

O5W—H5WB···O5	0.95 (5)	1.96 (3)	2.853 (7)	157 (7)
O6W—H6WA···O11 <sup>vi</sup>	0.95 (7)	2.10 (5)	2.948 (7)	149 (8)
N24—H24A···O4 <sup>viii</sup>	1.12 (1)	1.69 (1)	2.776 (6)	161 (1)
N24—H24B···O3 <sup>ix</sup>	1.07 (2)	1.69 (1)	2.746 (6)	167 (1)

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

All H atoms were found in the difference electron-density map. The H atoms of (cfH)<sup>+</sup> were included in the refinement at calculated positions, whereas the H atoms of the water molecules were refined with the O—H and H···H distances restrained (*DFIX* in *SHELXL93*; Sheldrick, 1993) to 0.95 (1) and 1.50 (2) Å, respectively. For all H atoms, the values of the isotropic displacement parameters were taken as 1.5 times the value of the attached heavy atom. The rather high value of the final *R* factor is due to the slight decomposition of the crystal during data collection. In addition to the computer programs mentioned below, *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989) and *PLUTON* (Spek, 1991) were used for data processing and the final interpretation of the structural geometry. Calculations were performed on a PC 486 computer.

Data collection: local program (Kratky, 1993). Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1971) and *PLUTON*. Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1358). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Tris(tetraphenylphosphonium) Decatelluriumtantalide Dimethylformamide (1/1), a Tetraphenylphosphonium Salt of TaTe<sub>10</sub><sup>3-</sup>

WALTER A. FLOMER, JOSEPH W. KOLIS AND  
WILLIAM T. PENNINGTON

Department of Chemistry, Clemson University, Clemson,  
SC 29634-1905, USA. E-mail: xraylab@clemson.edu

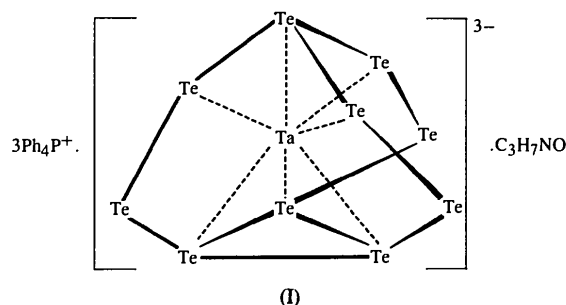
(Received 4 September 1995; accepted 7 May 1996)

## Abstract

The TaTe<sub>10</sub><sup>3-</sup> anion of the title compound, (C<sub>24</sub>H<sub>20</sub>P)<sub>3</sub>–[TaTe<sub>10</sub>].C<sub>3</sub>H<sub>7</sub>NO, consists of an extended ‘birdcage’ of Te atoms containing an interstitial Ta atom. The Ta atom is seven-coordinate, with Ta—Te distances ranging from 2.753 (3) to 2.870 (3) Å, arranged in a distorted capped trigonal prism geometry.

## Comment

The title compound, (I), is isomorphous with that of the niobium derivative (Flomer & Kolis, 1988). The geometric parameters of the two structures are identical within experimental error. In addition to the anion, the structure contains three tetraphenylphosphonium cations



and one badly disordered dimethylformamide (DMF) solvent molecule. The cations, anions and solvent molecules are well separated, with no close contacts between them.

The structure of the anion is similar to that of  $P_4S_3$  (Leung & Waser, 1957) and  $P_7^{3-}$  (Dahlmann & von Schnering, 1972), but with ditelluride rather than monotelluride bridges linking the apex and base. To the best of our knowledge, this is the only isolated tantalum–tellurium anion that has been structurally characterized, but the Ta–Te distances fall within the ranges of known tantalum tellurides,  $TaTe_2$  (Brown, 1966) and  $TaTe_4$  (Bjerkelund & Kjekshus, 1964), and are considerably longer than the value of 2.568 (1) Å found for a terminal Ta–Te bond in  $[TeTa\{(Me_3SiNCH_2CH_2)_3N\}]$  (Christou & Arnold, 1993). The Te–Te distances are grouped into two significantly different ranges, *i.e.* short

bonds [2.827 (5)–2.905 (4) Å] between bridging atoms and from the bridges to the base, and long bonds [3.068 (4)–3.171 (5) Å] involving the apical atom and within the  $Te_3$  base. The shorter bond lengths are similar to the value of 2.835 Å observed for elemental tellurium (Cherin & Unger, 1967), while the longer ones are experimentally within the limit of 3.14 Å considered to constitute a bonding interaction between Te atoms (Bogan, Rauchfuss & Rheingold, 1985).

