# A mixed valence heteropolyvanadate, $K_7Na[As_4^VV_7^VV_5^{IV}O_{43}H_3] \cdot 7H_2O$

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(Received June 6, 1991; revised September 11, 1991)

#### Abstract

The reaction of NaVO<sub>3</sub> with As<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>C<sub>6</sub>O<sub>6</sub> and KSCN in water at pH 4.4 yields shiny black crystals of K<sub>7</sub>Na[As<sub>4</sub><sup>V</sup>V<sub>7</sub><sup>V</sup>V<sub>5</sub><sup>IV</sup>O<sub>43</sub>H<sub>3</sub>]·7H<sub>2</sub>O. The structure consists of an  $\epsilon$ -Keggin core {AsV<sub>12</sub>O<sub>40</sub>} with three [VO] vertices removed and capped by three [VO] and three [AsOH] units. Crystal data: monoclinic C2/c, a = 40.048(9), b=13.326(1), c=18.074(3) Å,  $\beta$ =112.28(1)°, V=9005.9(14) Å<sup>3</sup>, D<sub>calc</sub>=2.986 g cm<sup>-3</sup>, Z=8, R=0.063 for 5999 reflections.

#### Introduction

In addition to the two large  $V^{v}$  isopolyanions,  $[V_{10}O_{28}]^{6-}$  [1] and  $[V_{12}O_{32}]^{4-}$  [2], a number of  $V^{1v}$ containing aggregates have been described [3–11]. Many of these latter complexes are hereroclusters containing arsenic [4, 7, 11] or phosphorus [12, 13] and displaying varying ratios of  $V^{1v}$  to  $V^{v}$  and unique structural types. As part of our investigations of the coordination chemistry of polyoxovanadates, we have isolated and structurally characterized the mixed valence heteropolyvanadate,  $K_7Na[As_4^{v}V_7^{v}V_5^{1v}O_{43}H_3] \cdot 7H_2O$ . The potassium salt of the one-electron oxidized form of this anion  $K_7[As_4^{v}V_8^{v}V_4^{u}O_{43}H_3] \cdot 9H_2O$  is also known and has been cited in a recent review by Pope and Müller [14].

#### Experimental

## **Synthesis**

The complex  $K_7Na[As_4V_{12}O_{43}H_3] \cdot 7H_2O$  was initially synthesized by dissolving NaVO<sub>3</sub> (0.732 g; 6 mmol) and arsenic(III) oxide (0.198 g; 1 mmol) in water (50 ml) and heating to 80 °C. This solution was treated with the disodium salt of rhodizonic acid, Na<sub>2</sub>C<sub>6</sub>O<sub>6</sub> (0.214 g; 1 mmol) and after 10 min with KSCN (1.00 g; 10.3 mmol). The pH of the reaction was adjusted to 4.4 by dropwise addition of dilute sulfuric acid (0.5 N) to give a dark bluish-black solution. After heating at 80 °C for 16 h, the solution was allowed to cool to room temperature and maintained at ambient temperature for 36 h. The green crystalline material of cubic habit which deposited was filtered, and the bright green filtrate allowed to evaporate at room temperature to give shiny black crystals of  $K_7Na[As_4^VV_7^VV_5^{IV}O_{43}H_3]$ . 7H<sub>2</sub>O.

Alternatively, the complex was isolated by adapting the above procedure with the addition of a large excess of KSCN (5.00 g; 51.5 mmol) and the exclusion of  $Na_2C_6O_6$ . The yield is significantly improved by the latter procedure to 40% (based on NaVO<sub>3</sub>). The rhodizonic acid presumably functions as a reducing agent [15] and may be replaced by the mild reductant KSCN.

Crystals of  $K_7$ Na $[As_4V_{12}O_{43}H_3] \cdot 7H_2O$  are indefinitely stable in air. The complex is soluble in warm water to give a bluish-black solution and insoluble in all common organic solvents. Upon heating, the characteristic bluishblack color of aqueous solutions of the complex is discharged to give a mustard yellow solution. IR (KBr pellet, cm<sup>-1</sup>): 3430(s), 1615(m), 972(s), 955(s), 897(s), 855(m), 835(s), 755(w), 719(w), 655(m), 577(w), 473(s). Anal. Calc. for  $H_{17}O_{50}NaK_7V_{12}As_4$ : K, 13.5; Na, 1.14. Found (atomic absorption): K, 13.6; Na, 1.14%. Thermogravimetric analysis was consistent with the presence of 7 water molecules of crystallization. Potentiometric titration revealed five V(IV) centers/formula unit.

#### X-ray crystallography

A crystal of the complex measuring  $0.25 \times 0.26 \times 0.28$  mm was mounted on a glass fiber and carefully centered at -40 °C on a Rigaku AFC5S diffractometer. Data

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TABLE 1. Atomic positional parameters for  $K_7Na$ - $[As_4^vV_7^vV_5^{tv}O_{43}H_3] \cdot 7H_2O$ 

Atom	<i>x</i>	y 2	:
As(1)	0.37000(3)	0.68308(9)	0.40330(7)
As(2)	0.45590(3)	0.5535(1)	0.38540(8)
As(3)	0.31352(3)	0.4375(1)	0.30516(7)
As(4)	0.34653(4)	0.7871(1)	0.18504(8)
V(Ì)	0.34598(5)	0.6919(2)	0.5778(1)
ví2í	0.33897(5)	0.8969(2)	0.4839(1)
v(3)	0.41749(5)	0.8130(2)	0.5928(1)
V(4)	0.40472(6)	0.9315(2)	0.4258(1)
V(5)	0.41950(6)	0.5757(2)	0.5933(1)
V(6)	0.28176(5)	0.7122(2)	0.4031(1)
V(7)	0.33452(5)	0.4900(2)	0.4888(1)
V(8)	0.31134(6)	0.8798(2)	0.3011(1)
V(9)	0.46464(6)	0.7415(2)	0.5001(1)
V(10)	0.28494(5)	0.6660(2)	0.2249(1)
V(11)	0.43264(6)	0.7771(2)	0.3144(1)
<b>V(12)</b>	0.39563(6)	0.4191(2)	0.4342(1)
K(1)	0.37183(8)	0.5290(2)	0.2142(2)
K(2)	1/2	0.6045(5)	1/4
K(3)	0.24131(8)	0.9456(2)	0.4285(2)
K(4)	0.19068(9)	0.6630(3)	0.2916(2)
K(5)	1/2	0.3136(5)	1/4
K(6)	0.5409(1)	0.8278(4)	0.4523(3)
K(7)	0.1945(2)	0.7347(6)	0.0480(3)
K(8)	0.9309(3)	0.7767(8)	0.2987(5)
Na(1)	0.9451(1)	0.5826(5)	0.3244(4)
$\mathcal{I}(\mathbf{I})$	0.3307(2)	0.6553(6)	0.6444(5)
J(2)	0.3204(2)	1.0022(0)	0.4874(5)
$\mathcal{J}(3)$	0.4301(2)	0.8342(7) 1.0482(6)	0.0707(3)
2(4)	0.4141(2) 0.4419(2)	0.5457(6)	0.4415(0)
D(6)	0.9462(2)	0.7307(6)	0.0040(5) 0.4222(5)
D(7)	0.3120(2)	0.4446(7)	0.5383(5)
2(8)	0.2941(2)	0.9890(6)	0.2853(5)
$\mathbf{D}(9)$	0.5016(2)	0.7668(7)	0.5688(5)
D(10)	0.2491(2)	0.6361(6)	0.1522(5)
$\mathcal{D}(11)$	0.4517(2)	0.8279(7)	0.2611(6)
D(12)	0.4145(2)	0.3123(7)	0.4461(5)
D(13)	0.4901(2)	0.4794(6)	0.3795(5)
D(14)	0.2852(2)	0.3440(7)	0.2512(5)
D(15)	0.3466(3)	0.8311(7)	0.0960(5)
D(16)	0.3272(2)	0.8162(6)	0.5527(5)
D(17)	0.3934(2)	0.7342(7)	0.6369(5)
D(18)	0.3872(2)	0.9163(7)	0.5608(6)
D(19)	0.4359(2)	0.8565(6)	0.5082(5)
D(20)	0.4141(2)	0.9022(7)	0.3457(5)
$\mathcal{J}(21)$	0.4717(2)	0.7872(6)	0.4184(5)
J(22)	0.4486(2)	0.6347(6)	0.3114(5)
D(23)	0.3543(2)	0.3880(0)	0.3298(5)
J(24)	0.2993(2) 0.3118(2)	0.4394(0)	0.3809(3)
2(26)	0.3116(2) 0.2986(2)	0.0311(0) 0.8364(6)	0.4782(3) 0.3923(5)
O(27)	0.3568(2)	0.9163(6)	0.3957(5)
D(28)	0.3717(2)	0.5630(6)	0.5737(5)
D(29)	0.4390(2)	0.6866(6)	0.5693(5)
2(30)	0.3689(2)	0.7456(6)	0.4841(5)
D(31)	0.4100(2)	0.7044(6)	0.3964(5)
<b>D</b> (32)	0.3367(2)	0.7228(6)	0.3219(5)
<b>D</b> (33)	0.3643(2)	0.5610(6)	0.4161(5)
D(34)	0.2677(2)	0.6531(6)	0.3140(5)
			. ,

TABLE 1. (continued)

Atom	x	у	z
O(35)	0.2765(2)	0.8086(6)	0.2315(5)
O(36)	0.3384(2)	0.8889(6)	0.2296(5)
O(37)	0.4262(2)	0.4841(7)	0.5351(5)
O(38)	0.4735(2)	0.6017(6)	0.4781(5)
O(39)	0.3624(2)	0.3938(6)	0.4874(5)
O(40)	0.3870(2)	0.7359(6)	0.2292(5)
O(41)	0.3146(2)	0.6999(6)	0.1602(5)
O(42)	0.3090(2)	0.5327(6)	0.2430(5)
O(43)	0.4200(2)	0.4820(6)	0.3681(5)
O(51)	0.3740(3)	0.0684(8)	0.2169(7)
O(52)	0.2323(3)	0.523(1)	-0.0900(6)
O(53)	0.4132(3)	0.109(1)	0.6056(7)
O(54)	0.3470(4)	0.171(1)	0.3739(9)
O(55)	0.5508(5)	0.685(1)	0.366(1)
O(56)	1.0048(7)	0.491(2)	0.368(2)
O(57)	0.2382(6)	0.220(2)	0.501(2)
O(58)	1.0000	1/2	1/2

collection was carried out as previously described [16]. Data was corrected for absorption using  $\Psi$  scans on 5 reflections with  $\chi$  angles near 90 or 270°. The structure was solved by direct methods and refined using full matrix least-squares techniques and a structural model which incorporated anisotropic thermal parameters for all atoms with the exception of hydrogens and oxygens of the water molecules of crystallization. Crystal data: monoclinic space group C2/c, a = 40.408(9), b =13.326(1), c = 18.074(3) Å,  $\beta = 112.28(1)^\circ, V = 9005.9(14)$ Å<sup>3</sup>,  $D_{calc} = 2.986$  g cm<sup>-3</sup>, Z = 8. Structure solution and refinement based on 5999 reflections with  $I_o \ge 6\sigma(I_o)$ (8343 collected; Mo K $\alpha$  radiation,  $\lambda = 0.71073$  Å) converged at R = 0.063,  $R_w = 0.069$ . Atomic positional parameters are listed in Table 1, and selected bond lengths and angles are given in Table 2.

