

1,3,5-Trimethyl-1,3,5-triazacyclohexane tricarbonyl complexes of Mo and W as sources of the *fac*-M(CO)₃ fragment. Mild syntheses of *fac*-[M(CO)₃(CH₃CN)₃] (M = Mo, W), [W(CO)₃(PR₃)₃], [W(CO)(alkyne)₃] and [W(CO)₃(π -arene)] complexes

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Abstract

The 1,3,5-trimethyl-1,3,5-triazacyclohexane (Me₃tach) group in [M(CO)₃(Me₃tach)] complexes (M = Mo, W) is easily displaced by a variety of ligands under mild conditions. The lability of the Me₃tach ligand allows the [M(CO)₃(Me₃tach)] complexes to be used as synthetic intermediates in convenient syntheses of products such as *fac*-[M(CO)₃(CH₃CN)₃] (M = Mo, W), [W(CO)(alkyne)₃], [W(CO)₃(PR₃)₃] and [W(CO)₃(η^6 -arene)] complexes. An improved synthesis of [W(CO)₃(Me₃tach)] from [W(CO)₆] and Me₃tach is described. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Molybdenum; Tungsten; 1,3,5-Triazacyclohexane; Alkynes; Phosphines

1. Introduction

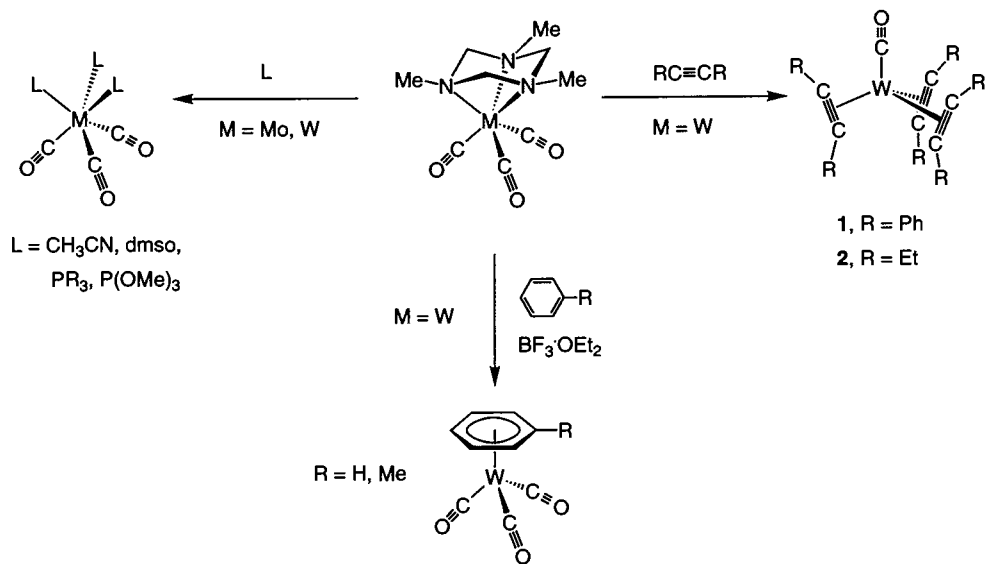
1,3,5-Triazacyclohexanes are the smallest members of the triazacycloalkane family, and there is considerable interest in the coordination chemistry of triazacyclohexanes with transition metals [1–6]. We recently reported the synthesis and some properties of a number of chromium, molybdenum, and tungsten tricarbonyl complexes of 1,3,5-trialkyl-1,3,5-triazacyclohexanes (R₃tach's) [7,8]. The small size of the triazacyclohexane system results in significant octahedral strain in facial triazacyclohexane complexes and in [M(CO)₃(Me₃tach)] (M = Cr, Mo, W) the Me₃tach ligand is labile.