## Experimental

The title compound was prepared by slow addition of a solution of  $TaCl_5$  (0.48 mmol) and  $PPh_4Br$  (1.19 mmol) in DMF (5 ml) to a stirred solution of  $K_2Te_2$  (1.40 mmol) in DMF (5 ml). The resulting brown solution was stirred (1 h) and then filtered. Crystals were grown by overnight storage of the filtrate, layered with tetrahydrofuran (10 ml), at reduced temperature (279 K).

### Crystal data

$(C_{24}H_{20}P)_3[TaTe_{10}]\cdot C_3H_7NO$

$M_r = 2548.16$

Triclinic

$P\bar{1}$

$a = 13.193$  (6) Å

$b = 14.523$  (7) Å

$c = 23.784$  (9) Å

$\alpha = 82.26$  (4)°

$\beta = 102.89$  (3)°

$\gamma = 109.25$  (3)°

$V = 4185$  (3) Å<sup>3</sup>

$Z = 2$

$D_x = 2.02$  Mg m<sup>-3</sup>

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 19

reflections

$\theta = 13$ – $14^\circ$

$\mu = 4.83$  mm<sup>-1</sup>

$T = 295$  K

Parallelepiped

$0.34 \times 0.29 \times 0.21$  mm

Black

### Data collection

Siemens R3m/V diffractometer

$\omega$ - $2\theta$  scans

Absorption correction:

empirical via  $\psi$  scans

(*SHELXTL-Plus*;

Sheldrick, 1990)

$T_{\min} = 0.21$ ,  $T_{\max} = 0.36$

11 475 measured reflections

10 909 independent

reflections

4544 observed reflections

$[F > 6\sigma(F)]$

$R_{\text{int}} = 0.0449$

$\theta_{\max} = 22.5^\circ$

$h = 0 \rightarrow 15$

$k = -16 \rightarrow 16$

$l = -26 \rightarrow 26$

3 standard reflections

monitored every 97

reflections

intensity decay: none

### Refinement

Refinement on  $F$

$R = 0.0598$

$wR = 0.0709$

$S = 1.502$

4544 reflections

287 parameters

$w = 1/[\sigma^2(F) + 0.0005F^2]$

$(\Delta/\sigma)_{\max} = 0.193$

$\Delta\rho_{\max} = 1.744$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.209$  e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *SHELXTL-Plus*

(Sheldrick, 1990)

