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SYNTHESIS AND SPECTROSCOPIC STUDIES OF RUTHENIUM(II) SELENOETHER COMPLEXES: STRUCTURE OF *TRANS*-$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]$

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Abstract—The complexes *trans*-$[Ru(L—L)_2X_2]$ [X = Cl, Br or I; L—L = bidentate ligand including $PhSe(CH_2)_2SePh$, $MeSe(CH_2)_2SeMe$ and *o*-$C_6H_4(TeMe)_2$] have been prepared by reaction of $RuCl_3 \cdot xH_2O$ and LiX with the ligand in alcoholic solvents in the presence of a reducing agent. The complexes have been characterized by analysis, IR, UV–vis and ^{77}Se or ^{125}Te NMR spectroscopies and the Ru^{II}–Ru^{III} redox potentials established by cyclic voltammetry. The crystal structure of *trans*-$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]$ has been determined, giving Ru—Cl 2.413(1), 2.444(1), Ru—Se 2.433(1)–2.460(1) Å.

Ruthenium(II) complexes of the form *trans*-$[Ru(L—L)_2X_2]$, where L—L is a bidentate dithioether and X are halogen ligands, are well known[1] but comparatively little has been reported for the corresponding selenoether and telluroether compounds.[2] Previous reports of compounds between diselenoether ligands and ruthenium are limited to ruthenium(III) species of the form $[\{Ru(L—L)Cl_3\}_n]$,[3] $[\{Ru_2(L—L)_3Cl_6\}_n]$[3,4] and the anionic $[Ru\{MeSe(CH_2)_2SeMe\}Cl_4]^-$.[3,5] We have recently reported[6] the synthesis of the ruthenium(III) diselenoether complexes *trans*-$[Ru(L—L)_2X_2]BF_4$ and now report a detailed study of the corresponding ruthenium(II) compounds and a rare example of a ruthenium telluroether complex.

EXPERIMENTAL

Physical measurements were made as described previously.[7] Hydrated "$RuCl_3 \cdot xH_2O$" was obtained from Johnson Matthey and was used as supplied. Ligands were prepared by literature routes. Ruthenium thioether chloride and bromide complexes were prepared by the method of Chatt *et al.*[1]

Synthesis

The ruthenium(II) complexes were prepared by two distinct routes as outlined below.

trans-$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]$. To a deoxygenated solution of $RuCl_3 \cdot xH_2O$ (0.24 g, 0.94 mmol) in ethanol (50 cm^3) and water (15 cm^3), PhSe$(CH_2)_2$SePh (0.90 g, 2.65 mmol) was added and the mixture heated. Hypophosphorous acid (2 cm^3) was added to the solution upon reaching reflux, and the solution changed from deep blue to pink in colour, and yielded a pink solid upon cooling. This precipitate was then filtered off, washed with diethyl ether and dried *in vacuo*. Yield 0.29 g, 36%. Found: C, 39.8; H, 3.2. Calc. for $C_{28}H_{28}Cl_2RuSe_4$: C, 39.4; H, 3.3%. ν(Ru—Cl) = 318 cm^{-1}.

trans-$[Ru\{MeSe(CH_2)_2SeMe\}_2Cl_2]$. A solution of $RuCl_3 \cdot xH_2O$ (0.31 g, 1.2 mmol) in ethanol was reduced, under an N_2 atmosphere, with five granules of zinc amalgam (prepared from zinc granules and a saturated $HgCl_2$ solution). This solution was stirred for 2 h and the zinc was removed under a steady flow of N_2. To this blue solution, MeSe$(CH_2)_2$SeMe (0.54 g, 2.5 mmol) was added by syringe and the mixture refluxed for 2 h, producing a brown suspension. The resultant solution was then cooled, filtered and the precipitate washed with ace-

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tone. The combined washings and filtrate were then treated with diethyl ether and cooled overnight, precipitating a fawn solid. This solid was filtered off, washed with diethyl ether and dried *in vacuo*. Yield 0.06 g, 19%. Found: C, 15.7; H, 3.4. Calc. for $C_8H_{20}Cl_2RuSe_4$: C, 15.9; H, 3.3%.

The bromo complexes were prepared by refluxing the analogous chloro complex with a large excess of LiBr in ethanol for 10 h. *trans*-$[Ru\{PhSe(CH_2)_2SePh\}_2Br_2]$. Found: C, 35.9; H, 3.3. Calc. for $C_{28}H_{28}Br_2RuSe_4$: C, 35.7; H, 3.0%). *trans*-$[Ru\{MeSe(CH_2)_2SeMe\}_2Br_2]$. Found: C, 13.9; H, 3.0. Calc. for $C_8H_{20}Br_2RuSe_4$: C, 13.8; H, 2.9%.

