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MIXED-LIGAND OXORHENIUM(V) COMPLEXES WITH RHENIUM-SELENIUM BONDS. MOLECULAR STRUCTURE OF (3-OXAPENTANE-1.5-DITHIOLATO)-(BENZENESELENOLATO)OXORHENIUM(V)

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MIXED-LIGAND OXORHENIUM(V) COMPLEXES WITH RHENIUM-SELENIUM BONDS. MOLECULAR STRUCTURE OF (3-OXAPENTANE-1.5-DITHIOLATO)- (BENZENESELENOLATO)OXORHENIUM(V)

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Mixed ligand oxorhenium(V) complexes [ReO(SXS)(SeR)] containing terdentate dithiol ligands HS-CH₂CH₂-X-CH₂CH₂-SH (HSXSH, X = O,S) and benzeneselenol or benzylselenol, RSeH (R = C₆H₅, CH₂-C₆H₅) as monodentate ligands have been synthesized and characterized by spectroscopic and X-ray crystallographic methods. X-ray structure analysis of complex 3 (X = O, R = C₆H₅) shows that the rhenium coordination number is five and the coordination polyhedron is a square pyramid. Structure and spectroscopic data of the complexes are close to those of the corresponding sulphur (RSH as monodentate ligand) compounds.

KEYWORDS: Rhenium complexes, selenols, X-ray structure

INTRODUCTION

Coordination compounds of the β -emitter rhenium-186 are of current interest as potential radiotherapeutics and there are considerable efforts to explore the coordination chemistry of this element for the design of new applicable complexes.^{1–3} As a route to such compounds the synthesis of mixed-ligand rhenium(V) complexes with sulphur-containing ligands according to the “3 + 1”-method is used.^{4,5} This procedure involves combined attack of both a terdentate dithiol and a monodentate thiol ligand on appropriate rhenium(V) precursors and delivers

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complexes of the type [ReO(SXS)(SR)] (“HSXSH” = HS-CH₂CH₂-X-CH₂CH₂-SH with X = O, S, NR’).

Rhenium complexes with selenium-containing ligands have generally been scarcely reported^{6–11} and there is no information available regarding the use of selenol-containing ligands or the properties of such mixed-ligand rhenium complexes. The present paper describes the synthesis of complexes [ReO(SSS)(SeR)]- (R = phenyl, benzyl) and [ReO(SOS)(SeC₆H₅)] and the solid-state structure of the latter. The physical properties of the complexes are compared to those of the sulphur analogues.

EXPERIMENTAL

Material and Measurements

Elemental analyses were performed on a LECO CHNS-932 elemental analyser. Proton NMR spectra were recorded using a Bruker WH-90 device (solvent CDCl₃). Infrared spectra (KBr pellets) were measured on a Specord M80 from Carl Zeiss Jena. Electronic spectra were measured with a Specord UVVIS S 10 device.

3-Thiapentane-1,5-dithiol and 3-oxapentane-1,5-dithiol were obtained from Fluka. Solvents were distilled before used.

Benzyltriethylammonium tetrachlorooxorhenate(V), BzEt₃N[ReOCl₄],⁵ and *chloro(3-thiapentane-1,5-dithiolato)oxorhenium(V)*¹² were synthesized according to literature procedures. Crude *sodium benzylselenolate*¹³ was used without purification. *Benzeneselenol*, C₆H₅-SeH, was synthesized from C₆H₅-Se-Se-C₆H₅.¹⁴ The solvent was reduced to a small volume by rotary evaporation and deaerated water was added. The mixture was acidified with dilute sulphuric acid. The selenol was extracted into chloroform, dried and stored under dinitrogen.

