Supplementary Material (ESI) for Chemical Communications


# Supporting Information

## Induction of Axially Chiral N-C Bonds in *N*-Aryl Acridane and Related Complexes by Chromium Tricarbonyl Migration Reactions

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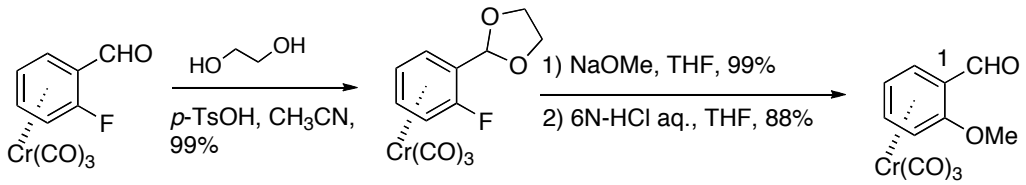
### General Remarks

All manipulations involving organometallics were carried out under an atmosphere of nitrogen and using an inert gas/vacuum double manifold techniques. NMR spectra were recorded in $CDCl_3$ or $C_6D_6$ solvent with tetramethylsilane as an internal reference. IR spectra were determined on a JASCO FT/IR-4100 spectrometer. Mass spectra were determined on a JEOL MS700 with EI mode. Optical rotations were obtained on a JASCO DIP-370 automatic polarimeter at wavelength 589 nm (sodium D line) using a 1.0-dm cell with a total volume of 5 mL.

### Absolute configuration in fluoro(arene)chromium complex

Optically active arene chromium complex **P1** was prepared by an optical resolution according to the reported procedure.[1] The absolute configuration of complex **P2** was determined as (1*S*) by a transformation to the literature known complex (1*S*)-**P3**.[2] Complex (1*S*)-**P2** was utilized as a starting material for the preparation of complexes **5f-5i**. Complexes **P2**, **P5** and **P6** were literature known compounds.[3]



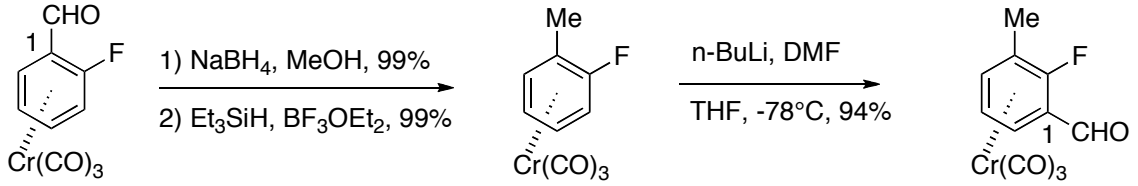

**P1**
$[\alpha]_D^{21}$ +1114
(*c* 0.5, $CHCl_3$)

**P2**
$[\alpha]_D^{23}$ +87.6
(*c* 0.5, $CHCl_3$)

(1*S*)-**P3**
$[\alpha]_D^{31}$ +1058
(*c* 0.1, $CHCl_3$)

lit. (1*S*)- **P3**, $[\alpha]_D^{22}$ +1029 ($CHCl_3$)

Next, complex (1*R*)- **P5** was prepared from complex (1*S*)-**P1** by following three steps.

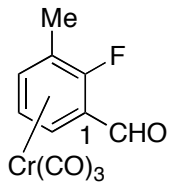



(1*S*)-**P1**
$[\alpha]_D^{21}$ +1114
(*c* 0.5, $CHCl_3$)

**P4**
$[\alpha]_D^{27}$ +20.4
(*c* 0.8, $CHCl_3$)

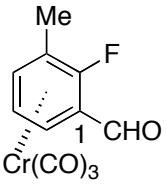
(1*R*)-**P5**
$[\alpha]_D^{22}$ +844.4
(*c* 0.04, $CHCl_3$)

On the other hand, both (1*S*) and (1*R*)-**P5** were prepared by an optical resolution. Complex (1*S*)-**P5** was transformed to complex **9**[3b] or other derivatives[3b], and utilized as a starting material for the preparation of complexes **3** or **5a-5e**.




*rac*-**P5**

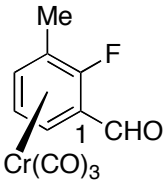
1) L-varinol, *p*-TsOH
2) optical resolution by slicagel
3) HCl aq.




(1*R*)-**P5**

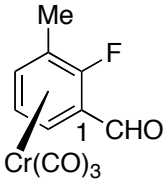
$[\alpha]_D^{22}$ +844.4
(*c* 0.04, $CHCl_3$)

+



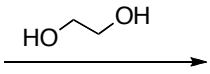

(1*S*)-**P5**

$[\alpha]_D^{22}$ -838.7
(*c* 0.06, $CHCl_3$)

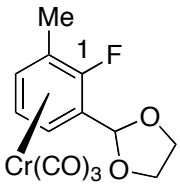



(1*S*)-**P5**

$[\alpha]_D^{22}$ -838.7
(*c* 0.06, $CHCl_3$)



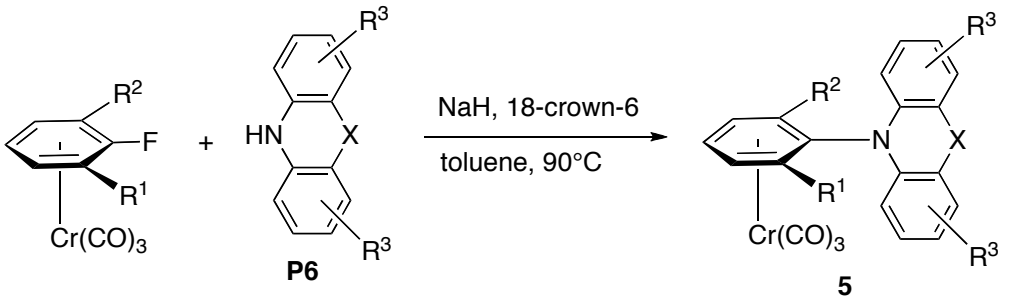

*p*-TsOH, $CH_3CN$,
99%




(1*R*)-**9**

$[\alpha]_D^{30}$ -378.6
(*c* 0.028, $CHCl_3$)

**General procedure for the preparation of complex 5 by a nucleophilic substitution reaction.**



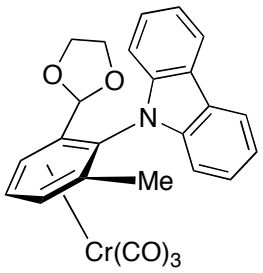

To a solution of **P6** (0.17 mmol) in toluene (5.0 mL), 18-crown-6 (0.24 mmol), NaH (0.24 mmol) was added at 25 °C under argon. The resulting mixture was stirred for 30 min at 25 °C, then a chromium complex (0.16 mmol) which was dissolved in toluene, was added to the reaction mixture and stirred for 2 h at 90 °C. The mixture was quenched with $H_2O$ at 0 °C, extracted with ethyl acetate, washed with brine. The organic layer was dried over anhydrous $MgSO_4$, and filtrate was concentrated under reduced pressure. The residue was purified by silica gel chromatography to give chromium complex **5**. Carbazole chromium complex **3** was also prepared by a same procedure.

**General procedure for the stereoselective chromium tricarbonyl migration reaction.**

The toluene solution of chromium complex **5** was degassed by three freeze/vacuum/thaw cycles and heated reflux for 2 h under nitrogen. Toluene was removed under reduced pressure, the residue was purified by silica gel chromatography.

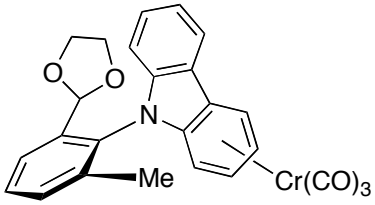
**Note:** Diastereomeric ratio was determined by $^1$H-NMR of the crude product. Crude products were essentially stable in air.

**Carbazole Complex 3**




$[\alpha]_D^{28}$ –67.6 (*c* 0.074, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 1.65 (3H, s), 3.60 (1H, d, *J* = 5.6 Hz), 3.74-3.80 (2H, m), 3.96 (1H, t, *J* = 5.6 Hz), 5.19 (1H, d, *J* = 6.1 Hz), 5.33 (1H, s), 5.47 (1H, d, *J* = 6.1 Hz), 5.79 (1H, t, *J* = 6.1 Hz), 6.91 (1H, d, *J* = 8.3 Hz), 7.27-7.39 (3H, m), 7.60 (1H, t, *J* = 8.3 Hz), 8.11 (2H, t, *J* = 8.3 Hz), 8.23 (1H, d, *J* = 8.3 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.04, 65.71, 65.91, 77.21, 84.44, 89.34, 93.55, 95.27, 99.43, 110.10, 111.52, 111.57, 120.10, 120.18, 120.20, 120.48, 120.55, 123.65, 126.07, 126.25, 128.13, 231.75; IR ($CHCl_3$) 1977, 1908, 1642, 1455 $cm^{-1}$; MS (relative intensity) *m/z* 465 ($M^+$, 20), 381 (80), 353, (88), 69 (100); HRMS calcd for $C_{25}H_{19}NO_5Cr$, 465.0668. found 465.0666.

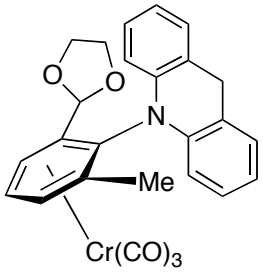
**Carbazole Complex 4**




mp. 172 °C; $[\alpha]_D^{29}$ –6.7 (*c* 0.060, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 1.57 (3H, s), 3.84 (1H, dq, *J* = 6.8, 1.4 Hz), 3.94 (1H, dq, *J* = 6.8, 1.4 Hz), 4.06 (1H, dq, *J* = 6.8, 1.4 Hz), 4.16 (1H, dq, *J* = 6.8, 1.4 Hz), 5.21 (1H, t, *J* = 6.5 Hz), 5.39 (1H, t, *J* = 6.5 Hz), 5.65 (1H, s), 5.68 (1H, d, *J* = 6.5 Hz), 6.50 (1H, d, *J* = 6.5 Hz), 6.93 (1H, d, *J* = 7.9 Hz), 7.31 (1H, dt, *J* = 7.6, 1.2 Hz), 7.38-7.44 (2H, m), 7.53, (1H, t, *J* = 7.9 Hz), 7.78 (1H, dd, *J* = 7.6, 1.2 Hz), 7.95 (1H, d, *J* = 7.9 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.3, 65.4, 65.5, 78.4, 85.7, 87.4, 94.1, 99.1, 111.2, 120.3, 121.6, 122.5, 124.0, 126.0, 126.1, 12.4, 130.1, 132.6, 133.1, 137.0, 138.1, 143.7, 233.9; IR ($CHCl_3$) 2995, 1958, 1884 $cm^{-1}$; MS (relative intensity) *m/z* 465 ($M^+$, 17), 381 (94), 353, (100); HRMS calcd for $C_{25}H_{19}NO_5Cr$, 465.0668. found 465.0668; HPLC condition; Chiralcel OD;

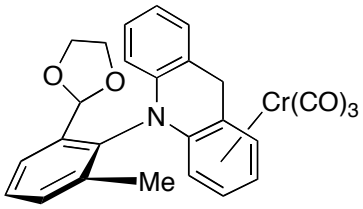
hexane/2-propanol = 20/1; flow rate 1.0 mL/min; column temperature 40 ˚C; UV detector 254 nm, retention time, 13.3 min for (+)-isomer, 15.8 min for (–)-isomer.

**Complex 5a**




mp. 155 °C; $[\alpha]_D^{29}$ –86.92 (*c* 1.3, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 2.02 (3H, s), 3.84-4.18 (6H, m), 5.21 (1H, d, *J* = 5.1 Hz), 5.56-5.61 (3H, m), 6.38 (1H, d, *J* = 8.0 Hz), 6.94 (1H, t, *J* = 8.0 Hz), 7.01 (2H, t, *J* = 7.0 Hz), 7.16 (2H, d, *J* = 7.0 Hz), 7.28 (1H, t, *J* = 8.0 Hz), 7.74 (1H, d, *J* = 8.0 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.5, 32.6, 66.1, 86.9, 91.2, 93.0, 99.9, 111.2, 116.1, 116.4, 117.7, 122.2, 124.4, 125.6, 126.3, 126.8, 127.6, 127.9, 141.2, 142.7, 231.8; IR ($CHCl_3$) 3007, 2956, 1975, 1905, 1597, 909 $cm^{-1}$; MS (relative intensity) *m/z* 479 ($M^+$, 7), 395 (43), 343 (12), 298 (100); HRMS calcd for $C_{26}H_{21}NO_5Cr$, 479.0825. found 479.0831.

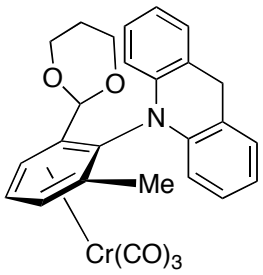
**Complex 6a**




mp. 149 °C; $[\alpha]_D^{26}$ +575.0 (*c* 0.040, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 1.99 (3H, s), 3.96-4.30 (6H, m), 4.67 (1H, d, *J* = 6.3 Hz), 5.00 (1H, t, *J* = 6.3 Hz), 5.26 (1H, t, *J* = 6.3 Hz), 5.45 (1H, d, *J* = 6.3 Hz), 5.80 (1H, s), 6.17 (1H, d, *J* = 7.3 Hz), 6.95-7.02 (2H, m), 7.14 (1H, d, *J* = 7.3 Hz), 7.41-7.49 (2H, m), 7.72 (1H, d, *J* = 7.3 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.5, 30.4, 65.5, 65.6, 81.3, 87.0, 91.9, 92.5, 93.6, 99.4, 115.2, 119.0, 122.7, 124.6, 126.6, 127.7, 128.6, 129.7, 133.7, 136.2, 136.4, 138.8, 139.5, 234.0; IR ($CHCl_3$) 3007, 1959, 1886, 1463, 909 $cm^{-1}$; MS (relative intensity) *m/z* 479 ($M^+$, 8), 395

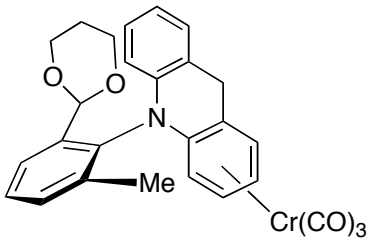
(43), 298 (100); HRMS calcd for $C_{26}H_{21}NO_5Cr$, 479.0825. found 479.0829; HPLC condition; Chiralcel OD; hexane/2-propanol = 20/1; flow rate 1.0 mL/min; column temperature 40 ˚C; UV detector 254 nm, retention time, 23.8min for (+)-isomer, 13.1 min for (–)-isomer.

**Complex 5b**




$[\alpha]_D^{30}$ –64.8 (*c* 0.040, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 1.27 (1H, d, *J* = 13.6 Hz), 1.96 (3H, s), 2.06-2.16 (1H, m), 3.60 (1H, t, *J* = 11.6 Hz), 3.75 (1H, t, *J* = 11.6 Hz), 3.93-4.13 (3H, m), 4.22 (1H, d, *J* = 6.8 Hz), 5.13 (1H, d, *J* = 6.0 Hz), 5.22 (1H, s), 5.57 (1H, t, *J* = 6.4 Hz), 5.71 (1H, d, *J* = 6.4 Hz), 6.37 (1H, d, *J* = 8.0 Hz), 6.90-7.03 (3H, m), 7.16 (2H, t, *J* = 7.6 Hz), 7.27 (1H, d, *J* = 7.6 Hz), 7.65 (1H, d, *J* = 8.0 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.3, 17.8, 22.8, 25.4, 31.7, 32.8, 67.7, 67.8, 87.7, 91.3, 93.2, 98.1, 116.6, 117.8, 122.3, 122.4, 126.4, 127.1, 127.8, 127.9, 232.2; IR ($CHCl_3$) 1955, 1871, 1597 $cm^{-1}$; MS (relative intensity) *m/z* 493 ($M^+$, 15), 409 (62), 298 (100); HRMS calcd for $C_{27}H_{23}NO_5Cr$, 493.0981. found 493.0976.

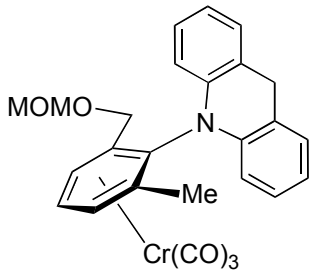
**Complex 6b**




$[\alpha]_D^{28}$ +23.7 (*c* 0.070, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 1.41 (1H, d, *J* = 12.4 Hz), 1.99 (3H, s), 2.21-2.27 (1H, m), 3.84 (1H, t, *J* = 12.4 Hz), 3.95 (1H, d, *J* = 19.6 Hz), 4.08 (1H, dd, *J* = 11.0, 4.0 Hz), 4.24-4.33 (3H, m), 4.68 (1H, d, *J* = 6.8 Hz), 5.00(1H, t,

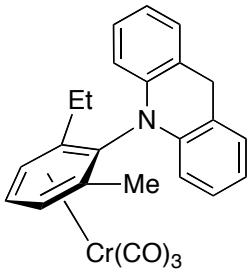
*J* = 6.0 Hz), 5.27 (1H, t, *J* = 6.8 Hz), 5.52 (1H, d, *J* = 6.0 Hz), 5.71 (1H, s), 6.26 (1H, d, *J* = 7.6 Hz), 6.98-7.02 (2H, m), 7.15 (1H, d, *J* = 8.4 Hz), 7.39 (1H, d, *J* = 6.8 Hz), 7.48 (1H, d, *J* = 7.6 Hz), 7.81 (1H, d, *J* = 7.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.8, 26.1, 30.5, 67.3, 67.5, 76.8, 81.2, 86.8, 93.1, 94.4, 98.1, 115.9, 122.9, 126.6, 127.6, 128.5, 130.0, 133.4, 234.4; IR ($CHCl_3$) 1954, 1869, 1638 cm$^{-1}$; MS (relative intensity) *m/z* 493 ($M^+$, 5), 409 (19), 357 (97), 298 (100); HRMS calcd for $C_{27}H_{23}NO_5Cr$, 493.0981. found 493.0944; HPLC condition; Chiralcel OD-H; hexane/2-propanol = 9/1; flow rate 1.0 mL/min; column temperature 40 ˚C; UV detector 254 nm, retention time, 15.9 min for (+)-isomer, 6.8 min for (–)-isomer.

**Complex 5c**



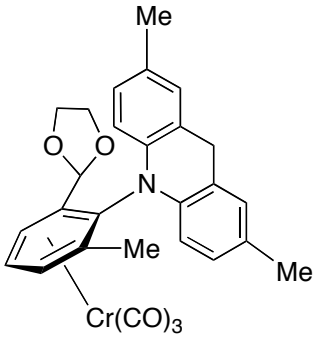

$^1$H NMR (400 MHz, $CDCl_3$) δ 2.08 (3H, s), 3.25 (3H, s), 4.07 (2H, s), 4.16 (1H, d, *J* = 4.0 Hz), 4.44 (1H, d, *J* = 4.0 Hz), 4.54 (1H, d, *J* = 6.8 Hz), 4.59 (1H, d, *J* = 6.8 Hz), 5.24 (1H, d, *J* = 5.6 Hz), 5.56 (1H, d, *J* = 5.6 Hz), 5.66 (1H, t, *J* = 6.0 Hz), 6.25 (1H, d, *J* = 7.6 Hz), 6.94-7.03 (3H, m), 7.17 (2H, d, *J* = 7.6 Hz), 7.27 (1H, d, *J* = 7.6 Hz), 7.72 (1H, d, *J* = 7.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.6, 32.6, 55.7, 64.6, 87.9, 90.8, 94.2, 96.7, 112.3, 112.5, 114.9, 117.4, 122.3, 122.4, 126.5, 127.1, 128.1, 128.5, 232.3; IR ($CHCl_3$) 2362, 2344, 1964, 1883 cm$^{-1}$; MS (relative intensity) *m/z* 481 ($M^+$, 6), 397 (25), 337 (60), 284 (100); HRMS calcd for $C_{26}H_{23}NO_5Cr$, 481.0981. found 481.0977.