The iodo complexes were prepared by refluxing the analogous chloro complexes with a large excess of LiI in ethanol for 10 h. *trans*-$[Ru\{PhS(CH_2)_2SPh\}_2I_2]$. Found: C, 39.4; H, 3.1. Calc. for $C_{28}H_{28}I_2RuS_4$: C, 39.7; H, 3.3%. *trans*-$[Ru\{MeS(CH_2)_2SMe\}_2I_2]$. Found: C, 15.9; H, 3.5. Calc. for $C_8H_{20}I_2RuS_4$: C, 16.0; H, 3.3%. *trans*-$[Ru\{PhSe(CH_2)_2SePh\}_2I_2]$. Found: C, 33.0; H, 2.9. Calc. for $C_{28}H_{28}I_2RuSe_4$: C, 32.5; H, 2.7%.

trans-$[Ru\{o\text{-}C_6H_4(TeMe)_2\}_2Cl_2]$. To a solution of $RuCl_3 \cdot xH_2O$ (0.22 g, 0.85 mmol), o-$C_6H_4(TeMe)_2$ (0.59 g, 1.65 mmol) was added and the reaction mixture refluxed for 2 h in 2-methoxyethanol (30 cm^3). Upon addition of the ligand, a light brown solid precipitated which dissolved upon reaching reflux and the solution paled over 2 h reflux. The 2-methoxyethanol was removed under vacuum and the resulting solid dissolved in CH_2Cl_2. A fawn coloured solid was precipitated with diethyl ether, filtered off, washed with diethyl ether and dried *in vacuo*. Yield 0.42 g, 55%. Found: C, 21.7; H, 2.4. Calc. for $C_{16}H_{20}Cl_2RuTe_4$: C, 21.5; H, 2.2%.

Refluxing this solid in ethanol with a large excess of LiBr for 10 h produced a brown solid which was found to be insoluble in organic solvents.

Crystal data

$C_{28}H_{28}Cl_2RuSe_4$, $M_r = 852.35$, triclinic, space group $P\bar{1}$, $a = 11.610(2)$, $b = 11.664(3)$, $c = 11.752(2)$ Å, $\alpha = 101.41(2)$, $\beta = 104.90(2)$, $\gamma = 106.56(2)^0$, $V = 1409.6$ Å^3, $Z = 2$, $D_c = 2.007$ g cm^{-3}, $F(000) = 820$, $\lambda = 0.71069$ Å, $\mu(\text{Mo-}K_\alpha) = 58.3$ cm^{-1}, room temperature.

Data collection, structure solution and refinement

Dark brown air-stable crystals were obtained from CH_2Cl_2 solution by vapour diffusion of diethyl ether and mounted in glass capillaries. Cell dimensions were obtained from 25 reflections using an Enraf–Nonius CAD-4 diffractometer fitted with Mo radiation and graphite monochromator. The data collection using the same room temperature crystal ($0.58 \times 0.23 \times 0.10$ mm^3) recorded 5843 reflections ($\theta \leqslant 26°$: h 0–14; k −14 to 14; l −14 to 14). No crystal decay was noted during the experiment and an empirical ψ-scan absorption correction based on four reflections was applied [transmission: 57.4 (min.), 100.0% (max.)]. The normalized structure factors (E values) favoured the centrosymmetric space group $P\bar{1}$ and application of the direct methods strategy in SHELX-76[8] yielded a solution showing seven large peaks ($RuSe_4Cl_2$). Subsequent structure factor and electron density calculations readily located the carbon atoms and a number of the expected hydrogen atoms, and all of the latter were introduced into the model in calculated positions [d(C—H) = 0.95 Å]. Full-matrix least-squares refinement converged to $R = 0.033$ {317 parameters, 4631 reflections [$F > 3\sigma(F)$], anisotropic (Ru, Se, Cl, C) and isotropic (H) atoms, $w = 1/\{\sigma^2(F) + 0.0003F^2\}$, $R_w = 0.050$, |max. shift/error| = 0.07}. The residual electron density was in the range 0.56 to −0.83 eÅ^{-3}.

