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Synthesis and structural characterisation of new cationic dinuclear ruthenium(II) thiolato complexes of the type $[Ru_2(\eta^6\text{-arene})_2(\mu\text{-}p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$

Frédéric Chérioux, Bruno Therrien *, Georg Süss-Fink

Institut de Chimie, Université de Neuchâtel, Case postale 2, CH-2007 Neuchâtel, Switzerland

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Abstract

Dinuclear dichloro complexes $[Ru(C_6H_6)Cl_2]_2$, $[Ru(p\text{-}MeC_6H_4\,{}^iPr)Cl_2]_2$, $[Ru(1,2,4,5\text{-}C_6H_2Me_4)Cl_2]_2$, and $[Ru(C_6Me_6)Cl_2]_2$ react in ethanol with *p*-bromothiophenol to give the corresponding cationic complexes $[Ru_2(C_6H_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**1**), $[Ru_2(p\text{-}MeC_6H_4\,{}^iPr)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**2**), $[Ru_2(1,2,4,5\text{-}C_6H_2Me_4)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**3**), and $[Ru_2(C_6Me_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**4**), which can be isolated in quantitative yield as their chloride salts. X-ray structure analysis of these complexes shows that the nature of the arene ligand influences the folding of the *p*-S–C_6H_4–Br units. In **1**, where the less hindered arene ligand is present, the three phenyl rings of the thiolato units are not constrained to a coplanar arrangement, whereas in **4** the C_6Me_6 forces the three phenyl rings to be in perfect planarity. Complexes **2** and **3** show an intermediary arrangement.

Keywords: Arene ligand; Ruthenium; Sulfur ligand

1. Introduction

During the past two decades, there has been a growing interest in the field of conjugated molecules containing metal centres because of their electronic, non-linear optical, magnetic, and catalytic properties [1–3] and more recently because of their utility for the development of sensors [4,5]. It is quite impossible to be exhaustive because of the plethora of new compounds synthesised in all these fields of research, but, to the best of our knowledge, all relevant molecules are built around mononuclear building blocks which are coordinated to different types of organic ligands such as metallocenes, salens, dithiolenes or "nitrogen bridges" (terpyridines, bipyridines or porphyrins), which are the most common in the literature because of their extraordinary ability to develop supramolecular structures [1,6]. However, there are still challenges to develop versatile and selective strategies in the view of creating new molecular design and new bridging ligands. On the other hand, star-shaped molecules can lead to a strong enhancement of the physical properties like non-linear optical susceptibilities [7,8] or electronic conductivities in hyperbranched conjugated polymers [9]. Moreover, there is a large interest for conjugated oligomers for their intrinsic physical properties [10,11] as well as they are model compounds for the study of their corresponding conductive polymers [12].

Recently, we have synthesised conjugated star-like oligophenylene molecules containing a dinuclear organometallic core with sulfur connectivities, $[Rh_2(C_5Me_5)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ and $[Ru_2(p\text{-}MeC_6H_4\,{}^iPr)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ [13], and used the bromo substituents to increase their conjugation length by Suzuki cross-coupling reactions [14]. In this paper, we report the synthesis of three new dinuclear organometallic complexes of the type $[Ru_2(\eta^6\text{-arene})_2(\mu\text{-}p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (arene = C_6H_6, 1,2,4,5-$C_6H_2Me_4$, C_6Me_6) with sulfur connectivities. A structural study including the previously reported

* Corresponding author. Tel.: +41-32-718-2499; fax: +41-32-718-2511.
E-mail address: bruno.therrien@unine.ch (B. Therrien).

analogue $[Ru_2(p\text{-}MeC_6H_4{}^iPr)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ [13] shows that the nature of the arene ligand influences the folding of the *p*-S–C_6H_4–Br units.

