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Complexes derived from the reactions of diphenylphosphinoacetic acid. Part 3[1]. Dimetal complexes from $[Mo_2Cl_8]^{4-}$ and $[Re_2Cl_8]^{2-}$

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Abstract

Diphenylphosphinoacetic acid (**1**) reacts with $K_4Mo_2Cl_8$ in refluxing methanol to afford the centrosymmetric dimolybdenum(II) complex $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$ (**2**) that contains two trans P,O bridging phosphine–ester ligands, an eclipsed rotational geometry, and a Mo–Mo quadruple bond distance of 2.1450(8) Å. The corresponding reactions of **1** with $(n\text{-}Bu_4N)_2Re_2Cl_8$ in refluxing methanol and ethanol yield the paramagnetic, dirhenium(III,II) complexes $Re_2Cl_5(Ph_2PCH_2CO_2R)_3$ (R = Me (**3**) or Et (**4**)) in which the phosphine–ester ligands are P-bound. A crystal structure determination of **3** shows it to have the 1,3,6-structure with a Re–Re bond length of 2.2318(3)Å in accord with a Re–Re bond order of 3.5.

Keywords: Crystal structures; Diphenylphosphinoacetic acid; Phosphine-ester ligands; Dimolybdenum(II) complexes; Dirhenium(III,II) complexes; Metal–metal multiple bonds

1. Introduction

One focus of our interest in incorporating multiply bonded dirhenium units into homometallic assemblies has been the use of bridging dicarboxylate ligands to generate 'dimers-of-dimers', supramolecular triangles, oligomers, and hydrogen-bonded polymeric chains in which paramagnetic $[Re_2]^{5+}$ and/or diamagnetic $[Re_2]^{4+}$ cores are present [1–3]. More recently, the strategy of using monocarboxylic acid ligands that also contain other accessible donor sites, such as 4-pyridinecarboxylic acid (isonicotinic acid), to obtain heterometallic assemblies has been successfully used to form molecular squares, in which there are alternating multiply bonded dirhenium $[Re(II)]_2$ and mononuclear Pt(II) units at the corners [4].

In the present report, we describe reactions utilizing diphenylphosphinoacetic acid (**1**) in which we attempted to form multiply bonded dimetal complexes that contain the diphenylphosphinoacetate ligand, which has previously been used quite extensively in mononuclear transition metal chemistry [5–17]. Our original intent had been to use the resulting products, which we expected would contain free P donors or carboxylic acid groups, to complex other metal centers and thereby generate new types of mixed-metal assemblies in which multiply bonded dimetal units would be present. This strategy has been used previously to access heterometallic complexes in which the $(\eta^5\text{-}C_5H_5)_2Ti(IV)$ unit is linked to Pd(II), Pt(II), Cu(I), Ag(I) and Au(I) centers [17]. Instead, our work led to some unanticipated results, details of which are now described.

2. Experimental

2.1. Starting materials and reaction procedures

The starting materials $K_4Mo_2Cl_8$ [18] and $(n\text{-}Bu_4N)_2Re_2Cl_8$ [19] were prepared by the standard literature methods. Diphenylphosphinoacetic acid (**1**) was prepared by a procedure different from that which has typically been used [5]. A 2.5 M hexane solution of n-BuLi (22 ml, 55 mmol) was added dropwise to a

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[1] Part 1 is ref [16]; Part 2 is ref [17].

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solution of Ph_2PH (9.30 g, 50 mmol) in THF (50 ml) at 0 °C. This mixture was stirred at 25 °C for 1 h and then added dropwise with stirring to a solution of $ClCH_2CO_2Na$ (5.60 g, 50 mmol) in THF at room temperature (r.t.). The solvent was evaporated and the residue dissolved in 100 ml of water, the extract filtered, and the filtrate washed with diethyl ether (2 × 10 ml) and the washings discarded. The careful addition of 2M HCl to this aqueous solution afforded a white crystalline solid that was washed with methanol and hexane. Yield, 11.1 g (91%). The ^1H and $^{31}P\{^1H\}$ NMR spectral data of this product were identical to the literature data [5,16]. All other reagents and solvents were obtained from commercial sources. Solvents were dried and degassed by standard methods and distilled prior to use. Reactions were carried out under an atmosphere of dry dinitrogen.