Neutral atom scattering factors and anomalous dispersion corrections were taken from SHELX 76[8] and ref. 9 (Ru, Se) and the calculations were carried out using SHELX 76 and ORTEP-II[10] on an IBM 3090 computer. Selected bond lengths and angles are given in Table 1 and the discrete molecule is shown in Fig. 1.

RESULTS AND DISCUSSION

The reaction of $RuCl_3 \cdot xH_2O$ with a 1:2 ratio of diselenoether in refluxing 2-methoxyethanol in the presence of a reducing agent, in particular hypophosphorous acid, produces complexes of the form *trans*-$[Ru(L—L)_2Cl_2]$. In the absence of a reducing agent, polymeric ruthenium(III) species are produced, as reported previously.[3] It was found that in the case of the ditelluroether complex no reducing agent was required, which is a reflection of the greater reducing ability of telluroethers compared to selenoethers and also the tendency of o-phenylene ligands to chelate rather than bridge metal centres. The *trans*-$[Ru\{MeSe(CH_2)_2SeMe\}_2Cl_2]$ was prepared from the reaction of a zinc amalgam-reduced ethanolic solution of $RuCl_3 \cdot xH_2O$ with an excess of the ligand, as it was found that the use of hypophosphorous acid as reductant produced intractable oils in this case. Analogous bromide and iodide compounds were prepared by refluxing the corresponding chloride in ethanol with a large excess of LiX. The *trans*-$[Ru(L—L)_2X_2]$ compounds were found to be soluble in common organic solvents, including dichloromethane and aceto-

Table 1. Selected bond lengths (Å) and angles (°) for *trans*-[Ru{PhSe(CH$_2$)$_2$SePh}$_2$Cl$_2$]

Ru—Se(1)	2.433(1)	Ru—Se(4)	2.443(1)
Ru—Se(2)	2.460(1)	Ru—Cl(1)	2.413(1)
Ru—Se(3)	2.460(1)	Ru—Cl(2)	2.444(1)
Se(1)—C(1)	1.951(6)	Se(3)—C(3)	1.977(6)
Se(1)—C(11)	1.928(6)	Se(3)—C(31)	1.935(6)
Se(2)—C(2)	1.976(6)	Se(4)—C(4)	1.974(6)
Se(2)—C(21)	1.930(6)	Se(4)—C(41)	1.932(6)
C(1)—C(2)	1.495(8)	C(3)—C(4)	1.503(9)
C—C(phenyl)	1.354(10)–1.406(9)		
Cl(1)—Ru—Se(1)	83.2(1)	Cl(1)—Ru—Cl(2)	178.3(1)
Cl(1)—Ru—Se(2)	100.5(1)	Se(1)—Ru—Se(2)	85.9(1)
Cl(1)—Ru—Se(3)	83.0(1)	Se(1)—Ru—Se(3)	166.2(1)
Cl(1)—Ru—Se(4)	96.8(1)	Se(1)—Ru—Se(4)	95.7(1)
Cl(2)—Ru—Se(1)	95.6(1)	Se(2)—Ru—Se(3)	95.8(1)
Cl(2)—Ru—Se(2)	80.6(1)	Se(2)—Ru—Se(4)	162.7(1)
Cl(2)—Ru—Se(3)	98.2(1)	Se(3)—Ru—Se(4)	86.7(1)
Cl(2)—Ru—Se(4)	82.1(1)		
Ru—Se(1)—C(1)	99.7(2)	Ru—Se(3)—C(3)	101.7(2)
Ru—Se(1)—C(11)	114.0(2)	Ru—Se(3)—C(31)	119.3(2)
Ru—Se(2)—C(2)	103.2(2)	Ru—Se(4)—C(4)	103.2(2)
Ru—Se(2)—C(21)	117.6(2)	Ru—Se(4)—C(41)	119.3(2)
C(1)—Se(1)—C(11)	100.8(2)	C(3)—Se(3)—C(31)	96.5(3)
C(2)—Se(2)—C(21)	97.7(2)	C(4)—Se(4)—C(41)	96.9(3)
Se(1)—C(1)—C(2)	107.4(4)	Se(3)—C(3)—C(4)	108.9(4)
Se(2)—C(2)—C(1)	111.3(4)	Se(4)—C(4)—C(3)	107.5(4)
Se(1)—C(1)—C(2)—Se(2)	58.7	Se(3)—C(3)—C(4)—Se(4)	62.9