2. Experimental

2.1. General remarks

All reactions were carried out under nitrogen, by using standard Schlenk techniques. Solvents were degassed prior to use. The dinuclear dichloro complexes $[Ru(C_6H_6)Cl_2]_2$, $[Ru(p\text{-}MeC_6H_4{}^iPr)Cl_2]_2$, $[Ru(1,2,4,5\text{-}C_6H_2Me_4)Cl_2]_2$, and $[Ru(C_6Me_6)Cl_2]_2$ were synthesised by previously described methods [15–17]. All other reagents were purchased (Acros, Fluka or Aldrich) and used as received. NMR spectra were recorded with a Varian Gemini 200 BB instrument and referenced to the signals of the residual protons in the deuterated solvents. The mass spectra were recorded at the University of Fribourg (Switzerland) by Prof. Titus Jenny. Microanalyses were carried out by the Laboratory of Pharmaceutical Chemistry, University of Geneva (Switzerland).

2.2. General method for **1**–**4**

The dinuclear dichloro complex $[Ru(C_6H_6)Cl_2]_2$ (50 mg, 0.1 mmol), $[Ru(p\text{-}MeC_6H_4{}^iPr)Cl_2]_2$ (60 mg, 0.1 mmol), $[Ru(1,2,4,5\text{-}C_6H_2Me_4)Cl_2]_2$ (60 mg, 0.1 mmol), or $[Ru(C_6Me_6)Cl_2]_2$ (67 mg, 0.1 mmol) was refluxed in technical grade ethanol (25 ml). Then, a solution of *p*-bromothiophenol (95 mg, 0.5 mmol) in 5 ml of ethanol was added drop-wise to the hot solution. The resulting mixture was refluxed in ethanol for 3 h. After cooling to 20 °C, the red solution was filtered on celite and the solvent was removed under reduced pressure. The oil obtained was purified by column chromatography (silica gel, dichloromethane/ethanol 5:1, R_f close to 0.8). Cations **1**, **2**, **3** and **4** were isolated in the form of the chloride salts by evaporation of the solvent as red–orange powders in quantitative yields.

2.2.1. $[Ru_2(C_6H_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**1**)

^{1}H NMR (200 MHz, $CDCl_3$, 21 °C): $\delta = 5.52$ (s, 12H, C*H*–Ar), 7.54 (d, $^3J_{H,H} = 8.2$ Hz, 6H, C*H*–Ar), 7.80 (d, $^3J_{H,H} = 8.2$ Hz, 6H, C*H*–Ar). $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$, 21 °C): $\delta = 87.02$ (Ru–*C*–Ar), 109.11 (*C*–Br), 132.25 (*C*–Ar), 134.44 (*C*–Ar), 142.49 (*C*–S). MS (ESI) *m*/*z*: 922 (M^+). Elemental Anal. Calc. for $C_{30}H_{24}Br_3ClRu_2S_3$ (958.01): C, 37.61; H, 2.53. Found: C, 37.48; H, 2.59%.

2.2.2. $[Ru_2(p\text{-}MeC_6H_4{}^iPr)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**2**)

^{1}H NMR (200 MHz, $CDCl_3$, 21 °C): $\delta = 0.82$ (d, $^3J_{H,H} = 7.0$ Hz, 6H, $(CH_3)_2CH$), 0.92 (d, $^3J_{H,H} = 7.0$ Hz, 6H, $(CH_3)_2CH$), 1.64 (s, 6H, CH_3), 1.97 (sept, $^3J_{H,H} = 7.0$ Hz, 2H, $(CH_3)_2CH$), 5.23 (d, $^3J_{H,H} = 6.2$ Hz, 2H, C*H*–Ar), 5.26 (d, $^3J_{H,H} = 5.9$ Hz, 2H, C*H*–Ar), 5.32 (d, $^3J_{H,H} = 5.9$ Hz, 2H, C*H*–Ar), 5.60 (d, $^3J_{H,H} = 6.2$ Hz, 2H, C*H*–Ar), 7.54 (d, $^3J_{H,H} = 8.8$ Hz, 6H, C*H*–Ar), 7.86 (d, $^3J_{H,H} = 8.8$ Hz, 6H, C*H*–Ar). $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$, 21 °C): $\delta = 18.09$ (CH_3), 22.12 ($(CH_3)_2CH$), 22.94 ($(CH_3)_2CH$), 30.00 ($(CH_3)_2CH$), 84.12 (Ru–*C*–Ar), 85.50 (Ru–*C*–Ar), 85.96 (Ru–*C*–Ar), 100.29 (Ru–*C*–Ar), 108.00 (Ru–*C*–Ar), 123.01 (*C*–Br), 132.60 (*C*–Ar), 134.61 (*C*–Ar), 137.16 (*C*–S). MS (ESI) *m*/*z*: 1036 (M^+). Elemental Anal. Calc. for $C_{38}H_{40}Br_3ClRu_2S_3$ (1070.23): C, 42.65; H, 3.77. Found: C, 42.39; H, 4.02%.