**Complex 5d**



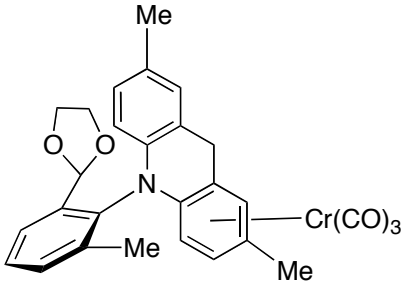

$^1$H NMR (400 MHz, $CDCl_3$) δ 0.74 (3H, t, *J* = 7.6 Hz), 1.25 (2H, s), 1.66 (3H, s), 1.88-2.16 (2H, m), 5.21-5.26 (2H, m), 5.92 (1H, t, *J* = 6.0 Hz), 7.26-7.30 (2H, m), 7.43 (1H, t, *J* = 8.0 Hz), 7.75 (2H, d, *J* = 8.0 Hz), 7.87 (1H, t, *J* = 8.0 Hz), 8.30 (2H, t, *J* = 5.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.3, 15.3, 19.4, 24.1, 27.8, 37.8, 60.7, 86.8, 87.4, 89.8, 97.4, 113.1, 113.2, 113.5, 120.5, 120.8, 128.1, 129.4, 131.5, 233.7; IR ($CHCl_3$) 3421, 1955, 1870 cm$^{-1}$; MS (relative intensity) *m/z* 435 ($M^+$, 8), 351 (36), 180 (100); HRMS calcd for $C_{25}H_{21}NO_3Cr$, 435.0927. found 435.0930.

**Complex 5e**



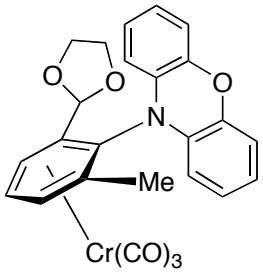

$[\alpha]_D^{24}$ –129.8 (*c* 0.25, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 1.99 (3H, s), 2.25 (3H, s), 2.30 (3H, s), 3.81-4.17 (6H, m), 4.69 (1H, dd, *J* = 5.6, 1.6 Hz), 5.54-5.59 (3H, m), 6.23 (1H, d, *J* = 8.0 Hz), 6.80 (1H, d, *J* = 8.0 Hz), 6.95 (2H, s), 7.07 (1H, d, *J* = 8.0 Hz), 7.60 (1H, d, *J* = 8.0 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.6, 20.6, 20.7, 32.7, 66.2, 66.3, 87.0, 91.4, 93.2, 100.1, 111.4, 111.5, 116.0, 117.6, 124.2, 125.6, 126.9, 127.4, 128.4, 128.7, 131.4, 131.5, 139.2, 140.6, 232.1; IR ($CHCl_3$) 3748, 2368, 1951, 1866 cm$^{-1}$; MS (relative intensity) *m/z* 507 ($M^+$, 7), 423 (34), 371 (96), 326 (100); HRMS calcd for $C_{28}H_{25}NO_5Cr$, 507.1138. found 507.1139.

**Complex 6e**




$[\alpha]_D^{25}$ +157.1 (*c* 0.014, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 2.12 (3H, s), 2.26 (3H, s), 2.36 (3H, s), 3.80-3.85 (2H, m), 3.93-4.05 (3H, m), 4.23 (1H, d, *J* = 19.6 Hz), 4.60 (1H, d, *J* = 6.8 Hz), 4.93 (1H, dd, *J* = 6.8, 1.6 Hz), 5.19 (1H, s), 5.58 (1H, s), 5.94 (1H, d, *J* = 8.8 Hz), 6.78 (1H, d, *J* = 8.0 Hz), 6.96 (1H, s), 7.43-7.47 (2H, m), 7.48-7.61 (1H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 18.7, 20.1, 20.7, 30.4, 65.4, 65.5, 81.4, 91.3, 93.0, 100.3, 114.4, 126.6, 128.4, 129.5, 133.3, 133.4, 235.2; IR ($CHCl_3$) 2889, 2377, 2347, 2305, 1951, 1873 $cm^{-1}$; MS (relative intensity) *m/z* 507 ($M^+$, 7), 423 (35), 371 (38), 326 (100); HRMS calcd for $C_{28}H_{25}NO_5Cr$, 507.1138. found 507.1130; HPLC condition; Chiralcel OD; hexane/2-propanol = 20/1; flow rate 1.0 mL/min; column temperature 40 ˚C; UV detector 254 nm, retention time, 14.3 min for (+)-isomer, 11.3 min for (–)-isomer.

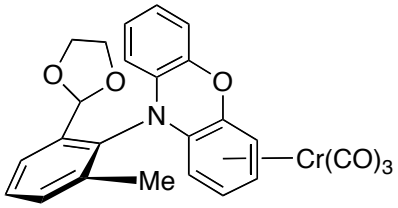
**Complex 5f**




$[\alpha]_D^{26}$ –217.97 (*c* 0.18, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 2.15 (3H, s), 3.93-4.17 (4H, m), 5.11 (1H, d, *J* = 5.6 Hz), 5.47 (1H, d, *J* = 5.6 Hz), 5.61 (1H, t, *J* = 5.6 Hz), 5.69 (1H, s), 6.10 (1H, d, *J* = 7.6 Hz), 6.70-6.87 (5H, m), 6.97 (1H, t, *J* = 7.6 Hz), 7.51 (1H, d, *J* = 7.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.7, 65.6, 66.3, 85.8, 90.5, 94.1, 99.9, 113.0, 115.5, 115.9, 116.5, 118.0, 121.5, 123.2, 123.3, 123.4, 123.6, 123.7, 231.7; IR ($CHCl_3$) 3654, 1970, 1893 $cm^{-1}$; MS (relative intensity) *m/z* 481 ($M^+$, 9), 397 (27), 345

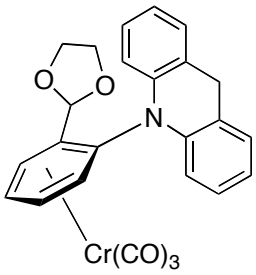
(100); HRMS calcd for $C_{25}H_{19}NO_6Cr$, 481.0617. found 481.0597.

**Complex 6f**



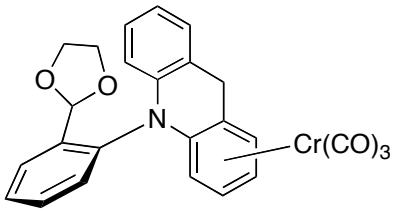

$[\alpha]_D^{26}$ +248.4 (*c* 0.12, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 2.20 (3H, s), 4.04-4.23 (4H, m), 4.47 (1H, d, *J* = 5.6 Hz), 4.89 (1H, s), 5.05 (1H, s), 5.26 (1H, d, *J* = 5.6 Hz), 5.93-5.98 (2H, m), 6.74-6.79 (3H, m), 7.46-7.49 (2H, m), 7.73 (1H, d, *J* = 5.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 17.8, 65.7, 65.8, 79.5, 80.2, 87.5, 88.4, 99.5, 114.8, 116.4, 123.5, 124.9, 126.9, 130.3, 134.0, 233.7; IR ($CHCl_3$) 2890, 1957, 1871 $cm^{-1}$; MS (relative intensity) *m/z* 481 ($M^+$, 2), 397 (6), 345 (100); HRMS calcd for $C_{25}H_{19}NO_6Cr$, 481.0617. found 481.0565; HPLC condition; Chiralcel OD; hexane/2-propanol = 9/1; flow rate 1.0 mL/min; column temperature 40 °C; UV detector 254 nm, retention time, 8.2 min for (+)-isomer, 7.0 min for (–)-isomer.

**Complex 5g**



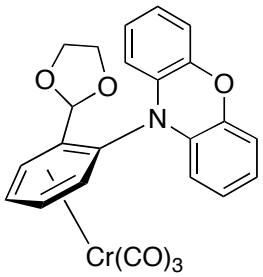

mp. 158 °C; $[\alpha]_D^{20}$ +16.8 (*c* 0.10, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 3.80-3.88 (2H, m), 3.95-4.10 (4H, m), 5.45-5.53 (4H, m), 5.71 (1H, d, *J* = 6.4 Hz), 6.98-7.05 (4H, m), 7.13-7.26 (4H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 33.2, 65.9, 66.1, 90.2, 90.3, 91.8, 96.1, 99.6, 110.5, 117.0, 118.0, 122.5, 126.2, 126.5, 127.7, 143.3, 231.6; IR ($CHCl_3$) 3006, 1978, 1907 $cm^{-1}$; MS (relative intensity) *m/z* 465 ($M^+$, 11), 381 (45), 284 (100); HRMS calcd for $C_{25}H_{19}NO_5Cr$, 465.0668. found 465.0671.

**Complex 6g**



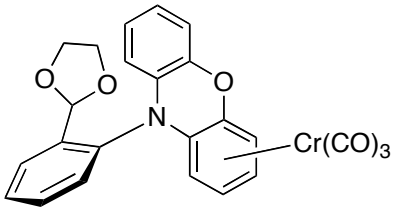

mp. 157 °C; $[\alpha]_D^{22}$ –335.2 (*c* 0.5, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 3.83-4.06 (5H, m), 4.24 (1H, d, *J* = 19 Hz), 4.46 (1H, d, *J* = 7.6 Hz), 4.98 (1H, t, *J* = 6.4 Hz), 5.28 (1H, t, *J* = 6.4 Hz), 5.51 (1H, d, *J* = 6.4 Hz), 5.72 (1H, s), 6.11-6.13 (1H, m), 6.97-6.99 (2H, m), 7.13-7.15 (1H, m), 7.58-7.61 (2H, m), 7.62 (1H, d, *J* = 7.6 Hz), 7.83 (1H, d, *J* = 7.6 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 30.7, 65.4, 65.6, 80.3, 86.1, 92.2, 94.1, 94.2, 99.9, 115.4, 118.7, 122.8, 124.9, 127.5, 128.6, 128.9, 129.8, 131.2, 131.8, 137.1, 137.5, 140.2, 234.4; MS (relative intensity) *m/z* 465 ($M^+$, 22), 381 (79), 284 (100); HRMS calcd for $C_{25}H_{19}NO_5Cr$, 465.0668. found 465.0667; HPLC condition; Chiralpak AD; hexane/2-propanol = 20/1; flow rate 1.0 mL/min; column temperature 40 °C; UV detector 254 nm, retention time, 15.1 min for (+)-isomer, 11.7 min for (–)-isomer.

**Complex 5h**



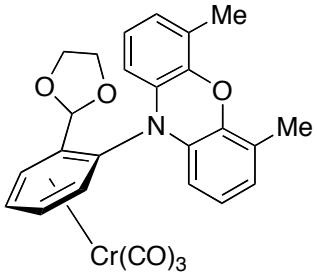

mp. 157 °C; $[\alpha]_D^{18}$ +118.0 (*c* 0.10, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 3.90-4.01 (2H, m), 4.07 (1H, q, *J* = 6.1 Hz), 4.16 (1H, q, *J* = 6.1 Hz), 5.27 (1H, t, *J* = 6.3 Hz), 5.49 (1H, t, *J* = 6.3 Hz), 5.58 (2H, t, *J* = 6.3 Hz), 5.68 (1H, s), 6.81-6.88 (8H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 66.2, 66.3, 88.0, 90.1, 91.8, 96.8, 99.5, 110.5, 116.0, 117.8, 118.1, 123.5, 123.6, 134.7, 147.0, 231.2; IR ($CHCl_3$) 1979, 1911, 1487 $cm^{-1}$; MS (relative intensity) *m/z* 467 ($M^+$, 19), 383 (100), 331 (48); HRMS calcd for $C_{24}H_{17}NO_6Cr$, 467.0461. found 467.0453.

**Complex 6h**



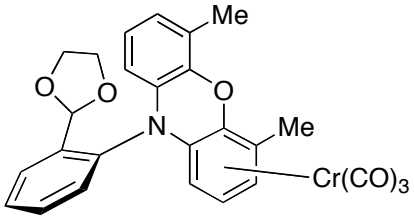

$[\alpha]_D^{22}$ –248 (*c* 0.10, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 3.95-4.13 (4H, m), 4.29 (1H, dd, *J* = 6.4, 0.8 Hz), 4.87 (1H, dt, *J* = 6.4, 0.8 Hz), 5.07 (1H, dt, *J* = 6.4, 0.8 Hz), 5.29 (1H, dd, *J* = 6.4, 0.8 Hz), 5.85 (1H, s), 5.93 (1H, dd, *J* = 7.6, 1.2 Hz), 6.71-6.81 (3H, m), 7.57-7.62 (1H, m), 7.65-7.73 (2H, m), 7.81 (1H, dd, *J* = 7.6, 1.2 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 65.6, 65.8, 79.8, 81.0, 87.8, 88.4, 100.2, 115.2, 116.4, 123.5, 124.8, 128.8, 130.2, 130.3, 132.4, 234.1; IR ($CHCl_3$) 3009, 2360, 1959, 1882 $cm^{-1}$; MS (relative intensity) *m/z* 467 ($M^+$, 28), 383 (91), 353 (51), 331 (41), 270 (100); HRMS calcd for $C_{24}H_{17}NO_6Cr$, 467.0461. found 467.0470; HPLC condition; Chiralcel OD; hexane/2-propanol = 9/1; flow rate 1.0 mL/min; column temperature 40 °C; UV detector 254 nm, retention time, 12.42 min for (+)-isomer, 21.51 min for (–)-isomer.

**Complex 5i**



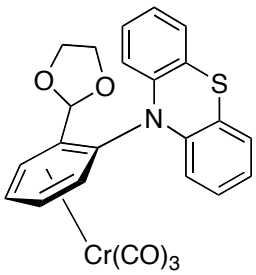

$[\alpha]_D^{22}$ +120 (*c* 0.42, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 2.29 (6H, s), 3.89-4.19 (4H, m), 5.27 (1H, t, *J* = 5.6 Hz), 5.47 (1H, t, *J* = 5.6 Hz), 5.55-5.60 (2H, m), 5.70 (1H, s), 6.67 (2H, d, *J* = 7.2 Hz), 6.74-6.79 (4H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) 15.5, 66.3, 88.2, 90.3, 91.6, 96.7, 99.5, 115.8, 122.6, 125.2, 125.5, 134.7, 231.4; IR ($CHCl_3$) 1972, 1898, 1637 $cm^{-1}$; MS (relative intensity) *m/z* 495 ($M^+$, 22), 411 (79), 359 (76), 298 (100); HRMS calcd for $C_{26}H_{21}NO_6Cr$, 495.0774. found 495.0776.

**Complex 6i**




$[\alpha]_D^{22}$ –234 (*c* 0.65, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 2.24 (3H, s), 2.32 (3H, s), 3.93-4.16 (5H, m), 4.95 (2H, d, *J* = 3.2 Hz), 5.76 (1H, d, *J* = 8.4 Hz), 5.86 (1H, s), 6.63 (1H, t, *J* = 8.0 Hz), 6.70 (1H, d, *J* = 8.0 Hz), 7.56-7.60 (1H, m), 7.68 (2H, d, *J* = 3.2 Hz), 7.79 (1H, d, *J* = 8.0 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 15.3, 15.6, 65.5, 65.7, 89.0, 89.4, 100.0, 112.7, 123.9, 125.3, 128.6, 130.1, 130.4, 132. 1, 234.6; IR ($CHCl_3$) 2348, 2307, 1952, 1862 $cm^{-1}$; MS (relative intensity) *m/z* 495 ($M^+$, 34), 411 (98), 298 (100); HRMS calcd for $C_{28}H_{21}NO_6Cr$, 495.0774. found 495.0770; HPLC condition; Chiralpak AS; hexane/2-propanol = 80/1; flow rate 1.0 mL/min; column temperature 40 ˚C; UV detector 254 nm, retention time, 17.71 min for (+)-isomer, 15.78 min for (–)-isomer.

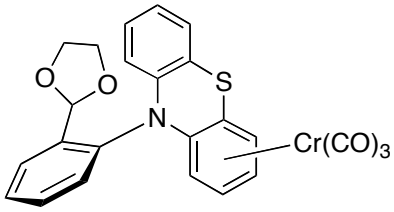
**Complex 5j**




mp. 92 °C; $[\alpha]_D^{22}$ –92.0 (*c* 0.10, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 3.82 (1H, s), 3.96 (2H, s), 4.05 (1H, s), 5.46 (2H, bs), 5.57 (1H, bs), 5.69 (1H, bs), 6.04 (1H, s), 6.99-7.06 (4H, m), 7.12-7.19 (4H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 65.9, 66.6, 89.1, 90.9, 91.3, 97.3, 100.0, 109.9, 120.2, 120.6, 124.6, 127.3, 127.4, 145.8, 231.4; IR ($CHCl_3$) 3585, 3059, 2362, 1979, 1913 $cm^{-1}$; MS (relative intensity) *m/z* 483 ($M^+$, 10),

399 (100), 371 (95); HRMS calcd for $C_{24}H_{17}NO_5SCr$, 483.0233. found 483.0233.

**Complex 6j**




mp. 147 °C; $[\alpha]_D^{18}$ +175.0 (*c* 0.12, $CHCl_3$); $^1$H NMR (400 MHz, $CDCl_3$) δ 3.94-4.11 (4H, m), 4.56 (1H, bs), 4.99 (1H, t, *J* = 6.4 Hz), 5.17 (1H, t, *J* = 6.4 Hz), 5.49 (1H, d, *J* = 6.4 Hz), 5.93 (1H, s), 6.19 (1H, bs), 6.93-6.95 (2H, m), 7.01-7.04 (1H, m), 7.58-7.67 (3H, m), 7.84 (1H, d, *J* = 6.8 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 65.6, 65.7, 82.1, 86.2, 92.5, 92.8, 100.2, 117.1, 122.6, 124.2, 127.0, 127.9, 129.6, 130.0, 131.3, 131.7, 142.0, 233.7; IR ($CHCl_3$) 3620, 2975, 2360, 1962, 1889 $cm^{-1}$; MS (relative intensity) *m/z* 483 ($M^+$, 21), 399 (92), 371 (100); HRMS calcd for $C_{24}H_{17}NO_5SCr$, 483.0233. found 483.0233.; HPLC condition; Chiralcel OD; hexane/2-propanol = 9/1; flow rate 1.0 mL/min; column temperature 40 °C; UV detector 254 nm, retention time, 12.85 min for (–)-isomer, 23.06 min for (+)-isomer.

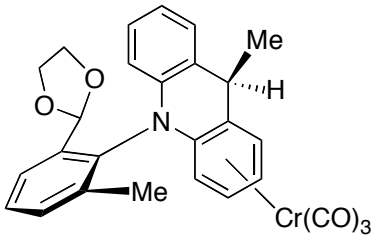
**Stereoselective synthesis of complex 8 from complex 6a.**

Complex **6a** (89 mg, 0.18mmol) and *N, N, N', N'*-tetramethylethylenediamine (26 mg, 0.22 mmol) were dissolved in THF (2.0 mL) under nitrogen atmosphere. The resulting solution was cooled to -78 °C and *n*-BuLi (0.14 mL, 1.6 N in hexane, 0.22 mmol) was added to the solution. The reaction mixture was stirred for 30 min at -78 °C and then gradually warmed to -60 °C. MeI (38 mg, 0.27 mmol) was added at -60 °C to the reaction mixture and gradually warmed to 0 °C. The reaction mixture was quenched with $H_2O$ and extracted with EtOAc. The organic layer was washed with brine and dried over anhydrous $MgSO_4$, and filtrate was concentrated under reduced pressure. The residue was purified by silica gel chromatography to give chromium complex **8** (75 mg, 0.15 mmol) in 85% yield as yellow amorphous.

**Stereoselective synthesis of complex 8 by a nucleophilic substitution reaction.**

Complex **9** (100 mg, 0.31 mmol), 9-methylacridane (73 mg, 0.37 mmol) and 18-crown-6 (122 mg, 0.46 mmol) were dissolved in toluene (2.0 mL) under nitrogen atmosphere. The solution was degassed by three freeze/vacuum/thaw cycles and heated to 85 °C. The suspension of NaH (18 mg, 0.46 mmol) and toluene (1.0 mL) was added to the solution, the resulting mixture was stirred at 85 °C for 1 h. The mixture was cooled to rt, and quenched with $H_2O$ and extracted with EtOAc. The organic layer was washed with brine and dried over anhydrous $MgSO_4$, and filtrate was concentrated under reduced pressure. The residue was purified by silica gel chromatography to give chromium complex **8** in 30 % yield as yellow amorphous.

