

HREIMS m/z (rel. int.) 285.2091 [M^+] (32) (calcd 285.2093 for $C_{19}H_{27}NO$), 200.1071 (41), 186.0915 (68), 173.0846 (100), 144.0832 (14); UV (MeOH) 214, 239, 322, 335 nm; IR (KBr) 3350, 2830, 1639, 1608, 1593, 1557, 1503, 1481, 1397, 1360, 1000, 758, 694 cm^{-1} ; 1H NMR (CD_3OD) δ 0.88 (t, 3H, $J=7.1$ Hz), 1.27-1.34 (m, 8H), 1.32-1.38 (m, 2H), 1.38-1.46 (m, 2H), 1.70-1.73 (m, 2H), 2.15 (s, 3H), 2.81 (t, 2H, $J=8.0$ Hz), 7.33 (ddd, 1H, $J=8.2, 6.9, 1.0$ Hz), 7.53 (dd, 1H, $J=8.4, 1.0$ Hz), 7.62 (ddd, 1H, $J=8.4, 6.9, 1.4$ Hz), 8.22 (dd, 1H, $J=8.2, 1.4$ Hz) ppm; ^{13}C NMR (CD_3OD) δ 179.6, 153.3, 140.6, 132.6, 126.2, 124.47, 124.42, 118.6, 116.2, 33.4, 33.0, 30.6, 30.5, 30.4, 30.3, 30.0, 23.7, 14.4, 10.8 ppm; HPLC Rt 11.5 min (same as the natural product,¹² Phenomenex μ -Bondapak C-18, 3.9×300 mm, UV 225 nm, 1 mL/min, 75:25 MeOH/H₂O).

2-Pentyl-4-quinolinone (4). Obtained as a white solid in 69% yield starting from ethyl 3-oxooctanoate (**11**)¹⁸: mp 139-140 °C (lit⁹ 141-142 °C, lit¹⁵ 134-138 °C); EIMS m/z (rel. int.) 215 [M^+] (17), 186 (8), 172 (26), 159 (100), 130 (12), 44 (29); UV (MeOH) 213, 234, 315, 327 nm; IR (KBr) 3350, 2900, 1628, 1592, 1548, 1495, 1473, 1439, 1315, 1249, 798, 750 cm^{-1} ; 1H NMR (CD_3OD) δ 0.92 (t, 3H, $J=7.0$ Hz), 1.37-1.42 (m, 4H), 1.76-1.78 (m, 2H), 2.70 (t, 2H, $J=7.7$ Hz), 6.22 (s, 1H), 7.37 (ddd, 1H, $J=8.2, 7.0, 1.1$ Hz), 7.57 (ddd, 1H, $J=8.4, 1.1, 0.4$ Hz), 7.62 (ddd, 1H, $J=8.4, 7.0, 1.5$ Hz), 8.20 (ddd, 1H, $J=8.2, 1.5, 0.4$ Hz) ppm; ^{13}C NMR (CD_3OD) δ 180.7, 157.1, 141.6, 133.4, 126.0, 125.5, 125.0, 119.0, 108.9, 35.0, 32.4, 29.8, 23.4, 14.2 ppm.

2-Heptyl-4-quinolinone (5). Obtained as a white solid in 75% yield starting from ethyl 3-oxodecanoate (**12**)¹⁸: mp 141-142 °C (lit¹⁴, 138-141 °C); EIMS m/z (rel. int.) 243 [M^+] (21), 172 (43), 159 (100), 130 (9); UV (MeOH) 213, 234, 315, 327 nm; IR (KBr) 3400, 2870, 1633, 1595, 1556, 1510, 1476, 1447, 1388, 1195, 1131, 763 cm^{-1} ; 1H NMR (CD_3OD) δ 0.89 (t, 3H, $J=7.0$ Hz), 1.29-1.34 (m, 4H), 1.32-1.43 (m, 4H), 1.76 (quintet, 2H, $J=7.7$ Hz), 2.71 (t, 2H, $J=7.7$ Hz), 6.22 (s, 1H), 7.38 (ddd, 1H, $J=8.2, 7.0, 1.1$ Hz), 7.57 (ddd, 1H, $J=8.4, 1.1, 0.3$ Hz), 7.62 (ddd, 1H, $J=8.4, 7.0, 1.5$ Hz), 8.20 (ddd, 1H, $J=8.2, 1.5, 0.3$ Hz) ppm; ^{13}C NMR (CD_3OD) δ 180.7, 157.1, 141.6, 133.4, 126.0, 125.5, 125.0, 119.0, 108.9, 35.0, 32.8, 30.2, 30.1, 30.0, 23.6, 14.3 ppm.