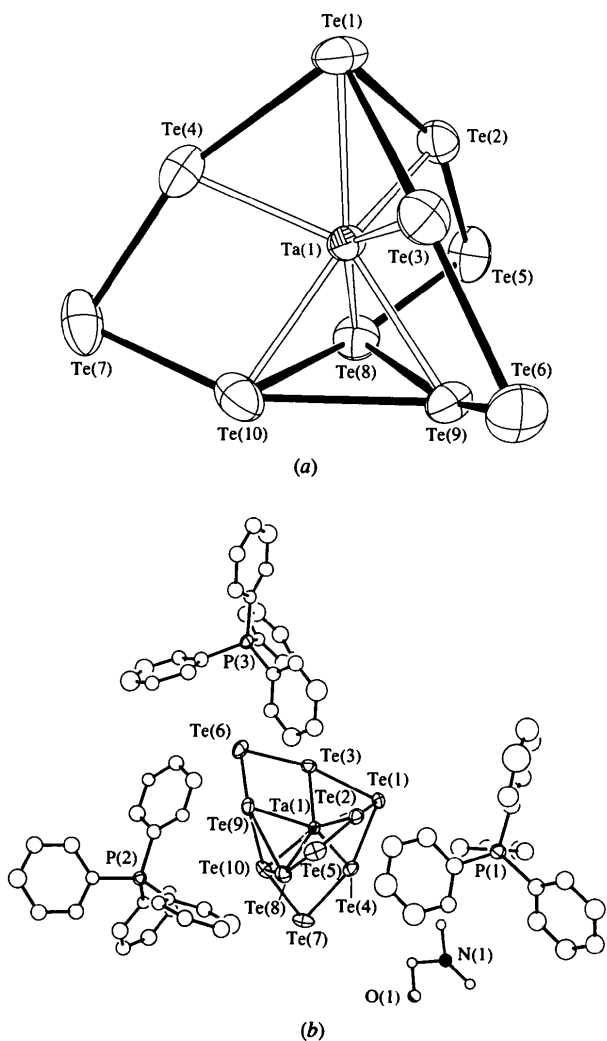


Fig. 1. View of (a) the  $TaTe_{10}^{3-}$  anion (50% probability ellipsoids) and (b) the full structure (35% probability ellipsoids), showing the labeling of the atoms.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
Ta(1)	0.4114 (1)	0.1880 (1)	0.2567 (1)	0.035 (1)
Te(1)	0.5420 (2)	0.1880 (2)	0.3708 (1)	0.057 (1)
Te(2)	0.3348 (2)	0.2618 (2)	0.3343 (1)	0.054 (1)
Te(3)	0.6260 (2)	0.3053 (2)	0.2598 (1)	0.053 (1)
Te(4)	0.4164 (2)	0.0052 (2)	0.3035 (1)	0.057 (1)
Te(5)	0.1295 (2)	0.2782 (2)	0.2657 (1)	0.071 (1)
Te(6)	0.6167 (2)	0.4062 (2)	0.1472 (1)	0.069 (1)
Te(7)	0.3028 (2)	-0.1139 (2)	0.2060 (2)	0.074 (1)
Te(8)	0.1896 (2)	0.1631 (2)	0.2000 (1)	0.057 (1)
Te(9)	0.3938 (2)	0.3091 (2)	0.1537 (1)	0.056 (1)
Te(10)	0.3513 (2)	0.0807 (2)	0.1565 (1)	0.062 (1)
P(1)	0.1155 (7)	0.2196 (7)	0.5468 (4)	0.046 (4)
P(2)	0.0269 (7)	0.3111 (6)	-0.0185 (4)	0.035 (4)
P(3)	0.6542 (7)	0.7625 (7)	0.2020 (4)	0.041 (4)
C(111)	0.0377 (18)	0.2356 (18)	0.4772 (9)	0.052 (10)
C(112)	-0.0679	0.1738	0.4588	0.047 (10)
C(113)	-0.1291	0.1943	0.4056	0.085 (14)
C(114)	-0.0849	0.2766	0.3708	0.093 (15)
C(115)	0.0207	0.3384	0.3891	0.080 (13)
C(116)	0.0820	0.3179	0.4424	0.067 (12)
C(121)	0.2152 (16)	0.1606 (15)	0.5451 (11)	0.035 (8)
C(122)	0.2460	0.1611	0.4926	0.065 (12)
C(123)	0.3212	0.1140	0.4897	0.072 (12)
C(124)	0.3656	0.0666	0.5392	0.068 (12)
C(125)	0.3347	0.0661	0.5917	0.087 (14)
C(126)	0.2595	0.1132	0.5946	0.062 (11)
C(131)	0.0258 (17)	0.1401 (16)	0.5943 (9)	0.050 (10)
C(132)	-0.0095	0.0393	0.5895	0.065 (12)
C(133)	-0.0793	-0.0222	0.6247	0.077 (13)
C(134)	-0.1138	0.0170	0.6646	0.061 (11)
C(135)	-0.0785	0.1179	0.6694	0.074 (13)
C(136)	-0.0087	0.1794	0.6342	0.057 (11)
C(141)	0.181 (2)	0.3372 (15)	0.5763 (11)	0.056 (10)
C(142)	0.2917	0.3642	0.6017	0.059 (11)
C(143)	0.3445	0.4580	0.6208	0.090 (15)
C(144)	0.2865	0.5248	0.6144	0.094 (15)
C(145)	0.1758	0.4978	0.5890	0.13 (2)
C(146)	0.1230	0.4040	0.5699	0.073 (13)
C(211)	-0.0320 (16)	0.3686 (14)	-0.0855 (8)	0.033 (8)
C(212)	-0.1400	0.3239	-0.1119	0.043 (9)
C(213)	-0.1869	0.3672	-0.1624	0.046 (9)
C(214)	-0.1258	0.4553	-0.1866	0.064 (11)
C(215)	-0.0178	1/2	-0.1603	0.051 (10)
C(216)	0.0291	0.4567	-0.1097	0.050 (10)
C(221)	0.0524 (16)	0.2001 (12)	-0.0312 (9)	0.033 (8)
C(222)	0.1368	0.1710	0.0051	0.038 (9)
C(223)	0.1563	0.0862	-0.0046	0.037 (8)
C(224)	0.0914	0.0307	-0.0506	0.056 (10)
C(225)	0.0071	0.0598	-0.0869	0.046 (9)
C(226)	-0.0124	0.1446	-0.0772	0.054 (10)
C(231)	-0.0631 (14)	0.2852 (15)	0.0312 (8)	0.026 (7)
C(232)	-0.1440	0.3301	0.0247	0.038 (9)
C(233)	-0.2085	0.3120	0.0667	0.043 (9)
C(234)	-0.1920	0.2489	0.1150	0.048 (10)
C(235)	-0.1111	0.2040	0.1215	0.069 (12)
C(236)	-0.0467	0.2222	0.0796	0.048 (10)
C(241)	0.1574 (12)	0.3946 (15)	0.0109 (9)	0.041 (9)
C(242)	0.2430	0.4179	-0.0192	0.055 (10)
C(243)	0.3460	0.4792	0.0041	0.034 (8)
C(244)	0.3633	0.5172	0.0576	0.048 (10)
