

Supporting information for

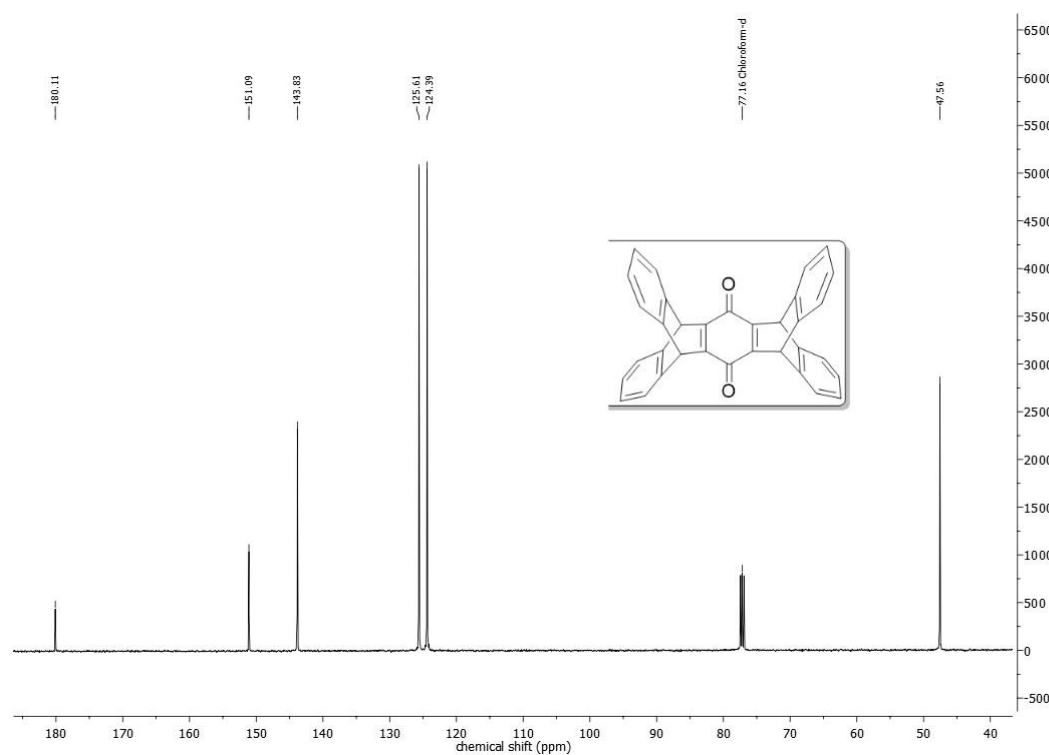
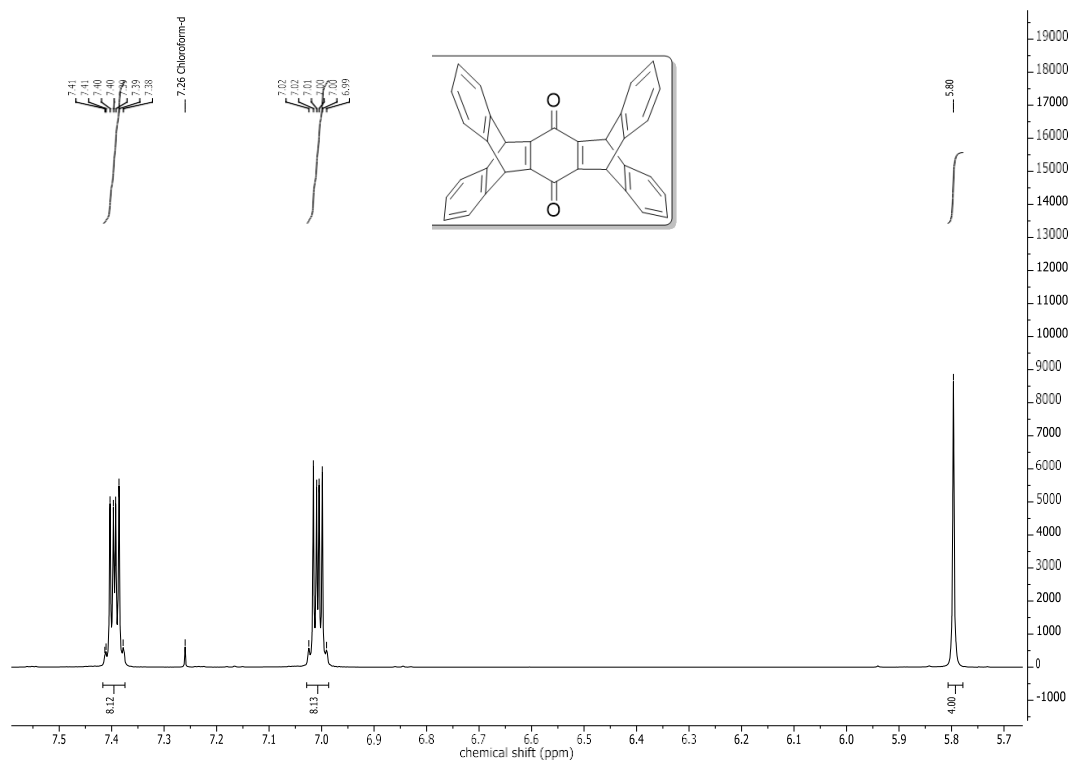
Penttiptycene-based concave NHC-metal complexes

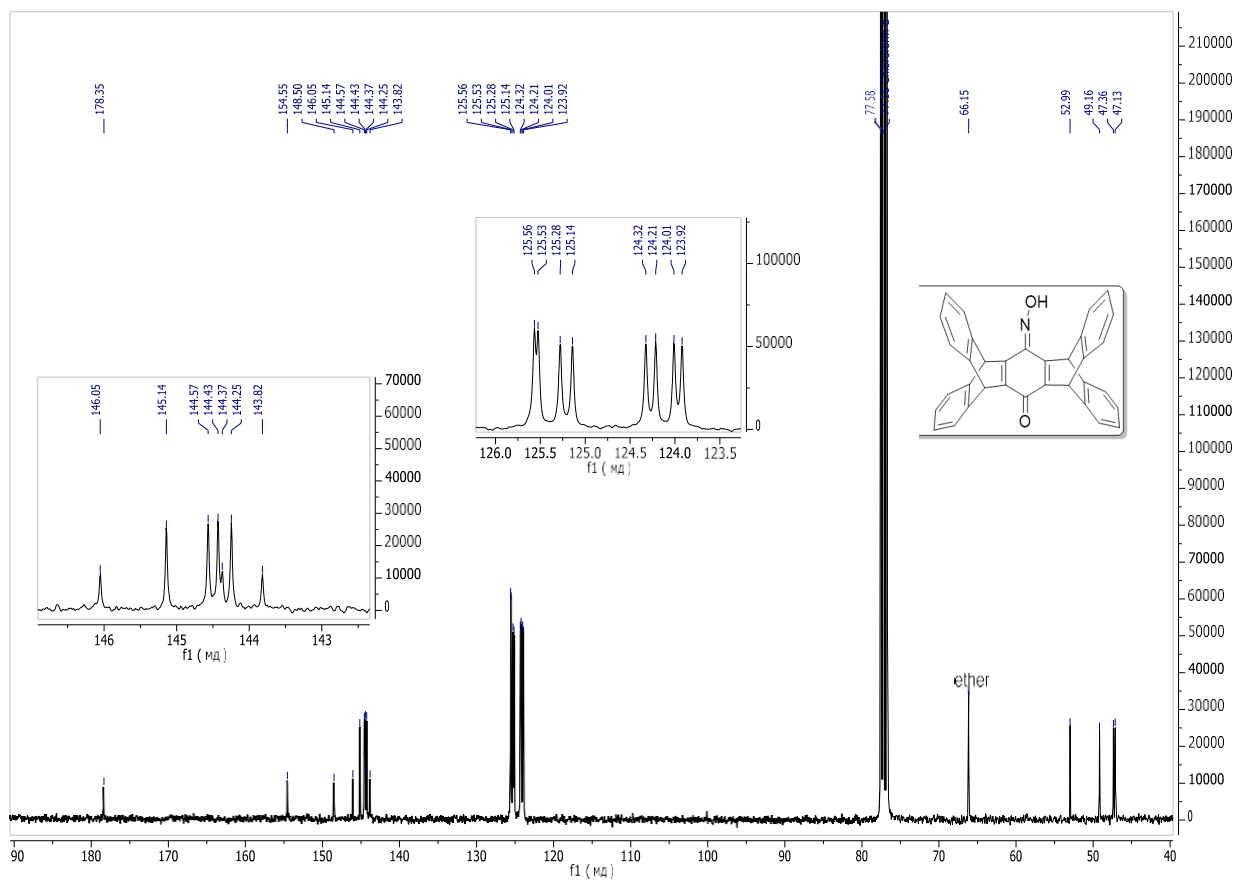
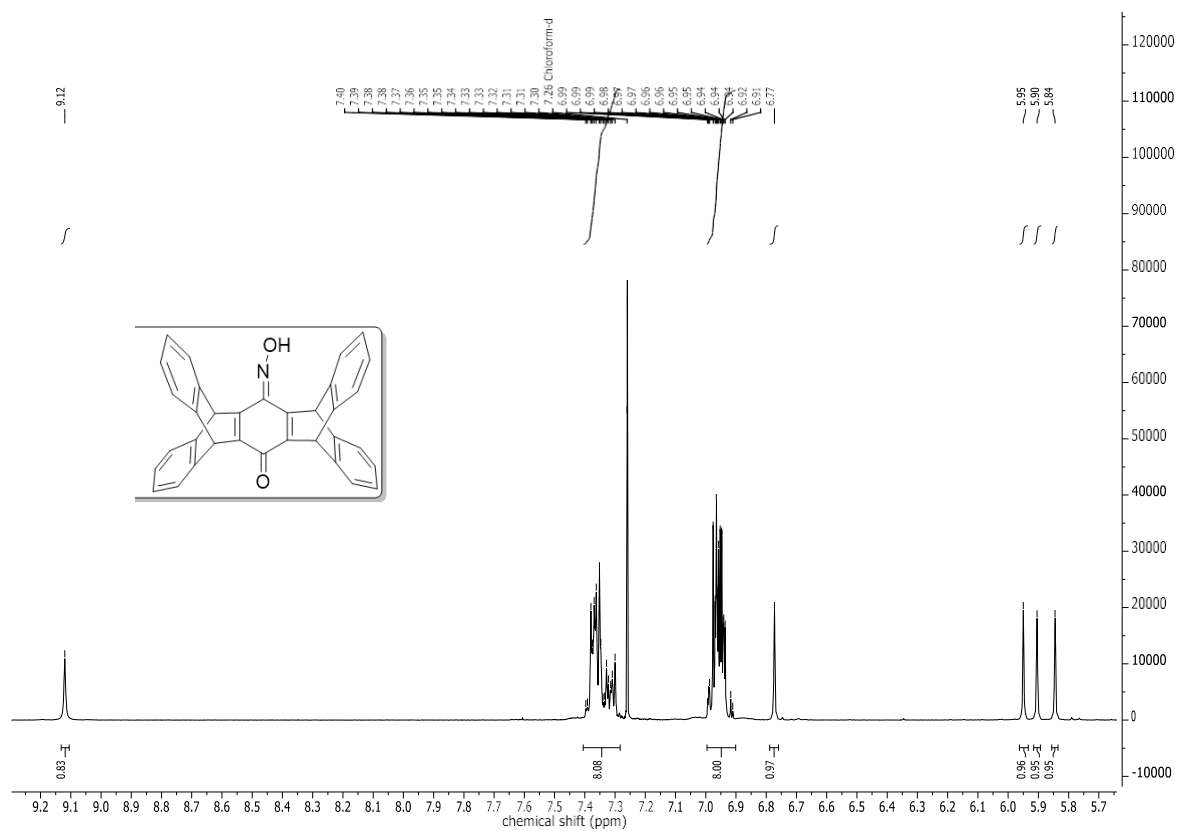
Roman Savka, Sabine Foro and Herbert Plenio*

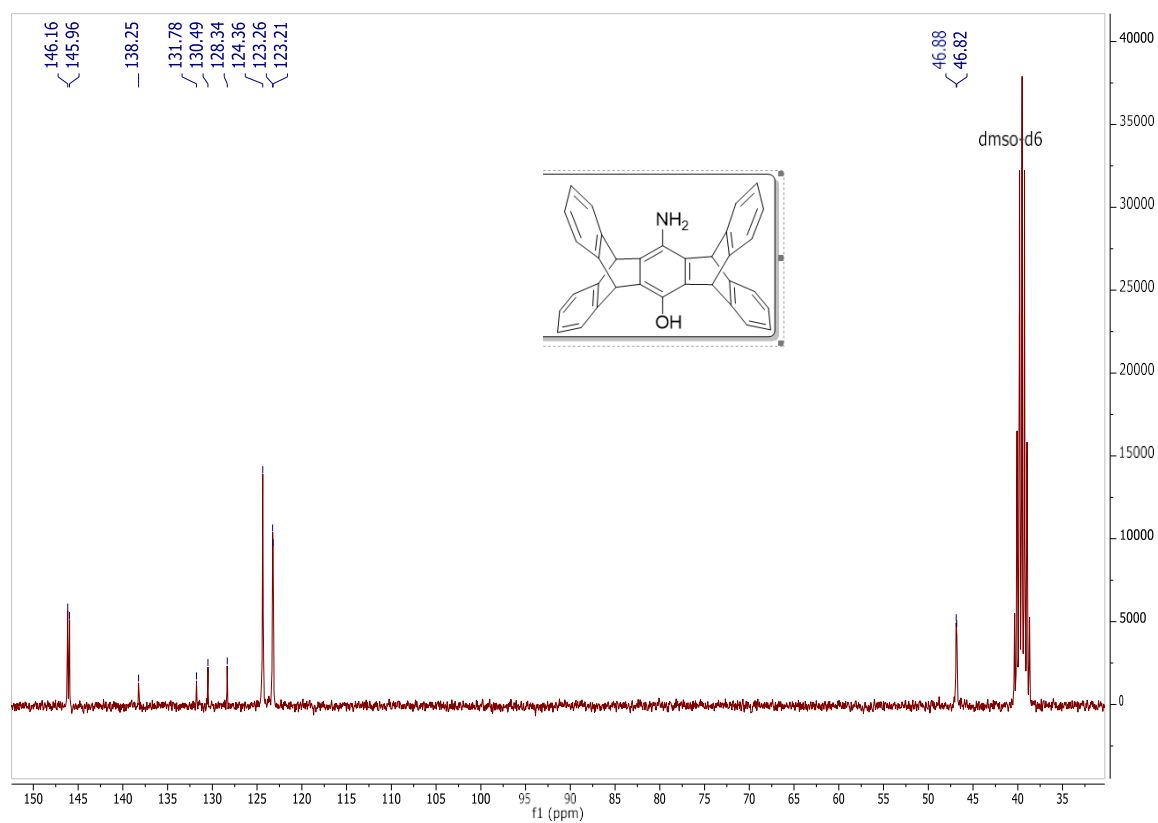
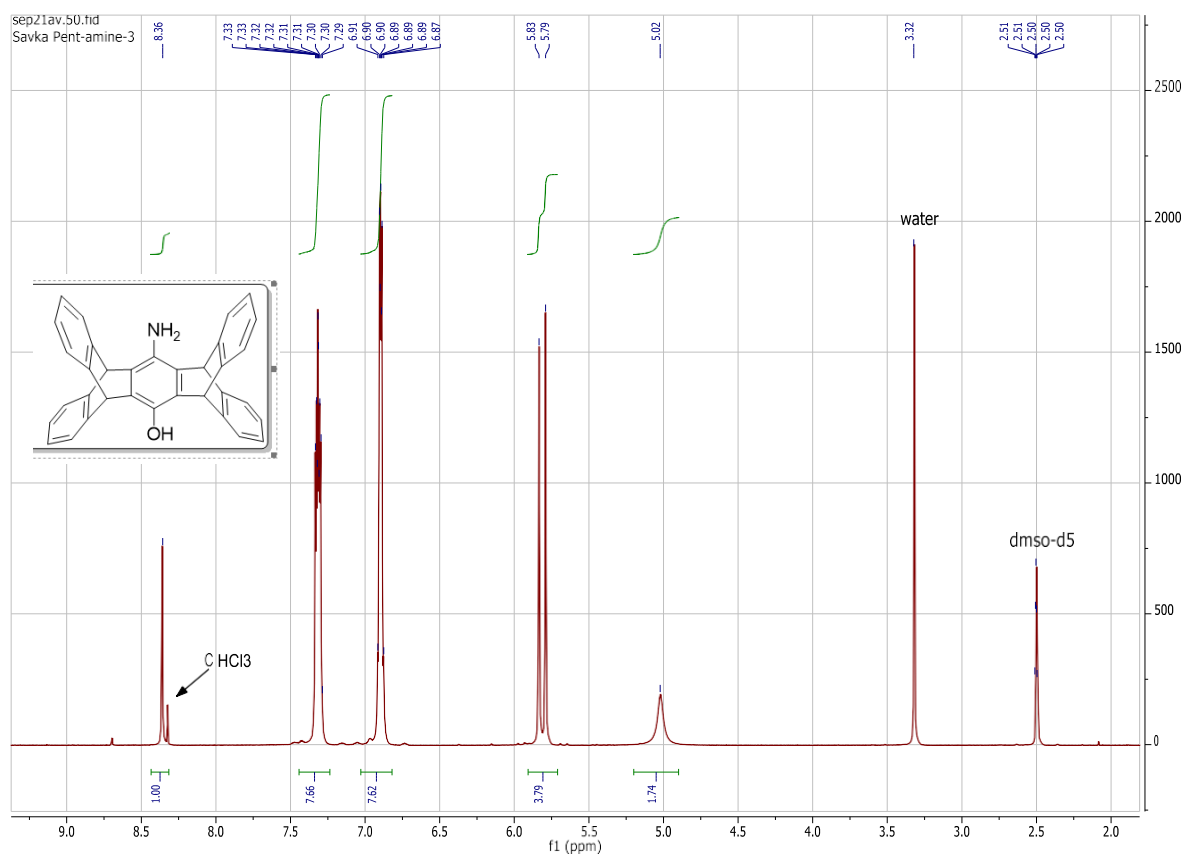
Organometallic Chemistry, Alarich-Weiss-Str. 12, TU Darmstadt, 64287 Darmstadt, Germany, e-mail:
plenio@tu-darmstadt.de

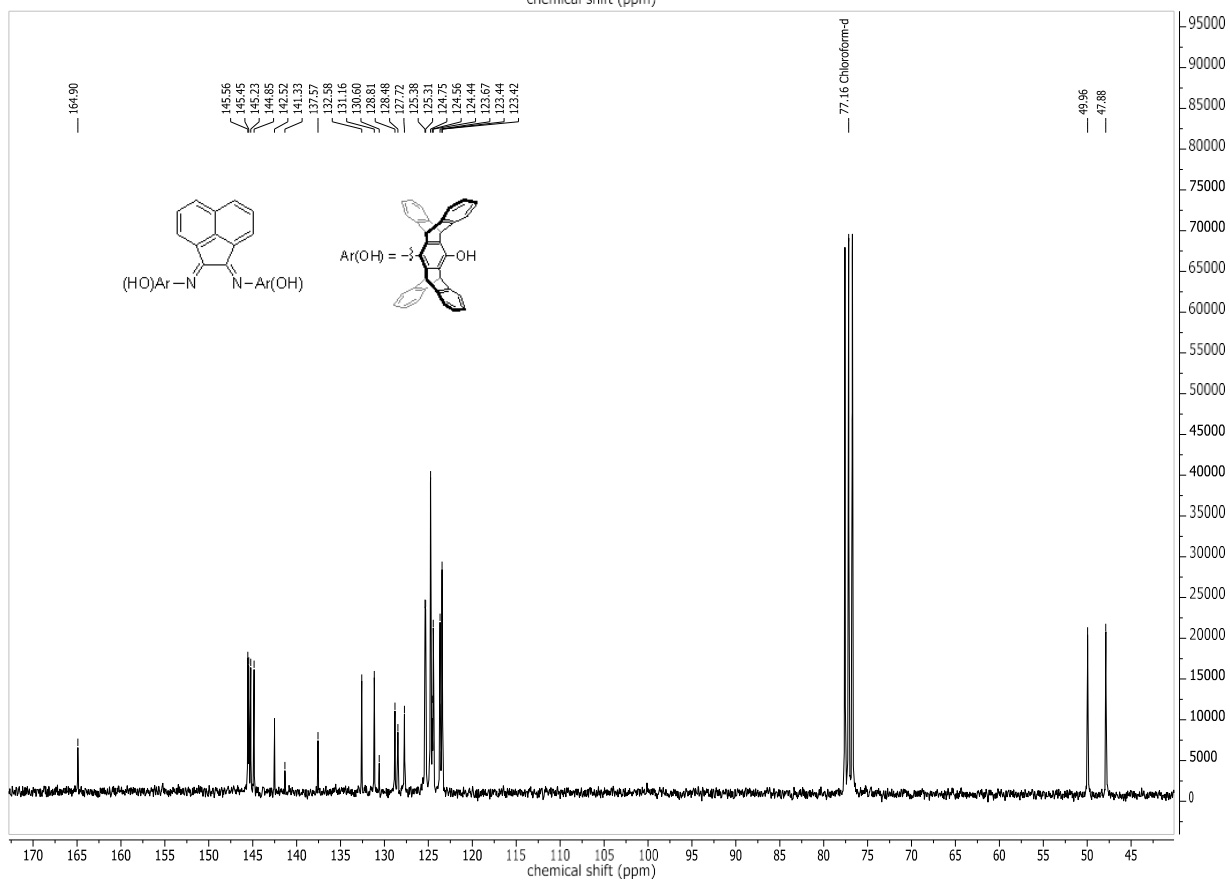
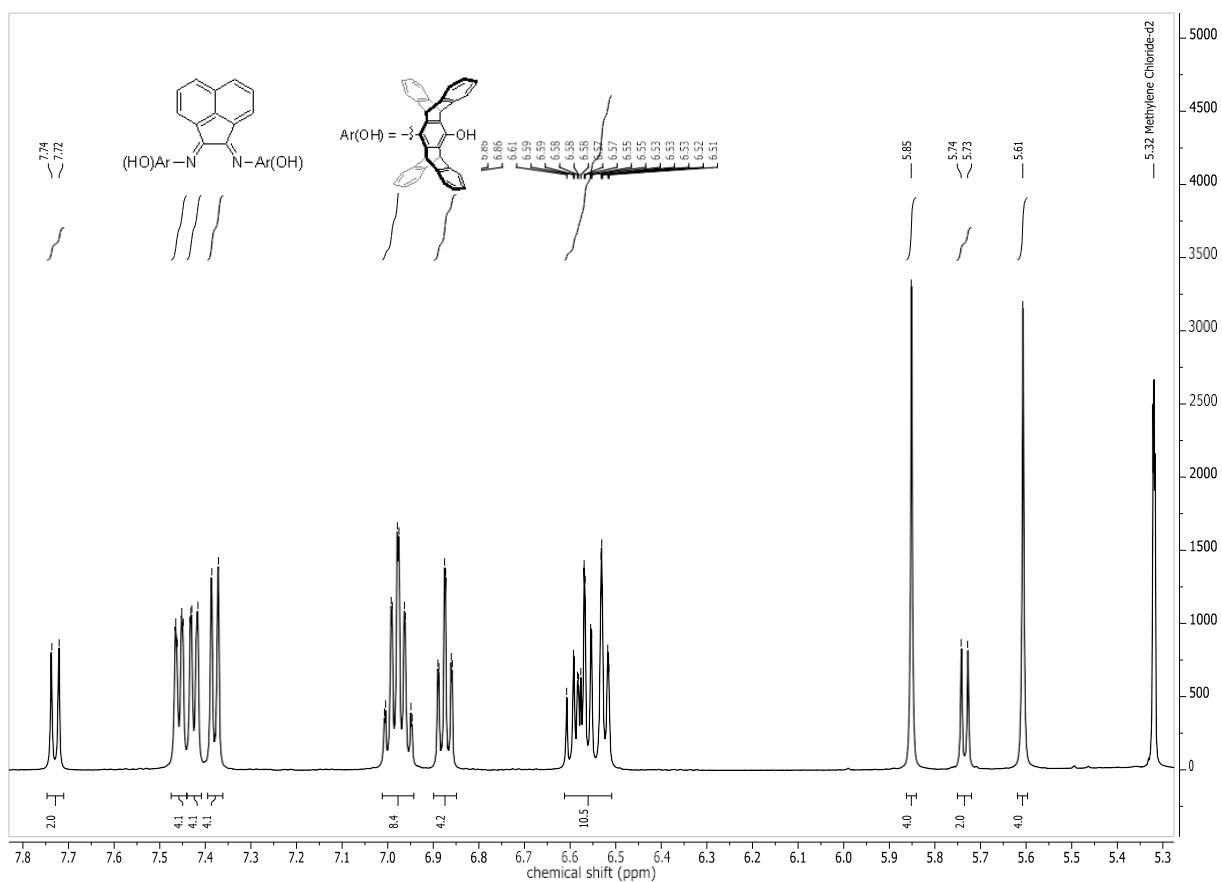
NMR spectra	p. 2 - 24
Mass spectra	p. 24-33
Cyclic voltammetry	p. 45
Infrared spectra	p. 46