2-Nonyl-4-quinolinone (6). Obtained as a white solid in 72% yield starting from ethyl 3-oxododecanoate (**13**)¹⁸: mp 131-132 °C (lit¹⁴, 129-132 °C); EIMS m/z (rel. int.) 271 [M^+] (20), 172 (58), 159 (100), 130 (10); UV (MeOH) 213, 234, 315, 327 nm; IR (KBr) 2800, 1638, 1593, 1552, 1503, 1473, 1444, 1353, 1327, 1137, 762 cm^{-1} ; 1H NMR (CD_3OD) δ 0.87 (t, 3H, $J=7.0$ Hz), 1.22-1.33 (m, 8H), 1.32-1.43 (m, 4H), 1.76 (quintet, 2H, $J=7.7$ Hz), 2.71 (t, 2H, $J=7.7$ Hz), 6.22 (s, 1H), 7.38 (ddd, 1H, $J=8.2, 7.0, 1.1$ Hz), 7.57 (ddd, 1H, $J=8.4, 1.1, 0.5$ Hz), 7.62 (ddd, 1H, $J=8.4, 7.0, 1.5$ Hz), 8.20 (ddd, 1H, $J=8.2, 1.5, 0.5$ Hz) ppm; ^{13}C NMR (CD_3OD) δ 180.7, 157.1, 141.6, 133.4, 126.0, 125.5, 125.0, 119.0, 108.9, 35.0, 33.0, 30.5, 30.4, 30.3, 30.1, 30.1, 23.7, 14.3 ppm.

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Efficient Synthetic Methods for $(\eta^5-C_5H_5)(CO)_2Cr \equiv C(C_6H_4Me-4)$

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Since the first transition metal alkylidyne complex was reported by Fischer and coworkers in 1973,¹ its chemistry has been extensively investigated in various aspects, *i.e.*, precursors for synthetic use,² active catalysts for alkyne me-

tathesis³ and polymerization.⁴ We and others have employed the group-6 alkylidyne complexes, $\text{Cp}(\text{CO})_2\text{M}\equiv\text{CTol}$ [$\text{M}=\text{Cr}$ (**1**), Mo (**2**) and W (**3**), $\text{Cp}=\eta^5\text{-C}_5\text{H}_5$, $\text{Tol}=\textit{p}\text{-C}_6\text{H}_4\text{Me}$], as reagents for the synthesis of mixed metal cluster compounds containing group-6 metals and bridging alkylidyne ligands.⁵ We could prepare complexes **2** and **3** without difficulties by the reported procedures from the bromo alkylidyne complexes as shown in eq. (1).⁶ We, however, could obtain the chromium alkylidyne complex **1** in very low yields (<5%) by

$$\text{Br}(\text{CO})_4\text{M}\equiv\text{CTol} + \text{Cp}^- \rightarrow \text{Cp}(\text{CO})_2\text{M}\equiv\text{CTol} + 2 \text{CO} + \text{Br}^- \quad (1)$$

the reported procedure which claims 25% yield for the formation of **1**.⁷ Herein we report efficient synthetic methods of chromium alkylidyne complexes, **1** and $\text{Tp}^*(\text{CO})_2\text{Cr}\equiv\text{CTol}$ (**6**) [$\text{Tp}^*=\text{hydrotris}(3,5\text{-dimethyl pyrazol-1-yl})\text{borato}$], via a bis(pyridine)-substituted bromo alkylidyne complex, $\text{Br}(\text{CO})_2(\text{py})_2\text{Cr}\equiv\text{CTol}$ (**5**).

