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	$U_{\rm eq}$
Ag	0.11664 (7)	0.48495 (6)	0.13126 (3)	0.0284 (1)
Co	0.72407 (11)	0.53483 (9)	0.89295 (5)	0.0192 (2)
O*W*	0.2185 (8)	0.3540 (7)	0.8962 (5)	0.049 (2)
O1	0.7494 (7)	0.7389 (5)	0.8896 (4)	0.030 (1)
O2	0.8958 (8)	0.9180 (5)	0.8338 (4)	0.040 (1)
O21	0.6390 (10)	0.6251 (8)	0.7334 (4)	0.056 (2)
O22	0.4813 (8)	0.4595 (8)	0.7753 (4)	0.046 (3)
O31	0.5941 (9)	0.2690 (6)	0.9377 (4)	0.048 (2)
O32	0.7990 (10)	0.2549 (6)	0.8533 (5)	0.071 (2)
O41	0.7873 (9)	0.4656 (8)	1.0591 (3)	0.049 (1)
O42	0.9864 (8)	0.5856 (7)	1.0037 (4)	0.048 (1)
N1	0.5130 (8)	0.5572 (7)	0.9552 (4)	0.030 (1)
N2	0.6018 (8)	0.5403 (7)	0.7881 (4)	0.027 (1)
N3	0.7038 (9)	0.3275 (6)	0.8954 (4)	0.029 (1)
N4	0.8459 (8)	0.5289 (7)	0.9978 (4)	0.028 (2)
N5	0.9361 (7)	0.5286 (6)	0.8312 (4)	0.020 (1)
C1	0.9940 (9)	0.6774 (8)	0.8092 (5)	0.025 (1)
C2	1.1750 (10)	0.7059 (9)	0.8379 (7)	0.041 (2)
C3	0.8729 (10)	0.7871 (7)	0.8462 (5)	0.024 (1)

Table 2. *Selected geometric parameters* (Å, °)

Co—O1	1.903 (5)	O22—N2	1.230 (9)
Co—N1	1.956 (6)	O31—N3	1.227 (9)
Co—N2	1.936 (6)	O32—N3	1.214 (10)
Co—N3	1.929 (6)	O41—N4	1.233 (9)
Co—N4	1.935 (6)	O42—N4	1.234 (9)
Co—N5	1.948 (6)	N5—C1	1.496 (9)
O1—C3	1.279 (10)	C1—C2	1.528 (11)
O2—C3	1.243 (8)	C1—C3	1.518 (10)
O21—N2	1.212 (9)		
O1—Co—N1	89.9 (3)	Co—N2—O21	121.3 (6)
O1—Co—N2	90.1 (3)	Co—N2—O22	121.1 (5)
O1—Co—N3	178.7 (3)	O21—N2—O22	117.6 (7)
O1—Co—N4	90.0 (3)	Co—N3—O31	120.7 (5)
O1—Co—N5	85.7 (2)	Co—N3—O32	119.3 (5)
N1—Co—N2	90.6 (3)	O31—N3—O32	120.0 (7)
N1—Co—N3	91.4 (3)	Co—N4—O41	121.0 (5)
N1—Co—N4	89.3 (3)	Co—N4—O42	120.2 (5)
N1—Co—N5	175.6 (3)	O41—N4—O42	118.8 (7)
N2—Co—N3	90.1 (3)	Co—N5—C1	110.9 (4)
N2—Co—N4	179.9 (3)	N5—C1—C2	112.1 (6)
N2—Co—N5	89.6 (3)	N5—C1—C3	109.4 (6)
N3—Co—N4	89.8 (3)	C2—C1—C3	111.1 (7)
N3—Co—N5	93.0 (3)	O1—C3—O2	122.6 (7)
N4—Co—N5	90.5 (3)	O1—C3—C1	117.4 (7)
Co—O1—C3	116.3 (5)	O2—C3—C1	120.0 (7)

The structure of the title compound was solved by Patterson and difference Fourier methods and refined by full-matrix least-squares methods. At a later stage of isotropic refinement, the absolute configuration was established by carrying out the refinement with two alternate configurations, with values of the polarity factor η of 1 and -1, giving R factors of 0.068 and 0.076, respectively. The model with the lower R value, which is in accordance with the configuration deduced from the configuration of (*S*)-alanine, was further anisotropically refined. The majority of the H atoms, including those belonging to the water molecule, were found in the difference Fourier map. The remaining H atoms were placed at calculated positions with C—H and N—H distances of 0.95 Å.