2.2.3. $[Ru_2(1,2,4,5\text{-}C_6H_2Me_4)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**3**)

^{1}H NMR (200 MHz, $CDCl_3$, 21 °C): $\delta = 1.73$ (s, 24H, CH_3–Ar), 5.43 (s, 4H, C*H*–Ar), 7.53 (d, $^3J_{H,H} = 8.2$ Hz, 6H, C*H*–Ar), 7.79 (d, $^3J_{H,H} = 8.2$ Hz, 6H, C*H*–Ar). $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$, 21 °C): $\delta = 29.88$ (CH_3–Ar), 86.89 (Ru–*C*–Ar), 109.52 (*C*–Br), 133.48 (*C*–Ar), 135.56 (*C*–Ar), 141.76 (*C*–S). MS (ESI) *m*/*z*: 1036 (M^+). Elemental Anal. Calc. for $C_{38}H_{40}Br_3ClRu_2S_3$ (1070.23): C, 42.65; H, 3.77. Found: C, 42.78; H, 3.92%.

2.2.4. $[Ru_2(C_6Me_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**4**)

^{1}H NMR (200 MHz, $CDCl_3$, 21 °C): $\delta = 1.71$ (s, 36H, CH_3–Ar), 7.52 (d, $^3J_{H,H} = 8.2$ Hz, 6H, C*H*–Ar), 7.77 (d, $^3J_{H,H} = 8.2$ Hz, 6H, C*H*–Ar). $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$, 21 °C): $\delta = 29.05$ (CH_3–Ar), 86.58 (Ru–*C*–Ar), 109.74 (*C*–Br), 132.69 (*C*–Ar), 135.48 (*C*–Ar), 141.14 (*C*–S). MS (ESI) *m*/*z*: 1091 (M^+). Elemental Anal. Calc. for $C_{42}H_{48}Br_3ClRu_2S_3$ (1126.33): C, 44.79; H, 4.30. Found: C, 44.61; H, 4.52%.

2.3. X-ray crystallographic study

The single-crystal structure analysis of **2** has been published previously [13]. Crystals of **1**, **3** and **4** were mounted on a Stoe Image Plate Diffraction system equipped with a ϕ circle goniometer, using Mo Kα graphite monochromated radiation ($\lambda = 0.71073$ Å with ϕ range 0°–200°, increment of 1.5°, 1.0° and 1.5°, respectively, 2θ range from 2.0° to 26°, D_{max}–D_{min} = 12.45–0.81 Å. The structures were solved by direct methods using the programme SHELXS-97 [18]. The refinement and all further calculations were carried out using SHELXL-97 [19]. The H-atoms were included in calculated positions and treated as riding atoms using the SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-square on F^2. Crystallographic details are summarised in Table 1. Figures were drawn with the ORTEP programme [20].

