$Ce_{16}Mo_{21}O_{56}$ is +2.95 which is close to that based on the stoichiomery, + 3.05, when considering all the Ce ions as trivalent. Bond-valence sums of the Ce—O bonds are 3.09, 3.30, 2.93, 2.97, 3.38, 3.11, 3.20 and 3.07 for Ce(1), Ce(2), Ce(3), Ce(4), Ce(5), Ce(6), Ce(7) and Ce(8), respectively. It is interesting to note that for the total valence sum Σs(Mo—O) + Σs(Ce—O), we obtain a value of 112.05 per formula unit, which is in very good agreement with the theoretical value of 112 based on the 56 O atoms.

Isostructural compounds are also formed with La, Pr and Nd.

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Structure of $[Ru_2Cl_4(Me_2SO)_5]$

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Abstract. *def*-Tri-μ-chloro-*a*-chloro-*bcghi*-pentakis(dimethyl sulfoxide-*S*)diruthenium(II), $[Ru_2Cl_4(C_2H_6OS)_5]$, $M_r = 734.62$, monoclinic, $P2_1/n$, $a = 11.085\,(1)$, $b = 15.647\,(1)$, $c = 15.167\,(2)$ Å, $\beta = 109.94\,(1)^\circ$, $V = 2472.9\,(5)$ Å^3, $Z = 4$, $D_x = 1.973$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 20.6$ cm^{-1}, $F(000) = 1464$, $T = 294$ K, $R = 0.023$, $wR = 0.032$ for 5581 unique observed reflections. The structure is characterized by a triple chloro-bridged diruthenium core [Ru···Ru distance 3.236 (1) Å] with five terminal S-bonded dimethyl sulfoxide ligands. Three are bonded to Rul in a *fac* arrangement, and two are *cis* bonded to Ru2. The distorted octahedral coordination of both metal atoms is completed by Cl ligands.

Introduction. As part of a general study on halide (X) dimethyl sulfoxide (Me_2SO, dmso) ruthenium complexes (Alessio, Balducci, Calligaris, Costa, Attia & Mestroni, 1991), we prepared the title complex and determined its crystal structure, in order to verify the spectroscopically proposed structure (Heath, Lindsay & Stephenson, 1982) and obtain further structural data on $RuX_x(dmso)_y$ complexes.

Experimental. Crystals of $[Ru_2Cl_4(dmso)_5]$, suitable for X-ray analysis, were directly obtained from the reaction when 0.25 g of *cis*-$RuCl_2(dmso)_4$ (0.5 mmol) was partially dissolved in 15 ml of methanol. This mixture was stirred for 8 h at room temperature and then filtered over fine filter paper to remove a small amount of unreacted starting material (checked by NMR). Orange-red crystals of the product formed from the deep yellow solution within a week, upon addition of 3 ml of diethyl ether. They were filtered off, washed with cold methanol and diethyl ether and

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vacuum dried at room temperature. Yield: 0.1 g (55%). The ^1H NMR spectrum of the product ($CDCl_3$, 360 MHz) was very similar to that already reported in the literature (CD_2Cl_2) (Heath *et al.*, 1982), consisting of one singlet and four unresolved multiplets of equal intensity in the region of S-bonded dmso: 3.53 (*s*), 3.50 (*m*), 3.48 (*m*), 3.46 (*m*), 3.40 (*m*) p.p.m.

As has been established (Heath *et al.*, 1982), traces of water (as from commercial methanol) in the reaction medium are essential for the synthesis reaction to occur. This must be connected to the formation, upon removal of coordinated dmso, of aquated species (Alessio, Mestroni, Nardin, Attia, Calligaris, Sava & Zorzet, 1988), probably *cis,fac*-$RuCl_2(dmso)_3(H_2O)$ and *cis,cis,cis*-$RuCl_2(dmso)_2(H_2O)_2$. These can react giving the triple chloro-bridge structure and reforming water, which therefore acts as a catalyst in the formation of the dimer.

On the other hand, it is interesting to observe that the NMR spectra in D_2O clearly show that, upon dissolution in water, the dimer undergoes decomposition mainly with formation of *cis,fac*-$RuCl_2(dmso)_3(H_2O)$ (peaks at 3.48, 3.46 and 3.38 p.p.m.) and *trans,cis,cis*-$RuCl_2(dmso)_2(H_2O)_2$ (peak at 3.34 p.p.m.) (Alessio *et al.*, 1988; Henn, Alessio, Mestroni, Calligaris & Attia, 1991) in equimolar amount.

