Inorganic Chemistry
including bioinorganic chemistry

Inorg. Chem., 1998, 37(21), 5439-5451, DOI:10.1021/ic980410c

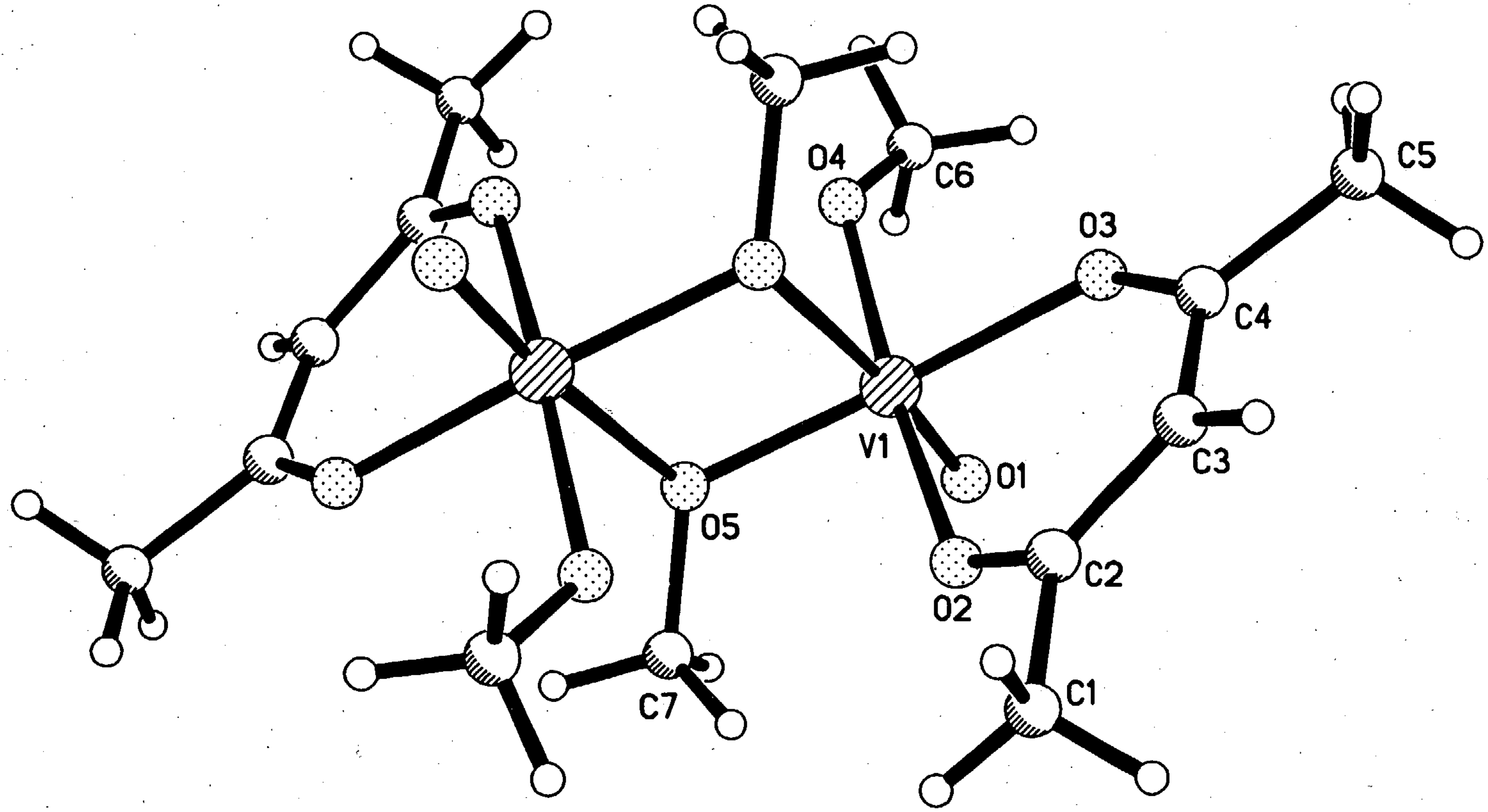

O4
C6
C5
O3
C4
C3
V1
O1
O5
C2
O2
C1
C7

Table 1. Crystal data and structure refinement for 1.

Identification code	dcccd5m
Empirical formula	$C_{14}H_{26}O_{10}V_2$
Formula weight	456.23
Temperature	159(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	*a* = 7.8668(5) Å alpha = 90°
	b = 15.1037(9) Å beta = 106.1500(10)°
	c = 8.5879(5) Å gamma = 90°
Volume, Z	980.13(10) $Å^3$, 2
Density (calculated)	1.546 Mg/m^3
Absorption coefficient	1.000 mm^{-1}
F(000)	472
Crystal size	0.09 x 0.26 x 0.38 mm
θ range for data collection	2.70 to 23.30°
Limiting indices	$-8 \leq h \leq 8$, $-15 \leq k \leq 16$, $-9 \leq l \leq 6$
Reflections collected	4331
Independent reflections	1413 (R_{int} = 0.0441)
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1413 / 0 / 119
Goodness-of-fit on F^2	0.757
Final R indices [$I>2\sigma(I)$]	R1 = 0.0395, wR2 = 0.1154
R indices (all data)	R1 = 0.0435, wR2 = 0.1213
Extinction coefficient	0.0000(13)
Largest diff. peak and hole	0.528 and −0.436 $eÅ^{-3}$

Table 3. Bond lengths [Å] and angles [°] for 1.

V(1)-O(1)	1.598(3)	V(1)-O(4)	1.783(2)
V(1)-O(5)	1.870(2)	V(1)-O(3)	2.001(3)
V(1)-O(2)	2.025(2)	V(1)-O(5)#1	2.254(2)
O(2)-C(2)	1.273(4)	O(3)-C(4)	1.277(5)
O(4)-C(6)	1.409(5)	O(5)-C(7)	1.433(4)
O(5)-V(1)#1	2.254(2)	C(1)-C(2)	1.498(5)
C(2)-C(3)	1.401(6)	C(3)-C(4)	1.384(6)
C(4)-C(5)	1.503(5)		
O(1)-V(1)-O(4)	99.63(13)	O(1)-V(1)-O(5)	100.24(12)
O(4)-V(1)-O(5)	101.23(11)	O(1)-V(1)-O(3)	97.82(12)
O(4)-V(1)-O(3)	87.16(11)	O(5)-V(1)-O(3)	158.45(11)
O(1)-V(1)-O(2)	96.03(12)	O(4)-V(1)-O(2)	162.20(11)
O(5)-V(1)-O(2)	84.15(10)	O(3)-V(1)-O(2)	82.35(10)
O(1)-V(1)-O(5)#1	174.78(11)	O(4)-V(1)-O(5)#1	83.60(10)
O(5)-V(1)-O(5)#1	75.03(10)	O(3)-V(1)-O(5)#1	86.39(9)
O(2)-V(1)-O(5)#1	81.43(9)	C(2)-O(2)-V(1)	131.4(2)
C(4)-O(3)-V(1)	132.3(2)	C(6)-O(4)-V(1)	127.4(2)
C(7)-O(5)-V(1)	120.7(2)	C(7)-O(5)-V(1)#1	121.4(2)
V(1)-O(5)-V(1)#1	104.97(10)	O(2)-C(2)-C(3)	122.8(3)
O(2)-C(2)-C(1)	116.4(3)	C(3)-C(2)-C(1)	120.8(3)
C(4)-C(3)-C(2)	121.9(3)	O(3)-C(4)-C(3)	124.4(3)
O(3)-C(4)-C(5)	115.1(3)	C(3)-C(4)-C(5)	120.4(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y+1,-z+2

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 1.

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
V(1)	20(1)	19(1)	18(1)	0(1)	6(1)	-1(1)
O(1)	22(2)	29(2)	28(2)	-4(1)	5(1)	-2(1)
O(2)	28(1)	20(1)	28(1)	3(1)	12(1)	0(1)
O(3)	30(1)	25(2)	25(1)	-5(1)	8(1)	-2(1)
O(4)	27(1)	29(2)	15(1)	2(1)	5(1)	-1(1)
O(5)	18(1)	19(1)	18(1)	0(1)	8(1)	2(1)
C(1)	38(2)	27(2)	33(2)	7(2)	10(2)	2(2)
C(2)	17(2)	20(2)	31(2)	3(2)	4(2)	-5(2)
C(3)	27(2)	21(2)	35(2)	-1(2)	2(2)	4(2)
C(4)	18(2)	22(2)	36(2)	-6(2)	4(2)	0(2)
C(5)	36(2)	32(2)	35(2)	-12(2)	3(2)	4(2)
C(6)	40(3)	57(3)	20(2)	2(2)	3(2)	-2(2)
C(7)	26(2)	26(2)	23(2)	0(2)	12(2)	2(2)

 5

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 1.

	x	y	z	U(eq)
H(1A)	9274(6)	3236(3)	13440(5)	49
H(1B)	10980(6)	2681(3)	13357(5)	49
H(1C)	9090(6)	2207(3)	12982(5)	49
H(3A)	10450(5)	1880(3)	10451(5)	35
H(5A)	9354(6)	2130(3)	6133(5)	53
H(5B)	8894(6)	1389(3)	7283(5)	53
H(5C)	10904(6)	1685(3)	7527(5)	53
H(6A)	8441(6)	5376(3)	4744(5)	60
H(6B)	6697(6)	5326(3)	5354(5)	60
H(6C)	7610(6)	4435(3)	4977(5)	60
H(7A)	7804(5)	6072(2)	11809(5)	36
H(7B)	7435(5)	5029(2)	11775(5)	36
H(7C)	6258(5)	5651(2)	10378(5)	36

