A new dinuclear ruthenium complex bridged by azide: [Ru$_2$Cl$_2$(μ-1,3-N$_3$)$_2$(Me$_2$SO)$_6$]

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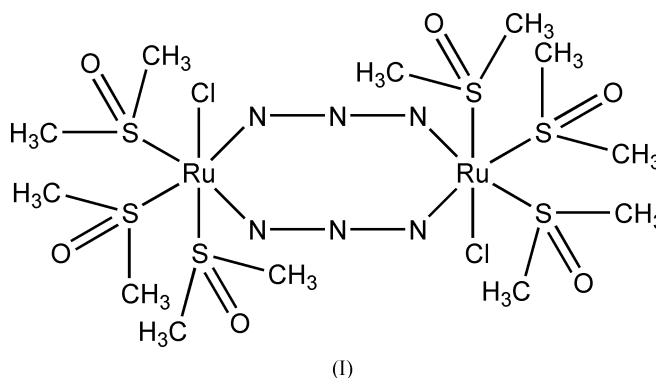
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Reaction of [RuCl$_2$(Me$_2$SO)$_4$] with sodium azide in water affords the title complex di-μ-1,3-azido-bis[chlorotris(dimethyl sulfoxide-κS)ruthenium(II)], [Ru$_2$Cl$_2$(N$_3$)$_2$(C$_2$H$_6$-OS)$_6$]. X-Ray crystal structure determination has been performed on the title compound. The result reveals that two [Ru(Me$_2$SO)$_3$]$^{2+}$ units are linked by two bridging azide ligands to form a centrosymmetric complex. The geometry about each Ru atom is essentially octahedral, with three S atoms of Me$_2$SO arranged in a facial configuration, two coordinated N atoms of bridging N$_3$$^-$ ligands and a chloride anion.

Comment

Studies of ruthenium(II)–dimethyl sulfoxide complexes have been well developed (Alessio *et al.*, 1998; Serli *et al.*, 2002). The chemistry of these complexes has wide potential in many fields, such as homogeneous catalysis (Srivastava *et al.*, 1992), antitumour activity (Mestroni *et al.*, 1987) and synthesis of ruthenium complexes (Lessing *et al.*, 1999). These reported complexes are almost all mononuclear, and only a few dinuclear ruthenium(II)–dimethyl sulfoxide complexes have been reported. During the course of our studies on ruthenium complexes containing dimethyl sulfoxide, we obtained the dinuclear ruthenium(II) complex [Ru$_2$Cl$_2$(μ-1,3-N$_3$)$_2$-(Me$_2$SO)$_6$], (I), and the synthesis and crystal structure are reported here.

(I)

In the title compound (Fig. 1), each Ru atom is situated in a distorted octahedral geometry, with six coordinating atoms, *viz.* three S atoms of dimethyl sulfoxide, one Cl atom and two coordinated N atoms of N$_3$$^-$. The two bridging N$_3$$^-$ anions are linked to two [RuCl(Me$_2$SO)$_3$]$^+$ units in the 1,3-bridge coordination model. The Ru1—N1 and Ru1—N3*A* (symmetry code as in Fig. 1) bond distances are 2.132 (3) and 2.135 (3) Å, respectively. These values are longer than those found in mononuclear complexes that contain Ru—N bonds (Masood

et al., 1994; Concepcio *et al.*, 2002). Moreover, the two N_3^- anions are almost linear, and the N1—N2—N3 bond angle is 176.6 (3)°. The Ru—S bond lengths range from 2.2669 (1) to 2.2772 (1) Å, which are very typical for bond distances of S from DMSO bonded to ruthenium(II) (Mercer & Trotter, 1975).

Experimental

Dichlorotetrakis(dimethyl sulfoxide)ruthenium (96.8 mg, 0.2 mmol) was dissolved in water (20 ml) containing sodium azide (13.0 mg, 0.2 mmol). The solution was stirred for 4 h. The resulting yellow solution was filtered and left to stand undisturbed. After a month, red crystals suitable for X-ray analysis were obtained by slow evaporation of the filtrate.

Crystal data

$[Ru_2Cl_2(N_3)_2(C_2H_6OS)_6]$	$D_x = 1.908$ Mg m^{-3}
$M_r = 412.93$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1005
$a = 8.141$ (2) Å	reflections
$b = 11.822$ (3) Å	$\theta = 3.0–26.2°$
$c = 14.934$ (4) Å	$\mu = 1.71$ mm^{-1}
$\beta = 90.217$ (5)°	$T = 293$ (2) K
$V = 1437.2$ (7) Å^3	Block, red
$Z = 4$	$0.26 \times 0.14 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	2915 independent reflections
	2276 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.038$
Absorption correction: multi–scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 26.4°$
	$h = -8 \rightarrow 10$
$T_{min} = 0.452$, $T_{max} = 0.843$	$k = -14 \rightarrow 12$
8004 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$+ 0.6144P]$
$wR(F^2) = 0.065$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
2915 reflections	$\Delta\rho_{max} = 0.49$ e Å^{-3}
161 parameters	$\Delta\rho_{min} = -0.46$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.0011 (2)

H atoms were included in the riding-model approximation, with C—H distances of 0.95 Å and $U_{iso}(H) = 1.5U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine
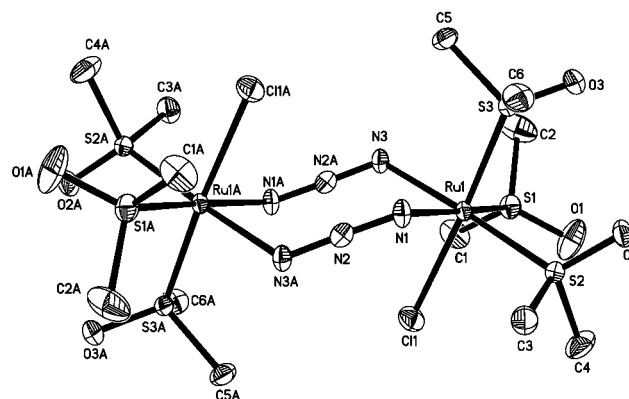

Figure 1
Perspective view of the title complex (I). Displacement ellipsoids are shown at the 30% probability level and H atoms have been omitted for clarity. [Symmetry code: (A) $-x + 1$, $-y + 1$, $-z + 1$.]

structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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