Preparation of (3-thiapentane-1,5-dithiolato)(benzeneselenolato)oxorhenium(V), 1

[ReO(SSS)Cl] (117 mg, 300 μmol) was dissolved in 5 cm³ of boiling acetonitrile and an approximately fivefold excess of the crude selenophenol solution was added. The colour of the mixture quickly turned from ink-blue to brown. The mixture was refluxed for 15 minutes and evaporated to dryness on a rotary evaporator. The residue was washed with methanol several times, dissolved in chloroform and filtered to remove a small amount of gray elemental selenium. The product was purified by column chromatography (20 × 3 cm; silica gel; chloroform). After addition of ethanol to the eluate, the solution was allowed to evaporate slowly to a small volume to give small brown needles. Yield 104.1 mg (69%). M.p. 220–222 °C. Anal. calc. (found) for C₁₀H₁₃OS₃SeRe: C, 23.58 (23.53); H, 2.39 (2.57); S, 18.61 (18.84)%.

Preparation of (3-thiapentane-1,5-dithiolato)[(phenyl)methaneselenolato]oxorhenium(V), 2

[ReO(SSS)Cl] (87 mg, 225 μmol) was dissolved in 8 cm³ of acetonitrile and an excess of crude sodium benzylselenolate solution was added. The colour of the mixture turned from blue to brown immediately and it was then stirred for

5 minutes. It was evaporated to dryness and the residue dissolved in water chloroform and filtered. The solution was passed through a silica gel column (15 × 1.5 cm; Kieselgel 60; MERCK) using chloroform as mobile phase. Much material was deposited near the starting point. To the brown eluate was added the same volume of ethanol and the solution slowly evaporated to near dryness to give crystals. The dark brown needles formed were washed with methanol and diethylether and dried. Yield: 41 mg (35%). M.p.: 176–179°C. Anal. calc. (found) for C₁₁H₁₅OS₃SeRe: C, 24.62 (25.19); H, 2.66 (2.88); S, 18.62 (18.33)%.

Preparation of (3-oxapentane-1,5-dithiolato)(benzenseenolato)oxorhenium(V), 3

A mixture of 3-oxapentane-1,5-dithiol (38.9 mg, 314 μmol) and an excess of the selenophenol solution was added to a cooled (0°C) and stirred solution of BzEt₃N[ReOCl₄] (168.3 mg, 314 μmol) in 3 cm³ of methanol. The colour of the mixture quickly turned to reddish brown. A clear solution was obtained after one hour of stirring. The mixture was evaporated to dryness and the residue washed with methanol several times to remove diphenyl diselenide. The residue was dissolved in chloroform and purified by column chromatography (15 × 1.5 cm; silica gel; chloroform). A few cm³ of ethanol were added to the eluate to promote crystallization. Then the eluate was slowly evaporated to near dryness to give large brown needles. The product was washed with methanol and diethylether. Yield: 74.2 mg (48%). M.p.: 177–179°C. Anal. calc. (found) for C₁₀H₁₃O₂S₂SeRe: C, 24.17 (24.17); H, 2.50 (2.65), S, 12.96 (12.97)%.

X-ray crystal structure analysis of compound 3

X-ray crystallographic data were obtained on a CAD4 diffractometer using graphite-monochromated Mo-K_α radiation at the Bundesanstalt für Materialforschung und -prüfung, Berlin. Large single crystals suitable for X-ray structure analyses were obtained by slow evaporation of a solution of 3 in chloroform and ethanol. The crystal used was a red brown prism (2.5 × 0.38 × 0.25 mm) which was mounted on a glass fibre with a viscous grease. X-ray intensity data were recorded at 296 K and the structure was solved by direct methods. Full-matrix least-square refinement with unit weights was performed. Hydrogen atoms were incorporated at their calculated positions (using a riding model) in the later stages of refinement which converged at $R = 0.56$ ($R_w = 0.067$). A final difference-Fourier synthesis revealed no unusual features (max. 1.65, min. -1.02 eÅ⁻³). Crystallographic calculations were performed using the MOLEN VAX suite of structure determination programs. For all structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from ref.¹⁵ Crystallographic data are summarized in Table 1.