C(245)	0.2777	0.4938	0.0876	0.054 (10)
C(246)	0.1747	0.4325	0.0643	0.036 (8)
C(311)	0.6979 (16)	0.8881 (11)	0.2222 (9)	0.034 (8)
C(312)	0.8037	0.9305	0.2524	0.045 (9)
C(313)	0.8372	1.0278	0.2662	0.073 (12)
C(314)	0.7649	1.0826	0.2498	0.055 (10)
C(315)	0.6592	1.0402	0.2196	0.048 (10)
C(316)	0.6257	0.9430	0.2058	0.049 (10)
C(321)	0.7700 (15)	0.7170 (15)	0.2150 (10)	0.038 (9)
C(322)	0.8672	0.7715	0.1968	0.055 (10)
C(323)	0.9578	0.7376	0.2112	0.081 (14)
C(324)	0.9511	0.6492	0.2439	0.066 (12)
C(325)	0.8539	0.5947	0.2621	0.055 (10)
C(326)	0.7634	0.6286	0.2477	0.055 (10)
C(331)	0.5904 (16)	0.7529 (16)	0.1247 (7)	0.044 (9)
C(332)	0.6466	0.7457	0.0831	0.042 (9)
C(333)	0.5980	0.7474	0.0247	0.074 (13)
C(334)	0.4931	0.7562	0.0079	0.079 (13)
C(335)	0.4369	0.7634	0.0494	0.040 (9)
C(336)	0.4855	0.7617	0.1079	0.048 (10)
C(341)	0.5485 (15)	0.6887 (14)	0.2393 (9)	0.042 (9)
C(342)	0.5166	0.7267	0.2809	0.048 (10)
C(343)	0.4326	0.6679	0.3087	0.085 (14)
C(344)	0.3805	0.5710	0.2949	0.058 (11)
C(345)	0.4124	0.5329	0.2534	0.068 (12)
C(346)	0.4963	0.5918	0.2256	0.043 (9)
O(1)	0.159 (5)	-0.374 (5)	0.380 (3)	0.27 (2)
N(1)	0.246 (7)	-0.294 (7)	0.443 (4)	0.27 (2)
C(1)	0.241 (9)	-0.290 (8)	0.384 (5)	0.27 (2)
C(2)	0.191 (8)	-0.363 (8)	0.479 (4)	0.27 (2)
C(3)	0.317 (8)	-0.193 (8)	0.445 (4)	0.27 (2)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Ta(1)—Te(1)	2.870 (3)	Te(2)—Te(5)	2.899 (4)
Ta(1)—Te(2)	2.766 (4)	Te(3)—Te(6)	2.873 (4)
Ta(1)—Te(3)	2.772 (3)	Te(4)—Te(7)	2.905 (4)
Ta(1)—Te(4)	2.753 (3)	Te(5)—Te(8)	2.827 (5)
Ta(1)—Te(8)	2.860 (3)	Te(6)—Te(9)	2.841 (4)
Ta(1)—Te(9)	2.829 (3)	Te(7)—Te(10)	2.834 (4)
Ta(1)—Te(10)	2.843 (4)	Te(8)—Te(9)	3.146 (4)
Te(1)—Te(2)	3.161 (4)	Te(8)—Te(10)	3.171 (5)
Te(1)—Te(3)	3.153 (4)	Te(9)—Te(10)	3.170 (4)
Te(1)—Te(4)	3.068 (4)		
Te(1)—Ta(1)—Te(2)	68.2 (1)	Te(9)—Ta(1)—Te(10)	68.0 (1)
Te(1)—Ta(1)—Te(3)	67.9 (1)	Te(2)—Te(1)—Te(3)	88.6 (1)
Te(2)—Ta(1)—Te(3)	105.5 (1)	Te(2)—Te(1)—Te(4)	89.7 (1)
Te(1)—Ta(1)—Te(4)	66.1 (1)	Te(3)—Te(1)—Te(4)	91.4 (1)
Te(2)—Ta(1)—Te(4)	105.5 (1)	Te(1)—Te(2)—Te(5)	157.4 (1)
Te(3)—Ta(1)—Te(4)	107.4 (1)	Te(1)—Te(3)—Te(6)	158.3 (1)
Te(1)—Ta(1)—Te(8)	140.5 (1)	Te(1)—Te(4)—Te(7)	159.3 (1)
Te(2)—Ta(1)—Te(8)	78.3 (1)	Te(2)—Te(5)—Te(8)	76.7 (1)
Te(3)—Ta(1)—Te(8)	144.2 (1)	Te(3)—Te(6)—Te(9)	76.0 (1)
Te(4)—Ta(1)—Te(8)	105.6 (1)	Te(4)—Te(7)—Te(10)	75.7 (1)
Te(1)—Ta(1)—Te(9)	141.1 (1)	Te(5)—Te(8)—Te(9)	104.2 (1)
Te(2)—Ta(1)—Te(9)	105.6 (1)	Te(5)—Te(8)—Te(10)	156.3 (1)
Te(3)—Ta(1)—Te(9)	77.8 (1)	Te(9)—Te(8)—Te(10)	60.3 (1)
Te(4)—Ta(1)—Te(9)	145.5 (1)	Te(6)—Te(9)—Te(8)	158.9 (1)
Te(8)—Ta(1)—Te(9)	67.1 (1)	Te(6)—Te(9)—Te(10)	108.4 (1)
Te(1)—Ta(1)—Te(10)	138.6 (1)	Te(8)—Te(9)—Te(10)	60.3 (1)
Te(2)—Ta(1)—Te(10)	145.1 (1)	Te(7)—Te(10)—Te(8)	103.1 (1)
Te(3)—Ta(1)—Te(10)	106.4 (1)	Te(7)—Te(10)—Te(9)	155.8 (2)
Te(4)—Ta(1)—Te(10)	78.0 (1)	Te(8)—Te(10)—Te(9)	59.5 (1)
Te(8)—Ta(1)—Te(10)	67.6 (1)		

