CONTRIBUTION FROM THE U. S. GEOLOGICAL SURVEY
WASHINGTON, D. C.

The Molecular Structure of the Isopoly Complex Ion, Decavanadate ($V_{10}O_{28}{}^{6-}$)[1]

BY HOWARD T. EVANS, JR.

Received November 1, 1965

The structure of the decavanadate ion $V_{10}O_{28}{}^{6-}$ has been found by a determination of the crystal structure of $K_2Zn_2V_{10}O_{28}\cdot 16H_2O$. The soluble, orange crystals are triclinic with space group $P\bar{1}$ and have a unit cell with $a = 10.778$ A, $b = 11.146$ A, $c = 8.774$ A, $\alpha = 104° 57'$, $\beta = 109° 32'$, and $\gamma = 65° 0'$ ($Z = 1$). The structure was solved from a three-dimensional Patterson map based on 5143 Weissenberg-film data. The full-matrix, least-squares refinement gave $R = 0.094$ and σ for V–O bond lengths of 0.008 A. The unit cell contains one $V_{10}O_{28}{}^{6-}$ unit, two $Zn(H_2O)_6{}^{2+}$ groups, two K^+ ions, and four additional water molecules. The decavanadate ion is an isolated group of ten condensed VO_6 octahedra, six in a rectangular 2 × 3 array sharing edges, and four more, two fitted in above and two below by sharing sloping edges. The structure, which is based on a sodium-chloride-like arrangement of V and O atoms, has a close relationship to other isopoly complex molybdates, niobates, and tantalates. Strong distortions in the VO_6 octahedra are analogous to square-pyramid and other special coordination features known in other vanadate structures.

Introduction

A peculiar property of aqueous vanadate(V) solutions (V_2O_5 dissolved in alkali) is that the colorless alkaline solutions, as acid is added, suddenly turn bright orange when the degree of acidity passes about pH 6.5. This phenomenon is associated with one of the condensation reactions that the vanadate ion undergoes in various regions of the pH scale. This behavior of forming higher and higher molecular weight, isopolynuclear complex anions as the solution becomes more and more acid is shared especially by the transition metals in groups V and VI (left side of the table; V, Nb, Mo, W, etc.). The complicated chemistry of vanadium and similar elements in such systems is the subject of a vast literature, a complete review of which cannot be attempted here.

The behavior of vanadium(V) in solution as a function of acidity was first clearly delineated by the diffusion rate studies of Jander and Jahr.[2] They showed that vanadium diffused through the solution at varying rates at different pH conditions but at fairly constant rate over certain ranges, so that the variation has a steplike character. By relating the diffusion rate to molecular weight, they proposed formulas for five different ionic species, each stable over a given range, and increasing in nuclearity and molecular weight with decreasing pH down to the isoelectric point at about pH 1.8. These have been referred to as orthovanadate (colorless, pH >12.6), pyrovanadate (colorless, pH 9.6–12.6), metavanadate (colorless, pH 6.5–9.6), polyvanadate (orange, pH 2.0–6.5), and pervanadyl (pale yellow, pH <0.8). Jander and Jahr proposed the formulation $V_5O_{16}{}^{7-}$ for the polyvanadate ion, but their method of molecular weight determination from diffusion rates has proved to be very unreliable. The proper structural formulations of all these species are still very controversial, in spite of the enormous amount of effort devoted to their elucidation by every physical and chemical method. The most dependable results so far have apparently been given by precision hydrogen ion analysis by emf methods of solutions containing 3 M $NaClO_4$ and varying concentrations of vanadate, as carried out by the highly developed techniques used by Professor L. G. Sillén's group at the Royal Institute of Technology at Stockholm. Vanadate(V) solutions in the pH range 1 to 6.5 have been studied in this way by Rossotti and Rossotti,[3] and in the range 7 to 10 by Brito and Ingri,[4a] and by Ingri and Brito.[4b] Previous chemical studies have been briefly reviewed in these papers.

The particular subject of this paper is the nature and structure of the complex occurring in the orange vanadate(V) solutions in the pH range 2 to 6.5. The brightly colored complex is immediately formed on adding acid as the pH passes below 6.5 and is largely destroyed when the pH is raised again above this point. A yellowish color persists for a long time, however, indicating that the reaction in the vicinity of pH 6.5 is sluggish. Below pH 2.0 the isoelectric point is reached and a brown precipitate of V_2O_5 hydrate is formed. This redissolves on further acidification, giving vanadyl(V) (pervanadyl) ion, usually written as $VO_2{}^+$. The orange solutions readily deposit beautiful, large, orange crystals, which apparently are generally various hydrates of salts of the orange complex contained in the solution. If these solutions are warmed, however, these crystals disappear, and a sparingly soluble trivanadate compound of the type KV_3O_8 precipitates as pale orange, flakelike crystals. This process also occurs at room temperature over a period of weeks, thus suggesting that the orange polyvanadate complex is actually metastable with respect to the solid trivanadate. Near the transformation point at pH 6.5 garnet-red crystals of a pentavanadate $K_3V_5O_{14}$ sometimes form on gentle heating of potassium vanadate solutions, before the trivanadate is formed. The crystal structures of KV_3O_8[5] and $K_3V_5O_{14}$[6] are

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[1] Publication authorized by the Director, U. S. Geological Survey.

[2] G. Jander and K. F. Jahr, *Z. Anorg. Allgem Chem.*, **212**, 1 (1933).

[3] F. J. C. Rossotti and H. Rossotti, *Acta Chem. Scand.*, **10**, 957 (1956).

[4] (a) F. Brito and N. Ingri, *Anales Real Soc. Espan. Fis. Quim.* (Madrid), **B56**, 165 (1960); (b) N. Ingri and F. Brito, *Acta Chem. Scand.*, **13**, 1971 (1959).

[5] S. Block, *Nature*, **186**, 540 (1960).

[6] A. M. Byström and H. T. Evans, Jr., *Acta Chem. Scand.*, **13**, 377 (1959).

both based on unusual types of layer structures and give no clue as to the nature of the ionic species in the solution.

The emf studies of Rossotti and Rossotti[3] established with fair certainty the predominant existence in the orange solutions of three complex ions: $V_{10}O_{28}^{6-}$, $HV_{10}O_{28}^{5-}$, and $H_2V_{10}O_{28}^{4-}$. In sodium solutions 3 *M* in $NaClO_4$, the constants for the equilibria between these ions in terms of concentrations were found to be

$$10VO_2^+ + 8H_2O \rightleftharpoons H_2V_{10}O_{28}^{4-} + 14H^+, \quad \log k = -6.75 \pm 0.15$$

$$H_2V_{10}O_{28}^{4-} \rightleftharpoons HV_{10}O_{28}^{5-} + H^+, \quad \log k = -3.6 \pm 0.3$$

$$HV_{10}O_{28}^{5-} \rightleftharpoons V_{10}O_{28}^{6-} + H^+, \quad \log k = -5.8 \pm 0.1$$

The existence of the decavanadate ion as a major species in the orange solutions has been confirmed by Schwartzenbach and Parissakis[7] from cryoscopic studies based on the $Na_2SO_4 \cdot 10H_2O$–Na_2SO_4 transition and from more extensive measurements by Naumann and Hallada.[8] Their results appear to be completely consistent with those of Rossotti and Rossotti.[3] Further confirmation has been provided by absorption and potentiometric studies of Chauveau,[9] by potentiometric and conductometric studies of Magee and Richardson,[10] and by absorption and potentiometric studies of Schiller and Thilo.[11] Crystallographic evidence for the tenfold multiplicity of the complex has been given by unit-cell determinations of $K_6V_{10}O_{28} \cdot 10H_2O$ (monoclinic and triclinic), $Ca_3V_{10}O_{28} \cdot 16H_2O$ (monoclinic), $K_2Mg_2V_{10}O_{28} \cdot 16H_2O$ (triclinic), and $K_2Zn_2V_{10}O_{28} \cdot 16H_2O$ (triclinic) by Evans, Mrose, and Marvin.[12]

Crystallized Decavanadates

Since Berzelius[13] first investigated the chemistry of vanadium, countless compounds have been described as forming well-formed, orange-red to garnet-red, soluble crystals from orange, alkali or alkaline earth vanadate(V) solutions that have been acidified, often with acetic acid. Berzelius formulated them as tetravanadates, for example, $(NH_4)_2V_4O_{11} \cdot 2H_2O$. Such products have since been assigned formulas varying over a wide range of multiplicity of the polyvanadate group and basicity; for example, Rammelsberg[14] offered $Li_4V_6O_{17} \cdot 15H_2O$, $Li_6V_8O_{18} \cdot 12H_2O$, $Li_6V_{10}O_{28} \cdot 14H_2O$, and $Li_{10}V_{12}O_{35} \cdot 30H_2O$, among others; Norblad[15] described $Li_2V_4O_{11} \cdot 9H_2O$ and $Ba_3V_{10}O_{28} \cdot 19H_2O$; all of these descriptions refer to the general features mentioned above. It is felt now that most of these polyvanadate salts are actually either $M^I{}_6V_{10}O_{28} \cdot nH_2O$ or $M^I{}_4H_2V_{10}O_{28} \cdot nH_2O$ or the corresponding M^{II} salts or double salts.

The first authors to describe a whole series of such compounds and correctly formulate them in this general way were Fock[16] and Radau[17] though the latter tended to prefer a halved formula: $M^IM^{II}V_5O_{14} \cdot 8H_2O$. These authors placed their trust in their analytical work and, especially on the basis of the double salt series $M^I{}_2M^{II}{}_2V_{10}O_{28} \cdot 16H_2O$, presented the best representation of these polyvanadates that was possible until the last two decades. The decavanadate formulation was then finally established for the solution by modern physical–chemical methods (see above) and for the solids by X-ray crystallographic methods.[12] It has been generally recognized that any further information concerning the structure and constitution of the decavanadate complex would probably come from a crystal structure analysis of one or more of its salts. The present paper reports a detailed structure analysis of *dipotassium dizinc decavanadate 16-hydrate* $K_2Zn_2V_{10}O_{28} \cdot 16H_2O$. A similar study[18] has been carried out independently by Swallow and Barnes on $Ca_3V_{10}O_{28} \cdot 16H_2O$ and will be reported elsewhere. The general results of these two studies are in complete agreement regarding the structure of the decavanadate ion.

Decavanadate complexes are the only ones of the large class of isopoly complexes (molybdates, tungstates, etc.) that are known to occur in nature. The calcium salt has been known as the mineral pascoite since 1914. The double salt $K_2Mg_2V_{10}O_{28} \cdot 16H_2O$ has been described as the mineral hummerite[19] occurring with pascoite in the Paradox Valley region in Colorado. The present study was originally undertaken to extend the general knowledge of the geochemistry of vanadium in the Colorado Plateau sandstones.[20] A considerable effort was expended in this laboratory and other laboratories to solve the structure from two-dimensional projections, but all efforts demonstrated the need for a full three-dimensional analysis.

The compounds studied in this laboratory were $K_2Mg_2V_{10}O_{28} \cdot 16H_2O$, $Cs_2Mg_2V_{10}O_{28} \cdot 16H_2O$, and $K_2Zn_2V_{10}O_{28} \cdot 16H_2O$, but the last was finally selected for the three-dimensional study because of its resistance to dehydration in the X-ray beam and the absence of excessively heavy X-ray scatterers in the crystal. These compounds all belong to the triclinic double salt series $M^I{}_2M^{II}{}_2V_{10}O_{28} \cdot nH_2O$ (M^I = K, Cs, NH_4; M^{II} = Mg, Zn, Mn, Co) first described by Fock[16] and Radau,[17] but the last (K–Zn) was the only one mentioned by them. Radau studied the crystallography of this compound, finding $a:b:c = 0.8025:1:0.9675$, $\alpha = 115° 2'$, $\beta = 109° 39'$, and $\gamma = 74° 57'$. There are inconsistencies in the values he gives for the direct cell and the five goniometrically measured angles from which it was determined, which make it difficult to reconcile his results with the X-ray diffraction results obtained in this work. The best internal consistency in Radau's data is obtained if his $a:b$ ratio is changed

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(9) F. Chauveau, *Bull. Soc. Chim. France*, 810 (1960).

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(11) K. Schiller and E. Thilo, *Z. Anorg. Allgem. Chem.*, **310**, 286 (1961).

(12) H. T. Evans, Jr., M. E. Mrose, and R. Marvin, *Am. Mineralogist*, **40**, 314 (1955).

(13) M. Berzelius, *Phil. Mag.*, **10**, 321 (1831).

(14) C. F. Rammelsberg, *Wiedemann's Ann. Chim.*, **20**, 938 (1883).

(15) J. A. Norblad, *Bull. Soc. Chim. France*, [2] **24**, 64 (1875).

(16) A. Fock, *Z. Krist.*, **17**, 1 (1897).

(17) C. Radau, *Ann. Chem. Liebigs*, **251**, 114 (1889).

(18) H. T. Evans, Jr., A. G. Swallow, and W. H. Barnes, *J. Am. Chem. Soc.*, **86**, 4209 (1964).

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(20) H. T. Evans, Jr., and R. M. Garrels, *Geochim. Cosmochim. Acta*, **15**, 131 (1958).

to 0.7869. The comparison with this work, after Raudau's cell is corrected and reoriented by the matrix $(00\bar{1}|010|\bar{1}00)$, is then

	$a:b:c$	α	β	γ
Radau	0.9675:1:0.7869	105° 3′	109° 39′	64° 58′
X-Ray	0.9670:1:0.7872	104° 57′	109° 32′	65° 0′

In spite of the errors in Radau's work, the agreement is excellent and there can be no question about the identity of the two crystals.

