Note

Synthesis and crystal structure of [Ru$_8$(μ$_5$-S)$_2$(μ$_4$-S)(μ$_3$-S)(μ-CNMe$_2$)$_2$(μ-CO)(CO)$_{15}$] formed via the double sulphur–carbon bond cleavage of dithiocarbamate ligands

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Abstract

Heating *cis*-[Ru(S$_2$CNMe$_2$)$_2$(CO)$_2$] and [Ru$_3$(CO)$_{12}$] in xylene affords octanuclear [Ru$_8$(μ$_5$-S)$_2$(μ$_4$-S)(μ$_3$-S)(μ-CNMe$_2$)$_2$(μ-CO)(CO)$_{15}$] resulting from the double carbon–sulfur bond cleavage of two dithiocarbamate ligands. The structure consists of a tri-edge-bridged square of ruthenium atoms with a further ruthenium atom being bound only to the central bridging atom. Studies suggest that it may be formed via the pentanuclear intermediate [Ru$_5$(μ$_4$-S)$_2$(μ-CNMe$_2$)$_2$(CO)$_{11}$] which is formed in trace amounts.
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1. Introduction

Dithiocarbamates are an important class of ligand known to form complexes with all of the transition elements to give complexes with many applications [1]. In by far the vast majority of these the dithiocarbamate acts as simple stabilising ligand, the ease with which it can be modified to tailor the electronic properties of the metal centre rendering it particularly attractive. However, in an increasing number of instances the dithiocarbamate has been shown to act in a non-innocent fashion. The first observation of this behaviour was made in 1973, with Ricard and co-workers showing that the product formed upon reaction of [Mo$_2$(μ-OAc)$_4$] and four equivalents of NaS$_2$CNPr$_2$ was the molybdenum(IV) complex [Mo(μ-S)(S$_2$CNPr$_2$)(η$_2$-SCNPr$_2$)]$_2$ [2]. This results from the cleavage of a single carbon–sulfur bond, generating sulfido and

thiocarboxamide ligands, and can be viewed as an oxidative-addition reaction, the oxidation state at molybdenum going from +2 to +4. In the following thirty years, the non-innocent behavior of dithiocarbamates, while still relatively rare, has become more prevalent. A number of different types of non-innocent behavior have been found, the cleavage of a single sulfur–carbon bond cleavage being most common [2–16], and these cleavage reactions have been exploited in the preparation of metal sulfides from volatile molecular precursors in MOCVD chemistry [1].

The cleavage of both sulfur–carbon bonds of a dithiocarbamate ligand is in theory a four-electron process, resulting in the formation of two sulfido groups and a single aminocarbyne ligand. Despite the importance of this process in the MOCVD process, it has only rarely been observed or postulated to have occurred [17–22]. Given the oxidative nature of the process it might be expected to occur primarily at low-valent centers. Recent work by Goh and co-workers has shed considerable light of this carbon–sulfur bond scission process [19]. When

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$[CpCr(CO)_2(S_2CNR_2)]$ and $[CpCr(CO)_3]_2$ are heated a number of products are obtained, including the thiocarboxamide and aminocarbyne complexes, $[CpCr(CO)_2(\eta_2\text{-}SCNR_2)]$ and $[CpCr(CO)_2(CNR_2)]$, respectively. While the former is the product of a single sulfur–carbon bond scission, the aminocarbyne results from a double bond cleavage. Importantly, further heating of $[CpCr(CO)_2(\eta_2\text{-}SCNR_2)]$ and $[CpCr(CO)_3]_2$ affords $[CpCr(CO)_2(CNR_2)]$ together with the cubane clusters, $[CpCr(\mu_3\text{-}S)]_4$ and $[Cp_4Cr_4(\mu_3\text{-}S)_2(\mu_3\text{-}CO)(\mu_3\text{-}CNR_2)]$, providing further evidence that the scission process occurs in a step-wise fashion.

In earlier work from our laboratory we found that heating the ruthenium(II) complex cis-$[Ru(CO)_2(S_2CNEt_2)_2]$ with an excess of $[Ru_3(CO)_{12}]$ at 130 °C yielded pentanuclear, $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNEt_2)_2(CO)_{11}]$, being postulated to result from a double sulfur–carbon bond cleavage reaction [17]. At the time of this work a second minor product was observed by TLC but could not be characterized because of the extremely small amounts generated. We have recently returned to this chemistry with the aim of elucidating more details of the precise transformations involved and products generated. Herein, we report the synthesis of octanuclear $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$, the major product of the thermolysis of cis-$[Ru(CO)_2(S_2CNMe_2)_2]$ with $[Ru_3(CO)_{12}]$.