All calculations were performed on a PDP 11/73 computer using *SDP* (Frenz, 1982). Data collection, cell refinement and data reduction were performed using *CAD*-4 *Software* (Enraf–Nonius, 1989). Molecular graphics were produced using *SDP*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and torsion angles have been deposited with the IUCr (Reference: KA1120). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Platinum(II) Diammine Complex: *cis*-$[\mathrm{Pt(C_8H_{11}N_5O_3)_2(NH_3)_2]Cl_2.2H_2O}$

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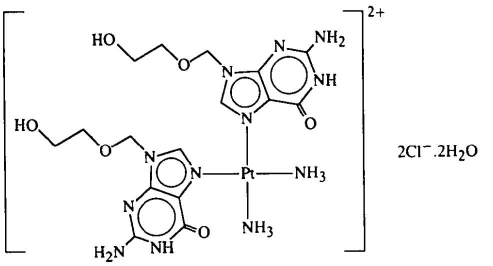
Abstract

The crystal structure of *cis*-diamminebis{9-[(2-hydroxyethoxy)methyl]guanine}platinum(II) chloride dihydrate (systematic name: bis{2-amino-1,9-dihydro-9-[(2-hydroxyethoxy)methyl]-6*H*-purin-6-one-N^7}diammineplatinum(II) chloride dihydrate), *cis*-$[\mathrm{Pt(C_8H_{11}N_5O_3)_2(NH_3)_2]Cl_2.2H_2O}$, was determined by single-crystal X-ray diffraction. The Pt atom is *cis*-coordinated by the N atoms of two ammine ligands and by an N atom from each of two 9-[(2-hydroxyethoxy)-methyl]guanine ligands. The geometry around the Pt atom deviates slightly from square-planarity. The complex crystallizes in the trigonal space group $P3_121$.

Comment

The title complex, (I), contains Acyclovir {ACV, 9-[(2-hydroxyethoxy)methyl]guanine}, a clinically licensed antiherpes agent (Keller, Fyfe, Beauchamp, Lubbers, Furman, Schaeffer & Elion, 1981), as a ligand.



(I)

The asymmetric unit of the complex is shown in Fig. 1. It contains three chloride ions, two water molecules and a mononuclear cationic unit in which the Pt atom is *cis*-coordinated by two ammine and two ACV ligands. The atoms Cl(1) and Cl(2) lie on twofold axes.

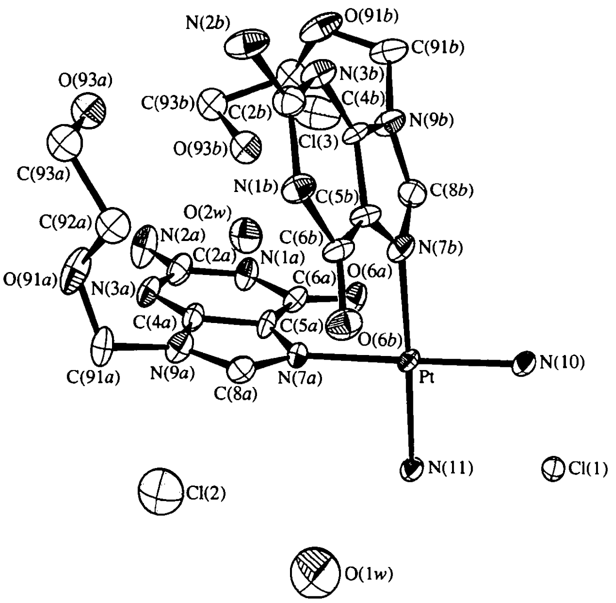


Fig. 1. An *ORTEP*II (Johnson, 1976) view of the asymmetric unit of the title compound, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 25% probability level.

