O1$^{\rm i}$—Ga1—O2	119.59 (9)	O1—Ga2—O2$^{\rm iii}$	91.87 (7)
O1$^{\rm i}$—Ga1—O3$^{\rm ii}$	106.79 (7)	O1—Ga2—O3	94.66 (7)
O2—Ga1—O3$^{\rm ii}$	105.92 (7)	O2—Ga2—O2$^{\rm v}$	94.14 (7)
O3$^{\rm ii}$—Ga1—O3$^{\rm iv}$	111.9 (1)	O2—Ga2—O2$^{\rm iii}$	80.91 (6)
O1—Ga2—O1$^{\rm v}$	103.22 (9)	O2—Ga2—O3	91.95 (7)
O1—Ga2—O2	80.91 (6)		

Symmetry codes: (i) $x, y, 1+z$; (ii) $-x, -y, 1-z$; (iii) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$; (iv) $-x, 1-y, 1-z$; (v) $x, 1+y, z$.

The starting parameters for the refinement were taken from Geller (1960). One reflection (234) with $\Delta F/\sigma = 53.6$ was removed since the next largest value (5.2 for $\bar{3}10$) is more than 90% smaller. The large residual electron density (2.89 e Å^{-3}) is situated between Ga1 and Ga2. Refinement including this position as possible disorder did not lead to an improved model. Anharmonic tensors were also used in an attempt to describe the residual electron density as thermal displacement. Third and fourth order (γ, δ) tensor components in the Edgeworth expansion were refined, but neither $\Delta\rho$ nor the R values were significantly improved.

Wolten & Chase (1976) proposed that the space group for β-Ga_2O_3 should be $P1$. No evidence has been found in the present study to support this, since the diffraction symmetry and systematic extinctions clearly indicate a C-centered monoclinic cell, as discussed by Geller (1977). Refinement of the structure in space group $P1$ gave no improvement.

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *LATCON* (Schwarzenbach & King, 1992). Data reduction: *CRYST* (Lundgren, 1982). Program(s) used to solve structure: *CRYST*. Program(s) used to refine structure: *CRYST*. Software used to prepare material for publication: *TEXSAN* (Molecular Structure Corporation, 1992).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: FG1144). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$Ba_3[V_{10}O_{28}].19H_2O$

Boris Kamenar, Marina Cindrić and Neven Strukan

Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, PO Box 153, 10000 Zagreb, Croatia. E-mail: neven.strukan@public.srce.hr

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Abstract

The crystal structure of barium decavanadate nonadecahydrate, $Ba_3[V_{10}O_{28}].19H_2O$, has been determined. The polyanion consists of ten distorted VO_6 edge-sharing octahedra and has a well established structure. There are two crystallographically independent decavanadate anions in the asymmetric unit, located around the centres of inversion at $\frac{1}{2},0,0$ and $0,\frac{1}{2},\frac{1}{2}$. Each of the three Ba^{2+} cations is surrounded by nine O atoms from water molecules and $[V_{10}O_{28}]^{6-}$ anions in a distorted square antiprism with one additional vertex.

Comment

Several structures containing the $[V_{10}O_{28}]^{6-}$ anion are known from the literature: $Na_6[V_{10}O_{28}].18H_2O$ (Durif, Averbuch-Pouchot & Guitel, 1980), $Ca_3[V_{10}O_{28}].17H_2O$ (Marvin & Magin, 1959; Swallow, Ahmed & Barnes, 1966), $Sr_3[V_{10}O_{28}].22H_2O$ (Nieto, Salagre, Medina, Sueritas & Solans, 1993), $Er_2[V_{10}O_{28}].25H_2O$ (Rivero, Rigotti, Punte & Navaza, 1984) and double salts such as $K_2Mg_2[V_{10}O_{28}].16H_2O$, $Cs_2Mg_2[V_{10}O_{28}].16H_2O$ and $K_2Zn_2[V_{10}O_{28}].16H_2O$ (Weeks, Cisney & Sherwood, 1951; Evans, 1966). However, although the title compound was prepared 120 years ago (Norblad, 1875), up to now its structure has remained unsolved.

