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Structural characterization of [$Ru_3Mo(\mu_3-\eta^1-CC\}(\mu-CO)_3(CO)_2(\eta-C_5H_5)_5$]: a revisitation

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Abstract

A re-examination, crystallographically, of the structure of [$MoRu_3C_2(CO)_5(\eta-C_5H_5)_4$] by low temperature single crystal X-ray studies of two forms, one the previously recorded tetragonal $P4/m$ dichloromethane solvate, and the other an un-solvated monoclinic $P2_1/c$ phase, is recorded, being undertaken in an attempt to establish with greater certainty by such means the disposition of the molybdenum atom among the [M_3(triangle)+M(pendant)] metal atom complement. The revised interpretation converges on [$\mu_3-\eta^1-\{(\eta-C_5H_5)(CO)_2Mo\equiv C-C\}Ru_3(CO)_5(\eta-C_5H_5)_3$] which ensures that the cluster is electron precise.

Keywords: Ruthenium; Cluster; Molybdenum

1. Introduction

In a recent report [1] we recorded the synthesis of [$Ru_3Mo(\mu_3-\eta^1-CC\}(\mu-CO)_3(CO)_2(\eta-C_5H_5)_4$] (**1**) supported by spectroscopic and single crystal X-ray studies, a key feature of interest being the distribution of the two types of metal atom within a skeleton readily defined as comprising a triangular metal cluster coupled to the fourth metal atom by the carbide group: [$\{(\eta-C_5H_5)_3(CO)_3M_3\}CC\{M(CO)_2((\eta-C_5H_5)\}$]. On the basis of spectroscopic evidence, the molybdenum atom was considered to constitute a component of the M_3 triangle, with Ru pendant; because of various difficulties, the crystallographic study was ambiguous in this aspect, supportive only of the gross connectivity/stereochemistry with any certainty. The crystallographic study recorded was carried out at room temperature with a single counter/sequential 'four-circle' instrument; as the original material had been capillary mounted and well-preserved thus, it was considered of interest to re-examine the problem with the advent of a CCD area-detector facility equipped with 'low'-temperature attachment in an attempt to obtain a more definitive result by such means.

One source of original difficulty was the inherent one of distinguishing between Mo ($Z = 42$) and Ru ($Z = 44$) meaningfully by this method in a situation where different sites might be occupied equally well by both elements. This was compounded by virtue of the asymmetric unit comprising a pair of half-molecules, the two molecules concerned being pseudo-symmetrically related, each disposed astride a mirror plane (in tetragonal space group $P4/m$), and disordered about it in that model, attempted refinement in lower symmetry being inherently unfruitful; disordered solvent (CH_2Cl_2) was also present. Crystals of this material were of adequate size; this study had been preceded by an attempted determination on a different un-solvated polymorph, obtained from hexane solution, which, for reasons of inadequate specimen size had been even less fruitful. This material had been similarly preserved. Specimens of both forms were thus re-examined using the 'low'-temperature CCD facility

2. Results and discussion

Despite the use of extensive low-temperature CCD diffractometer data, the redetermination of the tetra-

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gonal phase offers no improvement or increase in usefulness in the model. (It is our experience that not infrequently with such systems improved data at lower temperature simply results in the resolution of further components of disorder (etc.), obscuring further rather than assisting the understanding.) The converse applies with the monoclinic phase where, with an essentially uncomplicated structure, a useful model is now apprehensible, which we now discuss.

In the monoclinic phase, one formula unit comprising one discrete molecule, devoid of crystallographic symmetry and of solvent, comprises the asymmetric unit of the structure (Fig. 1(a)). Despite the straightforward nature of the structure, the crystal packing (Fig. 1(b)) hints at quasi-m symmetry about $y = 0.25$, 0.75, more closely realized in the tetragonal phase, the length of the a and b axes of the present $P2_1/c$ cell similar in length to that of tetragonal c of the $P4/m$ cell. Refinement behaviour of the $P2_1/c$ data is strongly indicative of near, if not total occupancy of the pendant site by molybdenum, although it is notable that minor disordered components in the tetragonal phase may be indicative of co-crystallization of some similar but different array, unlikely to be simply a permutation of the metal. The geometry of the indicated model is given in Table 1.