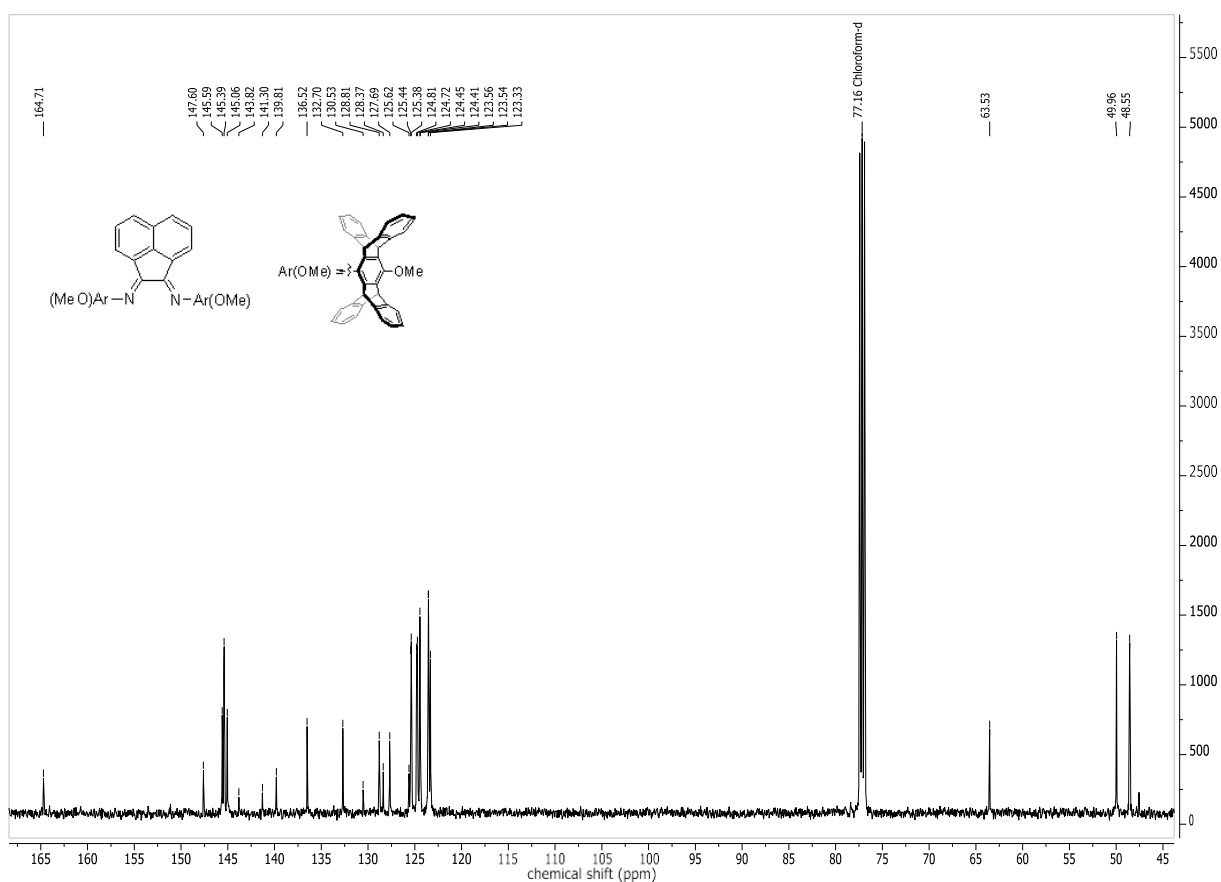
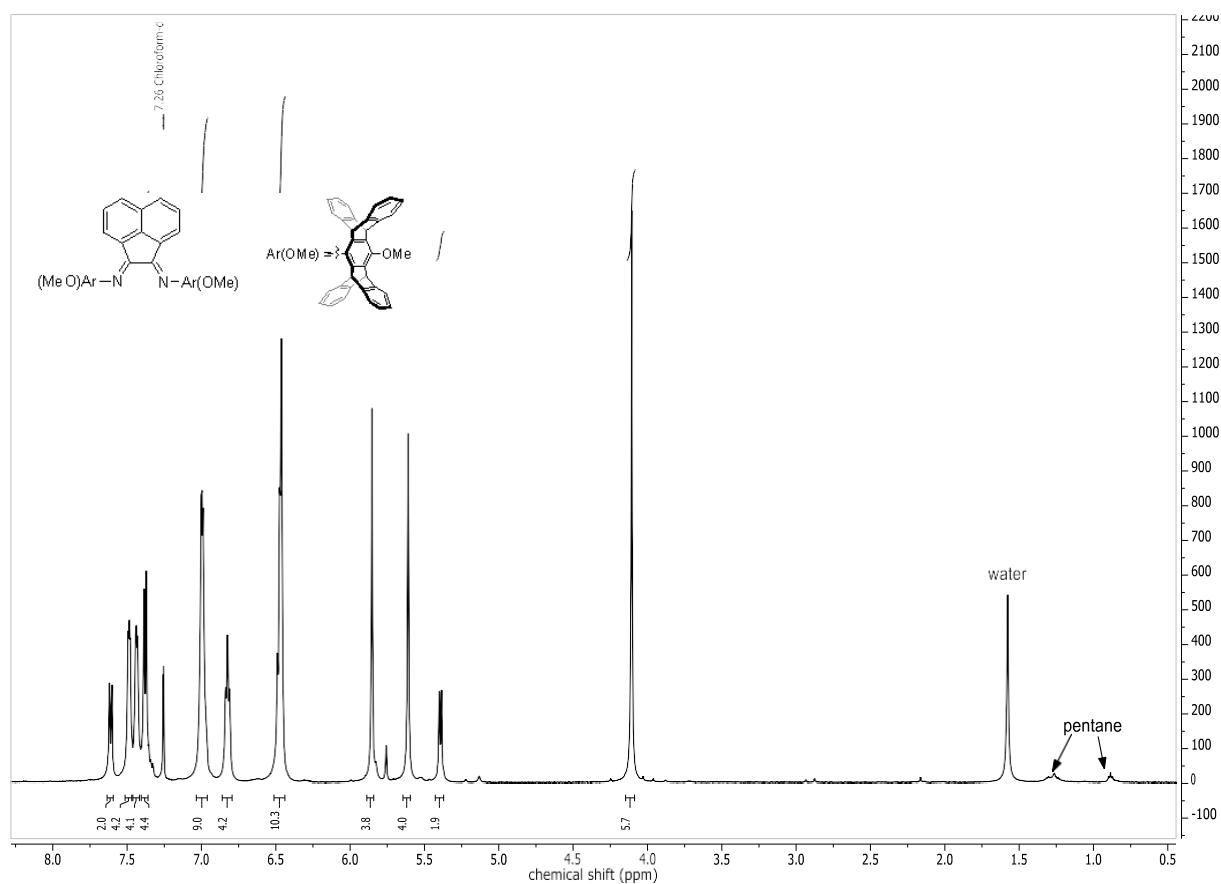
NMR spectra

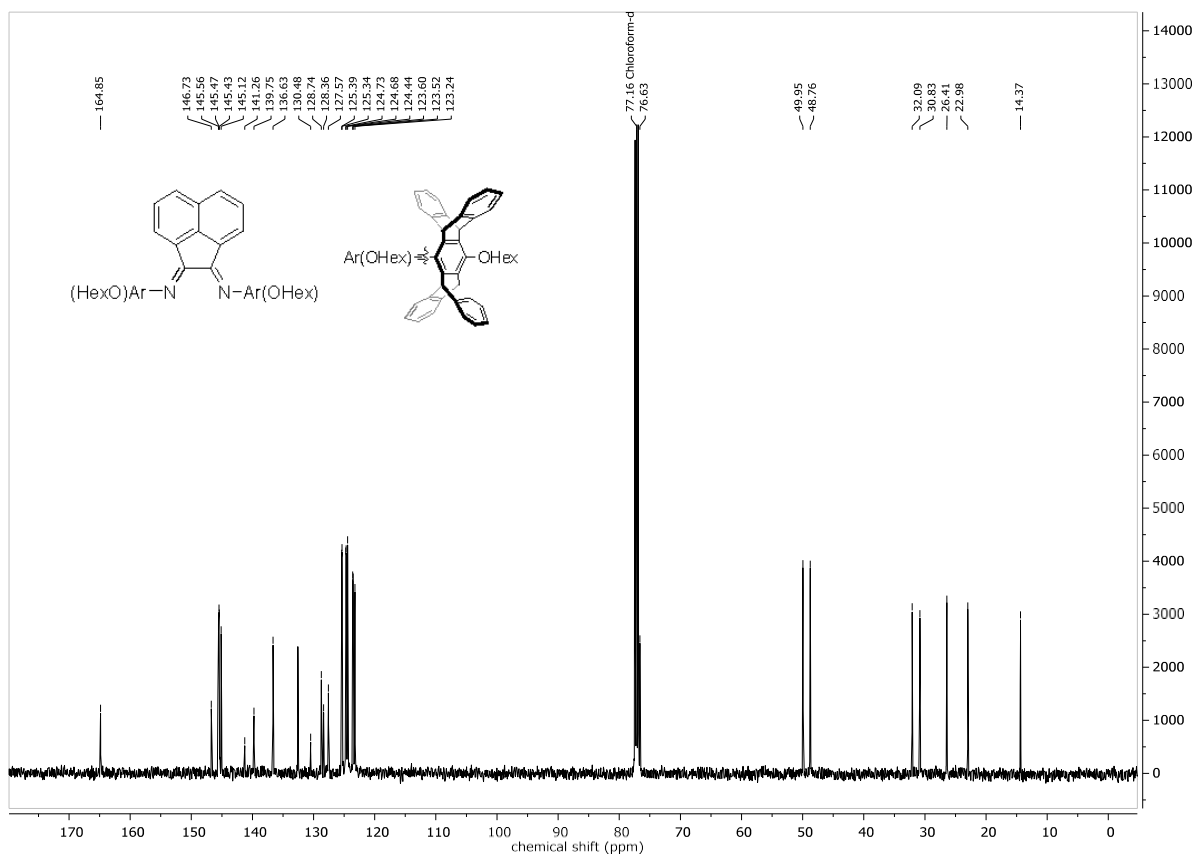
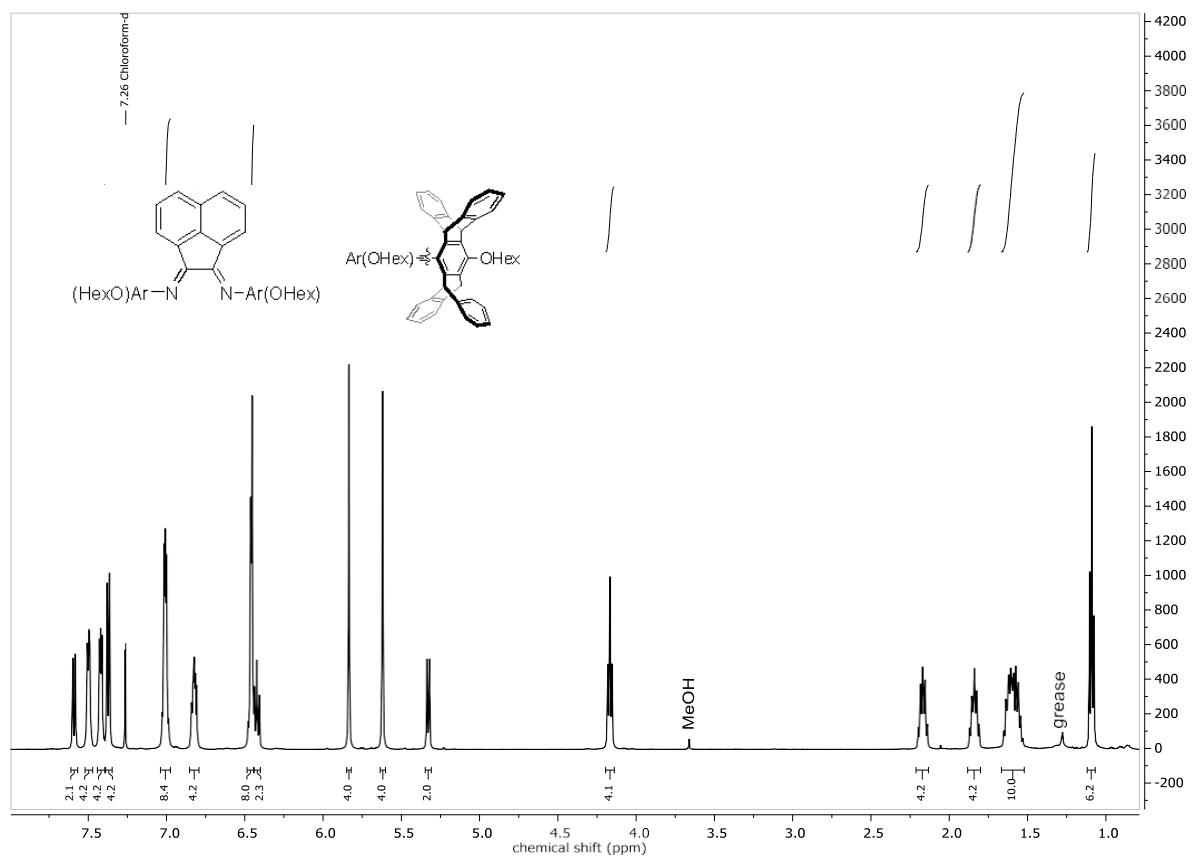


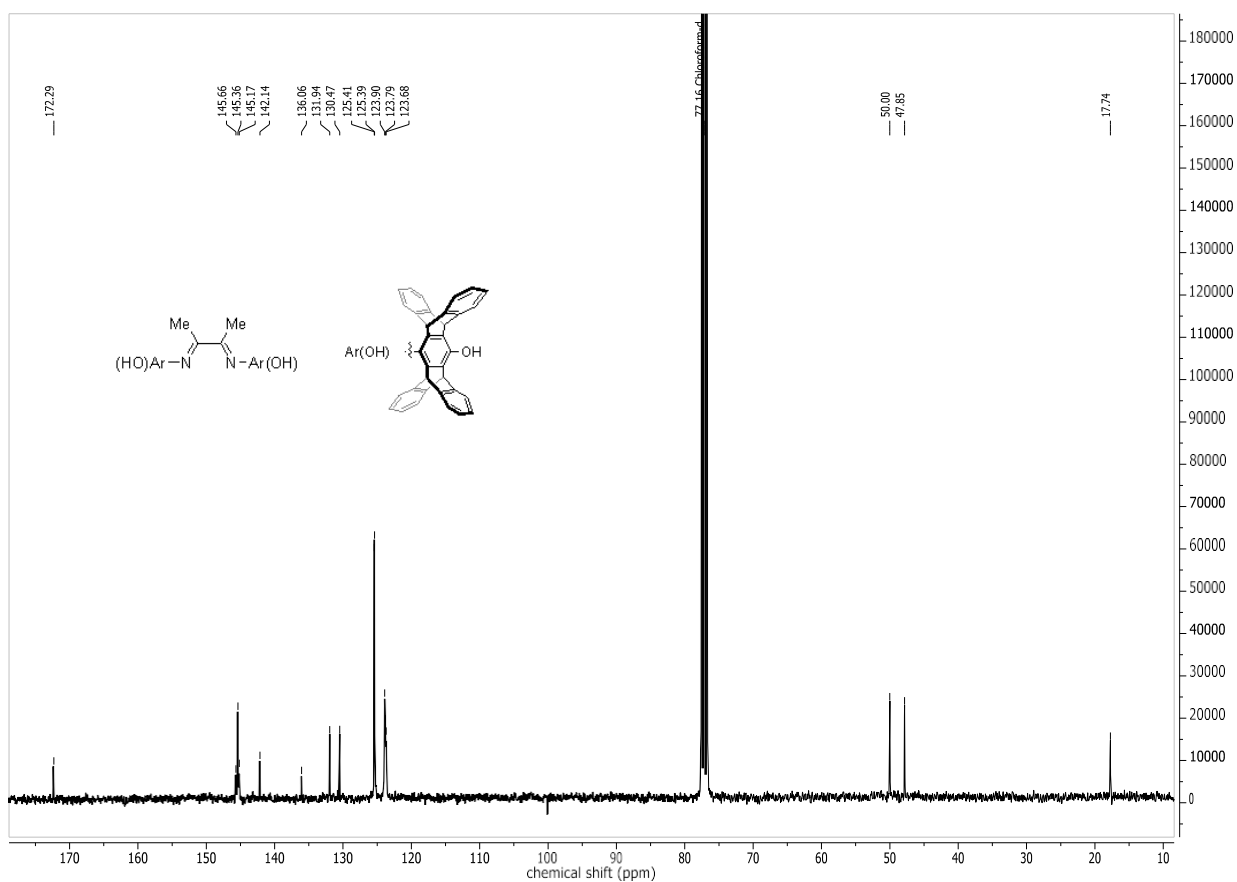
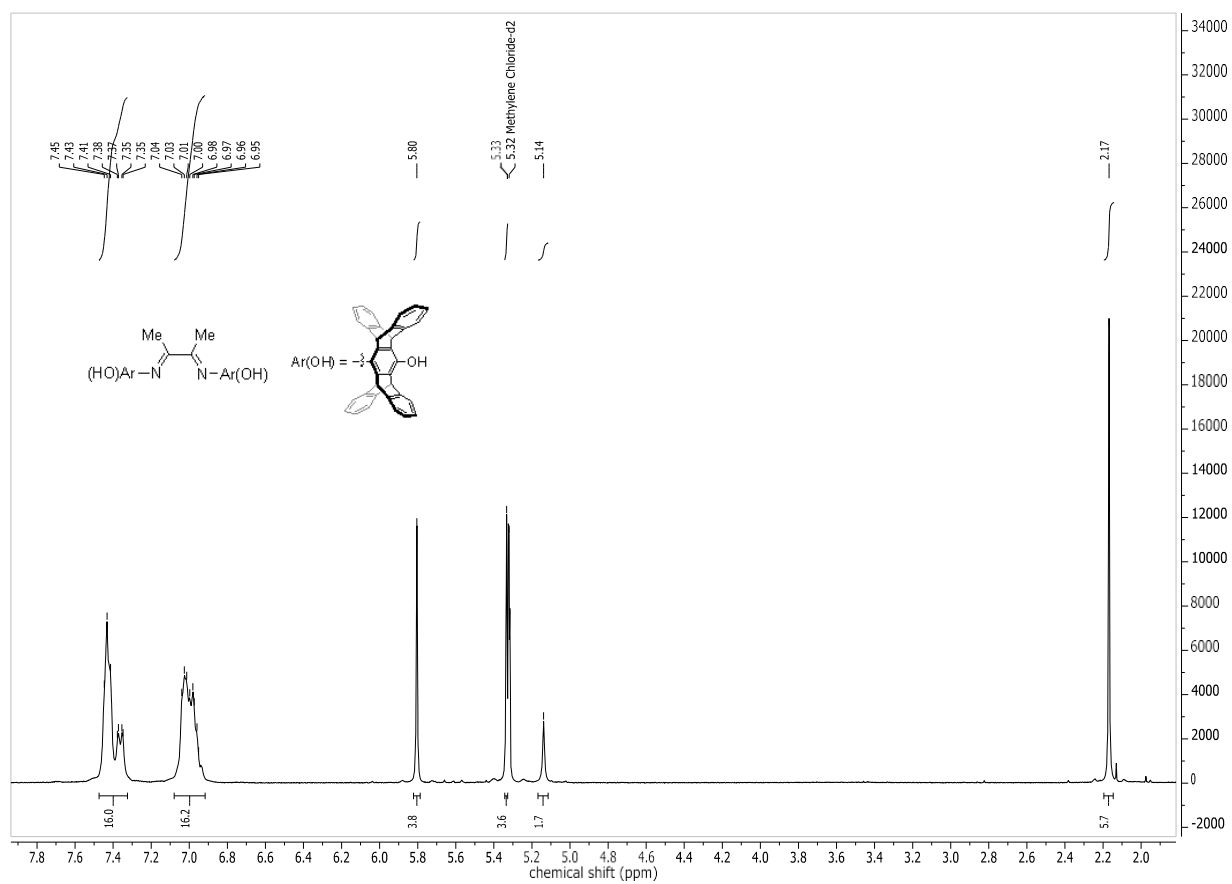