## **Results and discussion**

The complex  $K_7Na[As_4V_{12}O_{43}H_3] \cdot 7H_2O$  is synthesized from metavanadate and  $As_2O_3$  by exploiting the weak reducing ability of rhodizonic acid and/or SCN<sup>-</sup> in acidic media. Whereas under more reducing conditions  $V^{IV}/V^V$  clusters with high  $V^{IV}:V^V$  rations are isolated [14], these conditions favor the formation of

$$C_6 O_6^{2-} \xrightarrow{[0]} 3C_2 O_4^{2-}$$
$$2SCN^- \rightleftharpoons (CNS)_2 + 2e^{-1}$$

(continued)

species with fewer V<sup>IV</sup> centers. The IR spectrum of the dark blue crystals of the cluster exhibits characteristic bands at 972 and 958 cm<sup>-1</sup> assigned to  $\nu$ (V=O<sub>t</sub>) and a number of bands in the 750–900 cm<sup>-1</sup> range associated with  $\nu$ (V–O–V) and  $\nu$ (V–O–As).

TABLE 2. Selected bond lengths (Å) and angles (°) for  $K_7Na[As_4^vV_7^vV_5^{lv}O_{43}H_3]\cdot 7H_2O$ 

# TABLE 2. (continued)

Distances (Å)         V9-029         1.72(1)           ASI-030         1.696(9)         V9-031         224(1)           ASI-011         1.69(9)         V9-038         2.345(7)           ASI-023         1.66(7)         V10-010         1.566(9)           ASI-023         1.66(7)         V10-034         2.288(7)           AS2-012         1.358(9)         V10-041         1.941(9)           AS2-033         1.658(6)         V10-041         1.941(9)           V3-030         2.337(7)         V11-011         1.59(1)           V4-04         1.601(9)         V11-020         1.99(1)           V4-04         1.601(9)         V11-021         1.947(8)           V4-04         1.601(9)         V11-021         1.99(1)           V4-050         1.67(8)         V12-012         1.99(1)           V4-040         1.66(8)         V12-023         2.031(7)           V1-010         1.62(1)         V12-033         2.331(8)           V1-011         1.66(8)         V12-033         2.331(8)           V1-012         1.99(9)         X4-040         1.66(8)         V12-033         2.031(7)           V1-014         1.66(8)         V12-033         2.031(8)			V9–O21	1.961(9)
AST - 030         169(9)         V9-031         2240(1)           AST - 031         169(9)         V9-038         2245(7)           AST - 031         169(9)         V10-010         1566(9)           AST - 031         1671(8)         V10-034         2288(7)           ASZ - 011         1734(9)         V10-035         159(1)           ASZ - 032         1658(8)         V10-041         159(1)           ASZ - 033         1666(7)         V10-041         159(1)           ASZ - 033         165(8)         199(1)         V3-029         202(1)           V3-030         2.56(7)         V11-011         159(1)           V3-030         2.56(7)         V11-021         199(1)           V4-049         148(8)         V11-021         159(8)           V4-050         1.68(1)         V11-021         159(8)           AS4-040         167(8)         V12-033         2.231(7)           AS4-040         167(8)         V12-033         2.231(8)           V1-011         1.26(1)         V12-033         2.231(8)           V1-011         1.26(1)         V12-033         2.231(8)           V1-012         1.597(7)         K1-044         2.814(9)	Distances (Å)		V9-O29	1.72(1)
Axi-Dail         1.6900)         V9-038         2.245(7)           Axi-Dail         1.6900)         VI0-010         1.966(9)           Axi-Dail         1.691(8)         VI0-032         1.593(7)           Axi-Dail         1.734(9)         VI0-035         1.99(1)           Asz-O22         1.658(9)         VI0-041         1.941(9)           Asz-O38         1.665(8)         1.99(1)           V1-029         2.011(9)         V11-011         1.59(1)           V-044         1.601(9)         V11-021         1.947(8)           V4-04         1.601(9)         V11-022         2.011(9)           V4-04         1.601(8)         V12-012         1.947(8)           V4-019         1.840(8)         V11-021         1.947(8)           V4-010         1.68(1)         V11-021         1.947(8)           V4-010         1.68(1)         V11-031         2.24(1)           Ass-036         1.672(9)         V12-012         1.951(1)           V4-016         1.897(8)         V12-033         2.351(7)           V1-016         1.897(8)         V12-033         2.351(7)           V1-017         1.891(8)         V12-033         2.967(9)           V1-028 <td>A\$1_030</td> <td>1 606(0)</td> <td>V9-O31</td> <td>2.04(1)</td>	A\$1_030	1 606(0)	V9-O31	2.04(1)
$\begin{array}{c} \begin{array}{c} \lambda_{1} - 032 \\ \lambda_{1} - 033 \\ \lambda_{1} - 033 \\ \lambda_{1} - 033 \\ \lambda_{2} - 013 \\ \lambda_{2} - 013 \\ \lambda_{2} - 013 \\ \lambda_{2} - 022 \\ \lambda_{3} - 022 \\ \lambda_{4} - 033 \\ \lambda_{2} - 023 \\ \lambda_{2} - 023 \\ \lambda_{2} - 033 \\ \lambda_{2} - 033 \\ \lambda_{3} - 030 \\ \lambda_{3$	AS1_031	1.690(9)	V9-O38	2.345(7)
http://display.org/limits/picture/limits/pi	A\$1-032	1.650(7)	V10–O10	1.966(9)
https://doi.org/10.1034         2.288(7)           https://doi.org/10.1034         2.288(7)           https://doi.org/10.1034         1.99(1)           https://doi.org/10.1034         1.941(9)           https://doi.org/10.1034         1.941(9)           https://doi.org/10.1034         1.941(9)           https://doi.org/10.1034         1.941(9)           https://doi.org/10.1034         1.941(9)           https://doi.org/10.1034         1.941(1)           https://doi.org/10.1034         1.941	As1-033	1.671(8)	V10-O32	1.593(7)
https://doi.org/10.1001/10.1002/10.202/10.202/10.203/10.1002/10.1002/10.1002/10.1002/10.1002/10	AS2-013	1.734(9)	V10–O34	2.288(7)
AS2-O38       1.680(6)       VI0-O41       1.941(9) $V1-O29$ 2.011(9)       1.991(9)       1.991(9) $V1-O29$ 2.037(7)       VI1-O11       1.55(1) $V4-O1$ 1.601(9)       VI1-O22       2.011(9) $V4-O1$ 1.601(9)       VI1-O21       1.947(8) $V4-O19$ 1.860(8)       VI1-O21       1.947(8) $V4-O20$ 1.66(1)       VI1-O31       2.24(1) $AS4-O36$ 1.672(9)       VI2-O12       1.591(9) $AS4-O40$ 1.670(8)       VI2-O33       2.031(7) $AS4-O41$ 1.668(8)       VI2-O33       2.031(7) $AS4-O40$ 1.670(8)       VI2-O33       2.031(7) $V1-O11$ 1.662(1)       VI2-O33       2.031(7) $V1-O28$ 2.022(8)       K1-O1       2.967(9) $V1-O28$ 2.023(1)       K1-O42       2.78(1) $V2-O30$ 2.351(8)       K1-O42       2.78(1) $V2-O16$ 1.944(8)       K2-O5       2.967(9) $V2-O27$ 2.003(7)       K2-O13       3.02(1) $V2-O16$ 1.944(8)       K2-O22       2.73(1)	AS2022	1.658(9)	V10–O35	1.99(1)
As2- $0.43$ 1665(6)         VI0-O42         202(1)           V1-029         2011(9)         199(9)           V2-020         2.367(7)         VI1-011         1.59(1)           V4-04         1.601(9)         VI1-020         1.99(1)           V4-019         1.840(8)         VI1-021         1.947(8)           V4-020         1.68(1)         VI1-021         2.01(19)           V4-020         1.68(1)         VI1-031         2.24(1)           S4S-036         1.672(9)         VI2-012         1.591(9)           AS4-041         1.668(8)         VI2-023         2.031(7)           S4S-040         1.670(8)         VI2-033         2.231(8)           V1-016         1.897(8)         VI2-033         2.031(7)           V1-025         1.979(7)         VI2-033         2.09(1)           V1-026         2.022(8)         K1-01         2.967(9)           V1-030         2.33(1)         K1-028         2.82(1)           V2-020         1.633(9)         K1-043         2.759(8)           V2-026         2.003(7)         K2-022         2.73(1)           V2-027         2.0011         K2-022         2.73(1)           V2-026         2	AS2038	1.680(8)	V10-O41	1.941(9)