2.2. Synthesis of $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$ (**2**)

A mixture of $K_4Mo_2Cl_8$ (63 mg, 0.10 mmol) and **1** (110 mg, 0.45 mmol) in 40 ml of methanol was refluxed for 2 days. The mixture of blue crystals and red powder was filtered off and the solid products washed with dimethylformamide to dissolve the red powder and leave the blue crystals of **2**; yield 15 mg (18%). *Anal.* Found: C, 41.83; H, 3.56. Calc for $C_{30}H_{30}Cl_4Mo_2O_4P_2$: C, 42.38; H, 3.56%. This product, which was identified by X-ray crystallography, was insoluble in common polar and non-polar solvents. The red colored dimethylformamide extract was treated with an excess of diethyl ether to precipitate a red solid; yield 48 mg. *Anal.* Found: C, 37.70; H, 3.46%. This solid was soluble only in DMF and DMSO; $^{31}P\{^1H\}$ NMR spectrum (in $(CD_3)_2SO$) $\delta = -11.3$(s, br). The identity of this product was not established, but it does not appear to be closely related to **2** based upon a comparison of their IR spectra (KBr pellets).

2.3. Synthesis of $Re_2Cl_5(Ph_2PCH_2CO_2Me)_3$ (**3**)

A mixture of $(n\text{-}Bu_4N)_2Re_2Cl_8$ (114 mg, 0.10 mmol) and **1** (100 mg, 0.40 mmol) was refluxed in 70 ml of methanol for 24 h, then allowed to cool to r.t., and the green crystals of **3** filtered off and washed with methanol (2 × 5 ml) and diethyl ether (2 × 5 ml); yield 99 mg (75%). *Anal.* Found: C, 40.62; H, 3.31. Calc for $C_{45}H_{45}Cl_5O_6P_3Re_2$: C, 40.81; H, 3.42%.

2.4. Synthesis of $Re_2Cl_5(Ph_2PCH_2CO_2Et)_3$ (**4**)

The reaction of $(n\text{-}Bu_4N)_2Re_2Cl_8$ (118 mg, 0.10 mmol) and **1** (110 mg, 0.45 mmol) in 40 ml of refluxing ethanol for periods of up to 2 days gave **4** as green flakey crystals; yield 91 mg (71%). *Anal.* Found: C, 41.91; H, 3.68; Cl, 12.69. Calc. for $C_{48}H_{51}Cl_5O_6P_3Re_2$: C, 42.19; H, 3.76; Cl, 12.97%.

2.5. X-ray crystallography

Single crystals with the compositions $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$ (**2**) and $Re_2Cl_5(Ph_2PCH_2CO_2Me)_3$ (**3**), were obtained as described by use of the individual synthetic procedures outlined in Sections 2.2 and 2.3.

The data were collected at 173(±1) K for **2** and 170(±1)K for **3**. All measurements were carried out on a Nonius Kappa CCD diffractometer with graphite-monochromated Mo K_α radiation ($\lambda = 0.71073$ Å). Lorentz and polarization corrections were applied to the data sets. The important crystallographic data are given in Table 1.

The structure of **2** was solved using the structure solution program PATTY in DIRDIF92 [20] while DIRDIF99 [21] was used for **3**. The remaining atoms were located in succeeding difference Fourier syntheses. Hydrogen atoms were placed in calculated positions according to idealized geometries with $U(H) = 1.3\,U_{eq}(C)$. They were included in the refinement but constrained to ride on the atom to which they are bonded. An empirical absorption correction using SCALEPACK [22] was applied in both cases. The final refinements were performed by the use of the program SHELXL-97 [23]. All non- hydrogen atoms were refined with anisotropic thermal parameters unless indicated otherwise. Crystallographic drawings were done using the program ORTEP [24].

