Journal of Organometallic Chemistry, 241 (1983) 77–86
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

DIOLEFIN CATIONIC RHODIUM COMPLEXES WITH SULFUR DONORS. X-RAY STRUCTURE OF $[Rh(NBD)_2(SEt_2)]ClO_4$

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(Received July 23rd, 1982)

Summary

The preparation and properties of cationic rhodium complexes of the type $[Rh(diolefin)L_2]ClO_4$ (diolefin = COD, NBD; L = sulfur donor ligand) are described. Pentacoordinated complexes of general formula $[Rh(NBD)_2L]ClO_4$ (L = SMe_2, SEt_2, tetrahydrothiophen or trimethylene sulfide) are also reported. The crystal structure of $[Rh(NBD)_2(SEt_2)]ClO_4$ has been determined by X-ray methods. The crystals are rhombohedral, space group $R3c$, with a 14.530(7) Å and α 77.86(5)° ($Z = 6$). The structure was solved from diffractometer data by direct and Fourier methods and refined by full-matrix least-squares to $R = 0.054$ for 937 independent observed reflections. In the cationic $[Rh(NBD)_2(SEt_2)]^+$ complex the Rh atom is pentacoordinated, the geometry about the metal being a square pyramid whose base is defined by the midpoints of the olefin bonds from two norbornadiene molecules and the apex is occupied by a sulfur atom of the thioether ligand at a rather long distance [Rh–S 2.500(4) Å].

Introduction

A large number of cationic rhodium(I) complexes of the type $[Rh(diolefin)L_2]$ A have been reported [1]. Most of the isolated complexes contain nitrogen or phosphorous donor ligands, but a few containing oxygen [2–5] or sulfur [5–8] donor ligands have been described. In particular, Ainscough et al. [6] have reported some cationic complexes with phosphine sulfides. On the other hand, Maitlis et al. [7] prepared some related sulfur bonded complexes with 1,4-dithiacyclohexane, but

0022-328X/83/0000–0000/$03.00

when 2,5-dimethylthiophen or tetramethylthiophen were used as ligands, the isolated complexes were of the $[Rh(diolefin)(\eta^5\text{-}L)]PF_6$ type. The latter complexes were active catalysts for hydrogenation of olefins, but rapidly deposited rhodium metal [8]. Only a few related neutral rhodium complexes have been prepared [9–11].

In this paper we report the preparation of some cationic complexes of general formula $[Rh(diolefin)L_2]ClO_4$ and $[Rh(NBD)_2L]ClO_4$ (diolefin = 2,5-norbornadiene (NBD) or 1,5-cyclooctadiene (COD); L = sulfur donor ligand), along with the crystal structure of the pentacoordinated complex $[Rh(NBD)_2(SEt_2)]ClO_4$.

Results and discussion

$[Rh(diolefin)L_2]Clo_4$ complexes can be prepared by addition of $AgClO_4$ to dichloromethane solutions of $[RhCl(diolefin)]_2$ (diolefin = COD [12] or NBD [13]) and the corresponding sulfur ligand:

$$\tfrac{1}{2}[RhCl(diolefin)]_2 + 2\,L + AgClO_4 \rightarrow [Rh(diolefin)L_2]ClO_4 + AgCl \qquad (1)$$

(L = tetrahydrothiophen (tht), trimethylene sulfide (tms), SMe_2, SEt_2; L_2 = 1,4-dithiacyclohexane (dt), $(MeS)_2(CH_2)_3$ and $(t\text{-}BuS)_2(CH_2)_2$).

An alternative route which we used for the preparation of $[Rh(diolefin)L_2]ClO_4$ complexes, when L = tht, tms; L_2 = dt, $(MeS)_2(CH_2)_3$ and $(t\text{-}BuS)_2(CH_2)_2$, involves the addition to dichloromethane solutions of $[Rh(diolefin)_2]ClO_4$ [14] complexes of a slightly more than stoichiometric amount of the corresponding monodentate or bidentate sulfur ligand:

$$[Rh(diolefin)_2]ClO_4 + 2\,L \rightarrow [Rh(diolefin)L_2]ClO_4 + diolefin \qquad (2)$$

The yellow tht derivatives are stable in air at room temperature, but in all the other cases the products decompose in air. Elemental analyses, molar conductivities in acetone, (1/1 electrolytes) and yields of the isolated complexes are collected in Table 1.

