

O1	-0.3633 (6)	0.7271 (5)	0.4145 (3)	0.083 (2)
C2	-0.2636 (7)	0.6859 (6)	0.2110 (4)	0.058 (2)
O2	-0.3155 (6)	0.6300 (5)	0.1923 (4)	0.089 (2)
C3	-0.3358 (8)	0.9024 (7)	0.2093 (5)	0.071 (2)
O3	-0.4252 (7)	0.9783 (6)	0.1871 (5)	0.123 (3)
C4	-0.1321 (7)	0.8777 (5)	0.2885 (4)	0.057 (2)
O4	-0.1015 (6)	0.9380 (5)	0.3167 (4)	0.095 (2)
C5	0.1706 (8)	0.8153 (6)	0.1648 (4)	0.060 (2)
O5	0.2283 (7)	0.8654 (5)	0.1866 (4)	0.093 (2)
C6	0.0181 (8)	0.6383 (6)	0.0845 (4)	0.058 (2)
O6	-0.0160 (7)	0.5803 (5)	0.0567 (3)	0.086 (2)
C7	0.2695 (8)	0.6299 (6)	0.1062 (4)	0.061 (2)
O7	0.3821 (6)	0.5626 (5)	0.0958 (4)	0.093 (2)
C8	0.0954 (9)	0.8259 (6)	0.0254 (4)	0.067 (2)
O8	0.1018 (8)	0.8853 (5)	-0.0368 (3)	0.099 (2)
C11	0.1293 (5)	0.6532 (5)	0.3374 (3)	0.037 (1)
C12	0.2937 (6)	0.5858 (6)	0.3202 (4)	0.056 (2)
C13	0.3755 (7)	0.5981 (7)	0.3827 (5)	0.073 (2)
C14	0.3026 (8)	0.5636 (8)	0.4722 (5)	0.081 (2)
C15	0.1405 (7)	0.6294 (7)	0.4894 (4)	0.067 (2)
C16	0.0579 (7)	0.6181 (6)	0.4263 (4)	0.054 (2)
C21	0.1973 (7)	0.2465 (6)	0.1476 (5)	0.066 (2)
C22	0.3333 (10)	0.2796 (9)	0.1266 (6)	0.100 (3)
C23	0.4120 (10)	0.2754 (8)	0.0358 (5)	0.094 (3)
C24	0.4285 (12)	0.1709 (10)	0.0056 (6)	0.117 (4)
C25	0.2967 (10)	0.1373 (9)	0.0266 (6)	0.107 (3)
C26	0.2108 (7)	0.1386 (5)	0.1173 (4)	0.056 (2)
C311	0.1376 (13)	0.1679 (9)	0.3477 (8)	0.042 (3)
C32	0.1666 (9)	0.0455 (5)	0.3384 (5)	0.072 (2)
C331	0.2074 (19)	-0.0294 (15)	0.4229 (11)	0.081 (5)
C341	0.3417 (18)	-0.0081 (13)	0.4434 (11)	0.076 (4)
C35	0.3055 (10)	0.1087 (8)	0.4493 (5)	0.093 (3)
C361	0.2689 (13)	0.1911 (10)	0.3690 (7)	0.044 (3)
C312	0.1940 (14)	0.1566 (10)	0.3165 (8)	0.043 (3)
C332	0.2655 (17)	-0.0451 (12)	0.3904 (9)	0.066 (4)
C342	0.2713 (22)	-0.0060 (14)	0.4672 (10)	0.079 (5)
C362	0.2062 (16)	0.1938 (11)	0.3941 (8)	0.057 (3)
C41	-0.0968 (6)	0.2460 (5)	0.2518 (4)	0.0431 (13)
C42	-0.2083 (7)	0.2709 (7)	0.3327 (4)	0.069 (2)
C43	-0.3615 (8)	0.2585 (9)	0.3308 (6)	0.100 (3)
C44	-0.4228 (8)	0.3331 (8)	0.2584 (6)	0.090 (3)
C45	-0.3155 (7)	0.3080 (8)	0.1777 (6)	0.086 (3)
C46	-0.1633 (6)	0.3202 (6)	0.1768 (4)	0.059 (2)
H1	-0.099 (7)	0.829 (6)	0.152 (4)	0.080