The structure was solved by direct methods and refined by full-matrix least squares. The Ta, Te and P atoms were refined anisotropically, while phenyl rings were treated as rigid groups (C—C 1.395  $\text{\AA}$ ) and refined isotropically. Atoms representing a badly disordered DMF solvent molecule were refined with a group displacement parameter.

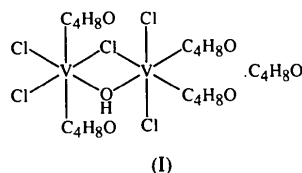
Data collection: *P3/P4-PC Diffractometer Control Software* (Siemens, 1976). Cell refinement: *P3/P4-PC Diffractometer Control Software*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Ziegler–Natta-type catalysts (Kahn, 1993; Poli & Torralba, 1993; Shaik, Hoffmann, Fisel & Summerville, 1980; Sobota *et al.*, 1995). Only a few examples of low-valent divanadium complexes are known, however, because of the lack of a useful synthetic method. Recently, Sobota and co-workers reported the synthesis, molecular structures and polymerization reactivity of two divanadium(III) complexes having two Cl bridges (Sobota, Ejfler, Szafert, Szczegot & Sawka-Dobrowolska, 1993; Sobota *et al.*, 1995). We obtained the asymmetric divanadium(III) complex having Cl and OH bridges from the reaction of  $[VCl_3(thf)_3]$  with  $NaCPh_3$  under an argon atmosphere. The structure of this complex, (I), was determined.



