₃)₂ and SeO₂ in the composition of

TABLE 4
Important Bond Distances and Bond Valence Sum (BVS) Calculations for $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$

Bond	Distance	BVS	Bond	Distance	BVS
Cd(1)–O(3)#1[a]	2.296(4)	0.3466	V(1)–O(15)	1.578(4)	1.837
O(2)	2.305(4)	0.3383	O(2)#3	1.805(4)	0.9946
OW	2.330(4)	0.3162	O(1)	1.807(4)	0.9892
O(3)	2.346(3)	0.3028	O(2)#2	1.918(4)	0.7329
O(4)	2.351(3)	0.2988	O(4)#2	2.054(4)	0.5074
O(1)	2.386(4)	0.2718		**1.82 ± 0.16**	**5.06**
O(5)	2.483(4)	0.2091			
	2.33(6)	**2.08**	V(2)–O(16)	1.583(4)	1.8123
			O(12)	1.801(4)	1.0054
Se(1)–O(7)	1.709(3)	1.3174	O(1)	1.871(4)	0.8321
O(6)	1.711(4)	1.3103	O(10)	1.962(4)	0.6507
O(8)	1.716(3)	1.2927	O(5)	1.986(3)	0.6098
	1.712(3)	**3.92**		**1.84 ± 0.14**	**4.91**
Se(2)–O(3)#1	1.667(3)	1.4758	V(3)–O(17)	1.584(4)	1.8074
O(4)#2	1.712(3)	1.3068	O(13)	1.857(3)	0.8642
O(9)	1.746(3)	1.1921	O(12)	1.886(4)	0.7991
	1.71(3)	**3.97**	O(6)#5	1.931(4)	0.7076
			O(8)	1.947(3)	0.6776
Se(3)–O(11)	1.704(3)	1.3353	O(13)#5	2.343(4)	0.2324
O(10)	1.714(4)	1.2997		**1.92 ± 0.22**	**5.09**
O(5)#2	1.716(4)	1.2927			
	1.711(5)	**3.93**	V(4)–O(14)	1.586(4)	1.7977
			O(13)#5	1.791(3)	1.033
			O(9)	1.933(3)	0.7037
			O(11)	1.952(3)	0.6685
			O(7)	1.958(4)	0.6578
			O(12)	2.392(4)	0.2035
				1.94 ± 0.24	**5.06**

[a]Symmetry transformations used to generate equivalent atoms: #1, $-x+1, -y+2, -z+1$; #2, $x+1, y, z$; #3, $-x+1, -y+1, -z+1$; #4, $-x+2, -y+1, -z+1$; #5, $-x+1, -y+1, -z$; #6, $x-1, y, z$.

the crystals was required for a pure phase synthesis, probably because of higher solubility of these compounds than V_2O_5. As long as the solution composition was kept within the range as in reactions 1–3, the $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals were obtained in high yields and high purity even at lower reaction temperatures (reactions 4 and 5). However, the crystals from these lower temperatures were much smaller than those from the 230°C reactions and were not suitable for single-crystal structure analysis. Reducing the V_2O_5 content in the starting solutions results in mixtures of colorless crystals and $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals (reactions 6–8). The impurity phase was determined to be the orthorhombic $CdSeO_3$ by single-crystal X-ray diffraction reported in the literature (19). On the other hand, when the reactions were run with smaller amount of SeO_2, unreacted orange-colored V_2O_5 powder was recovered as the major product (reactions 9 and 10).

The variations of composition of the reaction mixture inevitably induce the changes of pH. However, the pH values of the solutions before and after the reactions all were within the range of 0.4–1.0, suggesting that the pH was not a crucial factor for the phase formation. On the other hand, when the initial pH was adjusted to between 1 and 4 by adding KOH, a mixture of $CdSeO_3$ and $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ crystals was obtained. When the initial pH was higher than 4, dark greenish brown $KV_3Se_2O_{12}$ crystals in hexagonal rod-shape (16) were obtained in a high yield.