Table 2. Selected geometric parameters (Å, °)

Au—P2	2.3197 (14)	Mn1—H1	1.69 (8)
Au—P1	2.3217 (14)	Mn2—P1	2.334 (2)
Mn1—P1	2.334 (2)	Mn2—H1	1.86 (10)
Mn1—Mn2	2.9332 (13)		
P2—Au—P1	171.12 (5)	C11—P1—Mn1	115.3 (2)
P1—Mn1—Mn2	51.08 (4)	Au—P1—Mn1	123.23 (6)
P1—Mn2—Mn1	51.06 (4)	C11—P1—Mn2	115.0 (2)
Mn1—H1—Mn2	111 (4)	Au—P1—Mn2	112.89 (6)
C11—P1—Au	109.3 (2)	Mn1—P1—Mn2	77.86 (5)

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(triethylammonium) Bis(2-mercaptopbenzoato-*O,S*)dioxomolybdate(VI)

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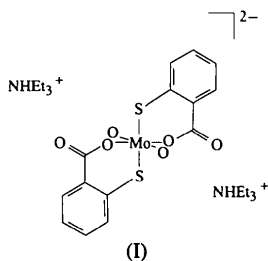
Abstract

Dioxobis(2,4-pentanedionato)molybdenum reacts with mercaptobenzoic acid neutralized with triethylamine in methanol to yield the ionic compound bis(triethylammonium) (*OC*-6-12)-bis[2-mercaptopbenzoato(2-)]-dioxomolybdate(VI), (C₆H₁₆N)₂[MoO₂(C₇H₄O₂S)₂]. This compound has been characterized by conventional spectroscopic methods and the results of the X-ray crystal structure analysis show the presence of a dianionic [MoO₂(SC₆H₄CO₂)₂]²⁻ complex and two Et₃NH⁺ cations.

Comment

The synthesis of the title compound, (I), was undertaken as part of our current research (Carrillo, Gouzerh & Jeannin, 1985; Bustos *et al.* 1991; Carrillo, Robert

& Gouzerh, 1992; Debenjak, Bustos, Carrillo, Robert & Gouzerh, 1992*a,b*; Li-Kao, Bustos, Carrillo, Robert & Gouzerh, 1993). The purpose was to obtain a new precursor for the preparation of molybdenum complexes containing organo-dinitrogen ligands, for use as models of the postulated intermediates in the conversion of coordinated dinitrogen into ammonia (Henderson, Leigh & Pickett, 1983; Leigh, 1992).



The complex is dianionic in nature (Fig. 1) and shows monomeric units composed of two mercaptobenzoate ligands coordinated to a *cis*- MoO_2 core through atoms S1, S2, O3 and O5. The coordination sphere around the Mo^{VI} ion is best described as a slightly distorted octahedron, where the basal plane is defined by Mo—O1 [1.700 (4) Å], Mo—O2 [1.699 (4) Å], Mo—O3 [2.140 (4) Å] and Mo—O5 [2.152 (4) Å]; r.m.s. deviations from the least-squares plane average 0.17 Å. The apical sites are occupied by atoms S1 and S2 of the organic anion, 5° away from the vertical due to the strain imposed by the close chelate structure. The anion presents a non-crystallographic twofold axis which bisects the O1—Mo—O2 and O3—Mo—O5 angles. These structural characteristics are similar

to those found in closely related compounds, mainly in $(NH_4)_2[MoO_2\{O_2CC(S)Ph_2\}_2] \cdot 2H_2O$ (Palanca *et al.*, 1990). The structure is completed by two Et_3NH^+ cations, the geometry of which posed some problems in the early stages of refinement. A soft similarity restraint applied to the C—C and C—N bond lengths was enough to stabilize the convergence. The resulting geometry was fairly regular, showing a strong coupling to the anion through two rather short hydrogen-bonded contacts (Fig. 1) (O4...H1A—N1, estimated length/angle 1.86 Å/175°; O6...H2A—N2, estimated length/angle 1.80 Å/171°), leading to somewhat independent neutral units connected to one another only through coulombian interactions (Fig. 2).