Experimental Section

Preparation and Measurement of Crystallographic and Intensity Data.—Crystals of $K_2Zn_2V_{10}O_{28}\cdot 16H_2O$ were obtained by evaporating solutions containing equivalent amounts of KVO_3 and $ZnAc_2$, adjusted to about pH 5 with acetic acid. The stubby, orange crystals are triclinic and have a prismatic habit that tends to become elongated parallel to the b axis. The unit cell was determined from Buerger-precession, single-crystal photographs, and the parameters refined by least-squares analysis of powder diffraction data recorded on a Norelco diffractometer. The cell parameters are given in Table I. The powder data have been reported by Swanson, Morris, Evans, and Ulmer.[21] The space group, to judge from the crystal habit, lack of piezoelectric response, and statistical criteria based on the normalized structure amplitudes, is centrosymmetric, namely $P\bar{1}$ (no. 2). Assuming that the unit cell contains one formula unit, the calculated density is 2.708 g cm^{-3}, which is in excellent agreement with the value of 2.70 ± 0.01 g cm^{-3} as measured with the Berman balance.

A complete set of 5143 three-dimensional data was collected from an equant crystal about 0.2 mm in diameter on nonintegrated, multiple-film, equiinclination Weissenberg photographs made with Mo Kα radiation, comprising all reflections for which $(\sin\theta)/\lambda < 0.7$ A^{-1}. Thirteen levels were recorded around the b axis and eight levels around the c axis. The intensities were estimated by visual comparison with a calibrated strip. The few low-angle reflections inaccessible on the Mo Kα patterns were measured from films made with Cu Kα radiation. All data were corrected for the Lorentz and polarization effects and then reduced to a common scale by intercomparison of reflections common to the two sets of photographs. No corrections were made for absorption or extinction.

Structure Determination.—The relative $|F|^2$ values were normalized and the resulting $(E^2 - 1)$ values were used to calculate a sharpened Patterson map with origin peak removed. This map showed heavy and recurrent peaks in a triangular array on two parallel sheets tilted slightly with respect to the x–y plane, parallel to which the Fourier sections were computed. One sheet passes through the origin and another sheet is at a distance of 2.9 A from the first. The peaks in these sheets were understood to represent V–V vectors arising from within the molecule, but because of the large number of superpositions of similar vectors it was not possible to discern the exact arrangement of vanadium atoms from them. Another group of ten smaller but quite sharp peaks was found elsewhere in the cell, which reflected the same triangular array of peaks found in the main system. This group was finite and possessed a center of symmetry, suggesting that these peaks represented the Zn–V vectors associated with one molecule. With the coordinates of the symmetry center of the group measured at x, y, z, a single peak was found at $2x$, $2y$, $2z$, of a height appropriate for a Zn–Zn vector. These eleven peaks were used to establish coordinates for a first trial arrangement of six atoms in the asymmetric unit, Zn + 5V. Structure factors based on these atoms alone gave $R = 0.33$ for all of the observed data. From this calculation, signs were selected for 2996 terms which were used for the first three-dimensional electron density calculation. This map revealed the positions of all 29 atoms in the asymmetric unit of structure as peaks of good shape and expected density, with no large, spurious background effects.

Coordinates of all atoms taken directly from the initial electron density map were submitted to refinement by full-matrix, least-squares analysis (with isotropic temperature factors and scale factor, 117 variables). Nine cycles were calculated, and in the last two cycles the data were weighted according to $\sqrt{w} = 4F_{\min}/F_o$ for $F_o < 4F_{\min}$ and $\sqrt{w} = F_o/4F_{\min}$ for $F_o < 4F_{\min}$, where $4F_{\min}$ was set at 18.2. The final standard error of F of weight unity was 2.3, and the final reliability factor (nonzero data only) was $R = 0.094$. The observed and calculated structure factors are listed in Table II.

TABLE I

STRUCTURAL PARAMETERS FOR $K_2Zn_2V_{10}O_{28}\cdot 16H_2O$

(TRICLINIC, SPACE GROUP NO. 2, $P\bar{1}$)

Unit cell

a = 10.778 ± 3 A	α = 104° 56.8 ± 1.3′
b = 11.146 ± 3 A	β = 109° 32.1 ± 1.5′
c = 8.774 ± 3 A	γ = 65° 0.0 ± 1.8′
V = 891.9 A^3	Z = 1

Atomic Parameters: x, y, z in Fractions of Cell Edges, B in A^2, σ (in parentheses) in Terms of Last Significant Figures (All Atoms in 2(i))

Atom	x	y	z	B
		$V_{10}O_{28}^{6-}$		
V_1	0.2190 (2)	0.5529 (2)	0.3845 (2)	0.95 (3)
V_2	0.4490 (2)	0.2756 (2)	0.3530 (2)	0.87 (3)
V_3	0.2718 (2)	0.8027 (2)	0.3655 (2)	1.10 (3)
V_4	0.5026 (2)	0.5225 (2)	0.3220 (2)	0.78 (3)
V_5	0.7372 (2)	0.2442 (2)	0.3083 (2)	1.07 (3)
O_1	0.6439 (7)	0.5898 (6)	0.4769 (8)	0.93 (9)
O_2	0.4380 (7)	0.8359 (7)	0.4912 (9)	1.40 (10)
O_3	0.5984 (7)	0.3797 (6)	0.4854 (8)	1.02 (9)
O_4	0.8065 (7)	0.1278 (7)	0.4593 (9)	1.49 (10)
O_5	0.8437 (7)	0.3443 (7)	0.4369 (8)	1.28 (10)
O_6	0.1010 (8)	0.4871 (8)	0.2936 (10)	1.86 (12)
O_7	0.3271 (8)	0.2163 (8)	0.2595 (10)	2.00 (12)
O_8	0.1605 (7)	0.6983 (7)	0.2786 (8)	1.34 (10)
O_9	0.3596 (7)	0.4469 (6)	0.2579 (8)	1.03 (9)
O_{10}	0.5682 (7)	0.2061 (7)	0.2223 (8)	1.27 (10)
O_{11}	0.1903 (9)	0.9231 (9)	0.2568 (11)	2.41 (14)
O_{12}	0.4018 (7)	0.6637 (7)	0.2298 (8)	1.36 (10)
O_{13}	0.6082 (7)	0.4171 (7)	0.2020 (8)	1.26 (10)
O_{14}	0.8237 (8)	0.1611 (8)	0.1736 (10)	1.96 (12)
K	0.0832 (3)	0.2255 (3)	0.3146 (3)	2.36 (4)
		$Zn(H_2O)_6^{2+}$		
Zn	0.7291 (1)	0.7900 (1)	0.2253 (1)	1.37 (2)
O_{15}	0.6273 (10)	0.8990 (9)	0.4087 (11)	2.61 (14)
O_{16}	0.8247 (9)	0.6305 (8)	0.3678 (10)	2.20 (13)
O_{17}	0.8958 (8)	0.8569 (8)	0.3755 (10)	1.82 (11)
O_{18}	0.5587 (11)	0.7316 (11)	0.0933 (13)	3.43 (18)
O_{19}	0.6564 (9)	0.9406 (8)	0.0828 (11)	2.27 (13)
O_{20}	0.8458 (10)	0.6632 (9)	0.0586 (11)	2.68 (15)
		Additional Water		
O_{21}	0.8461 (12)	0.4081 (11)	0.0694 (14)	3.55 (19)
O_{22}	0.9189 (12)	0.8884 (11)	0.0077 (14)	3.62 (19)

The Crystal Structure of $K_2Zn_2V_{10}O_{28}\cdot 16H_2O$

The final structural parameters for the crystal structure of $K_2Zn_2V_{10}O_{28}\cdot 16H_2O$ are listed in Table I. A detailed list of interatomic distances with associated standard errors is given in Table III. The arrangement of the atoms in the unit cell is shown in Figure 1a and may be compared with the final electron density

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(21) H. E. Swanson, M. C. Morris, E. H. Evans, and L. Ulmer, National Bureau of Standards Monograph No. 25, U. S. Government Printing Office, Washington, D. C., 1964, Section 3, p 45.

TABLE II

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR $K_2Zn_2V_{10}O_{28}\cdot16H_2O$

H	K	L	F-OBS	F-CAL
0	0	1	*	-132.7
0	0	2	*	-131.1
0	0	3	111.3	117.0
0	0	4	11.2	7.0
0	0	5	11.2	-9.1
0	0	6	15.9	16.5
0	0	7	58.6	-65.2
0	0	8	12.9	11.4
0	0	9	21.5	15.9
0	0	10		4.7
0	0	11		-11.1
1	0	0	*	-88.0
1	0	1	*	66.9
1	0	2	113.9	120.0
1	0	3	31.1	29.0
1	0	4	38.9	-41.4
1	0	5	61.2	66.5
1	0	6	29.4	-32.3
1	0	7	71.5	79.7
1	0	8	34.0	-34.5
1	0	9	43.8	-50.2
1	0	10	36.4	38.9
1	0	11		10.3
2	0	0	89.0	-98.2
2	0	1		-2.4
2	0	2	30.4	33.3
2	0	3	43.8	46.8
2	0	4	11.2	-11.4
2	0	5	52.3	-59.1
2	0	6	68.2	70.1
2	0	7	7.9	5.8
2	0	8	24.7	-26.2
2	0	9	41.3	40.5
2	0	10		-12.8
3	0	0	77.4	-78.6
3	0	1	30.1	-29.0
3	0	2	9.1	-5.1
3	0	3	43.3	47.3
3	0	4	9.1	4.8
3	0	5	6.4	-2.6
3	0	6	6.4	9.1
3	0	7	29.0	-32.4
3	0	8	33.1	31.2
3	0	9		15.1
3	0	10	27.1	-28.7
4	0	0	23.4	20.4
4	0	1	31.8	-29.6
4	0	2	75.7	-79.6
4	0	3	41.0	45.3
4	0	4	36.4	-36.4
4	0	5		3.3
4	0	6	42.0	44.4
4	0	7	38.4	-40.6
4	0	8		-5.0
4	0	9	34.0	28.5
4	0	10		3.0
5	0	0	23.4	-22.6
5	0	1		-2.5
5	0	2	103.9	104.4
5	0	3	130.2	-160.3
5	0	4		.3
5	0	5	53.3	72.1
5	0	6	50.3	-49.8
5	0	7		-5.0
5	0	8	25.5	27.2
5	0	9	41.5	-43.6
6	0	0	51.9	-50.7
6	0	1		-.5
6	0	2	12.1	10.6
6	0	3	14.5	14.0
6	0	4		3.9
6	0	5	41.8	-45.5
6	0	6	14.5	-13.9
6	0	7	41.8	42.6
6	0	8	26.3	-25.3
7	0	0	14.5	14.0
7	0	1	10.2	-9.7
7	0	2	12.9	-8.0
7	0	3	12.1	10.3
7	0	4		10.2
7	0	5	12.9	8.7
7	0	6	14.5	-8.5
7	0	7	25.5	-25.3
7	0	8		16.9
8	0	0	22.9	19.0
8	0	1	46.1	44.6
8	0	2	37.8	-36.9
8	0	3		-2.0
8	0	4		4.3
8	0	5		.2
8	0	6		10.9
8	0	7		2.9
9	0	0	34.6	-36.7
9	0	1	22.9	19.5
9	0	2	25.1	26.8
9	0	3	12.9	-11.5
9	0	4		-8.7
9	0	5	13.7	12.3
9	0	6	12.9	-15.8
10	0	0	14.5	-10.3
10	0	1	26.3	-25.9
10	0	2		-.4
10	0	3		5.2
10	0	4		14.0
10	0	5		-8.2
11	0	0	43.0	41.9
11	0	1	15.9	-14.1
11	0	2	22.5	-23.8
11	0	3		4.7
11	0	4		3.6
12	0	0	15.9	14.9
12	0	1	18.3	16.0
12	0	2		-5.6
13	0	0	25.5	-26.2
1	0	-1	27.9	23.9
1	0	-2	54.6	-62.8
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-8	3	-6	21.0	25.7
-9	3	-1	21.5	-45.0
-9	3	-2		-9.0
-9	3	-3	22.9	22.8
-9	3	-4		2.4
-9	3	-5		-11.3
-10	3	-1	35.2	34.5
-10	3	-2		-2.1
-10	3	-3	42.8	-44.8
-10	3	-4	24.3	25.5
-11	3	-1		6.6
-11	3	-2		9.8
0	4	0	57.7	63.6
0	4	1	25.1	-17.9
0	4	2	34.0	-33.0
0	4	3	49.4	47.5
0	4	4		5.2
0	4	5		8.9
0	4	6	24.7	-24.5
0	4	7		5.2
0	4	8		8.9
0	4	9		-2.3
0	4	10		6.2
1	4	0	79.0	-75.0
1	4	1	11.2	9.1
1	4	2	50.5	-50.0
1	4	3	133.5	-138.3
1	4	4	142.3	164.0
1	4	5	34.3	-37.3
1	4	6	48.6	-46.8
1	4	7	45.9	54.5
1	4	8	21.0	-19.0
1	4	9		-4.2
1	4	10	31.8	32.3

TABLE II (*Continued*)