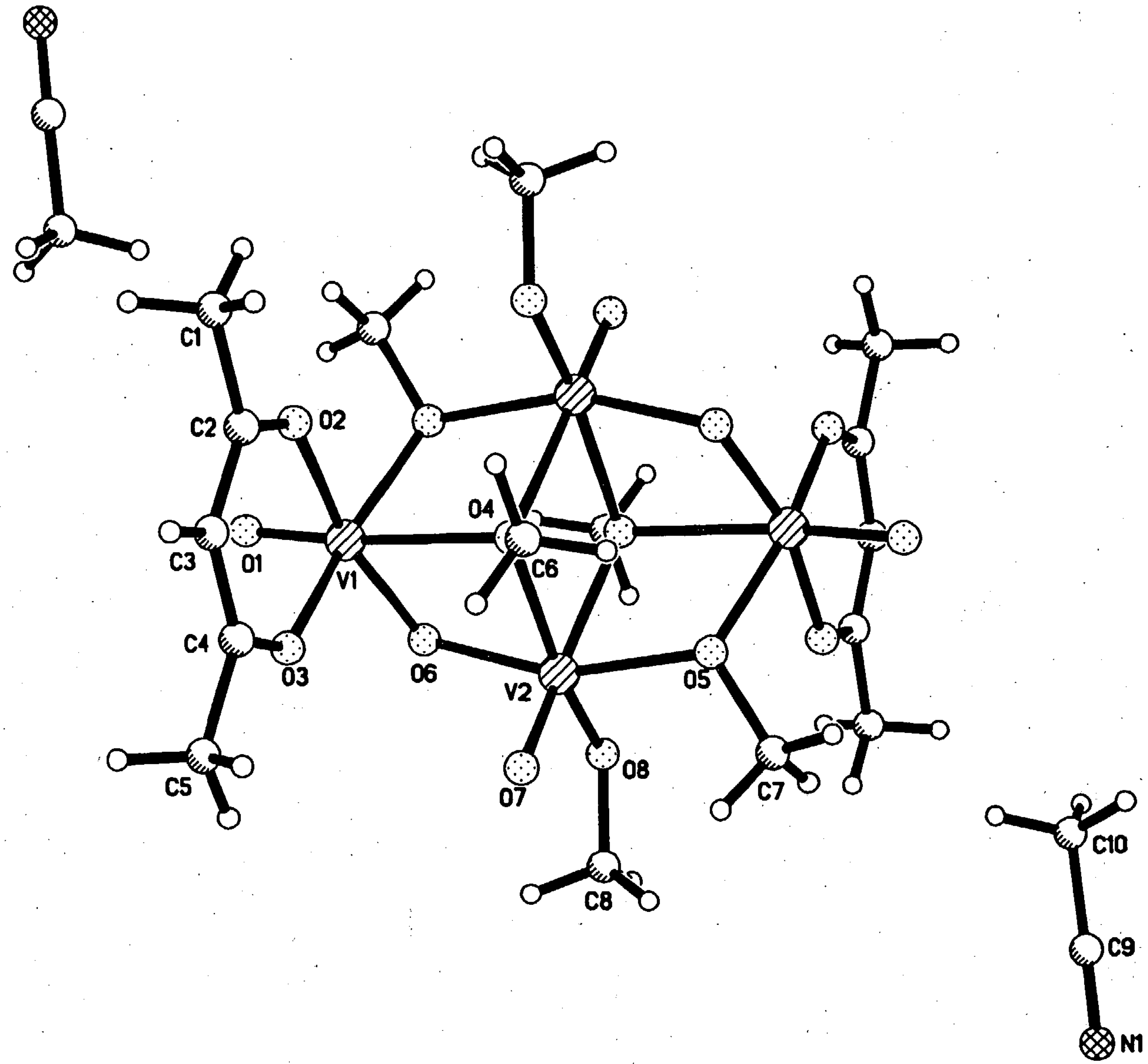

C1
C2
O2
C3
O1
V1
O4
C6
C4
O3
O6
V2
O5
C5
O7
O8
C7
C10
C8
C9
N1

Table 1. Crystal data and structure refinement for 1.

Identification code	dc20
Empirical formula	$C_{20}H_{38}N_2O_{16}V_4$
Formula weight	766.28
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	*a* = 8.531(2) Å alpha = 90°
	b = 14.703(3) Å beta = 95.95(2)°
	c = 12.574(2) Å gamma = 90°
Volume, Z	1568.7(5) $Å^3$, 2
Density (calculated)	1.622 Mg/m^3
Absorption coefficient	1.226 mm^{-1}
F(000)	784
Crystal size	0.20 x 0.14 x 0.12 mm
θ range for data collection	2.14 to 22.54°
Limiting indices	$-9 \le h \le 9$, $0 \le k \le 15$, $0 \le l \le 13$
Reflections collected	2170
Independent reflections	2065 (R_{int} = 0.0335)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2065 / 0 / 190
Goodness-of-fit on F^2	1.030
Final R indices [$I>2\sigma(I)$]	R1 = 0.0516, wR2 = 0.1062
R indices (all data)	R1 = 0.0906, wR2 = 0.1267
Largest diff. peak and hole	0.413 and -0.390 $eÅ^{-3}$

Table 3. Bond lengths [Å] and angles [°] for 1.

V(1)-O(1)	1.599(4)	V(1)-O(6)	1.747(4)
V(1)-O(5)#1	1.950(4)	V(1)-O(3)	1.964(5)
V(1)-O(2)	2.015(4)	V(1)-O(4)	2.317(4)
V(1)-V(2)	3.081(2)	V(2)-O(7)	1.597(4)
V(2)-O(8)	1.760(4)	V(2)-O(6)	1.911(4)
V(2)-O(5)	2.004(4)	V(2)-O(4)	2.059(4)
V(2)-O(4)#1	2.193(4)	O(2)-C(2)	1.287(8)
O(3)-C(4)	1.276(8)	O(4)-C(6)	1.425(7)
O(4)-V(2)#1	2.193(4)	O(5)-C(7)	1.450(7)
O(5)-V(1)#1	1.950(4)	O(8)-C(8)	1.431(8)
C(1)-C(2)	1.493(10)	C(2)-C(3)	1.382(9)
C(3)-C(4)	1.386(9)	C(4)-C(5)	1.495(10)
C(9)-N(1)	1.143(11)	C(9)-C(10)	1.437(12)
O(1)-V(1)-O(6)	102.8(2)	O(1)-V(1)-O(5)#1	97.1(2)
O(6)-V(1)-O(5)#1	94.7(2)	O(1)-V(1)-O(3)	101.7(2)
O(6)-V(1)-O(3)	91.5(2)	O(5)#1-V(1)-O(3)	158.3(2)
O(1)-V(1)-O(2)	97.5(2)	O(6)-V(1)-O(2)	159.7(2)
O(5)#1-V(1)-O(2)	83.2(2)	O(3)-V(1)-O(2)	83.8(2)
O(1)-V(1)-O(4)	171.2(2)	O(6)-V(1)-O(4)	75.8(2)
O(5)#1-V(1)-O(4)	74.5(2)	O(3)-V(1)-O(4)	87.0(2)
O(2)-V(1)-O(4)	84.2(2)	O(1)-V(1)-V(2)	137.1(2)
O(6)-V(1)-V(2)	34.30(14)	O(5)#1-V(1)-V(2)	88.69(12)
O(3)-V(1)-V(2)	84.75(13)	O(2)-V(1)-V(2)	125.37(13)
O(4)-V(1)-V(2)	41.94(10)	O(7)-V(2)-O(8)	100.4(2)
O(7)-V(2)-O(6)	101.0(2)	O(8)-V(2)-O(6)	94.8(2)
O(7)-V(2)-O(5)	94.7(2)	O(8)-V(2)-O(5)	95.2(2)
O(6)-V(2)-O(5)	159.4(2)	O(7)-V(2)-O(4)	96.5(2)
O(8)-V(2)-O(4)	162.8(2)	O(6)-V(2)-O(4)	79.3(2)
O(5)-V(2)-O(4)	85.9(2)	O(7)-V(2)-O(4)#1	166.7(2)
O(8)-V(2)-O(4)#1	90.4(2)	O(6)-V(2)-O(4)#1	85.7(2)
O(5)-V(2)-O(4)#1	76.4(2)	O(4)-V(2)-O(4)#1	73.2(2)
O(7)-V(2)-V(1)	97.2(2)	O(8)-V(2)-V(1)	125.5(2)
O(6)-V(2)-V(1)	31.01(12)	O(5)-V(2)-V(1)	134.08(13)
O(4)-V(2)-V(1)	48.75(11)	O(4)#1-V(2)-V(1)	82.53(11)
C(2)-O(2)-V(1)	132.5(4)	C(4)-O(3)-V(1)	133.4(4)
C(6)-O(4)-V(2)	118.3(3)	C(6)-O(4)-V(2)#1	117.9(4)
V(2)-O(4)-V(2)#1	106.8(2)	C(6)-O(4)-V(1)	124.3(4)
V(2)-O(4)-V(1)	89.3(2)	V(2)#1-O(4)-V(1)	94.8(2)
C(7)-O(5)-V(1)#1	119.8(4)	C(7)-O(5)-V(2)	116.9(4)
V(1)#1-O(5)-V(2)	114.3(2)	V(1)-O(6)-V(2)	114.7(2)
C(8)-O(8)-V(2)	130.2(4)	O(2)-C(2)-C(3)	123.2(6)
O(2)-C(2)-C(1)	115.8(6)	C(3)-C(2)-C(1)	121.0(6)
C(2)-C(3)-C(4)	122.7(7)	O(3)-C(4)-C(3)	124.4(6)
O(3)-C(4)-C(5)	114.2(6)	C(3)-C(4)-C(5)	121.4(6)
N(1)-C(9)-C(10)	178.8(10)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1

Table 4. Anisotropic displacement parameters [Å^2 x 10^3] for 1.