2. Results and discussion

An equimolar mixture of cis-$[Ru(S_2CNMe_2)_2(CO)_2]$ and $[Ru_3(CO)_{12}]$ was dissolved in xylene and heated under reflux for 4 h. After removal of volatiles a dark brown solid was obtained, which was purified by passing a dichloromethane solution through a small silica column. Further purification by TLC resulted in the isolation of one small red band, a green band and an uncharacterized yellow band. By comparison to the literature data for $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNEt_2)_2(CO)_{11}]$ [17], the red band was proposed by IR spectroscopy to be $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNMe_2)_2(CO)_{11}]$. The small amounts obtained precluded further characterization, a +ve FAB mass spectrum providing little useful information. The green solid was crystallized by slow evaporation of an acetone solution to give small dark green crystals. These were analyzed by single-crystal X-ray diffraction and shown to be an acetone solvate of the cluster $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ (Figs. 1 and 2).

The molecule has a crystallographic plane of symmetry which includes atoms S(2)–Ru(4)–S(3)–Ru(5)–C(12)–O(12). The cluster core consists of eight ruthenium atoms and four bridging sulfido ligands. The metal atoms are arranged in a very open fashion. A central square consisting of Ru(1), Ru(2) and their symmetry generated equivalents is edge-bridged along three sides, the unique edge-bridging atom, Ru(4), being further bound to a *spiked* ruthenium atom, Ru(5). Ruthenium–ruthenium bond lengths cover a considerable range varying between 2.6935(9) and 3.0082 (7) Å. Most of these are typical of ruthenium–ruthenium single
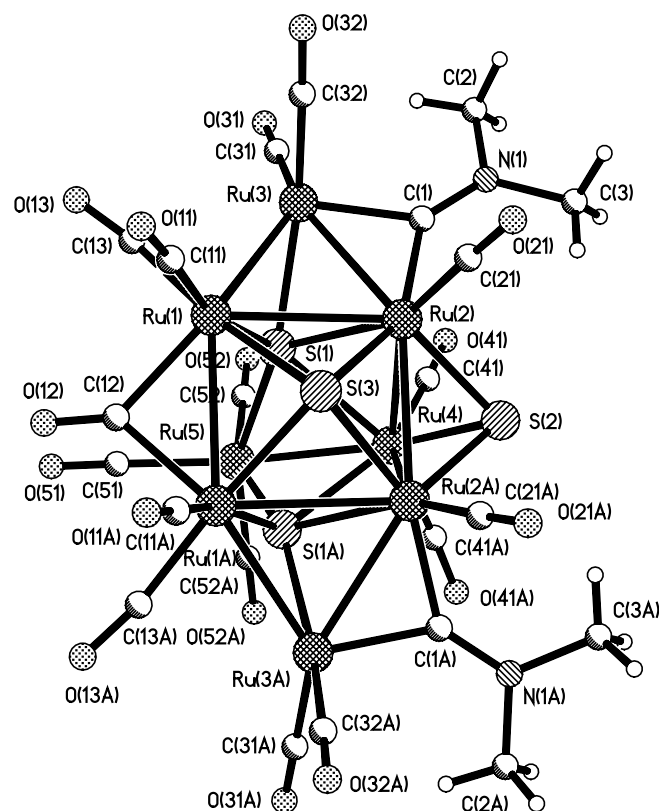

Fig. 1. Molecular structure of $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ with selected bond lengths (Å): Ru(1)–Ru(1A) 2.9135(8), Ru(1)–Ru(2) 2.9411(6), Ru(1)–Ru(3) 2.7731(6), Ru(2)–Ru(2A) 2.8077(8), Ru(2)–Ru(3) 2.7459(7), Ru(2)–Ru(4) 3.0082(7), Ru(4)–Ru(5) 2.6935(9), Ru(1)–S(1) 2.4012(14), Ru(1)–S(3) 2.4022(15), Ru(2)–S(1) 2.6533(15), Ru(2)–S(2) 2.3016(15), Ru(2)–S(3) 2.3777(15), Ru(3)–S(1) 2.3508(14), Ru(4)–S(1) 2.4732(14), Ru(4)–S(2) 2.334(2), Ru(5)–S(1) 2.4043(14), Ru(2)–C(1) 2.038(6), Ru(3)–C(1) 1.964(7).