The geometry around the Pt atom is approximately square planar. The bond angle N(7*a*)—Pt—N(7*b*) of 85.5 (6)° shows the greatest deviation from 90°. The Pt—N bond distances, ranging from 2.02 (2) to 2.03 (2) Å, are normal for this type of complex. The binding of the Pt atom to both ACV ligands is, as expected, through atom N(7). The bond lengths and angles within the purine rings are close to the average values obtained from a number of structures of guanine derivatives (Taylor & Kennard, 1982).

The ends of the acyclic chains are disordered, and as a result the bond lengths and angles thereof deviate from the expected values (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The ACV ligands adopt a head-to-tail arrangement. The guanine part of each ACV ligand is nearly planar. The largest deviation from the best plane is 0.02 (3) Å for atom C(2*a*) in the first ligand (ligand *a*) and −0.06 (2) Å for atom N(9*b*) in the second (ligand *b*). The largest deviation from the square coordination plane is 0.02 (2) Å for atom N(7*a*). The dihedral angle between the best planes through both nucleobases is 63.3 (3)°. The dihedral angles between the square coordination plane and the best planes through the nucleobases are 81.0 (4)° for ligand *a* and 76.8 (4)° for ligand *b*.

The structure is held together by a complicated network of hydrogen bonds involving both water molecules, both ammine ligands, the chloride ions and atoms N(1*a*), N(1*b*), N(2*a*), N(2*b*), O(6*a*) and O(6*b*) of the ACV ligands. Hydrogen-bond distances are given in Table 2.

Experimental

The synthesis of the title compound will be described elsewhere (Grabner & Bukovec, 1995). Crystals were obtained from aqueous solution. The crystal density D_m was measured by flotation.

Crystal data

$[Pt(C_8H_{11}N_5O_3)_2(NH_3)_2]$-$Cl_2.2H_2O$
$M_r = 786.5$
Trigonal
$P3_121$
$a = 20.270\ (9)$ Å
$c = 12.30\ (5)$ Å
$V = 4377\ (18)$ Å^3
$Z = 6$
$D_x = 1.790$ Mg m^{-3}
$D_m = 1.78\ (2)$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8.52$–$11.66°$
$\mu = 5.099$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.48 × 0.48 × 0.26 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical
$T_{min} = 0.1288$, $T_{max} = 0.3337$
22 441 measured reflections
3807 independent reflections

3097 observed reflections $[I > 2.5\sigma(I)]$
$R_{int} = 0.049$
$\theta_{max} = 27.98°$
$h = -26 \rightarrow 26$
$k = -26 \rightarrow 26$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 333 min
intensity decay: 10.87%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.056$
$S = 2.843$

$(\Delta/\sigma)_{max} = 0.080$
$\Delta\rho_{max} = 1.57$ e Å^{-3}
$\Delta\rho_{min} = -4.55$ e Å^{-3}
Extinction correction: none

3455 reflections
294 parameters
H-atom parameters not refined
$w = 6w_f w_s$ (see below)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