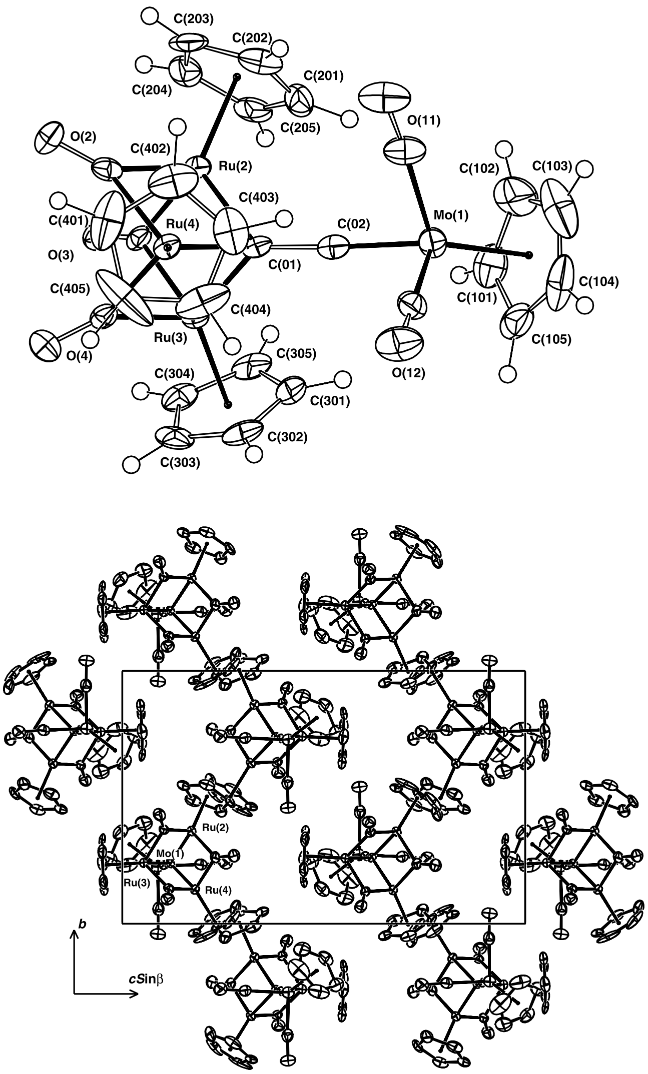

Fig. 1. (a) A single molecule of $[Ru_3Mo(\mu_3\text{-}\eta^1\text{-}CC\}(\mu\text{-}CO)_3(CO)_2(\eta\text{-}C_5H_5)_4]$ ($P2_1/c$ form). (b) Unit cell contents projected down a.

On geometrical rather than refinement considerations, support for the assignment of the pendant is more difficult-relevant determinations are sparse. Thus there are few structurally characterised $(\eta\text{-}C_5H_5)Mo(CO)_2$ residues available at present; particularly pertinent are the structural characterisation of $[(\eta\text{-}C_5H_3{}^tBu_2)_2Mo_2(CO)_4]$(**2**) [2] and $[(\eta\text{-}C_5H_5)(CO)_2Mo{\equiv}C{-}OB(NMe_2)B(NMe_2)Mo(CO)_3(\eta\text{-}C_5H_5)]$(**3**) [3]. This latter complex has a connectivity that has many similarities, at least in respect of the Mo–C bond, comprising a formal Mo–C triple bond. The geometries about the Mo atoms in **1** and **3** are almost identical with Mo≡C distances 1.830(9) and 1.825(4) Å, respectively, and Mo≡C–X angles essentially linear (177.6(6)° (**1**) and 174.9(3)° (**3**)). The most telling arbiter of connectivity is the subsequent CC bond in **1** which at 1.40(1) Å is clearly single bond in character and similar to the analogous bond (1.428(5) Å) in a tri-cobalt bis-methylidyne complex, $[(CO)_9Co_3C{-}CCo_3(CO)_9]$ [4]. Thus the pendant in **1** can be now formulated as $\{(\eta\text{-}C_5H_5)(CO)_2Mo{\equiv}C{-}C\}$ with the triply bridging alkylidyne carbon bound to the Ru_3 triangle, an example of the relatively rare tautomeric form of the carbide ligand best represented by $M{\equiv}C{-}C{\equiv}M_3$ as exemplified by the ditungsten complex of Schrock and co-workers, [5] $[({}^tBu_3O)_3W{\equiv}C{-}C{\equiv}W(O^tBu_3)_3]$. In the $[(\eta\text{-}C_5H_5)_3(CO)_3Ru_3]$ component, there are no systematic irregularities suggestive of any localised feature indicative of an ordered incorporation of molybdenum.