Experimental Section

General Comments. All reactions were carried out under an atmosphere of nitrogen with use of standard Schlenk techniques. Solvents were dried prior to use. ¹H NMR (300 MHz) and ¹³C NMR (75 MHz) spectra were recorded on a Bruker AM-300 spectrometer. Infrared spectra were obtained with a Bomem MB-100 FT-IR spectrophotometer. $(\text{CO})_5\text{Cr}=\text{C}(\text{OMe})\text{Tol}$ was prepared as described in the literature.⁸

Preparation of 5 from $(\text{CO})_5\text{Cr}=\text{C}(\text{OMe})\text{Tol}$. A petroleum ether solution (250 mL) of $(\text{CO})_5\text{Cr}=\text{C}(\text{OMe})\text{Tol}$ (2.00 g, 6.13 mmol) at -20°C was treated with BBr_3 (8.00 mL of 1.0 M solution in hexane, 8.00 mmol), whereby a yellow precipitate, $\text{Br}(\text{CO})_4\text{Cr}\equiv\text{CTol}$ (**4**), formed immediately. The reaction mixture was stirred at -20°C for 1.5 h. After the supernatant was decanted off, the yellow precipitate was washed with petroleum ether (3×10 mL) at -20°C and dried *in vacuo*. The yellow precipitate [$\text{Br}(\text{CO})_4\text{Cr}\equiv\text{CTol}$] was dissolved in dichloromethane (200 mL) at -30°C and then pyridine (2.50 mL, 30.91 mmol) was added. The solution was warmed to 0°C (ice bath), during which time the color changed to red, and stirred for 2 h. The solvent was removed to give a red solid, $\text{Br}(\text{CO})_2(\text{py})_2\text{Cr}\equiv\text{CTol}$ (**5**). The solid was recrystallized with a mixture of CH_2Cl_2 and petroleum ether to afford a red crystalline solid (2.55 g, 5.67 mmol, 93%).

¹H NMR (CDCl_3 , 25°C): δ 9.08 (m, 10H, pyridine), 7.11-7.68 (m, 4H, C_6H_4), 2.35 (s, 3H, Tol-CH_3); ¹³C NMR (CDCl_3 , 25°C): δ 304.2 ($\text{C}_{\text{carbyne}}$), 229.4 (2 CO), 153.2, 144.6, 139.3, 137.3, 128.8, 128.6, 124.0 (C_{aryl} of pyridine and Tol), 21.6 (Tol-CH_3); IR (CH_2Cl_2) $\nu(\text{CO})$ 1998 (s), 1923 (s) cm^{-1} .

Preparation of 1 from 5. A tetrahydrofuran (THF) solution of **5** (2.00 g, 4.44 mmol) was cooled to -20°C and NaCp (2.25 mL, 2.0 M solution in THF, 4.50 mmol) was added using a gas tight syringe. After stirring at -20°C for 4 h, the solvent was removed and the residue was extracted with cold petroleum ether (-20°C) to give an orange solution. The solvent of the filtrate was removed and the resulting orange solid (0.97 g, 3.51 mmol, 79%) was collected.

¹H NMR (CDCl_3 , 25°C): δ 7.05-7.41 (AB pattern, 4H, C_6H_4), 5.12 (s, 5H, Cp), 2.33 (s, 3H, Tol-CH_3); IR (cyclohexane) $\nu(\text{CO})$ 1995 (s), 1931 (s) cm^{-1} .