The structure solutions and refinements proceeded without major problem. During the latter stages of the refinement of the dirhenium complex **3**, residual electron

Table 1
Crystallographic data for $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$ (**2**) and $Re_2Cl_5(Ph_2PCH_2CO_2Me)_3$ (**3**)

	2	**3**
Empirical formula	$C_{30}H_{30}Cl_4Mo_2O_4P_2$	$C_{45}H_{45}Cl_5O_6P_3Re_2$
Formula weight	850.21	1324.37
Space group (No. 14)	$P2_1/n$	$P2_1/n$
a (Å)	10.3764(5)	11.27010(10)
b (Å)	8.6362(7)	22.0133(3)
c (Å)	18.4587(14)	20.5363(3)
α (°)	90	90
β (°)	95.818(4)	97.6124(6)
γ (°)	90	90
V (Å^3)	1645.6(3)	5049.99(11)
Z	2	4
ρ_{calcd} (g cm^{-3})	1.716	1.742
μ (Mo Kα) (mm^{-1})	1.201	5.193
$R(F_o)$[a]	0.037	0.043
$R_w(F_o^2)$[b]	0.075	0.089
Goodness-of-fit	1.012	1.152

[a] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ with $F_o^2 > 2\sigma(F_o^2)$.

[b] $R_w = [\Sigma w(|F_o^2| - |F_c^2|)^2/\Sigma w|F_o^2|^2]^{1/2}$.

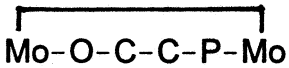
density ($\sim$5–6 e Å^{-3}) was found close to the multiply bonded dirhenium unit and orthogonal to it. The distance between these peaks was approximately 2.20 Å and they are almost certainly associated with the type of disorder commonly encountered in dimetal complexes that contain a metal-metal multiple bond and an eclipsed M_2L_8 geometry [25], in which two approximately orthogonal dimetal sets share the same set of ligand atoms. Among compounds that show this disorder are several of the type $Re_2Cl_5(PR_3)_3$ [26]. In the present case, inclusion of this very minor form in the refinement gave multiplicities for the major and minor forms of 0.968 and 0.032, respectively, and led to a reduction in the final R indices. The largest peak in the final difference Fouriers of **2** and **3** had a height of 0.51 and 1.97 e Å^{-3}, respectively.

2.6. Physical measurements

Infrared spectra were recorded as KBr pellets on a Perkin–Elmer 2000 FT-IR spectrometer, while ^{1}H and ^{31}P{^{1}H} NMR spectra were obtained on a Varian INOVA 300 spectrometer. Proton resonances were referenced internally to the residual protons in the incompletely deuteriated solvent. The ^{31}P{^{1}H} spectra were recorded at 121.6 MHz, with 85% H_3PO_4 as an external standard. The X-band ESR spectrum of compound **3** was recorded at 150 K on a Bruker ESP 300 instrument. Electrochemical measurements were carried out with the use of a BAS Inc. Model CV-27 instrument in conjunction with a BAS Model RXY recorder and were recorded on dichloromethane solutions that contained 0.1 M tetra-*n*-butylammonium hexafluorophosphate (TBAH) as supporting electrolyte. $E_{1/2}$ values, determined as $(E_{p,a}+E_{p,c})/2$, were referenced to the silver–silver chloride (Ag–AgCl) electrode at 25 °C and were uncorrected for junction potentials. Under our experimental conditions $E_{1/2} = +0.47$ V versus Ag–AgCl for the ferrocenium–ferrocene couple. Elemental microanalyses were performed by Dr. H. D. Lee of the Purdue University Microanalytical Laboratory.