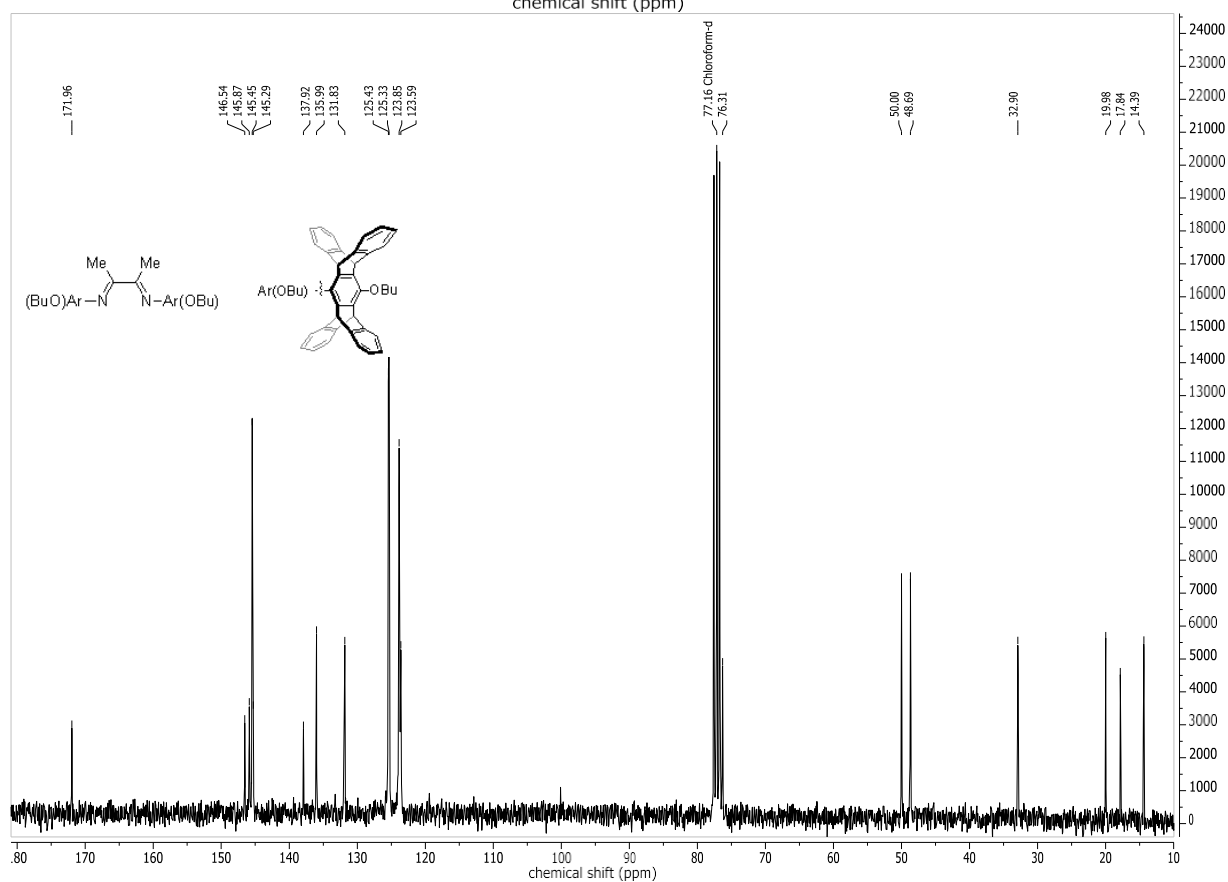
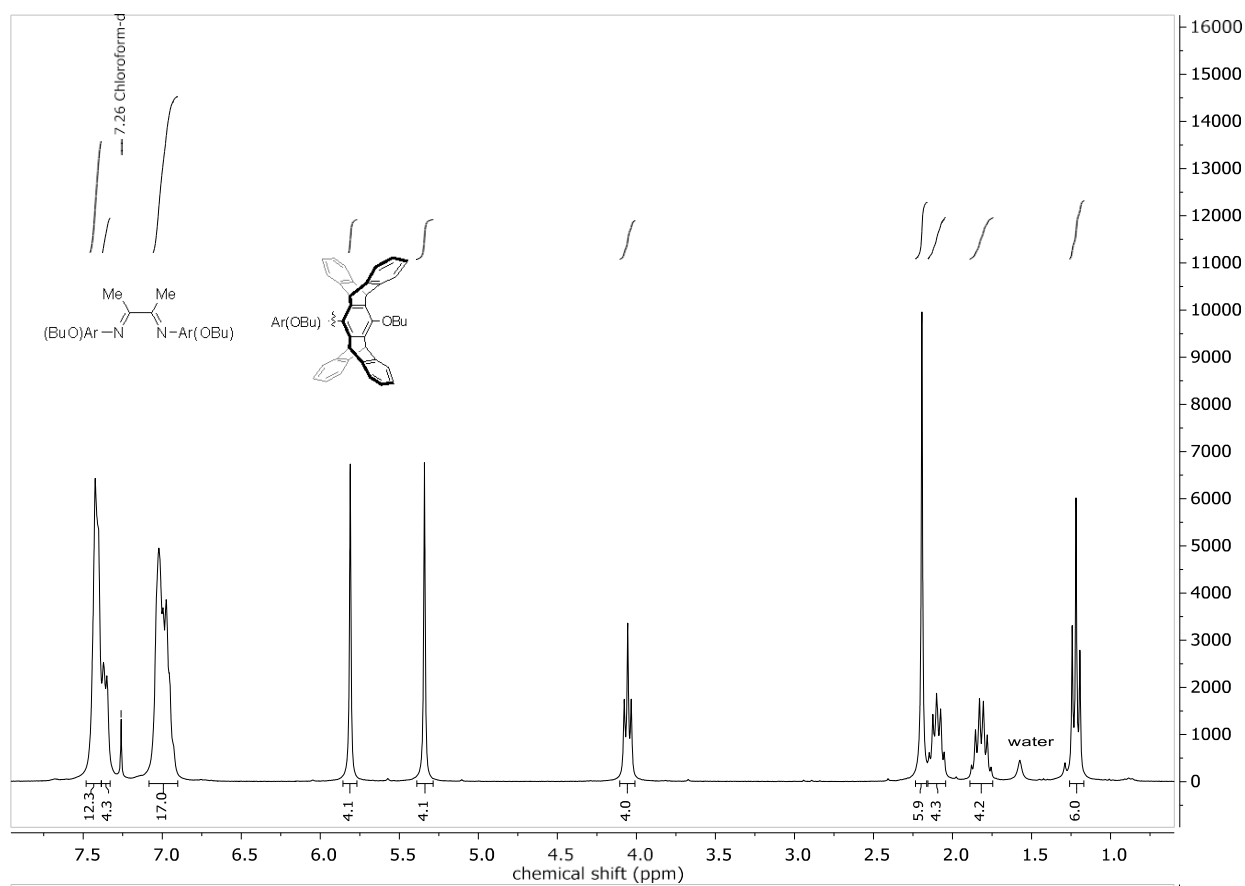


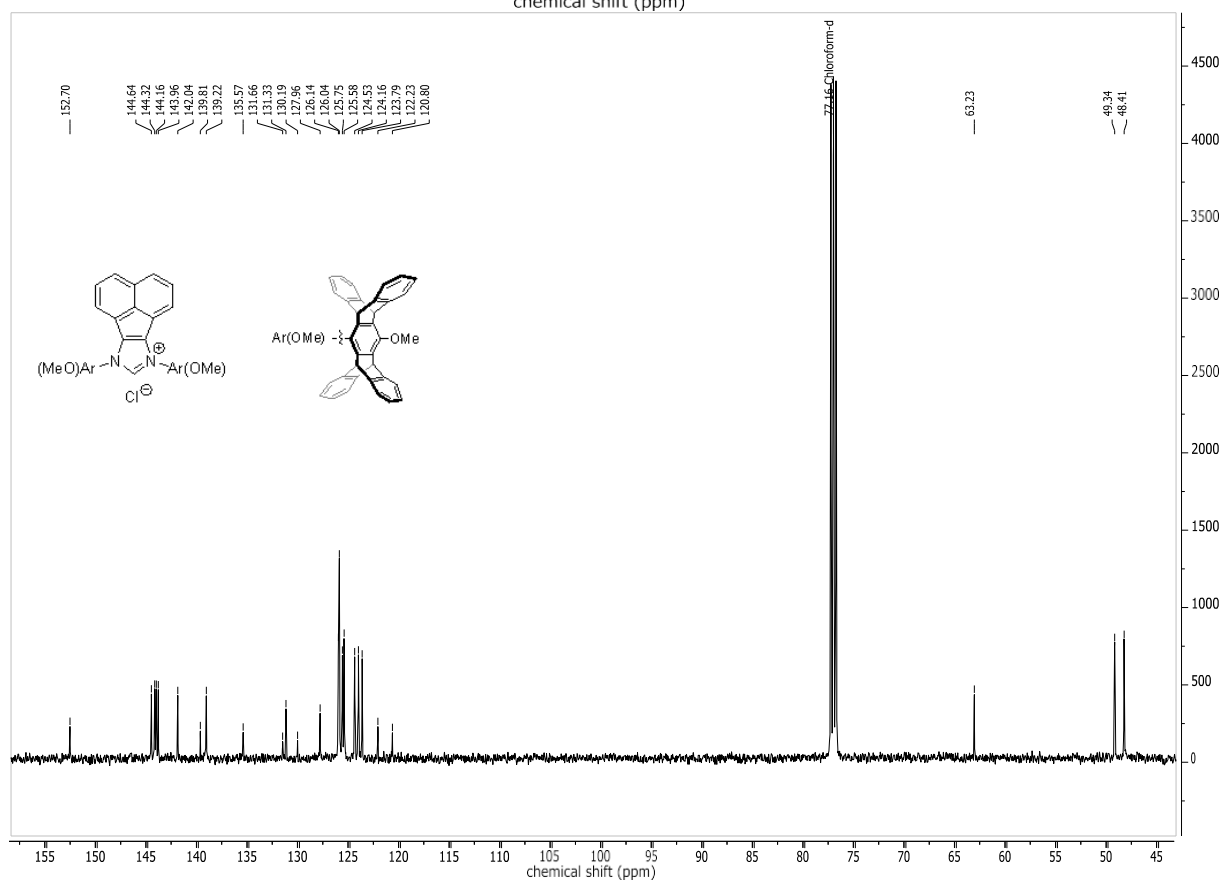
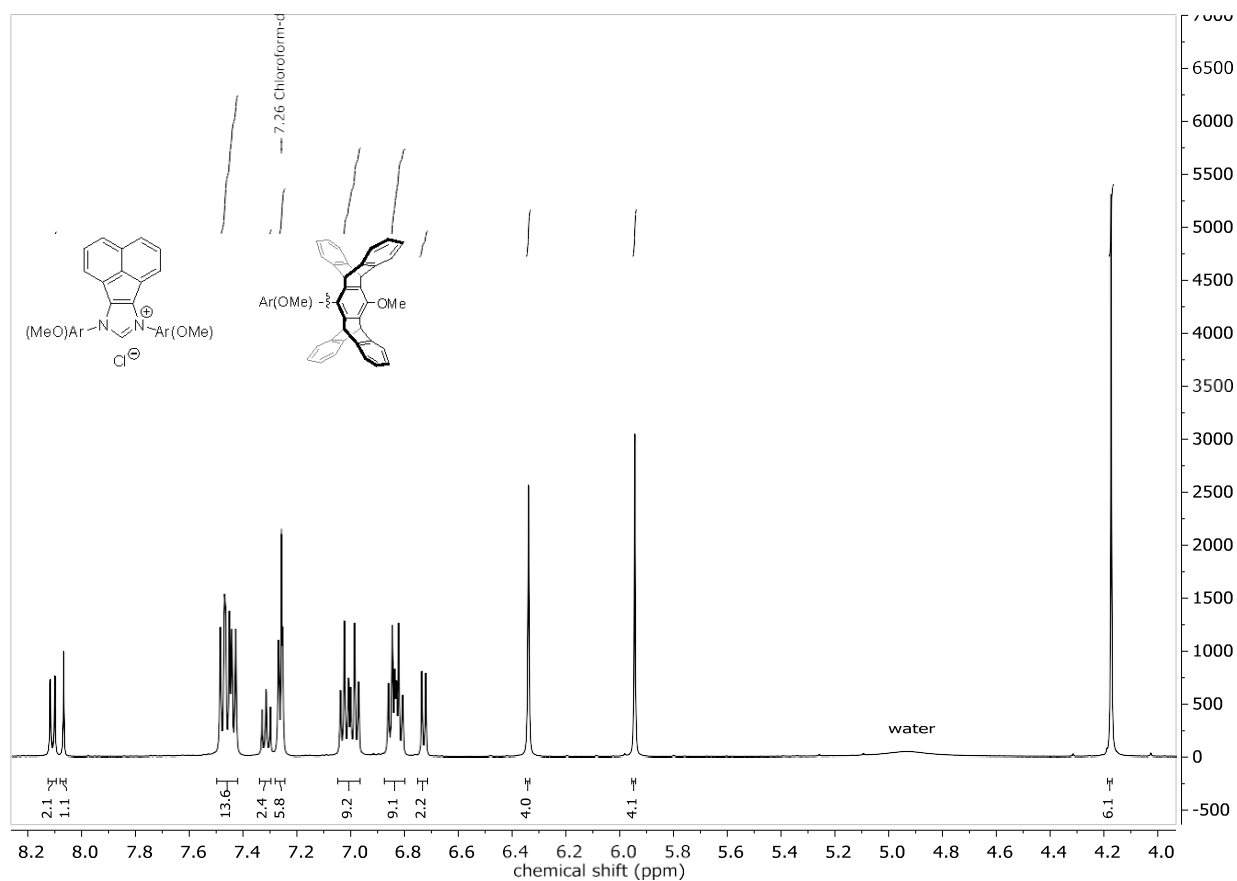


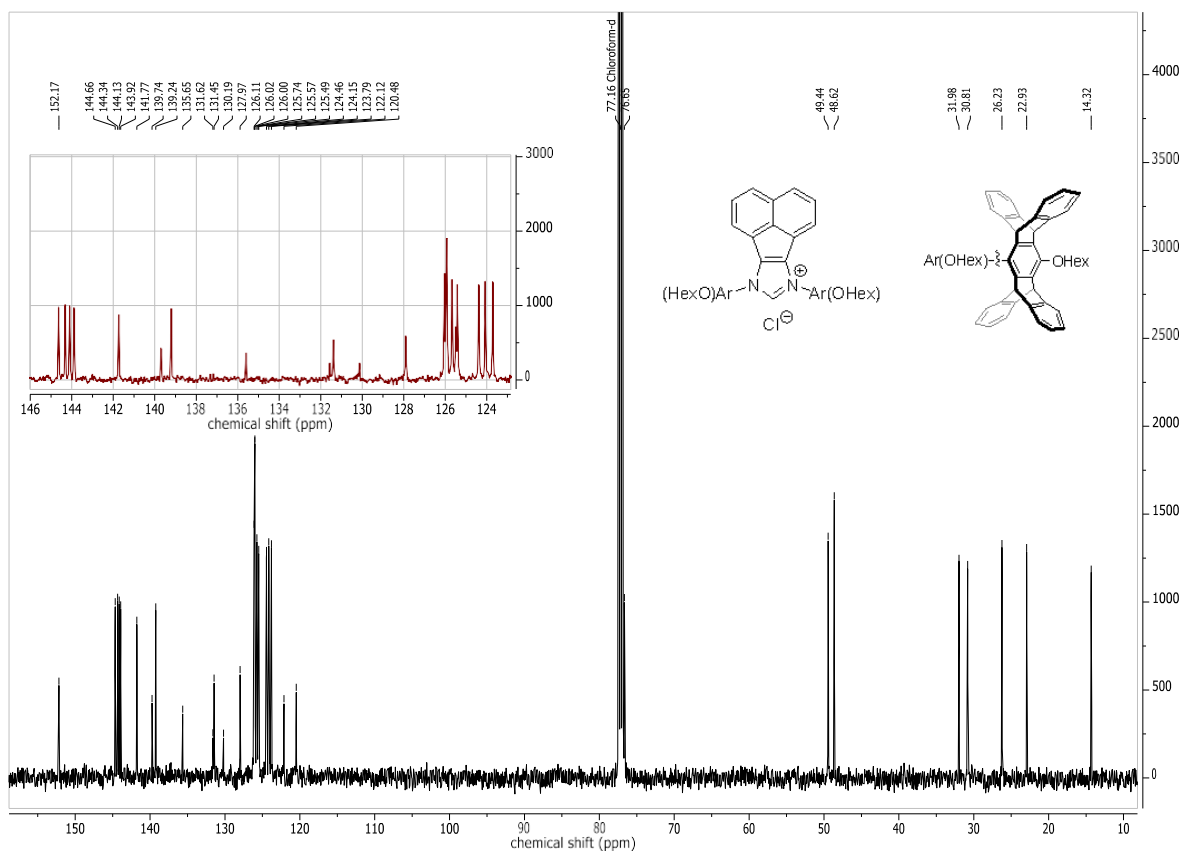
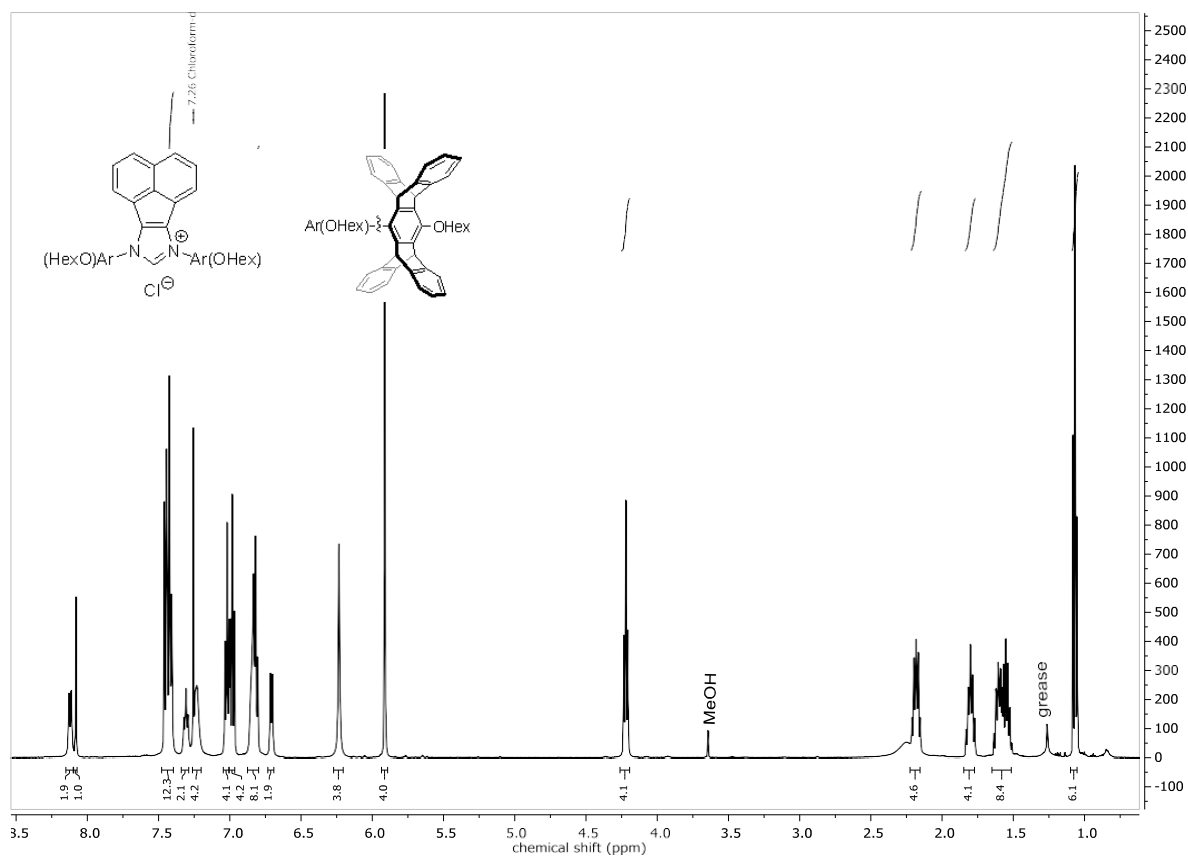


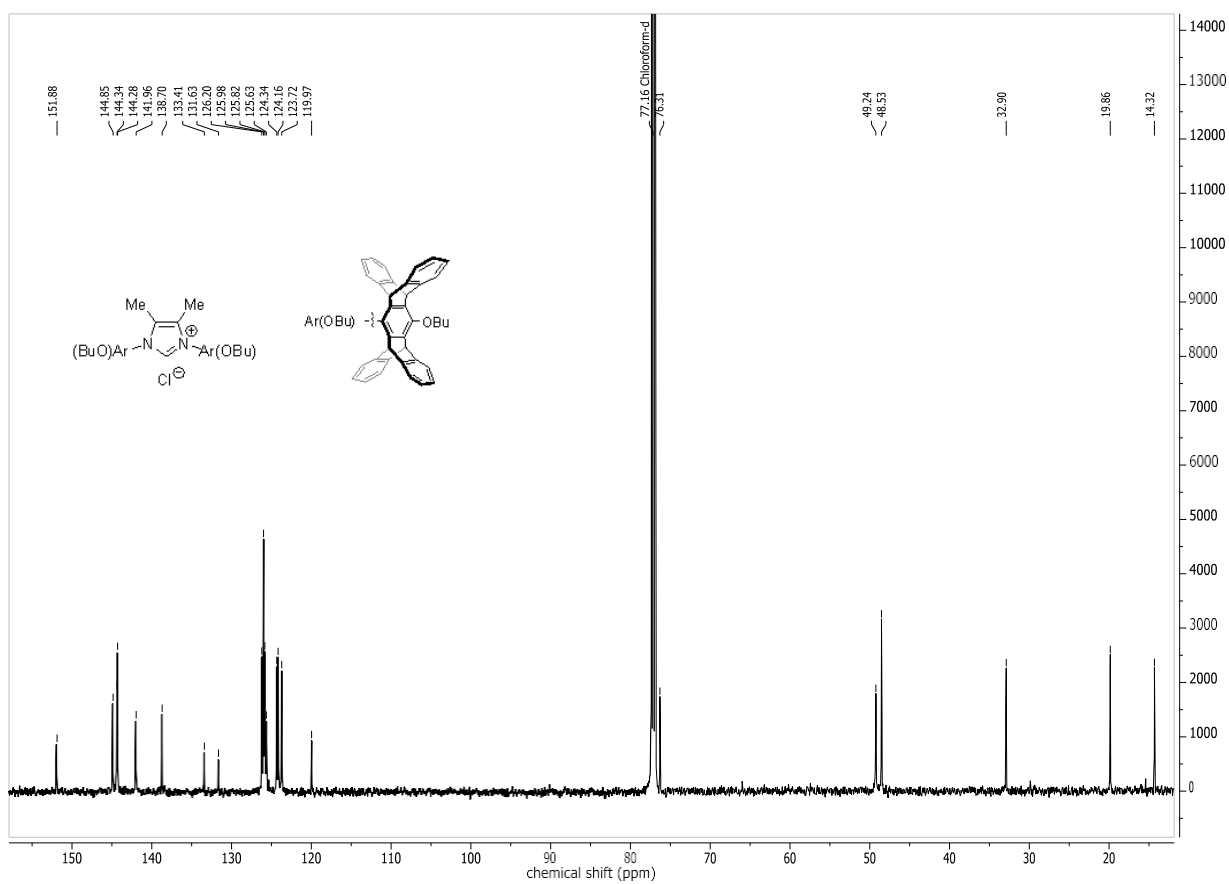
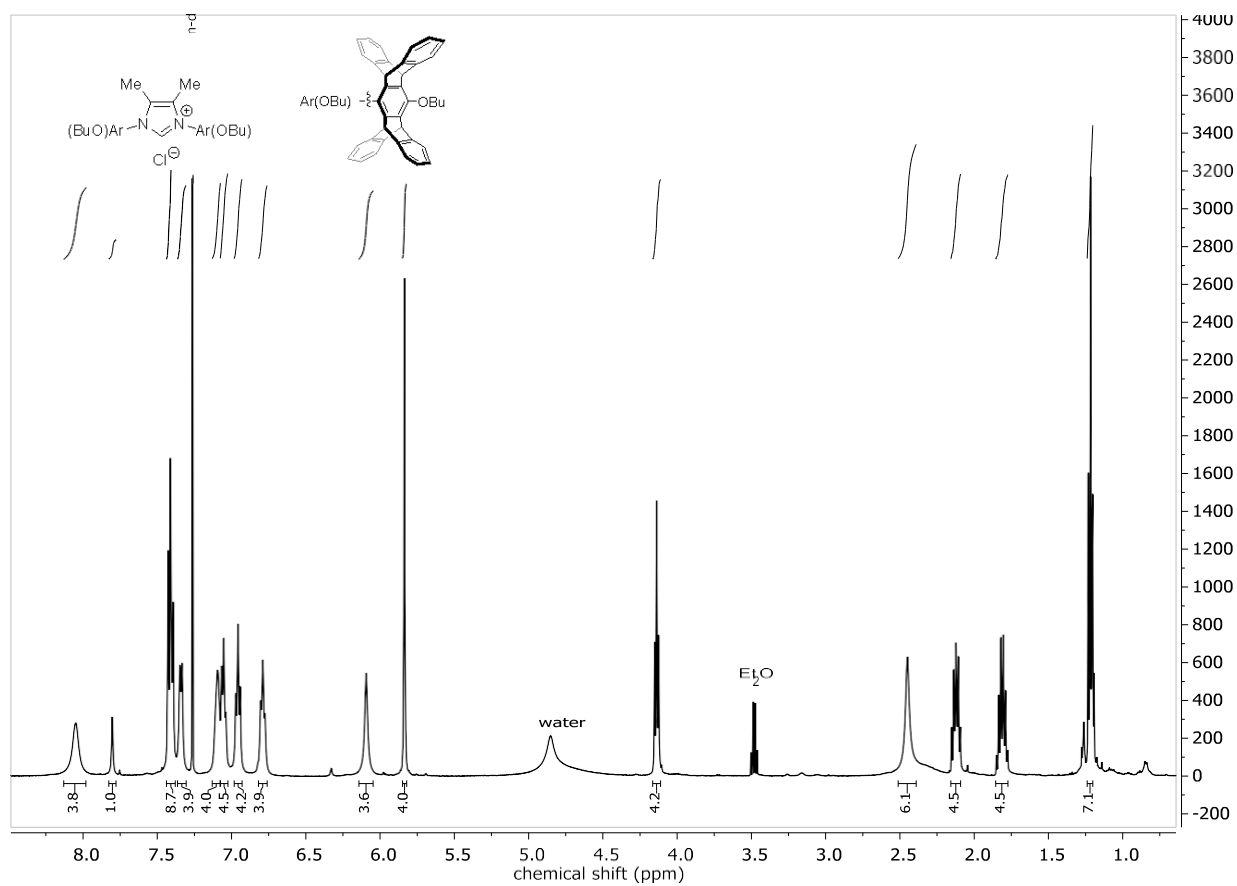