[M(CO)₃(Me₃tach)] (M = Cr, Mo, W) can be prepared easily and in high yield from the commercially available Me₃tach and the appropriate metal hexacar-

bonyl [7,8]. These complexes are air sensitive, but can be stored for extended periods (at least several months) under an inert atmosphere. The Me₃tach ligand is displaced instantly at room temperature by P(OMe)₃ to produce *fac*-[M(CO)₃{P(OMe)₃}₃] [7,8]. For [Mo(CO)₃(Me₃tach)] and [W(CO)₃(Me₃tach)], even dimethyl sulfoxide (DMSO) can displace the Me₃tach ligand; dissolution of the Mo and W complexes in DMSO results in solvolysis and the formation of *fac*-[M(CO)₃(DMSO)₃] [8].

The lability of the Me₃tach group in [Mo(CO)₃(Me₃tach)] and [W(CO)₃(Me₃tach)], and the ability of these complexes to be stored for long periods without decomposition, suggested that these complexes may be useful reagents for organometallic synthesis. This paper describes some ways in which the lability of the Me₃tach ligand in [Mo(CO)₃(Me₃tach)] and [W(CO)₃(Me₃tach)] allows these complexes to be used as reactive intermediates in the preparation of other molybdenum and tungsten carbonyl complexes (Scheme 1).

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Scheme 1.

2. Experimental

2.1. General aspects

Nuclear magnetic resonance spectra were recorded using a Bruker ARX-500 spectrometer (500.1 MHz for ¹H, 125.8 MHz for ¹³C, 202.5 MHz for ³¹P) at ambient temperature (ca. 295 K) and were referenced with respect to solvent resonances or external 85% H₃PO₄. Infrared spectra were recorded from KBr disks or as dichloromethane solutions using a Bio-Rad FTS45 FTIR spectrophotometer. Microanalyses were performed by CMAS, Victoria, Australia. Manipulations involving air-sensitive compounds were performed using Schlenk techniques or in a dry box under nitrogen. Solvents were distilled and deoxygenated prior to use. Deoxygenation of solvents and reagents was carried out either by distillation under nitrogen or by at least three freeze–pump–thaw cycles. Xylene (mixture of isomers) was dried by distillation from sodium under nitrogen.

[M(CO)₃(Me₃tach)] (M = Mo or W) were prepared as reported previously [8] and, in the case of tungsten, also as outlined below. The complexes [W(CO)₃L₃] (L = PMe₂Ph, PPh₃), [W(CO)₄(PⁱPr₂)₂], [W(CO)(RC≡CR)₃] (R = Et, Ph), and [W(CO)₃(η⁶-arene)] (arene = C₆H₆, MeC₆H₅) were identified by comparison of their spectroscopic data with that reported in the literature (see below).

2.2. Improved synthesis of [W(CO)₃(Me₃tach)]

2.2.1. Method A

A mixture of Me₃tach (4.0 g, 31 mmol) and [W(CO)₆] (5.0 g, 14.2 mmol) was heated in dry xylene (150 ml) at reflux for 4 h. During this time a yellow/brown precipitate

formed. The mixture was filtered whilst hot and the solid was washed with hexanes before drying in vacuo to afford [W(CO)₃(Me₃tach)] as a tan powder (4.11 g, 73%) which was identical to material prepared previously [8].

2.2.2. Method B

[W(CO)₃(CH₃CN)₃] (0.5 g, 1.3 mmol), prepared by refluxing W(CO)₆ in acetonitrile for 2 days according to the literature procedure [9], was added to a stirred solution of Me₃tach (0.25 g, 1.9 mmol) in THF (25 ml), and the mixture was stirred overnight. The yellow precipitate that formed was collected by filtration, washed with hexane, and dried in vacuo to afford [W(CO)₃(Me₃tach)] as a tan powder (0.49 g, 96%) which was identical to material prepared previously [8].