The anisotropic displacement factor exponent takes the form:
$-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + $2hka^*b^*U_{12}$]

	U11	U22	U33	U23	U13	U12
V(1)	23(1)	21(1)	18(1)	2(1)	0(1)	-4(1)
V(2)	21(1)	22(1)	15(1)	0(1)	3(1)	2(1)
O(1)	32(3)	35(3)	23(3)	6(2)	2(2)	-11(2)
O(2)	29(3)	32(3)	17(3)	1(2)	7(2)	-2(2)
O(3)	40(3)	24(3)	20(3)	6(2)	2(2)	5(2)
O(4)	15(2)	21(2)	18(2)	6(2)	-3(2)	1(2)
O(5)	16(2)	28(3)	24(3)	6(2)	6(2)	0(2)
O(6)	27(2)	22(3)	19(2)	0(2)	-3(2)	-3(2)
O(7)	25(3)	22(3)	30(3)	1(2)	5(2)	5(2)
O(8)	31(3)	33(3)	14(2)	-6(2)	6(2)	5(2)
C(1)	48(5)	57(5)	24(4)	-4(4)	5(4)	-3(5)
C(2)	22(4)	39(5)	18(4)	5(3)	-4(3)	-9(3)
C(3)	37(4)	30(4)	24(4)	9(3)	2(3)	-4(4)
C(4)	28(4)	27(4)	24(4)	9(3)	-1(3)	-5(3)
C(5)	64(6)	36(5)	32(4)	7(4)	0(4)	14(5)
C(6)	17(4)	38(4)	19(4)	3(3)	-3(3)	-3(3)
C(7)	31(4)	44(5)	50(5)	3(4)	19(4)	4(4)
C(8)	56(5)	56(6)	26(4)	-11(4)	9(4)	14(5)
C(9)	48(6)	37(5)	74(7)	21(5)	-9(5)	-3(5)
C(10)	40(5)	56(6)	58(6)	17(5)	1(4)	5(5)
N(1)	81(7)	62(6)	117(9)	-2(6)	19(6)	13(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 1.

	x	y	z	U(eq)
H(1A)	-1514(9)	4237(6)	9151(6)	80
H(1B)	224(9)	3879(6)	9535(6)	80
H(1C)	-1265(9)	3232(6)	9626(6)	80
H(3A)	775(8)	2301(5)	8554(6)	80
H(5A)	1415(10)	1297(5)	6034(6)	80
H(5B)	1038(10)	915(5)	7171(6)	80
H(5C)	2522(10)	1568(5)	7092(6)	80
H(6A)	1649(7)	4584(5)	7252(5)	80
H(6B)	2761(7)	4799(5)	6333(5)	80
H(6C)	2252(7)	3773(5)	6549(5)	80
H(7A)	4069(8)	4276(5)	3845(6)	80
H(7B)	4739(8)	5277(5)	4105(6)	80
H(7C)	3667(8)	5083(5)	3007(6)	80
H(8A)	671(9)	3725(6)	1333(6)	80
H(8B)	720(9)	2931(6)	2207(6)	80
H(8C)	2218(9)	3579(6)	2150(6)	80
H(10A)	4584(9)	6889(6)	852(7)	80
H(10B)	6069(9)	6909(6)	1740(7)	80
H(10C)	4776(9)	6122(6)	1760(7)	80

Table 1. Crystal data and structure refinement for 1.

Identification code	dcccd4m
Empirical formula	$C_{16}H_{32}O_{16}V_4$
Formula weight	684.18
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 8.5100(8) Å alpha = 110.7610(10)°
	b = 8.9714(8) Å beta = 103.1040(10)°
	c = 10.3708(10) Å gamma = 100.1550(10)°
Volume, Z	691.85(11) $Å^3$, 1
Density (calculated)	1.642 Mg/m^3
Absorption coefficient	1.377 mm^{-1}
F(000)	348
Crystal size	0.28 x 0.20 x 0.20 mm
θ range for data collection	2.21 to 23.29°
Limiting indices	$-7 \le h \le 9$, $-9 \le k \le 9$, $-11 \le l \le 11$
Reflections collected	3143
Independent reflections	1991 (R_{int} = 0.0167)
Absorption correction	SADABS (G.Sheldrick, private comm.)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1991 / 0 / 163
Goodness-of-fit on F^2	1.055
Final R indices [I>2σ(I)]	R1 = 0.0404, wR2 = 0.1000
R indices (all data)	R1 = 0.0491, wR2 = 0.1047
Largest diff. peak and hole	0.360 and -0.377 $eÅ^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 1. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
V(1)	6340(1)	4397(1)	12323(1)	46(1)
V(2)	4455(1)	2896(1)	8977(1)	43(1)
O(1)	6021(4)	4067(4)	13661(3)	64(1)
O(2)	8490(4)	6260(4)	13442(3)	55(1)
O(3)	7855(4)	3039(4)	11857(3)	58(1)
O(4)	3423(3)	4797(3)	9522(3)	38(1)
O(5)	4617(3)	3752(3)	7460(3)	43(1)
O(6)	4642(3)	2940(3)	10854(3)	48(1)
O(7)	2780(4)	1410(4)	8084(3)	60(1)
O(8)	6001(4)	1860(4)	8645(3)	57(1)
C(1)	11257(6)	7903(7)	14721(7)	83(2)
C(2)	10014(6)	6262(6)	13802(5)	55(1)
C(3)	10540(6)	4829(7)	13312(6)	63(1)
C(4)	9455(6)	3317(6)	12363(5)	56(1)
C(5)	10101(7)	1836(7)	11784(7)	84(2)
C(6)	1899(5)	4523(6)	9873(5)	52(1)
C(7)	4521(7)	2599(7)	6042(5)	70(1)
C(8)	5857(9)	129(7)	8087(7)	98(2)

Table 3. Bond lengths [Å] and angles [°] for 1.

V(1)-O(1)	1.588(3)	V(1)-O(6)	1.744(3)
V(1)-O(5)#1	1.941(3)	V(1)-O(3)	1.949(3)
V(1)-O(2)	2.018(3)	V(1)-O(4)#1	2.309(2)
V(2)-O(7)	1.588(3)	V(2)-O(8)	1.768(3)
V(2)-O(6)	1.903(3)	V(2)-O(5)	2.001(3)
V(2)-O(4)	2.017(3)	V(2)-O(4)#1	2.257(3)
O(2)-C(2)	1.265(5)	O(3)-C(4)	1.286(5)
O(4)-C(6)	1.428(4)	O(4)-V(2)#1	2.257(3)
O(4)-V(1)#1	2.309(2)	O(5)-C(7)	1.440(5)
O(5)-V(1)#1	1.941(3)	O(8)-C(8)	1.422(6)
C(1)-C(2)	1.481(7)	C(2)-C(3)	1.398(7)
C(3)-C(4)	1.365(7)	C(4)-C(5)	1.506(7)
O(1)-V(1)-O(6)	103.1(2)	O(1)-V(1)-O(5)#1	98.3(2)
O(6)-V(1)-O(5)#1	95.23(12)	O(1)-V(1)-O(3)	100.0(2)
O(6)-V(1)-O(3)	92.39(14)	O(5)#1-V(1)-O(3)	158.01(13)
O(1)-V(1)-O(2)	97.4(2)	O(6)-V(1)-O(2)	159.51(13)
O(5)#1-V(1)-O(2)	81.85(12)	O(3)-V(1)-O(2)	83.80(13)
O(1)-V(1)-O(4)#1	170.69(14)	O(6)-V(1)-O(4)#1	77.73(11)
O(5)#1-V(1)-O(4)#1	72.40(10)	O(3)-V(1)-O(4)#1	89.19(11)
O(2)-V(1)-O(4)#1	82.08(11)	O(7)-V(2)-O(8)	101.2(2)
O(7)-V(2)-O(6)	98.4(2)	O(8)-V(2)-O(6)	96.85(13)
O(7)-V(2)-O(5)	99.21(14)	O(8)-V(2)-O(5)	91.48(12)
O(6)-V(2)-O(5)	158.68(12)	O(7)-V(2)-O(4)	98.97(14)
O(8)-V(2)-O(4)	158.54(13)	O(6)-V(2)-O(4)	87.49(11)
O(5)-V(2)-O(4)	77.95(10)	O(7)-V(2)-O(4)#1	170.38(14)
O(8)-V(2)-O(4)#1	87.48(12)	O(6)-V(2)-O(4)#1	76.18(10)
O(5)-V(2)-O(4)#1	84.65(10)	O(4)-V(2)-O(4)#1	73.10(10)
C(2)-O(2)-V(1)	132.0(3)	C(4)-O(3)-V(1)	132.7(3)
C(6)-O(4)-V(2)	117.8(2)	C(6)-O(4)-V(2)#1	118.9(2)
V(2)-O(4)-V(2)#1	106.89(10)	C(6)-O(4)-V(1)#1	123.1(2)
V(2)-O(4)-V(1)#1	97.76(10)	V(2)#1-O(4)-V(1)#1	86.75(9)
C(7)-O(5)-V(1)#1	120.7(3)	C(7)-O(5)-V(2)	118.7(3)
V(1)#1-O(5)-V(2)	111.86(13)	V(1)-O(6)-V(2)	118.52(14)
C(8)-O(8)-V(2)	129.7(4)	O(2)-C(2)-C(3)	123.0(4)
O(2)-C(2)-C(1)	116.3(4)	C(3)-C(2)-C(1)	120.6(4)
C(4)-C(3)-C(2)	122.7(4)	O(3)-C(4)-C(3)	124.4(4)
O(3)-C(4)-C(5)	114.9(5)	C(3)-C(4)-C(5)	120.6(5)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+2