It is noteworthy that the reaction of $[Rh(COD)_2]ClO_4$ with SEt_2 affords a complex of formula $[Rh(COD)(SEt_2)]_n(ClO_4)_n$. Attempts to establish its nuclearity by measuring conductivities at various concentrations [15] were unsuccessful, but by analogy with the related complex $[Pt_2Br_4(SEt_2)_2]$ whose structure was determined by X-ray methods [16], a dimeric structure with bridging sulfur atoms, $[(COD)Rh(\mu\text{-}SEt_2)_2Rh(COD)](ClO_4)_2$, is suggested. However, $[Rh(COD)_2]ClO_4$ reacts with SMe_2 to give the mononuclear $[Rh(COD)(SMe_2)_2]ClO_4$ derivative.

On the other hand, the addition of a stoichiometric amount or an excess of SMe_2 or SEt_2 to $[Rh(NBD)_2]ClO_4$ (3/1 ratio) gives the pentacoordinated complexes $[Rh(NBD)_2(SMe_2)]ClO_4$ and $[Rh(NBD)_2(SEt_2)]ClO_4$. In particular the crystal structure of the latter complex has been determined by X-ray methods (see next section). The related pentacoordinated complexes $[Rh(NBD)_2(tht)]ClO_4$ and $[Rh(NBD)_2(tms)]ClO_4$ were prepared by treating $[Rh(NBD)_2]ClO_4$ with a stoichiometric amount of the corresponding sulfur ligand (1/1 ratio) (Table 1). If excess of tht or tms is added, the corresponding $[Rh(NBD)L_2]ClO_4$ complexes are formed.

The π-acceptor character [17] of 2,5-norbornadiene in comparison with 1,5-cyclooctadiene favours the formation of pentacoordinated complexes. Thus, while a few pentacoordinated complexes of the type $[Rh(NBD)_2L']ClO_4$ (L′ = CO, $SbPh_3$, $AsPh_3$, $AsMe_2Ph$ [18] or PPh_3 [19]) have been isolated, only the $[Rh(COD)_2(SbPh_3)]ClO_4$ complexes has been prepared [19].