**Complex 8**




$[\alpha]_D^{18}$ +371.8 (*c* 0.41, $CHCl_3$); $^1$H NMR (400 MHz, $C_6D_6$) δ 1.16 (3H, d, *J* = 7.2 Hz), 1.71 (3H, s), 3.42 (1H, q, *J* = 7.2 Hz), 3.54-4.00 (4H, m), 4.17 (1H, t, *J* = 6.0 Hz), 4.49 (1H, t, *J* = 6.0 Hz), 4.58 (1H, d, *J* = 7.0 Hz), 4.70 (1H, d, *J* = 6.0 Hz), 6.02 (1H, s), 6.46 (1H, *J* = 5.0 Hz), 6.80 (2H, t, *J* = 4.0 Hz), 6.88 (1H, t, *J* = 4.0 Hz), 7.01 (1H, d, *J* = 4.0 Hz), 7.18 (1H, m), 7.89 (1H, d, *J* = 7.2 Hz); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 18.1, 30.5, 36.2, 65.7, 81.7, 86.8, 92.4, 94.3, 99.5, 115.1, 123.2, 125.4, 126.9, 127.7, 128.7, 129.9, 134.0, 234.1; IR ($CHCl_3$) 2967, 2892, 1954, 1869 $cm^{-1}$; MS (relative intensity) *m/z* 493 ($M^+$, 13), 409 (83), 312 (100); HRMS calcd for $C_{27}H_{23}NO_5Cr$, 493.0981. found 493.0977.

## X-ray Crystallography

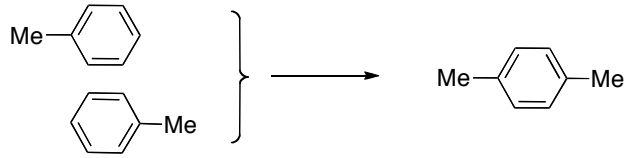
A crystal was mounted on a grass capillary with Paratone-N hydrocarbon oil and transferred to a Rigaku RAXIS Rapid diffractometer equipped with an imaging plate detector. The frame data were processed using the Rigaku PROCESS-AUTO program,[4] and the reflection data were corrected for absorption with an ABSCOR program.[5] The structures were solved by a direct method (SHELXS 97) and refinement on $F^2$ by full-matrix least-squares method by using SHELX97.[6]

**Complex 7 + 0.5($C_7H_8$)**

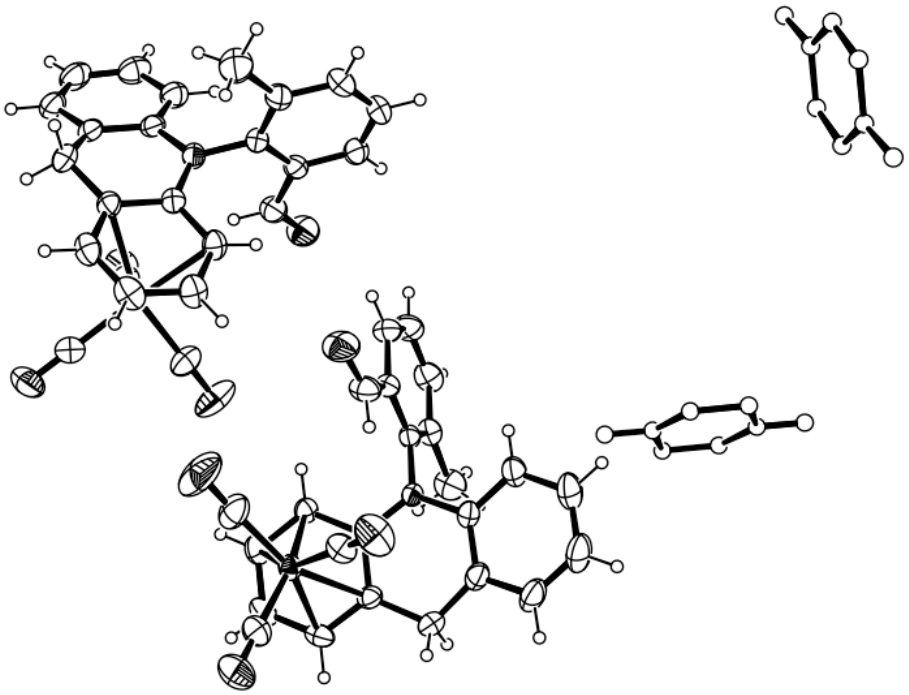
Crystallographic data: $C_{27.5}H_{23}CrNO_4$, $M$ = 483.48, triclinic, space group *P-1*, $a$ = 10.867(4), $b$ = 14.490(7), $c$ = 14.911(4), $V$ = 2293.2(1)$^3$, $Z$ = 4, Dc = 1.4000 g cm$^{-3}$ $\mu$(Mo K$\alpha$) = 0.71075 mm$^{-1}$, $F$(000) = 1004.00, $T$ = 296 K, 22335 reflections were corrected, 10174 unique ($R_{int}$ = 0.024), $R_1$ = 0.047 ($I>3\sigma(I)$), $wR_2$ = 0.145 ($I>3\sigma(I)$), $S$ = 1.03

***Special Remarks***

1. Two crystallographycally independent molecules are included in an asymmetric unit. These two molecules take almost same conformation.
2. Toluene is included as a crystal solvent. One toluene molecule exists in an asymmetric unit.
3. In an ortep diagram, the molecule looks like *p*-xylene is actually a toluene molecule partially overlapping two different orientations as shown below. This X-ray structure was analyzed on the basis of the assumption that a methyl group occupancy was 0.5 each.

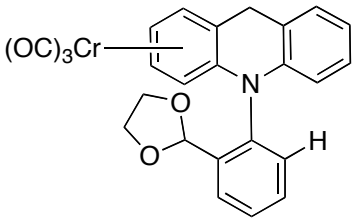



4. Level A alert appeared when our CIF file was checked by checkCIF, however it is due to the disorder of solvent molecules.

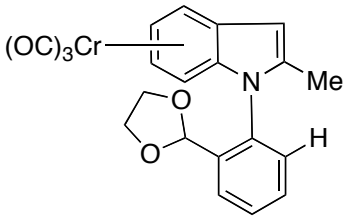


## Comparison of the rotational barrier for complex 6g and *N*-aryl indole P7

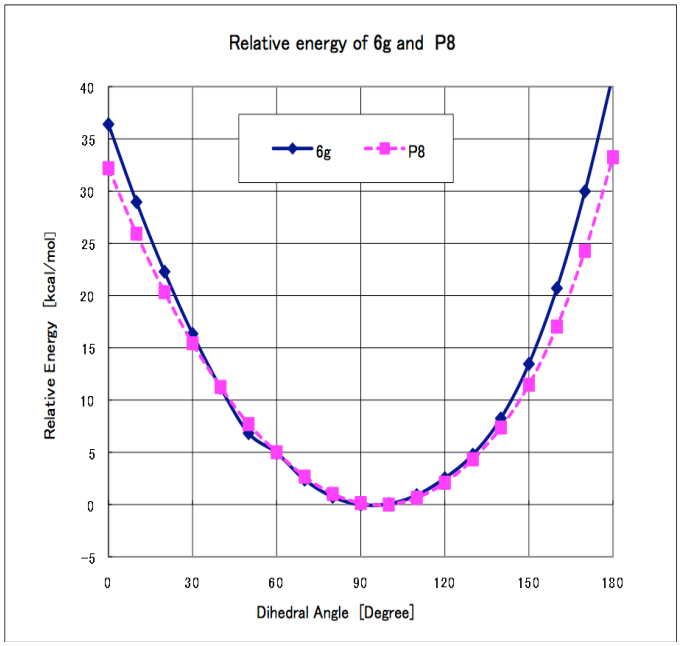
The relative energies for rotational barrier of *ortho*-trisubstituted chromium complexes **6g** and **P7** were carried out B3LYP/6-31(d, p) level calculations by using Gaussian 03 program.[7] Compared with the relative energies of these two complexes, it was revealed that **6g** have a higher rotational barrier than *N*-aryl indole complex **P7** with axial chiral N-C bond.




**6g**




**P7**



Cartesian coordinate for **6g**

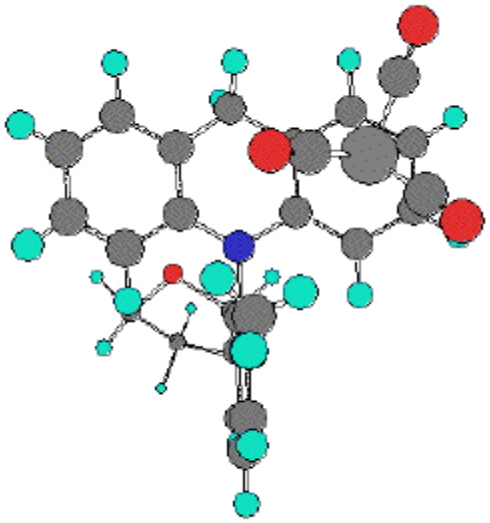
| | | | |
|---|---|---|---|
| C | -1.549808388582 | -0.875453666030 | 0.879805131071 |
| C | -2.649256765651 | -1.293530001744 | 0.089612204753 |
| C | -3.530049440086 | -2.267977138683 | 0.591663981007 |
| C | -3.328137219872 | -2.804572603647 | 1.875351975537 |
| C | -2.235987233936 | -2.383032577238 | 2.651032757828 |
| C | -1.322301388274 | -1.420021766515 | 2.166796991829 |
| C | -2.839705098262 | -0.723644211499 | -1.312367753652 |
| O | -3.127898080318 | 0.720433881719 | -1.308163883048 |
| O | -3.974542204635 | -1.365967255986 | -1.975121328293 |
| C | -4.906006399219 | -0.314320585682 | -2.429836138811 |
| C | -4.581301353244 | 0.863341910165 | -1.496563158650 |
| N | -0.646924794212 | 0.130041863555 | 0.337788997749 |
| C | 0.403856348379 | -0.273577641617 | -0.490534678149 |
| C | 1.170399761097 | 0.704156363151 | -1.228281287296 |
| C | 2.150085428573 | 0.264593214852 | -2.153021842860 |
| C | 2.432905566256 | -1.122408301740 | -2.352500631409 |
| C | 1.748258395038 | -2.068424490468 | -1.555751744305 |
| C | 0.753766200728 | -1.650292028989 | -0.619796880814 |
| C | -0.814984037487 | 1.507856622543 | 0.703633295950 |
| C | -1.726707181623 | 1.869919926993 | 1.718343604542 |
| C | -0.056471925336 | 2.510561026962 | 0.056187733051 |
| C | -1.864551334439 | 3.211445952383 | 2.105010893570 |
| C | -0.195145521569 | 3.846574904518 | 0.469421870823 |
| C | 0.821875285781 | 2.174579661440 | -1.134496965752 |
| C | -1.088807506731 | 4.211177146205 | 1.490157311935 |
| C | -0.126879246782 | -1.013896341427 | 3.006350661085 |
| H | -4.350099085445 | -2.600941620202 | -0.034401357869 |
| H | -4.012672507275 | -3.554518844772 | 2.263976036930 |
| H | -2.076644173315 | -2.807323613661 | 3.639569370023 |
| H | -1.940787456513 | -0.844377826173 | -1.926097306731 |

| | | | |
|---|---|---|---|
| H | -4.699459520552 | -0.071289001828 | -3.479433837893 |
| H | -5.920601024367 | -0.706708138894 | -2.322726385073 |
| H | -5.102223463746 | 0.779933454337 | -0.533648567812 |
| H | -4.753331987939 | 1.847053825592 | -1.937683638139 |
| H | 2.688891009384 | 1.009128934621 | -2.729617771435 |
| H | 3.182076354819 | -1.433953302606 | -3.069593174232 |
| H | 1.975852487326 | -3.124522269754 | -1.644419477936 |
| H | 0.232871308144 | -2.395157613208 | -0.033492246514 |
| H | -2.332411467598 | 1.110968413780 | 2.200565314288 |
| H | -2.570364999227 | 3.470605635464 | 2.890333935106 |
| H | 0.401036488986 | 4.611603788155 | -0.025203644453 |
| H | 0.284165014614 | 2.457798271832 | -2.055029835287 |
| H | 1.738872788874 | 2.776519428378 | -1.113694380561 |
| H | -1.180230377858 | 5.249628308925 | 1.796323050150 |
| H | -0.010518209822 | 0.073168163428 | 3.064981882437 |
| H | 0.805496309317 | -1.407542065857 | 2.582544535948 |
| H | -0.218903198108 | -1.403945338317 | 4.025460528114 |
| C | 4.493394808225 | 0.054169489278 | -0.447950588696 |
| O | 5.584155714968 | 0.477139241894 | -0.672003777210 |
| C | 3.521268093721 | -1.989077846526 | 0.779491283786 |
| O | 3.967884205589 | -2.924398304954 | 1.364362046657 |
| C | 2.755787425740 | 0.405908278001 | 1.391729497244 |
| O | 2.729728560579 | 1.054805326867 | 2.389693819978 |
| Cr | 2.812908035885 | -0.579694637025 | -0.144939428510 |

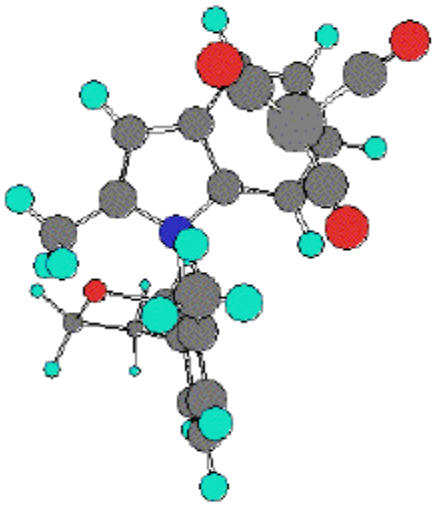
Cartesian coordinate for **P7**

| | | | |
|---|---|---|---|
| C | -1.544150453372 | -0.798754556204 | 1.061765863745 |
| C | -2.690692115695 | -1.220851703039 | 0.342377626919 |
| C | -3.616083369476 | -2.079052165263 | 0.957686300767 |
| C | -3.410356822090 | -2.494846878083 | 2.285450210852 |
| C | -2.288380167938 | -2.046751125487 | 2.998391359674 |
| C | -1.332300373272 | -1.190699917616 | 2.404350580947 |
| C | -2.934869110793 | -0.704243324346 | -1.060641612091 |
| O | -3.509085818411 | 0.674395208687 | -1.022548639684 |
| O | -3.914037568282 | -1.525073429355 | -1.747412039031 |
| C | -4.697192256389 | -0.620486382983 | -2.604489402650 |
| C | -4.797848537222 | 0.650648392995 | -1.742493632489 |
| N | -0.580202044522 | 0.055186971712 | 0.402313304192 |
| C | 0.453687417475 | -0.390948137070 | -0.426151568502 |
| C | 1.172785979334 | 0.767688305734 | -0.886427912031 |
| C | 2.268953466774 | 0.590902295192 | -1.792835062076 |
| C | 2.530632567737 | -0.703447351217 | -2.308740431788 |
| C | 1.785221858055 | -1.847069801445 | -1.862256518015 |
| C | 0.786184256924 | -1.705967357804 | -0.863287646485 |
| C | -0.522853840132 | 1.462091250487 | 0.477376806461 |
| C | -1.530015441783 | 2.239635475495 | 1.266386533527 |
| C | 0.544032111041 | 1.913176233309 | -0.285593044077 |
| C | -0.139777268433 | -0.724824019241 | 3.218157225776 |
| H | -4.474970682317 | -2.417572964048 | 0.388935371702 |
| H | -4.123217041146 | -3.163851731325 | 2.760912502759 |
| H | -2.136784649640 | -2.364973863138 | 4.027618419329 |
| H | -2.015598946049 | -0.629200351220 | -1.653465243668 |
| H | -4.161019613916 | -0.434088657107 | -3.544888346212 |
| H | -5.652993489141 | -1.109526022026 | -2.802484229670 |
| H | -5.621602347122 | 0.589945566936 | -1.020729035448 |
| H | -4.872368263069 | 1.572580732136 | -2.325699295994 |
| H | 2.850396852888 | 1.442139205745 | -2.126877649589 |

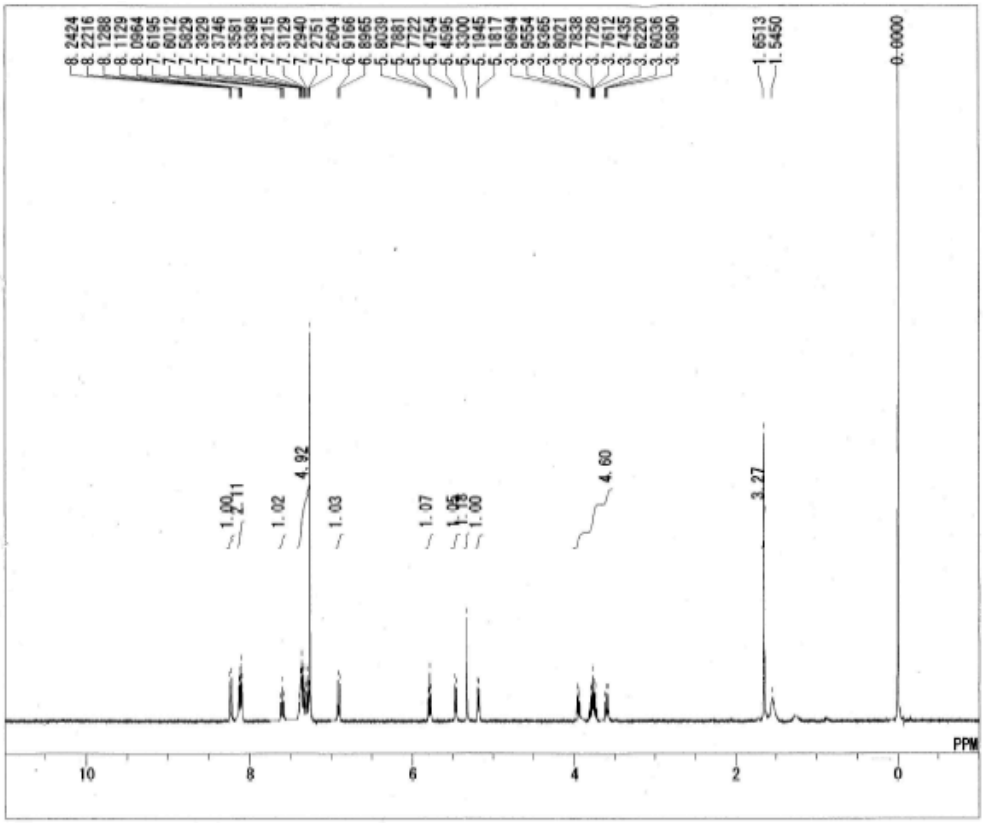
| | | | |
|---|---|---|---|
| H | 3.328643884488 | -0.841559090781 | -3.029074103949 |
| H | 2.022286343651 | -2.830109133401 | -2.250908723248 |
| H | 0.246308667857 | -2.569948725071 | -0.493716529428 |
| H | -1.309781853473 | 3.308970300755 | 1.198731823719 |
| H | -1.523869581309 | 1.955681248023 | 2.326753356181 |
| H | -2.538886491349 | 2.062697004769 | 0.873622704885 |
| H | 0.835964625355 | 2.945653273681 | -0.406822107662 |
| H | 0.534868611162 | -0.085323025959 | 2.644739202742 |
| H | 0.447025206113 | -1.580199469112 | 3.572837601663 |
| H | -0.474867466424 | -0.164924498232 | 4.101741236927 |
| C | 3.549410091177 | 0.416670671650 | 0.999007613722 |
| O | 4.012439754496 | 1.288652961202 | 1.664129457616 |
| C | 4.452724093101 | -1.615207790241 | -0.323301062560 |
| O | 5.526122698307 | -2.084911985728 | -0.539566798163 |
| C | 2.628470591898 | -1.971976477253 | 1.414867616172 |
| O | 2.507330621321 | -2.700771507902 | 2.352332025915 |
| C | 2.808643913611 | -0.892964656809 | -0.039242111678 |