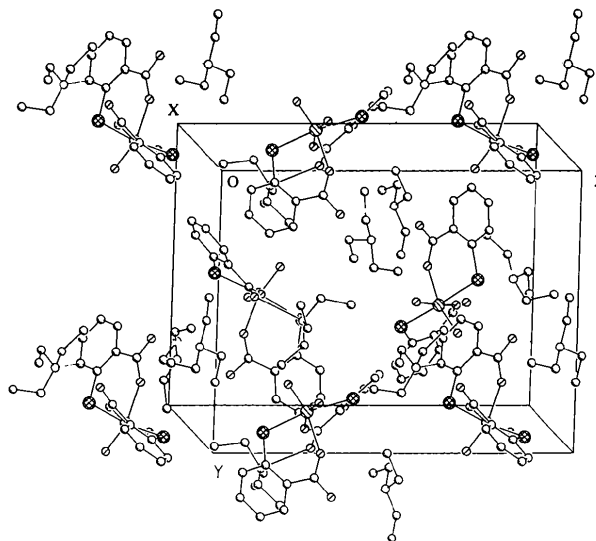


Fig. 2. Packing diagram showing the molecular arrangement and hydrogen-bond interactions within a unit cell.

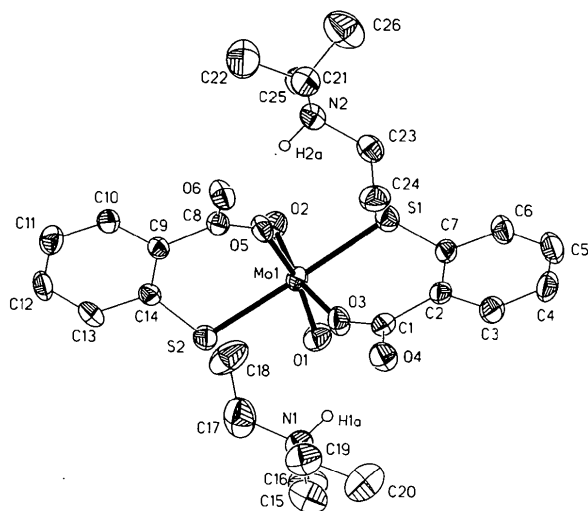


Fig. 1. View of the title molecule showing the atom-labelling scheme. Displacement ellipsoids are shown at the 40% probability level. H atoms are omitted for clarity, except for those involved in strong hydrogen bonds.

Experimental

The title compound was prepared by reaction of 10 mmol of $[MoO_2(acac)_2]$, where *acac* is acetylacetonato, in 25 ml of methanol with 20 mmol of triethylammonium mercaptobenzoate, $[NHEt_3]_2[SC_6H_4CO_2]$, obtained by neutralization of 20 mmol of mercaptobenzoic acid with 40 mmol of triethylamine in 25 ml of methanol. The reaction mixture was stirred for 1 h at room temperature. The crystalline solid that precipitated was filtered off, then washed with cold methanol and diethyl ether. Well formed orange crystals suitable for X-ray diffraction were obtained from a dichloromethane–hexane solution, the characterization of which gave: m.p. 421 K; 86.3% yield; calculated for $C_{26}H_{40}MoN_2O_6S_2$: C 49.05, H 6.44%; found: C 49.01, H 6.57%; IR (KBr): 1590 (s), 1570 (s), 1535 (s), 920 (s), 880 (s), 760 cm^{-1} (s); 1H NMR (DMSO) δ : 1.19 (t), 3.10 (q), 7.09 (m), 8.00 p.p.m. (m).

Crystal data

$(C_6H_{16}N)_2[MoO_2(C_7H_4O_2S)_2]$ Mo $K\alpha$ radiation
 $M_r = 636.66$ $\lambda = 0.71073$ Å

Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	$\theta = 7.5\text{--}12.5^\circ$
$a = 11.832 (3) \text{ \AA}$	$\mu = 0.616 \text{ mm}^{-1}$
$b = 14.050 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 18.049 (3) \text{ \AA}$	Prismatic
$V = 3000.5 (11) \text{ \AA}^3$	$0.46 \times 0.28 \times 0.20 \text{ mm}$
$Z = 4$	Orange
$D_x = 1.409 \text{ Mg m}^{-3}$	