H	K	L	F-OBS	F-CAL
2	4	0	36.4	34.3
2	4	1	49.2	-46.1
2	4	2	12.1	-8.9
2	4	3	41.0	40.0
2	4	4	93.5	-103.0
2	4	5	29.4	-28.8
2	4	6	73.7	78.6
2	4	7	36.4	-39.0
2	4	8	36.4	-30.9
2	4	9	39.2	36.0
2	4	10	19.4	-21.2
3	4	0	12.1	10.8
3	4	1	113.9	-99.1
3	4	2	33.1	26.9
3	4	3	68.8	68.9
3	4	4	50.9	-54.2
3	4	5		-1.0
3	4	6		-6.3
3	4	7		-2.6
3	4	8	23.8	19.9
3	4	9		-24.9
4	4	0	178.2	192.6
4	4	1	135.1	-148.8
4	4	2	25.1	-21.0
4	4	3	51.3	53.1
4	4	4	26.7	-25.4
4	4	5		5.6
4	4	6		-3.8
4	4	7	17.7	-18.3
4	4	8		3.1
4	4	9		13.1
5	4	0	140.6	-139.9
5	4	1	162.5	183.4
5	4	2	61.1	61.5
5	4	3	82.6	-88.6
5	4	4		-7.8
5	4	5	60.0	63.0
5	4	6	34.0	-30.0
5	4	7	14.5	14.3
5	4	8	21.0	-20.0
5	4	9		-10.3
6	4	0	50.3	-51.0
6	4	1	22.5	21.6
6	4	2	20.5	20.6
6	4	3	22.5	18.6
6	4	4		10.7
6	4	5	29.7	-33.5
6	4	6	51.1	55.3
6	4	7	28.6	-32.1
6	4	8		8.7
7	4	0		-3.8
7	4	1	50.3	-53.1
7	4	2	14.5	11.2
7	4	3	29.0	24.2
7	4	4		-1.4
7	4	5	12.9	10.2
7	4	6	17.1	-16.7
7	4	7		1.8
7	4	8		31.7
8	4	0	32.4	33.6
8	4	1		4.0
8	4	2	42.0	-39.8
8	4	3		-5.9
8	4	4	23.4	23.0
8	4	5		.6
8	4	6		-9.3
8	4	7		3.2
9	4	0		-1.2
9	4	1	33.1	32.0
9	4	2	15.9	15.6
9	4	3	50.3	-54.6
9	4	4	15.9	16.0
9	4	5	17.7	23.1
9	4	6	20.0	-21.7
10	4	0	39.7	-40.4
10	4	1		1.0
10	4	2	17.7	17.3
10	4	3	15.2	17.4
10	4	4	23.8	-28.9
10	4	5		-14.8
11	4	0	19.4	18.4
11	4	1		-3.9
11	4	2	20.0	-19.1
11	4	3	12.1	14.9
11	4	4	13.7	15.6
12	4	0	15.9	13.5
12	4	1	22.0	22.0
12	4	2	22.0	-19.4
12	4	3		-7.5
13	4	0		-.1
13	4	1		7.7
13	4	2		-.0
14	4	0		1.8
0	4	-1		-6.6
0	4	-2	226.8	-234.9
0	4	-3	144.5	151.7
0	4	-4	50.5	-47.6
0	4	-5	96.7	-97.8
0	4	-6	68.4	75.0
0	4	-7	17.7	-17.8
0	4	-8	46.3	-51.1
0	4	-9	54.9	55.3
0	4	-10		4.6
0	4	-11		-8.5
1	4	-1	6.4	-7.6
1	4	-2	77.5	-67.9
1	4	-3	39.2	-31.0
1	4	-4	85.4	84.6
1	4	-5	13.7	-12.3
1	4	-6	18.3	-15.7
1	4	-7		7.7
1	4	-8		6.8
1	4	-9	32.1	27.2
1	4	-10	20.5	-17.8
1	4	-11	22.0	-24.0
2	4	-1	40.5	-34.7
2	4	-2	32.4	27.2
2	4	-3	50.5	41.7
2	4	-4	11.2	10.0
2	4	-5	35.2	-33.4
2	4	-6	21.5	-25.4
2	4	-7	49.6	43.6
2	4	-8		-6.9
2	4	-9		-12.5
2	4	-10		-4.0
2	4	-11		.6
3	4	-1	42.0	41.9
3	4	-2	11.2	8.8
3	4	-3	22.9	-17.3
3	4	-4	15.9	-11.4
3	4	-5	20.0	20.6
3	4	-6	18.3	-21.0
3	4	-7	51.5	53.7
3	4	-8		2.3
3	4	-9	40.0	-44.5
3	4	-10		8.5
3	4	-11	30.1	31.4
3	4	-12	32.4	-27.8
4	4	-1	53.1	54.5
4	4	-2	48.6	-49.5
4	4	-3	16.5	-19.0
4	4	-4	27.9	27.0
4	4	-5	22.9	25.9
4	4	-6	52.5	61.7
4	4	-7	79.8	-87.0
4	4	-8	17.7	-9.3
4	4	-9	16.5	15.0
4	4	-10	23.4	20.3
4	4	-11	25.1	-21.1
4	4	-12	20.6	-17.7
5	4	-1	17.7	19.3
5	4	-2	12.9	12.1
5	4	-3		3.9
5	4	-4	23.4	22.1
5	4	-5	15.2	-14.7
5	4	-6	43.3	-47.3
5	4	-7		1.2
5	4	-8	25.9	17.8
5	4	-9	16.5	14.8
5	4	-10	25.5	-22.9
5	4	-11		-5.2
5	4	-12	19.2	22.6
6	4	-1	14.5	-14.2
6	4	-2	50.7	46.0
6	4	-3	21.5	-18.2
6	4	-4	9.1	-6.2
6	4	-5	14.5	-12.1
6	4	-6	24.7	-25.2
6	4	-7	41.3	40.8
6	4	-8	22.5	21.0
6	4	-9	44.2	-44.3
6	4	-10	20.0	10.4
6	4	-11	36.1	32.4
6	4	-12		-8.3
7	4	-1	40.0	38.9
7	4	-2		-6.7
7	4	-3	37.5	-35.6
7	4	-4		-2.2
7	4	-5	32.8	33.1
7	4	-6		2.1
7	4	-7		7.8
7	4	-8		-10.0
7	4	-9	15.9	11.0
7	4	-10		13.4
7	4	-11		.7
7	4	-12		-.8
8	4	-1	22.5	-18.8
8	4	-2		-2.6
8	4	-3	12.1	12.6
8	4	-4	10.2	9.7
8	4	-5	20.5	17.9
8	4	-6		-7.8
8	4	-7		5.0
8	4	-8		-8.3
8	4	-9	22.9	18.9
8	4	-10		13.5
8	4	-11	33.7	-32.0
9	4	-1	31.4	-33.2
9	4	-2	32.8	28.1
9	4	-3		5.0
9	4	-4	22.9	-17.2
9	4	-5	26.7	22.0
9	4	-6		2.3
9	4	-7	35.8	-29.1
9	4	-8	48.8	46.6
9	4	-9		3.6
9	4	-10	46.1	-43.9
9	4	-11	22.5	19.4
10	4	-1	18.9	13.9
10	4	-2	28.6	29.1
10	4	-3		-2.5
10	4	-4		1.6
10	4	-5		-8.5
10	4	-6	12.9	13.3
10	4	-7	18.9	14.5
10	4	-8	16.5	-9.3
10	4	-9	31.8	-27.6
10	4	-10		3.6
10	4	-11		13.2
11	4	-1	20.5	21.4
11	4	-2	15.9	-11.7
11	4	-3		-2.9
11	4	-4		-11.6
11	4	-5		-6.6
11	4	-6	31.1	29.0
11	4	-7	39.5	-32.6
11	4	-8	37.8	-31.7
11	4	-9	36.1	30.8
11	4	-10		-4.6
12	4	-1	24.3	-19.7
12	4	-2	31.1	-29.5
12	4	-3	17.7	6.4
12	4	-4	23.8	25.8
12	4	-5	24.7	-22.5
12	4	-6	16.5	-19.3
12	4	-7	24.7	24.5
12	4	-8	30.4	-22.5
12	4	-9	22.5	15.7
12	4	-10	25.1	23.8
13	4	-1	39.5	-41.2
13	4	-2	32.4	31.2
13	4	-3	22.5	17.4
13	4	-4	38.1	-36.1
13	4	-5	21.0	20.5
13	4	-6	17.7	-12.4
13	4	-7	25.9	-20.2
13	4	-8	44.7	46.4
13	4	-9		-20.9
14	4	-1	36.7	35.9
14	4	-2	34.3	-29.2
14	4	-3	14.5	7.5
14	4	-4		-4.0
14	4	-5	23.4	-22.0
14	4	-6	27.0	22.3
14	4	-7		12.1
-1	4	0	25.5	-28.2
-1	4	1		3.9
-1	4	2		.4
-1	4	3	55.4	52.6
-1	4	4	30.1	-28.8
-1	4	5	23.4	-24.2
-1	4	6		9.7
-1	4	7	22.5	20.4
-1	4	8		5.1
-1	4	9	22.5	-20.7
-1	4	10		-.8
-2	4	0	15.9	-14.8
-2	4	1	78.8	82.1
-2	4	2		2.1
-2	4	3		6.1
-2	4	4	37.0	-35.3
-2	4	5	11.2	8.5
-2	4	6	41.5	46.2
-2	4	7	29.7	-28.3
-2	4	8		-9.2
-2	4	9		2.0
-2	4	10		-13.1
-3	4	0		-1.2
-3	4	1	112.2	121.8
-3	4	2	73.9	-75.1
-3	4	3	86.4	-78.0
-3	4	4	92.3	99.5
-3	4	5		-3.4
-3	4	6	42.8	-42.0
-3	4	7		10.2
-3	4	8		-9.5
-3	4	9		2.6
-3	4	10		8.7
-4	4	0	37.0	34.3
-4	4	1	107.3	-113.9
-4	4	2	36.4	36.9
-4	4	3	20.5	21.2
-4	4	4		-2.1
-4	4	5		-.7
-4	4	6	37.3	-40.2
-4	4	7	18.3	15.4
-4	4	8		11.7
-4	4	9		-2.3
-4	4	10		4.8
-5	4	0	33.7	-34.7
-5	4	1	11.2	-11.0
-5	4	2	30.1	34.0
-5	4	3		3.1
-5	4	4	36.4	-39.2
-5	4	5		.6
-5	4	6	24.7	23.1
-5	4	7		-2.1
-5	4	8	18.9	20.1
-5	4	9		-8.8
-5	4	10		-9.9
-6	4	0	31.8	-30.0
-6	4	1	41.3	41.7
-6	4	2	19.4	18.4
-6	4	3	30.4	-25.9
-6	4	4	15.2	-12.6
-6	4	5	44.0	46.5
-6	4	6		6.0
-6	4	7	17.1	-17.0
-6	4	8		2.2
-6	4	9		3.0
-7	4	0	13.7	13.2
-7	4	1	20.5	19.6
-7	4	2	22.0	-19.3
-7	4	3	12.1	-10.0
-7	4	4	24.7	21.3
-7	4	5	13.7	10.7
-7	4	6	15.9	-13.1
-7	4	7		-11.4
-7	4	8		17.0
-7	4	9		4.9
-8	4	0	9.1	6.5
-8	4	1	47.5	-49.0
-8	4	2	15.9	10.1