The crystal had dimensions of approximately 0.2 × 0.2 × 0.7 mm. Weissenberg and precession photographs indicated the crystals to be monoclinic. Data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters were refined by least-squares fit of 25 reflections in the range $14 < \theta < 17°$. Intensity data were measured using $\omega/2\theta$ scans, with ω-scan angle $(0.60 + 0.35\tan\theta)°$ and ω-scan rate 1–7° min^{-1}. Three standard reflections, monitored every 83 min, showed no decay. 6390 reflections were collected ($2 \le \theta \le 28°$; $-14 \le h \le 14$, $0 \le k \le 20$, $0 \le l \le 20$), of which 5581 [$F_o \ge 3.0\sigma(F_o)$] were used in the final calculations. $\sigma(F_o)$ was based on counting statistics. An empirical absorption correction was applied to the data using ψ scans of three reflections (correction factors in the range 0.86–1.00, average transmission factor 0.97). Lorentz and polarization corrections were also made. An extinction correction in the final cycle had a value of $3.4(4) \times 10^{-8}$. The structure was solved using the heavy-atom method. H atoms, located at idealized positions on the basis of ideal bond lengths (C—H 0.95 Å) and angles, were fully confirmed by difference Fourier synthesis; H atoms were assigned thermal parameters B of 1.3 times $B_{\rm eq}$ of the attached C atom; they were not included in the least-squares refinement. Refinement using full-matrix least-squares method, including the extinction parameter and anisotropic thermal factors for non-H atoms (236 refined parameters), led to $R = 0.023$, $wR = 0.032$, $S = 0.602$, $(\Delta/\sigma)_{\rm max} = 0.36$. The function minimized was $\sum_i w_i(|F_o| - |F_c|)^2$, where $w = 1/[1 + \sigma|F_o|^2 + (0.02|F_o|)^2]$. Maximum positive and negative residuals $\Delta\rho$ were 0.67 and -4.23 e Å^{-3}. Atomic scattering factors, anomalous-dispersion terms and programs were as contained in *MolEN* (Enraf–Nonius, 1990). All computations were carried out on a MicroVAX 2000.

Discussion. Final fractional coordinates and equivalent isotropic thermal parameters of non-H atoms are given in Table 1 and bond lengths and angles in Table 2.* A sketch of the molecular structure is shown in Fig. 1. The observed triple chloro-bridged diruthenium structure, with exclusively terminal S-bonded dmso ligands (S-dmso), is in full agreement with the spectroscopic formulation (Heath *et al.*, 1982).

Both Ru atoms have a distorted octahedral geometry, sharing the trichloro face. Ru1 is further bonded to three S-dmso ligands in facial arrangment, while Ru2 is bonded to one Cl ligand and two *cis* S-dmso molecules.

The $Ru_2(\mu\text{-}Cl)_3$ structure is characterized by a Ru···Ru distance of 3.236 (1) Å, and Ru—Cl—Ru and Cl—Ru—Cl bond angles ranging, respectively, from 81.98 (3) to 83.34 (2)° [average 82.5 (8)°], and from 79.66 (3) to 82.24 (3)° [average 81.3 (9)°].

The present Ru···Ru distance is, to our knowledge, the shortest so far reported (3.35–3.44 Å) for Ru^{II}-$(\mu\text{-}Cl)_3$-Ru^{II} complexes (Seddon & Seddon, 1984). It lies within the values of 3.115 Å (Chioccola & Daly, 1968) and 3.28 Å (Contreras, Elliot, Gould, Heath, Lindsay & Stephenson, 1981) found in low-spin Ru^{II}-$(\mu\text{-}Cl)_3$-Ru^{III} phosphine derivatives.