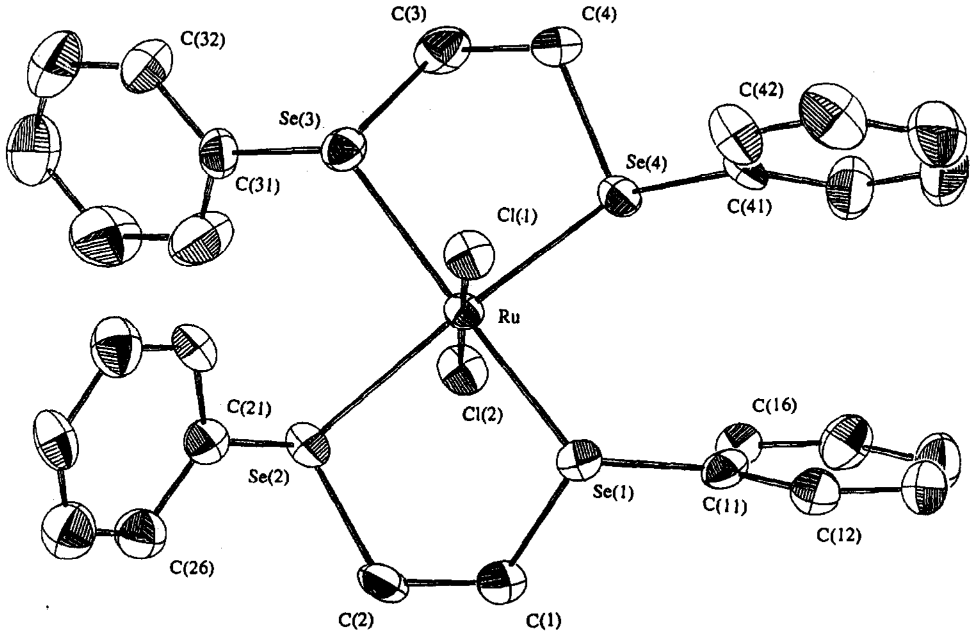

Fig. 1. The molecular structure of *trans*-[Ru{PhSe(CH$_2$)$_2$SePh}$_2$Cl$_2$] showing the atom labelling scheme. Thermal ellipsoids were drawn at the 50% probability level.

Table 2. Selected spectroscopic data

Compound	Colour	E_{max}[a], 10^3 cm^{-1} (ε, dm^3 mol^{-1} cm^{-1})
$Ru\{PhS(CH_2)_2SPh\}_2Cl_2$	orange	20.2 (105), 25.0 (150), 31.7 (1200)
$Ru\{PhS(CH_2)_2SPh\}_2Br_2$	pink	19.2 (20), 24.3 (50), 31.3 (460)
$Ru\{PhS(CH_2)_2SPh\}_2I_2$	orange	31.1 (660)
$Ru\{MeS(CH_2)_2SMe\}_2Cl_2$	yellow	23.3 (80), 29.7 (200) (sh)
$Ru\{MeS(CH_2)_2SMe\}_2Br_2$	lilac	21.0 (100), 28.1 (320)
$Ru\{MeS(CH_2)_2SMe\}_2I_2$	pink	19.5 (85), 27.2 (270)
$Ru\{PhSe(CH_2)_2SePh\}_2Cl_2$	pink	19.9 (330), 24.6 (460), 30.7 (1640)
$Ru\{PhSe(CH_2)_2SePh\}_2Br_2$	pink	19.5 (70), 24.6 (200), 30.8 (1200) (sh)
$Ru\{PhSe(CH_2)_2SePh\}_2I_2$	brown	20.7 (sh), 24.1 (2045), 31.3 (3070)
$Ru\{MeSe(CH_2)_2SeMe\}_2Cl_2$	fawn	23.5 (1020) (sh), 33.7 (4010) (sh)
$Ru\{MeSe(CH_2)_2SeMe\}_2Br_2$	brown	22.3 (670) (sh), 32.7 (3210) (sh)
$Ru\{o\text{-}C_6H_4(TeMe)_2\}_2Cl_2$	fawn	20.4 (270), 24.4 (820) (sh), 31.9 (sh)

[a] Recorded in CH_2Cl_2.

nitrile. The *trans* geometry of the complexes was established from the X-ray crystal structure of *trans*-$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]$ and by UV–vis and ^{77}Se or ^{125}Te NMR spectroscopies.