-8	4	3	41.0	41.7
-8	4	4		-3.2
-8	4	5	37.8	-33.1
-8	4	6	28.3	24.0
-8	4	7		13.0
-8	4	8		-11.5
-9	4	0		-5.8
-9	4	1		-3.5
-9	4	2	31.8	32.5
-9	4	3		-.2
-9	4	4	34.3	-32.4
-9	4	5	22.0	18.3
-9	4	6		6.8
-9	4	7	17.1	-15.0
-9	4	8		-6.7
-10	4	0		7.7
-10	4	1		9.8
-10	4	2		-8.6
-10	4	3	18.3	-19.7
-10	4	4		6.7
-10	4	5		1.2
-10	4	6		-6.0
-10	4	7		5.4
-11	4	0	19.4	15.2
-11	4	1		-10.8
-11	4	2	20.5	-18.1
-11	4	3		7.1
-11	4	4		10.7
-11	4	5		3.8
-1	4	-1	50.3	-53.1
-1	4	-2	137.9	142.0
-1	4	-3	67.3	-63.9
-1	4	-4	87.1	-88.6
-1	4	-5	46.3	46.0
-1	4	-6	13.7	16.0
-1	4	-7	34.3	-30.5
-1	4	-8	17.1	15.0
-1	4	-9	55.6	-63.2
-1	4	-10	39.5	39.0
-1	4	-11	22.9	21.0
-2	4	-1	55.6	-55.4
-2	4	-2	42.0	37.4
-2	4	-3		-5.3
-2	4	-4	11.2	6.8
-2	4	-5	11.2	-13.2
-2	4	-6	34.0	-34.1
-2	4	-7	32.8	24.4
-2	4	-8		3.4
-2	4	-9		4.8
-2	4	-10		-19.5
-3	4	-1	20.5	-19.1
-3	4	-2	57.5	63.3
-3	4	-3	52.7	-57.0
-3	4	-4	23.4	20.8
-3	4	-5	34.0	37.9
-3	4	-6	20.5	-19.4
-3	4	-7	34.3	-28.5
-3	4	-8		4.1
-3	4	-9	30.4	29.5
-3	4	-10	22.9	-21.0
-4	4	-1	76.9	80.8
-4	4	-2	48.6	-43.8
-4	4	-3	29.7	29.2
-4	4	-4		-4.6
-4	4	-5	27.5	-24.0
-4	4	-6	53.1	57.1
-4	4	-7		-7.3
-4	4	-8	35.5	-37.9
-4	4	-9	22.5	25.2
-5	4	-1	27.5	-25.6
-5	4	-2	41.0	39.9
-5	4	-3	25.1	22.9
-5	4	-4	35.2	-32.5
-5	4	-5	37.5	41.6
-5	4	-6	29.0	-29.9
-5	4	-7		.6
-5	4	-8	48.1	55.1
-6	4	-1	17.7	-17.0
-6	4	-2		.7
-6	4	-3	21.5	-17.5
-6	4	-4	27.1	25.7
-6	4	-5		-.1
-6	4	-6	25.5	-27.7
-6	4	-7	25.5	25.7
-6	4	-8		2.2
-7	4	-1		5.3
-7	4	-2		-1.1
-7	4	-3	19.4	-19.1
-7	4	-4		2.2
-7	4	-5	18.9	17.5
-7	4	-6		-.0
-7	4	-7	19.4	-24.7
-8	4	-1	38.7	39.2
-8	4	-2	26.7	-22.0
-8	4	-3		-8.2
-8	4	-4	14.5	14.9
-8	4	-5	24.7	-27.2
-8	4	-6	23.4	25.0
-9	4	-1		7.2
-9	4	-2		-.0
-9	4	-3		12.3
-9	4	-4		-10.5
-9	4	-5		-2.7
-10	4	-1		-9.2
-10	4	-2		-3.6
-10	4	-3		6.4
-11	4	-1		-2.2
0	5	0	46.3	44.3
0	5	1	35.2	-36.6
0	5	2	9.1	-5.1
0	5	3	7.9	-5.7
0	5	4	19.4	-21.0
0	5	5	36.7	39.7
0	5	6	10.2	-2.6
0	5	7	43.3	-48.5
0	5	8	15.2	18.6
0	5	9		.6
0	5	10		2.0
1	5	0	7.9	4.4
1	5	1	20.5	15.9
1	5	2	24.7	21.8
1	5	3	34.6	30.8
1	5	4	111.4	-126.9
1	5	5	24.7	28.3
1	5	6	42.3	40.4
1	5	7	12.9	-10.6
1	5	8	31.1	-28.8
1	5	9		5.7
1	5	10		17.1
2	5	0	44.5	-41.6
2	5	1		-4.9
2	5	2	63.4	66.0
2	5	3	43.5	-43.0
2	5	4	67.0	71.8
2	5	5	12.9	-13.7
2	5	6	60.0	-64.7
2	5	7	18.9	19.0
2	5	8	41.0	41.5
2	5	9	47.9	-49.0
3	5	0	7.9	-5.8
3	5	1	26.3	-22.2
3	5	2	31.4	-28.6
3	5	3	20.6	22.8
3	5	4	36.1	38.3
3	5	5	10.2	-13.1
3	5	6	17.1	18.7
3	5	7	13.7	-13.3
3	5	8		-10.4
3	5	9	37.8	41.9
4	5	0	27.1	26.2
4	5	1	15.9	-13.1
4	5	2	17.1	16.8
4	5	3	22.9	-22.2
4	5	4	23.4	-22.3
4	5	5	44.5	46.0
4	5	6	12.1	6.4
4	5	7	25.5	-25.5
4	5	8		12.4
4	5	9		3.8
5	5	0		-7.4
5	5	1	33.1	31.5
5	5	2		-5.1
5	5	3		-.3
5	5	4	12.9	-13.5
5	5	5	12.1	-11.6
5	5	6	22.5	21.1
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13	7	-9		.0
14	7	-1	21.0	-21.7
14	7	-2		-4.8
14	7	-3	18.3	16.9
14	7	-4		.0
14	7	-5	17.1	-14.4
14	7	-6		-2.0
14	7	-7		7.1
14	7	-8		4.9
15	7	-3		4.6
15	7	-4	22.5	-17.3
15	7	-5		7.5
-1	7	0	18.9	-20.1
-1	7	1	37.5	35.9
-1	7	2		.7
-1	7	3	21.0	-20.4
-1	7	4	15.9	16.9
-1	7	5		8.3
-1	7	6	10.2	6.3
-1	7	7	16.5	-12.8
-1	7	8	21.0	-18.7
-1	7	9		7.4
-2	7	0	33.1	28.2
-2	7	1	39.7	-35.6
-2	7	2	10.2	6.1
-2	7	3		-3.9
-2	7	4	12.1	-9.8
-2	7	5	17.7	17.5
-2	7	6	22.0	-18.1
-2	7	7	27.5	-26.1
-2	7	8	17.7	18.9
-2	7	9		13.3
-3	7	0	38.7	-39.3
-3	7	1	35.8	-37.8
-3	7	2	25.9	28.1
-3	7	3	10.2	10.4
-3	7	4	17.7	-16.1
-3	7	5		-6.6
-3	7	6		-2.3
-3	7	7		4.9
-3	7	8		2.0
-4	7	0	9.1	-7.8
-4	7	1	13.7	12.8
-4	7	2	14.5	17.0
-4	7	3	14.5	-12.0
-4	7	4	22.5	-21.2
-4	7	5		6.7
-4	7	6	15.2	-15.1
-4	7	7	16.5	15.5
-4	7	8		2.9
-5	7	0	32.8	35.6
-5	7	1	10.2	-8.1
-5	7	2	21.5	-24.3
-5	7	3		1.7
-5	7	4	18.9	-18.6
-5	7	5		1.4
-5	7	6	29.4	32.1
-5	7	7		-4.6
-5	7	8	31.4	-32.0
-6	7	0		8.5
-6	7	1	27.1	-27.9
-6	7	2	11.2	-8.7
-6	7	3		7.4
-6	7	4	30.4	32.4
-6	7	5		-6.7
-6	7	6		-4.3
-6	7	7	18.9	-19.9
-7	7	0	26.7	-24.9
-7	7	1	19.4	15.1
-7	7	2		-2.6
-7	7	3	24.3	21.2
-7	7	4		-1.7
-7	7	5	28.3	-28.8
-7	7	6	15.2	17.1
-8	7	0		-9.0
-8	7	1	23.4	24.3
-8	7	2	12.1	12.2
-8	7	3	34.3	-34.5
-8	7	4	18.3	16.5
-8	7	5	13.7	13.4
-9	7	0	40.0	47.8
-9	7	1	18.3	-17.2
-9	7	2	22.0	-20.3
-9	7	3	34.3	32.7
-1	7	-1	32.8	31.4
-1	7	-2	37.0	-36.6
-1	7	-3	15.2	-14.5
-1	7	-4	63.4	64.9
-1	7	-5	54.5	-57.3
-1	7	-6	25.5	-29.5
-1	7	-7	43.5	47.6
-1	7	-8	29.7	-30.6
-1	7	-9	12.1	14.7
-1	7	-10		8.1
-2	7	-1	35.8	37.4
-2	7	-2	29.4	-26.6
-2	7	-3		-5.9
-2	7	-4	17.1	16.1
-2	7	-5	15.9	15.2
-2	7	-6		-1.6
-2	7	-7	48.1	-47.9
-2	7	-8	16.5	19.6
-2	7	-9	15.2	16.2
-3	7	-1	35.5	36.7
-3	7	-2	12.1	10.2
-3	7	-3	24.7	-25.0
-3	7	-4	10.2	-3.4
-3	7	-5	22.9	21.3
-3	7	-6		-3.5
-3	7	-7		7.1
-3	7	-8		3.0
-3	7	-9	26.6	-27.4
-4	7	-1	29.4	-29.3
-4	7	-2	17.1	13.7
-4	7	-3	22.5	21.1
-4	7	-4	17.7	-19.6
-4	7	-5	11.2	-7.9
-4	7	-6		2.1
-4	7	-7		3.1
-4	7	-8		5.1
-5	7	-1	15.2	-17.0
-5	7	-2	38.7	-40.9
-5	7	-3	12.9	13.8
-5	7	-4	41.5	42.0
-5	7	-5	23.8	-24.5
-5	7	-6	13.7	-14.0
-5	7	-7	24.3	24.6
-6	7	-1	30.8	30.8
-6	7	-2	17.1	-18.9
-6	7	-3	31.8	-31.2
-6	7	-4		6.1
-6	7	-5		4.7
-6	7	-6		7.1
-7	7	-1		1.4
-7	7	-2	23.4	23.6
-7	7	-3		5.6
-7	7	-4	18.9	-22.2
-7	7	-5		-3.1
-8	7	-1	13.7	-16.0
-8	7	-2		2.7
-8	7	-3	15.9	18.8
-9	7	-1		-17.6
0	8	0	15.2	-11.8
0	8	1	60.5	-55.7
0	8	2	27.1	23.5
0	8	3	16.5	15.5
0	8	4	10.2	8.3
0	8	5	22.9	-20.8
0	8	6	13.7	-14.6
0	8	7	28.3	26.8
0	8	8		-6.9
1	8	0	15.9	17.6
1	8	1	104.9	112.5
1	8	2	127.6	-128.7
1	8	3	17.1	-13.5
1	8	4	72.7	79.2
1	8	5	34.0	-34.1
1	8	6	18.3	-13.5
1	8	7		8.3
1	8	8		-8.1
2	8	0	57.1	67.8