Molecular structure of **6g**



Molecular structure of **P7**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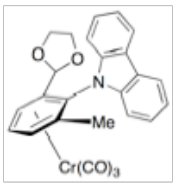
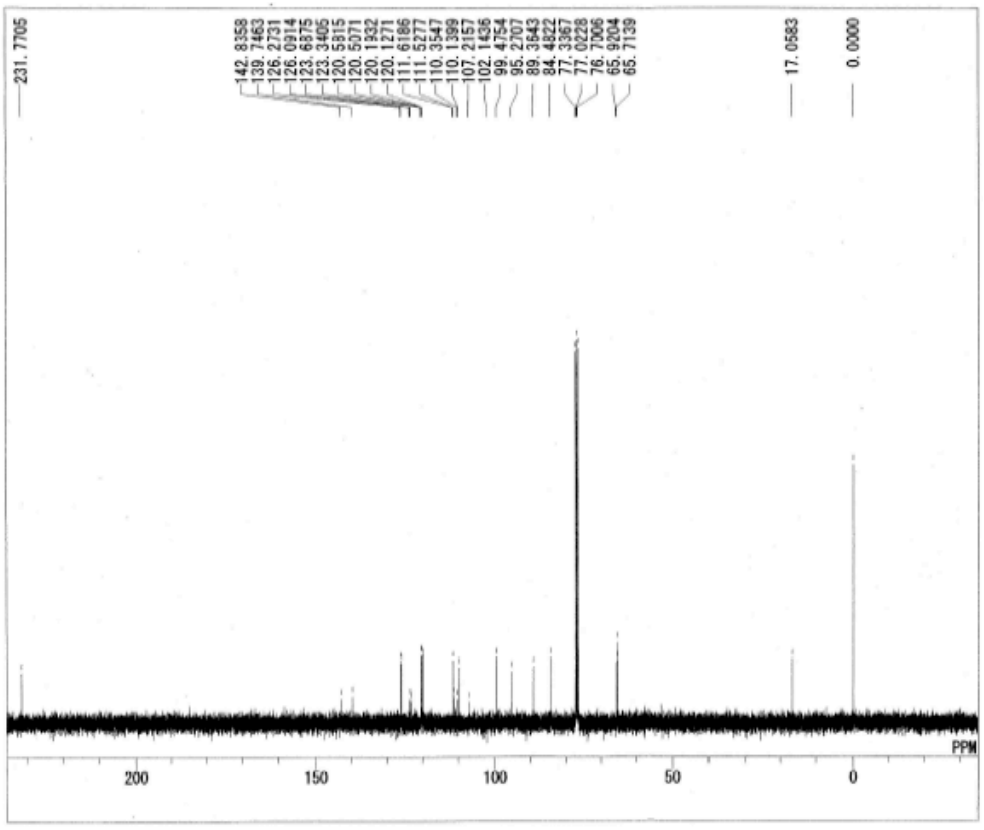
7.3581
7.3398
7.3215
7.3129
7.2940
7.2751
7.2694
7.2166
6.8965
6.8039
5.7881
5.7722
5.4754
5.4595
5.3300
5.1945
5.1817
3.9694
3.9554
3.9365
3.8021
3.7838
3.7728
3.7612
3.7435
3.6220
3.6036
3.5890
1.6513
1.5450
0.0000
1.00
2.11
1.02
4.92
1.03
1.07
1.05
1.00
4.60
3.27
PPM
10
8
6
4
2
0


O
O
N
Me
Cr(CO)3

**3**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139.7463
126.2731
126.0914
123.6875
123.3405
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120.5071
120.1932
120.1271
111.6186
111.5277
110.3547
110.1399
107.2157
102.1436
99.4754
95.2707
89.3643
84.4822
77.3367
77.0228
76.7006
65.9204
65.7139
17.0583
0.0000
PPM
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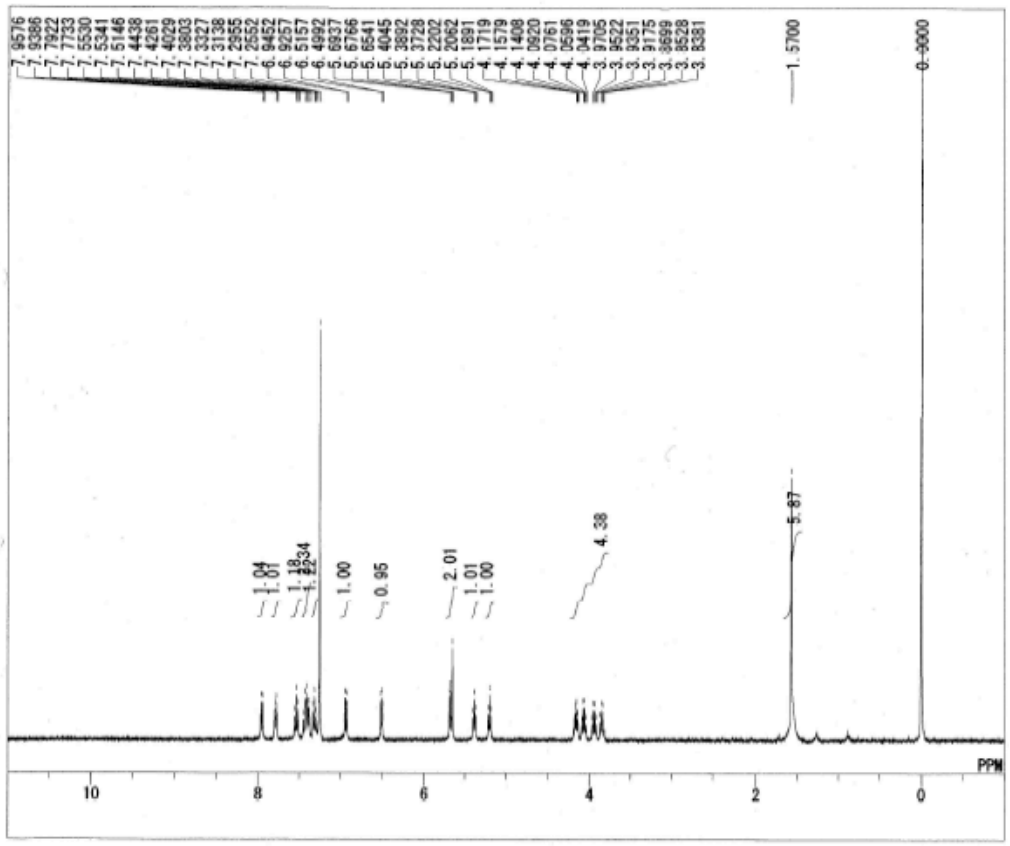
150
100
50
0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7.9386
7.7922
7.7733
7.5530
7.5341
7.5146
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7.4261
7.4029
7.3803
7.3327
7.3138
7.2855
7.2552
6.9452
6.9257
6.5157
6.4992
5.6837
5.6766
5.6541
5.4045
5.3892
5.3728
5.2202
5.2062
5.1891
4.1719
4.1579
4.1408
4.0920
4.0761
4.0596
4.0419
3.9705
3.9522
3.9351
3.9175
3.8699
3.8528
3.8381
1.5700
0.0000
1.04
1.07
1.18
1.22
1.34
1.00
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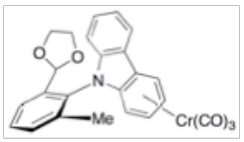
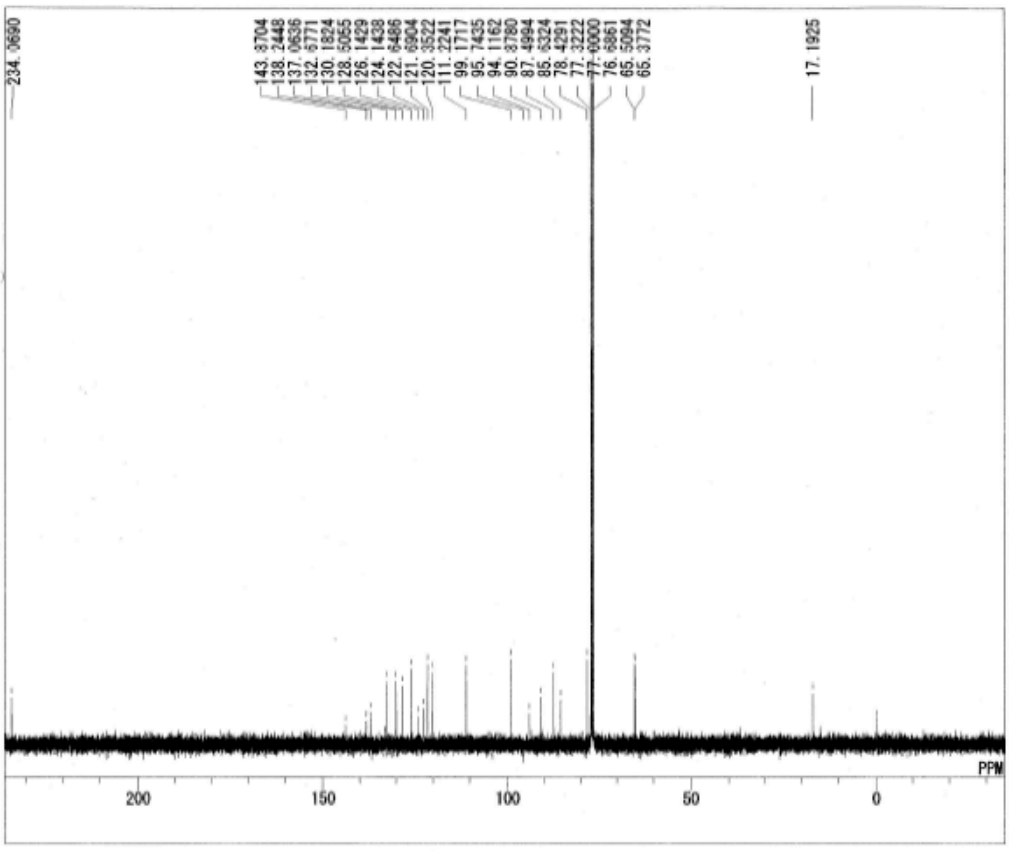
2.01
1.01
1.00
4.38
5.87
PPM
10
8
6
4
2
0


O
O
N
Me
Cr(CO)3

4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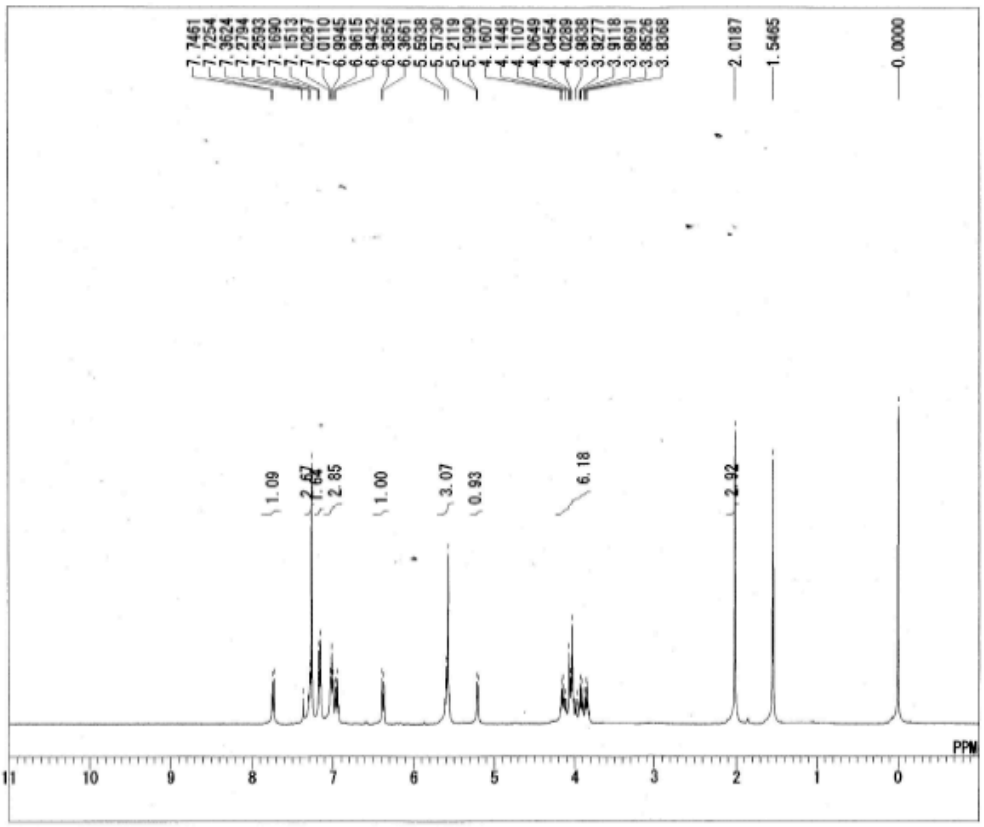
124.1438
122.6486
121.6904
120.3522
111.2241
99.1717
95.7435
94.1162
90.8780
87.4984
85.6324
78.4291
77.3222
77.0000
76.6861
65.5094
65.3772
17.1925
PPM
200
150
100
50
0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7.7254
7.3624
7.2794
7.2593
7.1690
7.1513
7.0287
7.0110
6.9945
6.9615
6.9432
6.3856
6.3661
5.5938
5.5730
5.2119
5.1990
4.1607
4.1448
4.1107
4.0649
4.0454
4.0289
3.9838
3.9277
3.9118
3.8691
3.8526
3.8368
2.0187
1.5465
0.0000
1.09
2.67
2.64
2.65
1.00
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2.92
PPM
11
10
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7
6
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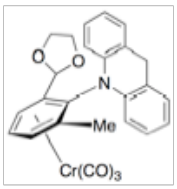
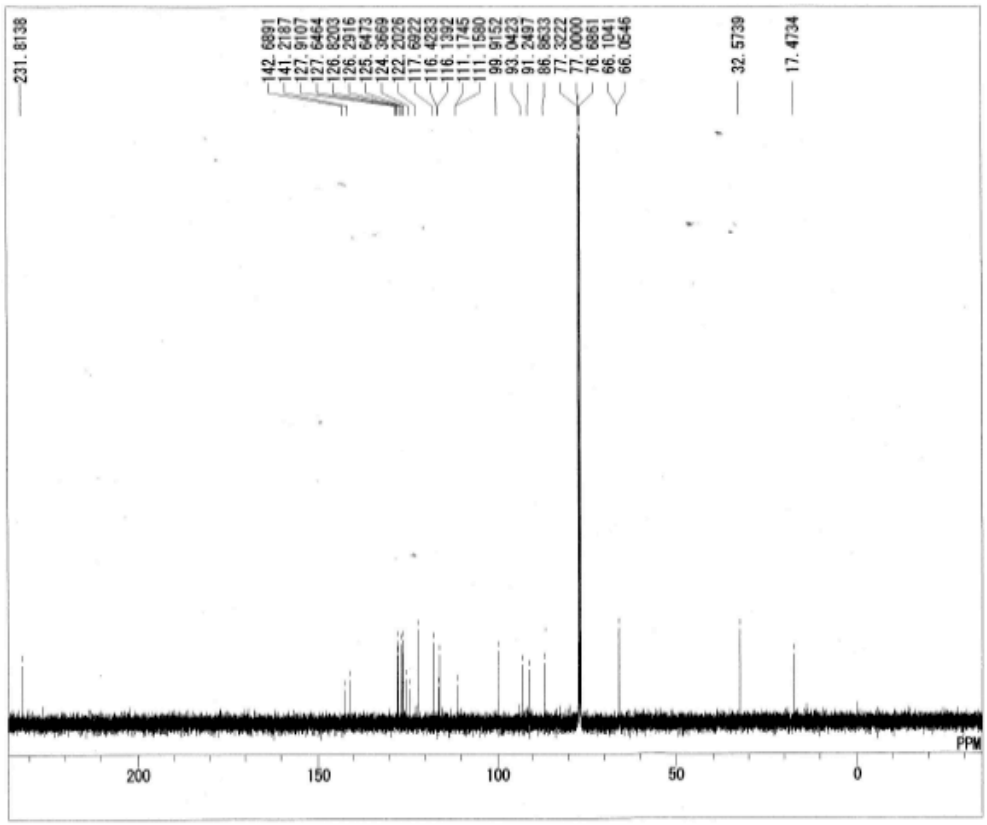
4
3
2
1
0


O
O
N
Me
Cr(CO)3

**5a**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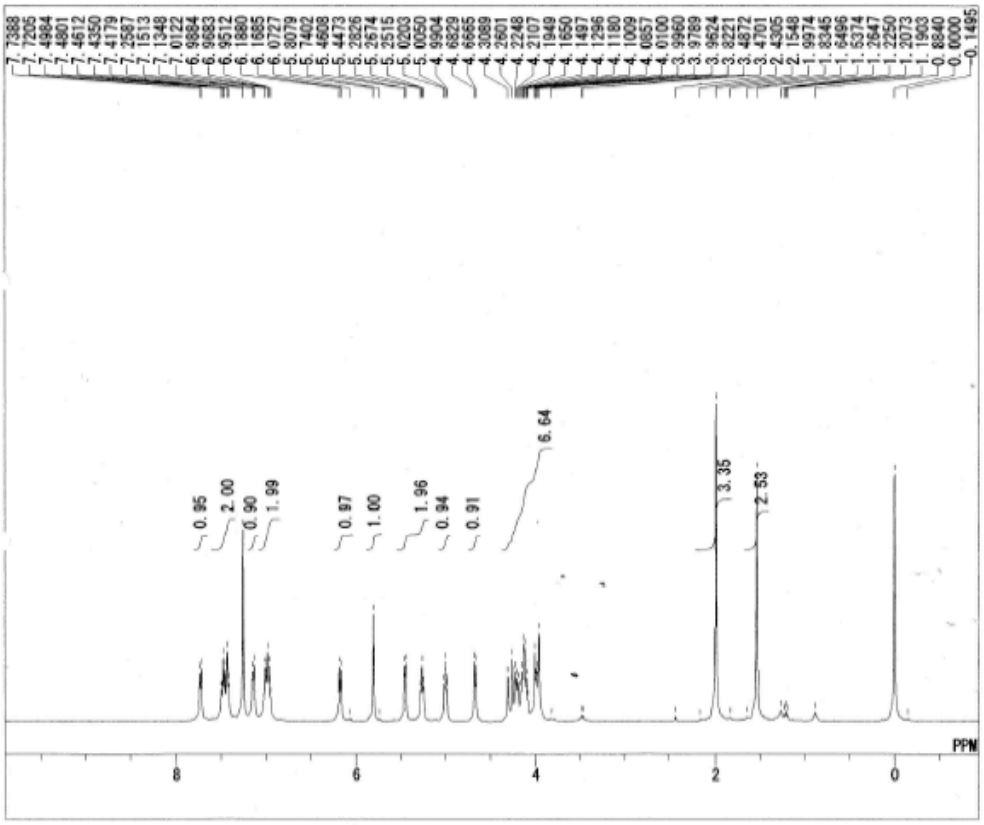
127.6464
126.8203
126.2916
125.6473
124.3669
122.2026
117.6922
116.4283
116.1392
111.1745
111.1580
99.9152
93.0423
91.2497
86.8633
77.3222
77.0000
76.6861
66.1041
66.0546
32.5739
17.4734
PPM
200
150
100
50
0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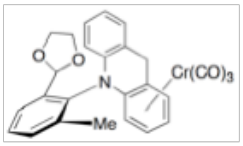
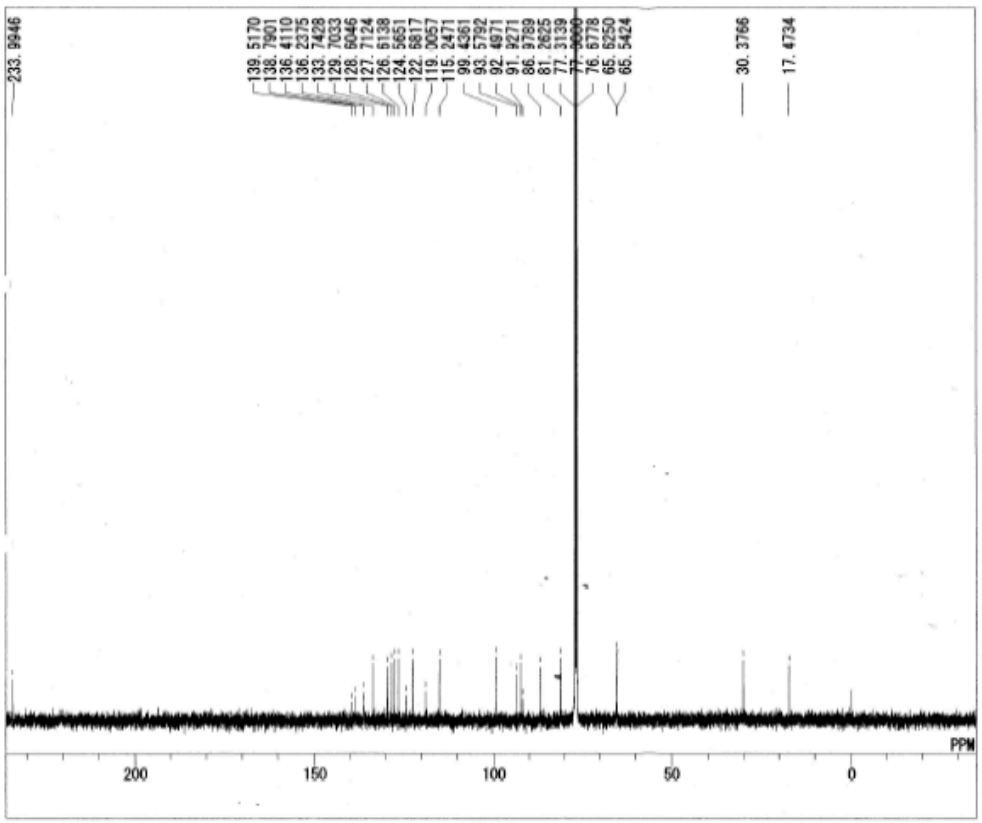
2.00
0.90
1.99
0.97
1.00
1.96
0.94
0.91
6.64
3.35
2.53
PPM
8
6
4
2
0