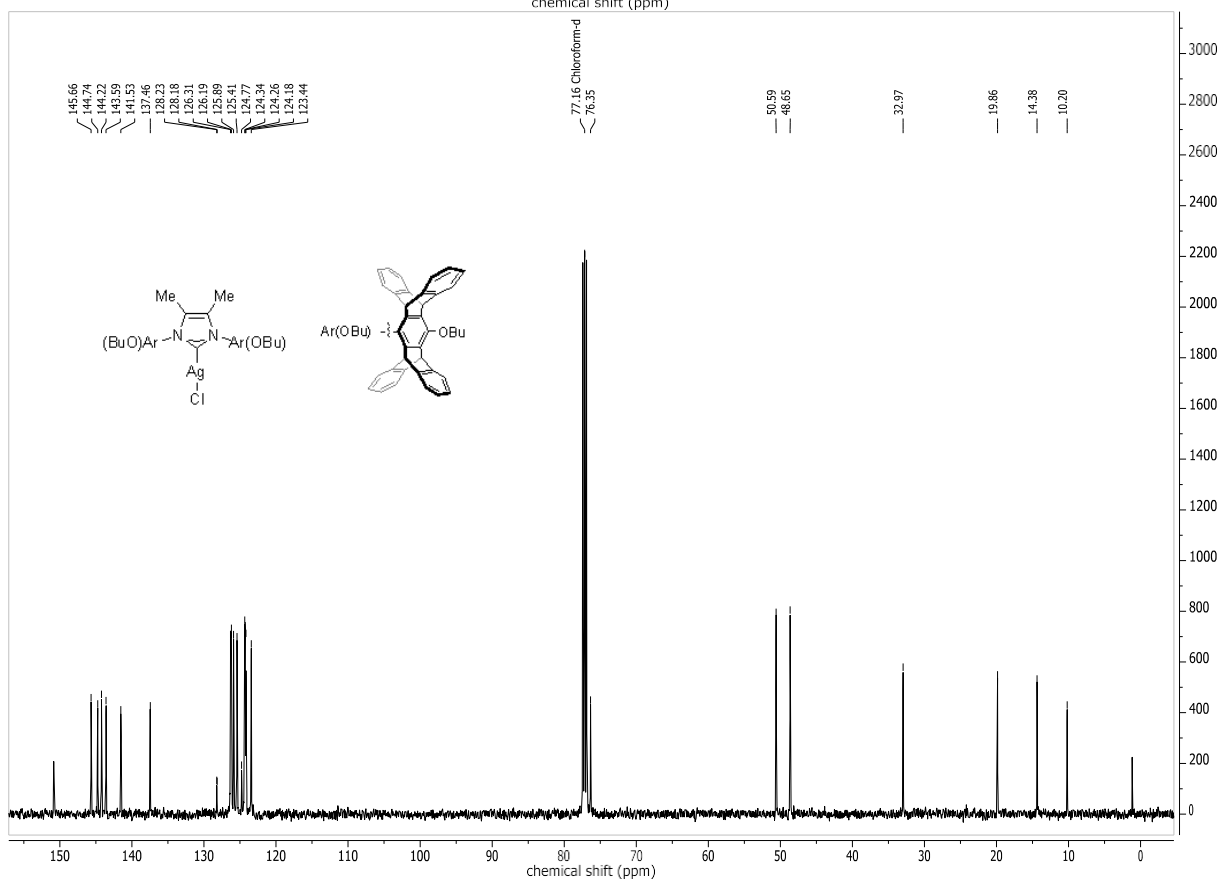
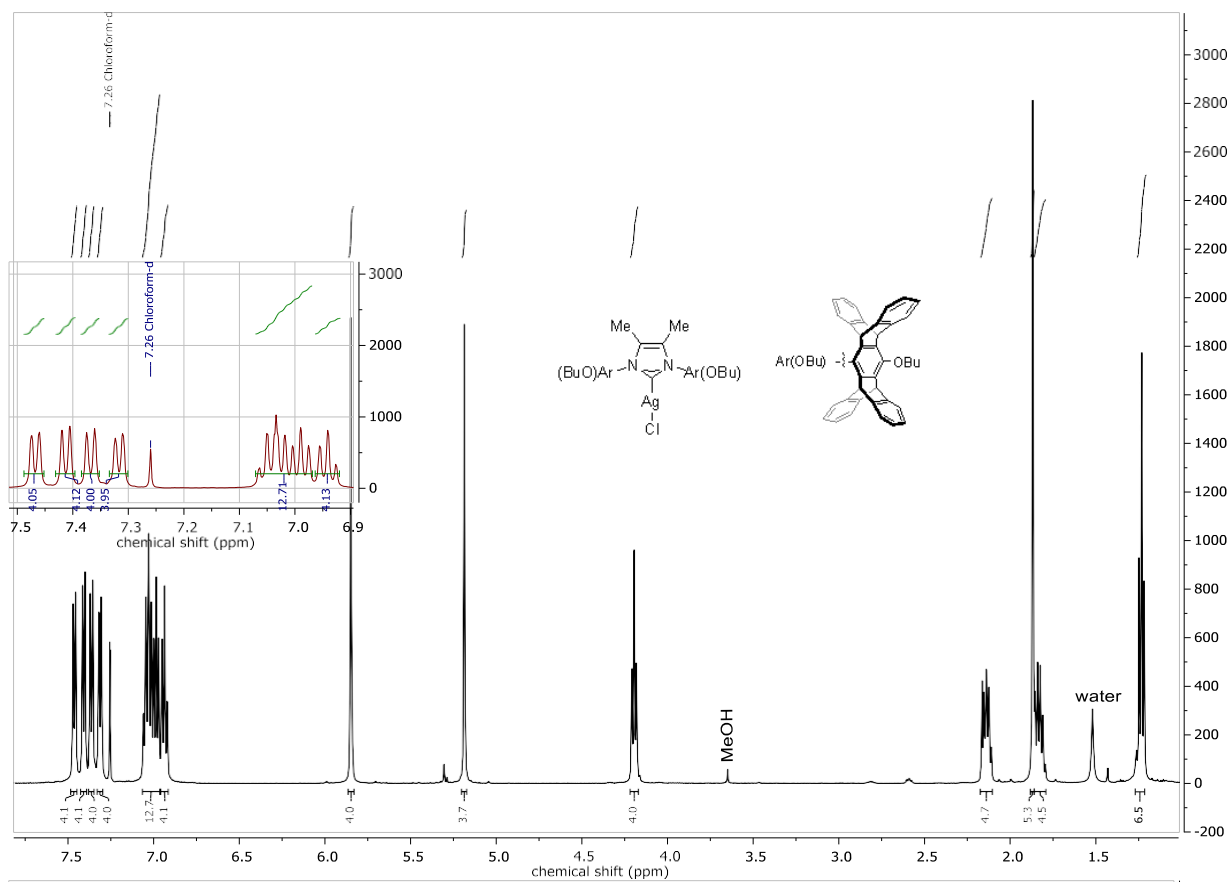


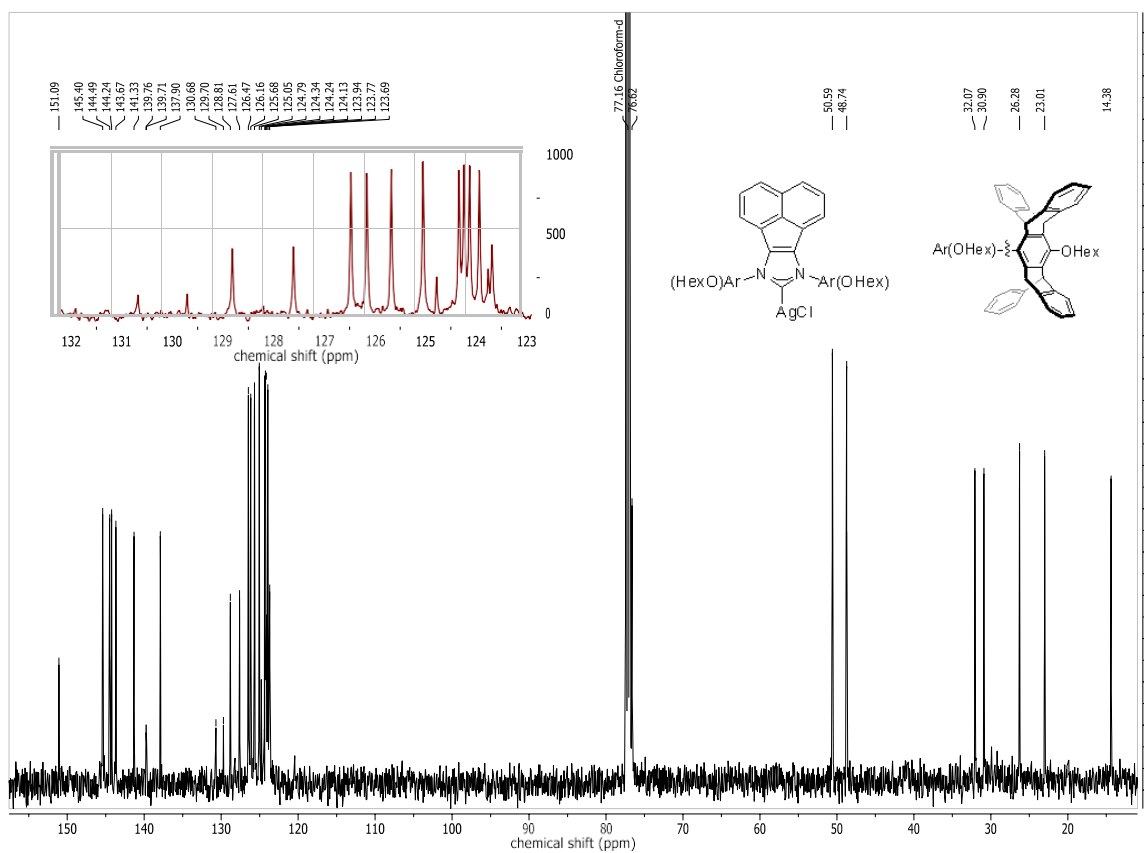
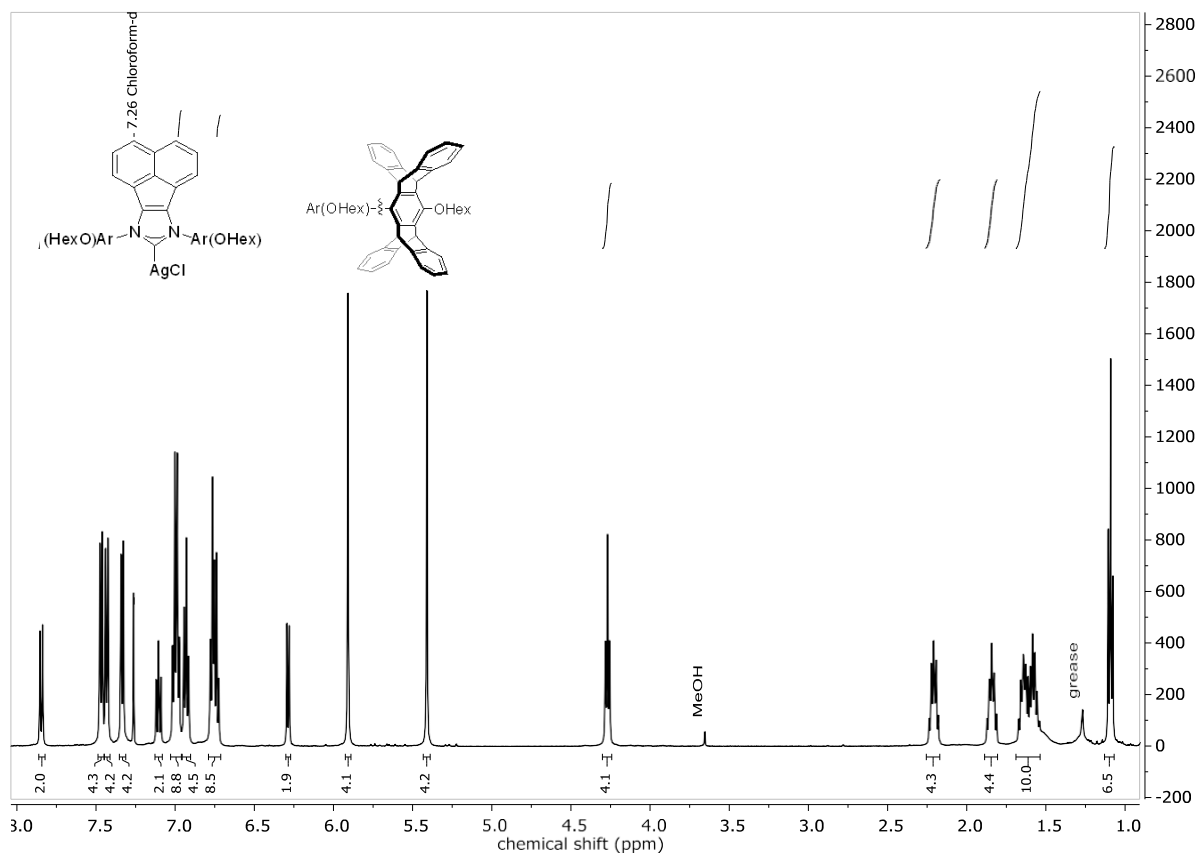


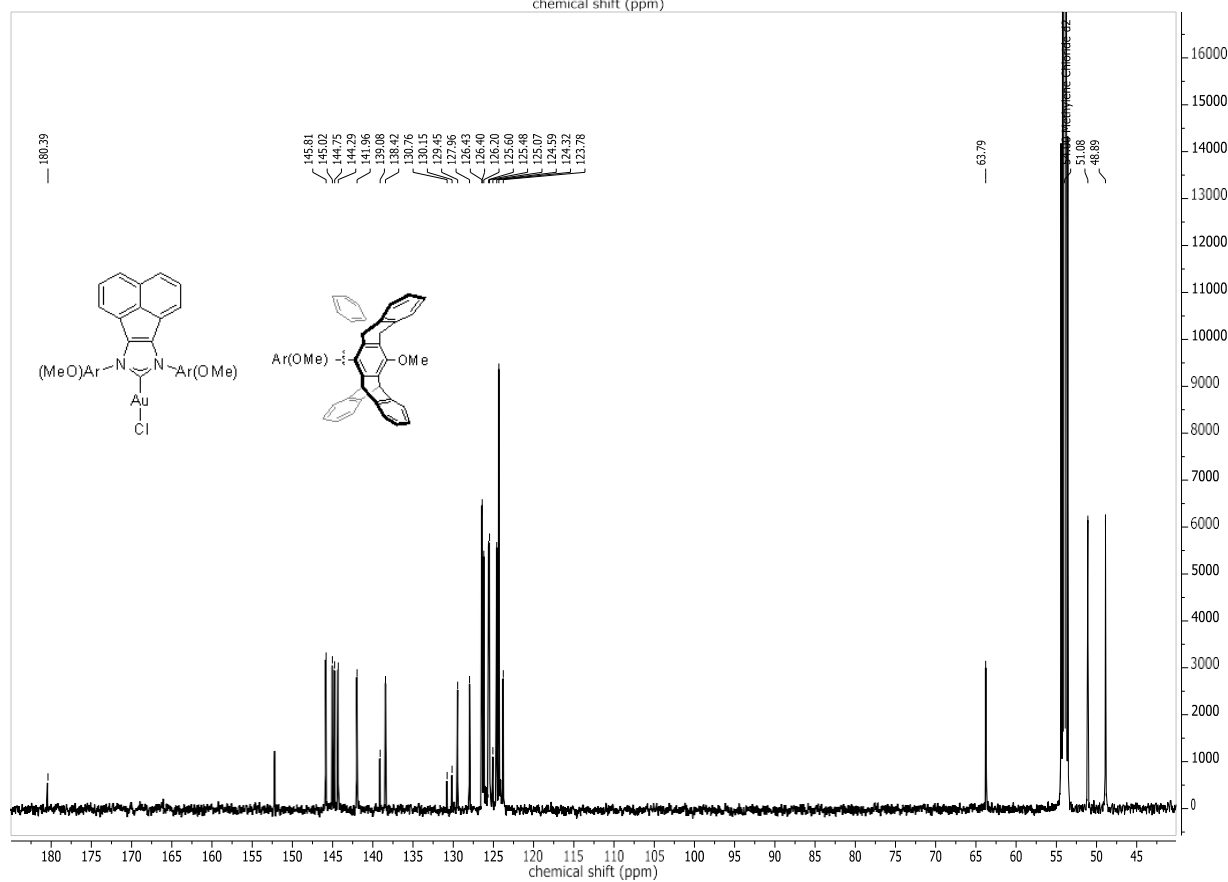
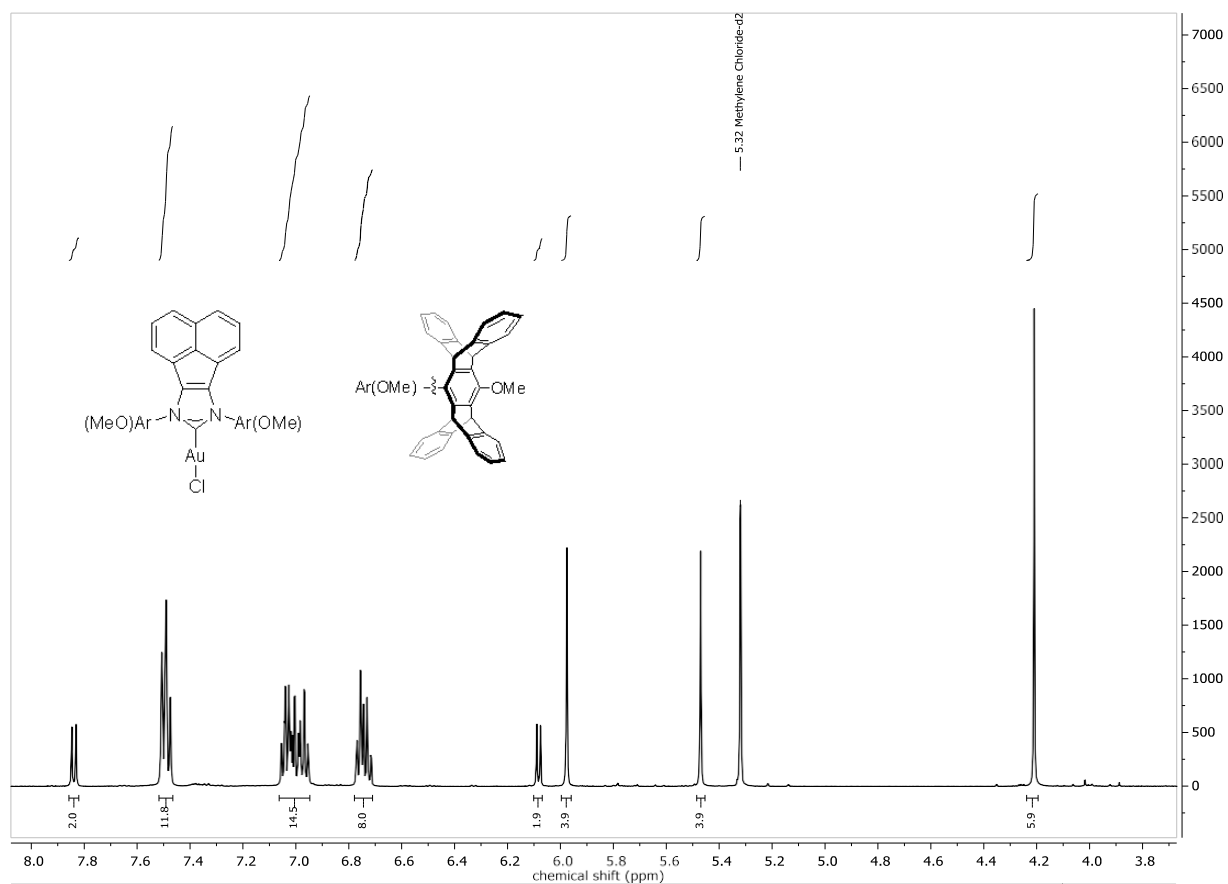


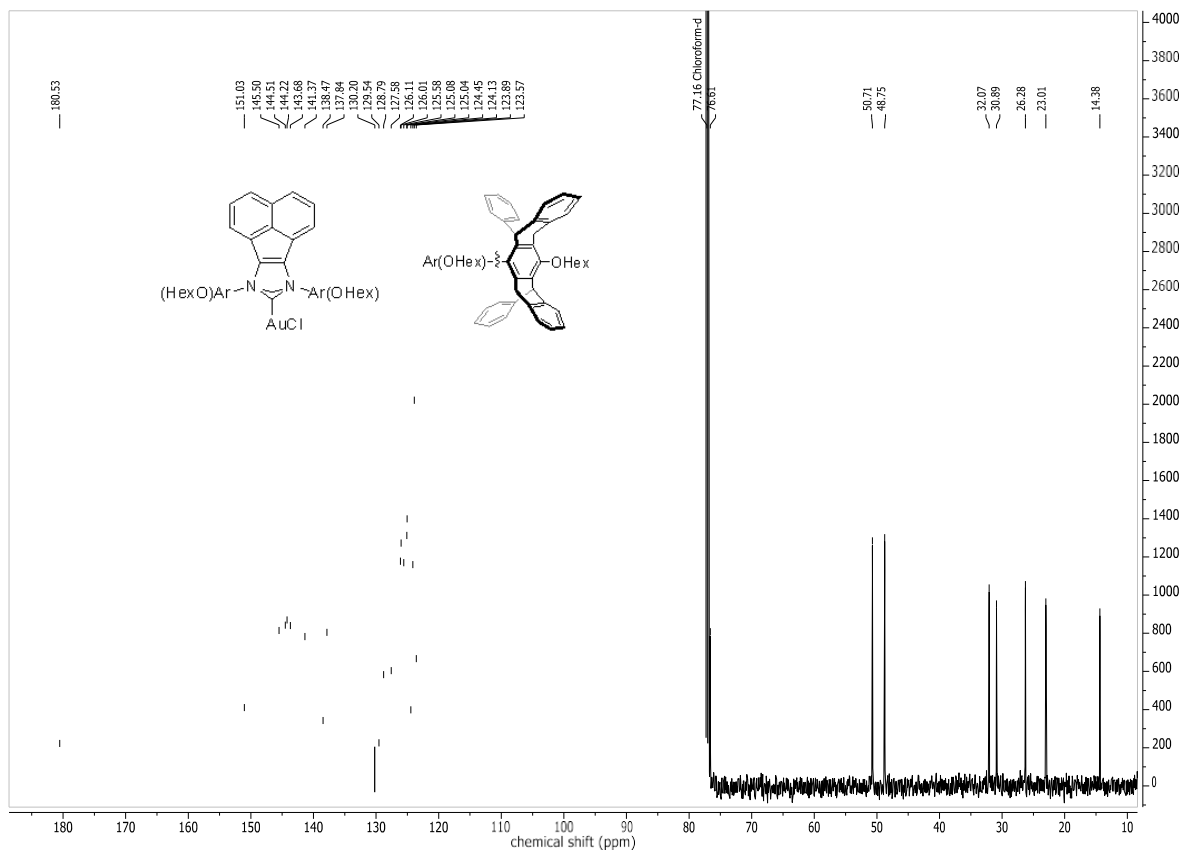
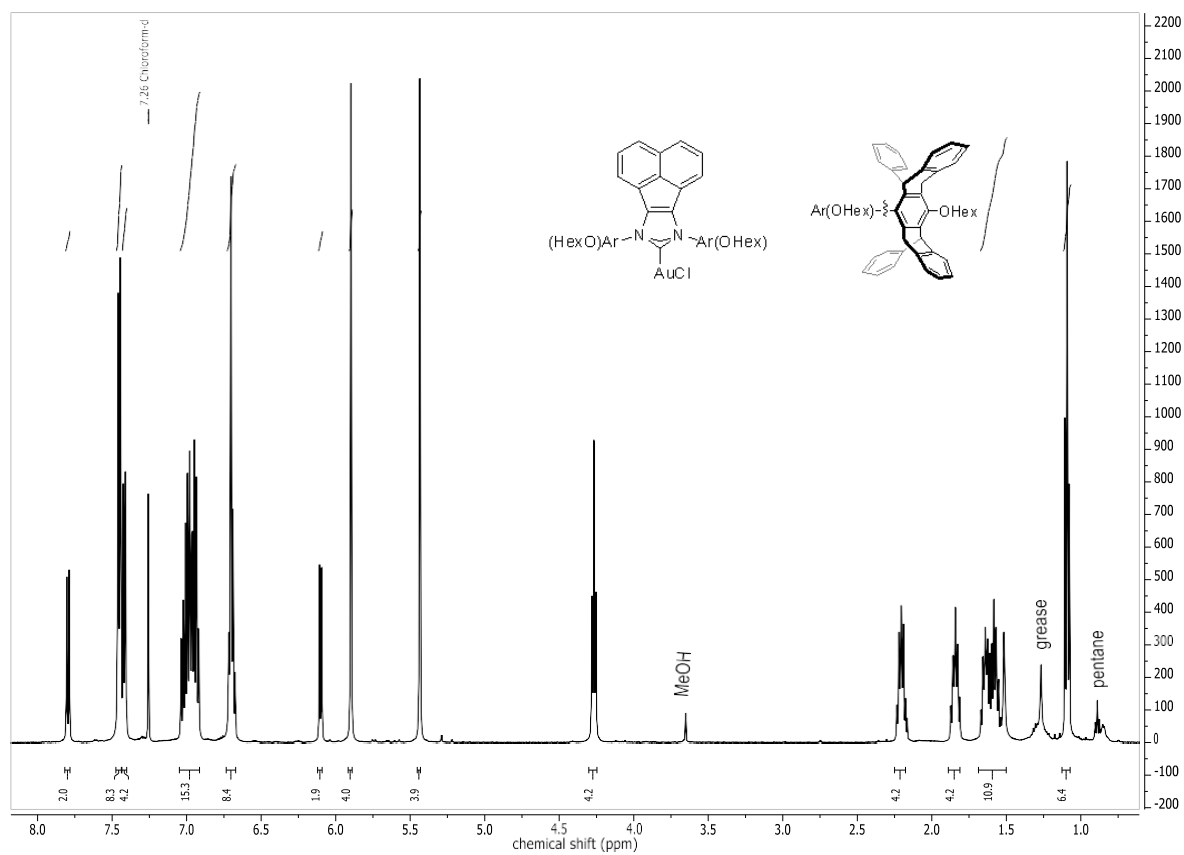