The title compound was prepared and studied as part of our research on molybdates, vanadates and mixed molybdovanadates (Kamenar, Cindrić & Strukan, 1994; Cindrić, Kamenar, Strukan & Veksli, 1995). The decavanadate anion consists of ten distorted VO_6 octahedra sharing edges and is basically the same as that found in other inorganic decavanadates. The two crystallographically independent $[V_{10}O_{28}]^{6-}$ anions in the asymmetric unit are located around the crystallographic centres of inversion at $\frac{1}{2},0,0$ and $0,\frac{1}{2},\frac{1}{2}$. The V—O distances are similar to those already observed and depend upon the type of oxo ligand: V═O$_t$ bond lengths to the terminal oxo O atoms vary between 1.599 (5) and 1.617 (5) Å, V—O$_b$ bond lengths to the O atoms bonded to two V atoms vary from 1.681 (4) to 2.094 (4) Å, V—O$_{3b}$ bond lengths to the O atoms bonded to three V atoms vary from 1.888 (4) to 2.043 (4) Å and V—O$_{6b}$ bond lengths to the O atoms shared between six V atoms range from 2.114 (4) to 2.336 (4) Å. The VO$_6$ octahedra are significantly distorted, with the bond angles at the V atoms ranging from 74.1 (2) to 107.3 (2)°.

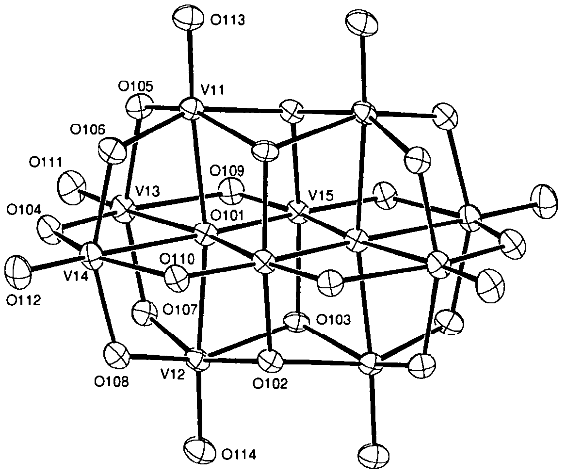

Fig. 1. A view of one of the two crystallographically independent $[V_{10}O_{28}]^{6-}$ anions in the title compound showing the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level. The other anion has essentially the same features and an analogous labelling scheme.

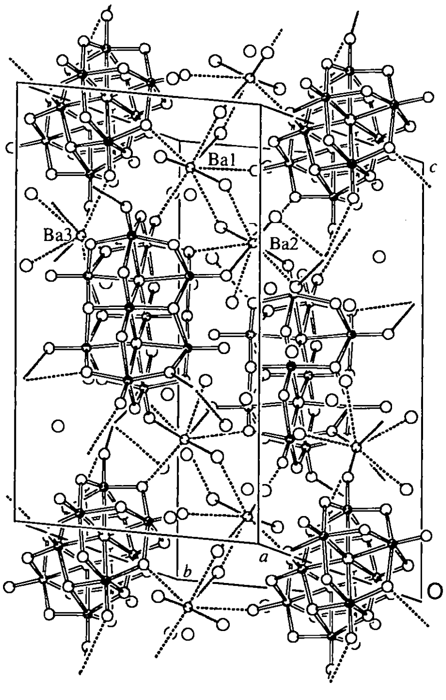

Fig. 2. A view of the bridging of the decavanadate anions by the hydrated Ba^{2+} cations.

The Ba^{2+} cations are surrounded by nine O atoms at distances varying from 2.744 (4) to 2.970 (6) Å. Ba1 and Ba3 are surrounded by six water O atoms and three O atoms from the polyanion, and Ba2 by five water O atoms and four O atoms from the polyanion. The coordination polyhedra around the Ba^{2+} ions can be described as highly distorted square antiprisms, each with one additional vertex. In spite of the suggestion that the charge-to-radius ratio of the cation determines the number of hydrate molecules of the compound (Nieto *et al.*, 1993), the present structure, that of the analogous sodium salt (Durif, Averbuch-Pouchot & Guitel, 1980) and those of the salts of some lanthanides or yttrium (Saf'yanov, Kuz'min & Belov, 1979; Rivero *et al.*, 1984) do not support such a conclusion.