Crystal Structure

An ORTEP drawing of the crystal structure of the title compound for the asymmetric unit and atom labels is given in Fig. 2. The crystal structure can be described with layers parallel to the ab-plane and columns between the planes (Fig. 3). The plane is composed of dimers of edge sharing VO_5 square pyramids and dimers of edge-sharing CdO_7 decahedra (heptacoordinated Cd^{2+}) connected with each other through corner- and edge-sharing modes (Fig. 4). These V_2O_8 and Cd_2O_{12} dimers have inversion centers at their respective centers so that half of each dimer is inversion related to the other half. The VO_5 square pyramid, therefore, shares an edge with the other (inversion-related) VO_5 within the same dimer, and shares an edge and a corner with the neighboring CdO_7 decahedra. Except the apical oxygen atom (O15), all of the coordinating oxygen atoms are shared with other V and/or Cd atoms. Similarly, the CdO_7 decahedron shares an edge with the other inversion-related CdO_7 and shares another edge and a corner with the neighboring VO_5 square pyramids. Except the water ligand, all of the other oxygen ligands in this layer are shared with the neighboring CdO_7, VO_5, and SeO_3 polyhedra by corner and edge-sharings.

TABLE 5
Bond Angles around Cd Ion in $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$

O(3)#1–Cd(1)–O(2)[a]	142.78(13)	O(2)–Cd(1)–O(1)	88.69(12)
O(3)#1–Cd(1)–OW	79.48(13)	OW–Cd(1)–O(1)	120.03(12)
O(2)–Cd(1)–OW	134.75(13)	O(3)–Cd(1)–O(1)	110.63(12)
O(3)#1–Cd(1)–O(3)	68.55(14)	O(4)–Cd(1)–O(1)	143.92(13)
O(2)–Cd(1)–O(3)	81.35(13)	O(3)#1–Cd(1)–O(5)	117.31(12)
OW–Cd(1)–O(3)	114.36(12)	O(2)–Cd(1)–O(5)	89.33(13)
O(3)#1–Cd(1)–O(4)	133.79(12)	OW–Cd(1)–O(5)	76.71(12)
O(2)–Cd(1)–O(4)	63.43(12)	O(3)–Cd(1)–O(5)	168.76(12)
OW–Cd(1)–O(4)	74.41(12)	O(4)–Cd(1)–O(5)	92.96(12)
O(3)–Cd(1)–O(4)	88.48(12)	O(1)–Cd(1)–O(5)	62.60(12)
O(3)#1–Cd(1)–O(1)	82.27(12)		

[a]Symmetry transformations used to generate equivalent atoms: #1, $-x+1, -y+2, -z+1$; #2, $x+1, y, z$; #3, $-x+1, -y+1, -z+1$; #4, $-x+2, -y+1, -z+1$; #5, $-x+1, -y+1, -z$; #6, $x-1, y, z$.
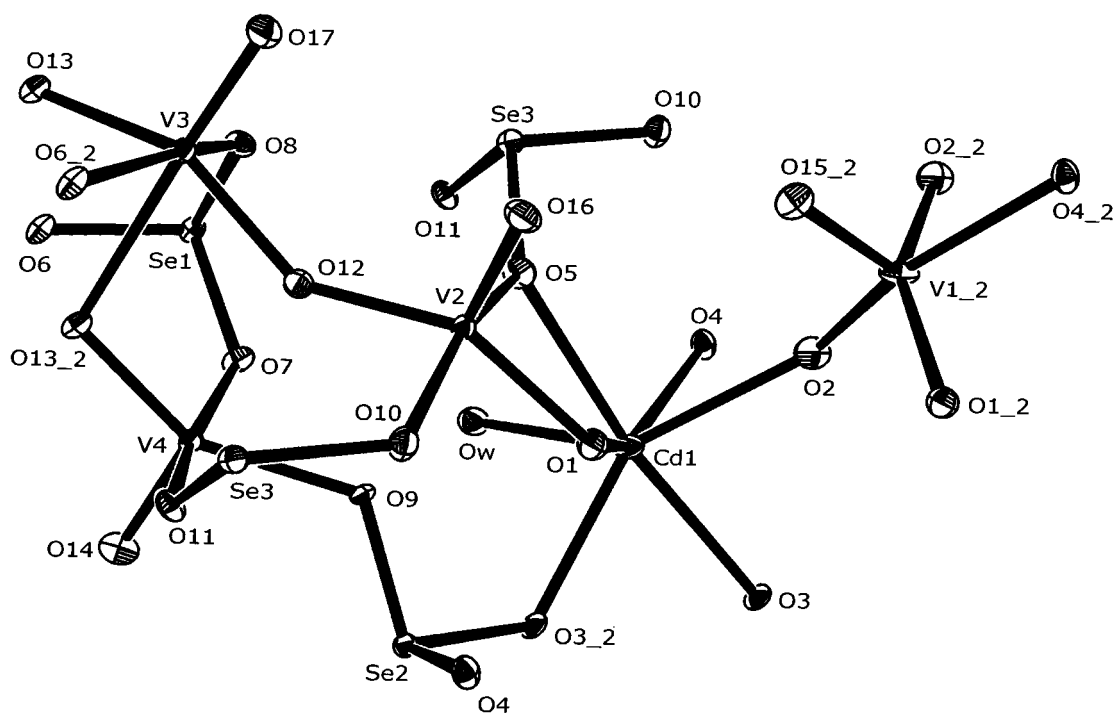
FIG. 2. ORTEP drawing and atom labels of the crystal structure of Cd(VO$_2$)$_4$(SeO$_3$)$_3$·H$_2$O.