TABLE II (*Continued*)

H	K	L	F-OBS	F-CAL
2	8	1	12.1	12.7
2	8	2	13.7	8.9
2	8	3	41.0	39.4
2	8	4	62.3	-67.0
2	8	5	28.6	26.7
2	8	6	43.6	45.0
2	8	7	55.8	-57.7
2	8	8		13.5
3	8	0	22.0	-22.0
3	8	1	25.5	-24.9
3	8	2	58.2	51.8
3	8	3	9.1	8.4
3	8	4		-3.2
3	8	5		-7.1
3	8	6		-.7
3	8	7	36.4	38.8
3	8	8	15.7	-14.2
4	8	0	30.4	33.8
4	8	1	54.9	-52.5
4	8	2	12.9	-9.5
4	8	3	34.3	31.5
4	8	4	15.9	14.9
4	8	5	25.5	-26.6
4	8	6		-2.1
4	8	7		13.6
4	8	8		-5.4
5	8	0	44.5	-43.3
5	8	1	13.7	8.2
5	8	2	12.9	11.8
5	8	3	42.0	-40.6
5	8	4	56.4	55.5
5	8	5	11.2	-12.0
5	8	6		-8.7
5	8	7		7.3
6	8	0		-1.1
6	8	1	13.7	-11.6
6	8	2		6.4
6	8	3		9.3
6	8	4	77.9	-85.4
6	8	5	50.1	-51.6
6	8	6	36.7	38.3
6	8	7	46.1	-51.0
7	8	0	24.3	-21.3
7	8	1	24.3	-21.4
7	8	2	28.6	26.8
7	8	3		.0
7	8	4	24.3	-20.4
7	8	5		1.8
7	8	6	24.3	-22.4
8	8	0	20.0	18.9
8	8	1	14.5	-10.4
8	8	2	23.4	-22.2
8	8	3	11.2	7.5
8	8	4	24.3	24.4
8	8	5	12.9	-16.3
8	8	6		-10.7
9	8	0	12.1	-10.6
9	8	1	65.2	65.0
9	8	2		-12.1
9	8	3	44.7	-44.7
9	8	4	21.5	19.0
9	8	5	23.8	26.3
10	8	0	22.9	-19.8
10	8	1	15.9	-18.3
10	8	2	29.7	32.5
10	8	3		4.4
10	8	4		-8.1
11	8	0		1.8
11	8	1	15.9	-13.4
11	8	2		-4.4
11	8	3	22.9	22.2
11	8	4		-3.9
12	8	0	12.9	13.1
12	8	1		6.6
12	8	2	28.6	-30.0
12	8	3		1.9
13	8	0	20.0	16.5
13	8	1		13.3
0	8	-1	38.4	40.4
0	8	-2	7.9	-2.9
0	8	-3	18.9	21.8
0	8	-4		-2.1
0	8	-5	61.1	-67.6
0	8	-6	81.9	92.2
0	8	-7	20.5	19.6
0	8	-8	56.8	-63.7
0	8	-9	25.9	27.5
0	8	-10	16.5	17.9
1	8	-1	38.9	-43.6
1	8	-2	75.1	81.4
1	8	-3	47.2	-45.0
1	8	-4	11.2	9.5
1	8	-5	68.1	67.2
1	8	-6	25.9	-24.5
1	8	-7	16.5	-15.5
1	8	-8		-1.7
1	8	-9		6.1
1	8	-10		6.7
2	8	-1	23.8	-22.2
2	8	-2	34.9	-35.5
2	8	-3	43.0	44.6
2	8	-4	11.2	10.4
2	8	-5	17.1	-17.9
2	8	-6	32.1	-32.3
2	8	-7		2.0
2	8	-8	26.3	27.1
2	8	-9		-11.4
2	8	-10		-3.7
2	8	-11		-3.7
3	8	-1	9.1	4.3
3	8	-2	12.1	-8.9
3	8	-3	31.1	30.3
3	8	-4	16.5	-16.5
3	8	-5	39.5	-36.9
3	8	-6	26.3	23.5
3	8	-7	32.8	32.2
3	8	-8		-2.4
3	8	-9	51.5	-50.5
3	8	-10	18.3	23.3
3	8	-11	16.5	18.0
4	8	-1	72.9	76.8
4	8	-2	107.2	-107.4
4	8	-3	22.0	23.3
4	8	-4	26.7	28.0
4	8	-5	26.3	-24.5
4	8	-6		-.7
4	8	-7	9.1	5.7
4	8	-8	54.9	-51.8
4	8	-9	44.0	45.4
4	8	-10		1.4
4	8	-11		-5.4
5	8	-1		-2.0
5	8	-2	45.6	43.9
5	8	-3	89.2	-97.0
5	8	-4	21.5	23.7
5	8	-5	27.5	24.5
5	8	-6	12.1	-10.1
5	8	-7	25.9	-23.6
5	8	-8	25.1	19.7
5	8	-9	35.5	30.6
5	8	-10		-10.9
5	8	-11	18.9	-18.5
6	8	-1	36.4	-34.7
6	8	-2	9.1	5.1
6	8	-3	27.5	26.6
6	8	-4	6.4	7.8
6	8	-5		.0
6	8	-6	27.5	-26.0
6	8	-7	26.3	25.7
6	8	-8	27.1	26.9
6	8	-9	30.1	-29.3
6	8	-10	13.7	-13.7
6	8	-11	15.9	18.8
7	8	-1	27.9	25.7
7	8	-2	15.2	11.7
7	8	-3		3.8
7	8	-4	7.9	-4.8
7	8	-5	24.7	-25.9
7	8	-6	19.4	17.4
7	8	-7	20.5	19.5
7	8	-8	18.3	-15.3
7	8	-9	23.4	-20.5
7	8	-10		2.9
7	8	-11		7.7
8	8	-1	19.4	18.1
8	8	-2		3.7
8	8	-3	25.1	-24.6
8	8	-4	20.5	-21.1
8	8	-5	30.8	27.5
8	8	-6	22.9	22.5
8	8	-7	33.4	-34.9
8	8	-8	12.9	-9.2
8	8	-9	23.4	-19.3
8	8	-10	35.8	41.1
8	8	-11		-10.6
9	8	-1	20.5	-20.9
9	8	-2	27.5	-27.3
9	8	-3	22.0	23.6
9	8	-4		-1.7
9	8	-5	23.4	24.9
9	8	-6	36.7	-35.9
9	8	-7	32.4	-33.1
9	8	-8	29.0	30.4
9	8	-9	31.4	31.3
9	8	-10	36.4	-46.3
9	8	-11		3.8
10	8	-1		3.5
10	8	-2	23.8	19.9
10	8	-3	18.3	-15.9
10	8	-4	27.5	19.3
10	8	-5	23.8	-23.9
10	8	-6	23.8	-25.0
10	8	-7	29.7	30.4
10	8	-8	11.2	7.7
10	8	-9	25.1	-24.2
10	8	-10		-6.5
10	8	-11	16.5	18.9
11	8	-1		-3.5
11	8	-2	20.0	15.0
11	8	-3		-6.4
11	8	-4	35.2	-31.9
11	8	-5		.8
11	8	-6	28.6	26.0
11	8	-7		1.5
11	8	-8	24.7	-25.8
11	8	-9	22.0	20.0
11	8	-10		13.1
12	8	-1	20.0	-17.7
12	8	-2	13.7	10.2
12	8	-3	12.1	-12.3
12	8	-4		2.3
12	8	-5	25.1	24.9
12	8	-6		-5.1
12	8	-7		6.0
12	8	-8		-2.3
12	8	-9		-3.6
12	8	-10	28.3	29.5
13	8	-1	20.0	-20.7
13	8	-2		-.5
13	8	-3	33.7	31.0
13	8	-4	21.5	-20.3
13	8	-5	12.9	10.5
13	8	-6	16.5	16.1
13	8	-7	46.3	-45.1
13	8	-8	34.9	38.9
13	8	-9		11.5
14	8	-1	31.4	31.9
14	8	-2	26.7	-20.9
14	8	-3		1.0
14	8	-4	34.3	34.9
14	8	-5	36.1	-41.4
14	8	-6		3.7
14	8	-7	38.9	38.6
14	8	-8		-23.5
15	8	-4		-7.3
-1	8	0	11.2	-9.7
-1	8	1	13.7	-11.6
-1	8	2	34.6	30.3
-1	8	3	9.1	-10.5
-1	8	4	17.1	-15.0
-1	8	5		-2.9
-1	8	6	12.1	3.9
-1	8	7	21.5	20.9
-1	8	8	13.7	-11.8
-2	8	0	23.8	21.8
-2	8	1	15.9	-12.0
-2	8	2	20.0	17.1
-2	8	3	16.5	-14.8
-2	8	4	33.4	-31.5
-2	8	5	52.3	54.4
-2	8	6	12.1	-8.1
-2	8	7	29.4	-32.4
-2	8	8	33.1	28.3
-3	8	0	15.9	-10.2
-3	8	1	56.2	57.3
-3	8	2	56.8	-62.8
-3	8	3		-3.5
-3	8	4	58.2	56.2
-3	8	5	22.9	-19.8
-3	8	6	17.7	-15.9
-3	8	7		8.0
-3	8	8	13.7	9.5
-4	8	0	32.4	-32.9
-4	8	1	15.2	-14.1
-4	8	2	34.3	37.5
-4	8	3		-.2
-4	8	4		1.8
-4	8	5	22.9	-19.4
-4	8	6		3.2
-4	8	7	15.9	18.0
-5	8	0		-1.8
-5	8	1	11.2	-4.2
-5	8	2	25.5	24.5
-5	8	3	16.5	-14.5
-5	8	4	31.8	-30.8
-5	8	5	21.5	18.9
-5	8	6		5.1
-5	8	7	15.2	-12.8
-6	8	0		.0
-6	8	1		-6.3
-6	8	2		6.9
-6	8	3	24.3	-20.0
-6	8	4		-4.7
-6	8	5	26.7	27.3
-6	8	6		-4.3
-7	8	0		6.2
-7	8	1		-4.5
-7	8	2	21.0	-18.5
-7	8	3	19.4	15.9
-7	8	4	18.9	9.1
-7	8	5		-11.3
-8	8	0		-9.0
-8	8	1	15.9	-14.9
-8	8	2		1.0
-8	8	3	24.3	22.5
-1	8	-1	27.5	-26.6
-1	8	-2	7.9	11.8
-1	8	-3	39.7	38.9
-1	8	-4	36.4	-33.9
-1	8	-5	15.2	14.9
-1	8	-6		1.9
-1	8	-7	23.8	-21.5
-1	8	-8	32.4	35.9
-1	8	-9		3.7
-2	8	-1	23.8	-23.3
-2	8	-2	24.7	-24.7
-2	8	-3	13.7	10.1
-2	8	-4	9.1	7.4
-2	8	-5	16.5	17.7
-2	8	-6	32.4	-33.7
-2	8	-7		.9
-2	8	-8	26.3	26.7
-2	8	-9	13.7	-10.8
-3	8	-1	31.4	-32.0
-3	8	-2	42.5	43.1
-3	8	-3	38.9	-39.5
-3	8	-4	28.3	-31.9
-3	8	-5	55.6	60.8
-3	8	-6	18.3	-16.1
-3	8	-7	21.5	-27.5
-3	8	-8		9.8
-4	8	-1	44.2	49.7
-4	8	-2	17.7	-14.0
-4	8	-3	18.9	-22.8
-4	8	-4	25.6	28.0
-4	8	-5	51.5	-52.2
-4	8	-6	22.0	22.2
-4	8	-7	29.0	31.2
-5	8	-1	12.9	.9
-5	8	-2		-3.6
-5	8	-3	30.8	30.6
-5	8	-4	15.9	-14.3
-5	8	-5		-7.8
-5	8	-6		3.0
-6	8	-1		7.2
-6	8	-2		-9.7
-6	8	-3	15.2	-15.5
-6	8	-4	18.3	21.3
-6	8	-5	16.5	15.1
-7	8	-1		-3.0
-7	8	-2	17.1	16.8
-7	8	-3	13.7	-12.6
-7	8	-4	14.5	-16.3
-8	8	-1		11.6
0	9	0	34.9	32.5
0	9	1	31.1	-28.2
0	9	2	11.2	-10.4
0	9	3	12.9	11.6
0	9	4	22.0	19.4
0	9	5	17.1	15.5
0	9	6	23.4	-23.4
0	9	7		7.9
1	9	0	39.7	40.3
1	9	1	33.1	-37.7
1	9	2	25.1	24.3
1	9	3		-.5
1	9	4		-8.1
1	9	5		-5.8
1	9	6	28.3	26.1
1	9	7		-5.2
2	9	0	76.1	-77.8
2	9	1	82.9	81.9
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1	13	3		10.7
2	13	0	14.5	-14.2
2	13	1	34.3	32.0
2	13	2		3.2
2	13	3	18.9	-18.1
3	13	0		-8.0
3	13	1	23.8	-26.4
3	13	2	22.0	26.2
3	13	3	17.1	17.6
4	13	0	12.1	12.8
4	13	1		-16.0
4	13	2		-4.4
4	13	3		7.4
5	13	0	16.5	15.1
5	13	1		8.1
5	13	2	15.2	-10.3
5	13	3		-6.3
6	13	0	20.0	-19.7
6	13	1	33.9	29.8
6	13	2		-4.2
6	13	3	14.5	-15.1
7	13	0	34.3	-33.0
7	13	1	17.1	-17.0
7	13	2	26.7	29.8
7	13	3		4.6
8	13	0	26.7	24.5
8	13	1	17.1	-10.9
8	13	2	18.9	-20.5
9	13	0		10.4
9	13	1	17.7	18.8
9	13	2		-3.0
10	13	0		-6.5
10	13	1		-4.9
11	13	0	37.0	-32.2
0	13	-1	17.1	14.0
0	13	-2		-5.2
0	13	-3		-1.4
0	13	-4		2.7
0	13	-5	21.0	-16.6
1	13	-1	22.0	24.1
1	13	-2	50.5	-49.2
1	13	-3		4.0
1	13	-4		10.0
1	13	-5		8.7
1	13	-6		-4.6
2	13	-1	47.0	-44.6
2	13	-2	34.9	30.2
2	13	-3		5.8
2	13	-4		-8.5
2	13	-5		7.7
2	13	-6		-7.0
2	13	-7		-.8
3	13	-1		7.5
3	13	-2	29.0	21.1
3	13	-3		1.1
3	13	-4		-6.1
3	13	-5		-5.2
3	13	-6		-6.4
3	13	-7	21.0	20.8
3	13	-8	*	-3.7
4	13	-1	22.9	25.9
4	13	-2	20.5	-18.7
4	13	-3	18.3	-18.8
4	13	-4		6.0
4	13	-5		5.2
4	13	-6		-2.9
4	13	-7		5.2
4	13	-8	*	-25.2
5	13	-1	27.5	-24.9
5	13	-2		2.9
5	13	-3		2.8
5	13	-4	20.0	18.7
5	13	-5	18.9	-13.7
5	13	-6		1.0
5	13	-7		-5.8
5	13	-8	*	24.9
6	13	-1	13.7	11.2
6	13	-2	20.5	-18.7
6	13	-3	31.1	24.3
6	13	-4		-4.0
6	13	-5		-14.0
6	13	-6	19.4	13.9
6	13	-7		-7.5
6	13	-8	*	3.5
6	13	-9	*	19.5
7	13	-1	27.9	30.9
7	13	-2	24.7	20.7
7	13	-3	44.0	-42.7
7	13	-4		16.8
7	13	-5	15.9	13.6
7	13	-6	19.4	-22.9
7	13	-7	30.1	29.1
7	13	-8	*	-1.4
7	13	-9	*	-26.6
8	13	-1		-4.1
8	13	-2		-1.4
8	13	-3		-3.4
8	13	-4		-7.6
8	13	-5	18.9	22.3
8	13	-6		6.2
8	13	-7		-11.2
8	13	-8	*	-17.8
8	13	-9	*	14.0
9	13	-1	20.0	-22.1
9	13	-2		-2.3
9	13	-3	22.5	18.1
9	13	-4		.6
9	13	-5	16.5	18.0
9	13	-6	28.3	-27.1
9	13	-7		-9.4
9	13	-8	*	27.8
9	13	-9	*	-9.5
10	13	-1	15.9	-15.4
10	13	-2	21.5	17.2
10	13	-3	32.8	30.2
10	13	-4	35.8	-34.0
10	13	-5	23.4	-27.3
10	13	-6	41.5	43.2
10	13	-7	26.7	-28.5
10	13	-8	*	9.7
11	13	-1	39.2	46.1
11	13	-2		-4.2
11	13	-3	43.5	-46.4
11	13	-4	28.3	33.6
11	13	-5		-8.4
11	13	-6		-6.0
11	13	-7	29.4	30.3
11	13	-8	*	-30.0
12	13	-2		.4
12	13	-3		11.9
12	13	-4		-16.5
12	13	-5		17.1
12	13	-6		-4.3
12	13	-7	20.0	-14.3
-1	13	0		-.0
-1	13	1	41.5	-33.7
-1	13	2	33.1	36.9
-2	13	0		8.3
-1	13	-1	21.0	6.5
-1	13	-2		-10.7
-1	13	-3	14.5	14.9
-1	13	-4		-12.5
-2	13	-1	43.3	-50.7
-2	13	-2	34.6	33.7
0	14	0		13.0
1	14	0	12.9	11.4
1	14	1		1.0
2	14	0		-5.7
2	14	1		5.0
3	14	0		-2.8
3	14	1	17.7	-17.7
3	14	2	16.5	20.1
4	14	0	21.0	-22.3
4	14	1		7.5
4	14	2		11.7
5	14	0	12.1	10.6
5	14	1	17.7	15.8
5	14	2		-12.5
6	14	0		-10.5
6	14	1		-5.3
6	14	2		9.1
7	14	0	25.1	21.2
7	14	1		-5.8
8	14	0	36.4	-32.0
8	14	1		12.0
9	14	0	18.3	15.1
0	14	-1		13.3
0	14	-2		-9.3
0	14	-3	21.0	-20.6
1	14	-1	15.2	-13.3
1	14	-2		-8.9
1	14	-3		7.7
1	14	-4	22.5	18.7
2	14	-1		.8
2	14	-2		-2.4
2	14	-3	14.5	11.5
2	14	-4		-12.3
2	14	-5	17.7	12.4
2	14	-6	15.9	-10.5
3	14	-1	17.1	16.7
3	14	-2		.4
3	14	-3		4.3
3	14	-4	18.3	-17.8
3	14	-5		1.1
3	14	-6	36.1	32.7
4	14	-1	31.8	32.0
4	14	-2		-5.7
4	14	-3	46.6	-46.8
4	14	-4	39.5	44.1
4	14	-5		11.9
4	14	-6	30.1	-38.6
4	14	-7		14.5
5	14	-1	37.5	-34.2
5	14	-2		9.0
5	14	-3	15.9	18.4
5	14	-4		1.3
5	14	-5		-8.4
5	14	-6		-12.6
5	14	-7	16.5	15.9
6	14	-1		-9.0
6	14	-2	36.1	36.3
6	14	-3		-2.7
6	14	-4	25.1	-26.4
6	14	-5	17.1	9.0
6	14	-6		3.9
6	14	-7		10.0
7	14	-1		4.3
7	14	-2		-12.6
7	14	-3	13.7	13.0
7	14	-4	35.8	-36.3
7	14	-5	20.5	22.1
7	14	-6	45.4	45.6
7	14	-7	64.3	-62.5
7	14	-8	*	8.9
8	14	-1	34.9	36.5
8	14	-2	25.9	-26.2
8	14	-3	21.5	-23.1
8	14	-4	60.9	59.2
8	14	-5		-20.5
8	14	-6		-21.6
8	14	-7	32.4	27.4
8	14	-8	*	-13.0
9	14	-1	45.9	-41.9
9	14	-2	22.0	20.7
9	14	-3	23.8	25.1
9	14	-4	18.9	-16.4
9	14	-5		-8.2
9	14	-6		-.9
9	14	-7		6.5
10	14	-1		-5.5
10	14	-2		8.4
10	14	-3		2.8
10	14	-4	18.9	-24.3
10	14	-5		2.2
10	14	-6		9.5
10	14	-7		3.2
11	14	-2		-7.1
11	14	-3	14.5	12.1
11	14	-4	22.5	-24.1
11	14	-5	17.7	23.3
11	14	-6		11.7
4	15	0		13.1
5	15	0	44.5	-43.5
6	15	0		13.3
3	15	-1		-4.9
3	15	-2		-2.2
3	15	-3	14.5	-15.9
3	15	-4		4.7
4	15	-1	33.4	-35.9
4	15	-2		-1.1
4	15	-3	41.3	44.3
4	15	-4	37.1	-34.6
4	15	-5		9.0
5	15	-1	36.4	41.5
5	15	-2		-5.1
5	15	-3	12.1	-13.6
5	15	-4	37.8	34.6
5	15	-5	25.5	-32.0
6	15	-1		-6.0
6	15	-2	22.9	16.9
6	15	-3		-5.0
6	15	-4		-9.5
6	15	-5		7.1
6	15	-6		8.5
7	15	-1		-7.0
7	15	-2	22.9	-15.5
7	15	-3		5.6
7	15	-4		.5
7	15	-5		4.9
7	15	-6		-12.5
8	15	-1	30.8	-32.7
8	15	-2		7.8
8	15	-3	26.7	27.3
8	15	-4	27.1	-28.5
8	15	-5		2.9
8	15	-6		10.2
9	15	-2	22.9	-21.5
9	15	-3		-5.5
9	15	-4		16.0
9	15	-5	21.5	-25.1

* Indicates an observation that was not recorded or was rejected because of severe extinction effects.