3. Results and discussion

The dimolybdenum(II) complex $K_4Mo_2Cl_8$ failed to react with diphenylphosphinoacetic acid ($Ph_2PCH_2CO_2H$, **1**) at room temperature in a variety of polar and non-polar solvents, and in refluxing acetone, dichloromethane or THF. However, in refluxing methanol a slow reaction ensues to afford a mixture of red powder and blue crystals. The blue crystals were insoluble in polar and non-polar solvents and could easily be separated from the unidentified DMF-soluble red material (Section 2.3). A single crystal X-ray structure determination showed the blue crystals to be those of the dimolybdenum(II) complex $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$ (**2**) which is formed by the esterification of the diphenylphosphinoacetic acid in the methanol solvent and the coordination of this neutral ligand in a bridging mode through its phosphine and carbonyl groups (Fig. 1). The important bond distances and bond angles for this complex are given in the caption to Fig. 1. The molecule lies on a crystallographic inversion center and retains a rigorously eclipsed rotational geometry like the parent $[Mo_2Cl_8]^{4-}$ anion [27]. The conformational demands of the puckered



Mo-O-C-C-P-Mo

ring do not enforce a staggered or partially staggered geometry as occurs, for example, in the case of β-$Mo_2Cl_4(dppe)_2$, where dppe = $Ph_2PCH_2CH_2PPh_2$, for which the average torsional (twist) angle is approximately 30° [27]. Consequently, the Mo–Mo distance of 2.1450(8)Å in **2** is shorter than that in β-$Mo_2Cl_4(dppe)_2$ (2.183(3)Å) [28], in which the δ component of the Mo–Mo quadruple bond has been weakened, but is very similar to that in α-$Mo_2Cl_4(dppe)_2$ (2.140(2)Å) [29], in which the dppe ligands are chelating and the molecule has an eclipsed geometry. The IR spectrum of **2** (KBr pellet) shows ν(COO) modes of the methyl ester group at 1630(s) and 1341(m-s) cm^{-1}.

In the analogous reaction between the dirhenium(III) complex $(n\text{-}Bu_4N)_2Re_2Cl_8$ and **1**, the use of acetone or

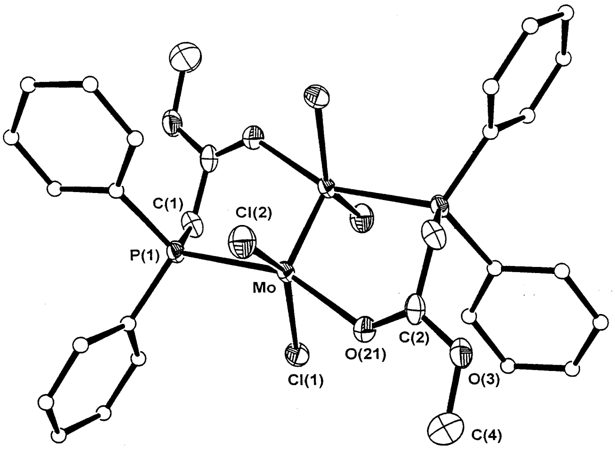


Fig. 1. ORTEP [24] representation of the structure of the dimolybdenum(II) complex $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$ (**2**). Thermal ellipsoids are drawn at the 50% probability level except for the carbon atoms of the phenyl rings which are circles of arbitrary radius. The molecule contains an inversion center at the mid-point of the Mo–Mo bond; the unlabeled Mo atom is Mo′. Selected bond distances (Å) and bond angles (°) are as follows: Mo–Mo′ 2.1450(8), Mo–O(21) 2.198(3), Mo–Cl(1) 2.3914(11), Mo–Cl(2) 2.4006(12), C(2)–O(3) 1.313(5), C(2)–O(21) 1.234(5); Mo′–Mo–O(21) 110.48(8), Mo′–Mo–Cl(1) 105.62(5), Mo′–Mo–Cl(2) 108.57(4), Mo′–Mo–P(1) 92.26(3), Mo–O(21)–C(2) 130.2(3), O(21)–C(2)–C(1) 123.1(4), C(2)–C(1)–P(1) 107.0(3), C(1)–P(1)–Mo 109.43(15).