UV–vis spectra

The UV–vis spectra of the complexes are typical of ruthenium(II) d^6 complexes of the form *trans*-RuL_4X_2 and the recorded data are shown in Table 2. The spectra consist of two weak absorptions to low energy, *ca* 24,000 and *ca* 20,000 cm^{-1}, which are assigned as *d–d* bands. A further, more intense absorption is observed at higher energies, *ca* 31,000 cm^{-1}, which is attributed to an M → L charge transfer (CT) or, in those cases where a phenyl substituted ligand is present, the $\pi \rightarrow \pi^*$ transition. Of the *d–d* bands, the lower energy band is assigned as $^1A_{1g} \rightarrow {}^1E_g$ and the higher as $^1A_{1g} \rightarrow {}^1A_{2g}$, as has been reported previously for the thioether compounds[1] and for the analogous diphosphine complexes.[11,12] The separation between the *d–d* bands is lower for the thio-, seleno- and telluroether complexes than for the corresponding phosphine complexes, consistent with the lower-field strength of the group 16 donor ligands. For some complexes the higher energy *d–d* band is obscured by the "tail" of the more intense M → L CT band and appears as a shoulder or not at all. In one case, that of *trans*-$[Ru\{PhS(CH_2)_2SPh\}_2I_2]$, both *d–d* bands are obscured by the higher energy CT band.

Electrochemistry

Cyclic voltammograms were recorded for the complexes and the Ru^{II}–Ru^{III} redox potentials are shown in Table 3. All the complexes gave reversible Ru^{II}–Ru^{III} couples, as had been observed previously for the corresponding ruthenium(III) cations,[6] except that of *trans*-$[Ru\{PhS(CH_2)_2SPh\}_2I_2]$, which was found to be irreversible. No evidence for a Ru^{III}–Ru^{IV} couple was observed for the seleno- or telluroether compounds, though the thioether complexes exhibited a number of irreversible oxidative waves to high potentials, greater than +1.5 V. The major variation in the redox potential is caused by changing the substituent on the donor atom from

Table 3. Electrochemical data for *trans*-$[Ru(L—L)_2X_2]BF_4$ and *trans*-$[Ru(L—L)_2X_2]$ complexes with Group 16 donor ligands, E_e°(V *vs* SCE)

Compound	Ru^{II}–Ru^{III}[a]
$[Ru\{PhS(CH_2)_2SPh\}_2Cl_2]^{0/+}$	+0.65[b,c]
$[Ru\{PhS(CH_2)_2SPh\}_2Br_2]^{0/+}$	+0.70[b]
$[Ru\{PhS(CH_2)_2SPh\}_2I_2]^{0/+}$	(+0.69)[d,e]
$[Ru\{MeS(CH_2)_2SMe\}_2Cl_2]^{0/+}$	+0.55[b,c]
$[Ru\{MeS(CH_2)_2SMe\}_2Br_2]^{0/+}$	+0.57[b,c]
$[Ru\{MeS(CH_2)_2SMe\}_2I_2]^{0/+}$	+0.60
$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]^{0/+}$	+0.57[b,c]
$[Ru\{PhSe(CH_2)_2SePh\}_2Br_2]^{0/+}$	+0.59[b,c]
$[Ru\{PhSe(CH_2)_2SePh\}_2I_2]^{0/+}$	+0.71[d]
$[Ru\{MeSe(CH_2)_2SeMe\}_2Cl_2]^{0/+}$	+0.43
$[Ru\{MeSe(CH_2)_2SeMe\}_2Br_2]^{0/+}$	+0.56[b,c]
$[Ru\{o\text{-}C_6H_4(TeMe)_2\}_2Cl_2]^{0/+}$	+0.52

[a] MeCN solution containing 0.1 mol dm^{-3} $Bu^n{}_4NBF_4$. The $Fe(\eta\text{-}C_5H_5)_2/Fe(\eta\text{-}C_5H_5)_2{}^+$ couple is at +0.41 V.

[b] From ref. 6.

[c] Data obtained from the ruthenium(III) complex.

[d] Recorded in CH_2Cl_2 solution, due to insolubility in MeCN, containing 0.1 mol dm^{-3} $Bu^n{}_4NBF_4$. The $Fe(\eta\text{-}C_5H_5)_2/Fe(\eta\text{-}C_5H_5)_2{}^+$ couple is at +0.57 V.

[e] Irreversible couple.