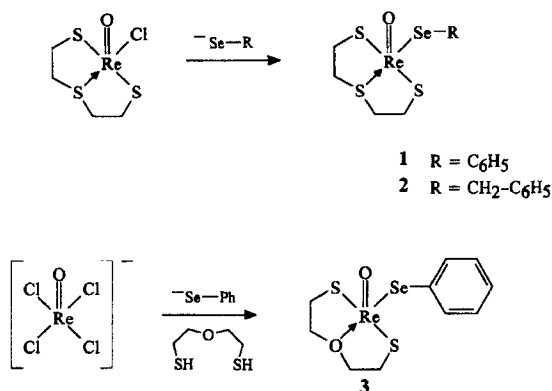
RESULTS AND DISCUSSION

Oxorhenium(V) complexes with '3 + 1' mixed-ligand coordination 1 and 2 have been obtained by exchange of chloride in [ReO(SSS)Cl]¹² by the selenol ligand. An alternative route involving common action of both 3-oxa-1,5-dithiol as terdentate dithiol and benzeneselenol on an appropriate oxorhenium(V) precursor⁵ delivers

Table I Crystallographic and refinement details for **3**.

		3
molecular formula		$C_{10}H_{13}O_2S_2SeRe$
formula weight		494.50
a (Å)		18.122(4)
b (Å)		12.668(3)
c (Å)		12.008(8)
α (°)		90.00
β (°)		90.00
γ (°)		90.00
V (Å ³)		2756.89
space group		<i>Pbca</i>
Z ; $F(000)$; D_{calc} .		8; 1840; 1.996
T(K)		296
crystal size (mm)		2.52 × 0.38 × 0.25
λ (Mo K α) (Å)		0.71073
μ (cm ⁻¹)		118.26
data sphere (°)		3 < 2 θ ≤ 50
scan mode		Ω -2 θ
scan rate (° min)		1.1-4.8°; variable
Miller index range		H 0-21 K 0-15 L 0-14
unique reflexions measured		2759
unique reflexions used ($I \geq 3\sigma(I)$)		2052
check reflexions		6 0 0 4 3 -6 5 3 -6
absorption correction		yes
$R = \Sigma[F_o - F_c] / \Sigma F_o $		0.056

compound **3** (Figure 1). The appropriate seleno-compounds were synthesized using two different methods. Crude selenophenol was obtained by alkaline cleavage of diphenyl diselenide in ethanol;¹⁴ sodium benzylselenolate was synthesized by

**Figure 1** Reaction scheme for the synthesis of complexes 1–3.

reducing selenium powder with sodium borohydride and subsequent reaction with benzyl chloride in ethanol.¹³ The selenols were not isolated because of their sensitivity towards air. So, excess crude solutions of them were used for complexation experiments. Since the solutions contained varying but significant amounts of the respective diselenides, it is impossible to determine the exact stoichiometry of the selenol components involved. In both routes there is no indication of reduction of the oxorhenium(V) core to lower oxidation states by the selenol. Spectroscopic data for complexes 1–3 and the corresponding sulphur analogues 1a–3a (Y = S) are listed in Table II. As found for the sulphur analogues, the selenium-containing complexes [ReO(SXS)SeR] are stable in air and soluble in organic solvents. The neutral and lipophilic species are insoluble in water and non-polar solvents.

The data clearly demonstrate that replacement of sulphur by selenium in the complexes does not drastically change structural or spectroscopic properties of the complexes. Clear differences between compounds 1–3 and their sulphur analogues 1a–3a are observed in electronic spectra, where the first intensive band is shifted to higher wavelengths when going from S to Se. Bathochromic shifts in UV spectra seems to be generally observed for compounds where sulphur is substituted by the more polarizable selenium atom.¹⁰

Small but significant changes have further been found in ¹H NMR and infrared data as well as in molecular structure. The proton NMR spectra of complexes show patterns similar to those of the corresponding sulphur analogues. The patterns for the terdentate ligand in compounds 1–3 resemble those found for compounds with 3-thiapentane-1,5-dithiolato ligands.⁵ Shifts in the monodentate ligand part, with respect to the methylene group in 2 and 2a as well as to aromatic protons may be caused by longer Re–Se–R bond distances (see below).