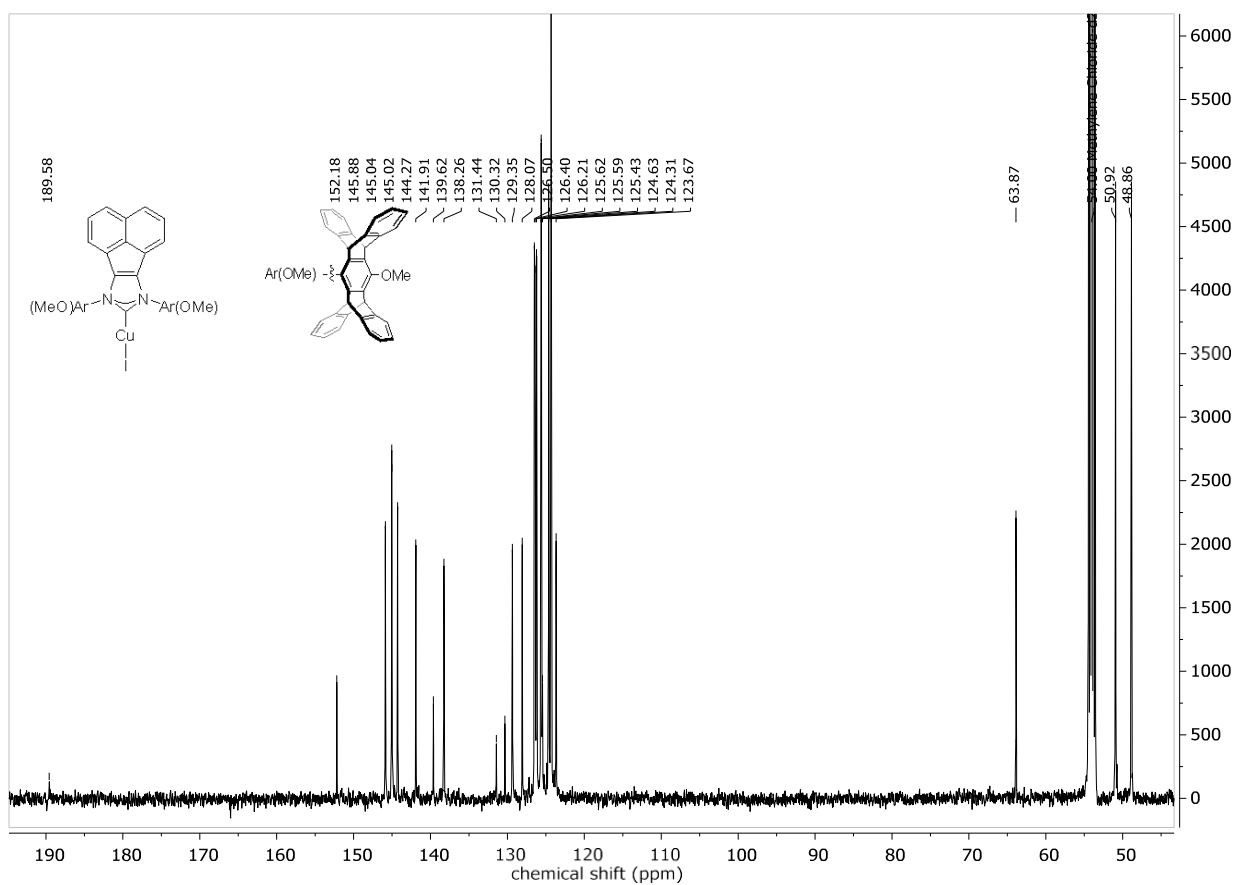
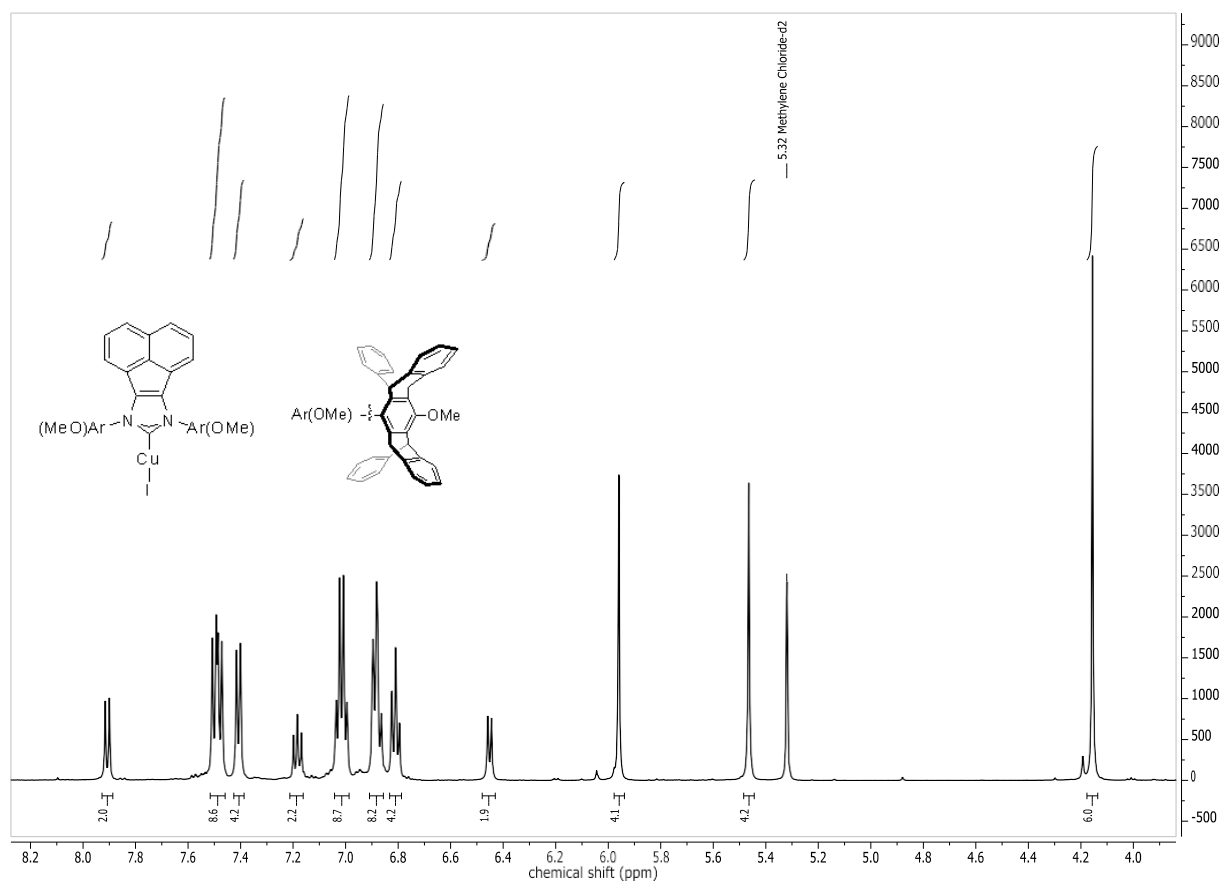


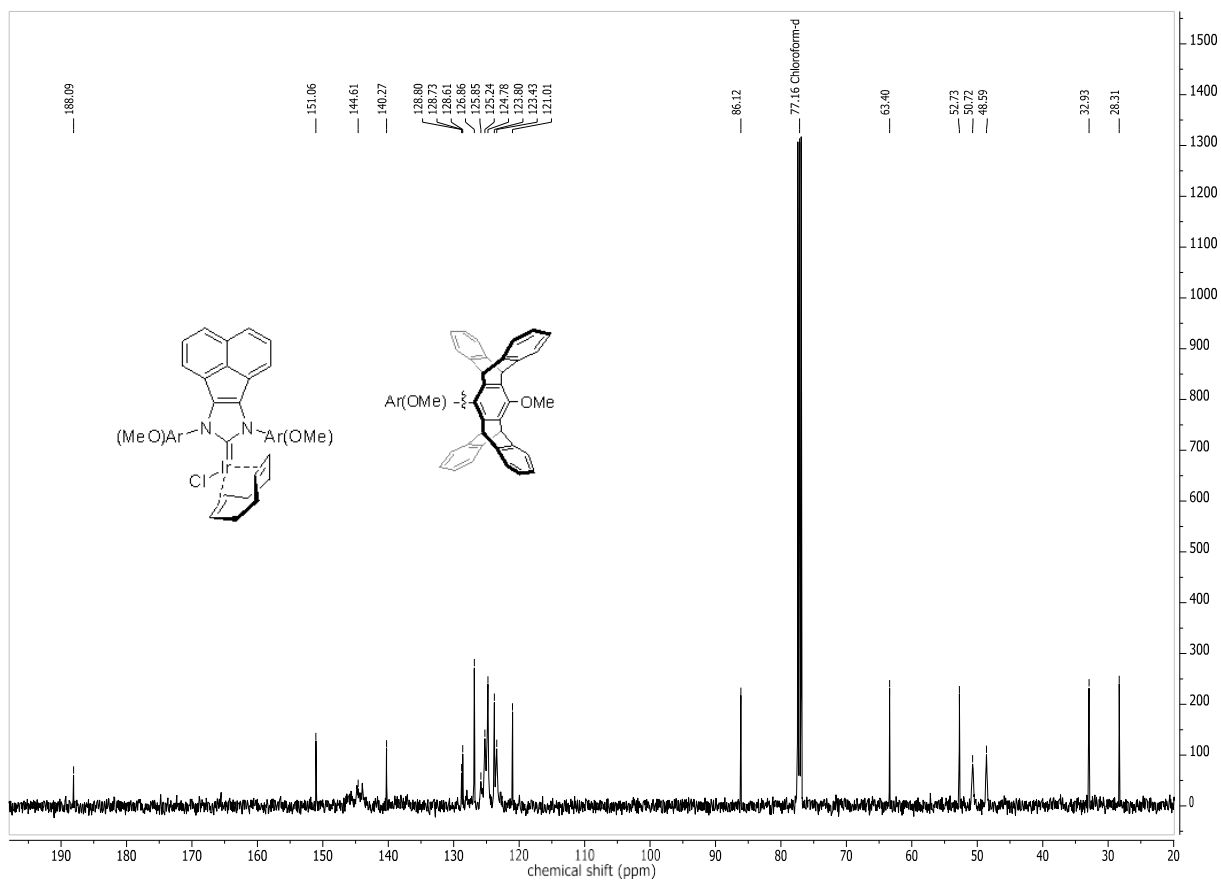
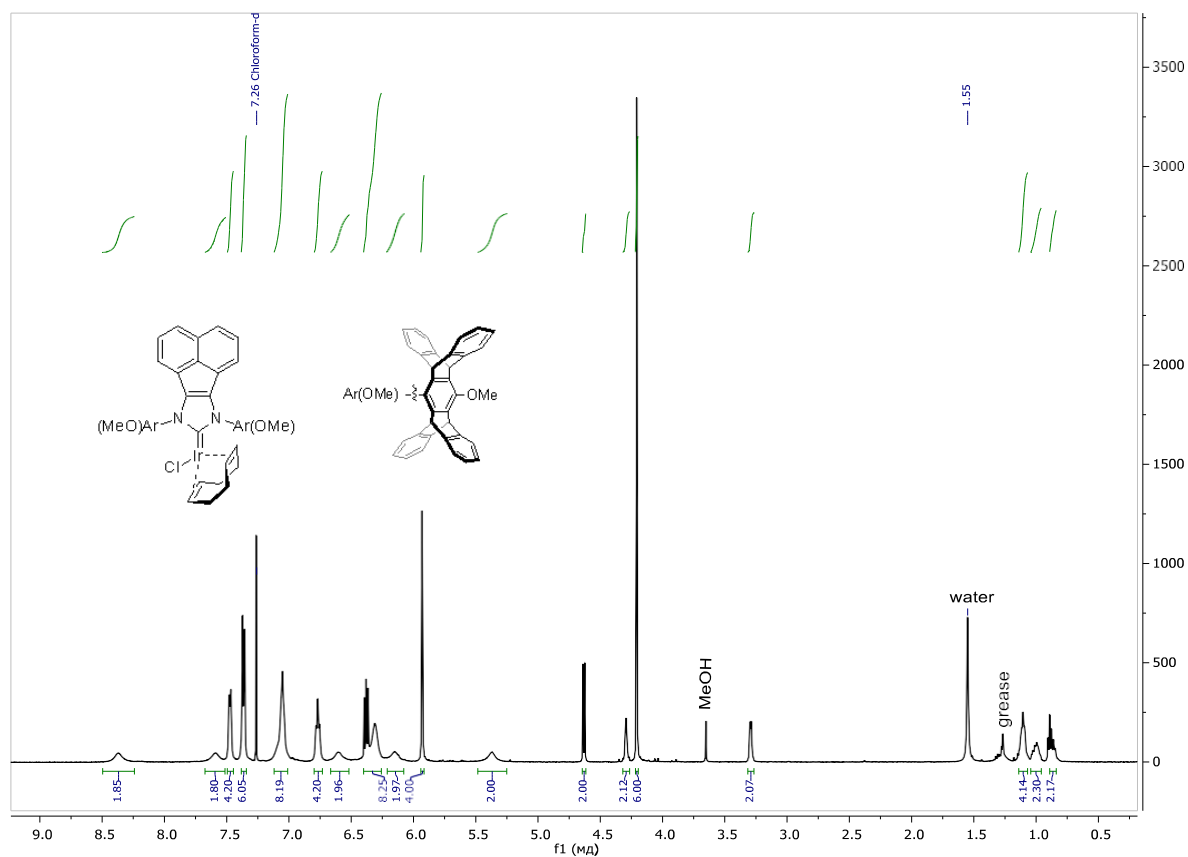


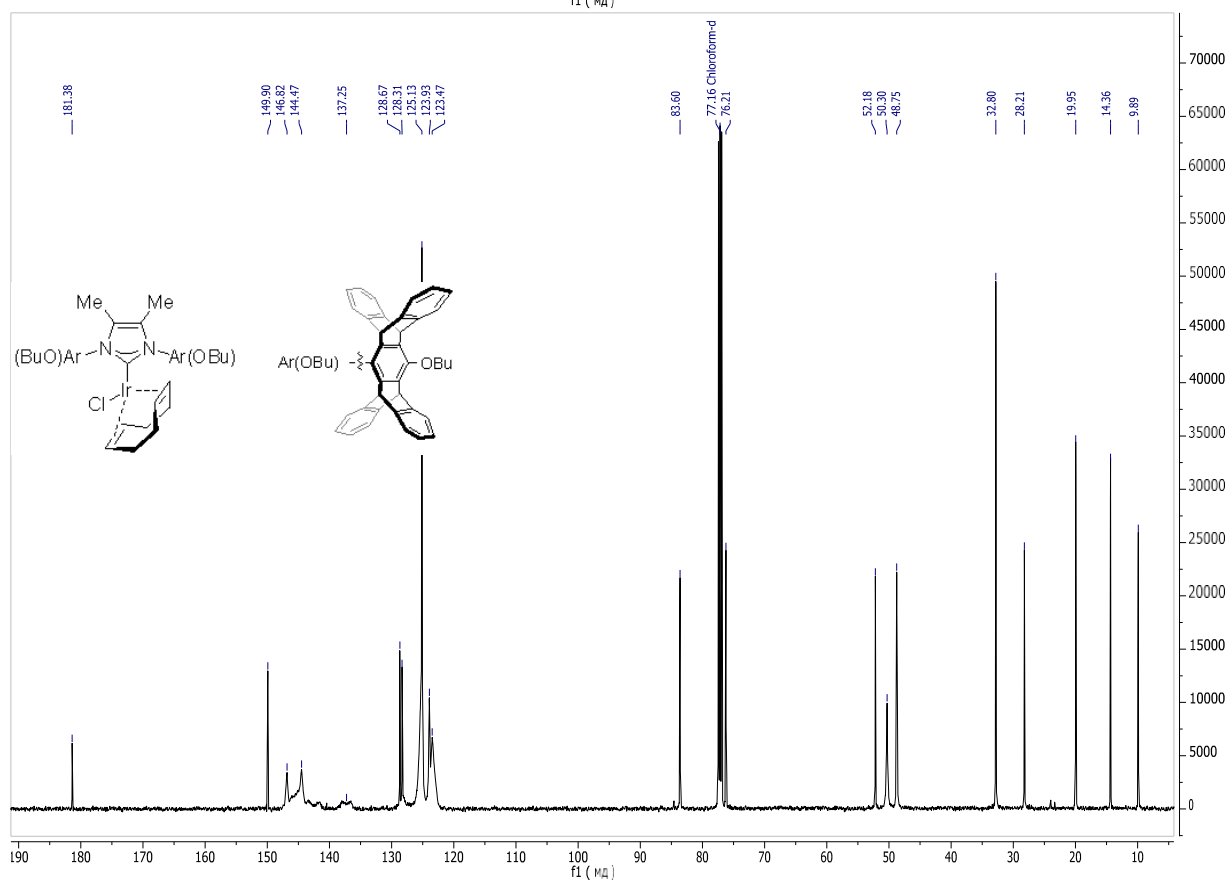
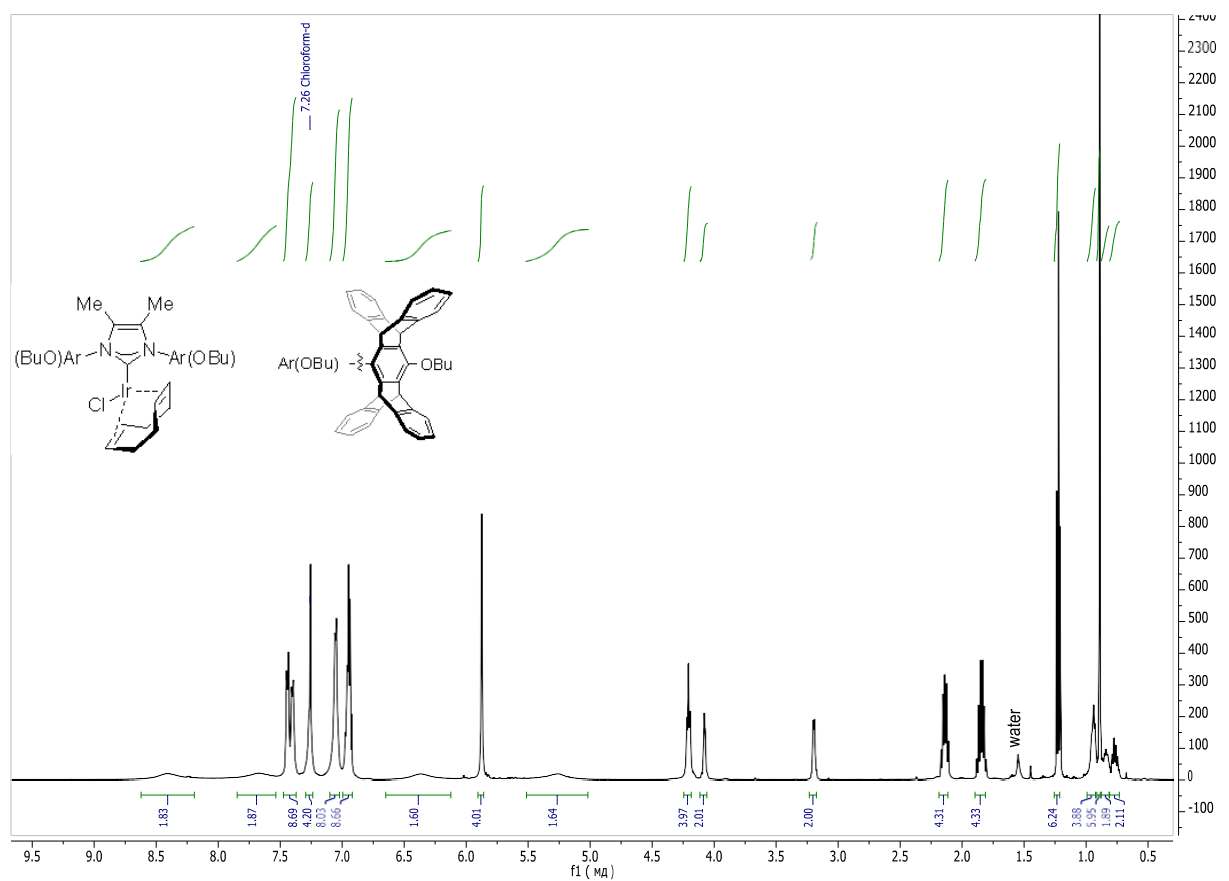