Table 4. ^{77}Se and ^{125}Te NMR spectroscopic data for *trans*-[Ru(L—L)$_2$X$_2$] complexes

Compound	^{77}Se δ (ppm)[a]	$\Delta\delta$ Se (av.)[b]
Ru{PhSe(CH$_2$)$_2$SePh}$_2$Cl$_2$	488, 491, 495, 498	153
Ru{PhSe(CH$_2$)$_2$SePh}$_2$Br$_2$	488, 491, 494, 495, 496, 498	154
Ru{PhSe(CH$_2$)$_2$SePh}$_2$I$_2$	487, 491, 494, 498	153
Ru{MeSe(CH$_2$)$_2$SeMe}$_2$Cl$_2$[c]	344.5, 341.5, 339, 334	219
Ru{MeSe(CH$_2$)$_2$SeMe}$_2$Br$_2$[c]	346, 343, 342, 340, 336, 334, 330, 328	216

Compound	^{125}Te δ (ppm)[d]	$\Delta\delta$ Te (av.)[b]
Ru{*o*-C$_6$H$_4$(TeMe)$_2$}$_2$Cl$_2$	885, 890, 893, 894, 908, 917	526

[a] Recorded at 300 K in CH$_2$Cl$_2$ using an external Me$_2$Se reference.

[b] Coordination shift, i.e. δ(complex) − δ(ligand).

[c] Recorded at 300 K in acetone using an external Me$_2$Se reference.

[d] Recorded at 300 K in CH$_2$Cl$_2$ using an external Me$_2$Te reference.

methyl to phenyl, the couple moving to more positive potentials with a phenyl-substituted ligand. This is consistent with observations made for the analogous diphosphine complexes.[6] Varying the donor atom from sulphur to selenium results in a shift of the Ru^{II}–Ru^{III} to less positive potentials and this trend can be extended to the single telluroether complex. It can be seen from the potentials that variation of the halide ligand from chloride to bromide to iodide has little effect upon the Ru^{II}–Ru^{III} redox potentials. Most of the complexes can be chemically oxidized[1,6] to the ruthenium(III) analogues. However, the attempts to oxidize *trans*-[Ru{PhS(CH$_2$)$_2$SPh}$_2$I$_2$] resulted in decomposition. Chlorine in CCl$_4$ decomposed the ditelluroether complex, whilst treatment with HNO$_3$–HBF$_4$ produced no visible change.

^{77}Se *and* ^{125}Te *NMR spectra*

^{77}Se{^{1}H} and ^{125}Te{^{1}H} NMR spectra were recorded for the appropriate complexes and the recorded data are shown in Table 4. The spectra are consistent with a *trans* configuration since the resonances were observed over a small range of values, whereas *cis* isomers would exhibit resonances in two distinct regions corresponding to δ(Se *trans* X) and δ(Se *trans* Se).[13] The complexes have five possible invertomers, which could give a maximum of eight signals.[14] The spectra generally consist of one intense band and a number of weaker signals (see Fig. 2 for a typical example). For the complex *trans*-

Fig. 2. ^{77}Se{^{1}H} NMR spectrum of *trans*-[Ru{PhSe(CH$_2$)$_2$SePh}$_2$Cl$_2$] in CH$_2$Cl$_2$.

$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]$, the isomer characterized by X-ray crystallography (see below) has all four selenium atoms equivalent and is probably the major species present in solution. Although one would not necessarily expect the predominant invertomer found in solution and the invertomer found in the crystal to be the same, with one exception[15] in all previous examples studied this has been found to be the case.[2]

X-ray structure of trans-$[Ru\{PhSe(CH_2)_2SePh\}_2Cl_2]$

The structure consists of discrete species containing six-coordinate ruthenium with *trans* stereochemistry (Fig. 1); the molecule has no crystallographic symmetry. The Ru—Cl bond lengths (Table 1) may be compared with the values found[16] in $[Ru(C_6H_{12}S_2)_2Cl_2]$ [2.445(1) Å] and $[Ru(C_6H_{12}S_2O)_2Cl_2]$ [2.413(4) Å], being longer than the values found[5] in the Ru^{III} anion $[Ru\{MeSe(CH_2)_2SeMe\}Cl_4]^-$ [2.353(2), 2.344(2) Å for Cl *trans* Cl]. This latter compound also provides Ru—Se bond lengths [2.446(1), 2.457(1) Å] for comparison with the present compound [2.433(1)–2.460(1) Å]. The intraligand bond lengths are unexceptional and in addition to establishing the *trans* stereochemistry, the only other feature of note is the ligand conformation. Figure 1 shows that the coordinated diselenoether ligands have the DL conformation with all four selenium centres having the "*S*" absolute configuration. The ligand "bite" is about 86° and the Se—C—C—Se torsion angles close to the idealized 60°.

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