$\begin{array}{c} 1-0.02^{-} & 2.011(0) & 1.991(9) & 1.991(9) \\ V2-030 & 2.35(7) & V11-011 & 1.59(1) \\ V4-04 & 1.601(9) & V11-021 & 1.947(8) \\ V4-019 & 1.840(8) & V11-021 & 2.011(9) \\ V4-020 & 1.66(1) & V11-031 & 2.24(1) \\ AS4-015 & 1.71(1) & V11-040 & 1.961(8) \\ AS4-040 & 1.670(8) & V12-012 & 1.591(9) \\ AS4-040 & 1.670(8) & V12-023 & 2.031(7) \\ V1-011 & 1.62(1) & V12-033 & 2.20(1) \\ V1-011 & 1.62(1) & V12-033 & 2.00(1) \\ V1-012 & 1.991(8) & V12-033 & 2.00(1) \\ V1-028 & 2.022(8) & K1-01 & 2.95(7) \\ V1-028 & 2.032(8) & K1-01 & 2.95(7) \\ V1-030 & 2.33(1) & K1-028 & 2.82(1) \\ V2-021 & 1.63(9) & K1-042 & 2.78(1) \\ V2-030 & 2.33(1) & K1-043 & 2.96(7) \\ V2-018 & 1.994(8) & K2-03 & 2.96(7) \\ V2-018 & 1.994(8) & K2-03 & 2.96(7) \\ V2-018 & 1.994(8) & K2-03 & 3.02(1) \\ V2-027 & 2.00(1) & K2-022 & 2.73(1) \\ V2-027 & 2.00(1) & K2-022 & 2.73(1) \\ V2-017 & 1.81(1) & 030-AS1-031 & 108.6(4) \\ V3-019 & 2.02(1) & 030-AS1-032 & 110.9(4) \\ AS3-024 & 1.69(8) & 031-AS1-033 & 107.9(4) \\ AS3-024 & 1.69(8) & 031-AS1-033 & 107.9(4) \\ AS3-024 & 1.69(8) & 013-AS2-022 & 103.0(5) \\ V4-027 & 1.813(9) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.836(9) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.836(9) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.836(9) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.68(8) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.68(8) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.68(8) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.68(9) & 013-AS2-038 & 103.4(4) \\ V5-028 & 1.68(7) & 014-AS3-042 & 106.9(4) \\ V5-028 & 1.68(9) & 013-AS2-043 & 116.8(4) \\ V5-028 & 1.68(9) & 013-AS2-043 & 116.8(4) \\ V5-028 & 1.68(7) & 014-AS3-042 & 106.9(4) \\ V5-028 & 1.59(7) & 014-AS3-042 & 106.9(4) \\ V5-038 & 1.59(7)$	AS2-043	1.665(8)	V10-O42	2.02(1)
Va-030 2.367(7) V1-011 1.59(1) V1-020 1.99(1) V4-04 1.60(19) V1-020 1.99(1) V4-020 1.68(1) V1-021 1.947(8) V4-020 1.68(1) V1-021 2.20(1)(9) V4-020 1.68(1) V1-021 2.20(1)(9) V3-020 1.68(1) V1-031 2.24(1) V1-031 2.24(1) V1-031 2.24(1) V1-040 1.98(18) V12-036 1.67(20) V1-01 1.62(1) V12-012 1.59(19) V1-01 1.62(1) V12-013 2.23(18) V1-016 1.68(8) V12-023 2.20(1) V1-016 1.68(1) V12-033 2.23(18) V1-016 1.507(8) V12-039 1.95(1) V1-025 1.979(7) V12-043 2.20(1) V1-028 2.202(8) K1-01 2.967(9) V1-030 2.33(1) K1-040 2.81(49) V2-030 2.35(18) K1-042 2.78(1) V2-030 2.35(18) K1-042 2.78(1) V2-030 2.35(18) K1-043 2.2795(8) V2-016 1.84(1) K1-043 2.2795(8) V2-016 1.84(1) K1-043 2.2795(8) V2-016 1.994(8) K2-05 2.967(9) V2-026 2.003(7) K2-013 3.02(1) V3-017 1.81(1) 0.30-AS1-033 109.2(4) V3-018 1.787(9) 0.30-AS1-033 109.2(4) V3-019 2.02(1) 0.33-AS1-033 109.2(4) V3-027 1.83(9) 0.33-AS1-033 109.2(4) V3-027 1.83(9) 0.33-AS1-033 109.2(4) V3-027 1.83(9) 0.33-AS1-034 108.7(4) V3-027 1.83(9) 0.33-AS1-034 108.7(4) V3-027 1.83(9) 0.33-AS1-034 108.7(4) V3-027 1.83(9) 0.33-AS1-034 108.7(4) V3-027 2.20(9(8) 0.13-AS2-043 108.7(4) V3-027 2.20(9(8) 0.13-AS2-043 108.7(4) V3-027 2.20(9(8) 0.13-AS1-044	V3O29	2.011(9)		1.991(9)
V4-O4 $160[0)$ V1-O20 $1.99(1)$ V4-O19 $1.80(8)$ V11-O21 $1.947(8)$ V4-O20 $1.68(1)$ V11-O21 $2.011(9)$ AS4-O36 $1.67(9)$ V12-O12 $1.591(9)$ AS4-O40 $1.670(8)$ V12-O12 $1.591(9)$ AS4-O40 $1.670(8)$ V12-O33 $2.031(7)$ AS4-O41 $1.668(8)$ V12-O37 $1.974(8)$ V1-O11 $1.62(1)$ V12-O33 $2.031(7)$ V1-O17 $1.891(8)$ V12-O33 $2.09(1)$ V1-O28 $2.022(8)$ K1-O1 $2.967(9)$ V1-O20 $1.603(9)$ K1-O42 $2.78(1)$ V1-O28 $2.022(8)$ K1-O42 $2.78(1)$ V2-O16 $1.84(1)$ K2-O5 $2.967(9)$ V2-O18 $1.994(8)$ K2-O5 $2.967(9)$ V2-O18 $1.934(8)$ K2-O5 $2.967(9)$ V2-O18 $1.934(8)$ K2-O13 $3.02(1)$ V2-O27 $2.004(1)$ K2-O22<	V3-O30	2.367(7)	V11-O11	1.59(1)
$V4-019 = 1.840(5) = V11-021 = 1.947(8) \\V4-020 = 1.840(5) = V11-022 = 2.011(9) \\AS4-045 = 1.71(1) = V11-031 = 2.24(1) \\AS4-046 = 1.672(9) = V12-012 = 1.591(9) \\AS4-041 = 1.668(8) = V12-023 = 2.031(7) \\AS4-041 = 1.668(8) = V12-023 = 2.031(7) \\AS4-041 = 1.668(8) = V12-033 = 2.231(8) \\V1-016 = 1.807(8) = V12-037 = 1.974(8) \\V1-016 = 1.807(8) = V12-037 = 1.974(8) \\V1-025 = 1.979(7) = V12-043 = 2.001(1) \\V1-025 = 2.022(8) = K1-01 = 2.967(9) \\V1-030 = 2.33(1) = K1-040 = 2.814(9) \\V2-030 = 2.33(1) = K1-040 = 2.814(9) \\V2-030 = 2.351(8) = K1-043 = 2.795(8) \\V2-016 = 1.84(1) = K2-05 = 2.967(9) \\V2-026 = 2.003(7) = K2-013 = 3.02(1) \\V3-018 = 1.787(9) = 0.30-AS1-032 = 109.2(4) \\V3-018 = 1.787(9) = 0.30-AS1-032 = 109.2(4) \\V3-019 = 2.02(1) = 0.30-AS1-032 = 109.2(4) \\V3-019 = 2.02(1) = 0.30-AS1-033 = 107.9(4) \\AS3-024 = 1.668(8) = 0.31-AS1-033 = 107.9(4) \\AS3-024 = 1.668(8) = 0.31-AS1-033 = 107.9(4) \\AS3-042 = 1.668(8) = 0.31-AS1-033 = 107.9(4) \\AS3-042 = 1.668(8) = 0.31-AS1-033 = 109.0(4) \\V5-028 = 1.36(9) = 0.22-AS2-043 = 103.4(4) \\V5-028 = 1.36(9) = 0.22-AS2-043 = 103.4(4) \\V5-028 = 1.83(9) = 0.1-4-AS3-042 = 105.4(4) \\V7-024 = 1.99(1) = 0.4-AS3-042 = 105.4(4) \\V7-024 = 1.99$	V404	1.601(9)	V11–O20	1.99(1)
$\begin{split} & V4-020 & 1.68(1) & V1-022 & 2.01(9) \\ & AS4-036 & 1.672(9) & V11-031 & 2.24(1) \\ & AS4-040 & 1.670(8) & V12-012 & 1.591(9) \\ & AS4-040 & 1.670(8) & V12-023 & 2.031(7) \\ & AS4-041 & 1.668(8) & V12-023 & 2.031(7) \\ & AS4-041 & 1.668(8) & V12-033 & 2.231(8) \\ & V1-011 & 1.821(8) & V12-037 & 1.974(8) \\ & V1-017 & 1.891(8) & V12-039 & 1.95(1) \\ & V1-028 & 2.022(8) & K1-01 & 2.967(9) \\ & V1-028 & 2.022(8) & K1-028 & 2.82(1) \\ & V1-028 & 2.022(8) & K1-040 & 2.814(9) \\ & V2-030 & 2.33(1) & K1-040 & 2.814(9) \\ & V2-030 & 2.351(8) & K1-042 & 2.78(1) \\ & V2-016 & 1.84(1) & K1-043 & 2.795(8) \\ & V2-018 & 1.934(8) & K2-05 & 2.967(9) \\ & V2-018 & 1.934(8) & Angles (°) \\ & V2-018 & 1.934(8) & Angles (°) \\ & V2-018 & 1.787(9) & 030-AS1-031 & 108.6(4) \\ & V3-018 & 1.787(9) & 030-AS1-033 & 107.9(4) \\ & AS3-024 & 1.668(8) & 031-AS1-033 & 107.9(4) \\ & AS3-024 & 1.69(1) & 031-AS1-033 & 109.2(4) \\ & V3-019 & 2.02(1) & 030-AS1-033 & 107.9(4) \\ & AS3-024 & 1.69(1) & 031-AS1-033 & 109.4(4) \\ & AS3-024 & 1.69(1) & 031-AS1-033 & 109.4(4) \\ & V3-019 & 2.02(1) & 030-AS1-033 & 109.4(4) \\ & V3-019 & 0.22(1) & 030-AS1-033 & 109.4(4) \\ & V3-018 & 1.787(9) & 013-AS2-023 & 108.2(4) \\ & V3-018 & 1.787(9) & 013-AS2-023 & 109.2(4) \\ & V3-018 & 1.787(9) & 013-AS1-033 & 109.4(4) \\ & V3-018 & 1.787(9) & 013-AS1-033 & 109.4(4) \\ & V3-018 & 1.787(9) & 013-AS2-023 & 108.2(4) \\ & V3-018 & 1.787(9) & 013-AS2-023 & 109.2(4) \\ & V3-018 & 1.787(9) & 013-AS2-023 & 109.2(4) \\ & V3-018 & 1.787(9) & 013-AS2-023 & 109.2(4) \\ & V3-018 & 1.787(9) & 013-AS2-043 & 100.3(5) \\ & V4-027 & 1.818(9) & 013-AS2-023 & 109.2(4) \\ & V3-028 & 1.366(9) & 022-AS2-043 & 110.3(4) \\ & V5-028 & 1.458(9) & 013-AS2-043 & 108.7(4) \\ & V5-028 & 1.458(9) & 013-AS2-043 & 108.7(4) \\ & V5-028 & 1.58(9) & 013-AS2-043 & 108.7(4) \\ & V5-028 & 1.58(9) & 013-AS3-042 & 106.5(4) \\ & V7-02 & 1.687(9) & 035-AS3-042 & 106.5(4) \\ & V7-02 & 1.687(9) & 035-AS3-042 & 106.5(4) \\ & V7-02 & 1.56(8) & 014-AS3-042 & 105.5(4) \\ & V7-03 & 1.768(7) & 014-AS3-042 & 105.5(4) \\ & V7-03 & 1.768(7) & 01-V1-028 & $	V4019	1.840(8)	V11–O21	1.947(8)