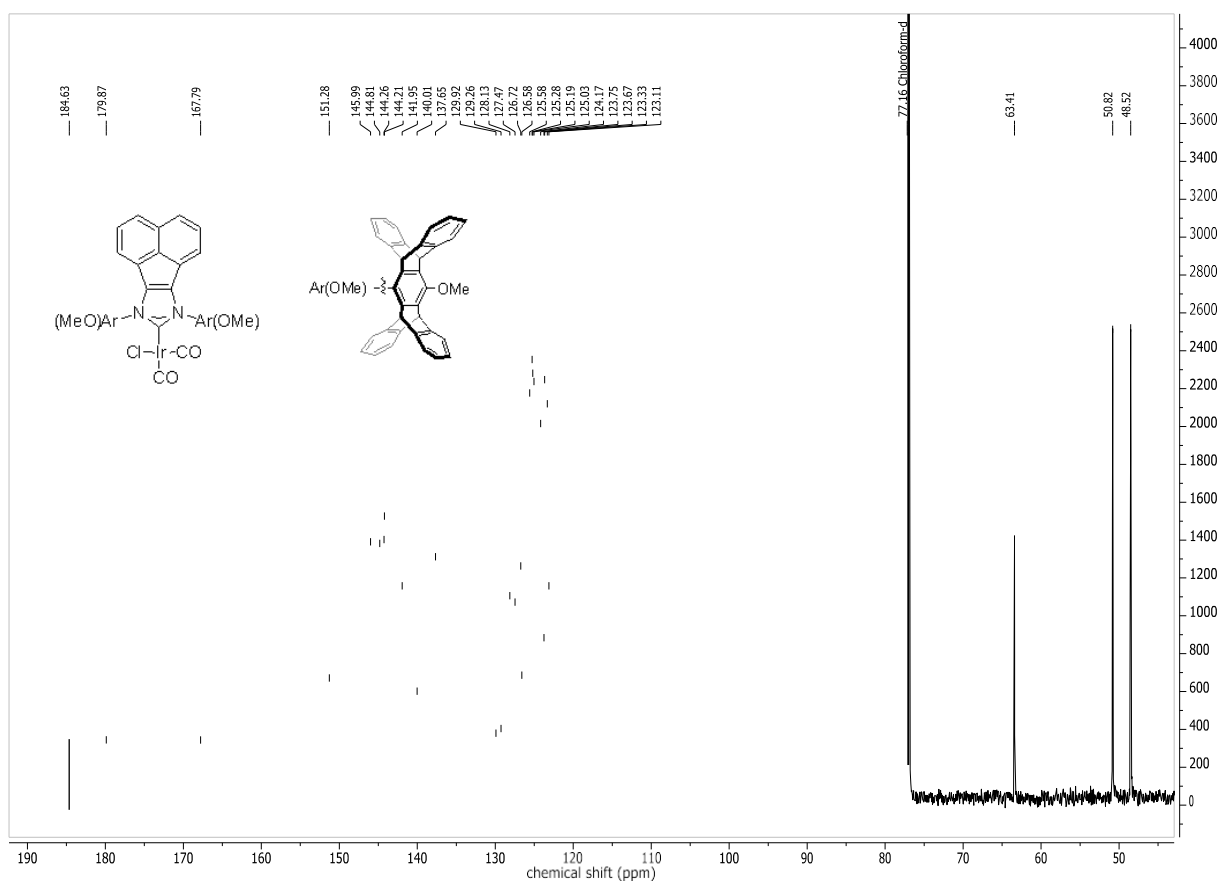
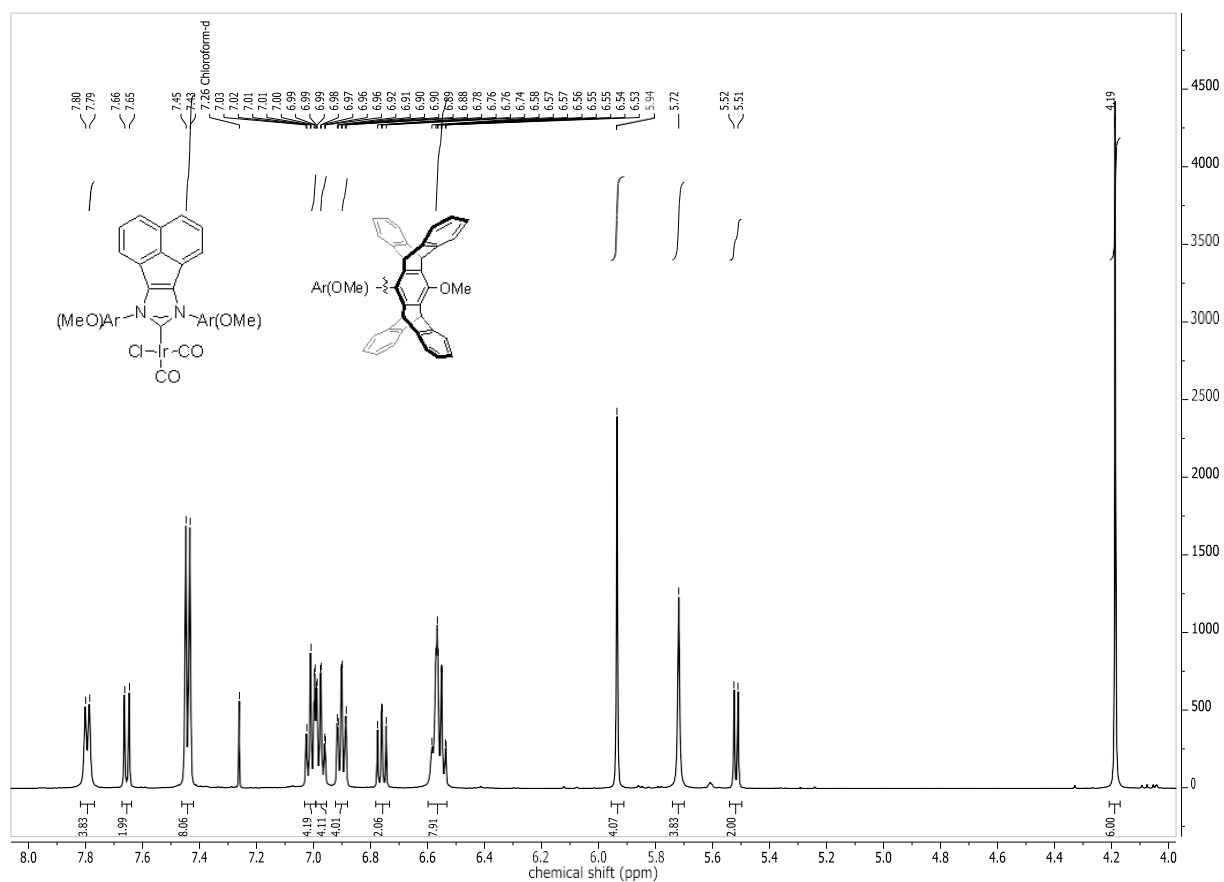


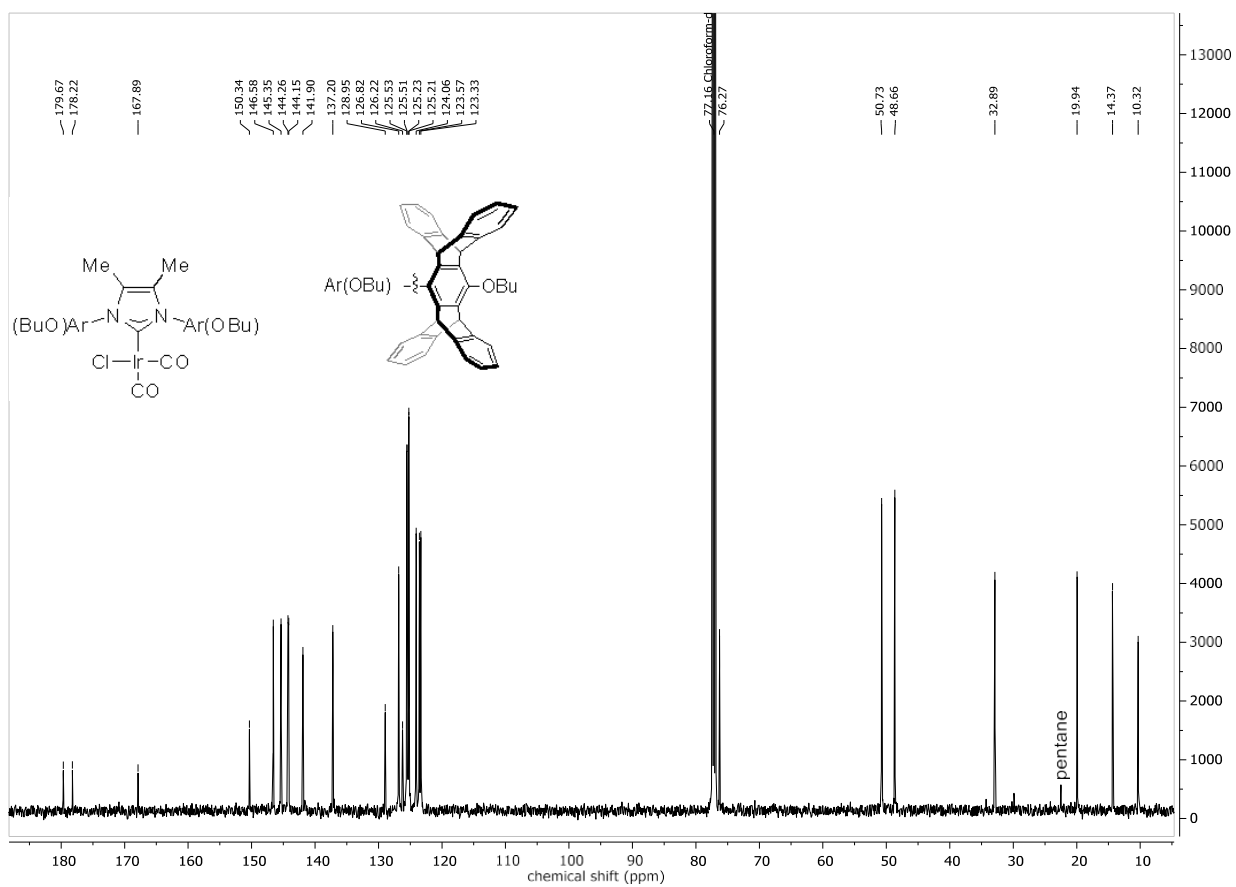
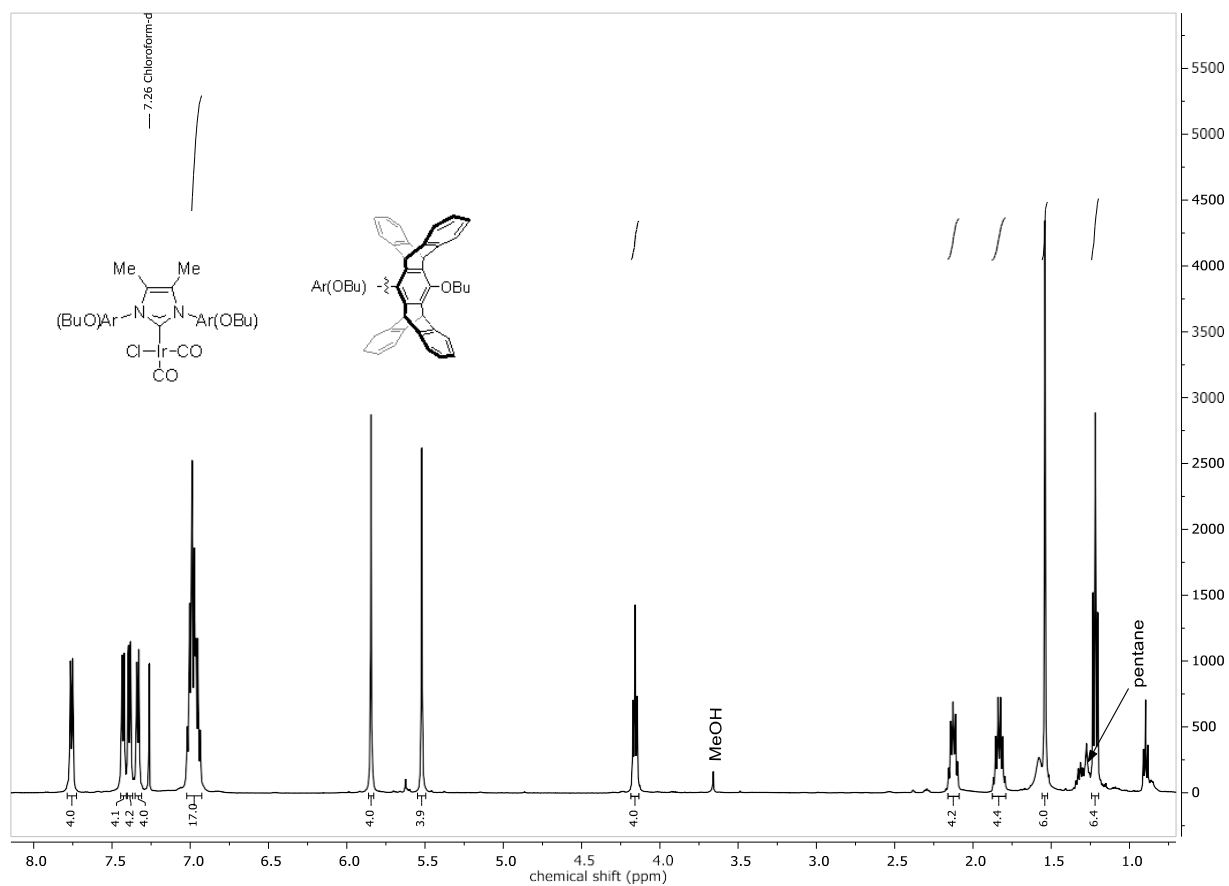


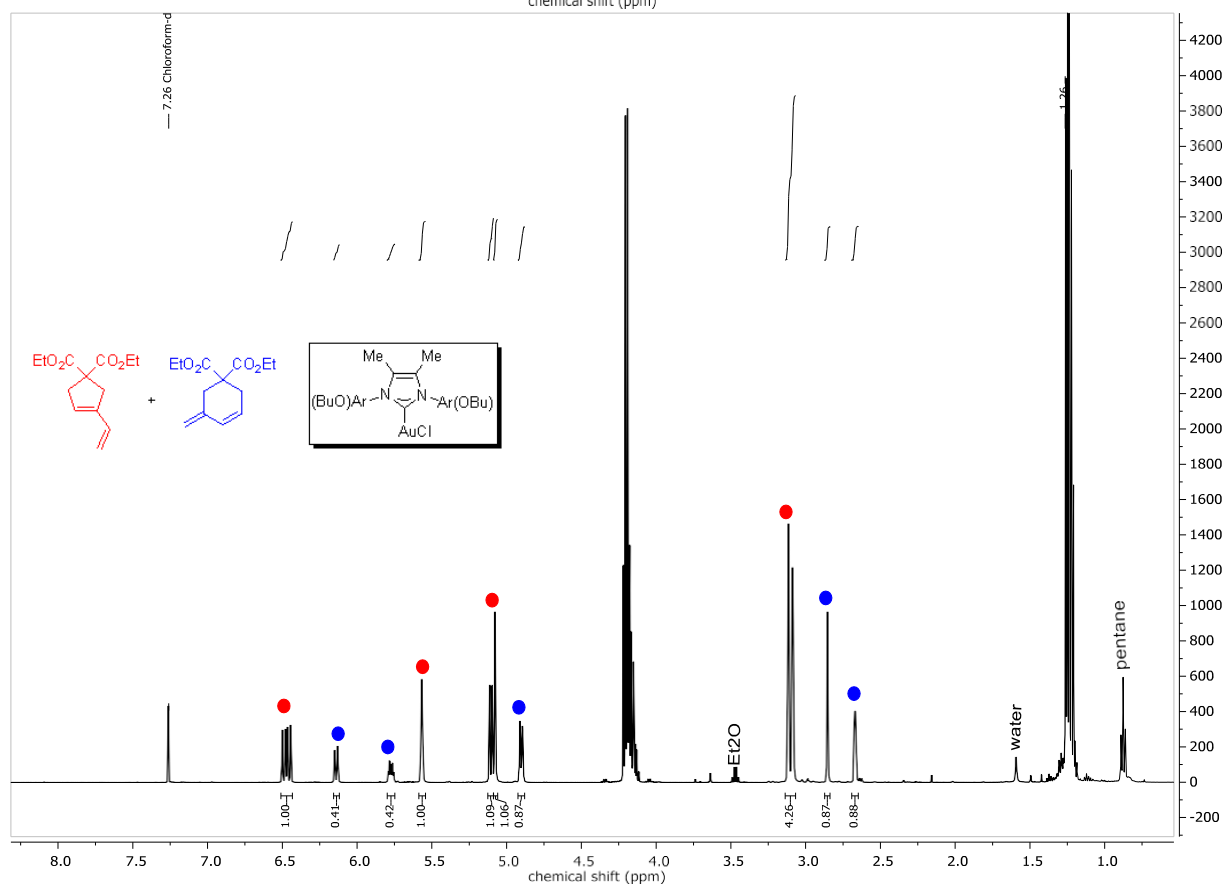
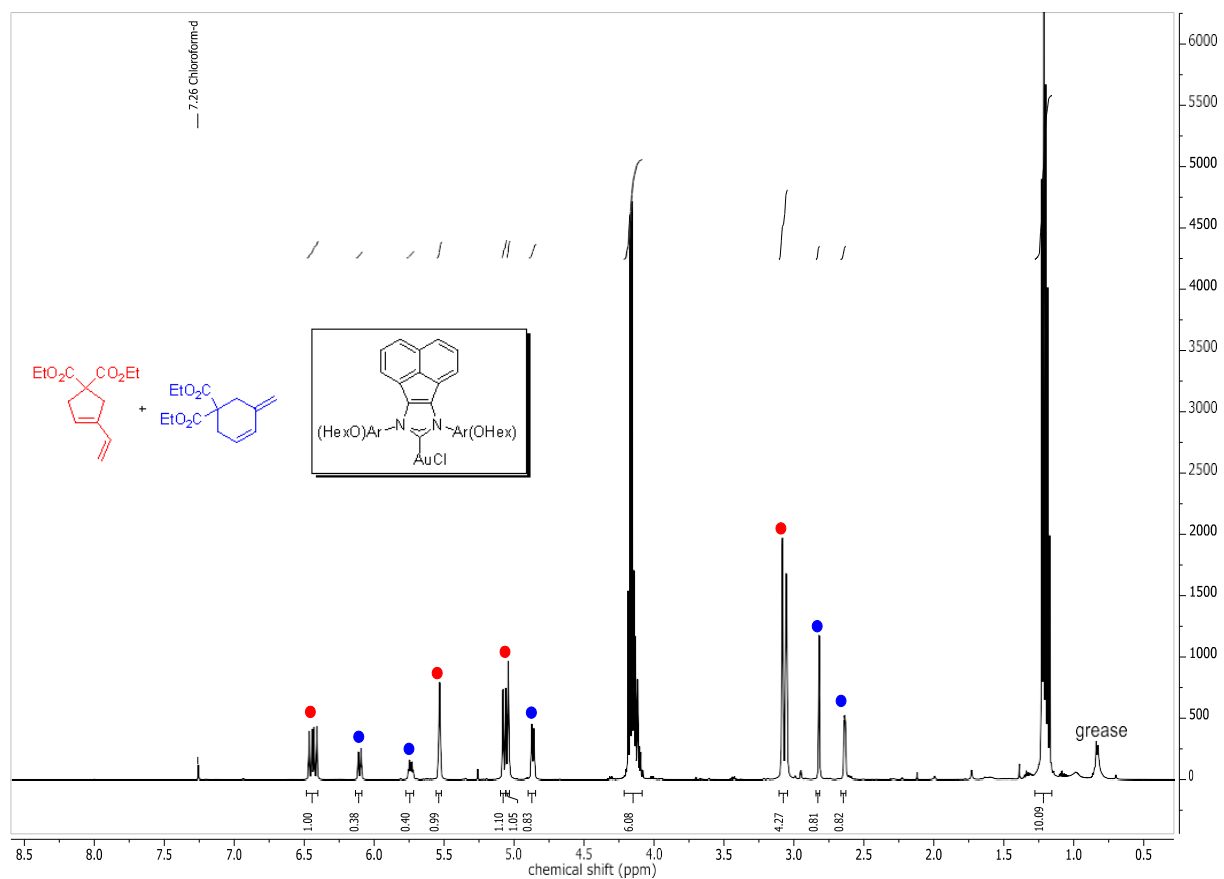


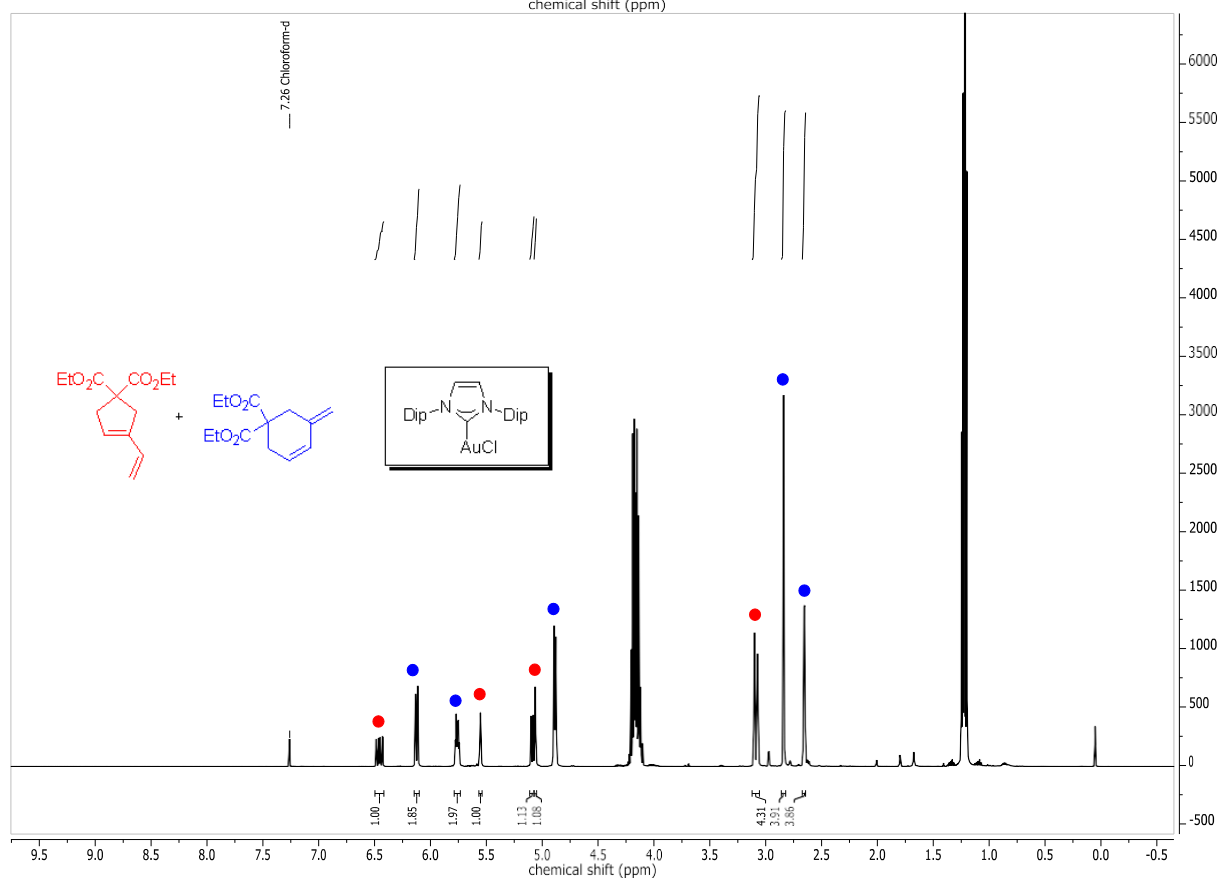
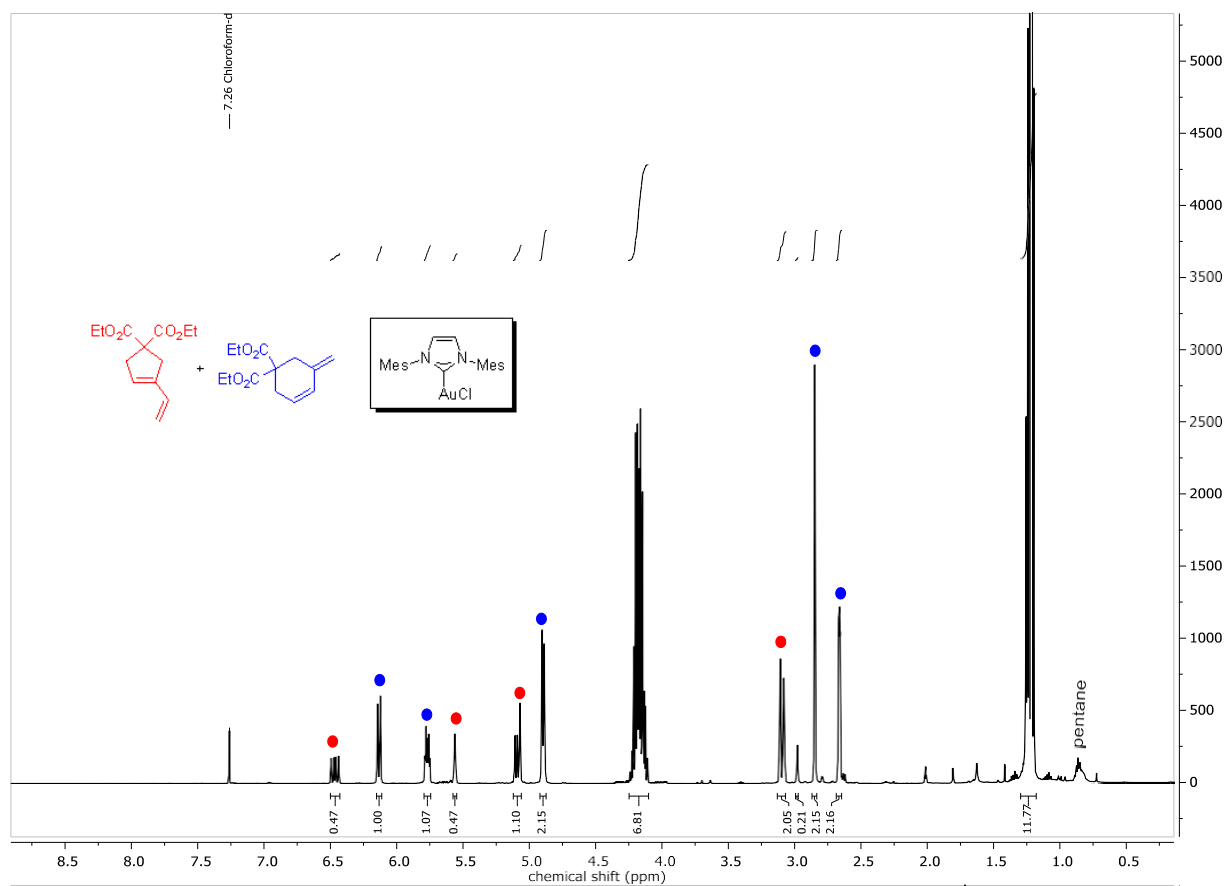