C18	0.5264 (7)	0.6474 (7)	-0.0065 (5)	0.111 (4)
C19	0.4286 (6)	0.8127 (4)	-0.0886 (5)	0.095 (3)
C20	0.3646 (4)	0.9049 (4)	-0.0851 (4)	0.114 (3)
C21	0.6640 (4)	0.6698 (4)	0.3372 (4)	0.068 (2)
C22	0.7666 (6)	0.6071 (7)	0.3289 (6)	0.118 (4)
C23	0.4658 (5)	0.6980 (4)	0.3043 (4)	0.063 (2)
C24	0.4856 (7)	0.7693 (5)	0.2434 (4)	0.077 (2)
C25	0.5214 (8)	0.5407 (4)	0.3525 (3)	0.084 (2)
C26	0.4948 (11)	0.5587 (7)	0.4329 (4)	0.124 (4)

Data collection

Siemens R3m diffractometer diffractometer	2273 observed reflections
$\theta/2\theta$ scans (4.19–29.3° min ⁻¹)	$[I > 2\sigma(I)]$
Absorption correction: semi-empirical	$\theta_{\text{max}} = 24.04^\circ$
$T_{\text{min}} = 0.72$, $T_{\text{max}} = 0.94$	$h = 0 \rightarrow 13$
2680 measured reflections	$k = 0 \rightarrow 16$
2680 independent reflections	$l = 0 \rightarrow 20$
	2 standard reflections monitored every 98 reflections
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.035$
$R(F) = 0.0330$	$\Delta\rho_{\text{max}} = 0.325 \text{ e \AA}^{-3}$
$wR(F^2) = 0.0879$	$\Delta\rho_{\text{min}} = -0.356 \text{ e \AA}^{-3}$
$S = 1.044$	Atomic scattering factors from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
2680 reflections	
335 parameters	
H atoms refined as a riding model	
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 1.2564P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$				
	x	y	z	U_{eq}
Mo1	0.24870 (5)	0.47995 (3)	0.14600 (3)	0.0451 (2)
S1	0.18931 (15)	0.56099 (12)	0.25962 (9)	0.0570 (4)
S2	0.33665 (14)	0.41886 (12)	0.03158 (9)	0.0547 (4)
N1	0.3612 (4)	0.7306 (4)	-0.0637 (3)	0.0602 (14)
N2	0.5575 (4)	0.6267 (3)	0.3109 (3)	0.0530 (13)
O1	0.1253 (4)	0.4709 (4)	0.0982 (3)	0.0681 (13)
O2	0.2492 (5)	0.3821 (3)	0.2013 (2)	0.0659 (11)
O3	0.2688 (4)	0.6233 (3)	0.1082 (2)	0.0608 (12)
O4	0.2966 (4)	0.7722 (3)	0.0794 (2)	0.0654 (13)
O5	0.4225 (3)	0.5093 (3)	0.1729 (2)	0.0561 (11)
O6	0.6024 (4)	0.5483 (3)	0.1780 (2)	0.0652 (13)
C1	0.2574 (6)	0.7099 (4)	0.1220 (3)	0.0471 (13)
C2	0.1917 (5)	0.7417 (4)	0.1886 (3)	0.0454 (14)
C3	0.1619 (6)	0.8383 (4)	0.1909 (4)	0.059 (2)
C4	0.0925 (7)	0.8739 (6)	0.2467 (5)	0.076 (2)
C5	0.0524 (7)	0.8136 (6)	0.3005 (5)	0.076 (2)
C6	0.0840 (6)	0.7196 (5)	0.3002 (4)	0.062 (2)
C7	0.1541 (5)	0.6821 (4)	0.2453 (3)	0.0477 (14)
C8	0.5239 (5)	0.4998 (4)	0.1511 (3)	0.0469 (15)
C9	0.5542 (5)	0.4249 (4)	0.0959 (3)	0.0412 (13)
C10	0.6654 (5)	0.3918 (5)	0.0967 (3)	0.052 (2)
C11	0.7024 (6)	0.3227 (5)	0.0484 (4)	0.066 (2)
C12	0.6274 (6)	0.2834 (5)	-0.0007 (4)	0.061 (2)
C13	0.5159 (6)	0.3150 (5)	-0.0036 (3)	0.056 (2)
C14	0.4787 (5)	0.3858 (4)	0.0454 (3)	0.0416 (13)
C15	0.2616 (6)	0.7148 (6)	-0.1116 (4)	0.098 (3)
C16	0.1850 (8)	0.6356 (6)	-0.0865 (5)	0.102 (3)
C17	0.4311 (7)	0.6434 (5)	-0.0623 (5)	0.107 (3)