The $[(\eta\text{-}C_5H_5)_3(CO)_3Ru_3C_2]$ residue in its present form has not been previously structurally characterized; the mean geometry of the present system is compared with that of the most closely related species, $[(\eta\text{-}C_5H_5)_3(CO)_3Ru_3CPh]$ [6], in Table 2, the differences being insignificant, those of the latter compound being the more dispersed. Perhaps the most persuasive comparator is the recently recorded geometry for the $(\eta\text{-}C_5H_5)Ru(CO)_2C$ residue in the recently reported $[\{(\eta\text{-}C_5H_5)Ru(CO)_2\}_2(\mu\text{-}C{\equiv}C)]$ [7] in a local more precise determination [8] for which $(\eta\text{-}C_5H_5){-}Ru$ range between 2.237(8) and 2.253(9) Å (⟨ ⟩ (ten values) 2.244(6) Å), Ru–C(CO) 1.852(8)–1.871(7) (⟨ ⟩ (four values) 1.862(8) Å) and Ru–C 2.035(7), 2.050(7) Å; it is of interest to note that in the latter all Ru–X distances are considerably shorter than the present excepting Ru–C≡ which is appreciably longer, perhaps a consequence of considerable carbenoid character in the present putative Mo–C≡ bond.

Table 1
Selected geometries for the monoclinic form of **1** ‘C(*n*00)’ are the cyclopentadienyl centroids

	The Ru_3 array, $n = 2, 3, 4$ (cyclic)			The Mo geometry	
Bond lengths (Å)					
C(01)–Ru(*n*)	2.052(8)	2.018(8)	2.021(8)	Mo(1)–C(02)	1.830(9)
Ru(*n*)–Ru(*n*+1)	2.707(1)	2.701(1)	2.700(1)	Mo(1)–C(11)	1.989(10)
Ru(*n*)–C(*n*)	2.019(8)	2.045(9)	2.032(9)	Mo(1)–C(12)	1.983(10)
Ru(*n*)–C(*n*+1)	2.022(9)	2.037(9)	2.041(9)	C(01)–C(02)	1.40(1)
Ru(*n*)–C(*n*00)	1.901	1.885	1.899	Mo(1)–C(100)	2.064
Ru(*n*)–C(*n*0*m*)	2.222(9) −2.257(9)	2.201(9) −2.248(9)	2.216(13) −2.250(11)	Mo(1)–C(100)	2.343(11) −2.402(14)
⟨Ru(*n*)–C(*n*0*m*)⟩	2.24(1)	2.23(2)	2.23(2)	⟨Mo(1)–C(10*n*)⟩	2.38(3)
Bond angles (°)					
C(01)–Ru(*n*)–C(*n*00)	1259	1274	1267	C(100)–Mo(1)–C(02)	1332
C(01)–Ru(*n*)–C(*n*)	96.6(3)	96.8(3)	96.5(3)	C(100)–Mo(1)–C(11)	1246
C(01)–Ru(*n*)–C(*n*+1)	96.4(3)	96.4(3)	96.9(3)	C(100)–Mo(1)–C(12)	1217
C(*n*00)–Ru(*n*)–C(*n*)	1222	1195	1234	Mo(1)–C(02)–C(01)	177.6(6)
C(*n*00)–Ru(*n*)–C(*n*+1)	1211	1222	1209	C(02)–Mo(1)–C(11)	84.5(4)
C(*n*)–Ru(*n*)–C(*n*+1)	84.7(3)	84.3(3)	81.1(3)	C(02)–Mo(1)–C(12)	88.8(4)
Ru(*n*)–C(*n*)–O(*n*)	139.8(7)	137.4(7)	139.0(7)	C(11)–Mo(1)–C(12)	91.6(4)
Ru(*n*1)–C(*n*)–O(*n*)	136.6(7)	138.6(7)	137.7(7)		
Ru(*n*)–C(*n*)–Ru(*n*+1)	83.4(3)	83.4(3)	83.2(3)		
Ru(*n*)–C(01)–Ru(*n*+1)	83.3(3)	83.9(3)	83.0(3)		
C(02)–C(01)–Ru(*n*)	128.1(6)	131.3(6)	129.9(6)		