2.3. Synthesis of [M(CO)₃(CH₃CN)₃] from [M(CO)₃(Me₃tach)] (M = Mo, W)

[Mo(CO)₃(Me₃tach)] (140 mg) was dissolved in CH₃CN (8 ml) to give a tan solution. The solution was poured into toluene (50 ml) and the resulting tan precipitate of *fac*-[Mo(CO)₃(CH₃CN)₃] was collected, washed with hexanes and dried in vacuo. Yield (117 mg, 85%); $\tilde{\nu}_{\max}/\text{cm}^{-1}$ (CO, KBr disk) 1905 (s) and 1790 (s, br) (lit. [10], 1915 and 1783 cm⁻¹). Anal. Found: C, 35.97; H, 3.16; N, 14.19. Calc. for C₉H₉MoN₃O₃: C, 35.66; H, 2.99; N, 13.86.

An analogous procedure starting with [W(CO)₃(Me₃tach)] afforded *fac*-[W(CO)₃(CH₃CN)₃]. Yield (82%); $\tilde{\nu}_{\max}/\text{cm}^{-1}$ (CO, KBr disk) 1900 (s) and 1790 (s, br) (lit. [11,12], 1910 and 1790 cm⁻¹). Anal. Found: C, 27.97; H, 2.60; N, 10.81. Calc. for C₉H₉N₃O₃W: C, 27.64; H, 2.32; N, 10.75.

2.4. Synthesis of *fac*-[W(CO)₃(PMe₂Ph)₃]

A mixture of [W(CO)₃(Me₃tach)] (85 mg, 0.21 mmol) and PMe₂Ph (0.10 g, 0.11 ml, 0.74 mmol) in tetrahydrofuran (THF) (10 ml) was stirred at room temperature for 20 min to give a yellow solution. Activated alumina was added and the solvent was removed in vacuo. The alumina was washed with pentane and then extracted with CH₂Cl₂ (4 × 10 ml). The combined extracts were filtered through celite and the yellow filtrate was reduced in volume, layered with pentane and cooled to –20°C, to produce *fac*-[W(CO)₃(PMe₂Ph)₃] as off-white micro-crystalline needles (90 mg, 62%). δ_H (acetone-*d*₆) 1.56 (18H, m, Me), 7.39–7.32 (15H, m, ArH) (lit. [13], δ_H (CD₂Cl₂) 1.53 and 7.34–7.25). δ_P (acetone-*d*₆) –24.1 (*J*_{31P–183W} = 217 Hz) (lit. [14], δ_P (CD₂Cl₂) –25.8 (*J*_{31P–183W} = 218 Hz). ν_{max}/cm^{–1} (CO, CH₂Cl₂) 1928 (s) and 1825 (s, br) (lit. [11,12], 1926 and 1823 cm^{–1}).

2.5. Synthesis of *fac*-[W(CO)₃(PPh₃)₃]

A mixture of [W(CO)₃(Me₃tach)] (200 mg, 0.50 mmol) and PPh₃ (463 mg, 1.76 mmol) in THF (15 ml) was stirred at room temperature for 1 h. The resultant white precipitate was collected, washed with THF, then hexanes, and dried in vacuo to give *fac*-[W(CO)₃(PPh₃)₃] as a white micro-crystalline powder (510 mg, 96%). δ_P (CH₂Cl₂) 21.2 (*J*_{31P–183W} = 214 Hz) (lit. [15] δ_P (CH₂Cl₂) 21.5 (*J*_{31P–183W} = 214 Hz). ν_{max}/cm^{–1} (CO, CH₂Cl₂) 1935 (s) and 1838 (s, br) (lit. [16] 1935 and 1840 cm^{–1}), ν_{max}/cm^{–1} (CO, KBr disk) 1945 (s) and 1840 (s, br) (lit. [16] β form: 1945 and 1840 cm^{–1} α form: 1930 and 1830 cm^{–1}).

2.6. Synthesis of *trans*-[W(CO)₄(P^{*i*}Pr₃)₂]

This compound was prepared in 43% yield according to the procedure used for *fac*-[W(CO)₃(PMe₂Ph)₃], except that the mixture was refluxed for 30 min. δ_H (acetone-*d*₆) 1.33 (12H, m, Me), 2.45 (2H, m, CH). δ_P (acetone-*d*₆) 41.5 (*J*_{31P–183W} = 260 Hz) (lit. [17] (CDCl₃) δ_P 40.8 (*J*_{31P–183W} = 262 Hz). ν_{max}/cm^{–1} (CO, CH₂Cl₂) 1917 (sh) and 1860 (vs, br).