Experimental

The title compound was prepared by mixing a hot water solution of MoO$_3$ and Ba(OH)$_2$ and a suspension of NH$_4$VO$_3$ in water, using hydrochloric acid to adjust to pH 5. The mixture was filtered off and solid BaCl$_2$ was added to the resulting yellow solution. After several days at room temperature, transparent orange crystals were isolated. The water content was determined thermogravimetrically.

Crystal data

$Ba_3[V_{10}O_{28}].19H_2O$
$M_r = 1711.68$
Triclinic
$P\bar{1}$
$a = 9.491\ (3)$ Å
$b = 12.201\ (6)$ Å
$c = 18.786\ (12)$ Å
$\alpha = 81.27\ (3)°$
$\beta = 81.02\ (2)°$
$\gamma = 71.12\ (2)°$
$V = 2021.2\ (18)$ Å^3
$Z = 2$
$D_x = 2.81$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 40 reflections
$\theta = 7.7–14.7°$
$\mu = 5.13$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.56 × 0.48 × 0.15 mm
Orange

Data collection

Philips PW1100 diffractometer
ω scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.0796$, $T_{\max} = 0.2084$
11 054 measured reflections
11 037 independent reflections

8545 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0617$
$\theta_{\max} = 30.08°$
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 17$
$l = 0 \rightarrow 26$
3 standard reflections
frequency: 90 min
intensity decay: 6.7%