TABLE 1

ANALYTICAL RESULTS, MOLAR CONDUCTIVITIES AND YIELDS FOR THE COMPLEXES

Complex	Analysis (found (calcd.) (%))			Λ_M (ohm^{-1} cm^2 mol^{-1})	Yield (%)
	C	H	S		
$[Rh(COD)(tht)_2]ClO_4$	39.62	5.80	12.63	130	92
	(39.47)	(5.80)	(13.17)		
$[Rh(NBD)(tht)_2]ClO_4$	38.65	4.98	13.56	132	86
	(38.26)	(5.14)	(13.62)		
$[Rh(COD)(tms)_2]ClO_4$	36.65	5.35	13.94	128	84
	(36.65)	(5.27)	(13.98)		
$[Rh(NBD)(tms)_2]ClO_4$	34.63	4.76	14.29	105	70
	(35.26)	(4.55)	(14.48)		
$[Rh(COD)(SMe_2)_2]ClO_4$	33.64	5.66	14.45	138	75
	(33.12)	(5.56)	(14.65)		
$[Rh(NBD)(SMe_2)_2]ClO_4$	31.78	4.79	14.87	125	50
	(31.55)	(4.81)	(15.31)		
$[Rh(COD)(SEt_2)_2]ClO_4$	38.71	6.23	12.77	130	15
	(39.11)	(6.52)	(13.06)		
$[Rh(NBD)(SEt_2)_2]ClO_4$	38.52	5.66	–	115	15
	(37.94)	(5.94)	(13.50)		
$[Rh(COD)(dt)]ClO_4$	32.86	4.70	14.57	135	80
	(33.46)	(4.68)	(14.89)		
$[Rh(NBD)(dt)]ClO_4$	32.40	4.05	15.53	122	73
	(31.86)	(3.89)	(15.46)		
$[Rh(COD)((MeS)_2(CH_2)_3)]ClO_4$	34.26	5.32	14.34	132	85
	(34.94)	(5.41)	(14.35)		
$[Rh(NBD)((MeS)_2(CH_2)_3)]ClO_4$	32.81	5.09	–	127	68
	(33.46)	(4.68)	(14.89)		
$[Rh(COD)((t\text{-}BuS)_2(CH_2)_2)]ClO_4$	41.34	6.67	12.46	129	86
	(41.78)	(6.58)	(12.40)		
$[Rh(NBD)((t\text{-}BuS)_2(CH_2)_2)]ClO_4$	40.15	5.82	–	138	83
	(40.76)	(6.04)	(12.80)		
$[Rh(COD)(SEt_2)]_2(ClO_4)_2$	35.77	5.49	7.96	211	15
	(35.93)	(5.49)	(8.00)		
$[Rh(NBD)_2(SMe_2)]ClO_4$	41.97	4.91	7.44	135	88
	(42.82)	(4.94)	(7.14)		
$[Rh(NBD)_2(SEt_2)]ClO_4$	44.32	5.54	7.30	118	77
	(45.34)	(5.50)	(6.73)		
$[Rh(NBD)_2(tht)]ClO_4$	45.16	5.27	7.27	127	73
	(45.53)	(5.09)	(6.75)		
$[Rh(NBD)_2(tms)]ClO_4$	43.42	5.04	7.53	139	91
	(44.31)	(4.81)	(6.96)		

The IR spectra of the above complexes show bands due to the coordinated diolefins, together with those due to the corresponding sulfur ligands and to the uncoordinate perchlorate anion (1090s and 620m cm^{-1}) [20].

Description of the structure of the compound $[Rh(NBD)_2(SEt_2)]ClO_4$

The crystal structure of the compound $[Rh(NBD)_2(SEt_2)]ClO_4$ consists of monomeric cationic rhodium(I) complexes with ClO_4^- counter ions. Selected bond distances and angles in the Rh complex and in the perchlorate anion are given in Table 2. A view of the structure of the Rh complex with the atomic numbering scheme is shown in Fig. 1.

The rhodium atom is in a square pyramidal arrangement whose base is formed by the midpoints of the olefin bonds from two norbornadiene molecules and the apex is occupied by the sulfur atom of the thioether ligand. The Rh–C distances are 2.19 and 2.12 Å for one double bond and 2.24 and 2.22 Å for the other double bond in a NBD molecule; 2.24, 2.20 and 2.30, 2.26 Å are the values of the corresponding distances in the second NBD molecule. The Rh–C distances with NBD ligands vary considerably, and in the present complex they are longer than in other square-planar complexes with a unique NBD molecule [21–23]. Indicating by M(1), M(2), M(3) and M(4) the midpoints of the C(11)–C(21), C(31)–C(41), C(12)–C(22) and C(32)–C(42) bonds, the Rh–M(1), Rh–M(2), Rh–M(3) and Rh–M(4) distances are 2.04, 2.12, 2.10 and 2.17 Å respectively. The angles M(1)–Rh–M(2), M(2)–Rh–M(4), M(3)–Rh–M(4) and M(1)–Rh–M(3) in the basal coordination plane are 69.2, 108.2, 67.2 and 102.7°, respectively. A least-squares plane passing through the midpoints M(1), M(2), M(3) and M(4) shows deviations of 0.01–0.04 Å, with the Rh atom deviating by 0.48 Å from this plane towards the apex of the pyramid. The olefinic carbon atoms C(11), C(21), C(31), C(41) and C(12), C(22), C(32), C(42) in the two molecules are coplanar. The planes passing through the two groups form a dihedral angle of 29°.