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 1.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + $2hka^*b^*U_{12}$]

	U11	U22	U33	U23	U13	U12
V(1)	47(1)	44(1)	53(1)	28(1)	14(1)	16(1)
V(2)	46(1)	33(1)	52(1)	20(1)	14(1)	14(1)
O(1)	76(2)	67(2)	60(2)	40(2)	17(2)	22(2)
O(2)	45(2)	50(2)	62(2)	21(2)	6(1)	14(1)
O(3)	60(2)	50(2)	69(2)	27(2)	13(2)	28(2)
O(4)	36(1)	38(2)	47(2)	22(1)	16(1)	13(1)
O(5)	47(2)	43(2)	47(2)	20(1)	18(1)	19(1)
O(6)	52(2)	40(2)	58(2)	31(1)	15(1)	11(1)
O(7)	62(2)	40(2)	65(2)	19(2)	7(2)	2(2)
O(8)	67(2)	45(2)	64(2)	22(2)	21(2)	30(2)
C(1)	51(3)	79(4)	97(4)	27(3)	7(3)	7(3)
C(2)	48(3)	65(3)	49(3)	27(2)	9(2)	13(2)
C(3)	47(3)	74(4)	71(3)	34(3)	14(2)	25(3)
C(4)	63(3)	66(3)	68(3)	45(3)	32(3)	37(3)
C(5)	89(4)	74(4)	118(5)	49(4)	49(4)	48(3)
C(6)	38(2)	63(3)	66(3)	33(2)	22(2)	15(2)
C(7)	93(4)	73(4)	60(3)	28(3)	40(3)	36(3)
C(8)	124(5)	50(3)	111(5)	19(3)	24(4)	50(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 1.

	x	y	z	U(eq)
H(1B)	10680(6)	8742(7)	14953(7)	124
H(1C)	11999(6)	8149(7)	14206(7)	124
H(1D)	11897(6)	7881(7)	15602(7)	124
H(3A)	11676(6)	4910(7)	13646(6)	75
H(5A)	9172(7)	882(7)	11135(7)	126
H(5B)	10686(7)	1622(7)	12577(7)	126
H(5C)	10855(7)	2060(7)	11270(7)	126
H(6A)	1544(5)	5518(6)	10111(5)	79
H(6B)	2086(5)	4232(6)	10690(5)	79
H(6C)	1043(5)	3635(6)	9055(5)	79
H(7B)	5053(7)	1768(7)	6138(5)	106
H(7C)	5083(7)	3184(7)	5584(5)	106
H(7D)	3363(7)	2076(7)	5460(5)	106
H(8A)	6929(9)	-36(7)	8035(7)	147
H(8B)	5048(9)	-416(7)	7133(7)	147
H(8C)	5497(9)	-327(7)	8715(7)	147

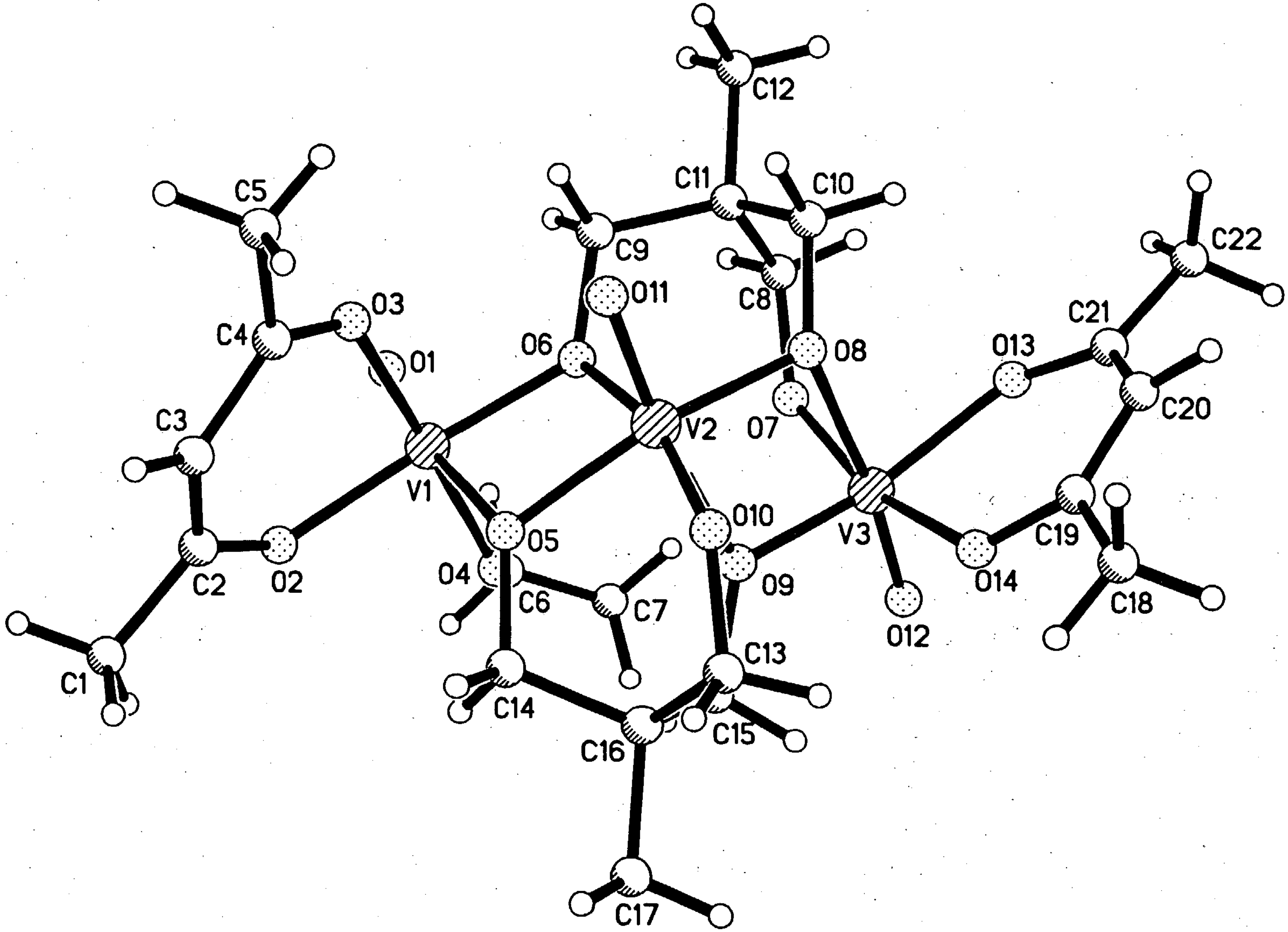

C12
C11
C10
C5
C9
C22
C8
O11
O3
C21
C4
O8
O13
O1
O6
C20
C3
V2
O7
V1
C19
O10
V3
O5
O9
O14
C2
O2
O4
C6
C7
C18
O12
C1
C13
C14
C15
C16
C17

Table 1. Crystal data and structure refinement for 1.

Identification code	dc22
Empirical formula	$C_{22}H_{37}O_{14}V_3$
Formula weight	678.34
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/n
Unit cell dimensions	a = 14.019(2) Å alpha = 90°
	b = 11.171(2) Å beta = 109.180(10)°
	c = 19.447(3) Å gamma = 90°
Volume, Z	2876.5(8) $Å^3$, 4
Density (calculated)	1.566 Mg/m^3
Absorption coefficient	1.019 mm^{-1}
F(000)	1400
Crystal size	0.24 x 0.16 x 0.04 mm
θ range for data collection	2.13 to 22.55°
Limiting indices	$-15 \le h \le 14$, $0 \le k \le 12$, $0 \le l \le 20$
Reflections collected	3908
Independent reflections	3780 (R_{int} = 0.0640)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3778 / 0 / 352
Goodness-of-fit on F^2	1.043
Final R indices [$I>2\sigma(I)$]	R1 = 0.0618, wR2 = 0.1187
R indices (all data)	R1 = 0.1410, wR2 = 0.1569
Largest diff. peak and hole	0.486 and -0.391 $eÅ^{-3}$

Table 3. Bond lengths [Å] and angles [°] for 1.