Refinement

Refinement on F^2
$R(F) = 0.0587$
$wR(F^2) = 0.1557$
$S = 1.051$
10 996 reflections
541 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1226P)^2 + 7.3131P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 3.32$ e Å^{-3}
$\Delta\rho_{\min} = -3.74$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
V11	0.06785 (10)	0.66500 (9)	0.40039 (5)	0.0170 (2)
V12	−0.07633 (11)	0.34605 (9)	0.43587 (6)	0.0175 (2)
V13	0.15492 (11)	0.43592 (9)	0.32801 (6)	0.0190 (2)
V14	−0.17306 (11)	0.58879 (10)	0.34493 (6)	0.0196 (2)
V15	0.17555 (10)	0.41820 (8)	0.49118 (5)	0.0150 (2)
O101	−0.0024 (4)	0.5065 (4)	0.4283 (2)	0.0162 (7)
O102	−0.1906 (4)	0.4252 (4)	0.5208 (2)	0.0167 (7)
O103	0.0739 (4)	0.3052 (4)	0.5082 (2)	0.0169 (7)
O104	−0.0113 (5)	0.5179 (4)	0.2822 (2)	0.0231 (9)
O105	0.1950 (4)	0.5782 (4)	0.3342 (2)	0.0190 (8)
O106	−0.0946 (5)	0.7084 (4)	0.3496 (3)	0.0220 (8)
O107	0.0823 (5)	0.3129 (4)	0.3624 (2)	0.0196 (8)
O108	−0.2064 (5)	0.4441 (4)	0.3770 (2)	0.0198 (8)
O109	0.2802 (4)	0.3786 (4)	0.4130 (2)	0.0200 (8)
O110	−0.2887 (4)	0.6337 (4)	0.4419 (2)	0.0192 (8)
O111	0.2772 (5)	0.3843 (5)	0.2619 (3)	0.0271 (10)
O112	−0.3024 (5)	0.6517 (5)	0.2930 (3)	0.0278 (10)
O113	0.1258 (5)	0.7778 (4)	0.3858 (3)	0.0238 (9)
O114	−0.1274 (6)	0.2313 (4)	0.4490 (3)	0.0278 (10)
V21	0.68216 (10)	−0.16351 (8)	−0.08468 (5)	0.0156 (2)
V22	0.55147 (10)	−0.06635 (9)	0.14050 (5)	0.0161 (2)
V23	0.85008 (11)	−0.16637 (10)	0.04610 (6)	0.0213 (2)
V24	0.61082 (11)	−0.28834 (9)	0.06619 (6)	0.0195 (2)
V25	0.63230 (10)	0.06440 (9)	−0.01159 (5)	0.0158 (2)
O201	0.5988 (4)	−0.1008 (4)	0.0247 (2)	0.0177 (8)
O202	0.3461 (4)	−0.0080 (4)	0.1031 (2)	0.0178 (8)
O203	0.5424 (4)	0.0880 (4)	0.0861 (2)	0.0174 (8)
O204	0.8029 (5)	−0.3033 (4)	0.0733 (2)	0.0214 (8)
O205	0.8612 (5)	−0.1881 (4)	−0.0526 (2)	0.0221 (8)
O206	0.6462 (5)	−0.2937 (4)	−0.0356 (2)	0.0216 (8)
O207	0.7535 (5)	−0.1073 (4)	0.1328 (2)	0.0216 (8)
O208	0.5382 (5)	−0.2142 (4)	0.1503 (2)	0.0201 (8)
O209	0.8111 (5)	0.0038 (4)	0.0067 (3)	0.0225 (9)
O210	0.3903 (5)	−0.2062 (4)	0.0424 (2)	0.0210 (8)
O211	1.0241 (5)	−0.2023 (5)	0.0570 (3)	0.0316 (11)
O212	0.5920 (5)	−0.4154 (4)	0.0930 (3)	0.0274 (10)
O213	0.7225 (6)	−0.1971 (5)	−0.1671 (3)	0.0297 (10)
O214	0.4945 (6)	−0.0288 (5)	0.2212 (3)	0.0283 (10)
Ba1	0.39943 (4)	0.44510 (3)	0.11925 (2)	0.02452 (10)
Ba2	0.39430 (4)	0.70960 (3)	0.28091 (2)	0.02006 (10)
Ba3	0.24677 (5)	0.10221 (4)	0.31434 (2)	0.03116 (12)
Ow1	0.4655 (6)	0.5183 (5)	0.7641 (3)	0.0302 (10)
Ow2	0.8034 (6)	0.0694 (5)	0.7663 (3)	0.0295 (10)
Ow3	0.6601 (6)	0.3946 (5)	0.0081 (3)	0.0328 (11)
Ow4	0.3889 (6)	0.7673 (6)	0.8392 (3)	0.0397 (13)
Ow5	0.7415 (6)	0.3171 (5)	0.8352 (3)	0.0345 (11)
Ow6	0.5843 (7)	0.1279 (5)	0.6317 (3)	0.0378 (12)
Ow7	0.5833 (6)	0.3318 (6)	0.3671 (4)	0.0394 (13)
Ow8	0.5411 (6)	0.4189 (6)	0.5872 (3)	0.0392 (14)
Ow9	0.3071 (8)	0.1072 (6)	0.4560 (4)	0.049 (2)
Ow10	0.0878 (7)	0.5308 (7)	0.1319 (4)	0.053 (2)
Ow11	0.9223 (8)	0.4145 (7)	0.0492 (5)	0.062 (2)
Ow12	0.4753 (9)	0.8488 (6)	0.6948 (4)	0.051 (2)
Ow13	0.9142 (9)	0.1021 (8)	0.1305 (5)	0.063 (2)
Ow14	0.8432 (8)	0.4014 (7)	0.2039 (4)	0.058 (2)
Ow15	0.5996 (9)	0.1329 (7)	0.4818 (4)	0.057 (2)
Ow16	0.6712 (10)	0.9209 (8)	0.3734 (6)	0.076 (3)
Ow17	0.9649 (11)	0.8454 (13)	0.2282 (5)	0.119 (6)
Ow18	0.0460 (10)	0.8326 (9)	0.7327 (6)	0.078 (3)
Ow19	0.0539 (13)	0.0079 (7)	0.4133 (6)	0.097 (4)