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## A Divanadium(III) Complex Bridged by $Cl^-$ and $OH^-$ Anions

MITSURU KONDO, TOMOO OHRUI, SATOSHI KAWATA AND SUSUMU KITAGAWA\*

Department of Chemistry, Faculty of Science, Tokyo Metropolitan University, 1-1 Minamiohsawa Hachioji, Tokyo 192-03, Japan. E-mail: kitagawa-susumu@c.metro-u.ac.jp

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### Abstract

The crystal structure of the complex  $\mu$ -chloro- $\mu$ -hydroxo-bis[dichlorobis(tetrahydrofuran-*O*)vanadium(III)] tetrahydrofuran solvate,  $[V_2(\mu-Cl)(\mu-OH)Cl_4(thf)_4] \cdot thf$  (where thf is tetrahydrofuran,  $C_4H_8O$ ), was determined. It was obtained from a tetrahydrofuran solution containing  $[VCl_3(thf)_3]$  and  $NaCPh_3$ . The two octahedral vanadium(III) centers are bridged by a  $Cl^-$  and an  $OH^-$  anion, with a  $V \cdots V$  separation of 3.281 (1) Å. One vanadium(III) center has two axial Cl and two equatorial tetrahydrofuran ligands, while the other vanadium(III) center has two axial tetrahydrofuran and two equatorial Cl ligands. A tetrahydrofuran molecule which is contained as crystalline solvent is involved in hydrogen bonding with the bridging  $OH^-$  anion.

### Comment

Dinuclear metal complexes bridged by halogen atoms or hydroxy groups have been extensively studied with regard to metal–metal magnetic interactions, stereoisomerism and stereoselective reactivity, and their use as

An *ORTEP*II (Johnson, 1976) drawing of (I) is shown in Fig. 1. Two vanadium(III) centers based on the octahedron are linked by Cl and OH bridges. The V(1) atom has two axial Cl atoms and two equatorial tetrahydrofuran molecules, while V(2) has two axial tetrahydrofuran molecules and two equatorial Cl atoms. Thus, the configurations of both vanadium(III) centers are different. The V(1)—O(2) and V(1)—O(3) bond distances [2.024 (4) and 2.030 (4) Å, respectively] are shorter than the V(2)—O(4) and V(2)—O(5) bond distances [2.088 (4) and 2.100 (4) Å, respectively], indicating that axial tetrahydrofuran ligation results in a stronger V—O bond compared with equatorial ligation.

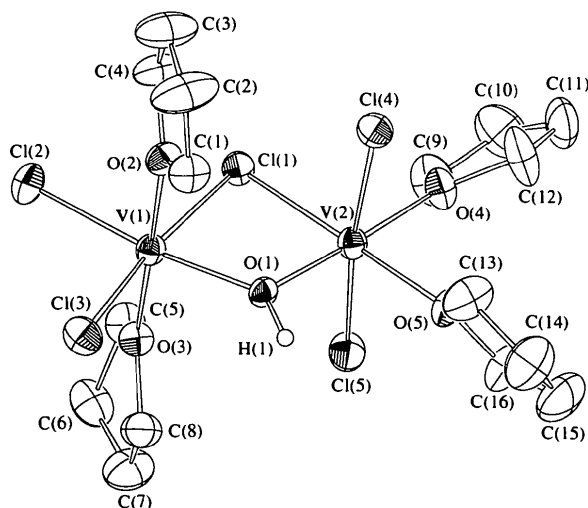


Fig. 1. An *ORTEP*II view of (I) with the atom-numbering scheme and ellipsoids plotted at the 30% probability level. H atoms of the tetrahydrofuran ligands have been omitted for clarity and the tetrahydrofuran solvate molecule is not shown.