Table 2
Comparative mean geometries, $[(\eta-C_5H_5)_3(CO)_3Ru_3CX]$

	X = –C≡Mo(CO)$_2$-(η–C$_5$H$_5$) (this work)	X = Ph$_6$
Bond lengths (Å)		
Ru–Ru	2.703(4)	2.697(8)
Ru–(cp centroid)	1.895(9)	1.901(2)
Ru–(μ$_3$-C)	2.03(2)	2.021(2)
Ru–C(carbonyl)	2.03(1)	2.02(1)
Bond angles (°)		
≡C–(μ$_3$-C)–Ru	130(2)	130(5)
(μ$_3$-C)–Ru–C(carbonyl)	96.6(2)	96.3(4)
(μ$_3$-C)–Ru–(cp centroid)	126.7(8)	129(2)
(cp cent.)–Ru–C(carb.)	122(1)	121(1)
Ru–C(carb.)–Ru	83.3(1)	83.6(3)
Ru–C(carb.)–O(carb.)	138(1)	138(1)
C(carb.)–Ru–C(carb.)	83(2)	83(2)

2.1. Further synthetic efforts

The reaction of the complex $[\{(\eta-C_5H_5)Ru(CO)_2\}_2(\mu-C\equiv C)]$ and $[Mo_2(CO)_4(\eta-C_5H_5)_2]$ has been the subject of the previous communication [1]. Although the general connectivity of complex **1** was seemingly established by the X-ray structural determination, it was envisaged that a functionalisation of the respective starting materials would allow the unequivocal assignment of the position of the molybdenum atom within the cluster framework through the introduction of a degree of asymmetry to the cluster but this avenue was thwarted by the inability to obtain unproblematic crystals for the X-ray work. Attempts to prepare the tungsten analogue of complex **1** were unsuccessful, with the ethyne-1,2-diyl complex $[\{(\eta-C_5H_5)Ru(CO)_2\}_2(\mu-C\equiv C)]$ seemingly unreactive towards $[\{W(CO)_2(\eta-C_5H_5)\}_2]$ over extended periods.

However, utilising the substituted ethyne-1,2-diyl complex $[\{(\eta-C_5H_4Me)Ru(CO)_2\}_2(\mu-C\equiv C)]$ the reaction with $[Mo_2(CO)_4(\eta-C_5H_5)_2]$ proceeded in an analogous manner giving $[Ru_3Mo(\mu_3-\eta^1-CC\}(\mu-CO)_3(CO)_2(\eta-C_5H_4Me)_3(\eta-C_5H_5)]$ (**1′**). This major red–black fraction isolated from this reaction provided infrared spectra containing four *ν*(CO) absorptions at 1957m, 1891m, 1816s and 1771m; the latter band indicating the presence of one or more semi-bridging carbonyl ligands. The FAB mass spectra of **1′** consisted of a base peak at *m*/*z* 868 corresponding to $[M+2H]^+$. We were unable to grow suitable crystals of **1′** to undertake a single crystal experiment. The ^{1}H-NMR spectrum of **1′** contained resonances characteristic of Mo(η-C$_5$*H*$_5$) moieties at *δ* 5.77 accompanied, in a 1:3 ratio, by additional resonances assigned to three Ru(η-C$_5$H$_4$Me) moieties displaying virtual triplets of an AA′:BB′ spin system centred at *δ* 4.98 and 4.92. A singlet resonance at *δ* 1.90 was attributed to the methyl protons of the η-C$_5$H$_4$Me rings. The three Ru(η-C$_5$H$_4$Me) groups are magnetically equivalent as evident from the ^{13}C-NMR spectrum with singlet resonances observed at *δ* 107.7, 94.5 and 93.1. The methyl carbons of the η-C$_5$H$_4$Me ligands of are attributed to a singlet resonance at *δ* 12.7 with a resonance at *δ* 90.6 assigned to Mo(η-*C*$_5$H$_5$). The NMR data discussed so far is fully consistent with the new formulation of complex **1** and **1′**. The remaining resonances observed in the ^{13}C-NMR spectra of **1** and **1′** can be assigned to the carbide and carbonyl ligands. The assignment of these should now be possible given the new formulation. Molybdenum η^1-alkylidyne complexes are well known [9] and are easily accessed by a number of methodologies [10,11]. The defining spectroscopic features of the new formulation should be the downfield ^{13}C resonances of the μ_3- and η^1- alkylidyne carbons. Related tri-ruthenium μ_3-alkylidyne clusters and mononuclear alkylidyne[12] complexes display ^{13}C-NMR resonances between δ 243–321 and δ 244–333, respectively [9]. The two singlet resonances at 240.6 and 233.0 ppm for **1′** (similar values were found for **1**) appear to indicate only two types of carbonyl ligand to be present for the solution state species. Singlet resonances at δ 87.2 and 83.7 for **1** and **1′**, respectively, were identified as quaternary carbons and are noted as comparable to the acetylenic carbon resonance of the starting ethyne-1,2-diyls but in this case were not assigned.