AS4-015 $1.71(1)$ V1-031 $2.24(1)$ AS4-036 $1.670(8)$ V12-012 $1.591(8)$ AS4-040 $1.670(8)$ V12-023 $2.031(7)$ AS4-041 $1.668(8)$ V12-023 $2.031(7)$ AS4-041 $1.668(8)$ V12-023 $2.031(7)$ V1-016 $1.807(8)$ V12-037 $1.974(8)$ V1-017 $1.891(8)$ V12-039 $1.95(1)$ V1-025 $1.979(7)$ V12-043 $2.00(1)$ V1-028 $2.022(8)$ K1-040 $2.814(9)$ V2-030 $2.351(8)$ K1-042 $2.78(1)$ V2-030 $2.351(8)$ K1-042 $2.78(1)$ V2-016 $1.84(1)$ K1-043 $2.795(8)$ V2-017 $1.81(8)$ Angles (°) $V2-026$ $2.003(7)$ K2-013 $3.02(1)$ V2-027 $2.00(1)$ $C30-AS1-O31$ $108.6(4)$ $V3-019$ $2.02(1)$ $C30-AS1-O32$ $109.2(4)$ AS3-014 $1.724(8)$ $O30-AS1-O33$ $10.92(4)$ $AS3-O42$ $1.068(8)$ $O31-AS1-O33$ $111.0(4)$ <	V4O20	1.68(1)	V11–O22	2.011(9)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	AS4-015	1.71(1)	V11–O31	2.24(1)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	AS4-036	1.672(9)	V11-O40	1.981(8)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	AS4-040	1.670(8)	V12-012	1.591(9)
VI-OI       1.62(1)       V12-O37       1.974(8)         VI-O16       1.897(8)       V12-O37       1.974(8)         VI-O17       1.891(8)       V12-O39       1.95(1)         VI-O25       1.979(7)       K1-O1       2.967(9)         VI-O26       2.022(8)       K1-O1       2.967(9)         VI-O30       2.33(1)       K1-O40       2.814(9)         V2-O2       1.603(9)       K1-O40       2.814(9)         V2-O30       2.351(8)       K1-O42       2.78(1)         V2-O16       1.84(1)       K2-O5       2.967(9)         V2-O26       2.003(7)       K2-O13       3.02(1)         V2-O26       2.003(7)       K2-O12       2.73(1)         V3-O17       1.81(1)       O30-AS1-O31       108.6(4)         V3-O18       1.787(9)       O30-AS1-O32       109.2(4)         V3-O19       2.02(1)       O30-AS1-O32       109.2(4)         V3-O14       1.724(8)       O31-AS1-O33       111.1(4)         AS3-O23       1.668(8)       O31-AS1-O33       109.0(4)         AS3-O42       1.658(9)       O13-AS2-O22       103.0(5)         V4-O27       1.813(9)       O13-AS2-O43       108.7(4)      V	AS4041	1.668(8)	V12-023	2.031(7)
VI-016       1.807(8)       V12-037       1.974(8)         VI-017       1.891(8)       V12-039       1.95(1)         VI-025       1.979(7)       V12-043       2.00(1)         VI-028       2.022(8)       K1-040       2.85(7)         V2-02       1.603(9)       K1-040       2.814(9)         V2-030       2.351(8)       K1-042       2.78(1)         V2-016       1.84(1)       K2-05       2.967(9)         V2-018       1.934(8)       K2-013       3.02(1)         V2-022       2.00(1)       K2-022       2.73(1)         V2-026       2.003(7)       K2-013       3.02(1)         V2-027       2.00(1)       K2-022       2.73(1)         V3-017       1.81(1)       O30-AS1-031       108.6(4)         V3-018       1.787(9)       O30-AS1-033       107.9(4)         AS3-021       1.668(8)       O31-AS1-033       111.0(4)         AS3-023       1.668(8)       O31-AS1-033       111.0(4)         AS3-024       1.69(1)       O32-AS1-033       109.0(4)         AS3-024       1.69(1)       O32-AS2-043       108.7(4)         V5-028       1.836(9)       O13-AS2-024       103.0(5)	V101	1.62(1)	V12-033	2.231(8)
VI-017       1.891(8)       V12-039       1.95(1)         VI-025       1.979(7)       V12-043       2.00(1)         VI-028       2.022(8)       K1-01       2.957(9)         VI-030       2.33(1)       K1-040       2.81(9)         V2-02       1.603(9)       K1-042       2.78(1)         V2-030       2.351(8)       K1-042       2.78(1)         V2-016       1.84(1)       K2-05       2.967(9)         V2-026       2.003(7)       K2-022       2.73(1)         V3-03       1.618(8)       Angles (*)       V2-027         V3-017       1.81(1)       030-AS1-031       108.6(4)         V3-018       1.757(9)       030-AS1-032       109.2(4)         V3-019       2.02(1)       030-AS1-033       107.9(4)         AS3-014       1.724(8)       031-AS1-033       111.1(4)         AS3-023       1.668(8)       031-AS1-033       111.1(4)         AS3-042       1.69(1)       032-AS1-033       109.0(4)         V5-05       1.603(8)       013-AS2-038       103.4(4)         V5-05       1.603(8)       013-AS2-038       103.4(4)         V5-037       1.70(1)       022-AS2-038       115.7(4)	V1O16	1.807(8)	V12-037	1.974(8)
$\begin{split} & VI-025 & 1.979(7) & V1-2045 & 2.0011) \\ VI-028 & 2.022(8) & K1-01 & 2.967(9) \\ VI-030 & 2.33(1) & K1-028 & 2.82(1) \\ V2-030 & 2.33(1) & K1-042 & 2.78(1) \\ V2-02 & 1.603(9) & K1-042 & 2.78(1) \\ V2-016 & 1.84(1) & K1-043 & 2.795(8) \\ V2-016 & 1.84(1) & K2-05 & 2.967(9) \\ V2-018 & 1.934(8) & K2-05 & 2.967(9) \\ V2-026 & 2.003(7) & K2-013 & 3.02(1) \\ V2-027 & 2.00(1) & K2-022 & 2.73(1) \\ V3-03 & 1.618(8) & Angles (*) \\ V3-017 & 1.81(1) & 030-AS1-031 & 108.6(4) \\ V3-018 & 1.787(9) & 030-AS1-032 & 110.2(4) \\ V3-018 & 1.787(9) & 030-AS1-033 & 107.9(4) \\ AS3-024 & 1.69(1) & 031-AS1-033 & 111.1(4) \\ AS3-024 & 1.69(1) & 031-AS1-033 & 109.0(4) \\ AS3-042 & 1.658(9) & 013-AS2-022 & 103.0(5) \\ V5-025 & 1.603(8) & 013-AS2-038 & 103.4(4) \\ V5-025 & 1.603(8) & 013-AS2-038 & 108.7(4) \\ V5-029 & 1.836(9) & 022-AS2-043 & 110.3(4) \\ V5-029 & 1.836(9) & 022-AS2-043 & 110.3(4) \\ V5-025 & 1.603(8) & 014-AS3-023 & 100.3(4) \\ V5-025 & 1.798(7) & 014-AS3-023 & 104.2(4) \\ V6-026 & 1.62(1) & 014-AS3-024 & 103.2(4) \\ V6-026 & 1.62(1) & 014-AS3-023 & 104.2(4) \\ V6-026 & 1.62(1) & 014-AS3-024 & 103.2(4) \\ V6-026 & 1.62(1) & 014-AS3-024 & 106.8(4) \\ V7-07 & 1.62(1) & 023-AS3-024 & 116.8(4) \\ V7-07 & 1.62(1) & 023-AS3-042 & 106.9(4) \\ V6-026 & 1.827(8) & 014-AS3-042 & 106.9(4) \\ V7-027 & 1.62(1) & 023-AS3-042 & 116.8(4) \\ V7-028 & 1.99(07) & 015-AS4-041 & 114.9(5) \\ V7-028 & 1.99(07) & 015-AS4-041 & 114.9(5) \\ V7-028 & 1.99(07) & 015-AS4-041 & 114.9(5) \\ V8-033 & 2.99(1) & 01-V1-017 & 105.0(4) \\ V8-035 & 1.798(7) & 01-V1-030 & 178.9(3) \\ V9-09 & 1.576(8) & 01-V1-030 & 178.9(3) \\ V9-09 & 1.576(8) & 01-V1-030 & 178.9(3) \\ V9-09 & 1.576(8) & 01-V1-030 &$	V1-O17	1.891(8)	V12-039	1.95(1)
VI-O28         2.022(8)         NI-O1         2.30(9)           VI-O30         2.33(1)         KI-O28         2.82(1)           V2-O2         1.603(9)         KI-O42         2.78(1)           V2-O30         2.351(8)         KI-O42         2.78(1)           V2-O16         1.84(1)         KI-O43         2.795(8)           V2-O18         1.934(8)         K2-O13         3.02(1)           V2-O26         2.003(7)         K2-O13         3.02(1)           V2-O27         2.00(1)         K2-O22         2.73(1)           V3-O3         1.618(8)         Angles (°)         V           V3-O17         1.81(1)         O30-AS1-O31         108.6(4)           V3-O18         1.787(9)         O30-AS1-O32         109.2(4)           V3-O19         2.02(1)         O30-AS1-O33         107.9(4)           AS3-O14         1.724(8)         O31-AS1-O33         111.0(4)           AS3-O23         1.668(8)         O31-AS1-O33         109.0(4)           AS3-O42         1.658(9)         O13-AS2-O43         103.0(5)           V4-O27         1.813(9)         O13-AS2-O43         103.4(4)           V5-O5         1.603(8)         O13-AS2-O43         103.4(4) </td <td>V1-O25</td> <td>1.979(7)</td> <td>V12-043</td> <td>2.00(1)</td>	V1-O25	1.979(7)	V12-043	2.00(1)