Crystallographic and selected experimental data of cations **1**, **2**, **3** and **4**

	[**1**]BPh_4	[**2**]$Cl \cdot CHCl_3$	[**3**]BF_4	[**4**]BF_4
Chemical formula	$C_{54}H_{44}BBr_3Ru_2S_3$	$C_{39}H_{41}Br_3Cl_4Ru_2S_3$	$C_{38}H_{40}BBr_3F_4Ru_2S_3$	$C_{40}H_{40}BBr_3F_4RuS_3$
Formula weight	1241.75	1189.57	1121.56	1044.51
Crystal system	triclinic	orthorhombic	monoclinic	hexagonal
Space group	$P\bar{1}$	$P2_12_12_1$	$P2_1/n$	$P6_3/m$
Crystal colour and shape	orange rod	red block	red block	orange needle
Crystal size	$0.50 \times 0.15 \times 0.15$	$0.48 \times 0.26 \times 0.26$	$0.50 \times 0.40 \times 0.40$	$0.30 \times 0.15 \times 0.15$
a (Å)	13.020(1)	10.7690(6)	17.380(1)	15.383(1)
b (Å)	13.453(1)	15.0037(8)	14.4876 (9)	15.383(1)
c (Å)	15.257(2)	26.664(2)	21.363(2)	13.808(1)
α (°)	73.77(1)	90	90	90
β (°)	77.16(1)	90	112.124(8)	90
γ (°)	71.94 (1)	90	90	120
V (Å^3)	2412.3(4)	4308.3(4)	4983(3)	2829.6(4)
Z	2	4	4	2
T (K)	153(2)	153(2)	153(2)	153(2)
D_c (g cm^{-3})	1.710	1.834	1.495	1.226
μ (mm^{-1})	3.277	3.905	3.175	2.540
Scan range (°)	$2.0 < 2\theta < 52.0$	$4.1 < 2\theta < 51.9$	$3.8 < 2\theta < 51.8$	$4.4 < 2\theta < 51.8$
Unique reflections	8758	8018	9637	1899
Reflections used [$I > 2\sigma(I)$]	5776	5392	6620	1458
R_{int}	0.0465	0.0719	0.0690	0.0988
Final R indices [$I > 2\sigma(I)$]	0.0316, wR_2 0.0572	0.0433, wR_2 0.0766	0.0628, wR_2 0.1803	0.0545, wR_2 0.1276
R indices (all data)	0.0615, wR_2 0.0623	0.0761, wR_2 0.0827	0.0905, wR_2 0.1914	0.0807, wR_2 0.1396
Goodness-of-fit	0.826	0.828	1.055	1.120
Maximum, minimum $\Delta\rho$ (e Å^{-3})	0.634, −0.615	0.557, −0.463	2.998, −1.711	0.835, −0.520