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

Mo1—O2	1.699 (4)	C2—C7	1.395 (8)
Mo1—O1	1.700 (4)	C2—C3	1.403 (9)
Mo1—O3	2.140 (4)	C3—C4	1.392 (10)
Mo1—O5	2.152 (4)	C4—C5	1.374 (11)
Mo1—S1	2.449 (2)	C5—C6	1.373 (10)
Mo1—S2	2.467 (2)	C6—C7	1.396 (9)
S1—C7	1.771 (6)	C8—C9	1.492 (8)
S2—C14	1.761 (6)	C9—C10	1.390 (8)
N1—C19	1.473 (8)	C9—C10	1.396 (8)
N1—C17	1.478 (9)	C10—C11	1.376 (9)
N1—C15	1.479 (9)	C11—C12	1.371 (10)
N2—C21	1.477 (7)	C12—C13	1.394 (9)
N2—C23	1.481 (7)	C13—C14	1.403 (8)
N2—C25	1.485 (8)	C15—C16	1.505 (10)
O3—C1	1.250 (7)	C17—C18	1.514 (10)
O4—C1	1.254 (7)	C19—C20	1.502 (10)
O5—C8	1.270 (7)	C21—C22	1.506 (10)
O6—C8	1.251 (7)	C23—C24	1.505 (10)
C1—C2	1.499 (8)	C25—C26	1.507 (10)
O2—Mo1—O1	103.9 (3)	C7—C2—C1	125.1 (5)
O2—Mo1—O3	161.5 (2)	C3—C2—C1	116.3 (6)
O1—Mo1—O3	90.2 (2)	C4—C3—C2	121.2 (7)
O2—Mo1—O5	91.1 (2)	C5—C4—C3	119.6 (7)
O1—Mo1—O5	161.6 (2)	C6—C5—C4	119.8 (7)
O3—Mo1—O5	77.6 (2)	C5—C6—C7	121.9 (7)
O2—Mo1—S1	83.4 (2)	C2—C7—C6	119.0 (6)
O1—Mo1—S1	102.3 (2)	C2—C7—S1	127.5 (5)
O3—Mo1—S1	82.02 (11)	C6—C7—S1	113.5 (5)
O5—Mo1—S1	89.77 (11)	O6—C8—O5	121.6 (5)
O2—Mo1—S2	102.1 (2)	O6—C8—C9	117.7 (5)
O1—Mo1—S2	84.9 (2)	O5—C8—C9	120.5 (5)
O3—Mo1—S2	90.80 (11)	C14—C9—C10	118.8 (5)
O5—Mo1—S2	81.53 (11)	C14—C9—C8	124.2 (5)
S1—Mo1—S2	169.76 (6)	C10—C9—C8	117.0 (5)
C7—S1—Mo1	113.1 (2)	C11—C10—C9	121.9 (6)
C14—S2—Mo1	112.1 (2)	C12—C11—C10	119.2 (6)
C19—N1—C17	110.6 (5)	C11—C12—C13	120.6 (6)
C19—N1—C15	111.7 (5)	C12—C13—C14	119.9 (6)
C17—N1—C15	109.4 (6)	C9—C14—C13	119.5 (6)
C21—N2—C23	112.0 (4)	C9—C14—S2	127.0 (4)
C21—N2—C25	114.6 (5)	C13—C14—S2	113.4 (5)
C23—N2—C25	112.4 (5)	N1—C15—C16	114.5 (6)
C1—O3—Mo1	147.3 (4)	N1—C17—C18	113.4 (7)
C8—O5—Mo1	144.3 (4)	N1—C19—C20	113.0 (5)
O3—C1—O4	121.2 (6)	N2—C21—C22	114.6 (5)
O3—C1—C2	120.4 (5)	N2—C23—C24	113.3 (5)
O4—C1—C2	118.4 (5)	N2—C25—C26	114.2 (6)
C7—C2—C3	118.6 (6)		

The structure was solved through a combination of direct methods and difference Fourier synthesis using the *SHELXTL/PC* package (Sheldrick, 1991). Refinement was performed with *SHELXL93* (Sheldrick, 1993) using the whole data set. H atoms were included at their expected positions with fixed displacement parameters.

