

- BERTAUT, E. F., BLUM, P. & SAGNIERES, A. (1959). *Acta Cryst.* **12**, 149–159.
- COLVILLE, A. (1970). *Acta Cryst.* **B26**, 1469–1473.
- COTTON, F. A. & WILKINSON, G. (1988). *Advanced Inorganic Chemistry*, 5th ed. New York: John Wiley.
- GELLER, S., GRANT, R. W. & GONSER, V. (1969). *Prog. Solid State Chem.* **5**, 1–26.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SMITH, D. K. (1962). *Acta Cryst.* **15**, 1146–1152.
- VAUGHNEY, J. T., WILEY, J. B. & POEPELMEIER, K. R. (1990). *Z. Anorg. Allg. Chem.* In the press.
- VIDYASAGAR, K., GOPALAKRISHNAN, J. & RAO, C. N. R. (1984). *Inorg. Chem.* **23**, 1206–1210.
- WEHE, D. J., BUSING, W. R. & LEVY, H. A. (1962). *ORABS*. Report ORNL-TM-229. Oak Ridge National Laboratory, Tennessee, USA.
- WELLS, A. F. (1984). *Structural Inorganic Chemistry*, 5th ed. Oxford: Clarendon Press.
- WILEY, J. B., SABAT, M., HWU, S.-J., POEPELMEIER, K. R., RELLER, A. & WILLIAMS, T. (1990). *J. Solid State Chem.* **87**, 250–260.

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## Structure of $[\text{VF}_3(\text{H}_2\text{O})_3]^*$

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**Abstract.** Triaquatrifluorovanadium(III),  $M_r = 161.98$ , trigonal,  $R3m$ ,  $a = 7.233$  (4),  $c = 8.329$  (5) Å,  $V = 377.3$  Å<sup>3</sup>,  $Z = 3$ ,  $D_m = 2.10$ ,  $D_x = 2.146$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 18.4$  cm<sup>-1</sup>,  $F(000) = 240$ ,  $T = 293$  K,  $R = 0.019$  for 218 unique observed reflections. Discrete complex octahedral molecules  $\text{VF}_3(\text{OH}_2)_3$  with an ordered facial arrangement of the two types of ligand and  $3m$  symmetry are linked to six other molecules by twelve strong O—H...F hydrogen bonds. As, in the rhombohedral axial setting,  $a_R = 5.015$  (3) Å and  $\alpha_R = 92.30$  (5)°, the distortion of the V-atom partial structure from the arrangement of lattice points in a primitive cubic lattice is only slight.

**Experimental.** Green single crystals were obtained by reaction of vanadium powder with 48% hydrofluoric acid at room temperature (Petersen, 1889). The experimental density is quoted from Fischer & Weiss (1964). The crystal used for data collection was a cube of 0.125 mm edge length. Data were collected on a Siemens *AED2* diffractometer with graphite monochromator. The lattice parameters were determined by a least-squares analysis of 30 selected reflections in the range  $19 < 2\theta < 35^\circ$  and agreed within the mutual experimental error with those reported earlier (Fischer & Weiss, 1964). Reflection conditions  $hkl$  for  $-h + k + l = 3n$  proved the  $R$  centering. Intensities were measured using a variable

$\omega$ - $\theta$  scan. Three standard reflections monitored every 2 h were constant within 3%. No corrections for absorption were applied. 1147 reflections were measured with  $\sin\theta_{\text{max}}/\lambda < 0.805$  Å<sup>-1</sup> in the index range  $-11 \leq (h,k) \leq 11$ ,  $0 \leq l \leq 13$ . The reflections were averaged to yield 230 independent data, internal  $R = 0.037$ . Twelve reflections with  $I < 1.96\sigma(I)$  were treated as unobserved.

The crystal structure was solved by the heavy-atom method. Least-squares refinement was carried out only with the observed reflections using  $F$  magnitudes weighted by  $w = [\sigma^2(F)]^{-1}$ . The noncentrosymmetric space group had been proved before by piezoelectricity measurements (Fischer & Weiss, 1964). The H atom was located from a difference electron density map. Atomic scattering factors for the neutral atoms and terms for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations and drawings were performed using the Nicolet *SHELXTL* program system (Sheldrick, 1984) on a Data General Eclipse S/140 computer.

With 19 free parameters, *i.e.* the scale factor, coordinates of all atoms, an isotropic displacement

Table 1. Atomic coordinates and isotropic displacement parameters with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^*/U_{\text{H}}(\text{Å}^2)$
V	0.0	0.0	0.0	0.0176 (1)
F	0.1291 (4)	2 <i>x</i>	0.1271 (4)	0.034 (1)
O	2 <i>y</i>	0.1294 (4)	-0.1377 (5)	0.025 (1)
H	0.311 (4)	0.232 (3)	-0.159 (3)	0.047 (7)

$$* U_{\text{eq}} = \frac{1}{3}(U_{11}a^*a^2 + U_{22}b^*c^2 \text{bccos } \gamma + \dots)$$

\* Part 27 of the series Fluorides and Fluoro Acids. For part 26 see Mootz & Bartmann (1991).