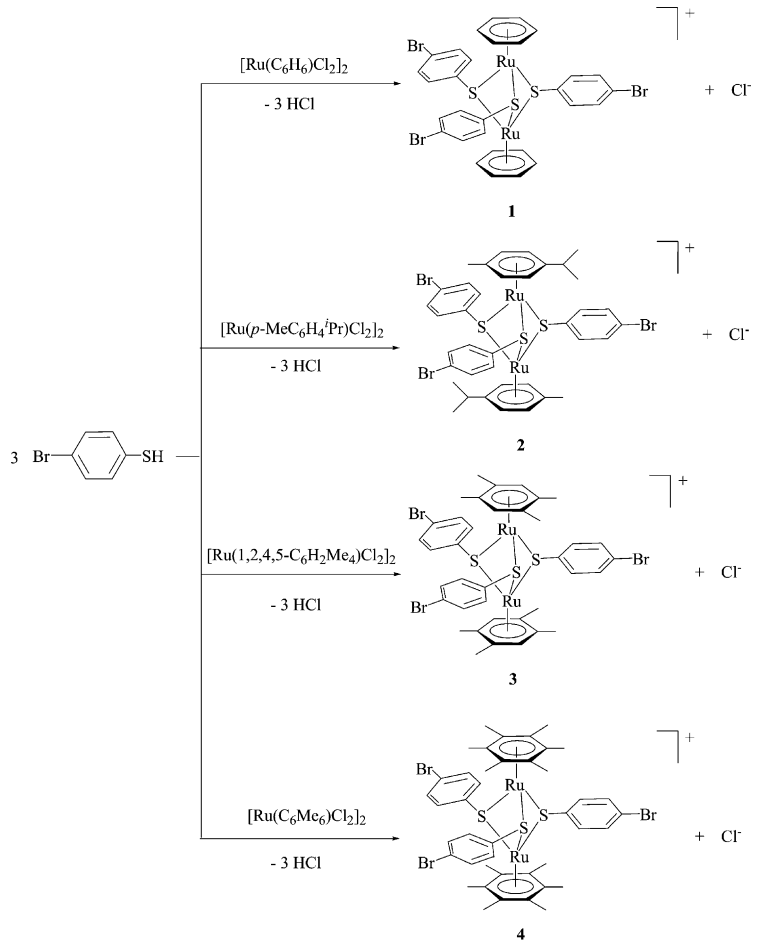
3. Results and discussion

In accordance with our previously reported studies, the dinuclear dichloro complexes $[Ru(C_6H_6)Cl_2]_2$, $[Ru(p\text{-}MeC_6H_4{}^iPr)Cl_2]_2$, $[Ru(1,2,4,5\text{-}C_6H_2Me_4)Cl_2]_2$, and $[Ru(C_6Me_6)Cl_2]_2$ are found to react in ethanol with *p*-bromothiophenol to give the cationic complexes $[Ru_2(C_6H_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**1**), $[Ru_2(p\text{-}MeC_6H_4{}^iPr)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**2**), $[Ru_2(1,2,4,5\text{-}C_6H_2Me_4)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**3**), and $[Ru_2(C_6Me_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**4**), which can be isolated in quantitative yield as the chloride salts (Scheme 1). Cations **1**, **2**, **3** and **4** are unambiguously characterised by their MS, IR, ^1H, and $^{13}C\{^1H\}$ NMR data as well as by satisfactory elemental analysis data of the chloride salts. These cationic complexes are well soluble in alcohols, acetone, and chlorinated solvents.

The bromo-ended star-shaped arrangement adopted by these complexes is very interesting as these compounds represent conductive organometallic nodes. The intramolecular charge transfer (ICT) between the organometallic moieties and the *p*-bromophenyl units is shown by NMR spectroscopy. An important shielding of the signals of the arene ligand coordinated to a ruthenium atom is observed in the NMR spectra, whereas an important deshielding of the *p*-bromophenyl groups is observed. To gain further insight on the ICT effect shown by these complexes, a complete structural study of **1**, **2**, **3** and **4** has been performed. The molecular structures of **1**, **2**, **3** and **4** are presented in Fig. 1, whereas important bond lengths and angles are given in Table 2.

In complexes **1**, **2**, **3** and **4**, the ruthenium atoms are in a disordered octahedral geometry in which the two metal centres are bridged by three *p*-bromophenylthiolato units. The Ru–S bond distances (ranging from 2.385 to 2.435 Å) and Ru–S–Ru angles (ranging from 87.21° to 88.92°) are similar to those found in other dinuclear η^6-arene ruthenium complexes triply bridged by sulfur atoms: $[Ru_2(C_6Me_6)_2(S\text{–}C_6H_5)_3]^+$ [21] and $[Ru_2(p\text{-}MeC_6H_4{}^iPr)_2(S\text{-}C_6H_5)_3]^+$ [22]. In all complexes, the Ru–Ru distance is well outside the range (2.28–2.95 Å) for a metal–metal single bond [22]. Interestingly, the shortest Ru–Ru distance (3.3154(14) Å) has been observed for the hexamethylbenzene derivative, $[Ru_2(C_6Me_6)_2(p\text{-}S\text{–}C_6H_4\text{–}Br)_3]^+$ (**4**). In all cases, the phenyl rings of the η^6-arene ligands are almost parallel with an angle between the two planes of 4.30° in **1**, 4.31° in **2**, 3.39° in **3**, and 0° in **4** where the two hexamethylbenzene planes are related by symmetry.