dichloromethane as solvents failed to afford pure products. However, we obtained crystals of the pure dirhenium(III,II) complex $Re_2Cl_5(Ph_2PCH_2CO_2Me)_3$ (**3**) from hot methanol while the ethyl ester derivative $Re_2Cl_5(Ph_2PCH_2CO_2Et)_3$ (**4**) was obtained with the use of hot ethanol as the reaction solvent. Unlike, the reaction with $K_4Mo_2Cl_8$, the reactions involving $(n\text{-}Bu_4N)_2Re_2Cl_8$ lead to a one-electron reduction of the dimetal core and the resulting complexes **3** and **4** contain monodentate P-bound $PPh_2(CH_2CO_2R)$ (R = Me or Et) ligands as shown by a single crystal X-ray structure determination of **3** (Fig. 2). The important bond distances and bond angles for **3** are given in the caption to Fig. 2. This structure is of the previously known $Re_2Cl_5(PR_3)_3$ type [30], several examples of which have now been described by Cotton and co-workers [26,31]; these can exist in 1,3,6- and 1,2,7- isomeric forms. Compound **3** is the more commonly encountered 1,3,6-isomer, and its Re–Re bond length of 2.2318(3)Å is a little longer than the Re–Re distances reported for the previously characterized trialkylphosphine PR_3 (R = Me, Et or Pr^n) and PMe_2Ph derivatives that have the 1,3,6-structure (range 2.211–2.222 Å) [26,31]. This complex has the expected eclipsed rotational geometry with values for the torsional angles Cl(11)–Re(1)–Re(2)–Cl(21), P(2)–Re(1)–Re(2)–Cl(22), Cl(12)–Re(1)–Re(2)–P(3) and P(1)–Re(1)–Re(2)–Cl(23) of 0.8, −1.4, 1.1 and 3.4°, respectively.

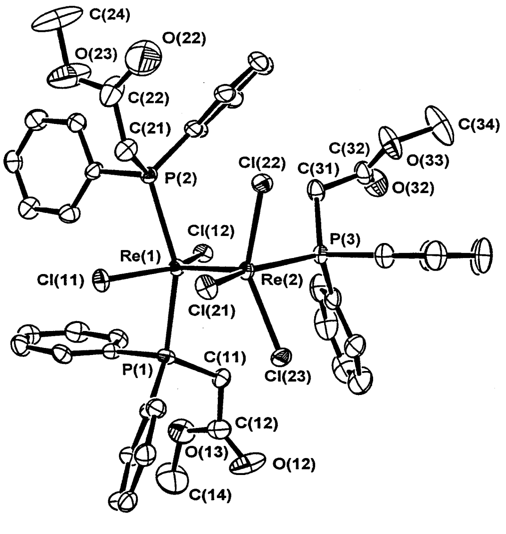


Fig. 2. ORTEP [24] representation of the structure of the dirhenium(III,II) complex $Re_2Cl_5(Ph_2PCH_2CO_2Me)_3$ (**3**). Thermal ellipsoids are drawn at the 50% probability level. Selected bond distances (Å) and bond angles (°) are as follows: Re(1)–Re(2) 2.2318(3), Re(1)–Cl(11) 2.3324(15), Re(1)–Cl(12) 2.3442(14), Re(1)–P(1) 2.4532(15), Re(1)–P(2) 2.4827(16), Re(2)–Cl(22) 2.3268(15), Re(2)–Cl(23) 2.3328(15), Re(2)–Cl(21) 2.3640(15), Re(2)–P(3) 2.4654(16), C(12)–O(12) 1.205(9), C(12)–O(13) 1.326(9); Cl(11)–Re(1)–Cl(12) 142.65(5), P(1)–Re(1)–P(2) 151.79(5), Cl(22)–Re(2)–Cl(23) 143.24(5), Cl(21)–Re(2)–P(3) 153.55(5).