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Table 2. Interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses

V—F	1.933 (4)	F—V—F <sup>ii</sup>	92.9 (1)
V—O	1.985 (4)	F—V—O	88.5 (1)
O—H	0.66 (2)	O—V—F <sup>ii</sup>	177.9 (2)
H...F <sup>i</sup>	1.96 (2)	O—V—O <sup>ii</sup>	90.0 (2)
O...F <sup>i</sup>	2.620 (3)	H—O—H <sup>iii</sup>	112 (5)
		O—H...F <sup>i</sup>	176 (3)

Symmetry code: (i)  $x + \frac{1}{3}, x - y + \frac{2}{3}, z - \frac{1}{3}$ ; (ii)  $-y, -x, z$ ; (iii)  $x, x - y, z$ .

parameter for the H atom and anisotropic ones for the non-H atoms, the refinement resulted in  $R = 0.019$ ,  $wR = 0.017$  and  $S = 1.077$  with  $\Delta/\sigma < 0.004$  in the last cycle. A final difference map yielded  $\Delta\rho_{\min}$  and  $\Delta\rho_{\max}$  values of  $-0.34$  and  $0.36 \text{ e \AA}^{-3}$ , respectively. The atomic parameters are listed in Table 1.\* The polarity of the crystal specimen as defined by these was slightly preferred in the refinement, the inverted one yielding  $R = 0.020$ ,  $wR = 0.018$  and  $S = 1.150$ . Table 2 contains interatomic distances and angles and Fig. 1 shows the complex octahedral molecule and its complete hydrogen-bonding environment in the crystal.

**Related literature.** Related compounds  $MF_3(OH_2)_3$  have been reported to crystallize either in a rhombohedral ( $R\bar{3}m$ ) structure with ligand and orientational disorder of discrete complex octahedral molecules or in a tetragonal ( $P4/n$ ) structure with chains of edge-sharing octahedra, bridged by hydrogen-bonded water molecules (Herbstein, Kapon & Reisner, 1985). A representative of the

\* Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53889 (3 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Tetracarbonyl- $\mu$ -(diphenylphosphanido)-di- $\mu$ -iodo-iodo(iododiphenylphosphine)-dirhenium(II)(*Re-Re*) Chloroform Solvate

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**Abstract.**  $[\text{Re}_2\text{I}_3\{\text{P}(\text{C}_6\text{H}_5)_2\}(\text{CO})_4\{\text{P}(\text{C}_6\text{H}_5)_2\text{I}\}]\cdot\text{CHCl}_3$ ,  $M_r = 1481.1$ , monoclinic,  $P2_1$ ,  $a = 11.823$  (3),  $b = 13.641$  (2),  $c = 12.150$  (3) Å,  $\beta = 103.56$  (2)°,  $V = 1904.8$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.583 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) =$

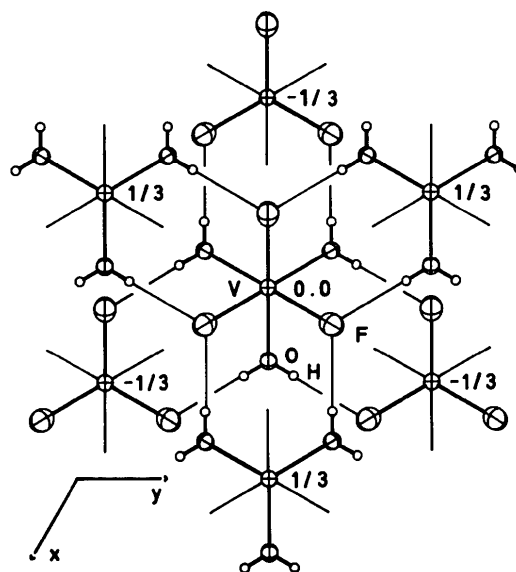


Fig. 1. The complex octahedral molecule  $\text{VF}_3(\text{OH}_2)_3$  with all intermolecular hydrogen bonds  $\text{O—H}\cdots\text{F}$  and  $\text{F}\cdots\text{H—O}$ . Projection down the  $c$  axis,  $z$  coordinates of V atoms as noted, 50% probability ellipsoids.

latter is also another form of  $\text{VF}_3\cdot 3\text{H}_2\text{O}$  itself (Maak, Eckerlin & Rabenau, 1961).

### References

- FISCHER, J. & WEISS, R. (1964). *Bull. Soc. Chim. Fr.* pp. 473.  
 HERBSTEIN, F. H., KAPON, M. & REISNER, G. M. (1985). *Z. Kristallogr.* **171**, 209–224.  
 MAAK, I., ECKERLIN, P. & RABENAU, A. (1961). *Naturwissenschaften*, **48**, 218.  
 MOOTZ, D. & BARTMANN, K. (1991). *Z. Anorg. Allg. Chem.* **592**, 171–178.  
 PETERSEN, E. (1889). *J. Prakt. Chem.* **40**, 44–52.  
 SHELDRICK, G. M. (1984). *SHELXTL Structure Determination System*. Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.

$0.71073$  Å,  $\mu = 98.9 \text{ cm}^{-1}$ ,  $F(000) = 1310$ ,  $T = 298$  (1) K,  $R = 0.027$ ,  $wR = 0.025$  for 2593 unique intensities ( $F > 4\sigma F$ ). The molecule is a 34 valence electron cluster with two face-sharing octahedrally