Table 2. *Selected geometric parameters (Å)*

V11—O113	1.612 (5)	V21—O213	1.617 (5)
V11—O105	1.796 (4)	V21—O205	1.810 (4)
V11—O106	1.830 (4)	V21—O206	1.815 (5)
V11—O102$^{\rm i}$	1.993 (4)	V21—O202$^{\rm ii}$	2.004 (5)
V11—O103$^{\rm i}$	2.011 (4)	V21—O203$^{\rm ii}$	2.032 (4)
V11—O101	2.208 (4)	V21—O201	2.238 (4)
V12—O114	1.599 (5)	V22—O214	1.611 (5)
V12—O108	1.802 (4)	V22—O207	1.807 (5)
V12—O107	1.861 (4)	V22—O208	1.828 (5)
V12—O102	1.976 (4)	V22—O203	1.984 (5)
V12—O103	2.017 (4)	V22—O202	2.043 (4)
V12—O101	2.263 (4)	V22—O201	2.228 (5)
V13—O111	1.612 (5)	V23—O211	1.603 (5)
V13—O104	1.836 (5)	V23—O204	1.846 (5)
V13—O107	1.839 (5)	V23—O207	1.863 (5)
V13—O105	1.917 (5)	V23—O205	1.894 (5)
V13—O109	2.041 (4)	V23—O209	2.029 (5)
V13—O101	2.302 (4)	V23—O201	2.336 (4)
V14—O112	1.608 (5)	V24—O212	1.613 (5)
V14—O104	1.839 (5)	V24—O204	1.797 (4)
V14—O106	1.858 (5)	V24—O208	1.866 (5)
V14—O108	1.887 (5)	V24—O206	1.897 (5)
V14—O110	2.028 (4)	V24—O210	2.094 (4)
V14—O101	2.311 (4)	V24—O201	2.275 (5)
V15—O109	1.681 (4)	V25—O209	1.684 (4)
V15—O110$^{\rm i}$	1.695 (4)	V25—O210$^{\rm ii}$	1.694 (5)
V15—O103	1.888 (4)	V25—O202$^{\rm ii}$	1.905 (5)
V15—O102$^{\rm i}$	1.939 (4)	V25—O203	1.921 (4)
V15—O101	2.114 (4)	V25—O201	2.135 (4)
V15—O101$^{\rm i}$	2.122 (4)	V25—O201$^{\rm ii}$	2.138 (4)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, -y, -z$.

Table 3. *Contact distances (Å)*

Ba1···O111	2.839 (5)	Ba2···Ow2$^{\rm iii}$	2.845 (5)
Ba1···O206$^{\rm i}$	2.771 (6)	Ba2···Ow5$^{\rm iii}$	2.809 (7)
Ba1···O212$^{\rm ii}$	2.821 (6)	Ba2···Ow6$^{\rm iii}$	2.832 (7)
Ba1···Ow1$^{\rm iii}$	2.861 (6)	Ba2···Ow8$^{\rm iii}$	2.766 (6)
Ba1···Ow3	2.941 (5)	Ba3···O107	2.744 (4)
Ba1···Ow3$^{\rm iv}$	2.856 (5)	Ba3···O213$^{\rm i}$	2.842 (5)
Ba1···Ow4$^{\rm iii}$	2.805 (6)	Ba3···O214	2.877 (5)
Ba1···Ow5$^{\rm iii}$	2.970 (6)	Ba3···Ow2$^{\rm vi}$	2.957 (7)
Ba1···Ow10	2.785 (6)	Ba3···Ow6$^{\rm vi}$	2.866 (6)
Ba2···O105	2.834 (5)	Ba3···Ow9	2.821 (8)
Ba2···O112$^{\rm v}$	2.771 (5)	Ba3···Ow12$^{\rm iii}$	2.863 (9)
Ba2···O113	2.938 (4)	Ba3···Ow18$^{\rm vii}$	2.874 (10)
Ba2···O208$^{\rm ii}$	2.799 (4)	Ba3···Ow19	2.789 (12)
Ba2···Ow1$^{\rm iii}$	2.861 (6)		

Symmetry codes: (i) $1-x, -y, -z$; (ii) $x, 1+y, z$; (iii) $1-x, 1-y, 1-z$; (iv) $1-x, 1-y, -z$; (v) $1+x, y, z$; (vi) $1-x, -y, 1-z$; (vii) $-x, 1-y, 1-z$.

41 reflections with $\Delta/\sigma > 5$ were omitted from the refinement. Most of the H atoms of the water molecules were found in a final difference Fourier map, but they were neither included in the refinement nor in the structure-factor calculations. The largest remaining difference peaks were located near the Ba^{2+} cations.

Data collection and cell refinement: *DIF*4 (Stoe & Cie, 1992*a*). Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). The structure was solved by Patterson and Fourier methods using *SHELXS*86 (Sheldrick, 1985) and refined by full-matrix least squares on F^2 using *SHELXL*93 (Sheldrick, 1993). Software used to prepare molecular graphics and material for publication: *ORTEP*II (Johnson, 1976), *PLUTON* (Spek, 1982), *SHELXL*93, *CSU* (Vicković, 1988).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1133). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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