The layers described above are connected to the neighboring ones by columns, composed of four edge-sharing VO$_6$ octahedra of V(3) and V(4) and capping SeO$_3$ flattened tetrahedral of Se(1). The square pyramid of V(2) serves as a bridge between the layer and the column.

An interesting feature of the present crystal structure is the hepta-coordination of Cd. While hepta-coordinated Cd compounds are not very unusual because of the large ionic size of Cd^{2+} (26–31), most of such examples are found from organometallic compounds with multidentate ligands and there is no example of oxide compound to the best of our knowledge (27–31). The coordination geometry of the ligands around Cd^{2+} can be described with a triangle of O2, O3, and O4 and a quadrangle of OW, O3_2, O1, and O5, or as a trigonal prism with basal triangles O1-O3-O3_2 and OW-O4-O5 and a capping O2 onto the quadrangular face of O1-O3-O4-O5 (Fig. 2). The bond angles in Table 5 suggest that the structure is heavily distorted from this idealized description.

Important bond distances of Cd(VO$_2$)$_4$(SeO$_3$)$_3$·H$_2$O and the bond valence sum (BVS) calculations (32) therefrom are summarized in Table 4. The BVS calculations show that the valence states of the ions are Cd^{2+}, V^{5+} and Se^{4+}. The Cd–O distances are in the range of 2.296–2.483 Å with an average of 2.36(6) Å. The crystal structure data of CdSeO$_3$ in two different crystal structures and 4CdSeO$_3$·H$_2$O give an average Cd–O distance 2.31(4) Å in the range of

2.238–2.436 Å. Although statistically indistinguishable within the standard deviations, the distances for the heptacoordinated Cd in the present case are slightly longer than those for the hexacoordinated Cd. The Cd–OH$_2$ bond distance in the present compound is 2.330(4) Å, in agreement with the average value of 2.32(7) Å over 73 data (33).

Cd(VO$_2$)$_4$(SeO$_3$)$_3$·H$_2$O has four crystallographically different V in square pyramidal and distorted octahedral geometries. The square pyramidal VO$_5$ units are found in the layer described above and bonded to the Cd atoms. They show typically short V=O in the apices and four long bonds in the bases of the square pyramids. The octahedral VO$_6$ units are in the column. These have a very long V–O bond in addition to the four long and one short distances of the square pyramidal VO$_5$ units. The short V=O bonds are not shared for all cases.