The identity of compound **3** as a complex with a paramagnetic $[Re_2]^{5+}$ core was confirmed by the presence of a band at 1410 nm in its electronic absorption spectrum (Nujol mull); this is assigned to the $\delta-\delta^*$ transition of a dirhenium(III,II) complex [32–34]. Its ESR spectrum (powder and CH_2Cl_2-toluene glass, 150 K) shows the typical complex hyperfine splitting pattern centered close to 2.7 kG ($g = 2.5$) that is seen for other $Re_2Cl_5(PR_3)_3$ complexes [26,31b] and closely related species [32].

The cyclic voltammetric properties of **3** and **4** are very similar, each showing reversible processes that correspond to one-electron oxidation ($E_{1/2}$(ox)) and a one-electron reduction ($E_{1/2}$(red)) of the bulk complexes. These half-wave potentials are at +0.66V and −0.46 V versus Ag–AgCl for **3** and at +0.61V and −0.44V versus Ag–AgCl for **4**, values which are similar to those reported previously for other $Re_2Cl_5(PR_3)_3$ complexes [26,31,32]. The uncomplexed ester groups in **3** and **4** exhibit ν(COO) modes in their IR spectra at 1734(s) and 1266(s) cm^{-1}, and 1725(s) and 1264(s) cm^{-1}, respectively.

4. Concluding remarks

The reactions of diphenylphosphinoacetic acid with the quadruply bonded dimolybdenum(II) and dirhenium(III) complexes $K_4Mo_2Cl_8$ and $(n\text{-}Bu_4N)_2Re_2Cl_8$ in refluxing alcohol solvents afford the phosphine-ester ligands $Ph_2PCH_2CO_2R$ (R = Me or Et) which display quite different bonding modes in the resulting dimetal complexes. While $K_4Mo_2Cl_8$ affords the centrosymmetric, non-redox product $Mo_2Cl_4(\mu\text{-}Ph_2PCH_2CO_2Me)_2$, the $[Re_2Cl_8]^{2-}$ anion is reduced in boiling alcohol solvents to produce the paramagnetic complexes 1,3,6-$Re_2Cl_5(Ph_2PCH_2CO_2R)$ (R = Me or Et) in which the phosphine-ester ligand is exclusively P-bound. These are the first multiply bonded dimetal complexes that contain ligands of the type $R'_2PCH_2CO_2R$.

5. Supplementary material

Tables giving full details for the crystal data and data collection parameters, atomic positional parameters, anisotropic thermal parameters, bond distances and bond angles for compounds **2** and **3** are available on request from the author (R.A.W.).

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References

[1] J.K. Bera, P. Angaridis, F.A. Cotton, M.A. Petrukina, P.E. Fanwick, R.A. Walton, J. Am. Chem. Soc. 123 (2001) 1515.

[2] J.K. Bera, R. Clérac, P.E. Fanwick, R.A. Walton, J. Chem. Soc., Dalton Trans. (2002) 2168.

[3] (a) S.-M. Kuang, P.E. Fanwick, R.A. Walton, Inorg. Chem. Commun. 4 (2001) 745;
(b) S.-M. Kuang, P.E. Fanwick, R.A. Walton, Inorg. Chem. 41 (2002) 405.

[4] J.K. Bera, B.W. Smucker, R.A. Walton, K.R. Dunbar, Chem. Commun. (2001) 2562.

[5] T. Jarolím, J. Podlahová, J. Inorg. Nucl. Chem. 38 (1976) 125.

[6] H.D. Empsall, E.M. Hyde, D. Pawson, B.L. Shaw, J. Chem. Soc., Dalton Trans. (1977) 1292.

[7] J. Podlahová, J. Loub, J. Ječny, Acta Cryst. B 35 (1979) 328.

[8] S. Civiš, J. Podlahová, J. Loub, J. Ječny, Acta Cryst. B 36 (1980) 1395.