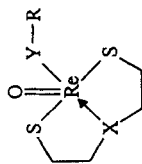
The Re = O vibration in the infrared spectra, in general rather sensitive to changes in the equatorial coordination sphere, is less affected. Slight shifts to higher wave numbers in going from sulphur to selenium are indicated. The trend is parallel to that observed for oxotechnetium(V) complexes with S/Se coordination.¹⁰

An X-ray crystal structure determination was carried out for complex 3. Fractional atom coordinates are listed in Table III and selected interatomic distances and angles are given in Table IV. An ORTEP diagram showing the solid-state conformation and atom numbering scheme of 3 is shown in Figure 2.

X-ray analysis reveals that the ligands bind the Re = O core to form a distorted tetragonal pyramidal coordination around the central atom. The structure presented here is the first example for a rhenium complex with an O,S,Se,S donor set and generally is one of the rare examples of both ether oxygen and basic selenium atom coordination. Whereas most of the data are similar to those observed in [ReO(SOS)(S-Ph-OCH₃)(p)],⁵ differences refer mainly to Re–X (2.406 Å for X = Se, 2.267 Å for X = S) and C–X bonds (1.92 Å for X = Se, 1.79 Å for X = S). Influence of S/Se replacement on the bond lengths involving rhenium and the donor atoms of the terdentate ligand and the terminal oxygen is considerably smaller and differences between 3 and its sulphur analogues do not exceed 0.02 Å.

The results indicate that the rhenium complexes with selenium which have been studied are very similar in their molecular properties to the appropriate sulphur species. This fact, together with the acute toxicity of many selenium compounds and the high sensitivity of –SeH groups towards air and humidity, makes selenium ligands less attractive for the design of rhenium-based radiotracers.

Table II Spectroscopic data for the sulphur and selenium analogues.



	X	Y	R	Infrared (KBr) [cm ⁻¹] ν _{Re-O}	UV-Vis(CH ₃ CN) [nm(εg)]	¹ H NMR shift (CDCl ₃) [ppm]	
						S-CH ₂ CH ₂ -X-CH ₂ CH ₂ -S	-Y-R
1	S	Se	C ₆ H ₅	961	301(3.66);417(3.56)	1.91(2H); 3.02(2H); 3.84(2H); 4.19(2H)	7.32(m,3H,arom.) 7.81(m,2H,arom.)
1a	S	S	C ₆ H ₅	960	330sh (3.19); 401 (3.37); 500 (2.20)	2.02 (2H); 2.98(2H); 3.87(2H); 4.07(2H)	7.02-7.64 (5H, arom.)
2	S	Se	CH ₂ -C ₆ H ₅	960	291(3.77);338sh(3.44) 417(3.57)	1.91(2H); 3.06(2H); 3.84(2H); 4.30(2H)	5.08(s,2H,S-CH ₂ ,Ph); 7.10-7.48(m,5H,arom.)
2a	S	S	CH ₂ -C ₆ H ₅	960	225sh(3.91);3.84(3.52); 508(2.20)	1.95(2H); 3.12(2H); 3.91(2H); 4.29(2H)	5.02(s,2H,S-CH ₂ ,Ph); 7.05-7.54(m,5H,arom.)
3	O	Se	C ₆ H ₅	971	287(3.69);392(3.68)	3.35(m,4H); 3.66(m,2H); 4.62(m,2H)	7.34(m,3H,arom.); 7.74(m,2H,arom.)
3a	O	S	C ₆ H ₅	968	225sh(4.2);254(3.9);359 (3.6);519(2.2)	3.34 (4H,broad signal); 3.70(2H); 4.68(2H)	7.08-7.67 (m,5H,arom.)