V1-030       2.33(1)       N1-028       2.021)         V2-02       1.603(9)       K1-040       2.814(9)         V2-030       2.351(8)       K1-043       2.795(8)         V2-016       1.84(1)       K1-043       2.967(9)         V2-018       1.934(8)       K2-013       3.02(1)         V2-026       2.003(7)       K2-022       2.73(1)         V3-03       1.618(8)       Angles (°)         V3-017       1.81(1)       030-AS1-031       108.6(4)         V3-018       1.787(9)       030-AS1-032       109.2(4)         V3-019       2.02(1)       030-AS1-033       107.9(4)         AS3-014       1.724(8)       031-AS1-033       109.0(4)         AS3-023       1.668(8)       031-AS1-033       109.0(4)         AS3-042       1.658(9)       013-AS2-022       103.0(5)         V4-027       1.813(9)       013-AS2-043       108.7(4)         V5-025       1.603(8)       013-AS2-043       108.7(4)         V5-025       1.603(8)       013-AS2-043       103.4(4)         V5-025       1.603(8)       013-AS2-043       103.4(4)         V5-025       1.603(8)       013-AS2-043       103.4(4) <tr< td=""><td>V1O28</td><td>2.022(8)</td><td>K1-01 K1 028</td><td>2.907(9)</td></tr<>	V1O28	2.022(8)	K1-01 K1 028	2.907(9)
V2-O2         1.603(9)         R1-042         2.514(7)           V2-O30         2.551(8)         K1-042         2.78(1)           V2-O16         1.84(1)         K2-O5         2.967(9)           V2-026         2.003(7)         K2-O13         3.02(1)           V2-026         2.003(7)         K2-O13         3.02(1)           V2-026         2.003(7)         K2-O13         3.02(1)           V2-027         2.00(1)         K2-O22         2.73(1)           V3-018         1.787(9)         030-AS1-O31         108.6(4)           V3-019         2.02(1)         030-AS1-O32         109.2(4)           V3-019         2.02(1)         030-AS1-O33         107.9(4)           AS3-023         1.668(8)         031-AS1-O33         111.1(4)           AS3-042         1.658(9)         013-AS2-033         109.0(4)           AS3-042         1.658(9)         013-AS2-043         108.4(4)           V5-05         1.603(8)         013-AS2-043         108.4(4)           V5-05         1.603(8)         013-AS2-043         103.4(4)           V5-05         1.603(8)         013-AS2-043         103.4(4)           V5-05         1.603(8)         013-AS2-043         103.4(	V1–O30	2.33(1)	K1-028	2.82(1) 2.814(0)
V2-030         2.351(8) $K1-043$ 2.795(8)           V2-016         1.84(1)         K2-05         2.967(9)           V2-018         1.934(8)         K2-013         3.02(1)           V2-026         2.003(7)         K2-022         2.73(1)           V3-03         1.618(8)         Angles (°)         V3-017         1.81(1)         0.30-AS1-031         108.6(4)           V3-018         1.787(9)         0.30-AS1-032         109.2(4)         V3-019         2.02(1)         0.30-AS1-032         110.9(4)           AS3-014         1.724(8)         0.31-AS1-032         111.1(4)         AS3-023         1.668(8)         0.31-AS1-033         109.0(4)           AS3-023         1.668(8)         0.31-AS2-022         103.0(5)         V4-027         1.813(9)         013-AS2-022         103.0(5)           V4-027         1.813(9)         013-AS2-023         108.7(4)         V5-028         1.836(9)         022-AS2-038         103.7(4)           V5-028         1.836(9)         022-AS2-043         110.3(4)         V5-028         1.836(9)         022-AS2-043         110.3(4)           V5-029         1.803(9)         022-AS2-043         110.3(4)         V5-028         1.667(1)         023-AS3-042         106.5(5) <td>V2-O2</td> <td>1.603(9)</td> <td>K1-040 K1 042</td> <td>2.014(9) 2.78(1)</td>	V2-O2	1.603(9)	K1-040 K1 042	2.014(9) 2.78(1)
V2-016       1.84(1)       R1-05       2.95(9)         V2-018       1.934(8)       K2-05       2.967(9)         V2-026       2.003(7)       K2-022       2.73(1)         V2-027       2.00(1)       K2-022       2.73(1)         V3-03       1.618(8)       Angles (°)       V3-03       1.018(8)       Angles (°)         V3-017       1.81(1)       030-AS1-032       109.2(4)         V3-018       1.787(9)       030-AS1-033       107.9(4)         AS3-014       1.724(8)       031-AS1-033       111.0(4)         AS3-023       1.668(8)       031-AS1-033       111.1(4)         AS3-024       1.69(1)       032-AS1-033       109.0(4)         AS3-042       1.658(9)       013-AS2-038       103.4(4)         V5-05       1.603(8)       013-AS2-043       108.7(4)         V5-05       1.603(8)       013-AS2-043       108.7(4)         V5-05       1.603(8)       013-AS2-043       108.7(4)         V5-05       1.603(8)       013-AS2-043       108.7(4)         V5-05       1.603(8)       014-AS3-024       103.2(4)         V5-05       1.603(8)       014-AS3-042       106.9(4)         V6-06       1.62(1)	V2O30	2.351(8)	K1-042 K1-043	2.70(1)
V2-018         1.934(8) $R^{2-O3}$ 2.03(7)           V2-026         2.003(7)         K2-022         2.73(1)           V2-027         2.00(1)         K2-022         2.73(1)           V3-03         1.618(8)         Angles (°)         V3-017           V3-018         1.787(9)         030-AS1-031         108.6(4)           V3-019         2.02(1)         030-AS1-032         110.92(4)           AS3-014         1.724(8)         031-AS1-032         111.1(4)           AS3-023         1.668(8)         031-AS1-033         109.0(4)           AS3-024         1.69(1)         032-AS1-033         109.0(4)           AS3-042         1.658(9)         013-AS2-022         103.0(5)           V4-027         1.813(9)         013-AS2-038         108.7(4)           V5-028         1.836(9)         022-AS2-038         115.7(4)           V5-029         1.803(9)         022-AS2-043         114.6(5)           V6-025         1.798(7)         014-AS3-023         104.2(4)           V6-026         1.827(8)         014-AS3-042         105.9(4)           V7-024         1.971(7)         024-AS3-042         106.9(4)           V7-025         2.069(8)         015-	V2O16	1.84(1)	K1=045 K2_05	2.755(0)