$U_{\rm iso}$ for C(92*a*), C(93*a*), O(93*a*), C(92*b*), C(93*b*), O(93*b*) and O(1*w*), $U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Pt	0.11023 (3)	0.20676 (2)	0.32873 (4)	0.03353 (17)
N(10)	0.1375 (7)	0.1234 (7)	0.3405 (11)	0.043 (4)
N(11)	−0.0015 (7)	0.1261 (7)	0.3295 (12)	0.044 (5)
N(1*a*)	0.1122 (11)	0.3879 (9)	0.0572 (11)	0.056 (7)
N(2*a*)	0.1088 (17)	0.4973 (12)	0.0217 (13)	0.083 (12)
N(3*a*)	0.0911 (9)	0.4532 (7)	0.1987 (12)	0.052 (6)
N(7*a*)	0.0861 (8)	0.2918 (7)	0.3206 (11)	0.041 (5)
N(9*a*)	0.0736 (9)	0.3871 (8)	0.3691 (12)	0.055 (6)
O(6*a*)	0.1183 (9)	0.2798 (7)	0.0762 (10)	0.059 (6)
O(91*a*)	0.1384 (12)	0.5126 (8)	0.4428 (14)	0.081 (8)
O(93*a*)	0.2870	0.6085	0.3895	0.069
C(2*a*)	0.1052 (12)	0.4474 (10)	0.0955 (16)	0.059 (8)
C(4*a*)	0.0864 (10)	0.3983 (8)	0.2587 (14)	0.048 (6)
C(5*a*)	0.0937 (10)	0.3352 (8)	0.2314 (13)	0.044 (6)
C(6*a*)	0.1080 (10)	0.3285 (8)	0.1170 (14)	0.048 (6)
C(8*a*)	0.0730 (9)	0.3259 (9)	0.4014 (12)	0.046 (6)
C(91*a*)	0.0701 (14)	0.4434 (10)	0.4424 (14)	0.061 (9)
C(92*a*)	0.1988	0.5106	0.4921	0.069
C(93*a*)	0.2644	0.5957	0.4923	0.069
N(1*b*)	0.2860 (8)	0.4024 (8)	0.5818 (10)	0.047 (5)
N(2*b*)	0.3978 (9)	0.5168 (9)	0.6065 (12)	0.062 (6)
N(3*b*)	0.3751 (9)	0.4600 (8)	0.4379 (11)	0.051 (5)
N(7*b*)	0.2206 (8)	0.2905 (7)	0.3301 (11)	0.046 (5)
N(9*b*)	0.3330 (8)	0.3814 (8)	0.2762 (11)	0.052 (5)
O(6*b*)	0.1740 (7)	0.2907 (7)	0.5679 (10)	0.057 (5)
O(91*b*)	0.4033 (10)	0.4989 (7)	0.1840 (10)	0.075 (6)
O(93*b*)	0.2704	0.4761	0.2226	0.067
C(2*b*)	0.3537 (10)	0.4601 (10)	0.5398 (13)	0.048 (6)
C(4*b*)	0.3249 (7)	0.3987 (6)	0.3809 (11)	0.036 (4)
C(5*b*)	0.2549 (7)	0.3395 (8)	0.4176 (12)	0.040 (5)
C(6*b*)	0.2320 (8)	0.3385 (7)	0.5250 (13)	0.042 (5)
C(8*b*)	0.2671 (10)	0.3166 (10)	0.2480 (14)	0.053 (7)
C(91*b*)	0.3961 (11)	0.4298 (12)	0.2033 (16)	0.066 (7)
C(92*b*)	0.3437	0.4933	0.1118	0.067
C(93*b*)	0.2982	0.5366	0.1488	0.067
Cl(1)	0	−0.00093 (20)	1/6	0.0415 (13)
Cl(2)	0	0.3098 (5)	2/3	0.087 (4)
Cl(3)	0.2859 (5)	0.4560 (8)	−0.1798 (6)	0.131 (6)
O(1*w*)	−0.0778 (22)	0.1449 (21)	0.527 (3)	0.140 (10)
O(2*w*)	0.1208 (14)	0.4210 (15)	−0.1713 (16)	0.102 (12)

Table 2. *Selected geometric parameters* (Å, °)