Because of the relatively large separation between the two ruthenium centres as well as the limited number of substituents attached to the arene ligands, in complexes **1**–**3**, the three phenyl rings of the *p*-bromophenylthiolato ligands are not constrained to a coplanar arrangement. However, as observed in $[Ru_2(Cp)_2(S\text{–}Ar)_3]^+$ [23], complex **4** which possesses six methyl groups attached to the arene ligands shows a



Scheme 1. Synthesis of star-like trisbromo complexes **1**, **2**, **3** and **4**.

Table 2
Selected bond lengths and angles for cations **1**, **2**, **3** and **4**

	1	**2**	**3**	**4**[a]
Distances (Å)				
Ru(1)–S(1)	2.394(1)	2.392(2)	2.378(2)	2.393(1)
Ru(1)–S(2)	2.384(1)	2.419(2)	2.394(2)	
Ru(1)–S(3)	2.397(1)	2.385(2)	2.391(2)	
Ru(2)–S(1)	2.401(1)	2.411(2)	2.395(2)	
Ru(2)–S(2)	2.398(1)	2.399(2)	2.435(2)	
Ru(2)–S(3)	2.401(1)	2.402(2)	2.388(2)	
Ru(1)–Ru(2)	3.3334(6)	3.3532(8)	3.3305(13)	3.3154(14)
Angles (°)				
Ru(1)–S(1)–Ru(2)	88.09(4)	88.56(7)	88.50(7)	87.71(7)
Ru(1)–S(2)–Ru(2)	88.39(3)	88.20(6)	87.21(7)	
Ru(1)–S(3)–Ru(2)	88.03(4)	88.92(8)	88.37(7)	

[a] Symmetry transformation: $x, y, -z - 1/2$.

perfect planarity of the three *p*-bromophenylthiolato units. In **1**, where we find the less hindered arene (C_6H_6), the three phenyl rings are rotated by 45.8°, 48.7°, and 51.0°, respectively, with respect to the plane formed by the three sulfur atoms. In **2**, the presence of a methyl and an isopropyl substituent on the arene ligands forces one phenyl ring to be closer to planarity 25.2°, whereas the two others show an angle of 42.4° and 43.5°, respectively. Cation **3** which possesses four methyl groups attached to the arene ligands presents two almost coplanar phenyl rings (2.1° and 4.3°) and one with an acute angle of 39.2°.