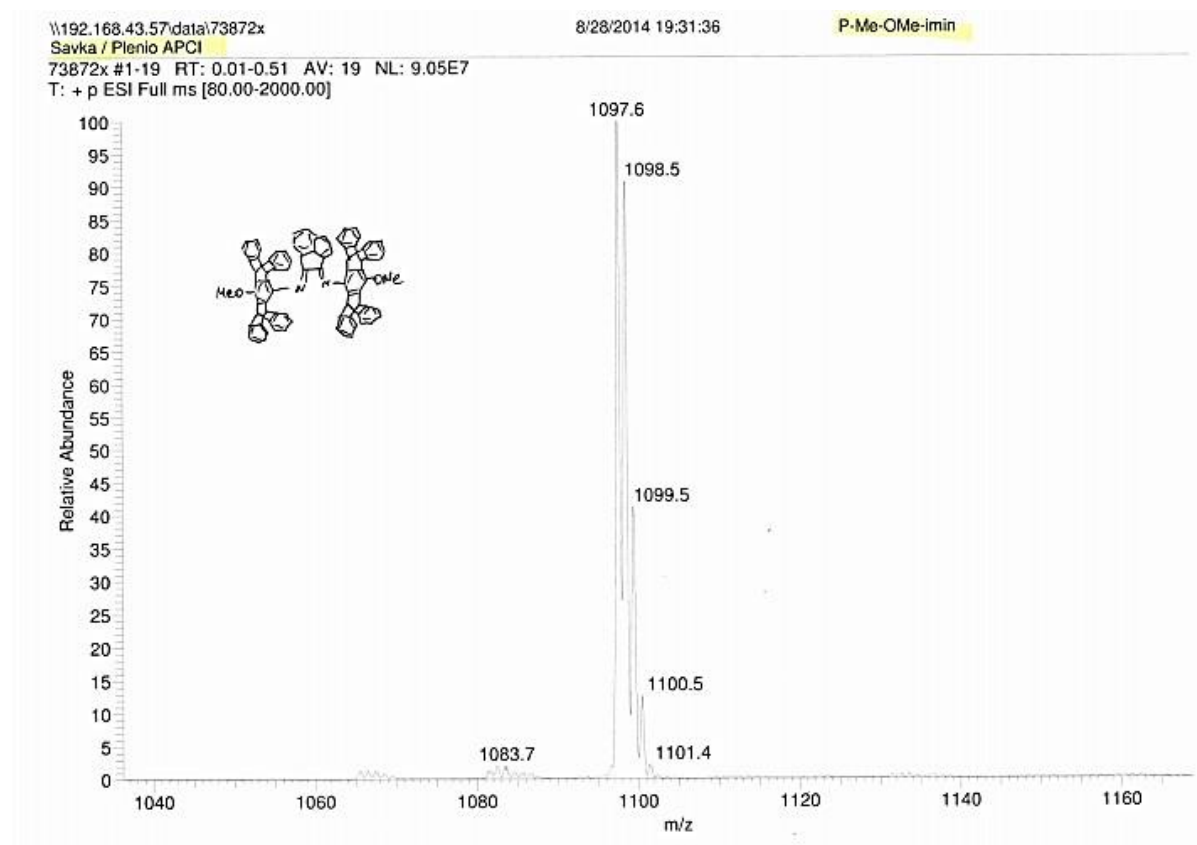
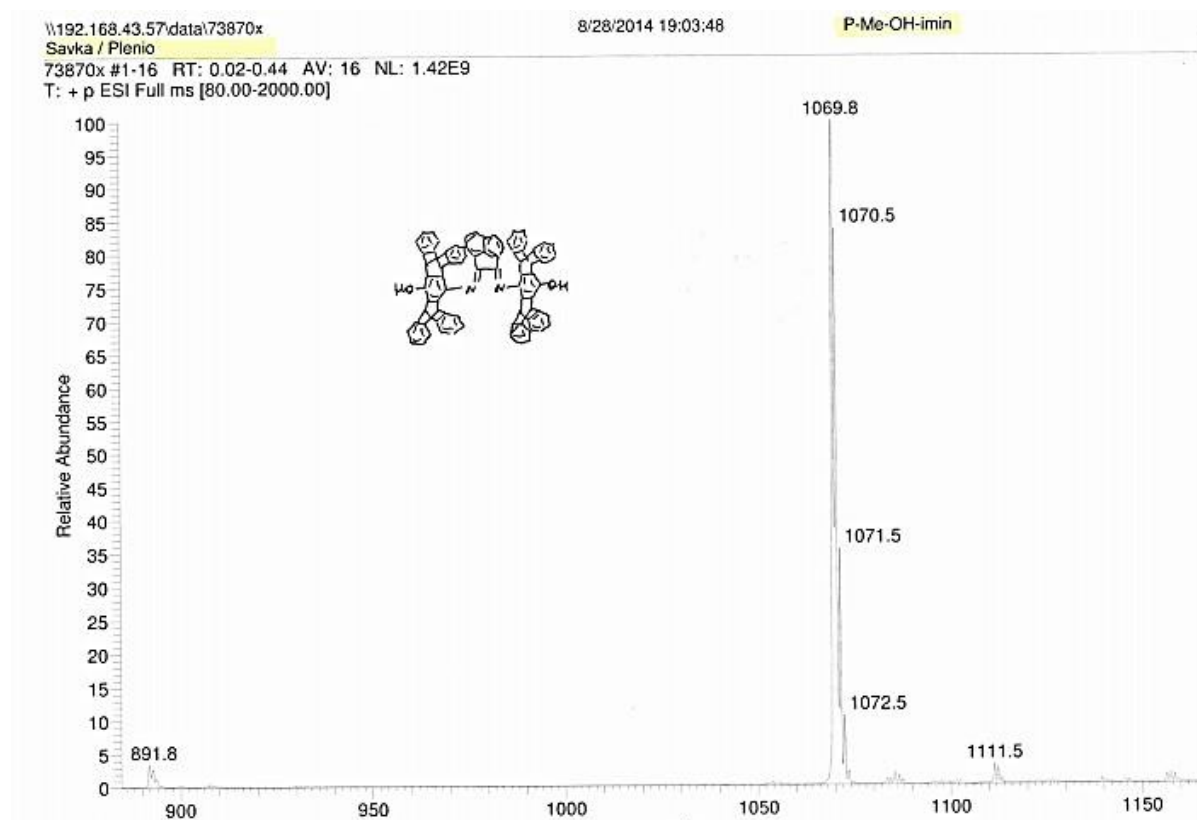


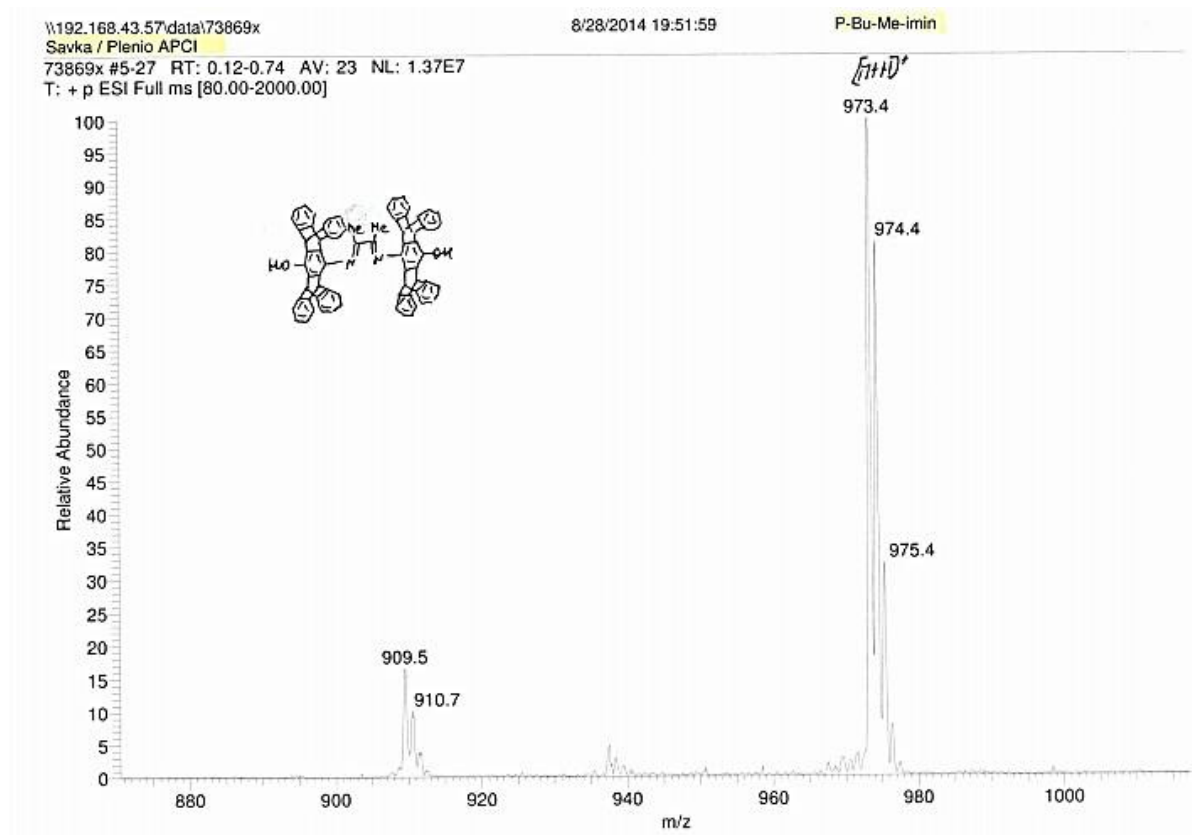
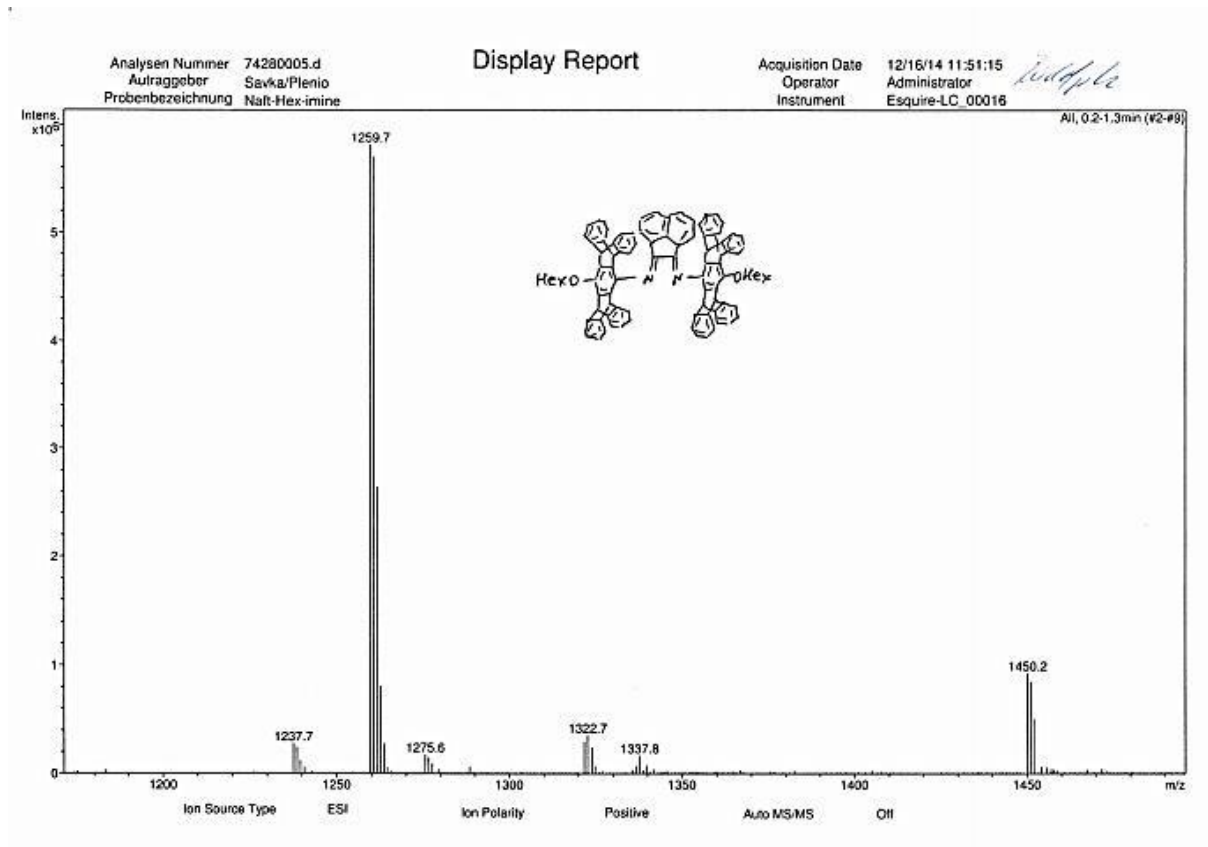






Mass spectra





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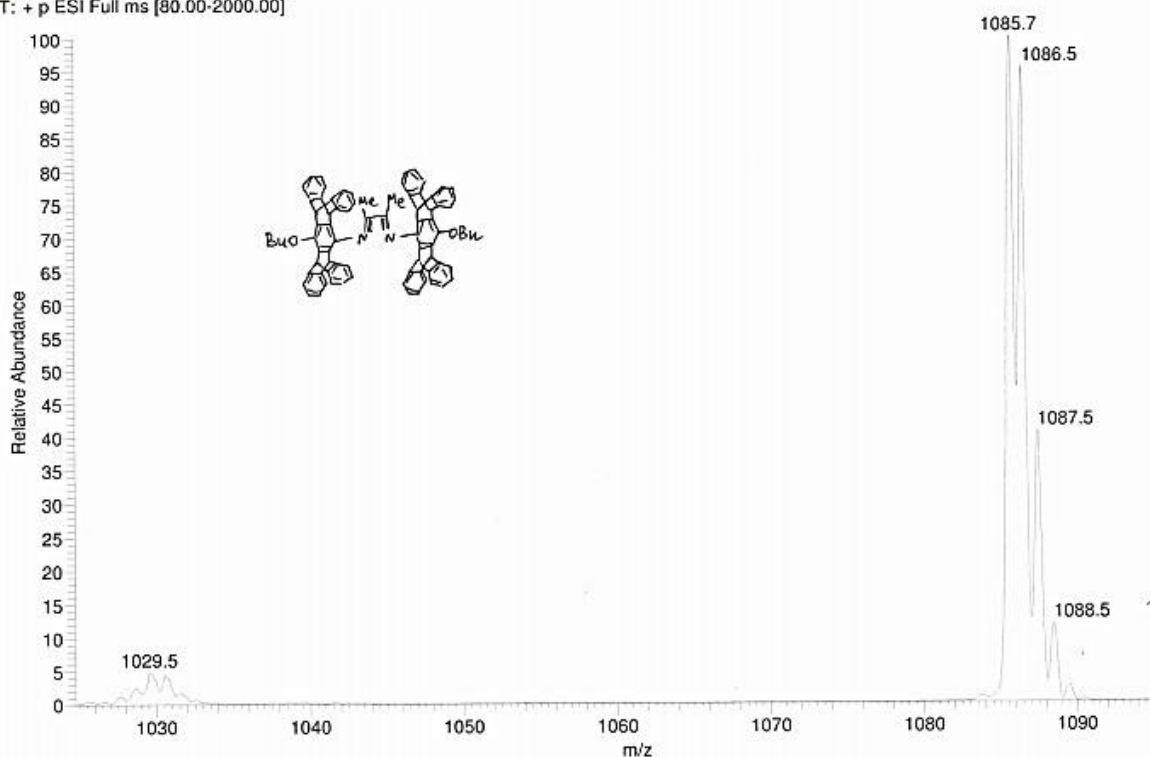
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P-Bu-OBu-imin

Savka / Plenio APCI

73871x #2-31 RT: 0.04-0.83 AV: 29 NL: 8.90E8

T: + p ESI Full ms [80.00-2000.00]



\\192.168.43.57\data\73874x

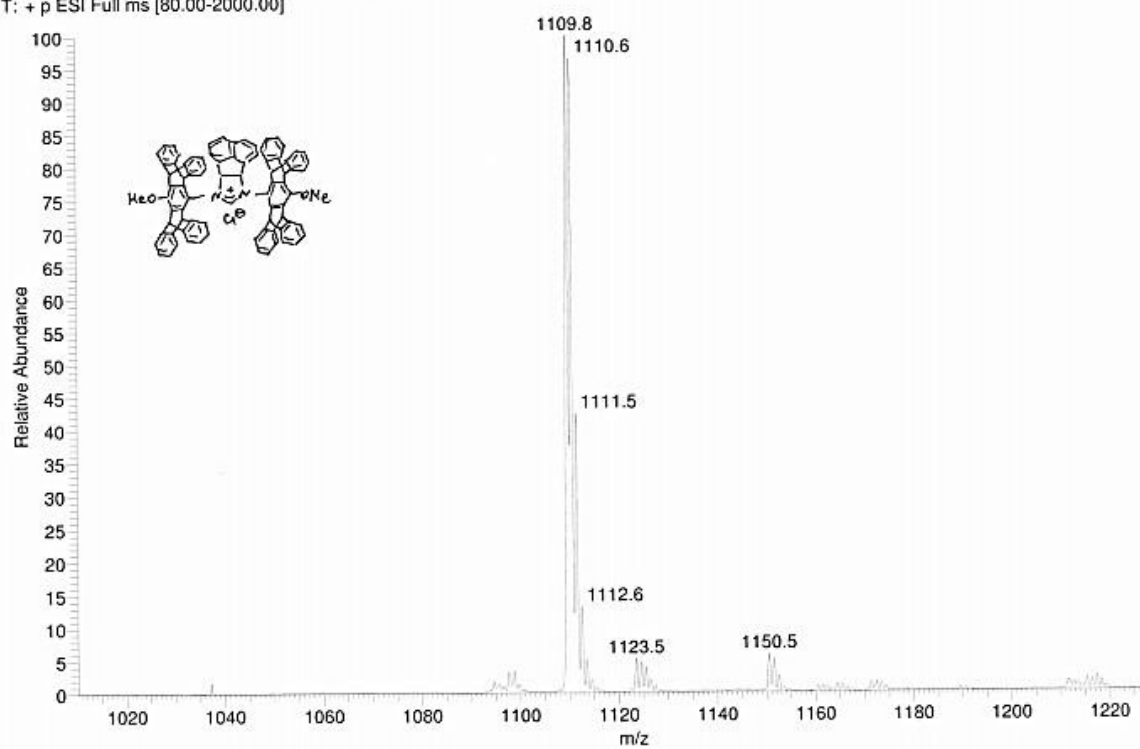
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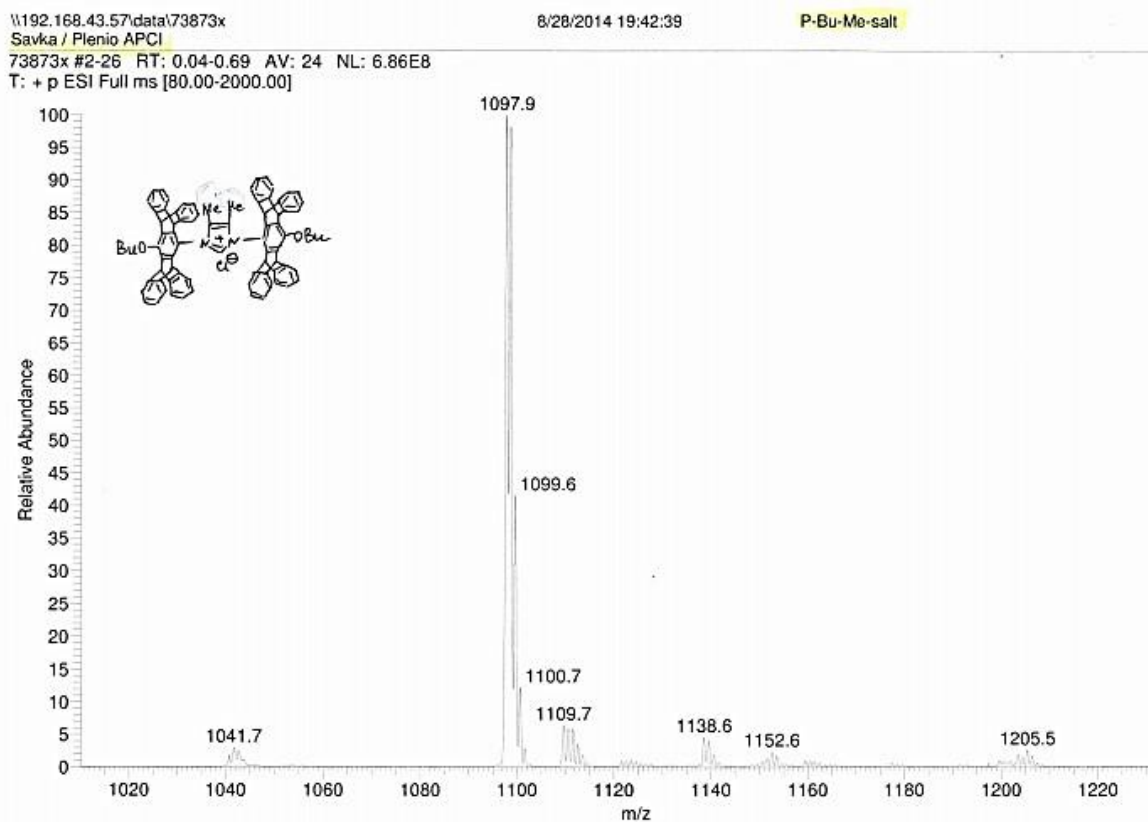
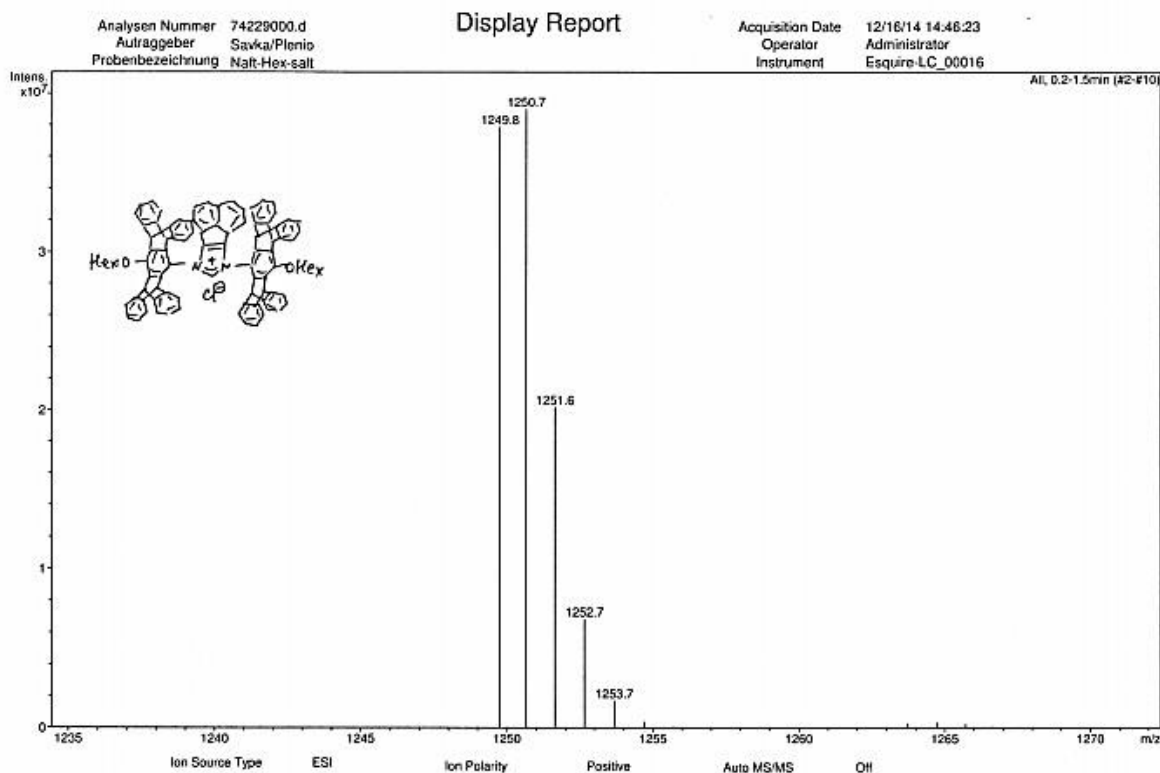
P-Me-salt