TABLE III

INTERATOMIC DISTANCES AND ANGLES IN $K_2Zn_2V_{10}O_{28} \cdot 16H_2O$[a-e]

A. Data for $V_{10}O_{28}^{6-}$ group

Atoms	Distances	Atoms	Distances	Atoms	Angles	Atoms	Angles
V_1-V_2	3.052(2)	O_1'-O_9	2.479(9)	O_6-V_1-O_1'	98.6(3)	O_3'-V_3-O_8	78.1(3)
V_1-V_4	3.148(2)	O_3'-O_9	2.613(9)	O_6-V_1-O_9	99.6(4)	O_4-V_5-O_5	92.8(4)
V_1-V_4'	3.187(2)	O_1-O_3	2.611(9)	O_7-V_2-O_1'	98.9(3)	O_4-V_5-O_{10}	90.9(4)
V_2-V_4	3.133(2)	O_3-O_9	2.622(9)	O_7-V_2-O_9	98.5(4)	O_4'-V_3-O_2	90.8(4)
V_2-V_4'	3.187(2)	O_1'-O_3	2.610(9)	O_6-V_1-O_5'	102.1(3)	O_4'-V_3-O_8	90.4(4)
V_1-V_3	3.122(2)	O_3'-O_8	2.686(9)	O_6-V_1-O_8	104.1(5)	O_{13}-V_5-O_5	84.4(3)
V_1-V_5'	3.084(2)	O_3-O_5	2.667(9)	O_7-V_2-O_2'	102.6(4)	O_{13}-V_5-O_{10}	82.2(3)
V_2-V_5	3.122(2)	O_3-O_{10}	2.628(9)	O_7-V_2-O_{10}	103.5(5)	O_{12}-V_3-O_2	83.7(3)
V_2-V_3'	3.137(2)	O_2'-O_3	2.660(9)	O_3'-V_1-O_1'	75.6(2)	O_{12}-V_3-O_8	84.8(3)
V_3-V_5'	3.078(3)	O_3-O_4	2.767(9)	O_3'-V_1-O_9	76.4(3)	O_3-V_5-O_{14}	174.4(5)
V_3-V_4	3.089(2)	O_3'-O_{12}	2.666(9)	O_3-V_2-O_1'	76.0(2)	O_3'-V_3-O_{11}	176.4(5)
V_4'-V_5'	3.068(2)	O_3-O_{13}	2.667(9)	O_3-V_2-O_9	76.7(3)	O_4-V_5-O_{13}	157.0(4)
V_4-V_4'	3.311(3)	O_3-O_3'	2.638(12)	O_3'-V_1-O_5'	81.0(3)	O_4'-V_3-O_{12}	156.1(5)
		O_8-O_9	2.721(9)	O_3'-V_1-O_8	81.4(3)	O_5-V_5-O_{10}	154.1(5)
V_1-V_6	1.612(8)	O_9-O_{10}	2.701(9)	O_3-V_2-O_2'	81.4(3)	O_2-V_3-O_8	154.2(5)
V_2-V_7	1.603(8)	O_1-O_2	2.707(9)	O_3-V_2-O_{10}	80.9(3)		
V_1-O_3'	2.246(6)	O_1-O_5	2.698(9)	O_1'-V_1-O_9	77.4(3)	O_{12}-V_4-O_{13}	106.8(4)
V_2-O_3	2.218(6)	O_1-O_4'	2.762(10)	O_1'-V_2-O_9	76.3(3)	O_3-V_4-O_3'	77.1(3)
V_1-O_1	2.000(6)	O_1-O_6'	2.747(10)	O_5'-V_1-O_8'	93.4(4)	O_3'-V_4-O_{12}	87.4(3)
V_1-O_9	1.965(7)	O_7-O_9	2.741(10)	O_2'-V_2-O_{10}	95.7(4)	O_3-V_4-O_{13}	88.7(3)
V_2-O_1'	2.014(6)	O_6-O_9	2.743(10)	O_8-V_1-O_9	91.1(4)	O_3-V_4-O_1	80.0(3)
V_2-O_9	2.000(6)	O_1-O_{12}	2.740(9)	O_9-V_2-O_{10}	90.3(3)	O_3-V_4-O_9	81.0(3)
V_1-O_5'	1.831(7)	O_1-O_{13}	2.698(9)	O_1'-V_1-O_8'	156.1(4)	O_3'-V_4-O_1	79.7(3)
V_1-O_8	1.846(7)	O_9-O_{12}	2.724(9)	O_9-V_1-O_5'	156.0(4)	O_3'-V_4-O_9	80.4(3)
V_2-O_2'	1.839(7)	O_9-O_{13}	2.752(9)	O_1'-V_2-O_{10}	155.4(4)	O_{13}-V_4-O_1	96.0(4)
V_2-O_{10}	1.803(7)	O_4-O_2	2.659(10)	O_9-V_2-O_2'	156.0(4)	O_{13}-V_4-O_9	99.7(4)
		O_4-O_{10}	2.674(10)	O_3'-V_1-O_6	173.3(5)	O_{12}-V_4-O_1	96.6(4)
V_5-O_{14}	1.602(8)	O_4-O_5	2.672(10)	O_3-V_2-O_7	173.7(5)	O_{12}-V_4-O_9	97.1(4)
V_3-O_{11}	1.608(9)	O_4-O_8'	2.628(10)			O_1-V_4-O_9	155.1(4)
V_5-O_3	2.317(6)	O_2-O_{11}	2.733(11)	O_{11}-V_3-O_{12}	102.0(5)	O_{13}-V_4-O_3'	165.7(4)
V_3-O_3'	2.355(7)	O_5-O_{14}	2.676(11)	O_{13}-V_5-O_{14}	99.9(4)	O_{12}-V_4-O_3	164.4(4)
V_3-O_4'	1.837(7)	O_8-O_{11}	2.718(11)	O_3'-V_3-O_{12}	74.5(3)		
V_5-O_4	1.845(7)	O_{10}-O_{14}	2.743(10)	O_3-V_5-O_{13}	74.5(3)		
V_3-O_2	1.895(7)	O_2-O_{12}	2.619(10)	O_{14}-V_5-O_4	103.1(4)		
V_3-O_8	1.867(7)	O_5-O_{13}	2.640(10)	O_{11}-V_3-O_4'	101.9(4)		
V_5-O_5	1.845(7)	O_8-O_{12}	2.628(10)	O_{14}-V_5-O_5	101.6(4)		
V_5-O_{10}	1.907(7)	O_{10}-O_{13}	2.622(10)	O_{14}-V_5-O_{10}	102.5(4)		
V_3-O_{12}	2.027(7)	O_2-O_4'	2.691(11)	O_{11}-V_3-O_2	102.2(4)		
V_5-O_{13}	2.077(7)	O_5-O_6'	2.681(10)	O_{11}-V_3-O_8	102.6(4)		
		O_7-O_{10}	2.678(11)	O_3-V_5-O_4	82.5(3)		
V_4-O_{12}	1.713(7)	O_9-O_8	2.732(10)	O_3-V_3-O_4'	81.6(3)		
V_4-O_{13}	1.678(7)	O_{12}-O_{13}	2.723(9)	O_3-V_5-O_5	78.8(3)		
V_4-O_3	2.110(7)	O_2-O_{10}'	2.700(10)	O_3-V_5-O_{10}	76.3(3)		
V_4-O_3'	2.123(6)	O_5-O_8	2.676(10)	O_3'-V_3-O_2	76.7(3)		
V_4-O_1	1.945(6)	O_4-O_{11}'	2.680(11)				
V_4-O_9	1.917(6)	O_4-O_{14}	2.703(11)				
		O_{11}-O_{12}	2.837(11)				
		O_{13}-O_{14}	2.831(10)				
		O_6-O_7	2.991(11)				

B. Data for $Zn(H_2O)_6^{2+}$ group

Atoms	Distances
Zn-O_{15}	2.077(9)
Zn-O_{16}	2.127(9)
Zn-O_{17}	2.136(8)
Zn-O_{18}	2.083(11)
Zn-O_{19}	2.076(9)
Zn-O_{20}	2.093(10)
O_{15}-O_{16}	2.861(12)
O_{15}-O_{17}	2.833(12)
O_{15}-O_{18}	2.970(14)
O_{15}-O_{19}	3.146(13)
O_{20}-O_{16}	2.922(12)
O_{20}-O_{17}	3.093(12)
O_{20}-O_{18}	2.961(14)
O_{20}-O_{19}	2.896(12)
O_{16}-O_{17}	2.911(11)
O_{16}-O_{18}	3.056(13)
O_{19}-O_{17}	2.982(11)
O_{19}-O_{18}	2.965(14)

Atoms	Angles
O_{15}-Zn-O_{16}	85.7(4)
O_{15}-Zn-O_{17}	84.5(4)
O_{15}-Zn-O_{18}	91.1(5)
O_{15}-Zn-O_{19}	98.5(4)
O_{20}-Zn-O_{16}	87.6(3)
O_{20}-Zn-O_{17}	94.0(3)
O_{20}-Zn-O_{18}	90.3(4)
O_{20}-Zn-O_{19}	88.0(4)
O_{16}-Zn-O_{17}	86.1(4)
O_{17}-Zn-O_{19}	90.1(4)
O_{18}-Zn-O_{19}	91.0(5)
O_{16}-Zn-O_{18}	93.1(5)
O_{15}-Zn-O_{20}	173.8(6)
O_{16}-Zn-O_{19}	174.1(5)
O_{17}-Zn-O_{18}	175.6(6)

C. Data for inter-polyion O-O distances < 3.2 Å (See note *f*)

Atoms	Distances
O_4'-O_{17}(2)	2.731(10)
O_2-O_{15}'(2)	2.765(11)
O_{10}'-O_{19}'(2)	2.773(11)
O_2-O_{15}	2.705(11)
O_1-O_{16}	2.672(10)
O_{12}-O_{18}	2.788(13)
O_{10}'(2)-O_{18}	2.771(13)
O_{16}-O_6(3)	2.930(11)
O_{17}-O_9(3)	2.928(10)
O_{20}-O_4'(1)	3.067(13)
O_{20}-O_6'(1)	3.223(12)
O_{19}-O_{22}	2.904(14)
O_{22}-O_{14}(2)	2.914(14)
O_{20}-O_{21}	2.868(14)
O_{21}-O_8'(2)	2.965(13)

D. Data for K coordination

Atoms	Distances
K-O_5	2.792(7)
K-O_6	3.057(8)
K-O_7	2.784(8)
K-O_{11}	3.029(9)
K-O_{14}	2.965(9)
K-O_{15}	3.251(10)
K-O_{16}	2.918(9)
K-O_{17}	2.998(8)
K-O_{21}	3.080(11)
K-O_{22}	2.781(11)
K-O_1	3.977(7)
K-Zn	3.876(3)

[a] Distances are in angstrom units, angles in degrees. [b] Numeral subscripts to atoms correspond to those in Table I and Figures 1a and 3a. [c] Unprimed atoms have x, y, z as given in Table I; primed atoms have $1 - x$, $1 - y$, $1 - z$. [d] Numbers in parentheses following data indicate standard errors in terms of last significant figures, derived from variance–covariance matrix diagonals, including lattice parameters. [e] Distances and angles that are related by the pseudo-orthorhombic symmetry of the $V_{10}O_{28}^{6-}$ ion are grouped together. [f] In group C, numbers in parentheses following atom symbols indicate atoms in adjacent unit cells, as follows: no number, 0, 0, 0; (1), 0, 0, $\bar{1}$; (2), 0, 1, 0; (3), 1, 0, 0.

map shown in Figure 1b. The structure consists of an assemblage in one unit cell of one discrete $V_{10}O_{28}^{6-}$ ion, two K^+ ions, two $Zn(H_2O)_6^{2+}$ ions, and four additional H_2O molecules. These are held together in the crystal by ionic forces and hydrogen bonds.

Only isotropic thermal vibrations of the atoms were assumed in the refined model. A difference Fourier synthesis based on the final ΔF values revealed slight anisotropy of motion for some of the atoms, but the only pronounced effect observed was associated with the K^+ ion. There is, in fact, some ellipticity approximately normal to the *a–c* plane evident for this ion in the electron density map (Figure 1b). The zinc atom and the five vanadium atoms also all show some slight elongation of vibration generally normal to the *a–b* plane, but this effect could result from small, systematic errors in the original data. Otherwise, no interpretable features could be found on the ΔF map.