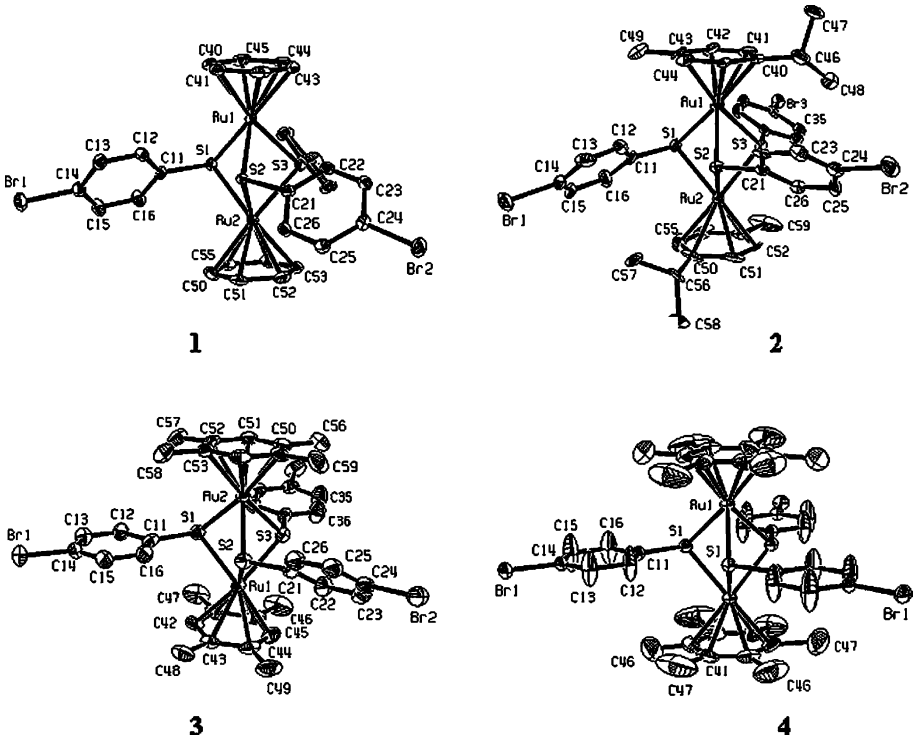


Fig. 1. Molecular structures of cations **1**, **2**, **3** and **4**. Displacement ellipsoids are drawn at the 35% probability level, anions, solvent molecules, and hydrogen atoms are omitted for clarity.

4. Supplementary material

CCDC-218215 (**1**), 197151 (**2**), 218216 (**3**), and 218217 (**4**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44-1223/336-033; e-mail: deposit@ccdc.cam.ac.uk).

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References

[1] J.-M. Lehn, Supramolecular Chemistry, Concepts and Perspectives, VCH, Weinheim, 1995.
[2] R. Ziessel, L. Charbonnière, M. Cesario, T. Prangé, H. Nierengarten, Angew. Chem. Int. Ed. 41 (2002) 975.
[3] K. Sénéchal, O. Maury, H. Le Bozec, I. Ledoux, J. Zyss, J. Am. Chem. Soc. 124 (2002) 4560.
[4] K.K.-W. Lo, W.-K. Hui, D.C.-M. Ng, J. Am. Chem. Soc. 124 (2002) 9344.
[5] D.T. MacQuade, A.E. Pullen, T.M. Swager, Chem. Rev. 100 (2000) 2537.
[6] U.S. Schubert, C. Eschbaumer, Angew. Chem. Int. Ed. 41 (2002) 2892, and references therein.
[7] J. Zyss, I. Ledoux, Chem. Rev. 94 (1994) 77.
[8] C. Dhenault, I. Ledoux, I.D.W. Samuel, M. Bourgault, H. Le Bozec, Nature 374 (1995) 339.
[9] F. Chérioux, L. Guyard, Adv. Func. Mater. 11 (2001) 305.
[10] R. Anémian, J.-C. Mulatier, C. Andraud, O. Stéphan, J.-C. Vial, Chem. Commun. (2002) 1608.
[11] K. Müllen, G. Wegner (Eds.), Electronic Materials: The Oligomer Approach, Wiley–VCH, Weinheim, 1998.
[12] L.L. Miller, Y. Yu, J. Org. Chem. 60 (1995) 6813, and references therein.
[13] F. Chérioux, B. Therrien, G. Süss-Fink, Eur. J. Inorg. Chem. (2003) 1043.
[14] N. Miyaura, A. Suzuki, Chem. Rev. 95 (1995) 2457.
[15] R.A. Zelonka, M.C. Baird, Can. J. Chem. 50 (1972) 3063.
[16] J.W. Wang, K. Moseley, P.M. Maitlis, J. Am. Chem. Soc. (1969) 5970.
[17] M.A. Bennett, A.K. Smith, J. Chem. Soc., Dalton Trans. (1974) 233.
[18] G.M. Sheldrick, Acta Crystallogr., Sect. A 46 (1990) 467.
[19] G.M. Sheldrick, SHELXL-97, University of Göttingen, Göttingen, Germany, 1999.
[20] L.J. Farrugia, J. Appl. Cryst. 30 (1997) 565.
[21] H.T. Schacht, R.C. Haltiwanger, M. Rakowski Dubois, Inorg. Chem. 31 (1992) 1728.
[22] K. Mashima, A. Mikami, A. Nakamura, Chem. Lett. (1992) 1795.
[23] S. Dev, K. Imagawa, Y. Mizobe, G. Cheng, Y. Wakatsuki, H. Yamazaki, M. Hidai, Organometallics 8 (1989) 1232.