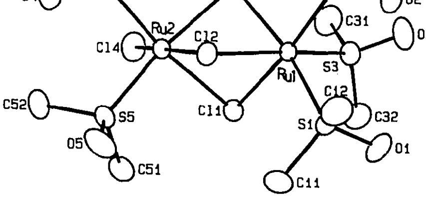

Fig. 1. *ORTEP*II (Johnson, 1976) drawing including the numbering scheme for $[Ru_2Cl_4(dmso)_5]$.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55745 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1015]

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Ru1	0.11647 (2)	0.00823 (1)	0.27478 (1)	1.589 (4)
Ru2	−0.13133 (2)	−0.11581 (1)	0.19985 (1)	1.704 (4)
Cl1	−0.10024 (6)	0.02593 (5)	0.27887 (5)	2.25 (1)
Cl2	−0.00040 (6)	−0.05044 (4)	0.11934 (4)	2.04 (1)
Cl3	0.08066 (7)	−0.13747 (5)	0.32218 (5)	2.46 (1)
Cl4	−0.24553 (8)	−0.16661 (6)	0.29742 (5)	3.26 (2)
S1	0.11496 (7)	0.13812 (4)	0.20774 (5)	2.10 (1)
S2	0.31478 (6)	−0.02555 (5)	0.27373 (5)	2.29 (1)
S3	0.20184 (7)	0.05989 (5)	0.42287 (5)	2.33 (1)
S4	−0.12642 (7)	−0.24395 (5)	0.13718 (5)	2.27 (1)
S5	−0.31682 (6)	−0.07579 (5)	0.09379 (5)	2.20 (1)
O1	0.1695 (3)	0.2123 (2)	0.2679 (2)	3.37 (5)
O2	0.4043 (2)	0.0437 (2)	0.2736 (2)	3.43 (5)
O3	0.3375 (2)	0.0849 (2)	0.4568 (2)	3.69 (6)
O4	−0.2237 (3)	−0.2672 (2)	0.0461 (2)	4.10 (6)
O5	−0.3152 (2)	−0.0358 (2)	0.0062 (2)	3.59 (5)
C11	−0.0447 (4)	0.1676 (2)	0.1383 (3)	3.65 (8)
C12	0.1857 (3)	0.1363 (2)	0.1188 (2)	3.36 (7)
C21	0.3060 (3)	−0.0931 (2)	0.1777 (3)	3.16 (6)
C22	0.3981 (3)	−0.0945 (3)	0.3681 (3)	3.67 (8)
C31	0.1805 (4)	−0.0142 (3)	0.5050 (2)	3.86 (8)
C32	0.1129 (4)	0.1480 (2)	0.4443 (2)	3.49 (7)
C41	−0.1248 (4)	−0.3265 (2)	0.2173 (3)	3.92 (8)
C42	0.0246 (3)	−0.2642 (2)	0.1247 (3)	3.28 (7)
C51	−0.3938 (3)	−0.0016 (3)	0.1461 (3)	3.33 (7)
C52	−0.4378 (3)	−0.1563 (3)	0.0617 (3)	3.80 (8)

Table 2. *Bond distances* (Å) *and angles* (°)

Ru1—Cl1	2.440 (1)	S1—O1	1.472 (2)
Ru1—Cl2	2.449 (1)	S1—C11	1.784 (3)
Ru1—Cl3	2.463 (1)	S1—C12	1.776 (4)
Ru1—S1	2.270 (1)	S2—O2	1.470 (3)
Ru1—S2	2.266 (1)	S2—C21	1.775 (4)
Ru1—S3	2.268 (1)	S2—C22	1.778 (4)
Ru2—Cl1	2.488 (1)	S3—O3	1.467 (3)
Ru2—Cl2	2.419 (1)	S3—C31	1.776 (4)
Ru2—Cl3	2.471 (1)	S3—C32	1.788 (4)
Ru2—Cl4	2.388 (1)	S4—O4	1.479 (3)
Ru2—S4	2.228 (1)	S4—C41	1.770 (4)
Ru2—S5	2.225 (1)	S4—C42	1.776 (4)
		S5—O5	1.474 (3)
		S5—C51	1.779 (4)
		S5—C52	1.782 (4)