2.7. Synthesis of *tris*(diphenylacetylene)tungsten(0) carbonyl

A suspension of [W(CO)₃(Me₃tach)] (150 mg, 0.38 mmol) and diphenylacetylene (215 mg, 5.7 mmol) in acetone (8 ml) was stirred and heated gently at reflux for 2 h to give a brown solution. Freshly activated alumina was added and the solvent was removed in vacuo. The dry-pack of alumina was extracted at room temperature with benzene (4 × 10 ml) to give a yellow solution. Evaporation of the solvent and recrystallisa-

tion of the residue from benzene/EtOH yielded *tris*(diphenylacetylene)tungsten(0) carbonyl as a yellow solid (229 mg, 81%). δ_H (acetone-*d*₆) 7.25–7.55 (10H, m, ArH) (lit. [18], δ_H (CDCl₃) 7.0–7.8) δ_C (acetone-*d*₆) 128.0, 128.3, 128.8, 129.4, 130.2, 130.7 (C2–C6, C2'–C6'), 139.8 and 141.3 (Cl and Cl'), 174.5 and 192.6 (2 × alkynyl C), and 220.2 (CO).

2.8. Synthesis of *tris*(3-hexyne)tungsten(0) carbonyl

Prepared in an analogous fashion to the diphenylacetylene analogue. Yield (80 mg, 77%); δ_H (acetone-*d*₆) 1.04 (9H, t, *J* = 7.5, 3 × Me), 1.28 (9H, t, *J* = 7.5, 3 × Me'), 2.98 (6H, q, *J* = 7.5, 3 × CH₂) and 3.45 (6H, q, *J* = 7.5, 3 × CH₂') [lit. [19], δ_H (acetone-*d*₆) 1.09, 1.34, 3.07 and 3.54]. δ_C (acetone-*d*₆) 14.7 (Me), 15.5 (Me'), 26.8 (CH₂), 30.5 (CH₂'), 170.8 and 192.2 (2 × alkynyl C), and 228.2 (CO).

2.9. Synthesis of (η⁶-toluene)tungsten(0) tricarbonyl

BF₃·OEt₂ (0.23 ml, 1.9 mmol) was added to a stirred suspension of [W(CO)₃(Me₃tach)] (0.25 g, 0.629 mmol) in toluene (20 ml), and the mixture was heated at reflux for 2 h. The resulting yellow solution was filtered (alumina/celite) whilst hot and evaporated to dryness to give a yellow solid, which was sublimed in vacuo to yield [W(CO)₃(η⁶-toluene)] as a yellow powder (0.12 g, 53%). δ_H (CDCl₃) 2.39 (3H, s, Me), 5.16–5.54 (5H, m, ArH) [lit. (CDCl₃) [13], δ_H 2.41 and 5.21–5.54]. δ_C (CDCl₃) 20.7 (Me), 86.9 (C4), 89.8 (C2 and C2'), 92.0 (C3 and C3'), 109.0 (Cl) and 209.9 (CO).

2.10. Synthesis of (η⁶-benzene)tungsten(0) tricarbonyl

This compound was prepared using the same procedure as for [W(CO)₃(η⁶-toluene)] given above. Yield (50 mg, 23%); δ_H (CDCl₃) 5.36 (6H, s, C₆H₆) [lit. (CDCl₃) [13] δ_H 5.38]. δ_C (CDCl₃) 89.89 (C₆H₆) and 208.81 (CO).