Pt—N(10)	2.03 (2)	C(5*a*)—C(6*a*)	1.46 (2)
Pt—N(11)	2.02 (1)	C(92*a*)—C(93*a*)	1.57
Pt—N(7*a*)	2.02 (2)	N(1*b*)—C(2*b*)	1.38 (2)
Pt—N(7*b*)	2.02 (1)	N(1*b*)—C(6*b*)	1.39 (2)
N(1*a*)—C(2*a*)	1.37 (3)	N(2*b*)—C(2*b*)	1.33 (2)
N(1*a*)—C(6*a*)	1.38 (3)	N(3*b*)—C(2*b*)	1.33 (2)
N(2*a*)—C(2*a*)	1.33 (3)	N(3*b*)—C(4*b*)	1.34 (2)
N(3*a*)—C(2*a*)	1.32 (3)	N(7*b*)—C(5*b*)	1.39 (2)
N(3*a*)—C(4*a*)	1.30 (2)	N(7*b*)—C(8*b*)	1.30 (2)
N(7*a*)—C(5*a*)	1.37 (2)	N(9*b*)—C(4*b*)	1.37 (2)
N(7*a*)—C(8*a*)	1.31 (2)	N(9*b*)—C(8*b*)	1.37 (2)
N(9*a*)—C(4*a*)	1.38 (2)	N(9*b*)—C(91*b*)	1.47 (2)
N(9*a*)—C(8*a*)	1.30 (3)	O(6*b*)—C(6*b*)	1.21 (2)
N(9*a*)—C(91*a*)	1.48 (3)	O(91*b*)—C(91*b*)	1.36 (3)
O(6*a*)—C(6*a*)	1.21 (3)	O(91*b*)—C(92*b*)	1.46
O(91*a*)—C(91*a*)	1.39 (2)	O(93*b*)—C(93*b*)	1.40
O(91*a*)—C(92*a*)	1.38	C(4*b*)—C(5*b*)	1.40 (2)
O(93*a*)—C(93*a*)	1.33	C(5*b*)—C(6*b*)	1.40 (2)
C(4*a*)—C(5*a*)	1.40 (3)	C(92*b*)—C(93*b*)	1.62
N(10)—Pt—N(11)	89.3 (5)	N(9*a*)—C(91*a*)—O(91*a*)	112 (2)
N(10)—Pt—N(7*a*)	178.0 (5)	O(91*a*)—C(92*a*)—C(93*a*)	104
N(10)—Pt—N(7*b*)	92.9 (6)	O(93*a*)—C(93*a*)—C(92*a*)	104
N(11)—Pt—N(7*a*)	92.2 (6)	C(2*b*)—N(1*b*)—C(6*b*)	126 (1)
N(11)—Pt—N(7*b*)	177.6 (7)	C(2*b*)—N(3*b*)—C(4*b*)	113 (1)
N(7*a*)—Pt—N(7*b*)	85.5 (6)	Pt—N(7*b*)—C(5*b*)	124 (1)
C(2*a*)—N(1*a*)—C(6*a*)	127 (2)	Pt—N(7*b*)—C(8*b*)	128 (1)
C(2*a*)—N(3*a*)—C(4*a*)	113 (2)	C(5*b*)—N(7*b*)—C(8*b*)	108 (1)
Pt—N(7*a*)—C(5*a*)	126 (1)	C(4*b*)—N(9*b*)—C(8*b*)	108 (1)
Pt—N(7*a*)—C(8*a*)	128 (1)	C(4*b*)—N(9*b*)—C(91*b*)	126 (1)
C(5*a*)—N(7*a*)—C(8*a*)	105 (2)	C(8*b*)—N(9*b*)—C(91*b*)	126 (1)
C(4*a*)—N(9*a*)—C(8*a*)	112 (2)	C(91*b*)—O(91*b*)—C(92*b*)	112
C(4*a*)—N(9*a*)—C(91*a*)	123 (2)	N(1*b*)—C(2*b*)—N(2*b*)	117 (1)
C(8*a*)—N(9*a*)—C(91*a*)	125 (1)	N(1*b*)—C(2*b*)—N(3*b*)	123 (1)
C(91*a*)—O(91*a*)—C(92*a*)	115	N(2*b*)—C(2*b*)—N(3*b*)	120 (1)
N(1*a*)—C(2*a*)—N(2*a*)	116 (2)	N(3*b*)—C(4*b*)—N(9*b*)	127 (1)
N(1*a*)—C(2*a*)—N(3*a*)	122 (2)	N(3*b*)—C(4*b*)—C(5*b*)	127 (1)
N(2*a*)—C(2*a*)—N(3*a*)	121 (2)	N(9*b*)—C(4*b*)—C(5*b*)	107 (1)
N(3*a*)—C(4*a*)—N(9*a*)	129 (2)	N(7*b*)—C(5*b*)—C(4*b*)	107 (1)
N(3*a*)—C(4*a*)—C(5*a*)	131 (2)	N(7*b*)—C(5*b*)—C(6*b*)	133 (1)
N(9*a*)—C(4*a*)—C(5*a*)	101 (2)	C(4*b*)—C(5*b*)—C(6*b*)	120 (1)
N(7*a*)—C(5*a*)—C(4*a*)	112 (2)	N(1*b*)—C(6*b*)—O(6*b*)	122 (1)
N(7*a*)—C(5*a*)—C(6*a*)	133 (2)	N(1*b*)—C(6*b*)—C(5*b*)	111 (1)
C(4*a*)—C(5*a*)—C(6*a*)	116 (2)	O(6*b*)—C(6*b*)—C(5*b*)	127 (1)
N(1*a*)—C(6*a*)—O(6*a*)	122 (2)	N(7*b*)—C(8*b*)—N(9*b*)	111 (1)
N(1*a*)—C(6*a*)—C(5*a*)	111 (2)	N(9*b*)—C(91*b*)—O(91*b*)	114 (2)
O(6*a*)—C(6*a*)—C(5*a*)	127 (2)	O(91*b*)—C(92*b*)—C(93*b*)	117
N(7*a*)—C(8*a*)—N(9*a*)	111 (2)	O(93*b*)—C(93*b*)—C(92*b*)	78
O(1*w*)···N($10^{\rm i}$)	2.95 (4)	N($2b^{\rm iii}$)···Cl(3)	3.28 (2)
O(1*w*)···N(11)	3.00 (4)	N($2b^{\rm iv}$)···N(3*a*)	2.93 (3)
O($2w^{\rm ii}$)···Cl(2)	3.09 (3)	N($10^{\rm v}$)···Cl(1)	3.29 (2)
O(2*w*)···Cl(3)	3.06 (4)	N(10)···Cl(1)	3.42 (1)
N(1*a*)···O(2*w*)	2.88 (3)	N(10)···O($6b^{\rm vi}$)	2.96 (2)
N($1b^{\rm iii}$)···Cl(3)	3.13 (2)	N(11)···Cl(1)	3.27 (2)
N(2*a*)···O(2*w*)	2.91 (4)	N($11^{\rm vii}$)···Cl(1)	3.31 (2)
N(2*a*)···N($3b^{\rm iv}$)	3.04 (4)	N(11)···O($6a^{\rm ii}$)	2.94 (3)