Preparations of 1 and 6 from $\text{Cr}(\text{CO})_6$. ToLi [*in*

situ generation from *p*-bromotoluene (1.00 g, 5.85 mmol) and *n*-butyl lithium (2.40 mL of 2.5 M solution in hexane, 6.00 mmol) in ether] was added to a suspension of $\text{Cr}(\text{CO})_6$ (1.21 g, 5.50 mmol) in diethyl ether at room temperature. The reaction mixture was stirred for 2 h and oxalyl dibromide, $\text{BrC}(\text{O})\text{C}(\text{O})\text{Br}$ (3.00 mL of 2.0 M solution in CH_2Cl_2 , 6.00 mmol), was added at -78°C . The resulting solution was allowed to warm to -40°C and stirred for 4 h. The solvent was removed at -20°C to give a brown-yellow residue. The residue was redissolved in dichloromethane at -40°C and treated with pyridine (2.22 mL, 27.50 mmol). The color of solution changed to yellow immediately. The solution was warmed to 0°C and stirred for 2 h during which time the yellow solution turned to dark red. The resulting red solution was reduced in volume and cold petroleum ether was added until precipitation of pyridine-substituted complex was complete. The supernatant was decanted off and the residue washed with petroleum ether three times (3×10 mL). The solid was redissolved in cold THF and cooled to -20°C . Corresponding alkali salts [NaCp (3.00 mL of 2.0 M solution in THF, 6.00 mmol) and KTp^* (2.01 g, 6.00 mmol)] were added and the solution was stirred for 4 h. The solvent was removed and the residue purified by column chromatography on alumina at -20°C . Excess pyridine was first eluted with petroleum ether. Further elution with $\text{CH}_2\text{Cl}_2/\text{petroleum ether}$ (1:2) gave an orange-red solution of **1** or a red solution of **6**, from which micro crystalline solids were obtained after removal of the solvent *in vacuo* at -20°C , respectively, (**1**; 0.85 g, 3.08 mmol, 56%, **6**; 1.48 g, 2.91 mmol, 53%).

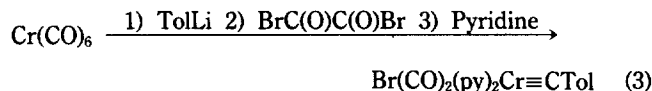
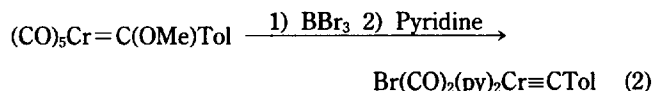
Compound **6**: ¹H NMR (CDCl_3 , 25°C): δ 7.56-7.10 (AB pattern, 4H, C_6H_4), 5.78 (s, 1H, $\text{Tp}^*\text{-CH}$), 5.77 (s, 2H, $\text{Tp}^*\text{-CH}$), 2.52 (s, 3H, $\text{Tp}^*\text{-CH}_3$), 2.49 (s, 6H, $\text{Tp}^*\text{-CH}_3$), 2.37 (s, 3H, $\text{Tp}^*\text{-CH}_3$ or Tol-CH_3), 2.34 (s, 3H, $\text{Tp}^*\text{-CH}_3$ or Tol-CH_3), 2.33 (s, 6H, $\text{Tp}^*\text{-CH}_3$); IR (cyclohexane) $\nu(\text{CO})$ 1987 (m), 1909 (s) cm^{-1} .

Results and Discussion

We have successfully utilized the cyclopentadienyl-substituted molybdenum and tungsten analogous, $\text{Cp}(\text{CO})_2\text{M}\equiv\text{CTol}$ [$\text{M}=\text{Mo}$ (**2**) and W (**3**)], for the synthesis of various MoOs_3 and WOs_3 mixed metal cluster complexes.^{5a,b,9} Complexes **2** and **3** have been conveniently prepared according to eq. (1) as described in the literature.⁶ In order to extend the scope of our cluster chemistry to presently unknown CrOs_3 clusters, we have been interested in the development of high yield synthetic method of $\text{Cp}(\text{CO})_2\text{Cr}\equiv\text{CTol}$ (**1**). Complex **1** has been recently prepared from the reaction of $\text{Br}(\text{CO})_4\text{Cr}\equiv\text{CTol}$ (**4**) and NaCp in Et_2O in 25% yield and reported to be somewhat unstable in contrast with complexes **2** and **3** by Stone and coworkers.⁷ Later they have also reported that $\text{Cp}(\text{CO})_2\text{Cr}=\text{C}(\text{C}_6\text{H}_3\text{Me}_2\text{-2,6})$ could be prepared in 80% yield via the trifluoroacetate derivative, $(\text{CF}_3\text{CO}_2)(\text{CO})_4\text{Cr}=\text{C}(\text{C}_6\text{H}_3\text{Me}_2\text{-2,6})$, instead of the bromo analogue.¹⁰ We have attempted both Stone's synthetic methods to prepare complex **1**, but have not been successful in our hands resulting in very low yields (<5%) of **1**.