S2—Ru1—S3	90.69 (3)	Ru1—Cl3—Ru2	81.98 (3)
S1—Ru1—S3	93.52 (3)	Ru1—S1—C12	112.8 (1)
S1—Ru1—S2	93.71 (3)	Ru1—S1—C11	110.5 (1)
Cl3—Ru1—S3	95.46 (3)	Ru1—S1—O1	119.3 (1)
Cl3—Ru1—S2	92.26 (3)	C11—S1—C12	99.3 (2)
Cl3—Ru1—S1	169.15 (3)	O1—S1—C12	106.8 (2)
Cl2—Ru1—S3	172.82 (3)	O1—S1—C11	106.1 (2)
Cl2—Ru1—S2	95.83 (3)	Ru1—S2—C22	112.0 (1)
Cl2—Ru1—S1	89.07 (3)	Ru1—S2—C21	111.3 (1)
Cl2—Ru1—Cl3	81.32 (3)	Ru1—S2—O2	119.0 (1)
Cl1—Ru—S3	90.93 (3)	C21—S2—C22	99.6 (2)
Cl1—Ru1—S2	172.96 (3)	O2—S2—C22	106.4 (2)
Cl1—Ru1—S1	93.03 (3)	O2—S2—C21	106.6 (2)
Cl1—Ru1—Cl3	80.77 (3)	Ru1—S3—C32	112.7 (1)
Cl1—Ru1—Cl2	82.24 (3)	Ru1—S3—C31	110.6 (1)
S4—Ru2—S5	95.50 (3)	Ru1—S3—O3	118.3 (1)
Cl4—Ru2—S5	89.27 (3)	C31—S3—C32	99.4 (2)
Cl4—Ru2—S4	93.45 (3)	O3—S3—C32	107.1 (2)
Cl3—Ru2—S5	171.53 (3)	O3—S3—C31	107.1 (2)
Cl3—Ru2—S4	92.38 (3)	Ru2—S4—C42	111.6 (1)
Cl3—Ru2—Cl4	93.35 (3)	Ru2—S4—C41	111.1 (1)
Cl2—Ru2—S5	94.68 (3)	Ru2—S4—O4	120.2 (1)
Cl2—Ru2—S4	93.38 (3)	C41—S4—C42	98.8 (2)
Cl2—Ru2—Cl4	171.75 (3)	O4—S4—C42	105.7 (2)
Cl2—Ru2—Cl3	81.74 (3)	O4—S4—C41	107.2 (2)
Cl1—Ru2—S5	92.26 (3)	Ru2—S5—C52	115.0 (1)
Cl1—Ru2—S4	171.20 (3)	Ru2—S5—C51	109.1 (1)
Cl1—Ru2—Cl4	90.79 (3)	Ru2—S5—O5	118.6 (1)
Cl1—Ru2—Cl3	79.66 (3)	C51—S5—C52	98.8 (2)
Cl1—Ru2—Cl2	81.83 (2)	O5—S5—C52	107.1 (2)
Ru1—Cl1—Ru2	82.09 (2)	O5—S5—C51	106.3 (2)
Ru1—Cl2—Ru2	83.34 (2)		

The Ru—μ-Cl bond distances *trans* to S atoms [2.440 (1)–2.488 (1) Å] are longer than that *trans* to Cl [2.419 (1) Å]. All are longer than the Ru—Cl(terminal) (Ru2—Cl4) bond distance of 2.388 (1) Å. This last distance is only slightly shorter than the average value of 2.41 (1) Å found in various *trans* Cl-Ru^{II}-Cl groups (Calligaris, Bresciani-Pahor & Srivastava, 1993).

It is interesting to observe that the Ru1—S bond distances [2.266 (1)–2.270 (1) Å] are slightly, but significantly, longer than the Ru2—S distances [2.225 (1)–2.228 (1) Å]. The former range of values, corresponding to three dmso ligands in a *fac* arrangement, are in the range of values found in *cis,fac*-$RuCl_2(dmso)_3(NH_3)$ [2.246 (1)–2.288 (1) Å (Henn *et al.*, 1991)], as well as in *fac*-$[RuCl_3(dmso)_3]^-$ [2.252 (4)–2.273 (5) Å (McMillan, Mercer, James & Trotter, 1975)], and *fac*-$[RuBr_3(dmso)_3]^-$ [2.23 (1)–2.283 (7) Å (Alessio, Milani, Calligaris & Bresciani-Pahor, 1992)]. The Ru2—S distances, corresponding to two *cis* dmso ligands, are shorter than the Ru—S, *trans* to Cl, distances in *cis*-$RuCl_2(dmso)_4$ [average 2.279 (7) Å (Alessio *et al.*, 1988)]. They are closer to the values found in *trans,cis,cis*-$RuCl_2(dmso)_2(NH_3)_2$ [2.235 (1)–2.247 (1) Å (Henn *et al.*, 1991)].

The non-equivalence of the two Ru–dmso groups is attributable to the different electron charge density on the two metal atoms and to their different steric environment.