V2-026         2.003(7)         K2-012         2.03(1)           V2-027         2.00(1)         K2-022         2.73(1)           V3-03         1.618(8)         Angles (°)         108.6(4)           V3-017         1.81(1)         030-AS1-031         108.6(4)           V3-018         1.787(9)         030-AS1-032         109.2(4)           V3-019         2.02(1)         030-AS1-033         117.9(4)           AS3-014         1.724(8)         031-AS1-033         111.0(4)           AS3-023         1.668(8)         031-AS1-033         111.0(4)           AS3-024         1.69(1)         032-AS1-033         109.0(4)           AS3-042         1.658(9)         013-AS2-038         103.4(4)           V5-05         1.603(8)         013-AS2-043         108.7(4)           V5-05         1.603(9)         022-AS2-043         110.3(4)           V5-028         1.836(9)         022-AS2-043         110.3(4)           V5-037         1.70(1)         022-AS2-043         110.3(4)           V6-026         1.827(8)         014-AS3-024         103.2(4)           V6-026         1.827(8)         014-AS3-024         105.6(4)           V7-024         1.971(7)         023-A	V2018	1.934(8)	K2-013	3.02(1)
V2-O27       2.00(1)       It. CLL       Distribution $V3-O3$ 1.618(8)       Angles (°) $V3-O17$ 1.81(1)       O30-AS1-O31       108.6(4) $V3-O18$ 1.787(9)       O30-AS1-O32       109.2(4) $V3-O19$ 2.02(1)       O30-AS1-O33       107.9(4)         AS3-O14       1.724(8)       O31-AS1-O33       111.1(4)         AS3-O22       1.668(8)       O31-AS1-O33       111.1(4)         AS3-O24       1.658(9)       O13-AS2-O22       103.0(5)         V4-027       1.813(9)       O13-AS2-O38       103.4(4)         V5-O5       1.603(8)       O13-AS2-O38       105.7(4)         V5-O28       1.836(9)       O22-AS2-O43       110.3(4)         V5-O29       1.803(9)       O22-AS2-O43       114.6(5)         V6-06       1.62(1)       O14-AS3-O24       103.2(4)         V6-025       1.798(7)       O14-AS3-O24       103.2(4)         V6-026       1.827(8)       O14-AS3-O24       103.2(4)         V7-O7       1.62(1)       O23-AS3-O24       116.8(4)         V7-O24       1.971(7)       O24-AS3-O42       105.3(4)         V7-O25       2.069(8)       O15-AS4-O36	V2O26	2.003(7)	K2-013	2.73(1)
$V3-03 \\ V3-017 \\ I.81(1) \\ V3-017 \\ V3-017 \\ V3-018 \\ V3-018 \\ V3-019 \\ 2.02(1) \\ V3-019 \\ 2.02(1) \\ O30-AS1-O32 \\ 030-AS1-O32 \\ 030-AS1-O32 \\ 030-AS1-O32 \\ 030-AS1-O33 \\ 031-AS1-O33 \\ 032-AS1-O33 \\ 042-(4) \\ 05-O37 \\ 1.70(1) \\ 022-AS2-O43 \\ 032-AS1-O43 \\ 032-(4) \\ 04-O25 \\ 015-AS1-O42 \\ 042-(4) \\ 042-(5) \\ 042-(4) \\ 042-(5)$	V2-O27	2.00(1)		200(1)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	V3-O3	1.618(8)	Angles (°)	
V3-018 $1.787(9)$ $O30-AS1-O32$ $109.2(4)$ $V3-019$ $2.02(1)$ $O30-AS1-O33$ $107.9(4)$ $AS3-014$ $1.724(8)$ $O31-AS1-O33$ $111.0(4)$ $AS3-O23$ $1.668(8)$ $O31-AS1-O33$ $111.1(4)$ $AS3-O24$ $1.69(1)$ $O32-AS1-O33$ $109.0(4)$ $AS3-O42$ $1.658(9)$ $O13-AS2-O22$ $103.0(5)$ $V4-027$ $1.813(9)$ $O13-AS2-O38$ $103.4(4)$ $V5-05$ $1.603(8)$ $O13-AS2-O43$ $108.7(4)$ $V5-028$ $1.836(9)$ $O22-AS2-O43$ $114.6(5)$ $V5-029$ $1.803(9)$ $O22-AS2-O43$ $114.6(5)$ $V5-037$ $1.70(1)$ $O22-AS2-O43$ $114.6(5)$ $V6-06$ $1.62(1)$ $O14-AS3-O24$ $103.2(4)$ $V6-025$ $1.798(7)$ $O14-AS3-O24$ $103.2(4)$ $V6-026$ $1.827(8)$ $O14-AS3-O24$ $106.9(4)$ $V7-07$ $1.62(1)$ $O23-AS3-O42$ $106.9(4)$ $V7-024$ $1.971(7)$ $O24-AS3-O42$ $105.5(4)$ $V7-025$ $2.069(8)$ $O15-AS4-O41$ $103.8(5)$ $V7-033$ $2.29(1)$ $O15-AS4-O41$ $103.3(4)$ $V8-O8$ $1.592(9)$ $O36-AS4-O41$ $114.9(5)$ $V8-O26$ $1.99(1)$ $O1-V1-O16$ $102.6(5)$ $V8-O36$ $1.99(1)$ $O1-V1-O17$ $105.0(4)$ $V8-O36$ $1.99(1)$ $O1-V1-O30$ $178.9(3)$ $V9-O9$ $1.576(8)$ $O1-V1-O17$ $96.1(4)$ $V9-O9$ $1.576(8)$ $O1-V1-O17$ $96.1(4)$ <	V3-017	1.81(1)	O30-AS1-O31	108.6(4)
$V_3$ -O19 $2.02(1)$ O30-AS1-O33107.9(4)AS3-O141.724(8)O31-AS1-O33111.10(4)AS3-O231.668(8)O31-AS1-O33111.11(4)AS3-O241.69(1)O32-AS1-O33109.0(4)AS3-O421.658(9)O13-AS2-O22103.0(5)V4-O271.813(9)O13-AS2-O43108.7(4)V5-O51.603(8)O13-AS2-O43108.7(4)V5-O281.836(9)O22-AS2-O43110.3(4)V5-O291.803(9)O22-AS2-O43114.6(5)V6-O61.62(1)O14-AS3-O23104.2(4)V6-O261.827(8)O14-AS3-O24103.2(4)V6-O261.827(8)O14-AS3-O24103.2(4)V6-O251.798(7)O14-AS3-O24103.2(4)V7-O71.62(1)O23-AS3-O42116.8(4)V7-O71.62(1)O23-AS3-O42115.5(4)V7-O252.069(8)O15-AS4-O40103.8(5)V7-O332.29(1)O15-AS4-O40103.8(5)V7-O391.714(9)O36-AS4-O40115.9(4)V8-O261.99(1)O40-AS4-O41111.2(4)V8-O261.99(1)O1-V1-O16102.6(5)V8-O361.99(1)O1-V1-O17105.0(4)V8-O361.99(1)O1-V1-O30178.9(3)V9-O191.961(9)O1-V1-O30178.9(3)V9-O191.961(9)O1-V1-O1796.1(4)	V3-018	1.787(9)	O30-AS1-O32	109.2(4)
$\begin{array}{c} AS3-O14 & 1.724(8) & O31-AS1-O32 & 111.0(4) \\ AS3-O23 & 1.668(8) & O31-AS1-O33 & 111.1(4) \\ AS3-O24 & 1.69(1) & O32-AS1-O33 & 109.0(4) \\ AS3-O42 & 1.658(9) & O13-AS2-O22 & 103.0(5) \\ V4-O27 & 1.813(9) & O13-AS2-O23 & 103.4(4) \\ V5-O5 & 1.603(8) & O13-AS2-O43 & 108.7(4) \\ V5-O28 & 1.836(9) & O22-AS2-O43 & 110.3(4) \\ V5-O29 & 1.803(9) & O22-AS2-O43 & 110.3(4) \\ V5-O37 & 1.70(1) & O22-AS2-O43 & 110.3(4) \\ V5-O25 & 1.798(7) & O14-AS3-O23 & 104.2(4) \\ V6-O26 & 1.827(8) & O14-AS3-O24 & 103.2(4) \\ V6-O26 & 1.827(8) & O14-AS3-O24 & 106.9(4) \\ V7-O7 & 1.62(1) & O23-AS3-O42 & 106.9(4) \\ V7-O24 & 1.971(7) & O24-AS3-O42 & 105.9(4) \\ V7-O25 & 2.069(8) & O15-AS4-O40 & 103.8(5) \\ V7-O28 & 1.590(7) & O15-AS4-O41 & 105.3(4) \\ V7-O28 & 1.590(7) & O15-AS4-O41 & 105.3(4) \\ V7-O33 & 2.29(1) & O15-AS4-O41 & 105.3(4) \\ V7-O39 & 1.714(9) & O36-AS4-O41 & 115.9(4) \\ V8-O32 & 1.99(1) & O15-AS4-O41 & 115.9(4) \\ V8-O32 & 2.297(8) & O1-V1-O16 & 102.6(5) \\ V8-O32 & 2.297(8) & O1-V1-O17 & 105.0(4) \\ V8-O36 & 1.99(1) & O1-V1-O16 & 102.6(5) \\ V8-O35 & 1.768(7) & O1-V1-O17 & 105.0(4) \\ V8-O36 & 1.99(1) & O1-V1-O28 & 96.8(4) \\ V9-O9 & 1.576(8) & O1-V1-O30 & 178.9(3) \\ V9-O19 & 1.96(19) & O1-V1-O17 & 105.0(4) \\ V8-O36 & 0.99(1) & O1-V1-O30 & 178.9(3) \\ V9-O19 & 1.96(19) & O1-V1-O17 & 002.6(5) \\ V8-O30 & 0.9(1) & O1-V1-O30 & 0.78.9(3) \\ V9-O19 & 1.96(19) & O1-V1-O17 & 0.6(1) \\ V2-O20 & V2-O20 &$	V3-019	2.02(1)	O30-AS1-O33	107.9(4)
AS3-0231.000(8)O31-AS1-O33111.1(4)AS3-0241.69(1)O32-AS1-O33109.0(4)AS3-0421.658(9)O13-AS2-O22103.0(5)V4-0271.813(9)O13-AS2-O38103.4(4)V5-051.603(8)O13-AS2-O43108.7(4)V5-0281.836(9)O22-AS2-O43110.3(4)V5-0291.803(9)O22-AS2-O43114.6(5)V6-061.62(1)O14-AS3-O23104.2(4)V6-0251.798(7)O14-AS3-O24103.2(4)V6-0261.827(8)O14-AS3-O42106.9(4)V7-071.62(1)O23-AS3-O42116.8(4)V7-071.62(1)O23-AS3-O42115.5(4)V7-0241.971(7)O24-AS3-O42103.8(5)V7-0332.29(1)O15-AS4-O41103.8(5)V7-0391.714(9)O36-AS4-O41115.9(4)V8-0261.99(1)O40-AS4-O41115.9(4)V8-0272.039(7)O1-V1-O17105.0(4)V8-0351.768(7)O1-V1-O17105.0(4)V8-0351.768(7)O1-V1-O17105.0(4)V8-0351.99(1)O1-V1-O25102.8(4)V9-091.576(8)O1-V1-O17105.0(4)V8-0361.99(1)O1-V1-O25102.8(4)V9-091.576(8)O1-V1-O1796.1(4)V9-091.576(8)O1-V1-O1796.1(4)V9-091.561(9)O1-V1-O1796.1(4)V9-0191.961(9)O1-V1-O1796.1(4)	AS3-014	1.724(8)	O31-AS1-O32	111.0(4)
AS3-024 $1.09(1)$ $O32-AS1-O33$ $109.0(4)$ AS3-042 $1.658(9)$ $O13-AS2-O22$ $103.0(5)$ V4-027 $1.813(9)$ $O13-AS2-O38$ $103.4(4)$ V5-05 $1.603(8)$ $O13-AS2-O43$ $108.7(4)$ V5-028 $1.836(9)$ $O22-AS2-O38$ $115.7(4)$ V5-029 $1.803(9)$ $O22-AS2-O43$ $110.3(4)$ V5-037 $1.70(1)$ $O22-AS2-O43$ $114.6(5)$ V6-06 $1.62(1)$ $O14-AS3-O23$ $104.2(4)$ V6-025 $1.798(7)$ $O14-AS3-O24$ $103.2(4)$ V6-026 $1.827(8)$ $O14-AS3-O24$ $106.9(4)$ V6-034 $1.687(9)$ $O23-AS3-O42$ $106.9(4)$ V7-07 $1.62(1)$ $O23-AS3-O42$ $109.0(4)$ V7-024 $1.971(7)$ $O24-AS3-O42$ $109.0(4)$ V7-025 $2.069(8)$ $O15-AS4-O40$ $103.8(5)$ V7-039 $1.714(9)$ $O36-AS4-O41$ $105.3(4)$ V7-039 $1.714(9)$ $O36-AS4-O41$ $115.9(4)$ V8-026 $1.99(1)$ $O40-AS4-O41$ $111.2(4)$ V8-035 $1.768(7)$ $O1-V1-O17$ $105.0(4)$ V8-035 $1.768(7)$ $O1-V1-O17$ $102.8(4)$ V9-09 $1.576(8)$ $O1-V1-O17$ $96.1(4)$ V9-09 $1.576(8)$ $O1-V1-O17$	AS3-023	1.008(8)	O31–AS1–O33	111.1(4)