O
O
N
Cr(CO)3
Me

**6a**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138.7901
136.4110
136.2375
133.7428
129.7033
128.6046
127.7124
126.6138
124.5651
122.5817
119.0057
115.2471
99.4361
93.5792
92.4971
91.9271
86.9789
81.2625
77.3139
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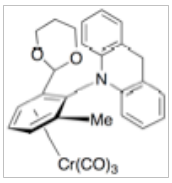
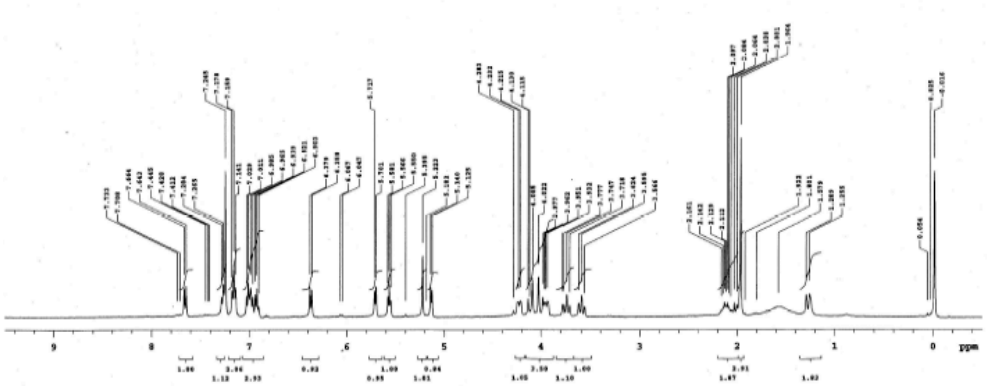
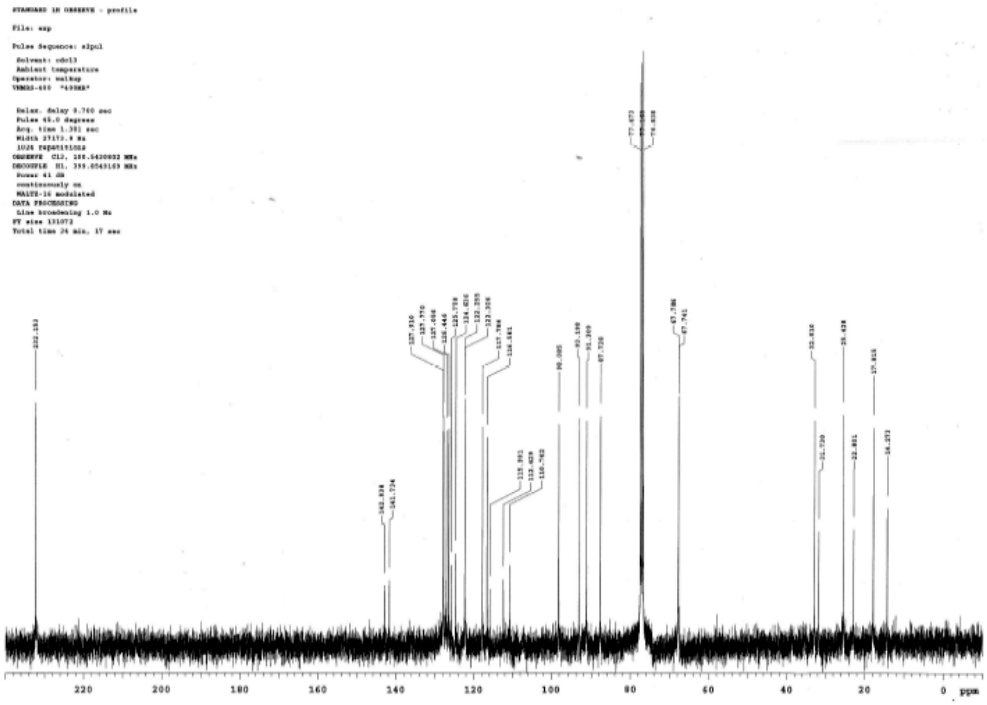
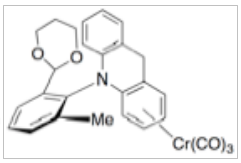
76.6778
65.6250
65.5424
30.3766
17.4734
PPM
200
150
100
50
0