3. Results and discussion

3.1. Synthesis of *fac*-[Mo(CO)₃(CH₃CN)₃] and *fac*-[W(CO)₃(CH₃CN)₃]

When [Mo(CO)₃(Me₃tach)] and [W(CO)₃(Me₃tach)] were dissolved in acetonitrile at room temperature, solvolysis occurred, producing *fac*-[Mo(CO)₃(CH₃CN)₃] and *fac*-[W(CO)₃(CH₃CN)₃] respectively. The products were precipitated as tan needles by addition of toluene to the solutions, and were easily isolated in analytically-pure form by filtration. Infrared spectroscopy (Fig. 1) indicated that the products consisted entirely of the facial isomers [ν_{max}

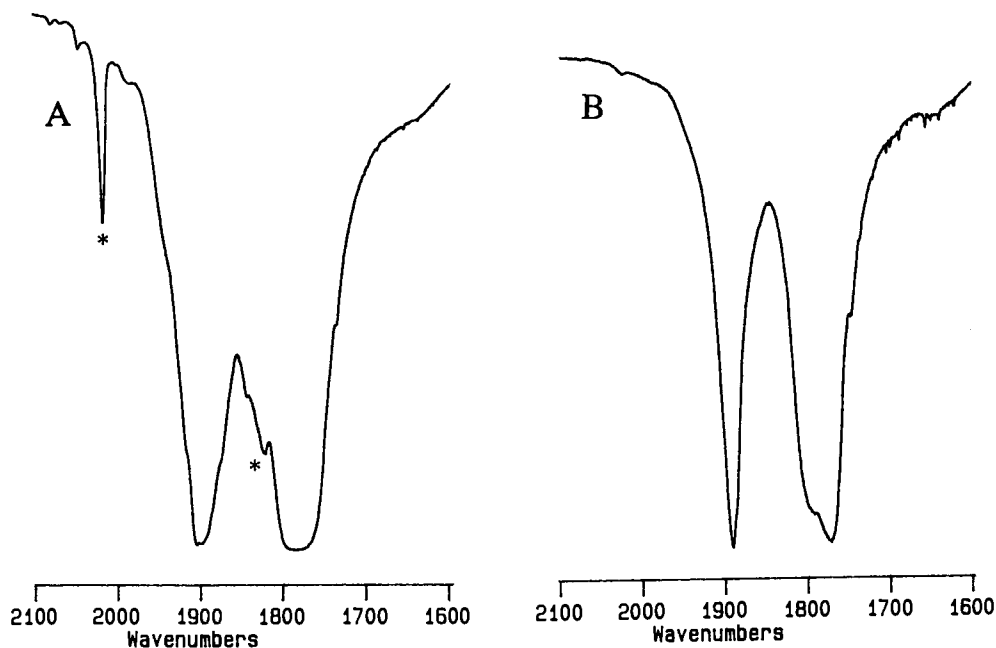


Fig. 1. Carbonyl region of the infrared spectra of samples of *fac*-[W(CO)₃(CH₃CN)₃] prepared from (A) W(CO)₆ in CH₃CN at reflux (B) [W(CO)₃(Me₃tach)] in CH₃CN at room temperature. Asterisks indicate bands due to *cis*-[W(CO)₄(CH₃CN)₂], an impurity in the sample in (A).

(CO) 1905 and 1790 cm⁻¹ (Mo), 1900 and 1790 cm⁻¹ (W)], with no evidence of contamination by other complexes. By contrast, when prepared by the standard method (i.e. from the appropriate metal hexacarbonyl in acetonitrile at reflux) [M(CO)₃(CH₃CN)₃] complexes (M = Mo, W) are almost invariably contaminated with the bis(acetonitrile) complexes [M(CO)₄(CH₃CN)₂], [$\tilde{\nu}_{\max}$ (CO) 2023, 1912, 1881 and 1833 cm⁻¹ (M = Mo), 2022, 1900, 1880 and 1839 cm⁻¹ (M = W)], presumably due to reactions resulting from the disproportionation of *fac*-[M(CO)₃(CH₃CN)₃] at elevated temperatures [11]. This problem is especially pronounced for *fac*-[W(CO)₃(CH₃CN)₃], the preparation of which can require up to 2 weeks in refluxing acetonitrile [9,20].