V(1)-O(1)	1.589(6)	V(1)-O(4)	1.821(6)
V(1)-O(6)	1.933(6)	V(1)-O(2)	1.952(6)
V(1)-O(3)	2.038(6)	V(1)-O(5)	2.157(6)
V(2)-O(11)	1.600(6)	V(2)-O(10)	1.781(5)
V(2)-O(8)	1.898(6)	V(2)-O(5)	1.937(5)
V(2)-O(6)	2.052(5)	V(2)-O(9)	2.184(6)
V(3)-O(12)	1.590(6)	V(3)-O(7)	1.808(6)
V(3)-O(9)	1.870(6)	V(3)-O(13)	1.984(6)
V(3)-O(14)	1.995(6)	V(3)-O(8)	2.253(6)
O(2)-C(2)	1.311(11)	O(3)-C(4)	1.253(11)
O(4)-C(6)	1.416(12)	O(5)-C(14)	1.435(9)
O(6)-C(9)	1.454(9)	O(7)-C(8)	1.444(10)
O(8)-C(10)	1.437(10)	O(9)-C(15)	1.427(9)
O(10)-C(13)	1.423(10)	O(13)-C(21)	1.282(9)
O(14)-C(19)	1.291(10)	C(1)-C(2)	1.490(13)
C(2)-C(3)	1.357(13)	C(3)-C(4)	1.423(13)
C(4)-C(5)	1.497(13)	C(6)-C(7)	1.36(2)
C(8)-C(11)	1.528(12)	C(9)-C(11)	1.501(12)
C(10)-C(11)	1.517(11)	C(11)-C(12)	1.528(11)
C(13)-C(16)	1.544(11)	C(14)-C(16)	1.520(11)
C(15)-C(16)	1.526(12)	C(16)-C(17)	1.539(11)
C(18)-C(19)	1.504(12)	C(19)-C(20)	1.381(12)
C(20)-C(21)	1.374(12)	C(21)-C(22)	1.500(12)
O(1)-V(1)-O(4)	97.6(3)	O(1)-V(1)-O(6)	98.1(3)
O(4)-V(1)-O(6)	96.7(3)	O(1)-V(1)-O(2)	102.1(3)
O(4)-V(1)-O(2)	91.9(3)	O(6)-V(1)-O(2)	156.7(3)
O(1)-V(1)-O(3)	93.2(3)	O(4)-V(1)-O(3)	169.1(3)
O(6)-V(1)-O(3)	83.1(3)	O(2)-V(1)-O(3)	84.5(3)
O(1)-V(1)-O(5)	170.3(3)	O(4)-V(1)-O(5)	87.5(3)
O(6)-V(1)-O(5)	73.0(2)	O(2)-V(1)-O(5)	85.8(2)
O(3)-V(1)-O(5)	82.0(2)	O(11)-V(2)-O(10)	99.5(3)
O(11)-V(2)-O(8)	96.1(3)	O(10)-V(2)-O(8)	105.1(3)
O(11)-V(2)-O(5)	107.2(3)	O(10)-V(2)-O(5)	90.5(2)
O(8)-V(2)-O(5)	149.4(2)	O(11)-V(2)-O(6)	92.6(3)
O(10)-V(2)-O(6)	163.6(3)	O(8)-V(2)-O(6)	84.3(2)
O(5)-V(2)-O(6)	75.4(2)	O(11)-V(2)-O(9)	169.7(3)
O(10)-V(2)-O(9)	86.7(2)	O(8)-V(2)-O(9)	74.2(2)
O(5)-V(2)-O(9)	80.8(2)	O(6)-V(2)-O(9)	83.0(2)
O(12)-V(3)-O(7)	100.5(3)	O(12)-V(3)-O(9)	98.4(3)
O(7)-V(3)-O(9)	98.3(3)	O(12)-V(3)-O(13)	101.7(3)
O(7)-V(3)-O(13)	86.4(3)	O(9)-V(3)-O(13)	158.2(3)
O(12)-V(3)-O(14)	96.4(3)	O(7)-V(3)-O(14)	162.0(3)
O(9)-V(3)-O(14)	85.3(2)	O(13)-V(3)-O(14)	84.1(2)
O(12)-V(3)-O(8)	170.8(3)	O(7)-V(3)-O(8)	84.2(2)
O(9)-V(3)-O(8)	73.1(2)	O(13)-V(3)-O(8)	86.3(2)
O(14)-V(3)-O(8)	79.9(2)	C(2)-O(2)-V(1)	129.5(6)
C(4)-O(3)-V(1)	129.1(6)	C(6)-O(4)-V(1)	126.7(6)
C(14)-O(5)-V(2)	120.8(5)	C(14)-O(5)-V(1)	126.5(5)
V(2)-O(5)-V(1)	103.5(2)	C(9)-O(6)-V(1)	123.4(5)
C(9)-O(6)-V(2)	118.9(5)	V(1)-O(6)-V(2)	107.7(3)
C(8)-O(7)-V(3)	131.5(5)	C(10)-O(8)-V(2)	120.0(5)
C(10)-O(8)-V(3)	118.8(5)	V(2)-O(8)-V(3)	104.4(2)
C(15)-O(9)-V(3)	126.6(5)	C(15)-O(9)-V(2)	120.5(5)

V(3)-O(9)-V(2)	108.1(2)	C(13)-O(10)-V(2)	127.1(5)
C(21)-O(13)-V(3)	131.7(6)	C(19)-O(14)-V(3)	130.7(6)
O(2)-C(2)-C(3)	124.9(9)	O(2)-C(2)-C(1)	113.0(9)
C(3)-C(2)-C(1)	122.1(9)	C(2)-C(3)-C(4)	122.8(9)
O(3)-C(4)-C(3)	122.8(9)	O(3)-C(4)-C(5)	116.4(9)
C(3)-C(4)-C(5)	120.9(9)	C(7)-C(6)-O(4)	116.6(11)
O(7)-C(8)-C(11)	115.9(7)	O(6)-C(9)-C(11)	112.4(7)
O(8)-C(10)-C(11)	112.0(7)	C(9)-C(11)-C(10)	111.5(7)
C(9)-C(11)-C(12)	107.3(7)	C(10)-C(11)-C(12)	107.5(7)
C(9)-C(11)-C(8)	111.8(7)	C(10)-C(11)-C(8)	111.0(7)
C(12)-C(11)-C(8)	107.5(7)	O(10)-C(13)-C(16)	111.4(7)
O(5)-C(14)-C(16)	113.6(7)	O(9)-C(15)-C(16)	110.3(6)
C(14)-C(16)-C(15)	110.9(7)	C(14)-C(16)-C(13)	111.7(7)
C(15)-C(16)-C(13)	108.4(7)	C(14)-C(16)-C(17)	109.1(7)
C(15)-C(16)-C(17)	109.0(7)	C(13)-C(16)-C(17)	107.7(7)
O(14)-C(19)-C(20)	123.3(8)	O(14)-C(19)-C(18)	113.4(8)
C(20)-C(19)-C(18)	123.3(8)	C(21)-C(20)-C(19)	123.1(8)
O(13)-C(21)-C(20)	123.9(8)	O(13)-C(21)-C(22)	114.2(8)
C(20)-C(21)-C(22)	122.0(8)		

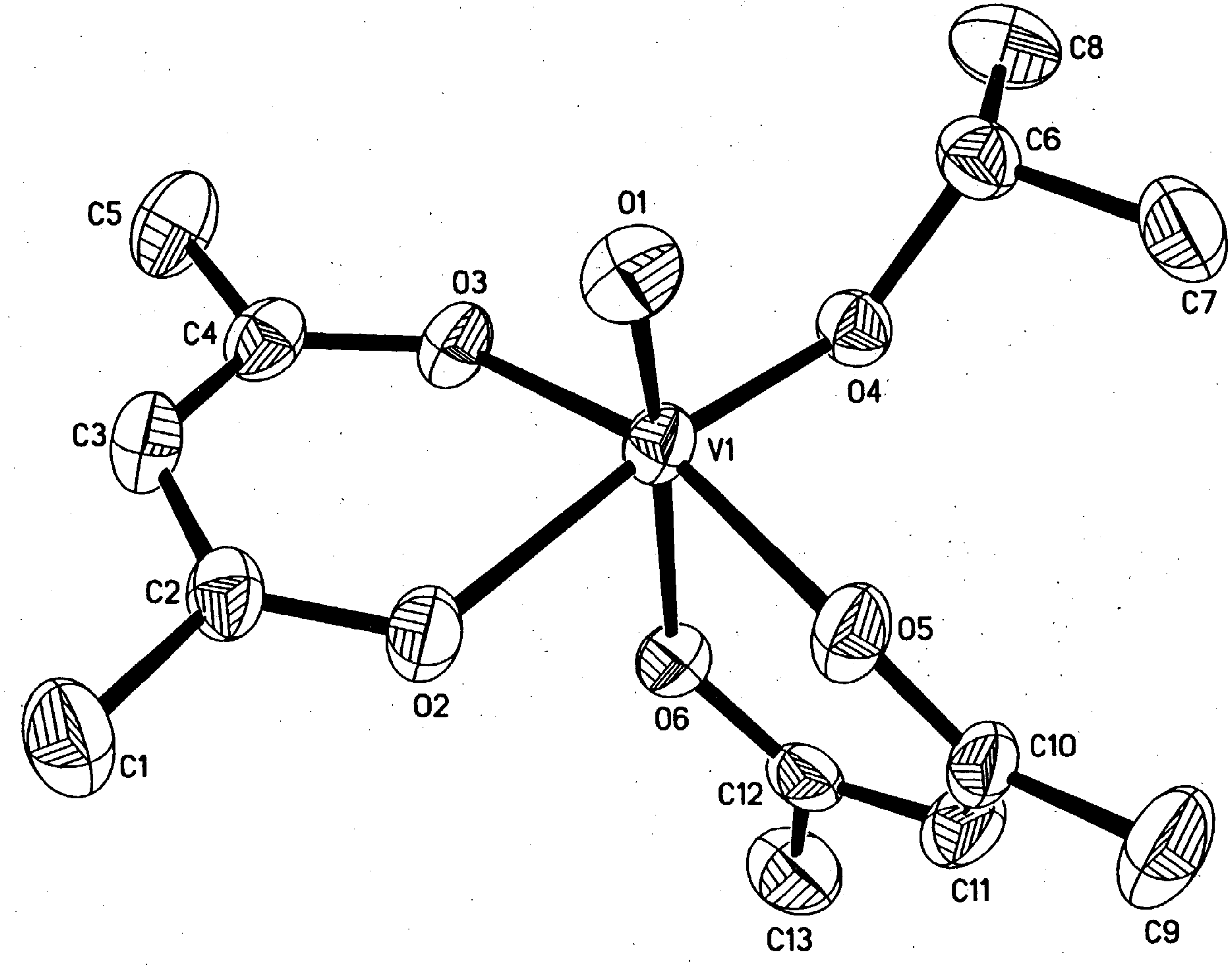
Symmetry transformations used to generate equivalent atoms:

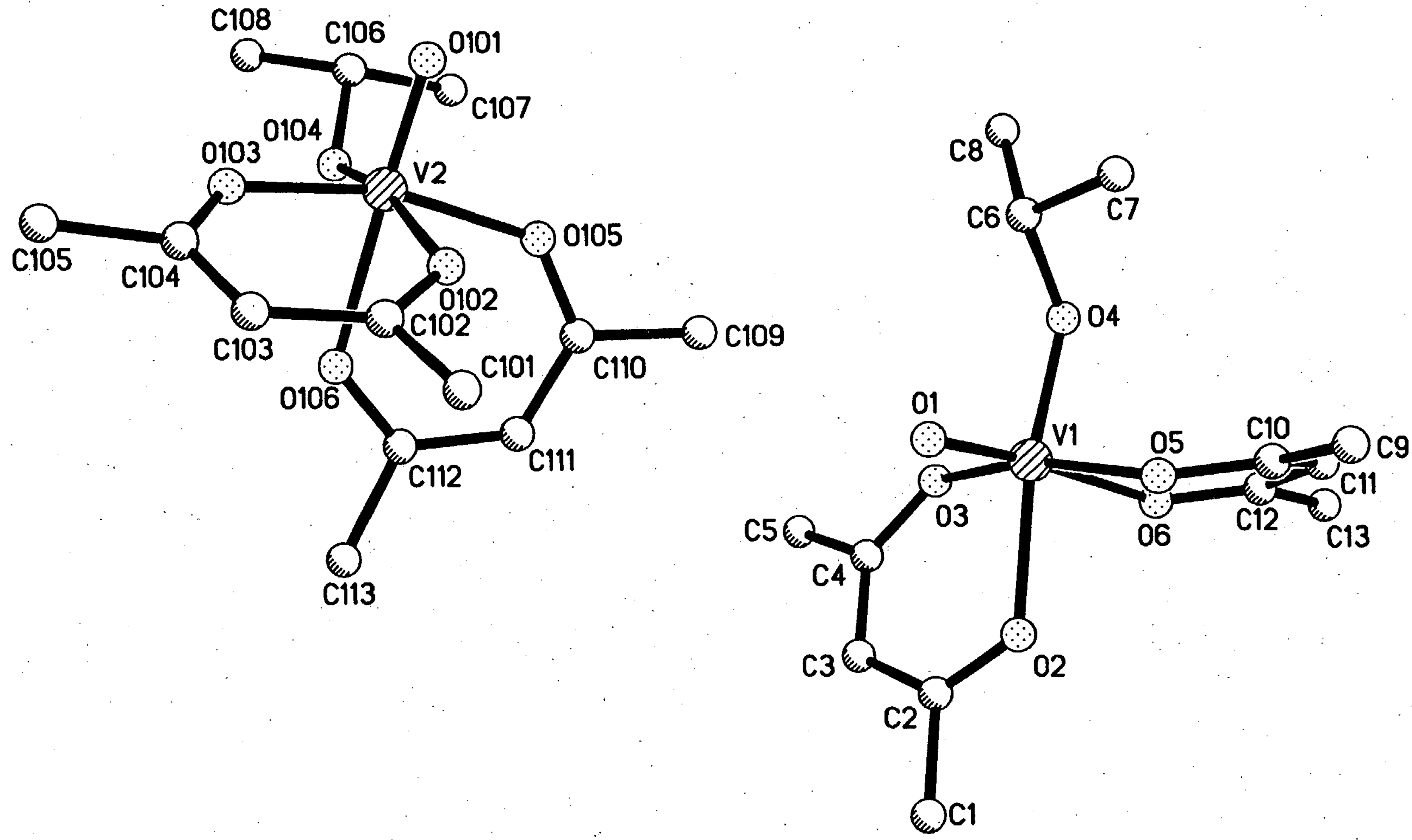
Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 1.
The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2U_{11} + ... + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
V(1)	20(1)	29(1)	34(1)	5(1)	12(1)	4(1)
V(2)	18(1)	19(1)	21(1)	1(1)	7(1)	0(1)
V(3)	22(1)	18(1)	25(1)	3(1)	7(1)	1(1)
O(1)	29(4)	35(4)	41(4)	6(3)	20(3)	7(3)
O(2)	19(4)	31(4)	41(4)	8(3)	4(3)	5(3)
O(3)	34(4)	27(4)	31(4)	2(3)	15(3)	0(3)
O(4)	29(4)	21(3)	32(4)	0(3)	6(3)	-5(3)
O(5)	17(3)	18(3)	17(3)	2(3)	2(3)	4(3)
O(6)	20(3)	24(4)	25(3)	3(3)	11(3)	3(3)
O(7)	26(4)	30(4)	33(4)	2(3)	12(3)	-5(3)
O(8)	16(3)	26(4)	12(3)	-1(3)	3(3)	-1(3)
O(9)	20(3)	19(4)	18(3)	-5(3)	1(3)	0(3)
O(10)	24(3)	29(4)	20(3)	1(3)	10(3)	1(3)
O(11)	28(4)	21(4)	25(4)	-9(3)	7(3)	-13(3)
O(12)	36(4)	20(4)	29(4)	4(3)	6(3)	1(3)
O(13)	27(4)	21(4)	29(4)	4(3)	10(3)	3(3)
O(14)	27(4)	20(4)	24(3)	4(3)	10(3)	3(3)
C(1)	31(6)	47(7)	51(7)	18(6)	14(5)	20(6)
C(2)	41(6)	25(6)	41(7)	9(5)	28(6)	11(5)
C(3)	37(6)	24(6)	44(7)	1(5)	25(5)	-10(5)
C(4)	34(6)	30(7)	31(6)	-4(5)	18(5)	3(5)
C(5)	57(7)	40(7)	48(7)	1(6)	24(6)	-15(6)
C(6)	81(9)	39(8)	88(10)	-13(7)	43(8)	-19(7)
C(7)	83(12)	60(11)	241(23)	41(13)	73(14)	18(10)
C(8)	37(6)	15(5)	26(6)	4(4)	17(5)	2(4)
C(9)	35(6)	36(6)	15(5)	5(5)	15(4)	3(5)
C(10)	25(5)	31(6)	21(5)	2(5)	3(4)	0(5)
C(11)	18(5)	37(6)	15(5)	3(5)	9(4)	8(5)
C(12)	34(6)	53(8)	29(6)	-3(5)	17(5)	-5(5)
C(13)	24(5)	29(6)	23(5)	7(4)	10(4)	-1(4)
C(14)	28(5)	19(5)	19(5)	3(4)	-1(4)	1(4)
C(15)	16(5)	20(5)	19(5)	-4(4)	2(4)	2(4)
C(16)	30(5)	22(5)	17(5)	-6(4)	11(4)	0(5)
C(17)	39(6)	26(6)	23(5)	4(5)	12(5)	8(5)
C(18)	34(6)	39(6)	39(6)	2(5)	18(5)	-2(5)
C(19)	32(6)	12(5)	31(6)	-6(4)	10(5)	-4(5)
C(20)	20(5)	29(6)	21(6)	1(5)	-1(4)	-4(5)
C(21)	17(5)	19(5)	24(5)	-4(4)	1(4)	1(4)
C(22)	41(6)	42(7)	32(6)	5(6)	8(5)	16(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 1.

	x	y	z	U(eq)
H(1A)	4573(7)	-686(9)	5634(5)	80
H(1B)	4481(7)	-2022(9)	5322(5)	80
H(1C)	3753(7)	-1583(9)	5756(5)	80
H(3A)	2511(7)	-2408(8)	4499(5)	80
H(5A)	830(8)	-1817(9)	2675(6)	80
H(5B)	726(8)	-2411(9)	3396(6)	80
H(5C)	1545(8)	-2911(9)	3057(6)	80
H(6A)	4374(10)	3345(10)	4645(7)	80
H(6B)	3764(10)	3353(10)	3793(7)	80
H(7A)	3908(11)	5163(12)	4257(10)	80
H(7B)	3354(11)	4748(12)	4816(10)	80
H(7C)	2749(11)	4760(12)	3961(10)	80
H(8A)	1357(7)	4214(7)	2153(5)	80
H(8B)	242(7)	4730(7)	1987(5)	80
H(9A)	788(7)	1145(9)	2208(4)	80
H(9B)	1677(7)	2078(9)	2264(4)	80
H(10A)	-1152(6)	3327(8)	2026(5)	80
H(10B)	-943(6)	1916(8)	2098(5)	80
H(12A)	-495(7)	3385(10)	1016(5)	80
H(12B)	-275(7)	1977(10)	1099(5)	80
H(12C)	610(7)	2886(10)	1105(5)	80
H(13A)	-212(6)	1087(8)	5349(5)	80
H(13B)	-469(6)	2434(8)	5067(5)	80
H(14A)	2427(6)	1264(8)	5380(4)	80
H(14B)	1564(6)	295(8)	5318(4)	80
H(15A)	864(6)	3873(7)	5204(4)	80
H(15B)	1944(6)	3444(7)	5179(4)	80
H(17A)	969(7)	2743(8)	6293(4)	80
H(17B)	2095(7)	2353(8)	6361(4)	80
H(17C)	1277(7)	1358(8)	6366(4)	80
H(18A)	-2039(7)	3465(9)	4592(5)	80
H(18B)	-2940(7)	3149(9)	3861(5)	80
H(18C)	-2914(7)	4423(9)	4243(5)	80
H(20A)	-3049(7)	4783(8)	2799(5)	80
H(22A)	-1762(7)	6456(9)	1793(5)	80
H(22B)	-2653(7)	6788(9)	2105(5)	80
H(22C)	-2748(7)	5631(9)	1602(5)	80