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991). Cell refinement: *P3/P4-PC Diffractometer Program*. Data reduction: *SHELXTL/PC XDISK*. Program(s) used to solve structure: *SHELXTL/PC XS*. Molecular graphics: *SHELXTL/PC XP*. Software used to prepare material for publication: *SHELXL93 CIFTAB*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including torsion angles and contact distances, have been deposited with the IUCr (Reference: BK1032). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$[(\eta^6-C_6H_3Me_3)Ru(D-pen)]_2Cl_2 \cdot 4H_2O$

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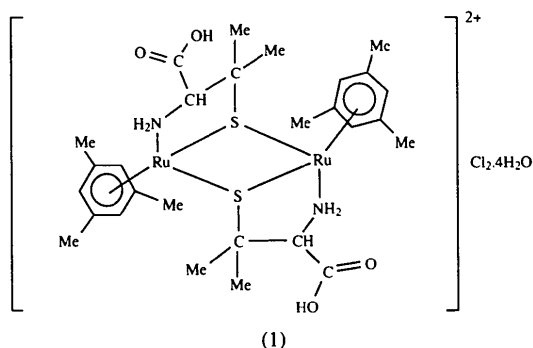
Abstract

The structure of the title compound, bis[μ -D-3-mercaptopivalinato(1-)]- $1\kappa N, 1:2\kappa^2 S; 2\kappa N, 1:2\kappa^2 S$ -bis[$(\eta^6$ -mesitylene)ruthenium] dichloride tetrahydrate, $[Ru_2(C_5-$

$H_{10}NO_2S)_2(\eta^6-C_9H_{12})_2]Cl_2 \cdot 4H_2O$, contains tridentate anionic amino acid ligands, where the S atoms adopt bridging positions between the two Ru atoms resulting in a near planar Ru—S—Ru—S ring. The coordination at each Ru atom is completed by an amino N atom of the chelating ligand and the η^6 -bonded mesitylene ring.

Comment

The determination of the structure of the title compound (1) was undertaken as part of a study of organometallic complexes with biologically important ligands (Carter, Davies, Fawcett & Russell, 1993). In particular, we have studied arene-ruthenium half-sandwich complexes, including amino acid complexes [(arene)Ru(aa)Cl] (aa = an anionic amino acid ligand) (Carter, Davies, Duffy, Fawcett & Russell, 1994) which contain a chiral metal centre.



Compound (1) is analogous to the η^6 -benzene-ruthenium(II)-l-penicillamine complex (2) reported by Sheldrick & Heeb (1989), the most significant difference being that compound (1) has four water molecules associated with each dimeric cation, where only one was found for (2). The central four-membered Ru—S—Ru—S ring, while essentially planar (Fig. 1) with deviations from the best least-squares plane of -0.15 for Ru(1), -0.15 for Ru(2), 0.38 for S(1) and 0.38 Å for S(2), is less so than that found by Sheldrick & Heeb (maximum deviation 0.009 Å). The Ru—S distances in the five-membered chelate rings [2.335 (3), 2.348 (3) Å] are shorter than the bridging Ru—S distances [2.394 (2), 2.401 (2) Å], as found for (2). The chelating rings have a *cis* arrangement which permits both chloride ions to form hydrogen bonds between the —OH group of one ligand and the —NH₂ group of the other. The O...Cl (2.874 , 2.917 Å) and the N...Cl (3.249 , 3.253 Å) distances are comparable with the distances found for a similar arrangement in compound (2). The chloride ions have further hydrogen-bond contacts with water molecules: Cl(1)...O(7) (3.273 Å) and Cl(2)...O(8) (3.235 Å) (Fig. 2). None of the H atoms of the —NH₂ or —OH groups or those of the water molecules were located with any certainty and were