Since tris(acetonitrile) complexes are frequently used as synthetic intermediates in organometallic synthesis, the synthesis of these complexes from [M(CO)₃(Me₃tach)] has many useful features: (i) *fac*-[M(CO)₃(CH₃CN)₃] can be synthesised in as little as 6 h from M(CO)₆ via [M(CO)₃(Me₃tach)]; (ii) the *fac*-[M(CO)₃(CH₃CN)₃] is obtained in highly pure form, a feature that may be useful for syntheses of certain *fac*-[M(CO)₃(ligand)₃] complexes [21,22]; (iii) [M(CO)₃(Me₃tach)] can be stored indefinitely under an inert atmosphere and can be rapidly converted to [M(CO)₃(CH₃CN)₃] as required. Further, although [M(CO)₃(cycloheptatriene)] also undergoes solvolysis to produce to [M(CO)₃(CH₃CN)₃] [23], [W(CO)₃(cycloheptatriene)] can itself only be obtained in low yields [24].

We have found that whilst [M(CO)₃(Me₃tach)] can be used to prepare *fac*-[M(CO)₃(CH₃CN)₃], the reverse is

also true. For example, when a sample of [W(CO)₃(CH₃CN)₃] prepared according to the literature procedure [9] was stirred with an excess of Me₃tach in THF, [W(CO)₃(Me₃tach)] formed in essentially quantitative yield (96%). Presumably the low solubility of the Me₃tach complex in THF drives the reaction to completion.

3.2. Synthesis of tungsten carbonyl phosphine complexes

A number of syntheses of [W(CO)₃L₃] complexes, where L is a phosphorus donor, have been reported in the literature [25]. Many of these syntheses, however, require long reaction times, are not always general for a range of phosphines, or require the preparation of a reactive intermediate. Direct synthesis of [W(CO)₃L₃] from W(CO)₆ usually requires forcing conditions to remove the carbon monoxide released and leads to a mixture of products ([W(CO)₅L], [W(CO)₄L₂] and [W(CO)₃L₃] species, *cis* and *trans* isomers) [26]. Some of the best indirect methods reported to date use the nitrile derivatives [W(CO)₃(RCN)₃] (R = Me, Et or Pr) or the cycloheptatriene complex [W(CO)₃(cycloheptatriene)], which give the facial phosphine complexes in good yield under mild conditions [27]. The disadvantages of these methods are, as outlined above, that the syntheses of the nitrile intermediates require long reaction times and the cycloheptatriene complex is itself prepared from the nitrile derivative [9,20]. More recently Angelici et al. [13], reported the reaction of [W(CO)₃(PMTA)] [PMTA = MeN(CH₂CH₂NMe₂)₂] with a range of phos-

phines in refluxing THF, with monodentate tertiary phosphines producing the corresponding *fac*-[W(CO)₃(PR₃)₃] complexes in good yield. However, the reaction of [W(CO)₃(PMTA)] with the bulkier and less basic phosphines such as PMePh₂ and the tridentate phosphine CH₃C(CH₂PPh₂)₃, required several hours at reflux and in the case of PPh₃, no reaction was observed [13].

[W(CO)₃(Me₃tach)] reacts with P(OMe)₃ in THF solution to produce the corresponding *fac*-[W(CO)₃{P(OMe)₃}₃] in good yield [8]. The reaction is rapid even when performed at -40°C. The Me₃tach ligand in [W(CO)₃(Me₃tach)] is also readily displaced by PMePh₂ and PPh₃ at room temperature to yield the expected facial phosphine complexes. The *fac* stereochemistry of the products was confirmed by the presence of a single resonance in the ³¹P NMR spectra and carbonyl absorbances characteristic of *fac* stereochemistry in the IR spectra (two bands corresponding to the *A*₁ and *E* stretching modes). The preparation of *fac*-[W(CO)₃(PPh₃)₃] in quantitative yield (96%), after only 1 h stirring at room temperature, represents a significant improvement over the previously reported procedure which used [W(CO)₃(η⁶-mesitylene)] as a starting material (36 h reflux, 32% yield of [W(CO)₃(PPh₃)₃]) [15]. *fac*-[W(CO)₃(PPh₃)₃] may adopt two different crystal forms (*α* and *β*) in the solid state [16]. Comparison of the carbonyl stretching frequencies measured for KBr disks of *fac*-[W(CO)₃(PPh₃)₃] with literature values [16] indicates that the *fac*-[W(CO)₃(PPh₃)₃] obtained from the reaction of [W(CO)₃(Me₃tach)] with PPh₃ is the less soluble *β* form.

