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/H_2O).

2-Pentyl-4-quinolinone (4). Obtained as a white solid in 69% yield starting from ethyl 3-oxooctanoate (**11**)[18] : mp 139-140 ℃ (lit[9] 141-142 ℃, lit[15] 134-138 ℃); 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 ℃ (lit[14]. 138-141 ℃); 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 ℃ (lit[14]. 129-132 ℃); 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 (η^5-C_5H_5)$(CO)_2$Cr≡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-

tathesis[3] and polymerization.[4] We and others have employed the group-6 alkylidyne complexes, $Cp(CO)_2M{\equiv}CTol$ [M=Cr (**1**), Mo (**2**) and W (**3**), Cp=η^5-C_5H_5, Tol=*p*-C_6H_4Me], 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

$$Br(CO)_4M{\equiv}CTol + Cp^- \rightarrow Cp(CO)_2M{\equiv}CTol + 2\ CO + Br^- \quad (1)$$

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 $Tp^*(CO)_2Cr{\equiv}CTol$ (**6**) [Tp^*=hydrotris(3,5-dimethyl pyrazol-1-yl)borato], *via* a bis(pyridine)-substituted bromo alkylidyne complex, $Br(CO)_2(py)_2Cr{\equiv}CTol$ (**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 ^{13}C NMR (75 MHz) spectra were recorded on a Bruker AM-300 spectometer. Infrared spectra were obtained with a Bomem MB-100 FT-IR spectrophotometer. $(CO)_5Cr{=}C(OMe)Tol$ was prepared as described in the literature.[8]

Preparation of 5 from $(CO)_5Cr{=}C(OMe)Tol$. A petroleum ether solution (250 mL) of $(CO)_5Cr{=}C(OMe)Tol$ (2.00 g, 6.13 mmol) at −20 ℃ was treated with BBr_3 (8.00 mL of 1.0 M solution in hexane, 8.00 mmol), whereby a yellow precipitate, $Br(CO)_4Cr{\equiv}CTol$ (**4**), formed immediately. The reaction mixture was stirred at −20 ℃ for 1.5 h. After the supernatant was decanted off, the yellow precipitate was washed with petroleum ether (3×10 mL) at −20 ℃ and dried *in vacuo*. The yellow precipitate [$Br(CO)_4Cr{\equiv}CTol$] was dissolved in dichloromethane (200 mL) at −30 ℃ and then pyridine (2.50 mL, 30.91 mmol) was added. The solution was warmed to 0 ℃ (ice bath), during which time the color changed to red, and stirred for 2 h. The solvent was removed to give a red solid, $Br(CO)_2(py)_2Cr{\equiv}CTol$ (**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 ℃): δ 9.08 (m, 10H, pyridine), 7.11-7.68 (m, 4H, C_6H_4), 2.35 (s, 3H, Tol-CH_3); ^{13}C NMR ($CDCl_3$, 25 ℃): δ 304.2 ($C_{carbyne}$), 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) ν(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 ℃ 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 ℃ for 4 h, the solvent was removed and the residue was extracted with cold petroleum ether (−20 ℃) 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 ℃): 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) ν(CO) 1995 (s), 1931 (s) cm^{-1}.

Preparations of 1 and 6 from $Cr(CO)_6$. TolLi [*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 $Cr(CO)_6$ (1.21 g, 5.50 mmol) in diethyl ether at room temperature. The reaction mixture was stirred for 2 h and oxalyl dibromide, BrC(O)C(O)Br (3.00 mL of 2.0 M solution in CH_2Cl_2, 6.00 mmol), was added at −78 ℃. The resulting solution was allowed to warm to −40 ℃ and stirred for 4 h. The solvent was removed at −20 ℃ to give a brown-yellow residue. The residue was redissolved in dichloromethane at −40 ℃ and treated with pyridine (2.22 mL, 27.50 mmol). The color of solution changed to yellow immediately. The solution was warm to 0 ℃ 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 ℃. 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 ℃. Excess pyridine was first eluted with petroleum ether. Further elution with CH_2Cl_2/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 ℃, respectively, (**1**; 0.85 g, 3.08 mmol, 56%, 6; 1.48 g, 2.91 mmol, 53%).

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

Results and Discussion

We have successfully utilized the cyclopentadienyl-substituted molybdenum and tungsten analogous, $Cp(CO)_2M{\equiv}CTol$ [M=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 $Cp(CO)_2Cr{\equiv}CTol$ (**1**). Complex **1** has been recently prepared from the reaction of $Br(CO)_4Cr{\equiv}CTol$ (**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 $Cp(CO)_2Cr{\equiv}C(C_6H_3Me_2\text{-}2,6)$ could be prepared in 80% yield via the trifluoroacetate derivative, $(CF_3CO_2)(CO)_4Cr{\equiv}C(C_6H_3Me_2\text{-}2,6)$, 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 Cr<Mo<W[11]; nevertheless, the calculated first carbonyl ligand dissociation energy of $M(CO)_6$ 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, $Br(CO)_2(py)_2Cr{\equiv}CTol$ (**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]

$$(CO)_5Cr{=}C(OMe)Tol \xrightarrow{1)\ BBr_3\ \ 2)\ Pyridine} Br(CO)_2(py)_2Cr{\equiv}CTol \quad (2)$$

$$Cr(CO)_6 \xrightarrow{1)\ TolLi\ \ 2)\ BrC(O)C(O)Br\ \ 3)\ Pyridine} Br(CO)_2(py)_2Cr{\equiv}CTol \quad (3)$$

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 $Br(CO)_2(py)_2Cr{\equiv}CPh$ (Ph=C_6H_5).[14] The higher energy absorption is assigned to the symmetric A_1 mode and the lower energy one to the asymmetric B_1 mode due to the C_{2v} local symmetry of the two carbonyl ligands.[15] The ^{13}C NMR spectrum ($CDCl_3$, −30 ℃) 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 $Cp(CO)_2Cr{\equiv}CTol$ (**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 $Tp^*(CO)_2Cr{\equiv}CTol$ (**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 ℃, $CDCl_3$) 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 $Tp(CO)_2W{\equiv}CNR_2$ [Tp=hydrotris(pyrazol-1-yl)borato; R=Me, Et] have been reported to be fluxional at 25 ℃.[16] The TMEDA (tetramethylethylene diamine) derivative, $Br(CO)_2(tmeda)Cr{\equiv}CTol$[17] 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 aminocarbyne complex, $Cp(CO)_2Cr{\equiv}CNEt_2$, has been recently developed by Filippou and coworkers by using a γ-picoline derivative, $Br(CO)_2(pic)_2Cr{\equiv}CNEt_2$.[18]

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