Symmetry codes: (i) $-y, x-y, \frac{1}{3}+z$; (ii) $-x, -x+y, \frac{1}{3}-z$; (iii) $x, y, z-1$; (iv) $-x+y, 1-x, z-\frac{1}{3}$; (v) $-x+y, -x, z-\frac{1}{3}$; (vi) $y, x, 1-z$; (vii) $x-y, -y, \frac{2}{3}-z$.

A scan width of $(0.9 + 0.3\tan\theta)^\circ$ was used. Background counts were recorded for one quarter of the scan on each side of a reflection. Intensity controls were checked every 20000 s of scanning time and orientation controls were checked every 600 reflections.

The data were corrected for crystal decay, and for absorption using an analytical method (de Meulenaer & Tompa, 1965). The Pt atom was located from a Patterson map and the positions of the remaining non-H atoms were found in electron density maps. The H atoms of the guanine rings and the methylene groups were placed at calculated positions; the remaining H atoms were not located.

All observed reflections [$I > 2.5\sigma(I)$] as well as those unobserved relfections for which $F_c > F_o$ were used in the refinement.

Full-matrix least-squares refinement with anisotropic displacement parameters for all non-H atoms with the exception of atoms C(92*a*), C(93*a*), O(93*a*), C(92*b*), C(93*b*), O(93*b*) and O(1*w*) was employed. These last atoms were included in the structure-factor calculations with isotropic displacement parameters. The parameters for O(1*w*) were also refined, but the ends of the (2-hydroxyethoxy)methyl groups in the ACV ligands were disordered and the refinement of population parameters for the atoms in these fragments was unsuccessful. Hence, the parameters for atoms C(92*a*), C(93*a*), O(93*a*), C(92*b*), C(93*b*) and O(93*b*) were not refined. H atoms were included in the structure-factor calculations, but were not refined. Weights were calculated according to $w = 6w_f w_s$, where $w_f(F_o < 35) = (F_o/35)^{1.3}$, $w_f(F_o > 92) = (92/F_o)^{1.3}$ and $w_f(35 \le F_o \le 92)$

= 1.0, and $w_s(\sin\theta < 0.543) = (\sin\theta/0.543)^{2.5}$ and $w_s(\sin\theta \geq 0.543) = 1.0$.