The mean dissociation enthalpy of group-6 metal hexacarbonyl complexes increases in the order of $\text{Cr} < \text{Mo} < \text{W}^{11}$; nevertheless, the calculated first carbonyl ligand dissociation energy of $\text{M}(\text{CO})_6$ is reported to increase in the order of

Mo < W < Cr.¹² We, therefore, thought that the carbonyl substitution is a rate-determining step with chromium derivatives and thus a starting chromium complex with more labile ligands than the carbonyl ligand is required. The bis(pyridine)-substituted complex, Br(CO)₂(py)₂Cr≡CTol (**5**), can be easily prepared from either (CO)₅Cr=C(OMe)Tol or Cr(CO)₆ without isolation of **4** as shown in eqs. (2) and (3).¹³



When decarbonylation of **4** is carried out at room temperature in the presence of excess (*ca.* 5 fold) pyridine, quantitative formation of **5** is observed. The synthetic method of eq. (3) is useful for one-pot synthesis of complex **5** from Cr(CO)₆. The IR spectrum of **5** exhibits two ν(CO) absorption bands of almost equal intensity at 1998 and 1923 cm⁻¹ indicating a *cis*-arrangement of the two carbonyl ligands as was proposed for the structure of Br(CO)₂(py)₂Cr≡CPh (Ph = C₆H₅).¹⁴ The higher energy absorption is assigned to the symmetric A₁ mode and the lower energy one to the asymmetric B₁ mode due to the C_{2v} local symmetry of the two carbonyl ligands.¹⁵ The ¹³C NMR spectrum (CDCl₃, -30 °C) of **5** shows an alkylidyne carbon resonance at δ 304.2 and a single resonance at δ 229.4 for the two equivalent *cis*-carbonyl ligands.

The reaction of **5** with NaCp indeed proceeds smoothly and *in situ* synthesis of Cp(CO)₂Cr≡CTol (**1**) results in a high yield of either 73% from (CO)₅Cr=C(OMe)Tol or 56% from Cr(CO)₆. Similarly, reaction of **5** with Tp*K results in the clean formation of Tp*(CO)₂Cr≡CTol (**6**), which can be prepared as a red solid from Cr(CO)₆ in 53% yield. The IR spectrum of **6** also reveals two absorption bands at 1909 and 1987 cm⁻¹, which is consistent with the *cis*-dicarbonyl ligands. The ¹H NMR spectrum (25 °C, CDCl₃) of **6** displays a 2 : 1 pattern for the hydrogens of the pyrazol-1-yl groups, implying that the Tp* ligand in **6** is not fluxional. However, the analogous tungsten complexes Tp(CO)₂W≡CNR₂ [Tp = hydrotris(pyrazol-1-yl)borato; R = Me, Et] have been reported to be fluxional at 25 °C.¹⁶ The TMEDA (tetramethylethylene diamine) derivative, Br(CO)₂(tmeda)Cr≡CTol¹⁷ does not undergo reaction with NaCp revealing the chelating effect of the TMEDA ligand. Mayr and coworkers have also made use of thermal stability and coordinative lability of group-6 alkylidyne complexes with nitrogen donor ligands in various substitution reactions.¹³ An analogous synthetic method for half-sandwich chromium aminocarbene complex, Cp(CO)₂Cr≡CNEt₂, has been recently developed by Filippou and coworkers by using a γ-picoline derivative, Br(CO)₂(pic)₂Cr≡CNEt₂.¹⁸

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