The C–C distances and angles within the NBD molecules are comparable with

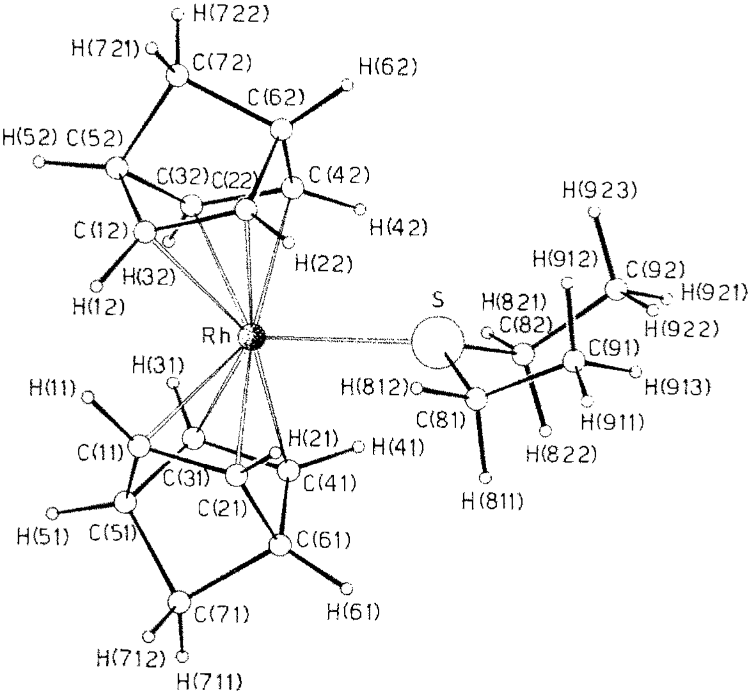

Fig. 1. View of the cationic complex $[Rh(NBD)_2(SEt_2)]^+$ with the atomic numbering scheme.

TABLE 2

SELECTED BOND DISTANCES (Å) AND ANGLES (°)