The X-ray results show that the decomposition reaction in water (see *Experimental*) requires the selective hydrolysis of the three chloro bridges at Ru2—Cl3, Ru2—Cl1 and Ru1—Cl2 bonds yielding, in order to form the spectroscopically detected compounds, *cis,fac*-$RuCl_2(dmso)_3(H_2O)$ and *trans,cis,cis*-$RuCl_2(dmso)_2(H_2O)_2$.

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Structure of a Nickel(II) Complex of the Deprotonated Anion of 3,6,6,9-Tetramethyl-4,8-diaza-3,8-undecadiene-2,10-dione Dioxime

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Abstract. [3,3′-(2,2-Dimethyl-1,3-propanediyldinitrilo)di(2-butanone oximato)(1 −)-*N*,*N*′,*N*″,*N*‴] nickel perchlorate, $[Ni(C_{13}H_{23}N_4O_2)](ClO_4)$, $M_r = 425.5$, monoclinic, $P2_1/c$, $a = 7.161\ (3)$, $b = 18.945\ (3)$, $c = 13.949\ (4)$ Å, $\beta = 103.47\ (1)°$, $V = 1840.2\ (9)$ Å^3, $Z = 4$, $D_x = 1.536$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0.7093$ Å, $\mu = 1.24$ mm^{-1}, $F(000) = 887.81$, $T = 298\ (4)$ K, $R = 0.039$, $wR = 0.040$, for 2729 significant reflections. The coordination about Ni$^{\rm II}$ is slightly distorted square planar. The Ni$^{\rm II}$ ion is a little (0.056 Å) out of the square coordination plane, towards the perchlorate ion. There are intramolecular and intermolecular hydrogen bonds between O atoms.

Introduction. Transition-metal complexes of deprotonated diaza-dioximes present considerable interest in the formation of strong intramolecular hydrogen bonds between *cis* oxime groups (Wang, Chung, Cheng & Wang, 1990; Fair & Schlemper, 1978; Gavel & Schlemper, 1979; Liss & Schlemper, 1975). We have recently reported the structure of a Ni$^{\rm II}$ complex with the deprotonated anion of 3,9-dimethyl-4,8-diaza-3,8-undecadiene-2,10-dione dioxime (Wang, Chung, Cheng & Wang, 1990).

Experimental. 2,3-Butanedione monoxime (115.4 mmol) was dissolved in ethanol (200 ml), to which 2,2-dimethyl-1,3-propanediamine (49 mmol) in ethanol (100 ml) was added dropwise; the solution was refluxed for 5 h, then allowed to cool to room temperature. The solvent was removed under reduced pressure, and the white product was washed with cold acetonitrile and recrystallized from benzene. A hot methanol solution of nickel(II) perchlorate hexahydrate (10 mmol in 30 ml) was added to a hot methanol solution of the ligand 3,6,6,9-tetramethyl-4,8-diaza-3,8-undecadiene-2,10-dione dioxime (10 mmol in 20 ml). The orange solution was cooled and precipitated into orange products which were collected by filtration, washed with diethyl ether, dried in air and then recrystallized from methanol. The crystal used for the X-ray study had dimensions 0.38 × 0.63 × 0.69 mm. The intensity data were collected on a Nonius CAD-4 diffractometer using the θ–2θ scan mode. Cell dimensions were obtained from 25 reflections with 2θ angles in the range 11–32°. The *hkl* ranges were $-8 \le h \le 8$, $0 \le k \le 22$, $0 \le l \le 16$. Maximum $\sin\theta/\lambda = 0.594$ Å^{-1}. Three normalized standard reflections ($\bar{2}\bar{5}\bar{3}$, $\bar{2}71$ and $\bar{2}80$) fluctuated within 1%. 3509 valid reflections were measured, of which 3234 were unique with 2729 significant $[I \ge 2.5\sigma(I)]$. An empirical absorption correction was applied based on azimuthal rotation from three reflections (364, 333 and 586) (North, Philips & Mathews, 1968); the maximum and minimum transmission factors were 0.759 and 0.758, respectively. Patterson synthesis, difference Fourier synthesis and least-squares fit were used to locate the Ni atom and other atoms. The function minimized in the full-matrix least-squares fit was $\sum(|F_o| - |F_c|)^2$, with unit weights. 262 parameters were refined with anisotropic temperature factors for non-H atoms and isotropic for H atoms, the H-atom positions

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