The substantially downfield ($\sim$308 ppm) singlet resonances observed for **1** and **1′** could now be assigned to either of these μ_3- or η^1-carbons, with the remaining carbon centre attributed to one of the two moderately downfield resonances (232.4/234.2 ppm, **1**; 233.0/240.6 ppm, **1′**) initially assigned to carbonyl ligands. This requires assignment of a single resonance to both terminal and bridging carbonyls of the clusters and also gives no plausible assignment for the singlet resonance at ca. 85 ppm for each complex. The structure, as currently formulated, does allow for an adequate explanation of the observed variable temperature NMR experiments as the three Ru(η-C_5H_4R) environments are essentially identical if the solid state structure persists in solution.

3. Experimental

3.1. General procedures

Manipulations of oxygen and moisture sensitive compounds were performed under an atmosphere of high purity argon using standard Schlenk techniques or in a dry box (Miller Howe).

Infrared spectra were recorded using a Bio-Rad FTS 45 or 40 FTIR spectrometer. ^{1}H- and ^{13}C-NMR spectra were acquired using Varian Gemini 200 or Bruker ARX 500 spectrometers. ^{31}P-NMR spectra were acquired using a Bruker ARX 500 spectrometer. ^{1}H- and ^{13}C-NMR spectra were referenced with respect to incompletely deuterated solvent signals.

Mass spectra were obtained on a VG AutoSpec spectrometer employing a fast atom bombardment (FAB) ionisation source in all samples unless otherwise specified.

Elemental analyses were performed by The Research School of Chemistry Microanalytical Unit, Australian National University, ACT.

n-Hexane and toluene were dried over sodium metal and distilled from sodium benzophenone ketyl under an atmosphere of argon. Distilled solvents were stored over sodium or potassium mirrors until use.

3.2. *Synthesis of* $[Ru_3Mo(\mu_3\text{-}\eta^1\text{-}CC\}(\mu\text{-}CO)_3(CO)_2(\eta\text{-}C_5H_5)(\eta\text{-}C_5H_4Me)_3]$ (**1′**)

A solution of $[\{Mo(CO)_2(\eta\text{-}C_5H_5)\}_2]$ (70 mg, 0.182 mmol) and $[\{Ru(CO)_2(\eta\text{-}C_5H_4Me)\}_2(\mu_2\text{-}C{\equiv}C)]$ (100 mg, 0.162 mmol) in toluene (50 ml) was stirred at reflux (25 h). After cooling the solvent was removed in vacuo, and the residue redissolved in CH_2Cl_2 (min.) and chromatographed on silica (2.5 × 6 cm; 60% CH_2Cl_2/40% *n*-hexanes eluant) to resolve four bands. The first orange band eluted was identified (IR and ^{1}H/^{13}C-NMR) as an unresolved mixture of unreacted $[\{Mo(CO)_2(\eta\text{-}C_5H_5)\}_2]$ and $[\{Ru(CO)_2(\eta\text{-}C_5H_4Me)\}_2]$ (10 mg total). The second yellow/brown band remains unidentified and the third red/brown band gave **1′** (88 mg, 67%). IR (toluene) ν(CO) 1957 m, 1891 m, 1816 s, 1774 m; ^{1}H-NMR (CD_2Cl_2): δ 5.77 (s, 5 H, C_5H_5), 4.99 (s, 1H, C_5H_4Me), 4.98 (s, 3H, C_5H_4Me), 4.97 (s, 2H, C_5H_4Me), 4.93 (s, 2H, C_5H_4Me), 4.92 (s, 3H, C_5H_4Me), 4.91 (s, 1H, C_5H_4Me), 1.90 (s, 9H, C_5H_4Me); ^{13}C-NMR (toluene-d_8) δ 308.6 (s, μ_3-C*C*), 240.6 (s, *C*O), 233.0 (s, *C*O), 107.7 (s, C_i), 94.5 (s, C_5H_4Me), 93.1 (s, C_5H_4Me), 90.6 (s, C_5H_5), 83.7 (s, ?), 12.7 (s, C_5H_4*Me*); MS (FAB) *m/z* 868 ($[M+H]^+$, 100%); Anal. Calc. for $C_{30}H_{26}Mo_1O_5Ru_3$: C, 41.62; H, 3.03. Found: C, 41.92; H, 2.91.