The three crystallographically different Se atoms show some variations of the bond distance patterns. All the three selenite groups bridge three metal atoms. The Se–O distances of Se(1) and Se(3) are more or less homogeneous in the range of 1.704–1.716 Å, while those of Se(2) show a broader range from 1.667(3) to 1.746(3) Å. Similarly distorted SeO$_3$ structures are found from hydrogen selenites, HSeO$_3$ and H$_2$SeO$_3$; the average Se–O and Se–OH bond lengths are 1.672(19) and 1.761(21) Å in HSeO$_3^-$ and 1.645(17) and 1.748(16) Å in H$_2$SeO$_3$, respectively. This different bond distances are probably due to the stronger acidity of
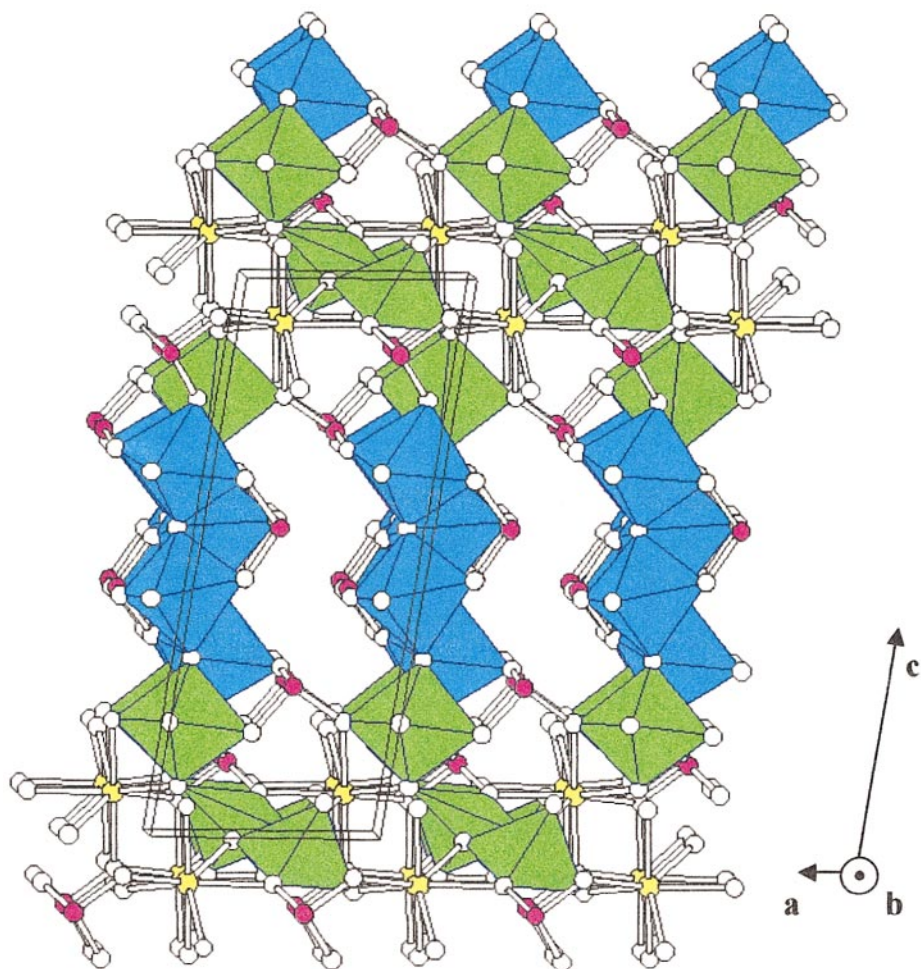
FIG. 3. Crystal structure of $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ viewed along the b direction. Yellow spheres are Cd, red spheres are Se, and white spheres are oxygen atoms. VO_6 octahedra and VO_5 pyramids are colored in blue and green, respectively.
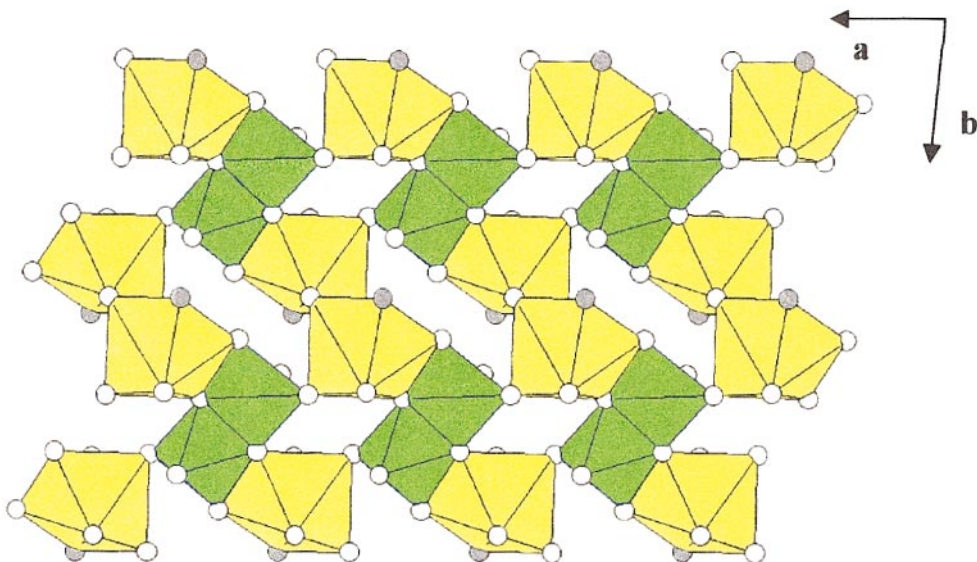
FIG. 4. A section of the crystal structure of $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ to show the layer parallel to the ab-plane of the unit cell. CdO_7 decahedra and VO_5 square pyramids are colored in yellow and green, respectively. Oxygen ligands are shown as open circles and water ligands are shown in gray circles.
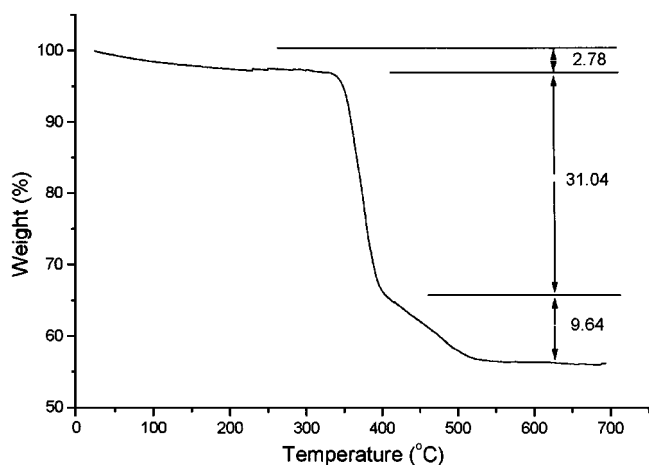
FIG. 5. Thermogravimetric data of $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$.