(a) around the Rh atom			
Rh–C(11)	2.19(1)	Rh–C(12)	2.24(1)
Rh–C(21)	2.12(1)	Rh–C(22)	2.20(2)
Rh–C(31)	2.24(2)	Rh–C(32)	2.30(2)
Rh–C(41)	2.22(2)	Rh–C(42)	2.26(1)
Rh–S	2.500(4)		
C(11)–Rh–C(21)	37.5(5)	C(12)–Rh–C(22)	37.5(4)
C(11)–Rh–C(31)	65.4(8)	C(12)–Rh–C(32)	62.3(6)
C(11)–Rh–C(41)	77.3(8)	C(12)–Rh–C(42)	76.7(6)
C(21)–Rh–C(31)	77.4(8)	C(22)–Rh–C(32)	74.3(8)
C(21)–Rh–C(41)	65.0(7)	C(22)–Rh–C(42)	64.4(8)
C(31)–Rh–C(41)	36.4(5)	C(32)–Rh–C(42)	35.8(4)
S–Rh–C(11)	127.7(7)	S–Rh–C(21)	125.8(6)
S–Rh–C(21)	90.2(6)	S–Rh–C(22)	88.3(8)
S–Rh–C(31)	115.1(8)	S–Rh–C(32)	116.3(6)
S–Rh–C(41)	80.5(8)	S–Rh–C(42)	81.2(7)
(b) within the norbornadiene and ethylthioether ligands			
C(11)–C(21)	1.39(2)	C(12)–C(22)	1.43(2)
C(31)–C(41)	1.39(2)	C(32)–C(42)	1.40(2)
C(11)–C(51)	1.58(2)	C(12)–C(52)	1.50(2)
C(31)–C(51)	1.55(2)	C(32)–C(52)	1.54(2)
C(21)–C(61)	1.54(2)	C(22)–C(62)	1.52(2)
C(41)–C(61)	1.54(2)	C(42)–C(62)	1.54(2)
C(51)–C(71)	1.58(2)	C(52)–C(72)	1.57(2)
C(61)–C(71)	1.57(2)	C(62)–C(72)	1.56(2)
S–C(81)	1.78(2)	C(81)–C(91)	1.55(2)
S–C(82)	1.73(2)	C(82)–C(92)	1.53(2)
C(21)–C(11)–C(51)	101(2)	C(22)–C(12)–C(52)	109(1)
C(11)–C(21)–C(61)	111(2)	C(12)–C(22)–C(62)	106(2)
C(41)–C(31)–C(51)	101(2)	C(42)–C(32)–C(52)	113(2)
C(31)–C(41)–C(61)	111(2)	C(32)–C(42)–C(62)	102(2)
C(71)–C(61)–C(21)	95(2)	C(72)–C(62)–C(22)	95(2)
C(41)–C(61)–C(21)	99(2)	C(42)–C(62)–C(22)	102(2)
C(71)–C(61)–C(41)	106(2)	C(72)–C(62)–C(42)	106(2)
C(71)–C(51)–C(11)	98(2)	C(72)–C(52)–C(12)	94(1)
C(31)–C(51)–C(11)	100(2)	C(32)–C(52)–C(12)	101(2)
C(71)–C(51)–C(31)	111(2)	C(72)–C(52)–C(32)	99(2)
C(51)–C(71)–C(61)	90(2)	C(52)–C(72)–C(62)	96(2)
C(81)–S–Rh	115(1)	C(82)–S–Rh	123(1)
C(82)–S–C(81)	114(1)	S–C(82)–C(92)	112(2)
S–C(81)–C(91)	118(2)		
(c) within the perchlorate anion			
Cl–O(1)	1.41(3)	Cl–O(3)	1.37(3)
Cl–O(2)	1.36(3)	Cl–O(4)	1.39(3)
O(1)–Cl–O(2)	109(1)	O(2)–Cl–O(3)	98(2)
O(1)–Cl–O(3)	114(2)	O(2)–Cl–O(4)	108(2)
O(1)–Cl–O(4)	117(2)	O(3)–Cl–O(4)	109(1)

those reported for several NBD complexes [24], the olefinic bonds (1.39–1.43 Å) being, as expected, longer than in free NBD molecule (1.338 Å) [25].

The Rh–S bond length, involving the apical coordination site, is rather long [2.500(4) Å]. This value can not be compared with values found for similar complexes since, while a few rhodium(I)-thioether complexes have been prepared [10,11,26], none has been fully characterized by X-ray analysis. Rh–S distances within the range 2.27–2.37 Å have been observed [27] in rhodium(I)-thiolate complexes, but the rhodium(I) was in a square planar environment; Rh–S bond lengths ranging from 2.368 to 2.450 Å have been reported [28] for Rh square pyramidal complexes with sulfur dioxide as apical ligand.

The crystal packing of the cationic rhodium complexes and of the perchlorate anions is shown in Fig. 2.