C8
C6
C5
O1
C7
O3
C4
O4
C3
V1
C2
O5
O2
O6
C1
C10
C12
C11
C9
C13


C108
C106
O101
C107
O104
O103
V2
C105
C104
O105
O102
C102
C103
C101
C110
C109
O106
C111
C112
C113
C8
C7
C6
O4
O1
V1
O5
C10
C9
C11
O3
O6
C12
C13
C5
C4
C3
O2
C2
C1

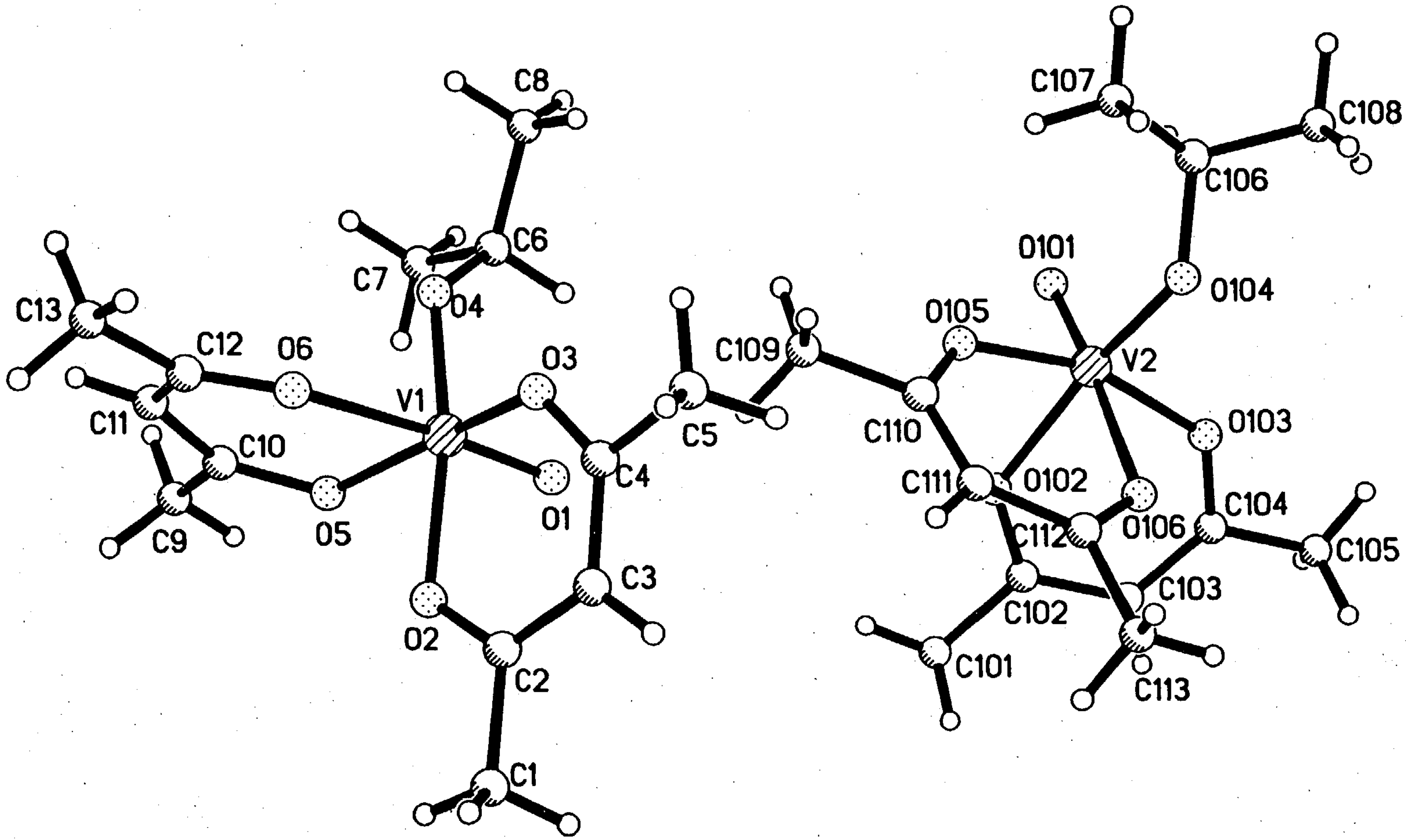

C8
C107
C108
C106
C6
O101
O104
C7
O4
C13
C12
O6
O3
C109
O105
V2
V1
C5
C110
O103
C11
C10
C4
C111
O102
C104
C9
O5
O1
C112
O106
C105
C3
C102
C103
O2
C2
C101
C113
C1

Table 1. Crystal data and structure refinement for 1.

Identification code	dc16m
Empirical formula	$C_{13}H_{21}O_6V$
Formula weight	324.24
Temperature	165(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 15.0023(3) Å alpha = 90°
	b = 8.13680(10) Å beta = 95.74°
	c = 26.5598(2) Å gamma = 90°
Volume, Z	3225.89(8) $Å^3$, 8
Density (calculated)	1.335 Mg/m^3
Absorption coefficient	0.634 mm^{-1}
F(000)	1360
Crystal size	0.40 x 0.34 x 0.20 mm
θ range for data collection	1.50 to 28.25°
Limiting indices	$-19 \le h \le 15$, $-9 \le k \le 10$, $-32 \le l \le 35$
Reflections collected	20621
Independent reflections	7721 (R_{int} = 0.0550)
Absorption correction	SADABS Tmax=0.98 Tmin=0.64
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7721 / 0 / 361
Goodness-of-fit on F^2	1.036
Final R indices [$I>2\sigma(I)$]	R1 = 0.0598, wR2 = 0.1308
R indices (all data)	R1 = 0.1062, wR2 = 0.1543
Largest diff. peak and hole	0.512 and -0.442 $eÅ^{-3}$

Table 3. Bond lengths [Å] and angles [°] for 1.