bonds in electron-precise clusters, although the longest [Ru(2)–Ru(4)] is more indicative of electron-rich complexes [23]. Three types of bridging sulfido groups are seen. One of these, S(2), is triply-bridging capping the closed-triangle, Ru(4)–Ru(2)–Ru(2A), in an approximately symmetrical fashion. A second, S(3), is quadruply bridging and caps the central square of ruthenium atoms, again approximately symmetrically. The final sulfido groups (S(1) and its symmetry related equivalent) bind in a highly asymmetric manner to five ruthenium atoms, Ru(1)–Ru(5). Ruthenium–sulfur bonds in the cluster vary between 2.3508(14) and 2.6533(15) Å. The longest [Ru(2)–S(1)] is outside of the range usually seen for sulfido-bridged ruthenium clusters [24], leading to an alternative view of S(1) as a quadruply bridging ligand. The dimethylaminomethylidyne ligands bridge the Ru(2)–Ru(3) vector in a somewhat asymmetric fashion [Ru(2)–C(1) 2.038(6), Ru(3)–C(1) 1.964(7) Å], restricted rotation about the carbon–nitrogen bond leading to the observation of two methyl singlets in the room temperature ^{1}H NMR spectrum. The remaining coordination sphere comprises sixteen carbonyls. All are terminally bound except for CO(12) which bridges the Ru(1)–Ru(1A) vector and is observed in the IR spectrum at 1836 cm^{-1}.
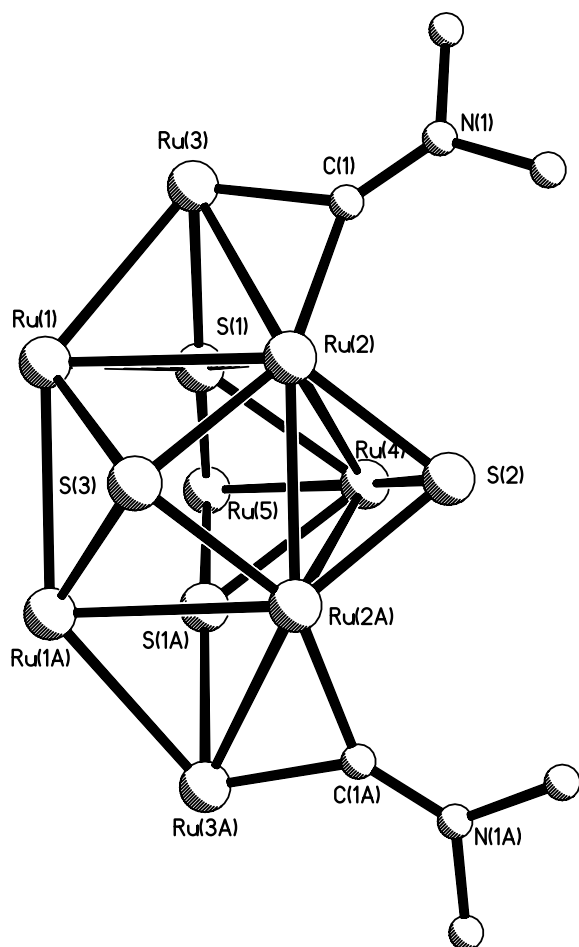
Fig. 2. Cluster core of $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ with carbonyls removed for clarity.
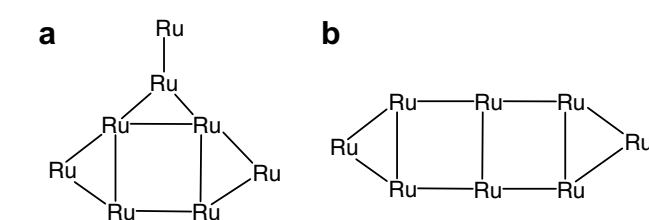

Fig. 3. Schematic representations of the metal core geometries of 112 valence electron octanuclear clusters: (a) $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ and (b) $[Ru_8(\mu\text{-}H)(\mu_4\text{-}ampy)(\mu_3\text{-}ampy)(\mu\text{-}CO)_2(CO)_{15}]$.

A handful of octaruthenium clusters have previously been crystallographically characterized [24–32] the majority containing quite closed octaruthenium cores. The cluster core of $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ is very open, containing a total of 122 valence electrons. This is in accord with the 18-electron rule which predicts the observed 11 M–M bonds. Very recently, Cabeza and co-workers have reported the synthesis of $[Ru_8(\mu\text{-}H)(\mu_4\text{-}ampy)(\mu_3\text{-}ampy)(\mu\text{-}CO)_2(CO)_{15}]$ $(H_2ampy = 2\text{-amino-6-methylpyridine})$ upon high temperature condensation of $[Ru_3(\mu\text{-}H)(\mu_3\text{-}ampy)(CO)_9]$ with $[Ru_3(CO)_{12}]$ [31]. This cluster is also characterized by 122 valance electrons and 11 M–M bonds, the core consisting of two edge-sharing squares, each square having its most distant edge bridged by an additional metal atom (Fig. 3).