AS3-042 $1.036(9)$ $013-AS2-022$ $103.0(5)$ V4-027 $1.813(9)$ $013-AS2-038$ $103.4(4)$ V5-05 $1.603(8)$ $013-AS2-038$ $108.7(4)$ V5-028 $1.836(9)$ $022-AS2-043$ $110.3(4)$ V5-029 $1.803(9)$ $022-AS2-043$ $110.3(4)$ V5-037 $1.70(1)$ $022-AS2-043$ $110.3(4)$ V6-06 $1.62(1)$ $014-AS3-023$ $104.2(4)$ V6-025 $1.798(7)$ $014-AS3-024$ $103.2(4)$ V6-026 $1.827(8)$ $014-AS3-042$ $106.9(4)$ V7-07 $1.62(1)$ $023-AS3-042$ $106.9(4)$ V7-07 $1.62(1)$ $023-AS3-042$ $109.0(4)$ V7-024 $1.971(7)$ $024-AS3-042$ $105.5(4)$ V7-025 $2.069(8)$ $015-AS4-040$ $103.8(5)$ V7-033 $2.29(1)$ $015-AS4-041$ $105.3(4)$ V7-039 $1.714(9)$ $036-AS4-041$ $114.9(5)$ V8-08 $1.592(9)$ $036-AS4-041$ $114.9(5)$ V8-026 $1.99(1)$ $01-V1-016$ $102.6(5)$ V8-035 $1.768(7)$ $01-V1-017$ $105.0(4)$ V8-036 $1.99(1)$ $01-V1-028$ $96.8(4)$ V9-09 $1.576(8)$ $01-V1-017$ $102.8(4)$ V8-036 $1.99(1)$ $01-V1-030$ $178.9(3)$ V9-019 $1.961(9)$ $01e-V1-017$ $96.1(4)$	AS3-024	1.69(1)	O32-AS1-O33	109.0(4)
V4-027 $1.613(9)$ $O13-AS2-O38$ $103.4(4)$ $V5-025$ $1.603(8)$ $O13-AS2-O43$ $108.7(4)$ $V5-028$ $1.836(9)$ $O22-AS2-O43$ $115.7(4)$ $V5-029$ $1.803(9)$ $O22-AS2-O43$ $110.3(4)$ $V5-037$ $1.70(1)$ $O22-AS2-O43$ $110.3(4)$ $V6-06$ $1.62(1)$ $O14-AS3-O23$ $104.2(4)$ $V6-025$ $1.798(7)$ $O14-AS3-O24$ $103.2(4)$ $V6-026$ $1.827(8)$ $O14-AS3-O42$ $106.9(4)$ $V6-034$ $1.687(9)$ $O23-AS3-O42$ $116.8(4)$ $V7-07$ $1.62(1)$ $O23-AS3-O42$ $119.0(4)$ $V7-077$ $1.62(1)$ $O23-AS3-O42$ $119.0(4)$ $V7-024$ $1.971(7)$ $O24-AS3-O42$ $115.5(4)$ $V7-025$ $2.069(8)$ $O15-AS4-O40$ $103.8(5)$ $V7-033$ $2.29(1)$ $O15-AS4-O40$ $103.8(5)$ $V7-039$ $1.714(9)$ $O36-AS4-O41$ $114.9(5)$ $V8-O26$ $1.99(1)$ $O40-AS4-O41$ $115.9(4)$ $V8-O26$ $1.99(1)$ $O1-V1-O17$ $105.0(4)$ $V8-O35$ $1.768(7)$ $O1-V1-O17$ $105.0(4)$ $V8-O35$ $1.768(7)$ $O1-V1-O28$ $96.8(4)$ $V9-O9$ $1.576(8)$ $O1-V1-O17$ $96.1(4)$ $V9-O19$ $1.961(9)$ $O16-V1-O17$ $96.1(4)$	A55-042 VA 027	1.036(9)	O13-AS2-O22	103.0(5)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	V4-027	1.613(9)	O13-AS2-O38	103.4(4)
$\begin{array}{c} 13503 & 13507 & 022-AS2-O38 & 115.7(4) \\ V5-029 & 1.803(9) & 022-AS2-O43 & 110.3(4) \\ V5-037 & 1.70(1) & 022-AS2-O43 & 114.6(5) \\ V6-06 & 1.62(1) & 014-AS3-O23 & 104.2(4) \\ V6-025 & 1.798(7) & 014-AS3-O24 & 103.2(4) \\ V6-026 & 1.827(8) & 014-AS3-O42 & 106.9(4) \\ V6-034 & 1.687(9) & 023-AS3-O24 & 116.8(4) \\ V7-07 & 1.62(1) & 023-AS3-O42 & 109.0(4) \\ V7-024 & 1.971(7) & 024-AS3-O42 & 115.5(4) \\ V7-025 & 2.069(8) & 015-AS4-O40 & 103.8(5) \\ V7-033 & 2.29(1) & 015-AS4-O40 & 103.8(5) \\ V7-039 & 1.714(9) & 036-AS4-O40 & 115.9(4) \\ V8-08 & 1.592(9) & 036-AS4-O41 & 114.9(5) \\ V8-026 & 1.99(1) & 040-AS4-O41 & 111.2(4) \\ V8-027 & 2.039(7) & 01-V1-016 & 102.6(5) \\ V8-035 & 1.768(7) & 01-V1-017 & 105.0(4) \\ V8-036 & 1.99(1) & 01-V1-028 & 96.8(4) \\ V9-09 & 1.576(8) & 01-V1-030 & 178.9(3) \\ V9-019 & 1.961(9) & 016-V1-017 & 96.1(4) \\ \hline \end{array}$	V5028	1.836(9)	O13-AS2-O43	108.7(4)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	V5_020	1.803(9)	O22-AS2-O38	115.7(4)
$\begin{array}{c} 1.16(1) & 022-AS2-O43 & 114.6(5) \\ V6-O6 & 1.62(1) & 014-AS3-O23 & 104.2(4) \\ V6-O25 & 1.798(7) & 014-AS3-O24 & 103.2(4) \\ V6-O26 & 1.827(8) & 014-AS3-O42 & 106.9(4) \\ V6-O34 & 1.687(9) & 023-AS3-O42 & 106.9(4) \\ V7-O7 & 1.62(1) & 023-AS3-O42 & 109.0(4) \\ V7-O24 & 1.971(7) & 024-AS3-O42 & 115.5(4) \\ V7-O25 & 2.069(8) & 015-AS4-O36 & 104.2(5) \\ V7-O28 & 1.950(7) & 015-AS4-O40 & 103.8(5) \\ V7-O33 & 2.29(1) & 015-AS4-O41 & 105.3(4) \\ V7-O39 & 1.714(9) & 036-AS4-O41 & 115.9(4) \\ V8-O8 & 1.592(9) & 036-AS4-O41 & 114.9(5) \\ V8-O26 & 1.99(1) & 040-AS4-O41 & 111.2(4) \\ V8-O27 & 2.039(7) & 01-V1-O16 & 102.6(5) \\ V8-O32 & 2.297(8) & 01-V1-O17 & 105.0(4) \\ V8-O35 & 1.768(7) & 01-V1-O25 & 102.8(4) \\ V8-O36 & 1.99(1) & 01-V1-O28 & 96.8(4) \\ V9-O9 & 1.576(8) & 01-V1-O30 & 178.9(3) \\ V9-O19 & 1.961(9) & 016-V1-O17 & 96.1(4) \\ \end{array}$	V5_037	1.303(7)	O22-AS2-O43	110.3(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V6-06	1.62(1)	O22-AS2-O43	114.6(5)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	V6025	1.798(7)	O14-AS3-O23	104.2(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V6-026	1.827(8)	014-AS3-024	103.2(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V6-034	1.687(9)	O14-AS3-O42	106.9(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V7-07	1.62(1)	023-AS3-024	116.8(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V7-O24	1.971(7)	023-AS3-042	109.0(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V7-O25	2.069(8)	015 4 54 026	113.3(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V7-O28	1.950(7)	O15 - A54 - O30	104.2(3) 103.8(5)
$\begin{array}{c ccccc} V7-O39 & 1.714(9) & O36-A54-O41 & 105.9(4) \\ V8-O8 & 1.592(9) & O36-A54-O40 & 115.9(4) \\ V8-O26 & 1.99(1) & O40-A54-O41 & 114.9(5) \\ V8-O27 & 2.039(7) & O1-V1-O16 & 102.6(5) \\ V8-O32 & 2.297(8) & O1-V1-O17 & 105.0(4) \\ V8-O35 & 1.768(7) & O1-V1-O25 & 102.8(4) \\ V8-O36 & 1.99(1) & O1-V1-O28 & 96.8(4) \\ V9-O9 & 1.576(8) & O1-V1-O30 & 178.9(3) \\ V9-O19 & 1.961(9) & O16-V1-O17 & 96.1(4) \\ \hline \end{array}$	V7O33	2.29(1)	O15 - A54 - O40	105.0(5) 105.3(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V7O39	1.714(9)	$O_{15} - A_{54} - O_{41}$	105.5(4) 115 9(4)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	V8-O8	1.592(9)	$O_{36} = A_{54} = O_{40}$	114.9(5)
V8-O27     2.039(7)     O1-V1-O16     102.6(5)       V8-O32     2.297(8)     O1-V1-O17     105.0(4)       V8-O35     1.768(7)     O1-V1-O25     102.8(4)       V8-O36     1.99(1)     O1-V1-O28     96.8(4)       V9-O9     1.576(8)     O1-V1-O30     178.9(3)       V9-O19     1.961(9)     O16-V1-O17     96.1(4)	V8O26	1.99(1)	$\bigcirc 40 - 4 S4 - 041$	111.7(3)
V8-O32         2.297(8)         O1-V1-O17         102.0(3)           V8-O35         1.768(7)         O1-V1-O17         105.0(4)           V8-O36         1.99(1)         O1-V1-O25         102.8(4)           V9-O9         1.576(8)         O1-V1-O30         178.9(3)           V9-O19         1.961(9)         O16-V1-O17         96.1(4)	V8O27	2.039(7)	01_V1_016	102 6(5)
V8-O35       1.768(7)       O1-V1-O17       103.0(4)         V8-O36       1.99(1)       O1-V1-O25       102.8(4)         V9-O9       1.576(8)       O1-V1-O28       96.8(4)         V9-O19       1.961(9)       O16-V1-O17       96.1(4)         (continued)	V8032	2.297(8)	01-V1-017	105.0(3)
V8-O36         1.99(1)         O1-V1-O28         96.8(4)           V9-O9         1.576(8)         O1-V1-O30         178.9(3)           V9-O19         1.961(9)         O16-V1-O17         96.1(4)	V8O35	1.768(7)	01-V1-025	102.8(4)
V9-O9         1.576(8)         O1-V1-O30         178.9(3)           V9-O19         1.961(9)         O16-V1-O17         96.1(4)           (continued)	V8O36	1.99(1)	01 - V1 - 028	96.8(4)
V9-O19 1.961(9) 016-V1-O17 96.1(4) (continued)	V9–O9	1.576(8)	Q1-V1-Q30	178.9(3)
(continued)	V9–O19	1.961(9)	016-V1-017	96.1(4)
		(c)	ontinued)	