The $Zn(H_2O)_6^{2+}$ and $V_{10}O_{28}^{6-}$ groups are linked together strongly by hydrogen bonds[22] into a sheet parallel to the *a–b* plane, as shown in Figure 2. The hexahydrated zinc cation $Zn(H_2O)_6^{2+}$ is a nearly regular octahedron lying in neat apposition to three adjacent $V_{10}O_{28}^{6-}$ groups, sharing two or three hydrogen bonds with each. The K^+ ion is also incorporated into this sheet fabric, so that there are relatively few bond forces between the sheets. This arrangement is consistent with the good cleavage observed parallel to (001).

Three water molecules of one face of the $Zn(H_2O)_6^{2+}$ octahedron (O_{15}, O_{16}, and O_{17}) are coordinated to the adjacent potassium ion. The six Zn–O distances (see Table III) average 2.101 A. The K^+ ion is irregularly coordinated to ten oxygen atoms, five from three neighboring $V_{10}O_{28}^{6-}$ groups, three water molecules in the adjacent $Zn(H_2O)_6^{2+}$ group, and the two additional water molecules. The average distance is 2.96 A (see Table III).

The Structure of the Decavanadate Ion

Ten vanadium atoms and 28 oxygen atoms are arranged in a highly condensed system of VO_6 octahedra, as shown in Figure 3. It may be described as a group of six VO_6 octahedra arranged in a 2 × 3 rectangular array by sharing edges, with two VO_6 octahedra joined from above and two more from below by sharing sloping edges with the central six octahedra. The structure conforms closely to an ideal arrangement with orthorhombic symmetry mmm. In

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[22] The hydrogen bonds are recognized by the shortness of O–O distances, that is, <2.95 A, and crystal chemical considerations; no direct evidence of hydrogen atoms was found in the structure analysis.

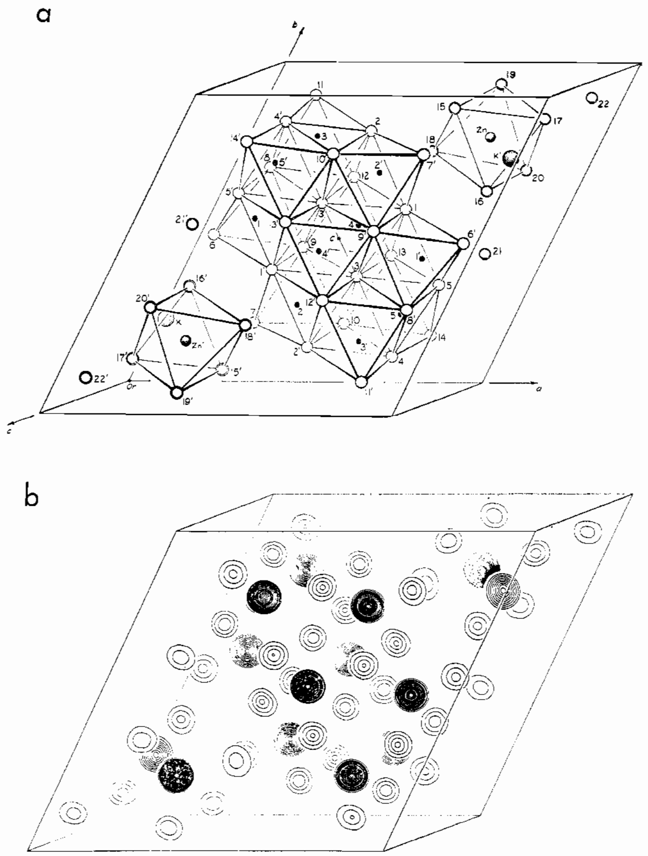

Figure 1.—Contents of the triclinic unit cell of $K_2Zn_2V_{10}O_{28}\cdot 16H_2O$, projected normal to the a–b plane. (a) Numbers correspond to the serial numbers given in Table I; primes indicate atoms at $1 - x$, $1 - y$, $1 - z$ in terms of parameters of Table I. (b) Composite of electron density sections in same view as part a. Contours at 5, 10, 15, etc. e/A^3.

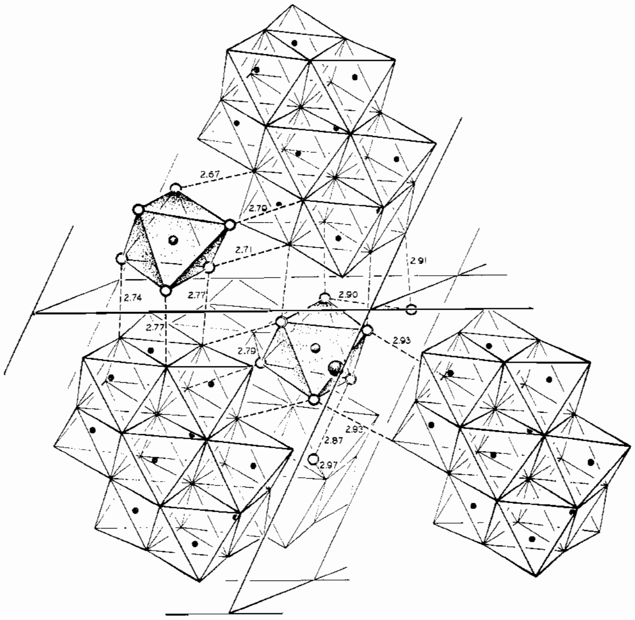

Figure 2.—Contents of several unit cells viewed as in Figure 1a, showing hydrogen bonding (lengths in A, ±0.01 A) among $V_{10}O_{28}^{6-}$ groups, $Zn(H_2O)_6^{2+}$ groups, and H_2O molecules. Atoms may be identified by reference to Figure 1a.

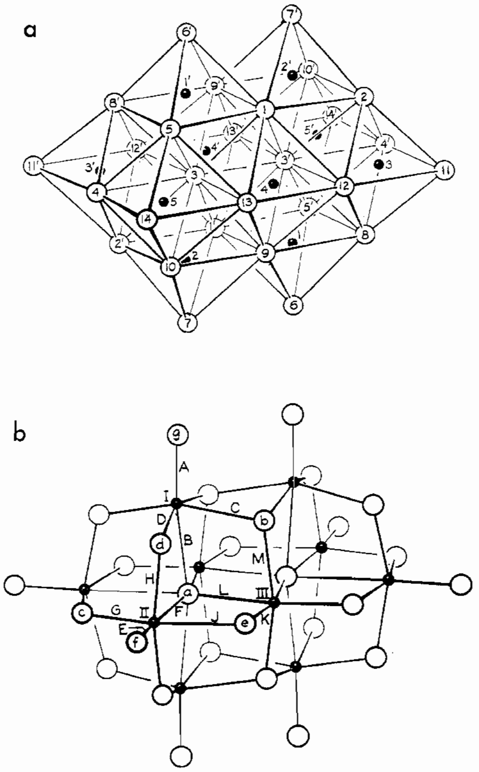

Figure 3.—Model structures of the $V_{10}O_{28}^{6-}$ group. (a) Polyhedral model with atoms numbered as in Figure 1a and Table I. (b) Bond model with symbols assigned according to idealized mmm symmetry, as used in Table IV.

this ideal structure there are three kinds of VO_6 octahedra: two octahedra (V_{III}) are in the center of the 2 × 6 rectangular group and share seven edges each with neighboring octahedra; four octahedra (V_{II}) are at the corners of the rectangular group and share four edges each with neighbors; and four octahedra (V_I) are at the top and bottom of the group sharing five edges each with neighbors. Two oxygen (O_a) atoms lie inside the $V_{10}O_{28}^{6-}$ group and are coordinated to six vanadium atoms each; four oxygen atoms (O_b) lie on the surface of the group and coordinate three vanadium atoms each; 14 oxygen atoms (O_c, O_d, O_e) lying on the surface coordinate two vanadium atoms each; and eight oxygen atoms (O_f, O_g) lying on the outer corners coordinate only one vanadium atom each.

All of the crystallographically independent interatomic distances have been averaged over the ideal, orthorhombic molecular symmetry and are listed in Table IV. Variations in the interatomic distances and angles equivalent to the averaged values in Table IV are minor, about ±0.02 A for distances and ±2° for angles. One of the more significant variations is in V_{III}–O_e (vector K), which has values 1.678 (V_4–O_{13}) and 1.713 A (V_4–O_{12}) (both ±0.007 Å) on opposite sides of the pseudo-mirror plane. This is a reasonable variation because O_{12} (O_e') is associated with a strong hydrogen bond to O_{18} (2.78 A) in the $Zn(H_2O)_6^{2+}$ group, while O_{13} is not associated with any hydrogen bond (Figure 2). The variations generally do not seem to be greater than what would be expected from the influence of neighboring atoms and groups, especially through hydrogen bonding. In other words, there appears to be no reason to believe that the free polyion does not have true orthorhombic symmetry.

TABLE IV

AVERAGE INTERATOMIC DISTANCES IN THE ORTHORHOMBIC DECAVANADATE ION[a]

Vanadium–oxygen bonds	Vectors	Lengths, A	Range (number averaged), A
$V_{I}-O_{g}$	A	1.61	1.607–1.608 (2)
$V_{I}-O_{a}$	B	2.23	2.218–2.246 (2)
$V_{I}-O_{b}$	C	1.99	1.965–2.014 (4)
$V_{I}-O_{d}$	D	1.83	1.803–1.846 (4)
$V_{II}-O_{f}$	E	1.61	1.602–1.608 (2)
$V_{III}-O_{a}$	F	2.34	2.317–2.355 (2)
$V_{II}-O_{c}$	G	1.84	1.837–1.845 (2)
$V_{II}-O_{d}$	H	1.88	1.845–1.907 (4)
$V_{II}-O_{e}$	J	2.05	2.027–2.077 (2)
$V_{III}-O_{e}$	K	1.70	1.678–1.713 (2)
$V_{III}-O_{a}$	L	2.12	2.110–2.123 (2)
$V_{III}-O_{b}$	M	1.93	1.917–1.945 (2)

Interbond angles	Vectors	Angles, deg	Range (number averaged), deg
$O_{g}-V_{I}-O_{b}$	$A \wedge C$	99	98.6–99.6 (4)
$O_{g}-V_{I}-O_{d}$	$B \wedge D$	103	102.1–104.1 (4)
$O_{a}-V_{I}-O_{b}$	$A \wedge C$	76	75.6–76.4 (4)
$O_{a}-V_{I}-O_{d}$	$B \wedge D$	81	81.0–81.4 (4)
$O_{b}-V_{I}-O_{b}'$	$C \wedge C$	77	76.3–77.4 (2)
$O_{d}-V_{I}-O_{d}'$	$D \wedge D$	95	93.4–95.7 (2)
$O_{b}-V_{I}-O_{d}$	$C \wedge D$	91	90.3–91.1 (2)
$O_{b}-V_{I}-O_{d}'$	$C \wedge D$	156	155.4–156.1 (4)
$O_{a}-V_{I}-O_{g}$	$A \wedge B$	174	173.3–173.7 (2)
$O_{f}-V_{II}-O_{e}$	$E \wedge J$	101	99.9–102.0 (2)
$O_{a}-V_{II}-O_{e}$	$F \wedge J$	75	74.5 (2)
$O_{f}-V_{II}-O_{c}$	$E \wedge G$	103	101.9–103.1 (2)
$O_{f}-V_{II}-O_{d}$	$E \wedge H$	102	101.6–102.5 (4)
$O_{a}-V_{II}-O_{c}$	$F \wedge G$	82	81.6–82.5 (2)
$O_{a}-V_{II}-O_{d}$	$F \wedge H$	77	76.3–78.8 (4)
$O_{c}-V_{II}-O_{d}$	$G \wedge H$	91	90.4–92.8 (4)
$O_{d}-V_{II}-O_{e}$	$H \wedge J$	84	82.2–84.8 (4)
$O_{a}-V_{II}-O_{f}$	$E \wedge F$	175	174.4–176.4 (2)
$O_{c}-V_{II}-O_{e}$	$G \wedge J$	156	156.1–157.0 (2)
$O_{d}-V_{II}-O_{d}$	$H \wedge H'$	154	154.1–154.2 (2)
$O_{e}-V_{III}-O_{e}'$	$K \wedge K'$	107	106.8 (1)
$O_{a}-V_{III}-O_{a}'$	$L \wedge L'$	77	77.1 (1)
$O_{a}-V_{III}-O_{e}$	$L \wedge K$	88	87.4–88.7 (2)
$O_{a}-V_{III}-O_{b}$	$L \wedge M$	80	79.7–80.4 (4)
$O_{b}-V_{III}-O_{e}$	$M \wedge K$	97	96.0–99.7 (4)
$O_{b}-V_{III}-O_{b}$	$M \wedge M'$	155	155.1 (1)
$O_{a}-V_{III}-O_{e}'$	$L \wedge K'$	165	164.4–165.7 (2)

Shared octahedral edges	Lengths, A	Range (number averaged), A
$O_{b}-O_{b}'$	2.48	2.479 (1)
$O_{a}-O_{b}$	2.61	2.610–2.622 (4)
$O_{a}-O_{d}$	2.66	2.628–2.686 (4)
$O_{a}-O_{c}$	2.76	2.767 (1)
$O_{a}-O_{e}$	2.67	2.666–2.667 (2)
$O_{a}-O_{a}'$	2.64	2.638 (1)
$O_{b}-O_{d}$	2.71	2.698–2.721 (4)

Unshared octahedral edges	Lengths, A	Range (number averaged), A
$O_{b}-O_{g}$	2.75	2.741–2.762 (4)
$O_{b}-O_{e}$	2.73	2.698–2.752 (4)
$O_{d}-O_{c}$	2.66	2.628–2.674 (4)
$O_{d}-O_{f}$	2.72	2.676–2.743 (4)
$O_{d}-O_{e}$	2.63	2.619–2.640 (4)
$O_{d}-O_{g}$	2.70	2.678–2.732 (4)
$O_{e}-O_{e}'$	2.72	2.723 (1)
$O_{d}-O_{d}'$	2.69	2.676–2.700 (2)
$O_{c}-O_{f}$	2.69	2.680–2.703 (2)
$O_{e}-O_{f}$	2.83	2.831–2.837 (2)
$O_{g}-O_{g}'$	2.99	2.991 (1)

Vanadium–vanadium distances	Lengths A	Range (number averaged), A
$V_{I}-V_{I}'$	3.05	3.052 (1)
$V_{I}-V_{III}$	3.16	3.133–3.187 (4)
$V_{I}-V_{II}$	3.12	3.084–3.137 (4)
$V_{II}-V_{II}'$	3.08	3.078 (1)
$V_{II}-V_{III}$	3.08	3.068–3.089 (2)
$V_{III}-V_{III}'$	3.31	3.311 (1)

[a] To identify atoms and vectors, see Figure 3.