vibrations of VO_6 and VO_5 groups. The CdO_7 group probably is responsible for the low-frequency peaks at 568 and 521 cm^{-1}.

In conclusion, we have synthesized a novel compound of $Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$. The crystal structure shows two novel features of this compound. First, it is the first example of a bimetallic selenite with vanadium and cadmium. The geometry of the SeO_3 groups shows variations depending on the nature of the metal ions that are bonded to the oxygen atoms. Second, the crystal structure of this compound shows an unusual heptacoordinated Cd^{2+} ion with all the seven oxygen ligand atoms.

H^+ than metal ions (mostly with low valence states) that are bonded to the oxygen atoms. The same argument can be applied to the present case. While the selenite groups of Se(1) and Se(3) are bridging three vanadium atoms, that of Se(2) bridges two vanadium and a cadmium atoms. The higher charge density of vanadium exerts stronger acidity than cadmium, causing the Se–O bond in the Se(2)–O–Cd linkage much shorter than the others.

Physical Properties

$Cd(VO_2)_4(SeO_3)_3 \cdot H_2O$ decomposes in two steps as shown in the thermogravimetric data (Fig. 5). The first weight loss step below about 200°C by 2.78% corresponds to the loss of water coordinated to Cd (theoretical value 2.14%). The second and third weight loss steps from about 320 to 530°C can be explained by the loss of SeO_2. The theoretical value for this step, 39.48%, agrees well with the observed loss of 40.68%. The loss of SeO_2 occurs in two steps while in most of the vanadium selenites the loss of SeO_2 occurs in a single step. Because there are two types of selenite groups with different local environments in 2 to 1 ratio with three neighboring vanadium and two vanadium and one cadmium, it is tempting to interpret these two step losses in these terms. However, the ratio of the weights lost in the two steps do not give a 2:1 ratio, but rather close to a 3:1 ratio, making this interpretation not very strong. Rather, the two-step weight losses are due to the kinetics of decomposition reactions that may involve an intermediate phase.

Infrared spectrum gives absorption peaks characteristic to the functional groups. The assignments of the peaks are as follows: The SeO_3 group gives peaks at 1002 cm^{-1}. The two split peaks at 808 and 783 cm^{-1} are assigned to the symmetric vibrations of VO_6 and VO_5 groups, and the peaks at 692 and 609 cm^{-1} are from the asymmetric

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