V(1)-O(1)	1.602(2)	V(1)-O(4)	1.776(2)
V(1)-O(3)	1.942(2)	V(1)-O(5)	1.951(2)
V(1)-O(2)	2.059(2)	V(1)-O(6)	2.140(2)
O(2)-C(2)	1.270(4)	O(3)-C(4)	1.295(4)
O(4)-C(6)	1.450(4)	O(5)-C(10)	1.300(4)
O(6)-C(12)	1.259(4)	C(1)-C(2)	1.515(5)
C(2)-C(3)	1.393(5)	C(3)-C(4)	1.379(5)
C(4)-C(5)	1.497(5)	C(6)-C(7)	1.511(5)
C(6)-C(8)	1.516(5)	C(9)-C(10)	1.494(5)
C(10)-C(11)	1.381(6)	C(11)-C(12)	1.405(6)
C(12)-C(13)	1.505(6)	V(2)-O(101)	1.600(2)
V(2)-O(104)	1.772(2)	V(2)-O(103)	1.936(2)
V(2)-O(105)	1.943(2)	V(2)-O(102)	2.052(3)
V(2)-O(106)	2.154(2)	O(102)-C(102)	1.269(4)
O(103)-C(104)	1.303(4)	O(104)-C(106)	1.447(4)
O(105)-C(110)	1.290(4)	O(106)-C(112)	1.259(4)
C(101)-C(102)	1.504(6)	C(102)-C(103)	1.406(6)
C(103)-C(104)	1.377(6)	C(104)-C(105)	1.498(5)
C(106)-C(108)	1.507(5)	C(106)-C(107)	1.510(5)
C(109)-C(110)	1.505(5)	C(110)-C(111)	1.368(5)
C(111)-C(112)	1.406(5)	C(112)-C(113)	1.512(5)
O(1)-V(1)-O(4)	99.02(12)	O(1)-V(1)-O(3)	97.73(12)
O(4)-V(1)-O(3)	94.83(10)	O(1)-V(1)-O(5)	95.01(12)
O(4)-V(1)-O(5)	93.89(11)	O(3)-V(1)-O(5)	163.23(11)
O(1)-V(1)-O(2)	91.85(12)	O(4)-V(1)-O(2)	169.10(11)
O(3)-V(1)-O(2)	84.50(10)	O(5)-V(1)-O(2)	84.23(10)
O(1)-V(1)-O(6)	175.10(12)	O(4)-V(1)-O(6)	85.88(10)
O(3)-V(1)-O(6)	81.99(10)	O(5)-V(1)-O(6)	84.38(10)
O(2)-V(1)-O(6)	83.24(10)	C(2)-O(2)-V(1)	126.8(2)
C(4)-O(3)-V(1)	130.3(2)	C(6)-O(4)-V(1)	127.1(2)
C(10)-O(5)-V(1)	129.5(2)	C(12)-O(6)-V(1)	126.4(3)
O(2)-C(2)-C(3)	124.2(3)	O(2)-C(2)-C(1)	116.3(3)
C(3)-C(2)-C(1)	119.5(3)	C(4)-C(3)-C(2)	122.9(3)
O(3)-C(4)-C(3)	123.9(3)	O(3)-C(4)-C(5)	115.2(3)
C(3)-C(4)-C(5)	120.8(3)	O(4)-C(6)-C(7)	108.5(3)
O(4)-C(6)-C(8)	108.7(3)	C(7)-C(6)-C(8)	112.7(3)
O(5)-C(10)-C(11)	124.8(3)	O(5)-C(10)-C(9)	113.7(4)
C(11)-C(10)-C(9)	121.5(3)	C(10)-C(11)-C(12)	123.9(3)
O(6)-C(12)-C(11)	124.0(4)	O(6)-C(12)-C(13)	117.2(4)
C(11)-C(12)-C(13)	118.7(3)	O(101)-V(2)-O(104)	99.37(12)
O(101)-V(2)-O(103)	98.67(11)	O(104)-V(2)-O(103)	95.93(10)
O(101)-V(2)-O(105)	93.71(11)	O(104)-V(2)-O(105)	94.83(10)
O(103)-V(2)-O(105)	162.04(10)	O(101)-V(2)-O(102)	94.35(12)
O(104)-V(2)-O(102)	165.91(11)	O(103)-V(2)-O(102)	85.09(10)
O(105)-V(2)-O(102)	81.02(10)	O(101)-V(2)-O(106)	175.33(12)
O(104)-V(2)-O(106)	85.27(10)	O(103)-V(2)-O(106)	81.28(9)
O(105)-V(2)-O(106)	85.32(9)	O(102)-V(2)-O(106)	80.98(10)
C(102)-O(102)-V(2)	129.9(2)	C(104)-O(103)-V(2)	131.8(2)
C(106)-O(104)-V(2)	129.3(2)	C(110)-O(105)-V(2)	132.1(2)
C(112)-O(106)-V(2)	128.0(2)	O(102)-C(102)-C(103)	122.9(4)
O(102)-C(102)-C(101)	117.0(4)	C(103)-C(102)-C(101)	120.0(4)
C(104)-C(103)-C(102)	123.7(4)	O(103)-C(104)-C(103)	124.1(3)
O(103)-C(104)-C(105)	114.6(3)	C(103)-C(104)-C(105)	121.3(3)

O(104)-C(106)-C(108)	108.4(3)	O(104)-C(106)-C(107)	108.8(3)
C(108)-C(106)-C(107)	113.2(3)	O(105)-C(110)-C(111)	125.8(3)
O(105)-C(110)-C(109)	113.5(3)	C(111)-C(110)-C(109)	120.7(3)
C(110)-C(111)-C(112)	124.8(3)	O(106)-C(112)-C(111)	123.9(3)
O(106)-C(112)-C(113)	116.6(3)	C(111)-C(112)-C(113)	119.5(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 1.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
V(1)	24(1)	32(1)	23(1)	-5(1)	1(1)	0(1)
O(1)	36(2)	48(2)	26(1)	1(1)	8(1)	3(1)
O(2)	28(1)	41(2)	35(1)	-13(1)	-2(1)	1(1)
O(3)	23(1)	42(2)	27(1)	-5(1)	0(1)	-1(1)
O(4)	28(1)	27(1)	27(1)	-3(1)	5(1)	2(1)
O(5)	24(1)	43(2)	36(1)	-13(1)	0(1)	0(1)
O(6)	37(2)	31(1)	27(1)	-1(1)	-4(1)	3(1)
C(1)	46(3)	44(2)	49(2)	-18(2)	8(2)	-2(2)
C(2)	37(2)	33(2)	23(2)	-4(2)	4(2)	-3(2)
C(3)	36(2)	42(2)	32(2)	-10(2)	-2(2)	-7(2)
C(4)	31(2)	38(2)	25(2)	6(2)	0(2)	-6(2)
C(5)	30(2)	64(3)	43(2)	-4(2)	-1(2)	-13(2)
C(6)	43(2)	28(2)	29(2)	1(2)	4(2)	-2(2)
C(7)	47(3)	31(2)	64(3)	-10(2)	3(2)	-3(2)
C(8)	50(3)	40(2)	58(3)	4(2)	10(2)	15(2)
C(9)	27(2)	75(3)	57(3)	-30(2)	-7(2)	3(2)
C(10)	26(2)	39(2)	45(2)	-23(2)	-10(2)	8(2)
C(11)	43(2)	49(3)	31(2)	-13(2)	-11(2)	11(2)
C(12)	49(2)	25(2)	28(2)	-6(2)	-2(2)	11(2)
C(13)	84(4)	42(3)	30(2)	2(2)	-2(2)	11(2)
V(2)	28(1)	20(1)	23(1)	0(1)	3(1)	1(1)
O(101)	39(2)	25(1)	31(1)	0(1)	0(1)	-3(1)
O(102)	31(1)	36(2)	37(1)	-1(1)	3(1)	1(1)
O(103)	39(2)	25(1)	25(1)	2(1)	6(1)	2(1)
O(104)	26(1)	26(1)	26(1)	-2(1)	4(1)	3(1)
O(105)	38(1)	26(1)	24(1)	1(1)	0(1)	3(1)
O(106)	34(1)	22(1)	26(1)	0(1)	3(1)	2(1)
C(101)	35(3)	80(4)	77(3)	-1(3)	16(2)	14(2)
C(102)	30(2)	39(2)	49(2)	-6(2)	10(2)	-2(2)
C(103)	43(3)	63(3)	38(2)	-4(2)	18(2)	4(2)
C(104)	46(2)	29(2)	27(2)	-2(2)	13(2)	-8(2)
C(105)	56(3)	48(3)	30(2)	3(2)	12(2)	-8(2)
C(106)	35(2)	30(2)	34(2)	-7(2)	8(2)	5(2)
C(107)	54(3)	46(2)	31(2)	-7(2)	18(2)	-1(2)
C(108)	41(2)	54(3)	52(2)	-7(2)	4(2)	21(2)
C(109)	65(3)	41(2)	34(2)	13(2)	-6(2)	9(2)
C(110)	28(2)	27(2)	31(2)	8(2)	3(1)	4(1)
C(111)	46(2)	25(2)	38(2)	7(2)	-4(2)	3(2)
C(112)	28(2)	24(2)	36(2)	-2(2)	5(2)	1(1)
C(113)	72(3)	24(2)	45(2)	-12(2)	3(2)	-2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 1.

	x	y	z	U(eq)
H(1B)	1611(3)	13201(5)	1010(2)	70
H(1C)	2498(3)	14306(5)	1073(2)	70
H(1D)	2330(3)	12954(5)	1491(2)	70
H(3A)	3953(3)	12331(5)	1226(1)	44
H(5A)	5404(3)	9993(6)	513(2)	69
H(5B)	5365(3)	10489(6)	1093(2)	69
H(5C)	5374(3)	11889(6)	671(2)	69
H(6A)	2934(3)	5443(4)	262(1)	40
H(7A)	1492(3)	5473(5)	-174(2)	72
H(7B)	1951(3)	3729(5)	-251(2)	72
H(7C)	1899(3)	5068(5)	-694(2)	72
H(8A)	4213(3)	5264(5)	-182(2)	74
H(8B)	3597(3)	4938(5)	-700(2)	74
H(8C)	3649(3)	3597(5)	-257(2)	74
H(9A)	-158(3)	7902(6)	-246(2)	81
H(9B)	-71(3)	7494(6)	-828(2)	81
H(9C)	-435(3)	9251(6)	-671(2)	81
H(11E)	962(3)	9672(5)	-1242(1)	51
H(13A)	2980(3)	11979(5)	-1235(2)	79
H(13B)	1947(3)	12382(5)	-1396(2)	79
H(13C)	2395(3)	10780(5)	-1614(2)	79
H(10P)	2173(3)	4893(7)	2406(2)	95
H(10Q)	2261(3)	5558(7)	2976(2)	95
H(10R)	1879(3)	3751(7)	2851(2)	95
H(10I)	3244(3)	3572(6)	3604(2)	56
H(10C)	5282(3)	1379(5)	3935(1)	66
H(10D)	4256(3)	1046(5)	4020(1)	66
H(10E)	4717(3)	2757(5)	4193(1)	66
H(10B)	6419(2)	1100(4)	2049(1)	39
H(10M)	6307(3)	3214(5)	1444(1)	64
H(10N)	7261(3)	2331(5)	1446(1)	64
H(10O)	7185(3)	4076(5)	1717(1)	64
H(10J)	7452(3)	1262(6)	2770(2)	74
H(10K)	7902(3)	2853(6)	2547(2)	74
H(10L)	7977(3)	1109(6)	2277(2)	74
H(10F)	4430(3)	5594(5)	980(1)	71
H(10G)	4984(3)	7263(5)	1079(1)	71
H(10H)	3942(3)	7209(5)	1160(1)	71
H(11D)	4820(3)	8379(5)	2006(1)	44
H(11A)	5361(3)	7890(5)	3337(2)	71
H(11B)	4663(3)	8973(5)	2983(2)	71
H(11C)	5706(3)	9030(5)	2906(2)	71