3.3. Structure determinations

Full spheres of 'low'-temperature CCD area-detector diffractometer data were measured (Bruker AXS instrument, ω-scans, T ca. 153 K; monochromatic Mo–K_α radiation, $\lambda = 0.7107_3$ Å) yielding $N_{(\mathrm{total})}$ reflections, these merging to N unique (R_{int} quoted) after 'empirical'/multiscan absorption correction (proprietary software). N_o with $F > 4\sigma(F)$ were considered 'observed' and used in the full-matrix least-squares refinements, refining anisotropic thermal parameter forms for the non-hydrogen atoms, $(x, y, z, U_{\mathrm{iso}})_H$ being constrained at estimated values. Conventional residuals R, R_w on $|F|$ are cited at convergence (reflection weights: $(\sigma^2(F)+0.0004F^2)^{-1}$). Neutral atom complex scattering factors were employed within the XTAL 3.7 program system [13]. Pertinent results are given below and in Tables 1 and 2 and Fig. 1, the latter showing 50% probability amplitude displacement envelopes for the non-hydrogen atoms, hydrogen atoms having arbitrary radii of 0.1 Å.

3.4. Crystal/refinement data: [$Ru_3Mo(\mu_3$-η^1-CC}(μ-CO$)_3$(CO$)_2(\eta$-$C_5H_5)_4$] $C_{27}H_{20}MoO_5Ru_3$

3.4.1. Unsolvated form

$M = 823.6$. Monoclinic, space group $P2_1/c$ (C^5_{2h}, no. 14), $a = 11.596(2)$, $b = 11.567(3)$, $c = 18.590(4)$ Å, $\beta = 93.250(4)°$, $V = 2509$ Å^3. D_{calc} ($Z = 4$) $= 2.180$ g cm^{-3}. $\mu_{Mo} = 2.3$ mm^{-1}; specimen: $0.19 \times 0.13 \times 0.04$ mm^3; $T_{min/max} = 0.80$. $2\theta_{max} = 50°$; $N_t = 24632$, $N = 5136$ ($R_{int} = 0.062$), $N_o = 3240$; $R = 0.046$, $R_w = 0.046$.

The principal test for the molybdenum location here was by refinement of the site occupancy of the pendant metal against the triangle, modelled as Ru_3, refinement of M(4) as Ru giving a site occupancy of 0.89, as Mo 0.95. The final model therefore assigns M(4) as the molybdenum site for this phase.

3.4.2. 0.25 CH_2Cl_2 solvate

$M = 844.8$. Tetragonal, space group $P4/m$ (C^1_{4h}, no. 83), $a = 20.787(2)$, $c = 12.501(1)$ Å, $V = 5403$ Å^3. D_{calc} ($Z = 8$) $= 2.077$ g cm^{-3}. $\mu_{Mo} = 2.2$ mm^{-1}; specimen: $0.36 \times 0.26 \times 0.18$ mm^3; $T_{min/max} = 0.85$. $2\theta_{max} = 65°$; $N_t = 112467$, $N = 9940$ ($R_{int} = 0.080$), $N_o = 7374$; $R = 0.060$, $R_w = 0.010$.

As noted previously, disorder and/or space group assignment problems are prevalent and not ameliorated by virtue of more extensive data measured at low temperature. Anisotropic displacement parameter forms were refined for Ru, Mo only. Cyclopentadienyl carbons C(11,21) associated with Mo(11,21) are modelled as disordered about the crystallographic mirror plane about which the molecule lies, disorder also being resolvable in Mo(21), i.e. lying off the plane. Beyond this, minor disordered metal atom components were resolved and refined for both molecules, site occupancies 0.9824(9), 0.9380(9) and complements. As before, solvent residues were refined in terms of disordered dichloromethane. As before, attempted refinement in lower symmetry was unfruitful.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 200702 and 200703. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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