Data collection and cell refinement were carried out using *CAD*-4 *Software* (Enraf-Nonius, 1989). The *Xtal*3.0 system (Hall & Stewart, 1990) was used for correlation and reduction of data, structure refinement and interpretation. Calculations were performed on VAX 8550 computers at the University Computer Centre, Ljubljana. *ORTEP*II (Johnson, 1976) was used for molecular graphics.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1142). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Nine-Membered Metallacyclic Metalloxycarbene Vanadium Complex

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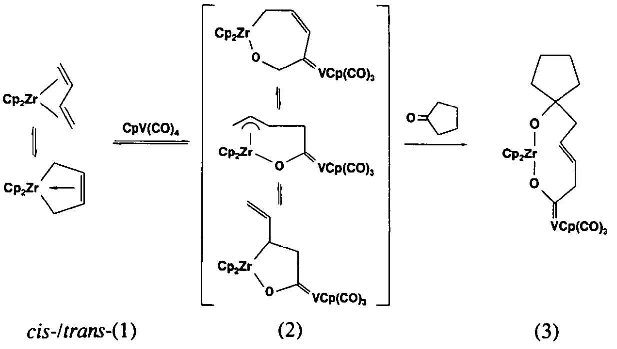
Abstract

The title compound, tricarbonyl-2κ^3C-tris[1,1,2(η^5)-cyclopentadienyl][μ-1-hydroxy-5-(1-hydroxycyclopentyl)-pent-3-en-1-ylidene(4−)-1κ^2O,O':2κC^1]vanadiumzirconium–toluene (1/0.5), $[\{Zr(C_5H_5)_2\}(\mu\text{-}C_{10}H_{14}O_2)\text{-}\{V(CO)_3(C_5H_5)\}].0.5C_7H_8$, is a carbene complex of the *d*-block elements and has an important role in organometallic chemistry. Only a few carbene–vanadium complexes have been characterized by X-ray diffraction so far. X-ray structure analysis showed that the central nine-membered metallacyclic framework contains a *trans*-configured C=C double bond between C(6) and C(7).

Comment

Carbene complexes of *d*-block elements are finding increasing application in stoichiometric organic synthesis and catalysis (Casey & Alper, 1976; Brown, 1980; Dötz, 1984; Dötz *et al.*, 1983). The new carbene synthesis described by Berlekamp & Erker (1993) makes use of the high reactivity of alkene–metallocene complexes and the high tendency of the Group IV elements to form thermodynamically favourable *M*—O bonds. Thus, (butadiene)ZrCp$_2$ (Cp = cyclopentadienyl) complexes react with metal carbonyls such as CpV(CO)$_4$ as shown in the scheme below. Subsequent addition of cyclopentanone converts the intermediate complex into the nine-membered metallacyclic metalloxycarbene vanadium complex (3), the structure of which is described here.



cis-/trans-(1) (2) (3)

(I)

The title complex is isostructural with the corresponding hafnium metalloxycarbene vanadium complex, the X-ray structure of which was determined previously as it shows greater thermodynamic stability. The central nine-membered metallacyclic framework of the zirconocene complex contains a *trans*-configured C=C double bond between C(6) and C(7) [1.312 (3) Å]. The adjacent C(5)—C(6) [1.493 (3) Å] and C(7)—C(8) [1.488 (3) Å] bond lengths are characteristic of C_{sp^2}—C_{sp^3} single-bond distances (Berlekamp & Erker, 1993; Allen *et al.*, 1987). These distances are equal, to within one e.s.d., to the bond lengths found in the corresponding hafnium complex. This arrangement of the *trans*-C=C bond inside the rigid nine-membered metallacycle means that the complex is chiral (Berlekamp & Erker, 1993). The Zr—O bonds are rather short at 2.084 (2) Å

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