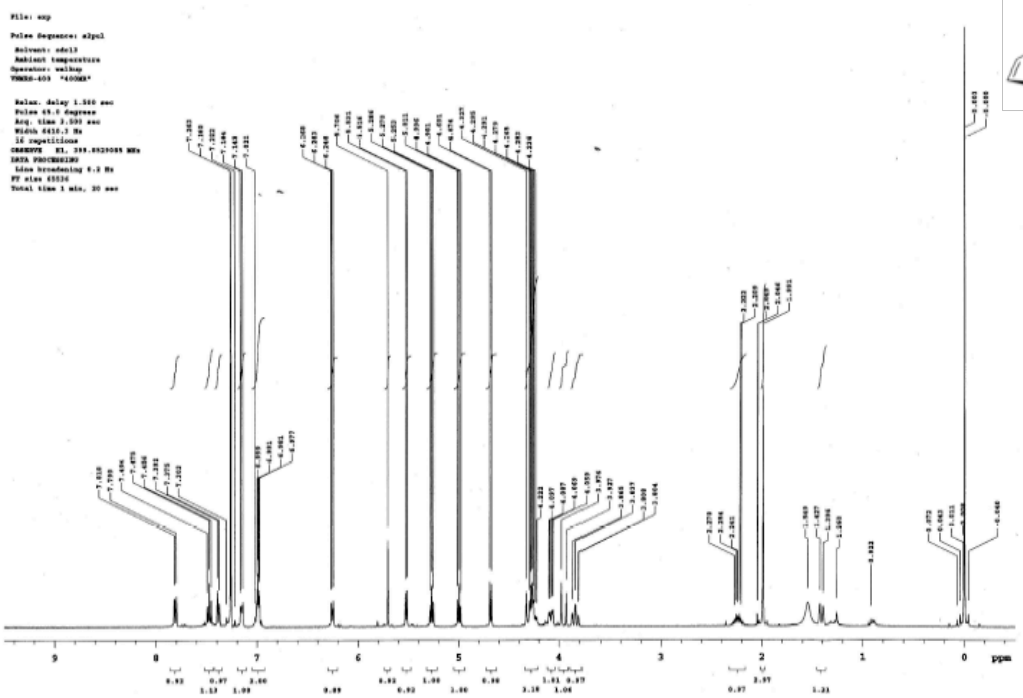

**5b**

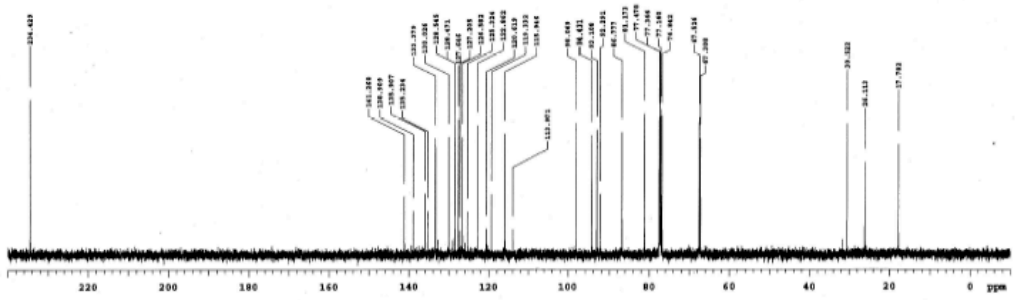


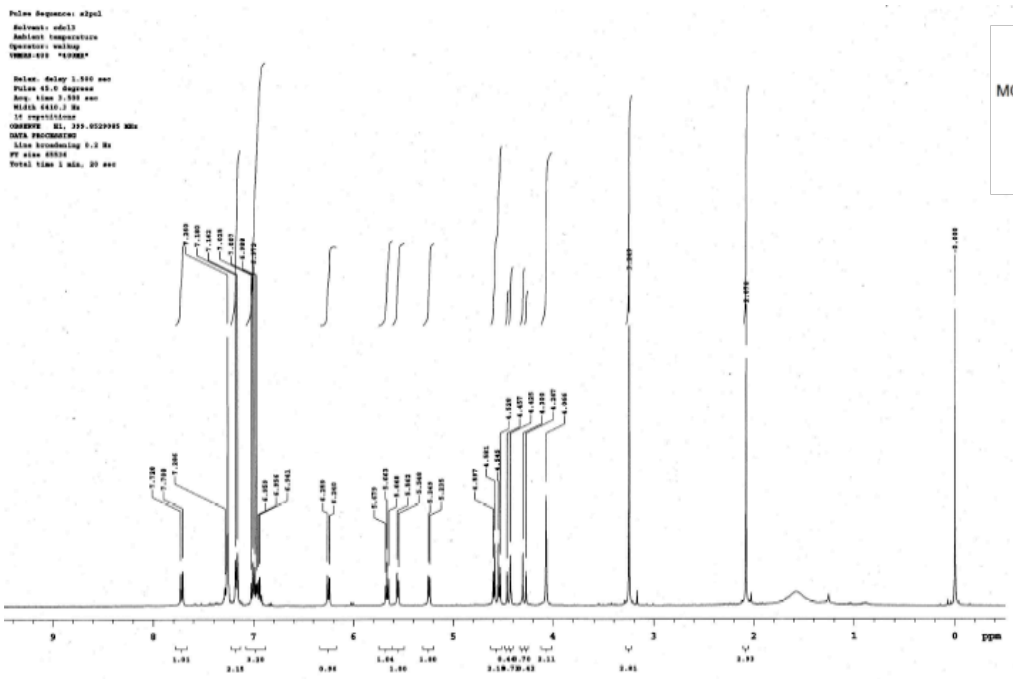





6b

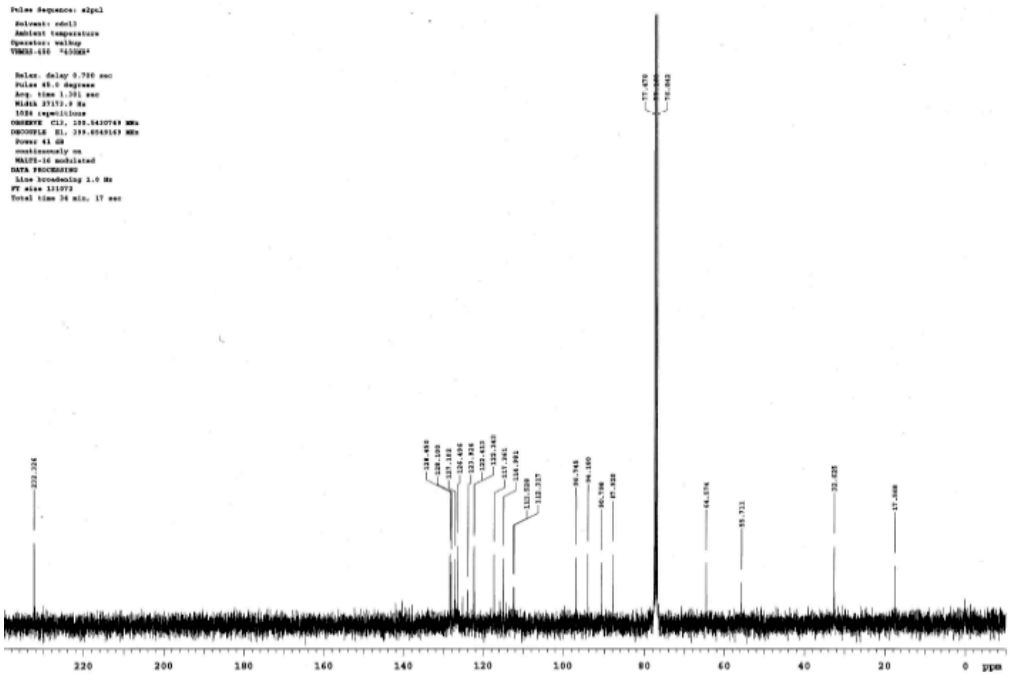


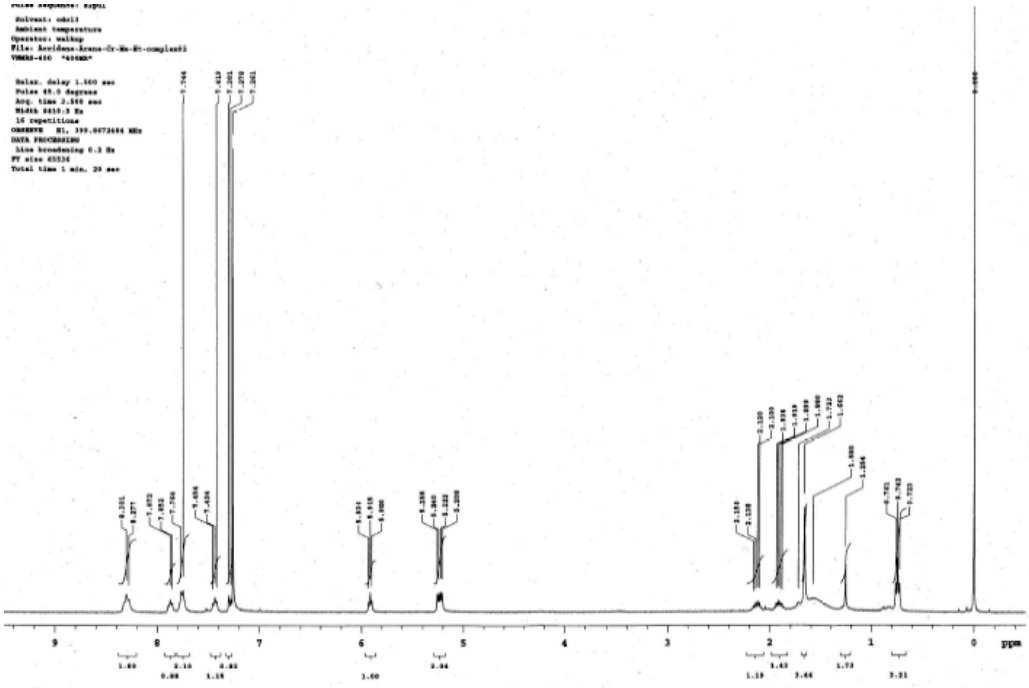


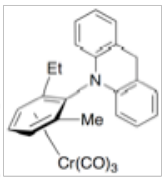



5c

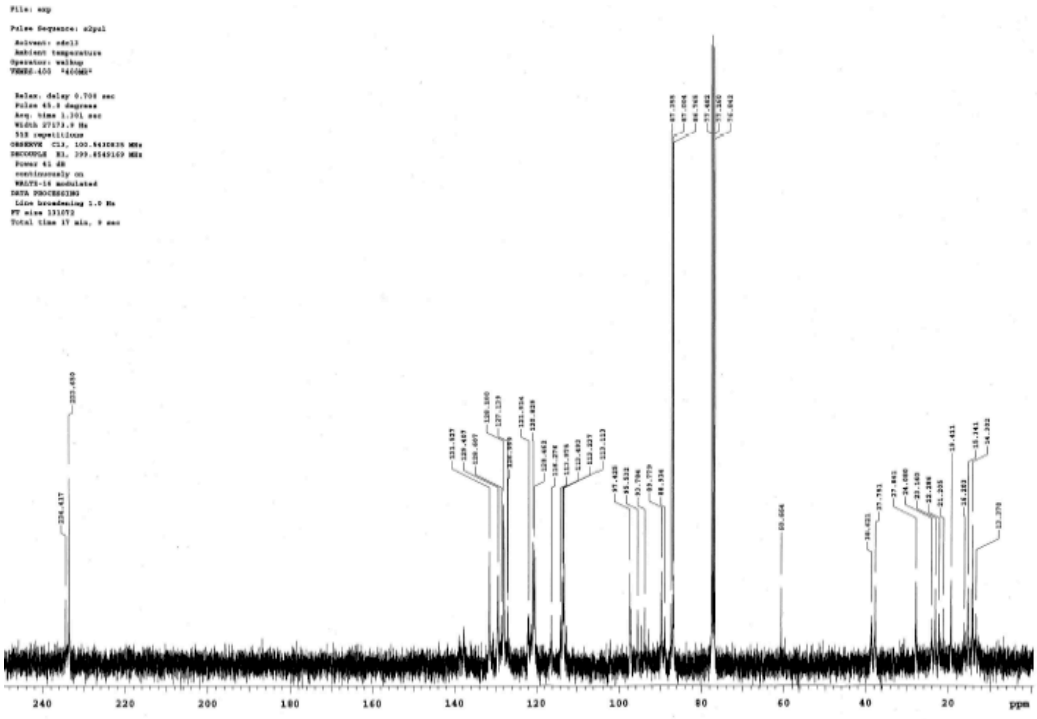


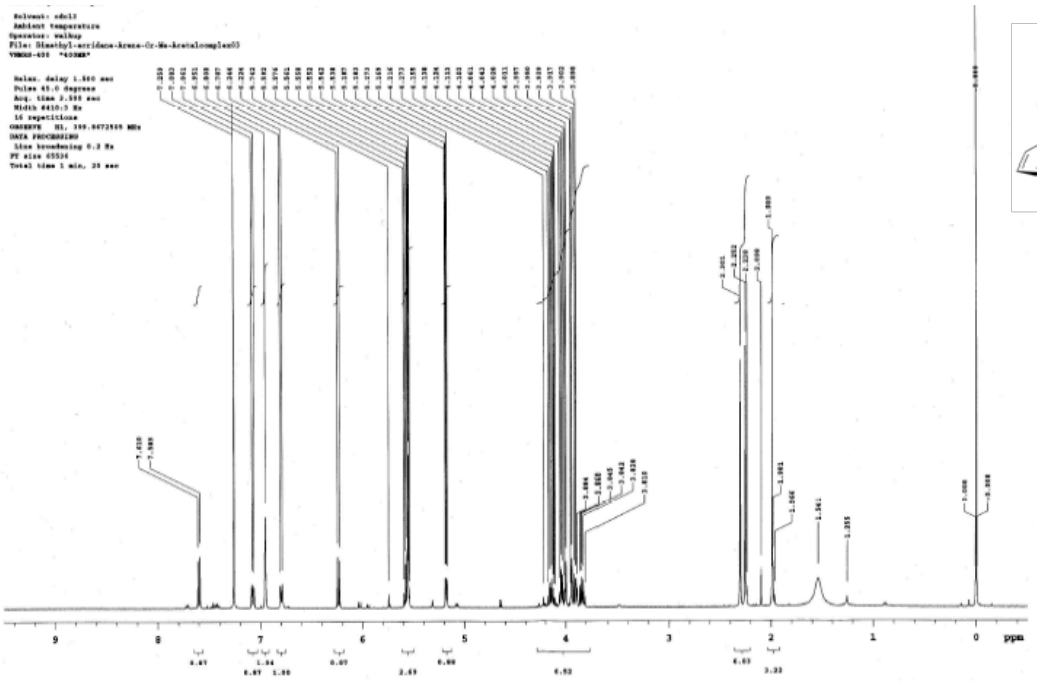


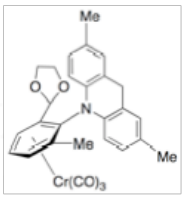


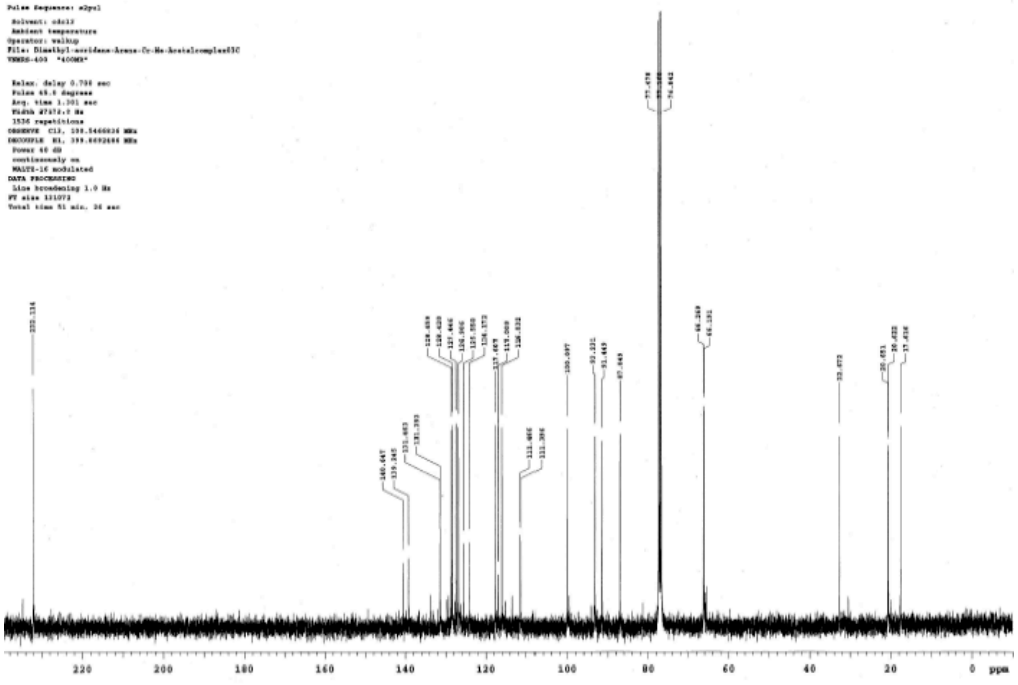

**5d**

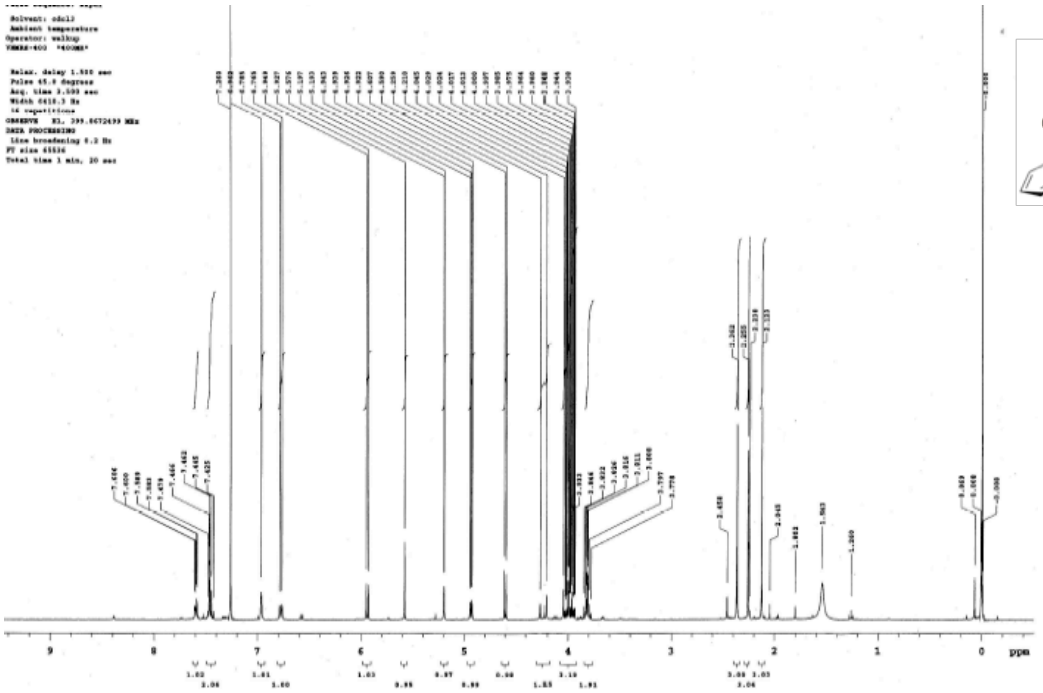


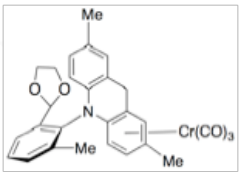




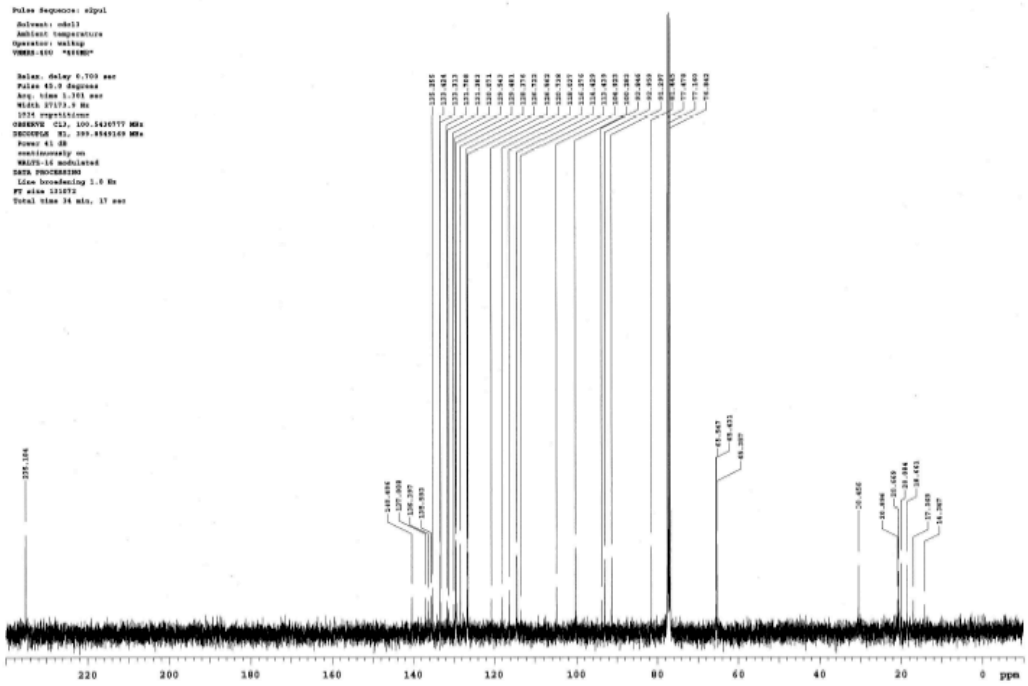

**5e**

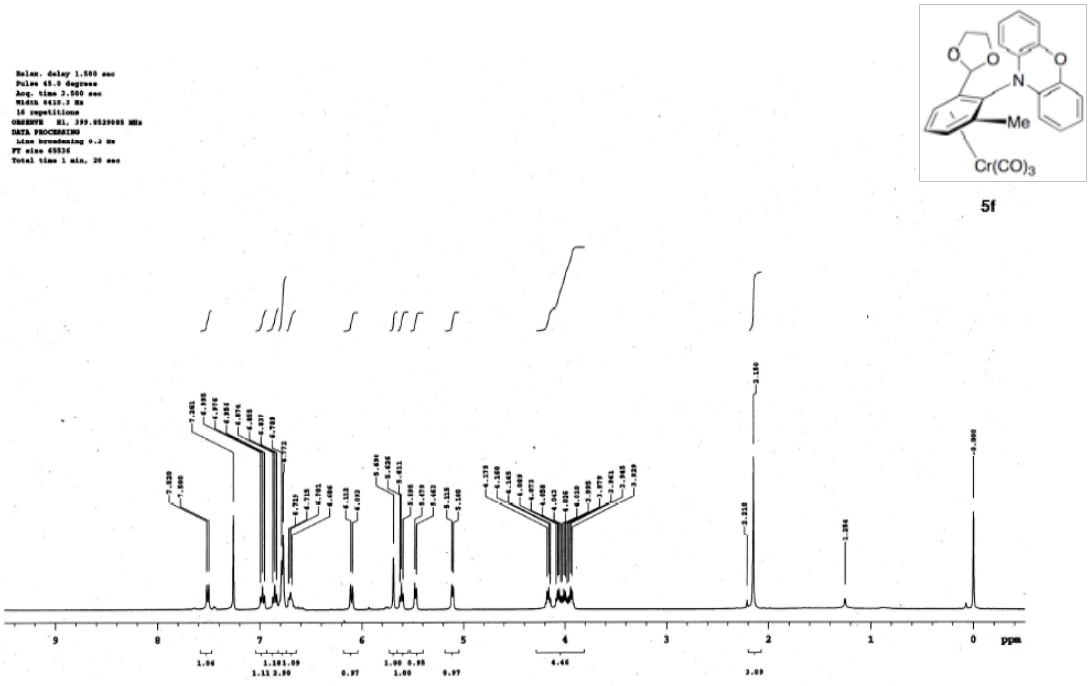


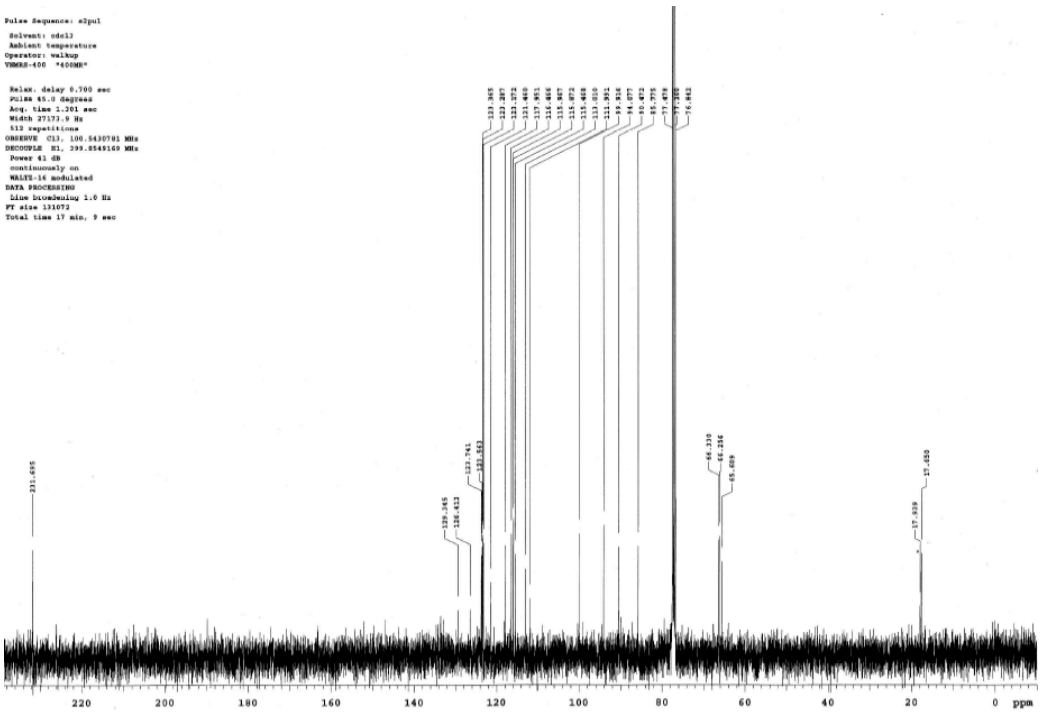


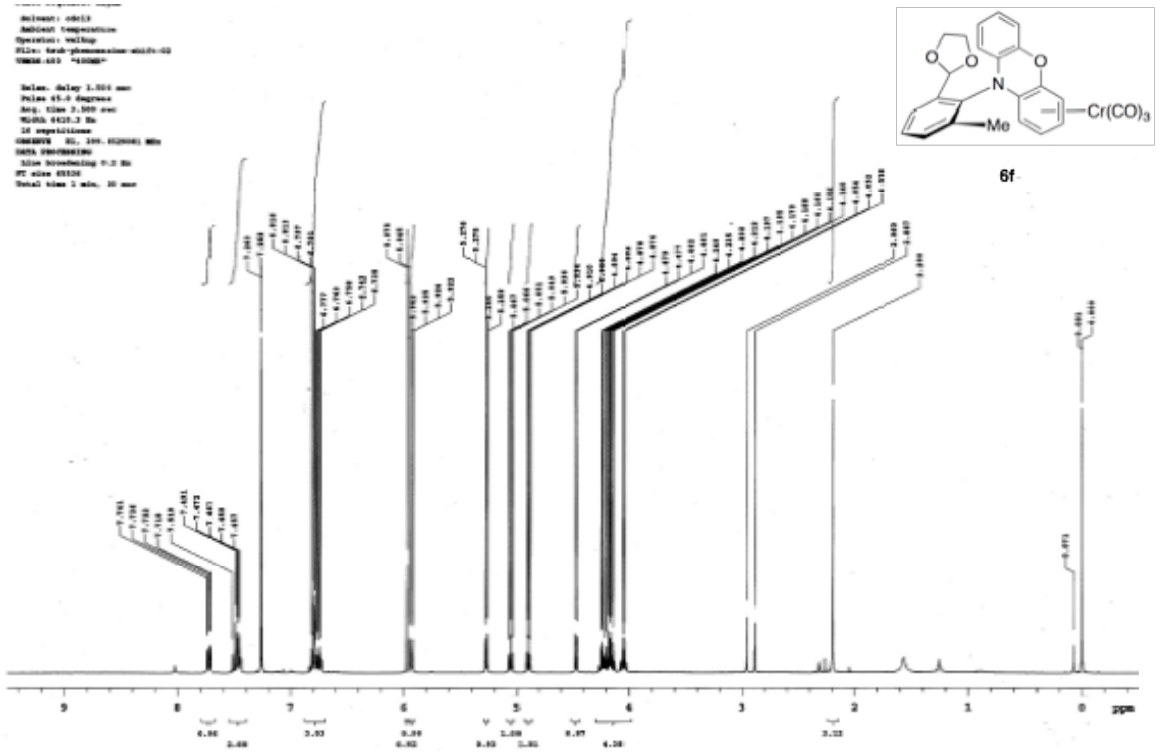


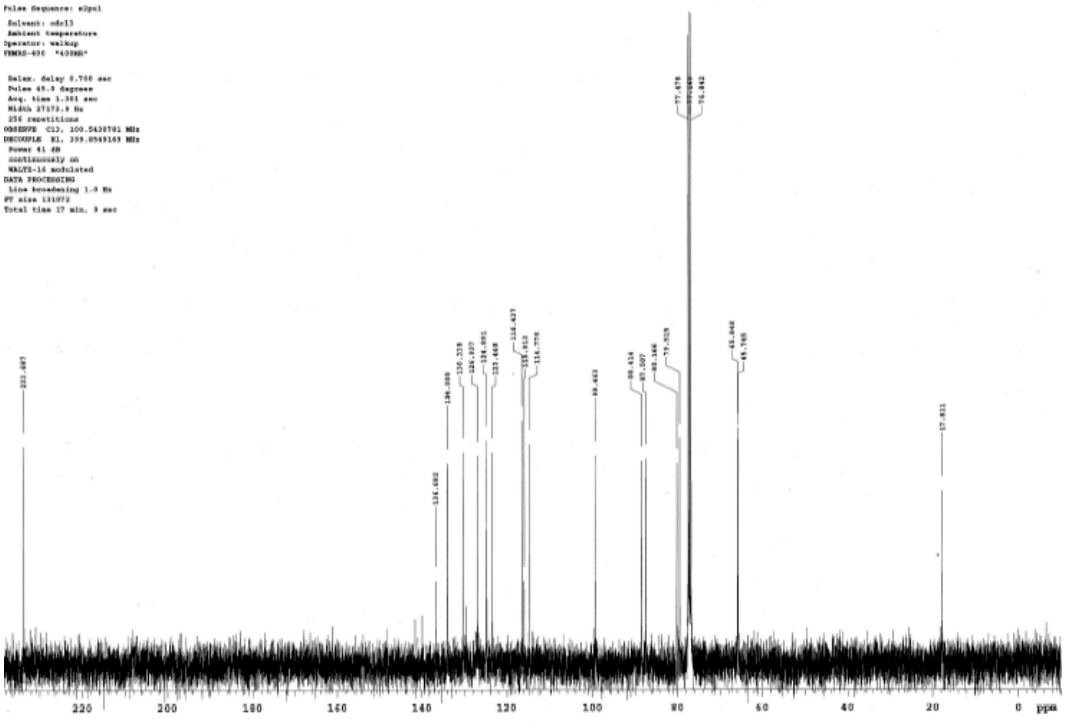

**6e**

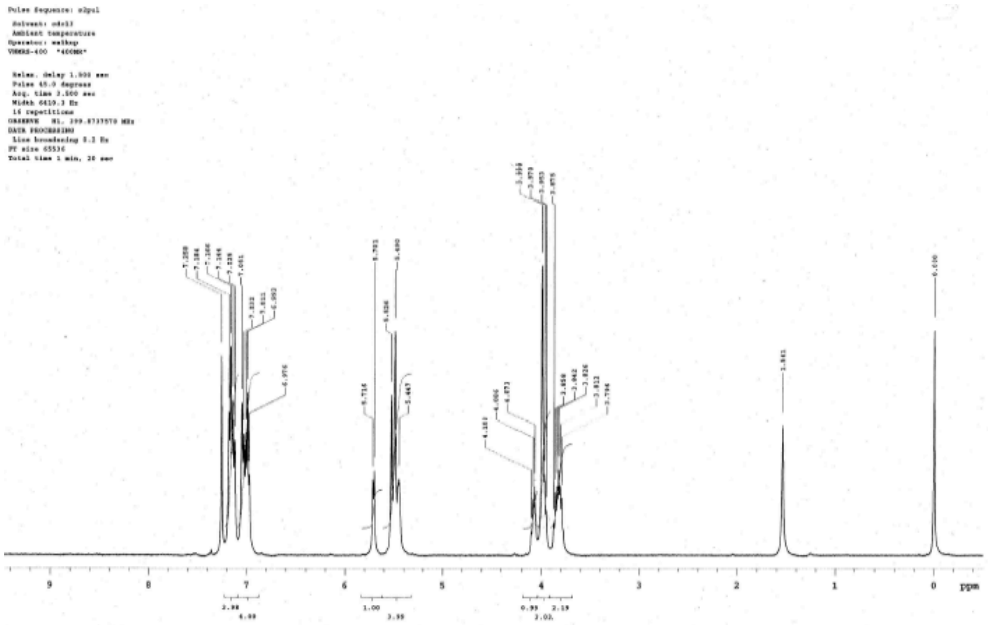


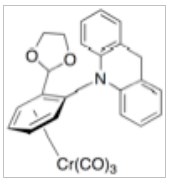

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.8529085 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 20 sec
O
O
N
O
Me
Cr(CO)3
5f
ppm


Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: walkup
VNMRS-400 "400MR"
Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.201 sec
Width 27173.9 Hz
512 repetitions
OBSERVE C13, 100.5430701 MHz
DECOUPLE H1, 399.8548169 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 17 min, 9 sec
ppm

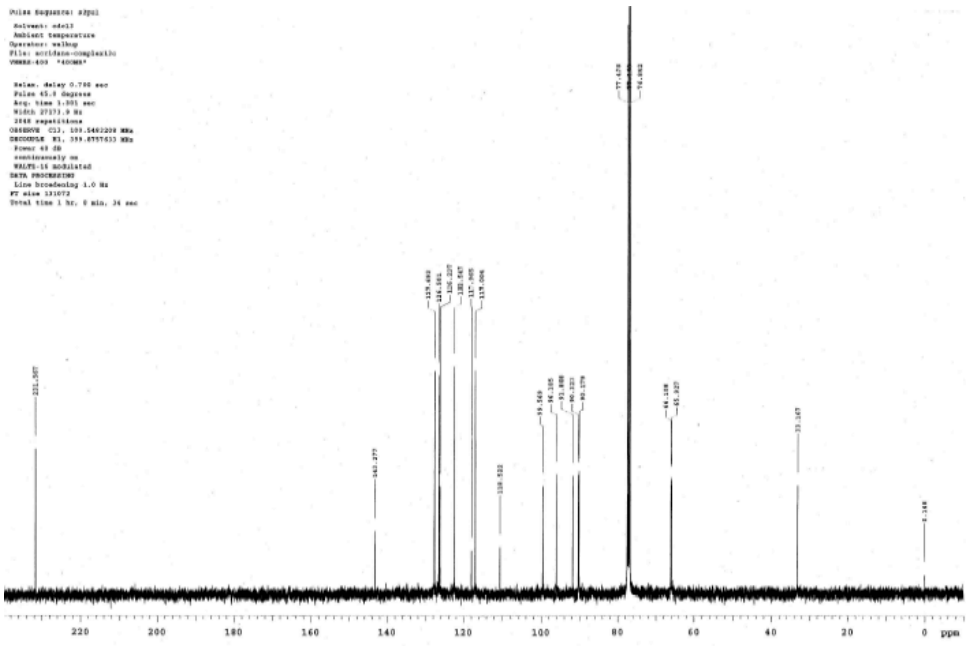

Cr(CO)3
Me
6f
ppm

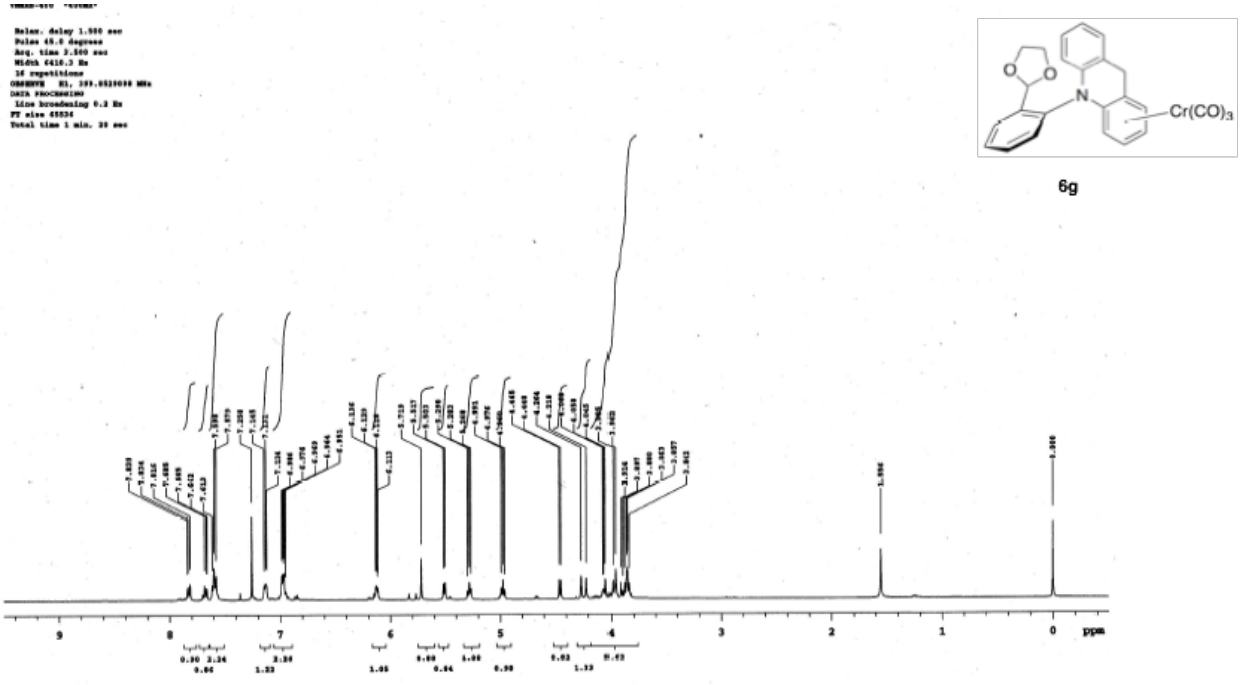

ppm

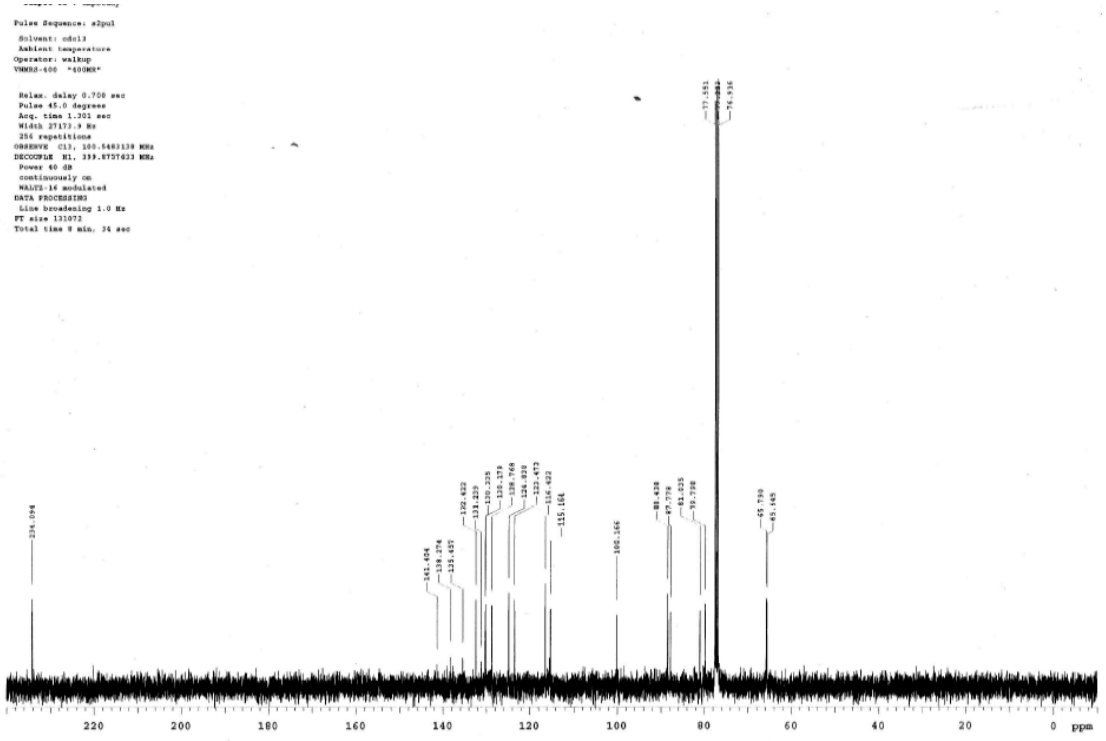


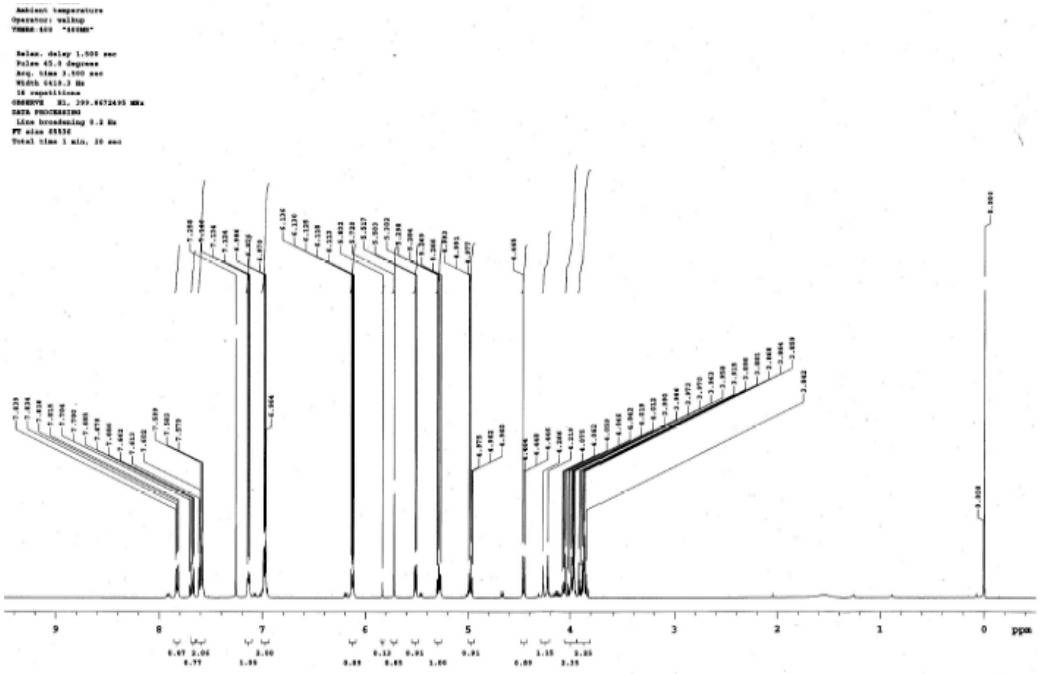


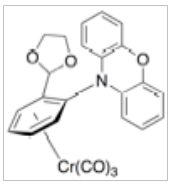

**5g**



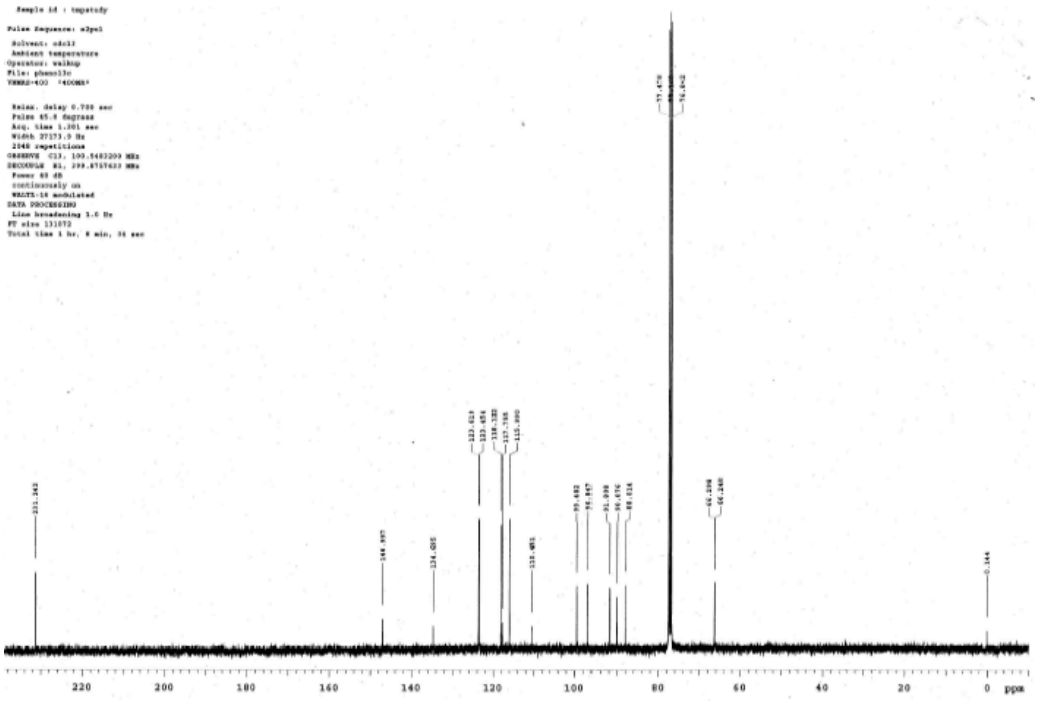

Cr(CO)3
6g
ppm

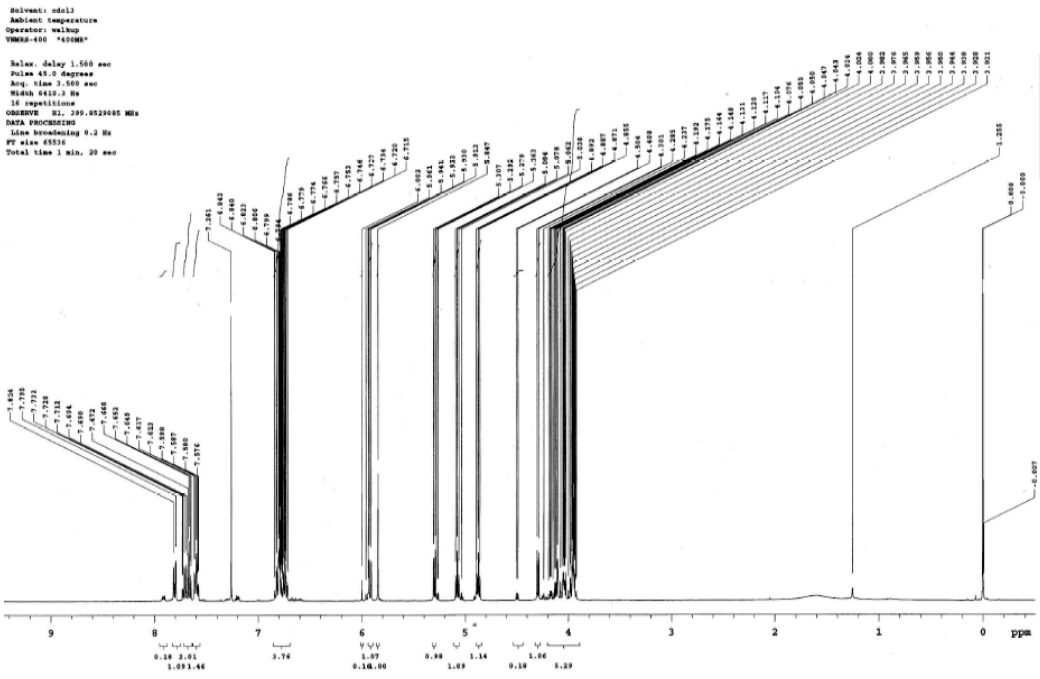

ppm

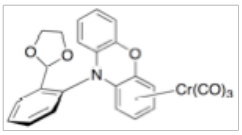




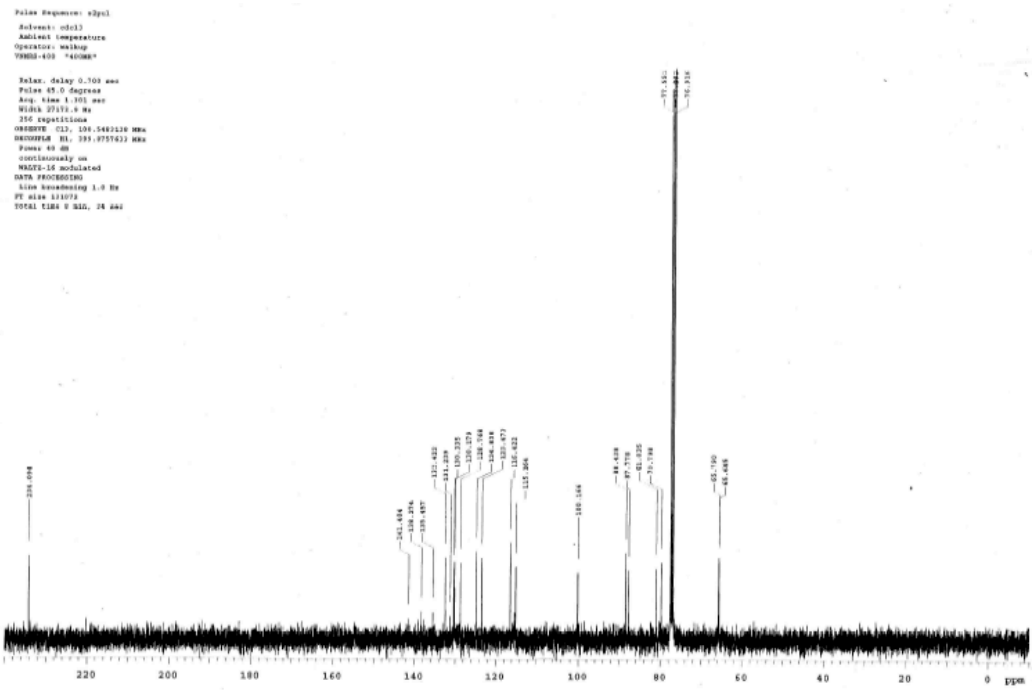

**5h**

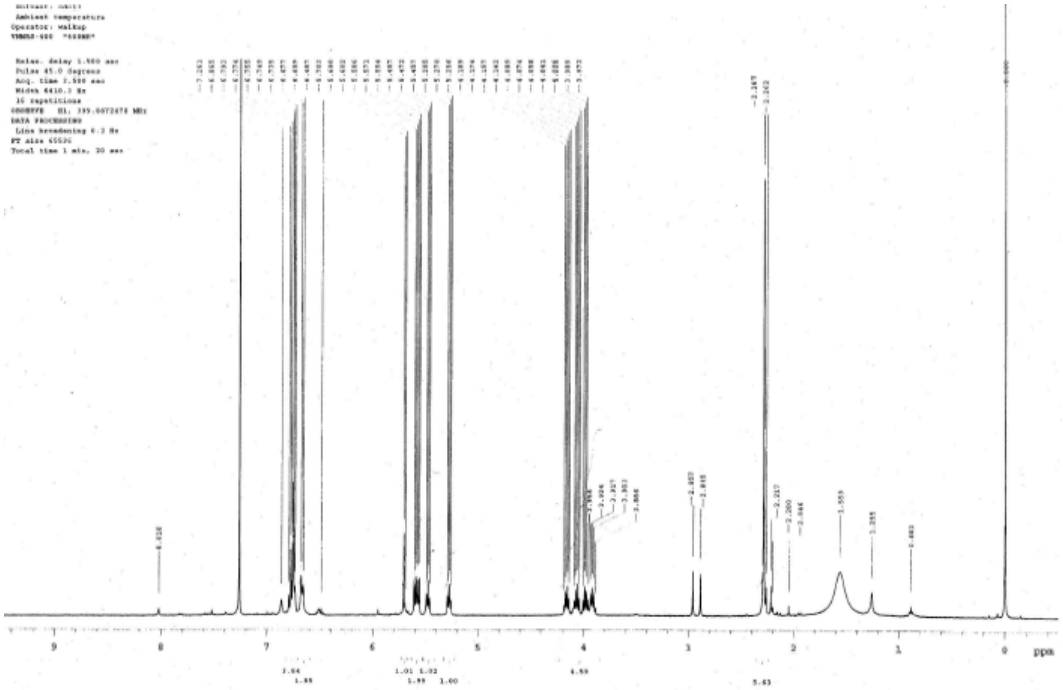


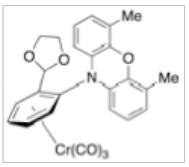