Formation of green $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ formally results from the double sulfur–carbon bond cleavage of both of the dithiocarbamate ligands in $cis\text{-}[Ru(S_2CNMe_2)_2(CO)_2]$, upon addition of $2\frac{1}{3}$ equivalents of $[Ru_3(CO)_{12}]$ with the overall loss of 14 carbonyls. This may, however, be a rather simplistic interpretation. In the analogous reaction with $cis\text{-}[Ru(S_2CNEt_2)_2(CO)_2]$ the major product was found to be red $[Ru_5(\mu_4\text{-}S)_2\text{-}$

$(\mu\text{-}CNEt_2)_2(CO)_{11}]$ with a minor green product being observed [17]. A minor product from the thermolysis of $cis\text{-}[Ru(S_2CNMe_2)_2(CO)_2]$ was also red and on the basis of IR spectroscopy was tentatively characterized as $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNMe_2)_2(CO)_{11}]$. It may be that the pentanuclear cluster is an intermediate in the formation of the final octanuclear product. This would require the addition of three ruthenium and two sulfur atoms (along with associated carbonyls) (Fig. 4). The most likely candidates for such a cluster expansion would be $[Ru_3(\mu_3\text{-}S)_2(CO)_9]$ or $[Ru_3(\mu_3\text{-}S)(\mu_3\text{-}CO)(CO)_9]$ which has been shown by Adams and co-workers to work well in this capacity [32–35] and in support of this, we have found that thermolysis of $cis\text{-}[Ru(S_2CNEt_2)_2(CO)(PEt_3)]$ with $[Ru_3(CO)_{12}]$ gives $[Ru_3(\mu_3\text{-}S)_2(CO)_8(PEt_3)]$ as the major product [36]. Unfortunately despite repeated attempts we were unable to isolate significant quantities of $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNMe_2)_2(CO)_{11}]$ in order to carry out this experiment.

As detailed in Section 1, Goh and co-workers have observed the double carbon–sulfur bond cleavage of dithiocarbamates using chromium(I) complexes [19]. During this work they found that similar products were formed when $[CpCr(CO)_3]_2$ was heated with $[Cr(S_2CNR_2)_3]$. This led us to attempt the thermolysis of $[Ru(S_2CNEt_2)_3]$ [37] with $[Ru_3(CO)_{12}]$ in refluxing xylene. After heating for 4 h, workup afforded a mixture of $cis\text{-}[Ru(CO)_2(S_2CNEt_2)_2]$ and $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNEt_2)_2(CO)_{11}]$ [17] suggesting that initial reduction of the ruthenium(III) complex may occur being followed by further reaction with $[Ru_3(CO)_{12}]$.

3. Experimental

3.1. Synthesis of $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$

A xylene solution (20 cm³) of $cis\text{-}[Ru(S_2CNMe_2)_2(CO)_2]$ (0.05 g, 0.126 mmol) and $Ru_3(CO)_{12}$ (0.08 g, 0.126 mmol) was refluxed for 4 h leading to a colour change from yellow to black. After cooling to room temperature, volatiles were removed under reduced pressure to give a black solid which was extracted into dichloromethane (10 cm³) and passed through a short silica column. This gave a brown solid which was purified by TLC on silica using light petroleum (40–60 °C) and dichloromethane (10:7). Three bands eluted
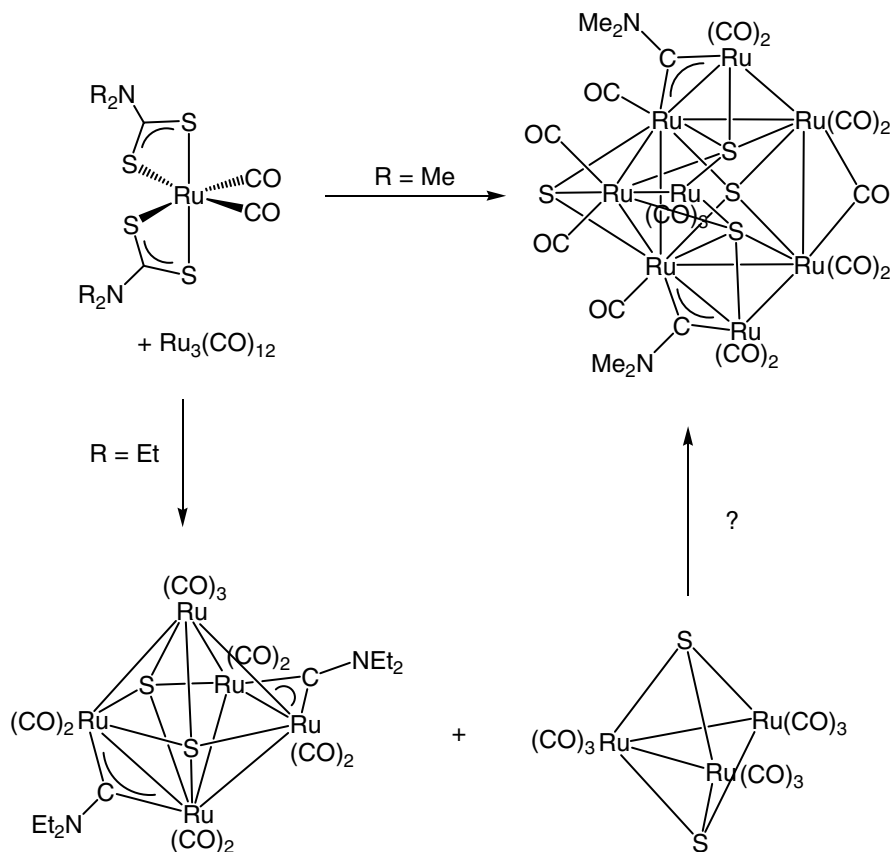
Fig. 4. Possible mode of formation of $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNR_2)_2(\mu\text{-}CO)(CO)_{15}]$ *via* $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNR_2)_2(CO)_{11}]$.