Reaction of [W(CO)₃(Me₃tach)] with the sterically hindered phosphine PⁱPr₃ was slower than reactions involving other phosphines and required elevated temperatures (refluxing THF). The formation of *trans*-[W(CO)₄(PⁱPr₃)₂] in this reaction rather than *fac*-[W(CO)₃(PⁱPr₃)₃] reflects the inability of this bulky phosphine to form a tris(phosphine) complex, a feature noted previously for PCy₃ [28].

3.3. Synthesis of tris(alkyne) complexes

The [W(CO)₃(Me₃tach)] complex reacted with internal alkynes in acetone at reflux to afford the known tris(alkyne)tungsten carbonyl compounds 1 and 2 (Scheme 1). Previously, tris(alkyne)tungsten carbonyl complexes have been prepared from either W(CO)₆ or [W(CO)₃(CH₃CN)₃] using a large excess of the appropriate alkyne (often as solvent/reagent) at reflux under reduced pressure [18,29]. The use of [W(CO)₃(Me₃tach)] as an intermediate in these syntheses holds the advantage that only a relatively small excess of the alkyne (we used five equivalents) is required and that the reaction can be carried out at

atmospheric pressure and at a lower temperatures. In the case of the 3-hexyne derivative 2, if a larger excess of alkyne was used (100 equivalents), then the reaction proceeded at room temperature without heating. Whilst there is little need for employing milder conditions of synthesis for the examples given (1 and 2), this method should be particularly useful for the synthesis of complexes of the more reactive terminal alkynes [29]. Preliminary investigations have shown that the synthesis can be extended to [W(CO)(PhC≡CH)₃] [29], but due to the reactive nature of the alkyne we have had difficulty in isolating the product in a pure form.

Attempts to prepare [Mo(CO)(PhC≡CPh)₃] starting from [Mo(CO)₃(Me₃tach)] were unsuccessful. When [Mo(CO)₃(Me₃tach)] was heated with diphenylacetylene in acetone solution there was no apparent reaction; neither molybdenum–diphenylacetylene complexes [30,31] nor oligomers of diphenylacetylene [18,30] were formed.

3.4. Synthesis of [W(CO)₃(η⁶-arene)] complexes

η⁶-Arene tungsten tricarbonyl complexes are generally difficult to prepare [13]. One of the better methods for the synthesis of these complexes was reported by Angelici et al. [13] and started from [W(CO)₃(PMTA)], but this method was complicated by the formation of [W(CO)₄(PMTA)] as a reaction by-product. We have found that if [W(CO)₃(Me₃tach)] is used as the starting material in the procedure of Angelici et al., the η⁶-arene complexes can be obtained in good yield without the formation of other tungsten containing by-products. For example, [W(CO)₃(η⁶-benzene)] and [W(CO)₃(η⁶-toluene)] were prepared in yields of 23 and 53%, respectively, by heating [W(CO)₃(Me₃tach)] and three equivalents of BF₃·OEt₂ in benzene or toluene at reflux. The products were easily purified by sublimation in vacuo.

4. Conclusions

The lability of the Me₃tach unit in [M(CO)₃(Me₃tach)] (M = Mo or W) allows convenient access to a variety of metal carbonyl complexes via unusually mild ligand displacement reactions. [M(CO)₃(Me₃tach)] complexes are particularly useful intermediates in the synthesis of Mo and W tricarbonyl complexes in which facial or meridional isomers are possible but where only the facial product is desired [21]. In addition to the ligand displacement reactions described above, the Me₃tach unit can be displaced by other R₃tach's and should also be easily displaced by other tridentate ligands.

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