There are rather severe distortions in the VO_6 octahedra in the polyion. There are two types of distortion: (1) a displacement of the vanadium atom toward one edge of the octahedron as shown by V_{III} and (2) a displacement of the vanadium atom toward one apex, as shown by V_{I} and V_{II}. These displacements are in a direction consistent with what would be expected by ordinary coulombic repulsion as predicted by the familiar Pauling rule. Accordingly, the O–O distances representing edges of octahedra shared with neighboring octahedra are somewhat shortened. Nevertheless, it cannot escape our attention that both these distortion configurations are already familiar to us in other oxovanadate structures. The square-pyramid coordination is now well known in such structures,[23] in many cases where coulombic repulsion cannot account for the observed distortion, and covalent forces must be active. Tetrahedral (fourfold) and trigonal bipyramidal (fivefold) coordinations are also well known for vanadium(V).[24] The coordinations in the decavanadate ion show features that have a strong relationship to all these types of coordination.

Two of the three types of vanadium in the decavanadate ion, V_{I} and V_{II}, tend toward square-pyramid coordination. In this coordination the vanadium atom has one short bond to the apex of the pyramid (type A), four intermediate bonds to the base atoms of the pyramid (type B), and often a sixth atom bonded at a greater distance through the base (type C). Considerable variation in these bonds has been observed from structure to structure (type A, 1.52–1.68 A; type B, 1.75–2.00 A; type C, 2.20 to >3.50 A). The two groups $V_{I}O_6$ and $V_{II}O_6$ in the decavanadate ion conform to this configuration with average type-A bonds of 1.61 A, type-B bonds of 1.91 A, and type-C bonds of 2.28 A.

The third type of vanadium atom in the decavanadate ion (V_{III}) has two short V–O bonds adjacent to

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[23] H. T. Evans, Jr., *Acta Cryst.*, **13**, 1019 (1960).

[24] H. T. Evans, Jr., *Z. Krist.*, **114**, 17 (1960).

each other in the polyhedral group. The lengths of these bonds (1.69 A) and the angle between them (106.8°) represent a bent VO_2 configuration which is nearly identical with that found in two other vanadate structures KVO_3 and $KVO_3 \cdot H_2O$.[24] The coordination configurations in these structures containing this VO_2 group are shown in Figure 4. Hanic[25] has pointed out that this group might be recognized as a separate VO_2^+ ion in these structures. If this group is considered to be a rather rigid covalently bonded entity, apparently it can coordinate at will two, three, or four other oxygen atoms on the exposed vanadium orbitals with weaker bonds.

At present it is impossible to understand why the structure found here is so especially stable. The only suggestion we may make here is that the stability of the $V_{10}O_{28}^{6-}$ group may in some way be related to the satisfactory way it accommodates the special coordination configurations that are known to be characteristic of vanadium(V).

No special association between the $V_{10}O_{28}^{6-}$ group and any neighboring cations can be discerned in this structure, except through ordinary crystal-structure-building hydrogen bonds. Thus, this crystal structure does not suggest any mechanism for the formation of cation–decavanadate supercomplexes, evidence for which has been advanced on the basis of conductometric studies by Jahr, Fuchs, and Preuss.[26] They found interactions between decavanadate and a number of cations, including Na^+, K^+, Rb^+, Cs^+, Be^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Zn^{2+}, Cu^{2+}, Co^{2+}, La^{3+}, and Ce^{3+}, and proposed that complexes such as $[Zn(OH)_3(H_2O)_{n-3}V_{10}O_{28}]^{7-}$ are formed in the solutions. Other crystal structure analyses may throw more light on this question. Jahr, *et al.*, went so far as to propose elegant cagelike structures for the $V_{10}O_{28}^{6-}$ complex which would be able to trap cations, but unfortunately they considered only tetrahedral coordination for vanadium(V) in spite of ample evidence for the prevalence of higher coordination at lower pH values.

Relation of the Decavanadate Structure to Those of Other Isopoly Ions

The structures of five isopoly ions of elements in groups V and VI have been reported, including the present one. The other four, all determined by Lindqvist, are: heptamolybdate(VI) (paramolybdate) ion in $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$;[27] octamolybdate(VI) ion in $(NH_4)_4Mo_8O_{26} \cdot 7H_2O$;[28] hexaniobate(V) and hexatantalate(V) in $Na_7HNb_6O_{19} \cdot 15H_2O$[29] and $K_8Ta_6O_{19} \cdot 16H_2O$;[30] and dodecatungstate(VI) (paratungstate) ion in $Na_{10}H_{10}W_{12}O_{46} \cdot 23H_2O$.[31] The last complex is not well established[32] and in any case does not appear to have any structural relationship to the others. The structure of the dodecavanadate polyion is essentially the same as that of sodium chloride. If the same condensed octahedral lattice were extended indefinitely in three dimensions (with reduction to vanadium(II)), the sodium chloride structure of VO would result. The structures of all the other polyions mentioned above (except paratungstate) can be derived from the decavanadate structure by merely removing two or more octahedra from the ten-nucleate group. This is illustrated in Figure 5. It can be seen that the paramolybdate ion structure is obtained by removing the front three octahedra from the decavanadate structure; the octamolybdate structure is obtained by removing the left-rear and right-front octahedra; and the hexaniobate structure is obtained by removing four octahedra from one end. This shows that all of these isopoly complexes are based fundamentally on the sodium chloride arrangement of transition-metal atoms and oxygen atoms. We may imagine that in the hydrolysis of their solutions there is a strong tendency to build up a sodium-chloride-like metal–oxygen structure, but this tendency is abruptly interrupted by some sort of electronic energy barrier at a configuration which is very specific for each type of

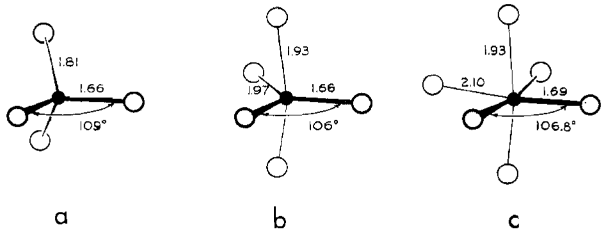

Figure 4.—Three vanadate coordination groups showing common VO_2^+ feature: (a) KVO_3; (b) $KVO_3 \cdot H_2O$; (c) $V_{10}O_{28}^{6-}$.

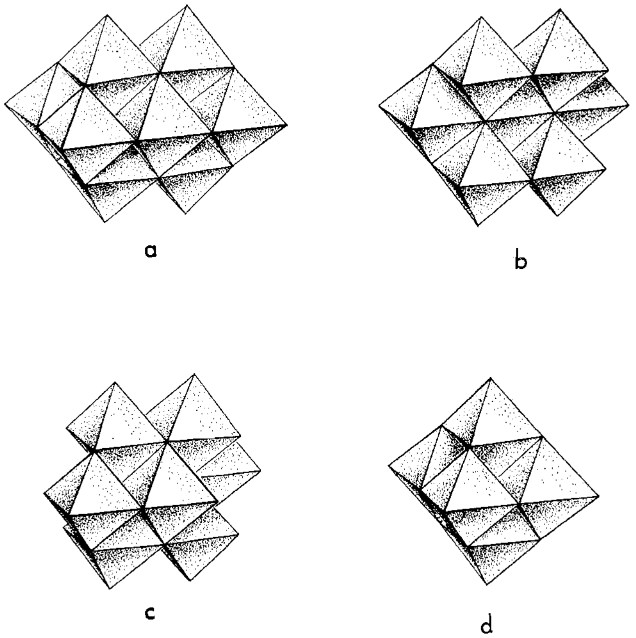

Figure 5.—Four isopoly complexes showing interrelationships of sodium-chloride-like structures: (a) $V_{10}O_{28}^{6-}$; (b) $Mo_7O_{24}^{6-}$; (c) $Mo_8O_{26}^{4-}$; (d) $Ta_6O_{19}^{8-}$.

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(25) F. Hanic, *Chem. Zvesti*, **12**, 579 (1958).
(26) K. F. Jahr, J. Fuchs, and F. Preuss, *Chem. Ber.*, **96**, 556 (1963).
(27) I. Lindqvist, *Arkiv Kemi*, **2**, 325 (1950).
(28) I. Lindqvist, *ibid.*, **2**, 349 (1950).
(29) I. Lindqvist, *ibid.*, **5**, 247 (1953).
(30) I. Lindqvist, *ibid.*, **7**, 49 (1954).
(31) I. Lindqvist, *Acta Cryst.*, **5**, 667 (1952).
(32) W. N. Lipscomb, *Inorg. Chem.*, **4**, 132 (1965).

metal ion. There is no suggestion at this time as to what the nature of that barrier is.

Acknowledgments. The author gratefully acknowledges the extensive assistance of Mary E. Mrose in the earlier stages of this structure study, the assistance of Howard E. Swanson and Eloise H. Evans of the American Society for Testing and Materials, in powder diffraction studies, the stimulating discussions with William N. Lipscomb, and especially the generous cooperation of A. Graham Swallow and William H. Barnes of the National Research Council of Canada in sharing the results of their determination of the structure of calcium decavanadate.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY AND DIVISION OF ENGINEERING,
BROWN UNIVERSITY, PROVIDENCE, RHODE ISLAND 02912

Structure and Properties of Nickel Vanadium Selenide, NiV_2Se_4[1]

BY ROBERT J. BOUCHARD, WARD T. ROBINSON, AND AARON WOLD

Received January 10, 1966

The structure of NiV_2Se_4 was determined by X-ray analysis of a powder sample. The structure is based on the monoclinic space group I2/m. The cell dimensions are $a = 6.142 \pm 0.006$, $b = 3.437 \pm 0.004$, $c = 11.55 \pm 0.01$ A, and $\beta = 91.5 \pm 0.1°$. There appear to be two V–V distances that are less than 3.44 A, and, according to Goodenough, metal–metal d-orbital overlap can occur and may account for the observed metallic behavior of this compound.

Introduction

Recently, a series of monoclinic chalcogenides of the type NiM_2X_4 was prepared and characterized[2] where M was V^{3+} or Cr^{3+} and X was sulfur or selenium. The vanadium compounds were reported to be metallic with positive temperature coefficients of resistivity and low Seebeck voltages. The corresponding chromium compounds appeared to be semiconductors. Unit cell volumes of the vanadium compounds should have been larger than those of the corresponding isostructural chromium compounds since both the ionic and covalent radii of vanadium exceed those of chromium. However, the reverse was true (Table I). An examination of several metal–metal distances and interactions has been carried out in order to investigate this anomaly.

TABLE I

UNIT CELL DIMENSIONS OF SOME ISOSTRUCTURAL MONOCLINIC CHALCOGENIDES

Formula	a, A	b, A	c, A	β, deg	V, A^3
Cr_3S_4[a]	5.95	3.42	11.24	91.5	229
$NiCr_2S_4$[a]	5.90	3.41	11.10	91.4	223
NiV_2S_4	5.82	3.28	11.05	92.0	211
$NiCr_2Se_4$	6.23	3.59	11.52	91.1	258
NiV_2Se_4	6.14	3.44	11.55	91.6	244

[a] Refined cell dimensions are in close agreement with those reported by Jellinek.[4]

Goodenough[3] has indicated that direct interaction of the d orbitals of metal atoms and consequent delocalization of the t_{2g} electrons can occur only if metal–metal distances are less than ≈3.34 A for the thiochromites and ≈3.44 A for the thiovanadites. For the compounds $NiCr_2S_4$ and NiV_2S_4 these metal–metal distances, measured along the b axes, are 3.41 and 3.28 A, respectively, and so this type of bonding could occur in NiV_2S_4 and account for the small unit cell size and metallic properties of this compound.

In order to extend studies of the effect of vanadium–vanadium interactions in these compounds, a powder sample of NiV_2Se_4 has been subjected to X-ray crystal structure analysis.

Experimental Section

NiV_2Se_4 was prepared by reaction of the elements in an evacuated silica tube. The tube was slowly heated to 500° and kept at this temperature until most of the selenium had reacted. The temperature was then raised to 1000° after the sample had been ground under dry nitrogen. Firings were continued at this temperature until no change was observed in the X-ray diffraction pattern and no impurity peaks were present. This diffraction pattern could be indexed approximately on the basis of a monoclinic unit cell of the type ascribed by Jellinek[4] to Cr_3S_4. Subsequent refinement of these parameters by a conventional least-squares procedure,[5] in which the differences between observed and calculated values of $\sin^2 \theta$ were minimized for the indexed reflections, resulted in the values $a = 6.142 \pm 0.006$, $b = 3.437 \pm 0.004$, $c = 11.55 \pm 0.01$ A, and $\beta = 91.5 \pm 0.1°$, where maximum errors in these quantities are indicated.

X-Rays from a high-intensity copper source were diffracted by the powder, then monochromatized (AMR-202 focusing monochromator) and detected by electronic pulse counting using a Norelco diffractometer. Relative intensities (I_o) for the various multiple reflections were obtained by fixed time scans from background to background on either side of each peak and then by subtracting the appropriate portions of an established curve of background intensity *vs.* angle. Lorentz and polarization[6,7] corrections were applied to these intensities, and the absolute

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[1] This work has been supported by ARPA and Grant No. AF 19(628)-3837.

[2] R. J. Bouchard and A. Wold, *J. Chem. Phys. Solids*, **27**, 591 (1966).

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[7] J. T. Norton, Advanced Metals Research Corp., Burlington, Mass., personal communication.