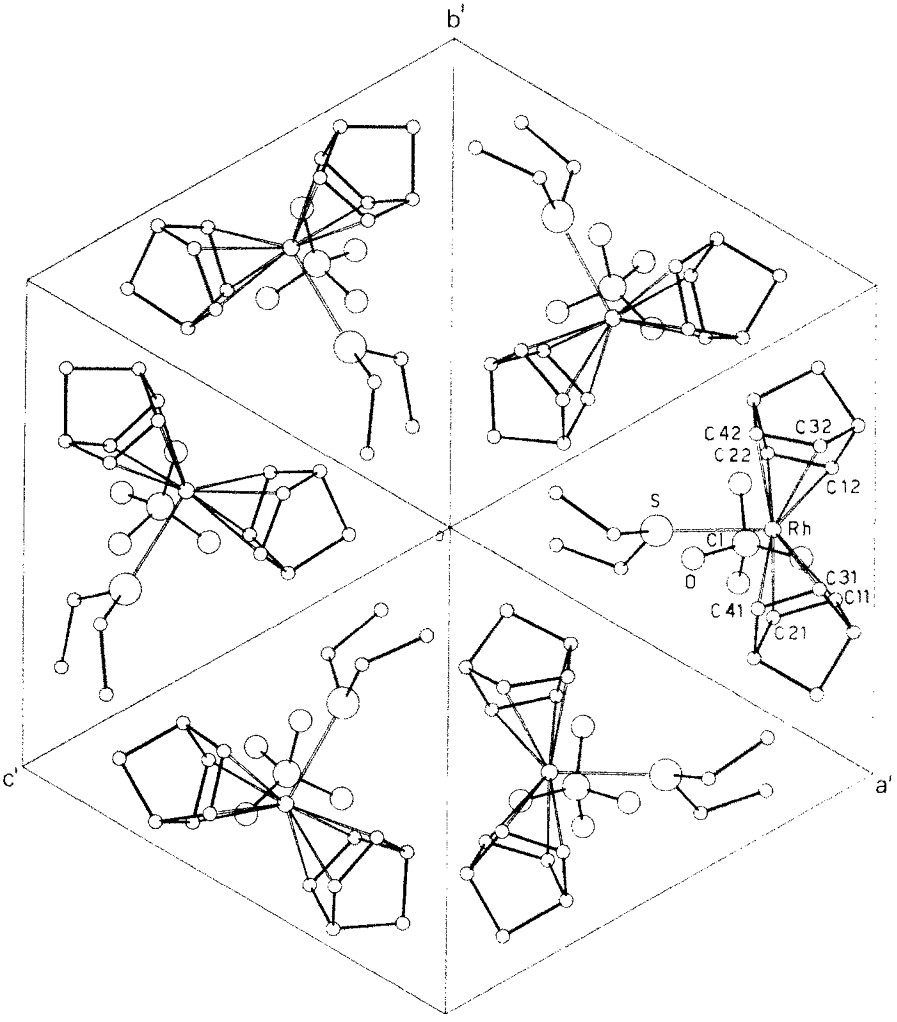

Fig. 2. Projection along [111] of the structure of the compound $[Rh(NBD)_2(SEt_2)]ClO_4$ showing the packing of the cationic complexes and of the perchlorate anions ($a' = b' = c' = a\sqrt{2/3(1-\cos\alpha)}$).

Experimental

Reactions with the tetrahydrothiophen ligand were carried out in air at room temperature, but a nitrogen atmosphere was used with all the others ligands. C, H and S analyses were carried out on a Perkin–Elmer 240B microanalyzer. The IR spectra were recorded on Beckman IR 4260 spectrophotometer using Nujol mulls between polyethylene sheets or NaCl plates. Conductivities were measured using approximately 3×10^{-4} *M* acetone solutions with a RED Copenh. CDM 3 conductimeter.

Preparation of [Rh(diolefin)L_2]ClO_4 complexes

The compounds were prepared by two routes, as described below: (i) The addition of a slight excess of ligand (0.45 mmol of L or 0.23 mmol of L_2) and the stoichiometric amount of $AgClO_4$ (0.20 mmol) to dichloromethane solutions of $[RhCl(COD)]_2$ or $[RhCl(NBD)]_2$ (0.10 mmol) produced a white precipitate of silver chloride, which was filtered off through Kieselguhr. Addition of ether to the filtrate precipitated the required complexes, which were filtered off, washed with ether, and vacuum dried. All the $[Rh(diolefin)L_2]ClO_4$ complexes can be prepared by this route.

(ii) Upon addition of slightly more than the stoichiometric amount of ligand (0.23 mmol (L), 0.12 mmol (L_2)) to dichloromethane solutions of $[Rh(COD)_2]ClO_4$ or $[Rh(NBD)_2]ClO_4$ (0.09 mmol) an immediate reaction was observed. The resulting complexes were precipitated by adding ether, and were filtered off, washed with ether and vacuum dried.