Table III Atom coordinates for **3** with standard deviations parentheses.

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i> (Å ²)
Re	0.20323(5)	0.03145(6)	0.13462(7)	2.17(1)
Se	0.1522(1)	0.0368(2)	0.3194(2)	3.38(5)
S1	0.2756(4)	0.1689(4)	0.1937(5)	3.4(1)
S2	0.0891(3)	0.0010(6)	0.0624(6)	4.1(1)
O1	0.2569(9)	-0.074(1)	0.133(1)	3.3(3)*
O2	0.2146(9)	0.112(1)	-0.017(1)	3.2(3)
C1	0.321(1)	0.202(2)	0.060(2)	3.6(5)*
C2	0.260(2)	0.203(2)	-0.032(2)	4.2(5)*
C3	0.155(2)	0.107(2)	-0.104(3)	5.2(6)*
C4	0.109(2)	0.014(3)	-0.085(3)	5.6(7)*
C5	0.061(1)	-0.045(2)	0.319(2)	3.1(4)*
C6	-0.004(2)	-0.004(2)	0.353(2)	4.4(5)*
C7	-0.064(1)	-0.062(2)	0.369(2)	3.7(5)*
C8	-0.061(2)	-0.166(2)	0.346(2)	4.4(5)*
C9	0.001(2)	-0.215(2)	0.310(2)	4.7(6)*
C10	0.064(2)	-0.152(2)	0.291(2)	4.5(6)*
H11	0.3631	0.1428	0.0402	4*
H12	0.3484	0.2783	0.0657	4*
H21	0.2267	0.2744	-0.0254	5*
H22	0.2846	0.2017	-0.1152	5*
H31	0.1793	0.1054	-0.1870	6*
H32	0.1201	0.1775	-0.0988	6*
H41	0.0586	0.0189	-0.1334	6*
H42	0.1397	-0.0559	-0.1147	6*
H6	-0.0069	0.0804	0.3687	5*
H7	-0.1150	-0.0252	0.3988	4*
H8	-0.1110	-0.2136	0.3570	5*
H9	0.0019	-0.2997	0.2975	6*
H10	0.1146	-0.1860	0.2540	5*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameters defined as $4/3[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{13} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$.

SUPPLEMENTARY DATA

Additional material consisting of H-atom coordinates, thermal parameters, and structure factors are available from P.L. (12489 Berlin)

Acknowledgements

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Table IV Bond distances and angles for **3** with estimated standard deviations in parentheses.