in order; (i) red band $[Ru_5(\mu_4\text{-}S)_2(\mu\text{-}CNMe_2)_2(CO)_{11}]$ (ca. 2 mg) IR (C_6H_{14}) $\nu(CO)$ 2064w, 2031m, 2016vs, 2000m, 1981m, 1931w cm^{-1}, (ii) green band $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)$-$(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$ (64 mg, 34%) IR (C_6H_{14}) $\nu(CO)$ 2076m, 2046s, 2023vs, 2008m, 1979s, 1952m, 1836w cm^{-1}, ^{1}H NMR (CDCl$_3$) δ 4.33 (s, 6H, Me), 3.64 (s, 6H, Me), and (iii) yellow band (ca. 5 mg). Crystallization of $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(CNMe_2)_2(CO)_{16}]$ upon slow evaporation of an acetone solution afforded a number of dark green crystals identified as $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}] \cdot$ acetone.

3.2. X-ray data collection and solution

A single crystal was mounted on a glass fibre and all geometric and intensity data were taken from this sample using a Bruker SMART APEX CCD diffractometer using graphite-monochromated Mo Kα radiation $(\lambda = 0.71073$ Å) at 298 ± 2 K. Data reduction was carried out with SAINT+ [38] and absorption correction applied using the programme SADABS [39]. The structure was solved by direct methods [40] and developed [41] using alternating cycles of least-squares refinement and difference-Fourier synthesis. All non-hydrogen atoms were refined anisotropically except those of the acetone solvate which were refined only isotropically and hydrogens were placed in calculated positions (riding model). The positions of the heavy atoms in the acetone

solvate were fixed in the final cycles as the central carbon atom, C(6), kept drifting. Structure solution used SHELXTL PLUS V6.10 program package [42].

4. Crystallographic data

Crystallographic data for $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)$-$(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}] \cdot$ acetone: green block, dimensions $0.39 \times 0.13 \times 0.07$ mm, orthorhombic, space group $Pmn2_1$, $a = 17.9106(11)$ Å, $b = 10.3076(6)$ Å, $c = 11.5564(7)$ Å, $V = 2133.5(2)$ Å^3, $Z = 2$, $F(000)$ 1468, $d_{calc} = 2.421$ g cm^{-3}, $\mu = 3.013$ mm^{-1}. 18 399 reflections were collected, 5206 unique [$R_{int} = 0.0296$] of which 4840 were observed [$I > 2.0\sigma(I)$]. At convergence, $R_1 = 0.0306$, $wR_2 = 0.0695$ [$I > 2.0\sigma(I)$] and $R_1 = 0.0344$, $wR_2 = 0.0712$ (all data), for 251 parameters.

5. Supplementary material

CCDC 617239 contains the supplementary crystallographic data for $[Ru_8(\mu_5\text{-}S)_2(\mu_4\text{-}S)(\mu_3\text{-}S)(\mu\text{-}CNMe_2)_2(\mu\text{-}CO)(CO)_{15}]$. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Acknowledgement

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