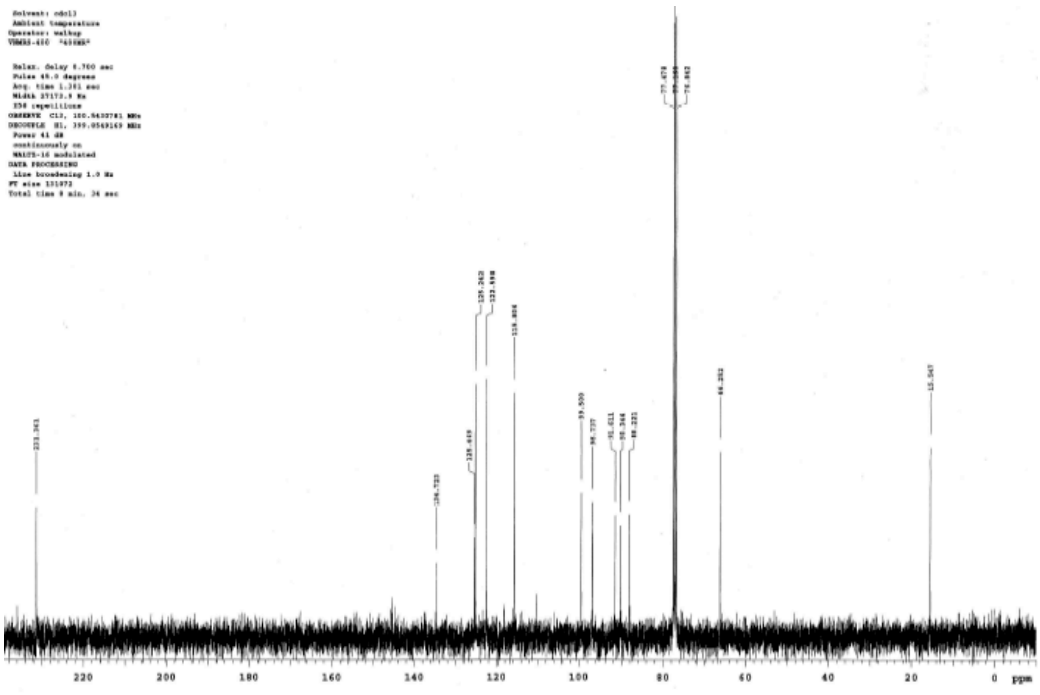

6h

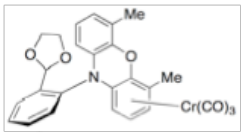




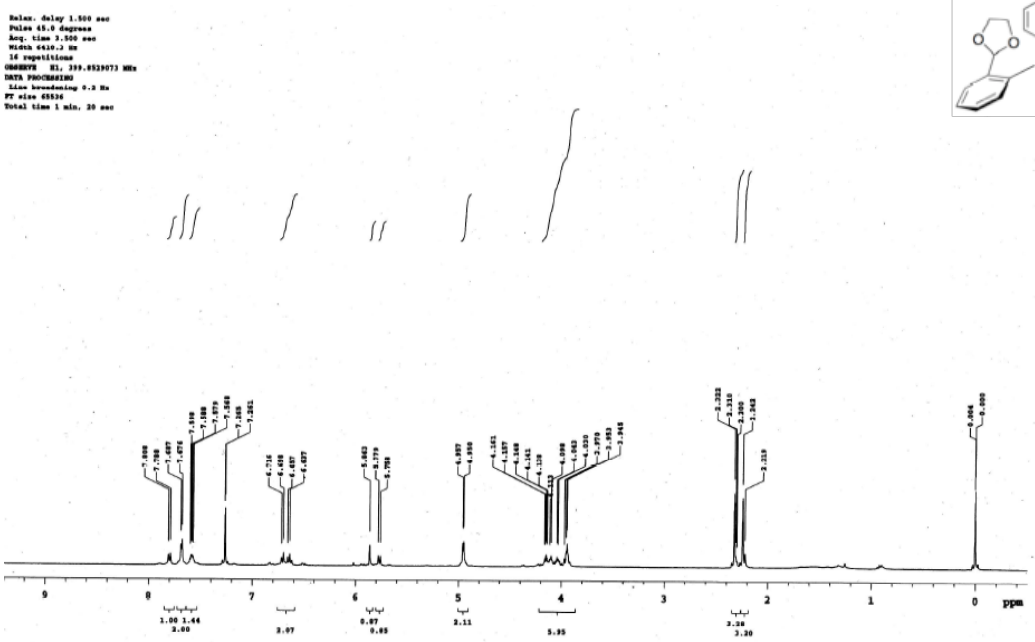


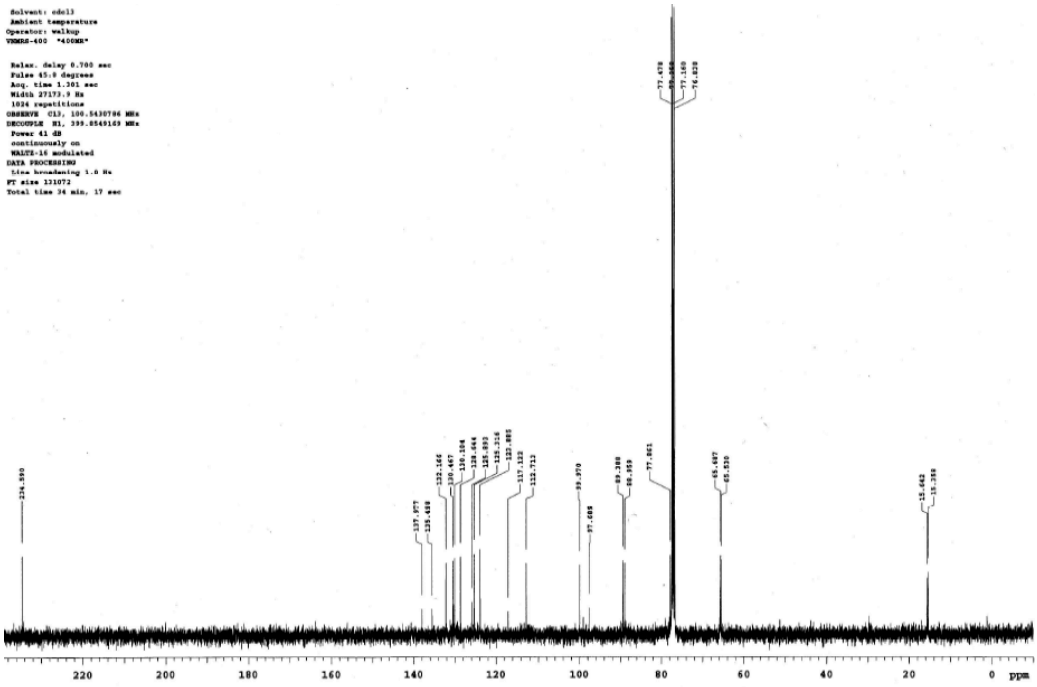

**5i**





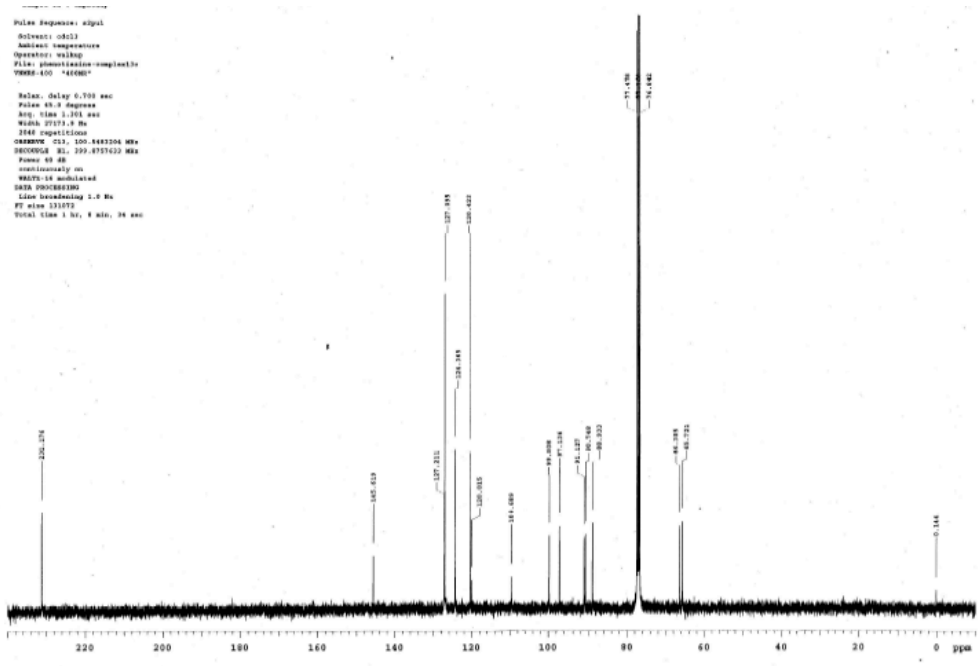

6i

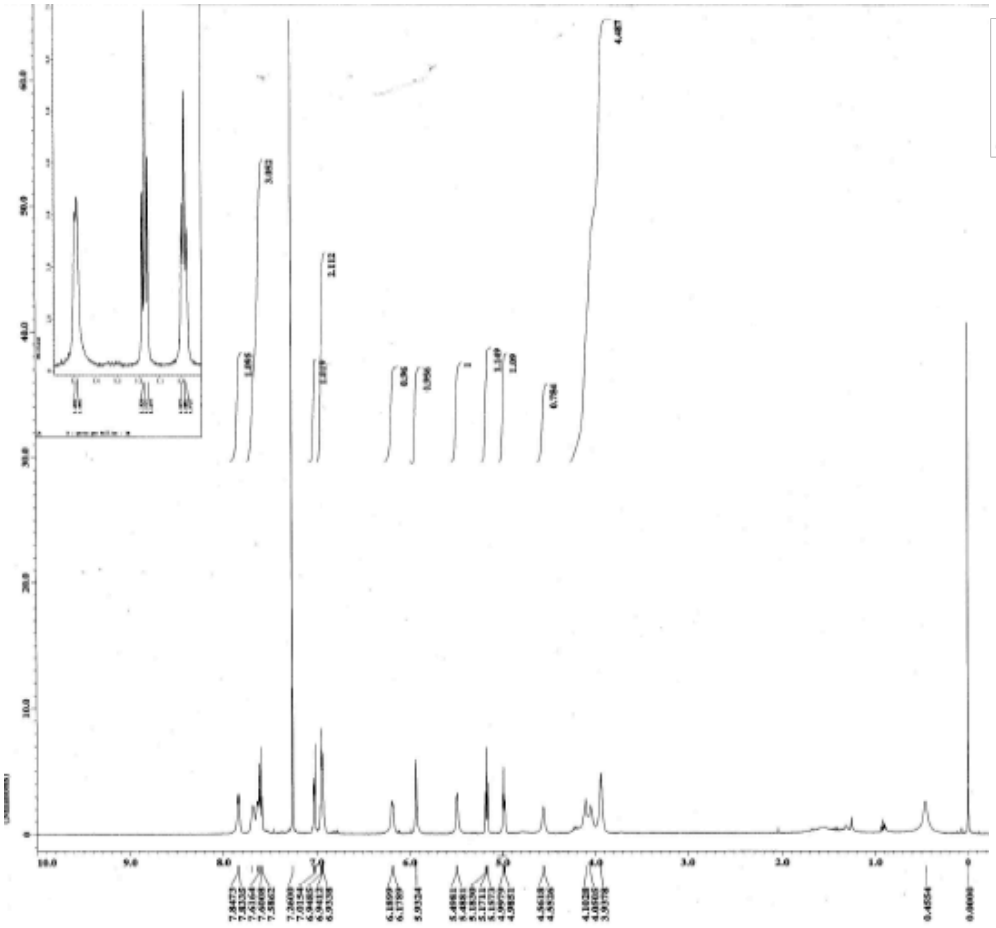


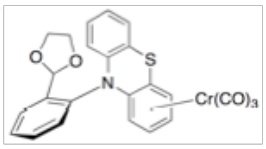




5j

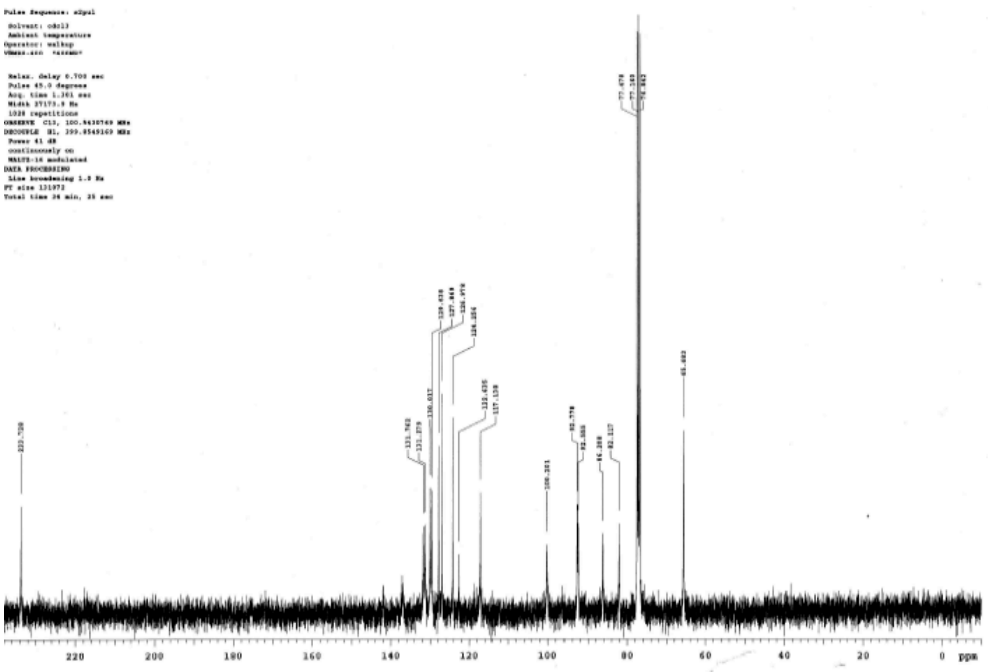


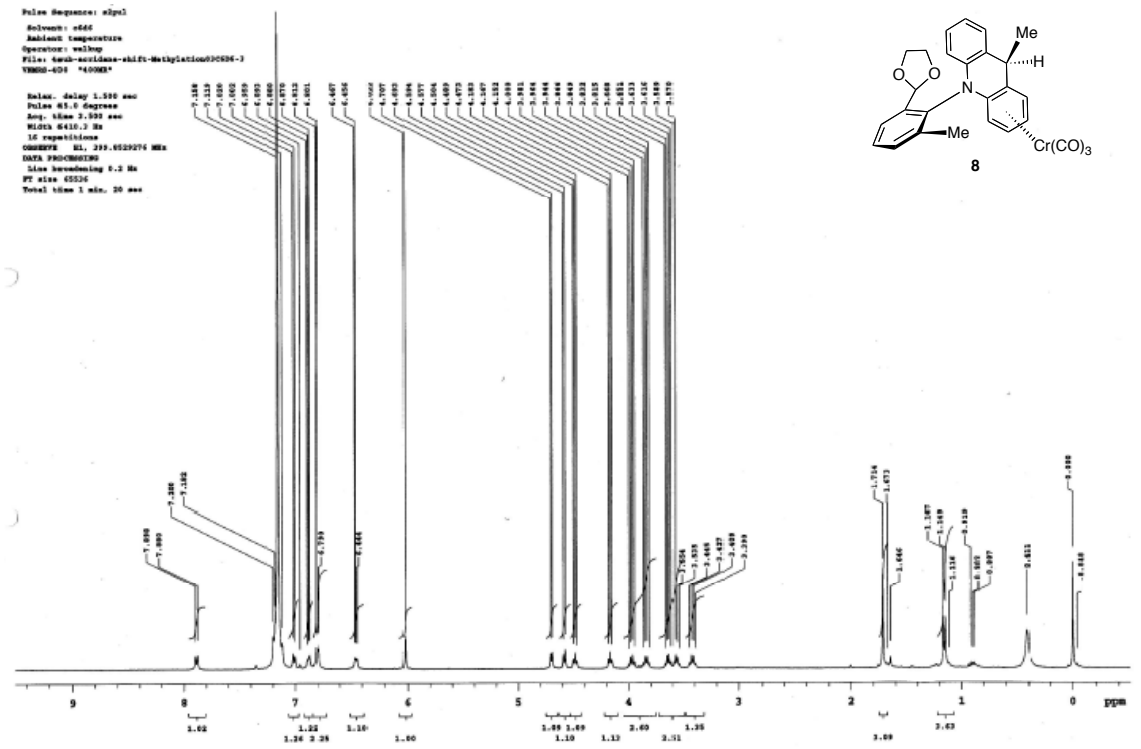


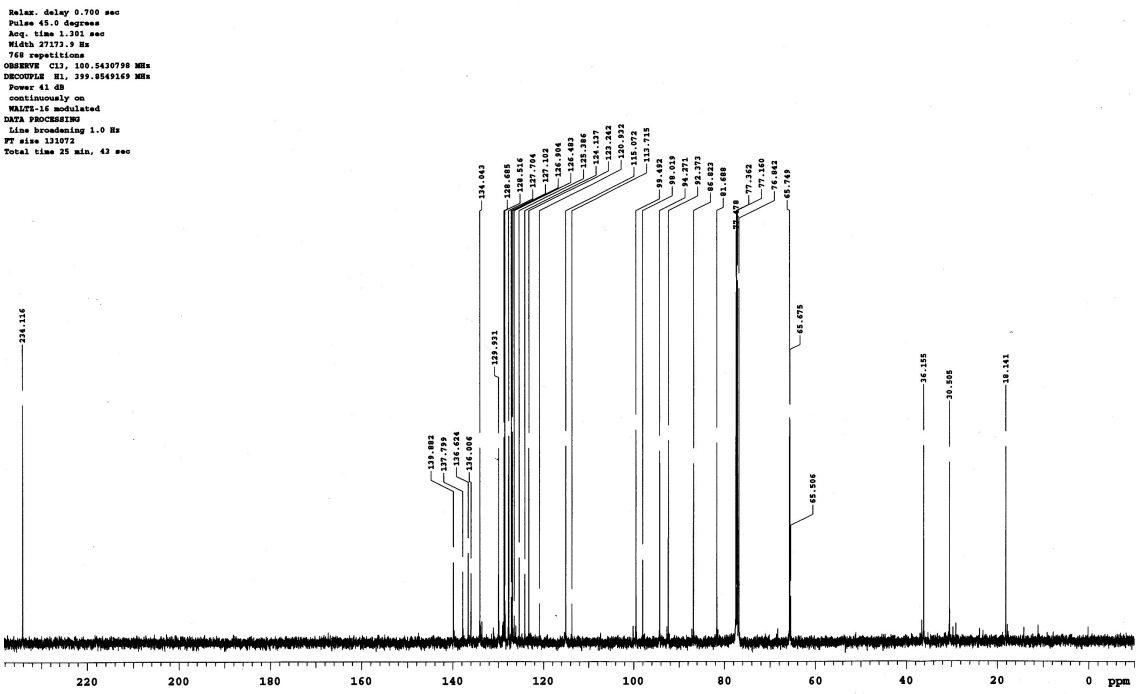





**6j**




Me
H
N
O
O
Me
Cr(CO)3
8
ppm


Relax. delay 0.700 sec
Pulse 45.0 degrees
Acq. time 1.301 sec
Width 27173.9 Hz
768 repetitions
OBSERVE C13, 100.5430798 MHz
DECOUPLE H1, 399.8549169 MHz
Power 41 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 25 min, 43 sec
ppm

## References

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