Distances (Å)			
Re-Se	2.406(2)	C3-H31	1.12(2)
Re-S1	2.288(5)	C3-H32	1.02(2)
Re-S2	2.275(5)	C4-H41	1.07(2)
Re-O1	1.64(1)	C4-H42	1.11(3)
Re-O2	2.10(1)	C5-C6	1.36(3)
Se-C5	1.92(2)	C5-C10	1.41(2)
S1-C1	1.83(2)	C6-C7	1.34(3)
S2-C4	1.82(2)	C6-H6	1.09(2)
O2-C2	1.42(2)	C7-C8	1.33(3)
O2-C3	1.45(3)	C7-H7	1.13(2)
C1-C2	1.50(3)	C8-C9	1.39(3)
C1-H11	1.13(2)	C8-H8	1.10(2)
C1-H12	1.11(2)	C9-C10	1.39(3)
C2-H21	1.06(2)	C9-H9	1.09(2)
C2-H22	1.14(2)	C10-H10	1.06(2)
C3-C4	1.51(4)		
Angles (°)			
Se-Re-S1	84.9(1)	O2-C3-H31	109.(2)
Se-Re-S2	90.6(1)	O2-C3-H32	115.(2)
Se-Re-O1	104.9(5)	C4-C3-H31	104.(2)
Se-Re-O2	145.1(3)	C4-C3-H32	107.(2)
S1-Re-S2	139.8(2)	H31-C3-H32	111.(2)
S1-Re-O1	108.8(4)	S2-C4-C3	107.(2)
S1-Re-O2	80.4(3)	S2-C4-H41	109.(2)
S2-Re-O1	111.0(4)	S2-C4-H42	106.(2)
S2-Re-O2	80.9(4)	C3-C4-H41	114.(2)
O1-Re-O2	109.8(6)	C3-C4-H42	111.(2)
Re-Se-C5	108.6(5)	H41-C4-H42	108.(2)
Re-S1-C1	98.3(6)	Se-C5-C6	121.(1)
Re-S2-C4	100.5(8)	Se-C5-C10	120.(1)
Re-O2-C2	123.(1)	C6-C5-C10	119.(2)
Re-O2-C3	121.(1)	C5-C6-C7	122.(2)
C2-O2-C3	110.(1)	C5-C6-H6	120.(2)
S1-C1-C2	109.(1)	C7-C6-H6	118.(2)
S1-C1-H11	107.(1)	C6-C7-C8	120.(2)
S1-C1-H12	109.(1)	C6-C7-H7	119.(2)
C2-C1-H11	114.(2)	C8-C7-H7	121.(2)
C2-C1-H12	114.(2)	C7-C8-C9	121.(2)
H11-C1-H12	104.(2)	C7-C8-H8	119.(2)
O2-C2-C1	108.(1)	C9-C8-H8	120.(2)
O2-C2-H21	111.(2)	C8-C9C10	120.(2)
O2-C2-H22	107.(1)	C8-C9-H9	117.(2)
C1-C2-H21	113.(2)	C10-C9-H9	123.(2)
C1-C2-H22	111.(2)	C5-C10-C9	117.(2)
H21-C2-H22	106.(2)	C5-C10-H10	121.(2)
O2-C3-C4	110.(2)	C9-C10-H10	122.(2)

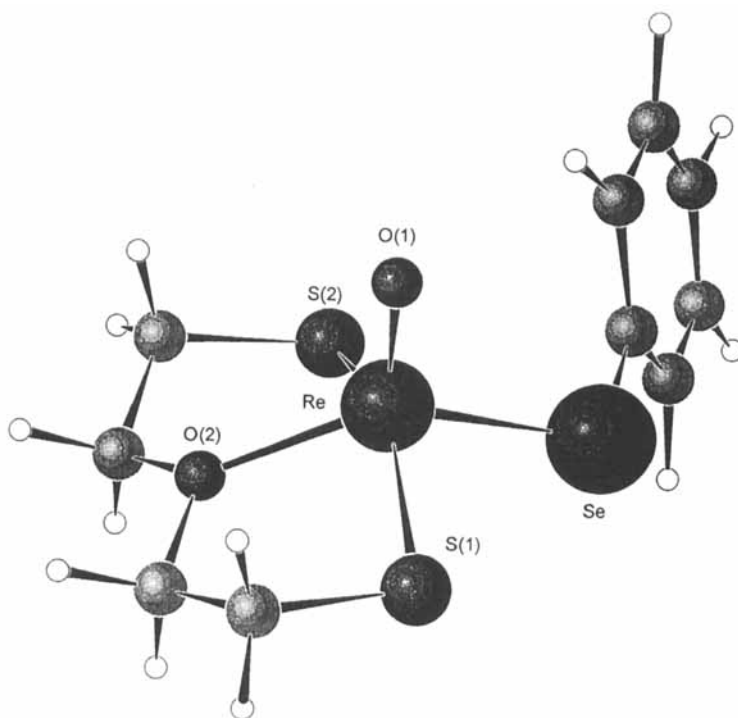


Figure 2 ORTEP drawing of (3-oxapentane-1,5-dithiolato)(benzeneselenolato)oxorhenium(V), **3**, showing the the solid-state conformation and atom numbering scheme.

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