[9] A.C. Hazell, R.G. Hazell, B. Kratochvíl, J. Podlahová, Acta Cryst. B 37 (1981) 2068.

[10] A. Jegorov, B. Kratochvíl, V. Langer, J. Podlahová, Inorg. Chem. 23 (1984) 4288.

[11] M.M. Taqui Khan, M.K. Nazeeruddin, Inorg. Chim. Acta 147 (1988) 33.

[12] A Jegorov, J. Podlaha, J. Podlahová, F. Tureček, J. Chem. Soc., Dalton Trans. (1990) 3259.

[13] D.J. Darensbourg, J.D. Draper, J.H. Reibenspies, Inorg. Chem. 36 (1997) 3648.

[14] M. Onishi, K. Hiraki, M. Yamaguchi, J. Morishita, Inorg. Chim. Acta 195 (1992) 151.

[15] F. Refosco, F. Tisato, G. Bandoli, E. Deutsch, J. Chem. Soc. (1993) 2901.

[16] D.A. Edwards, M.F. Mahon, T.J. Paget, Polyhedron 17 (1998) 4121.

[17] D.A. Edwards, M.F. Mahon, T.J. Paget, Polyhedron 19 (2000) 757.

[18] J.V. Brencic, F.A. Cotton, Inorg. Chem. 9 (1970) 351.

[19] (a) T.J. Barder, R.A. Walton, Inorg. Chem. 21 (1982) 2510;
(b) T.J. Barder, R.A. Walton, Inorg. Synth. 23 (1985) 116.

[20] P.T. Beurskens, G. Admirall, G. Beurskens, W.P. Bosman, S. Garcia-Granda, R.O. Gould, J.M.M. Smits, C. Smykalla, The DIRDIF-92 Program System, Crystallography Laboratory, University of Nijmegen, Nijmegen, The Netherlands, 1992.

[21] P.T. Beurskens, G. Beurskens, R. deGelder, S. Garcia-Granda, R.O. Gould, R. Israel, J.M.M. Smits, The DIRDIF-99 Program System, Crystallography Laboratory, University of Nijmegan, Nijmegen, The Netherlands, 1998.

[22] Z. Otwinowski, W. Minor, Methods Enzymol. 276 (1996) 307.

[23] G.M. Sheldrick, SHELXL-97. A Program for Crystal Structure Refinement, University of Göttingen, Göttingen, Germany, 1997.

[24] C.K. Johnson, ORTEP II, Report ORNL-5138 Oak Ridge National Laboratory, 1976.

[25] F.A. Cotton, R.A. Walton, Multiple bonds between metal atoms, 2nd ed., Oxford University Press, Oxford, 1993.

[26] (a) F.A. Cotton, E.V. Dikarev, Inorg. Chem. 35 (1996) 4738;
(b) F.A. Cotton, E.V. Dikarev, M.A. Petrukhina, Inorg. Chem. 38 (1999) 3384.

[27] See Ref. [25], Table 3.1.1, pp. 143–144.

[28] P.A. Agaskar, F.A. Cotton, Inorg. Chem. 23 (1984) 3383.

[29] P.A. Agaskar, F.A. Cotton, Inorg. Chem. 25 (1986) 15.

[30] J.R. Ebner, R.A. Walton, Inorg. Chem. 14 (1975) 1987.

[31] (a) F.A. Cotton, A.C. Price, K. Vidyasagar, Inorg. Chem. 29 (1990) 5143;
(b) F.A. Cotton, E.V. Dikarev, M.A. Petrukhina, Inorg. Chem. 37 (1998) 1949.

[32] P. Brant, D.J. Salmon, R.A. Walton, J. Am. Chem. Soc. 100 (1978) 4424.

[33] J.R. Ebner, R.A. Walton, Inorg. Chim. Acta 14 (1975) L45.

[34] B.E. Bursten, F.A. Cotton, P.E. Fanwick, G.G. Stanley, R.A. Walton, J. Am. Chem. Soc. 105 (1983) 2606.