All the $[Rh(diolefin)L_2]ClO_4$ complexes were prepared by this route except $[Rh(COD)(SEt_2)_2]ClO_4$, $[Rh(NBD)(SMe_2)_2]ClO_4$ and $[Rh(NBD)(SEt_2)_2]ClO_4$.

Preparation of $[Rh(COD)(SEt_2)]_n(ClO_4)_n$

Addition of an excess of SEt_2 (21 mg, 0.23 mmol) to dichloromethane solutions of $[Rh(COD)_2]ClO_4$ (31 mg, 0.07 mmol), produced an immediate colour change. Subsequent addition of ether precipitated out the desired complex, which was filtered off, washed with cold ether, and vacuum dried (yield 15%).

Preparation of $[Rh(NBD)_2L]ClO_4$ complexes

Upon addition of 0.1 mmol of the ligand (L = SMe_2, SEt_2, tht and tms) to dichloromethane solutions of $[Rh(NBD)_2]ClO_4$ (0.1 mmol), an immediate reaction was observed. The resulting pentacoordinated complex was precipitated out by adding ether and then filtered off, washed with ether, and vacuum dried.

X-Ray data collection of $[Rh(NBD)_2(SEt_2)]ClO_4$

A yellow prismatic crystal of the complex, having approximate dimensions of $0.10 \times 0.13 \times 0.30$ mm, was used for the data collection. The cell parameters were obtained by least-squares refinement from the θ values of 29 reflections accurately measured on a Siemens AED single crystal diffractometer. The crystal data are as follows: $C_{18}H_{26}ClO_4RhS$, $M = 476.82$, rhombohedral, a 14.530(7) Å, α 77.86(5)°, V 2887(3) Å^3, $Z = 6$, D_c 1.645 g cm^{-3}, Cu-K_α radiation (λ 1.54178 Å), μ(Cu-K_α) 98.15 cm^{-1}, space group $R\bar{3}c$ from systematic absences and structure determination.

A complete set of intensity data was collected within the angular range $3 < \theta < 60°$.

The $\theta/2\theta$ scan technique was used with a variable scan rate ranging from 2.5 to 10°/min. A standard reflection was remeasured after every 20 reflections as a check on crystal and instrument stability. No significant change in the measured intensities of this reflection was observed during the data collection. A total of 2349 independent reflections were measured, of which 937 were employed in the analysis, having $I > 2\sigma(I)$; the remaining 1412 were considered unobserved. The structure amplitudes were obtained after the usual Lorentz and polarization corrections. No correction for absorption was applied. The absolute scale and the mean temperature factor were established by Wilson's method.

Solution and refinement of the structure

The structure was solved by direct and Fourier methods. Refinement was carried out by full-matrix least-squares, using the SHELX system of computing programs [29], first with isotropic thermal parameters for all the non-hydrogen atoms, and in the last cycles with anisotropic thermal parameters for Rh, Cl and S atoms only. The hydrogen atoms were placed in their geometrically calculated positions and included in the final structure factor calculations. The function minimized in the least-squares calculations was $\Sigma w|\Delta F|^2$; unit weights were used in the first cycles of refinement, the weight calculated as $w = K/[\sigma^2(F_0) + gF_0^2]$ with $K = 0.7841$ and $g = 0.0023$ was chosen in the final cycles. The atomic scattering factors (corrected for anomalous