(continued)

TABLE 2. (continued)

TABLE 2. (continued)

		(continued)		(c
O7–V7–O39	103.8(5)	O20-V11-O22	162.7(4)	
O7V7O33	177.0(4)	O20-V11-O21	85.6(4)	
O7V7O28	100.3(4)	O11-V11-O40	99.6(4)	
O7–V7–O25	94.0(4)	O11–V11–O31	175.8(3)	
O7–V7–O24	97.3(4)	O11-V11-O22	98.7(5)	
O26-V6-O34	108.9(4)	O11-V11-O21	98.9(4)	
O25-V6-O34	109.1(4)	O11-V11-O20	97.8(5)	
O25-V6-O26	116.9(3)	O41-V10-O42	86.3(4)	
06-V6-O34	105.7(4)	O35-V10-O42	161.6(3)	
O6-V6-O26	106.3(4)	O35V10O41	88.5(4)	
O6-V6-O25	109.3(4)	O34–V10–O42	95.1(4)	
O29–V5–O37	104.7(5)	O34–V10–O41	162.9(3)	
O28V5O37	102.5(4)	O34–V10–O35	84.9(4)	
O28-V5-O29	123.5(4)	O32-V10-O42	85.6(3)	
O5-V5-O37	107.3(4)	O32-V10-O41	78.4(3)	
O5-V5-O29	108.9(4)	O32–V10–O35	76.1(3)	
O5-V5-O28	108.6(5)	O32-V10-O34	84.6(3)	
O20-V4-O27	106.3(4)	O10-V10-O42	98.4(4)	
O19-V4-O27	120.5(4)	O10-V10-O41	97.6(4)	
O19–V4–O20	105.2(4)	O10-V10-O35	99.8(4)	
O4-V4-O27	108.4(4)	O10-V10-O34	99.1(4)	
O4–V4–O20	105.3(5)	O10-V10-O32	174.3(4)	
O4–V4–O19	109.9(4)	O31-V9-O38	80.1(3)	
O29-V3-O30	78.2(3)	O29-V9-O38	87.7(4)	
O19–V3–)30	84.8(3)	O29-V9-O31	82.6(3)	
O19V3O29	76.7(4)	O21-V9-O38	93.4(4)	
O18V3O30	76.8(3)	O21-V9-O31	78.5(3)	
O18-V3-O29	151.0(4)	O21–V9–O29	160.5(3)	
O18-V3-O19	86.5(5)	019–V9–038	156.1(4)	
017-V3-030	74.3(3)	019-V9-031	79.6(3)	
017-V3-029	87.4(4)	019-V9-029	77.4(4)	
017-V3-019	156.0(4)	019-V9-021	94.7(4)	
017 - V3 - 018	99.9(5)	09-038	99.8(4)	
03 - V3 - 030	176.4(4)	O9-V9-O31	179.1(5)	
$O_3 = V_3 = O_2 Y_3$	101.1(4)	$O_{2} = V_{2} = O_{2}$	98.3(5) 170.1(5)	
$O_3 V_3 O_2^0$	90.3(4) 101.1(4)	$O_{9} - v_{9} - O_{21}$	08 2 (5)	
$O_3 V_3 O_{10}$	104.0(4)	$O_{9} = V_{9} = O_{19}$	100.7(4)	
03-V3-018	102.2(3)	$O_{0} V_{0} O_{10}$	100 7(4)	
027 - 72 - 030 03 - V3 - 017	102 2(5)	O35_V8_O36	93 2(4)	
$O_{27}-V_{2}-O_{30}$	76 8(3)	032	80.9(4)	
O26 - V2 - O30	85.0(3)	032-V8-035	79.2(3)	
O26-V2-O27	79-6(4)	027-V8-036	89.3(4)	
O18-V2-O30	74.7(3)	O27-V8-O35	161.2(4)	
O18-V2-O27	89.2(4)	O27V8O32	82.9(3)	
O18-V2-O26	158.7(4)	O26-V8-O36	159.2(3)	
O16-V2-O30	76.2(4)	O26-V8-O35	92.7(4)	
O16-V2-O27	151.5(4)	O26-V8-O32	80.7(3)	
O16-V2-O26	89.1(3)	O26-V8-O27	79.0(4)	
O16-V2-O18	92.3(4)	O8-V8-O36	98.0(5)	
O2-V2-O30	177.1(3)	O8-V8-O35	100.8(4)	
O2-V2-O27	103.5(5)	O8V8O32	178.9(5)	
O2-V2-O26	97.9(4)	O8V8O27	97.3(4)	
O2-V2-O18	102.3(4)	O8–V8–O26	100.4(5)	
O2-V2-O16	104.0(5)	O33–V7–O39	79.0(4)	
O28-V1-O30	83.3(3)	O28–V7–O39	94.0(3)	
O25-V1-O30	78.2(3)	O28–V7–O33	80.2(3)	
O25-V1-O28	79.0(3)	O25-V7-O39	161.7(4)	
O17-V1-O30	73.9(4)	O25-V7-O33	83.2(3)	
O17-V1-O28	81.4(4)	O25-V7-O28	78.6(3)	
O17-V1-O25	147.5(4)	O24–V7–O39	93.5(4)	
O16-V1-O30	77.3(4)	O24–V7–O33	81.6(3)	
O16V1O28	160.4(4)	O24–V7–O28	158.7(4)	
O16V1O25	93.7(3)	O24–V7–O25	88.2(3)	

(continued)

TABLE 2. (continued)

O20-V11-O31	83.2(4)	
O20-V11-O40	96.5(4)	
O21-V11-O22	86.6(3)	
O21-V11-O31	77.0(3)	
O21V11O40	160.9(4)	
O22V11O31	79.9(4)	
O22-V11-O40	86.0(3)	
O31-V11-O40	84.3(4)	
O12-V12-O23	97.5(4)	
O12-V12-O33	174.4(4)	
O12-V12-O37	99.7(4)	
O12-V12-O39	99.4(5)	
O12-V12-O43	97.9(5)	
O23-V12-O33	79.2(3)	
O23-V12-O37	162.3(4)	
O23-V12-O39	87.0(4)	
O23-V12-O43	86.9(3)	
O33-V12-O37	83.4(3)	
O33-V12-O39	76.1(3)	
O33-V12-O43	86.4(4)	
O37-V12-O39	85.8(4)	
O37-V12-O43	95.2(4)	
O39-V12-O43	162.3(3)	

The structure of the complex is shown in Fig. 1. The overall structure may be viewed in terms of the hypothetical  $\{AsV_{12}O_{40}\} \in Keggin structure with three$ [VO] vertices removed and capped by three [VO] and three [AsOH] units. The 'open' pole of the  $[As_4V_{12}O_{43}H_3]^{8-}$  cluster is capped by a potassium cation and two cluster units are bridged by a second potassium cation, as shown in Fig. 2. There are four distinct V environments: the tetrahedral centers (V4, V5 and V6), the pseudo-octahedral sites which participate in long range bonding to the quadruply bridging oxo group of the central [AsO<sub>4</sub>] group (V1, V2 and V3), the octahedral sites which participate in three V-O-As interactions (V10, V11 and V12), and the octahedral sites which exhibit two V-O-As interactions (V7, V8 and V9).

Solid-state magnetic susceptibility studies on  $K_7Na[As_4V_{12}O_{43}H_3] \cdot 7H_2O$  gave an effective magnetic moment of 3.59  $\mu_B$ , which corresponds to 1.61  $\mu_B$  per  $V^{IV}$  center. Thus, the five  $V^{IV} d^1$  centers are practically uncoupled, an observation consistent with the relatively small number of spins, which are effectively trapped as far apart as possible to give rise to approximately spin-only values for  $\mu_{eff}$  per  $V^{IV}$ . The one-electron oxidized form of the cluster  $[As_4V_{12}O_{43}H_3]^{7-}$  [14] has been reported to possess four  $V^{IV}$  centers with an effective moment of 1.75  $\mu_B$  per  $V^{IV}$ . Other examples of trapped  $V^{IV}$  sites in polyvanadate clusters include  $[V_{14}AsO_{40}]^{7-}$  [17] and  $[V_{12}As_8O_{40}(HCO)_2]^{3-}$  [18]. As demonstrated most elegantly by the recent work of Müller *et al.* [18], antiferromagnetically coupled systems are observed when the  $V^{IV}/V^V$  ratio is greater than



Fig. 1. ORTEP view of the structure of  $[KAs_4V_{12}O_{43}H_3]^7$ , showing the capping of the  $[As_4V_{12}O_{43}H_3]^{8^-}$  anion by K1.

0.5, resulting in complicated magnetic behavior arising from both trapped and delocalized  $V^{IV}$  centers.

Valence sum calculations [19] suggest that three spins are trapped on the V10, V11 and V12 centers, while two spins are delocalized in the cluster. The oxidized species  $[As_4V_{12}O_{43}H_3]^{7-}$  exhibits a pattern of three trapped spins and one delocalized. The isolation of the  $[As_4V_{12}O_{43}H_3]^{8-}$  mixed valence cluster demonstrates that the aggregates of the family of arseno-vanadates exist with different electron populations and that subtle variations in reaction conditions allow isolation of species in different overall cluster oxidation states.

#### Acknowledgement

This work was supported by NSF Grant No. CHE8815299.

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Fig. 2. Perspective view of two  $[KAs_4V_{12}O_{43}H_3]^{7-}$  units bridged by a potassium cation (K2) located on a crystallographic two-fold axis.

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