TABLE 3

FRACTIONAL ATOMIC COORDINATES ($\times 10^4$) WITH E.S.D.'S IN PARENTHESES FOR NON-HYDROGEN ATOMS

	x/a	y/b	z/c
Rh	9776 (1)	5975 (1)	2174 (1)
Cl	7944(15)	4162(15)	971(25)
S	8440(20)	5961(23)	3575(19)
O(1)	8923(33)	4195(28)	645(29)
O(2)	7785(30)	3272(31)	985(29)
O(3)	7659(22)	4182(23)	1929(25)
O(4)	7297(28)	4807(30)	471(25)
C(11)	10722(24)	4741(29)	1636(26)
C(21)	10080(22)	4457(25)	2451(22)
C(31)	11248(36)	5672(33)	2541(34)
C(41)	10610(34)	5342(26)	3342(31)
C(51)	11612(38)	4757(30)	2092(34)
C(61)	10533(24)	4302(24)	3351(24)
C(71)	11542(28)	3827(28)	2876(27)
C(81)	7821(19)	4978(19)	3788(21)
C(91)	6745(25)	5138(37)	4236(33)
C(12)	9614(17)	6391(15)	634(17)
C(22)	8700(25)	6524(28)	1236(32)
C(32)	10088(28)	7455(34)	1385(27)
C(42)	9213(26)	7564(32)	2015(28)
C(52)	9983(25)	7318(21)	390(29)
C(62)	8500(30)	7563(29)	1367(28)
C(72)	8975(25)	7952(25)	331(25)
C(82)	8447(24)	6402(26)	4592(24)
C(92)	7467(29)	6943(34)	4981(34)

TABLE 4

CALCULATED COORDINATES ($\times 10^4$) FOR THE HYDROGEN ATOMS

	x/a	y/b	z/c
H(11)	10635	4905	896
H(21)	9363	4359	2452
H(31)	11437	6374	2299
H(41)	10224	5759	3881
H(51)	12320	4764	1674
H(61)	10225	3937	4038
H(711)	12077	3668	3329
H(712)	11537	3198	2594
H(811)	8181	4406	4262
H(812)	7881	4759	3109
H(911)	6470	4484	4322
H(912)	6358	5697	3773
H(913)	6658	5344	4926
H(12)	9979	5733	394
H(22)	8245	5996	1536
H(32)	10764	7466	1576
H(42)	9075	7631	2760
H(52)	10588	7410	−177
H(62)	7786	7907	1629
H(721)	8642	7806	−208
H(722)	8992	8707	217
H(821)	8957	6882	4420
H(822)	8659	5811	5137
H(921)	7511	7202	5612
H(922)	6950	6469	5161
H(923)	7249	7540	4444

TABLE 5

THERMAL ATOMIC PARAMETERS ($\times 10^4$) WITH e.s.d.'s IN PARENTHESES

Anisotropic parameters are in the form: $\exp[-2\pi^2(h^2a^{\star 2}U_{11} + \ldots + 2hka^{\star}b^{\star}U_{12})]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh	504(49)	492(13)	484(50)	−100(36)	−96(10)	−19(36)
Cl	739(127)	703(149)	666(44)	−26(136)	−197(119)	−319(43)
S	1651(189)	954(63)	1208(186)	−429(131)	775(167)	−686(124)

	U		U		U		U
O(1)	1826(156)	C(31)	739(220)	C(91)	784(205)	C(52)	795(153)
O(2)	1391(163)	C(41)	983(164)	C(12)	572(66)	C(62)	688(155)
O(3)	1409(119)	C(51)	934(236)	C(22)	447(167)	C(72)	477(124)
O(4)	1761(168)	C(61)	543(113)	C(32)	871(155)	C(82)	990(108)
C(11)	444(128)	C(71)	750(163)	C(42)	831(151)	C(92)	1267(211)
C(21)	300(106)	C(81)	579(87)				

dispersion of Rh, Cl and S) were taken from the International Tables [30]. The final *R* value was 0.054 (observed reflections only). Final atomic coordinates for the non-hydrogen atoms and for the hydrogen atoms (in the calculated positions) are given in Tables 3 and 4, respectively. The thermal parameters are given in Table 5. A list of observed and calculated structure factors is available from the authors on request.

Calculations were performed on the CYBER 7600 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord-Orientale, Bologna, with financial support from the University of Parma.

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