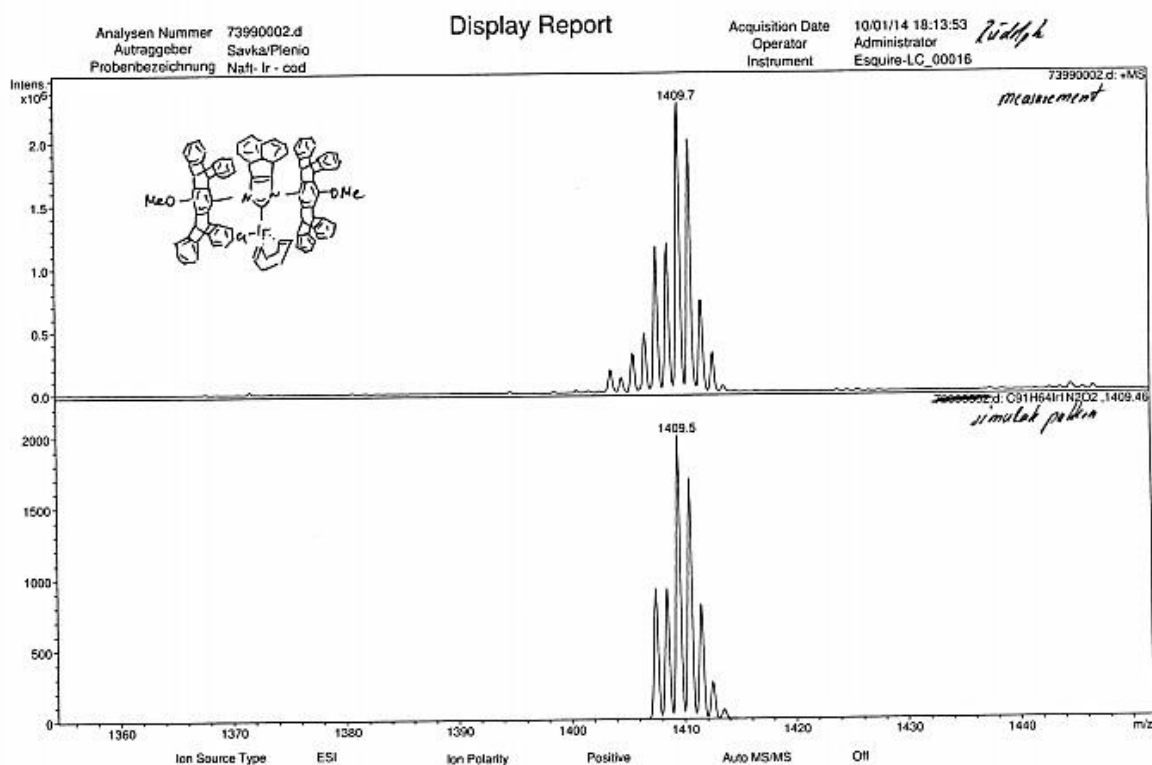
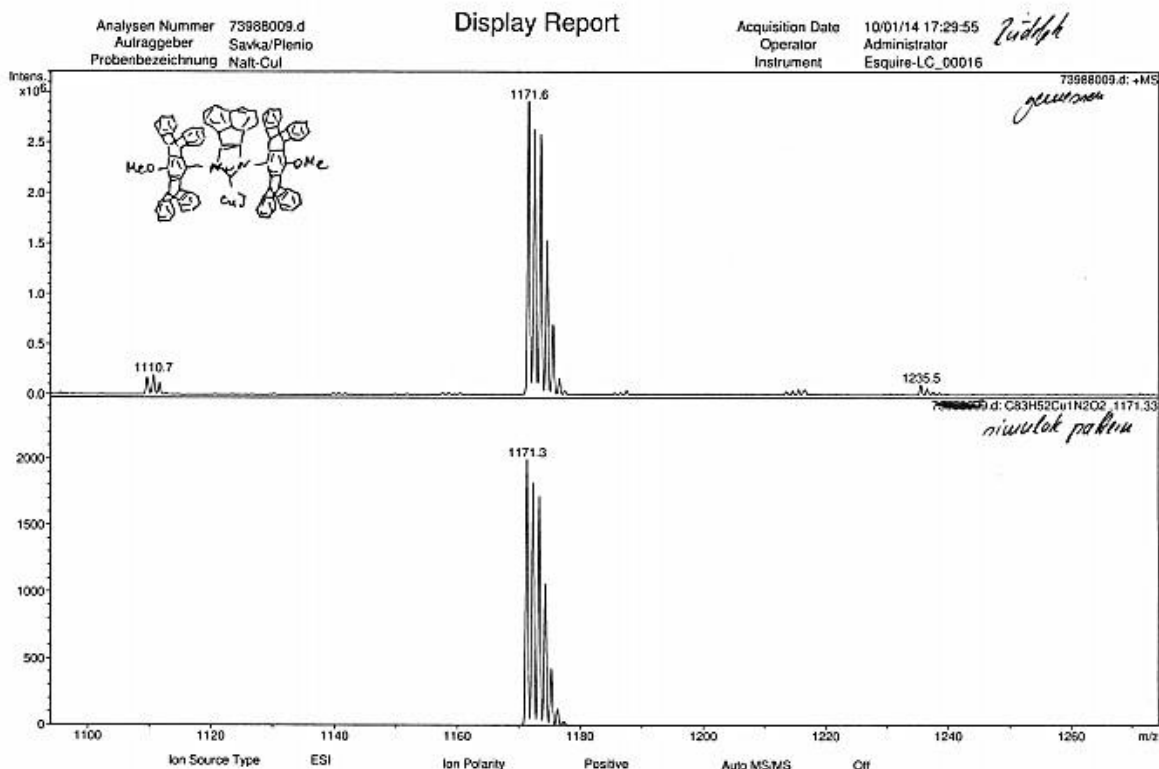
Savka / Plenio APCI

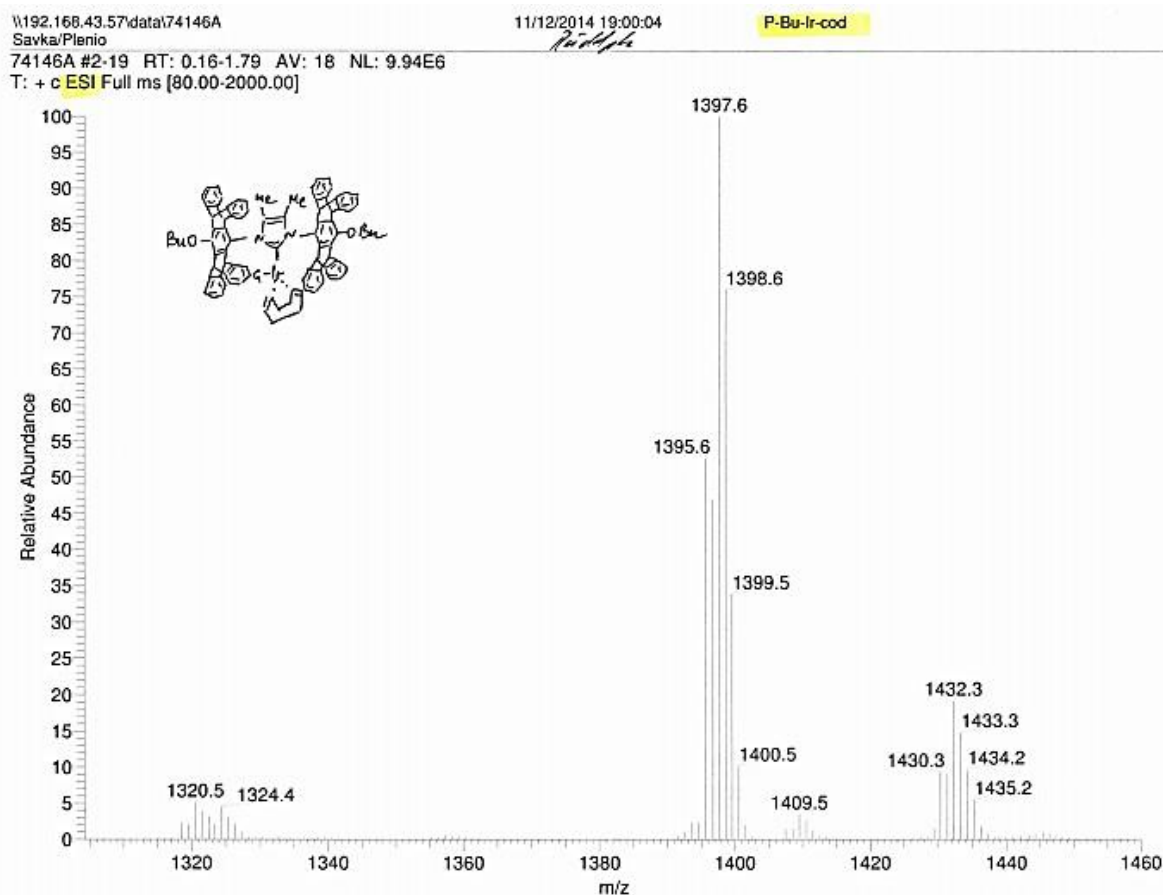
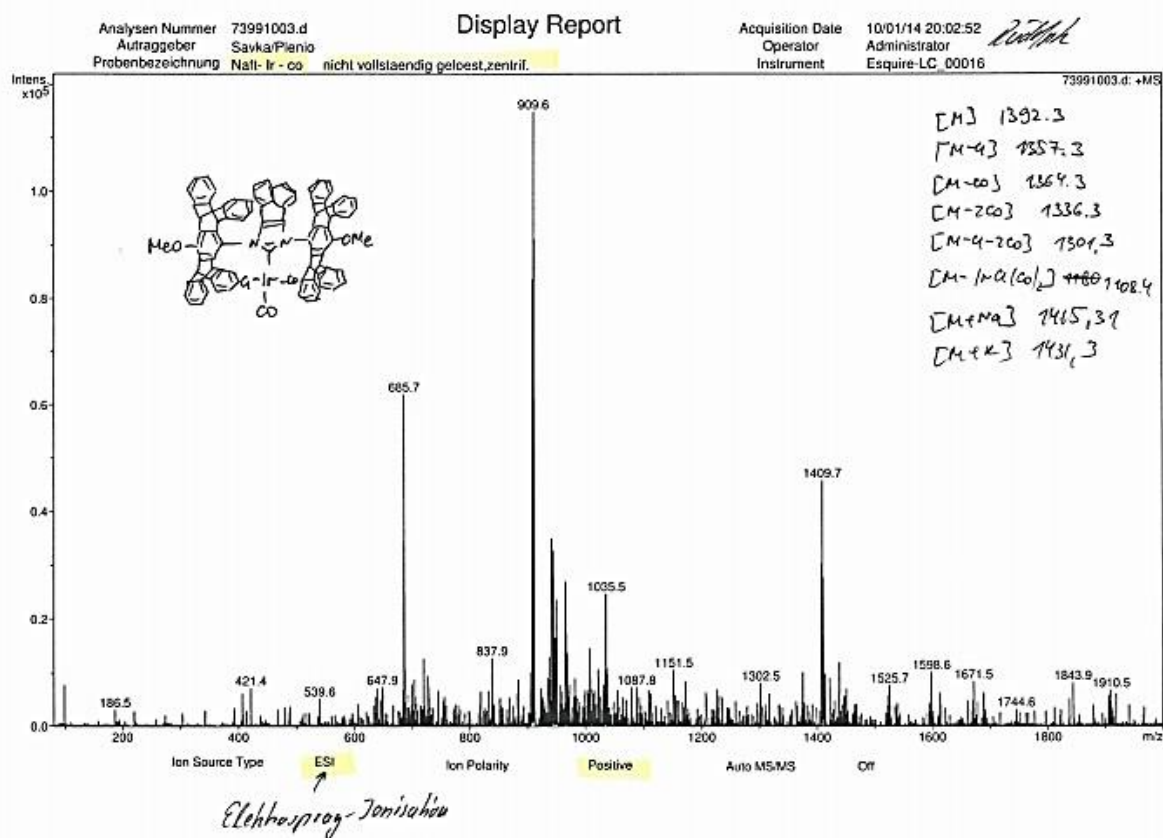
73874x #1-22 RT: 0.03-0.58 AV: 21 NL: 7.12E8

T: + p ESI Full ms [80.00-2000.00]



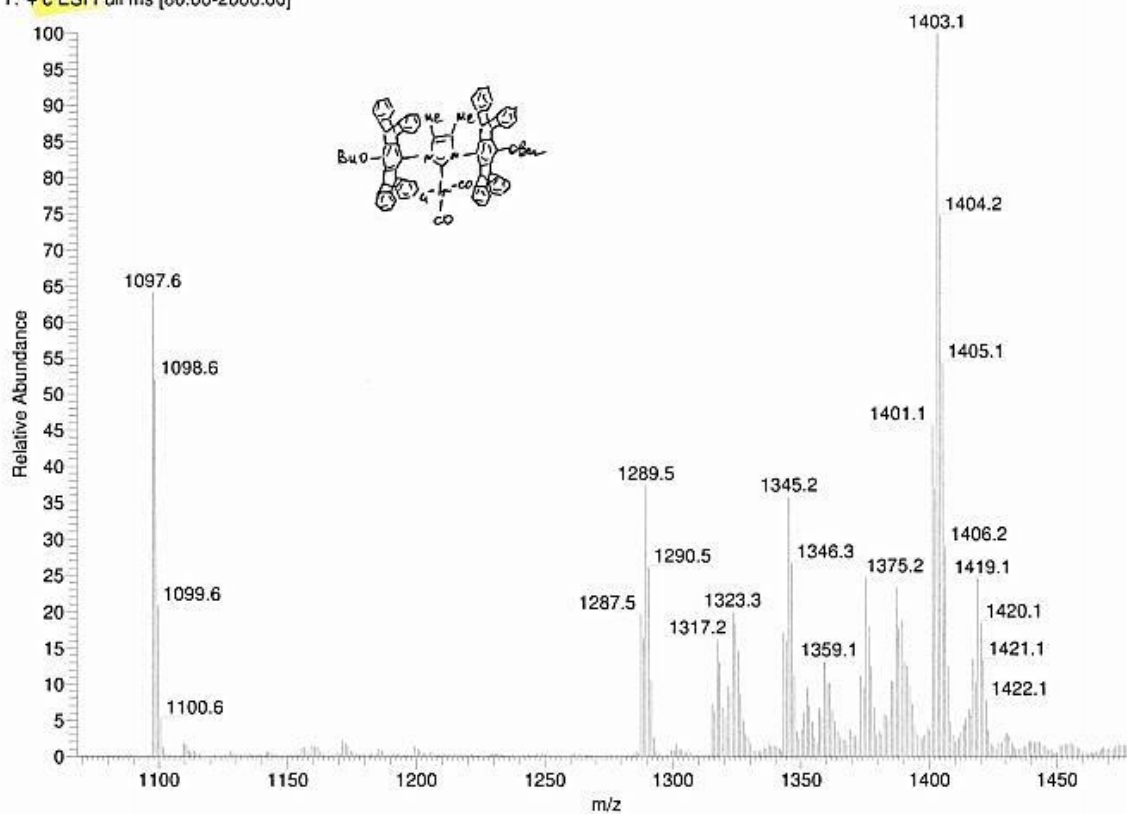






74145A #1-25 RT: 0.01-2.39 AV: 25 NL: 2.11E6

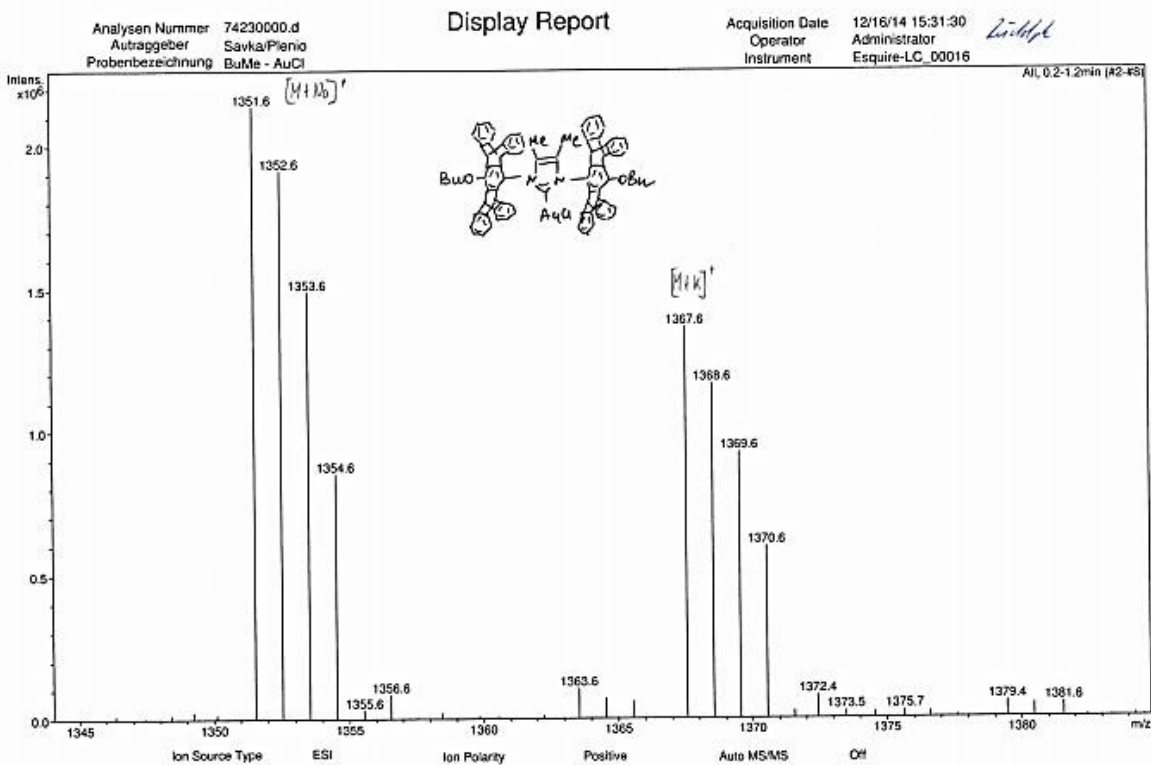
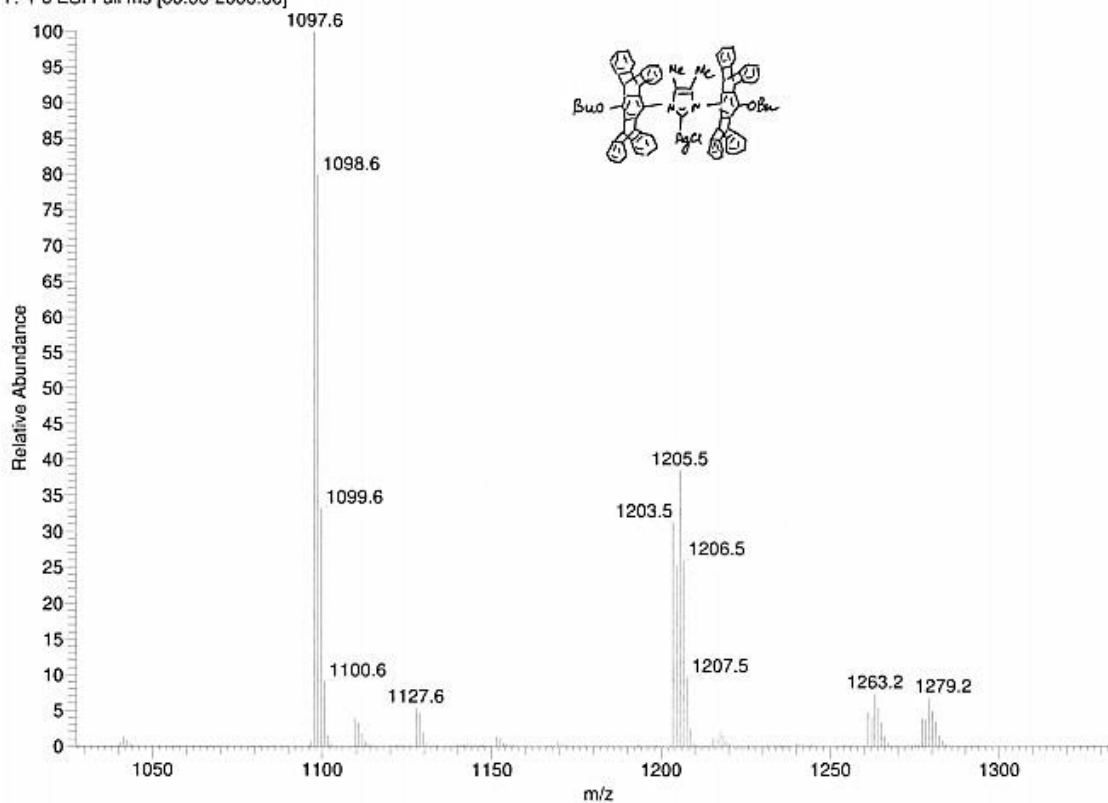
T: + c ESI Full ms [80.00-2000.00]

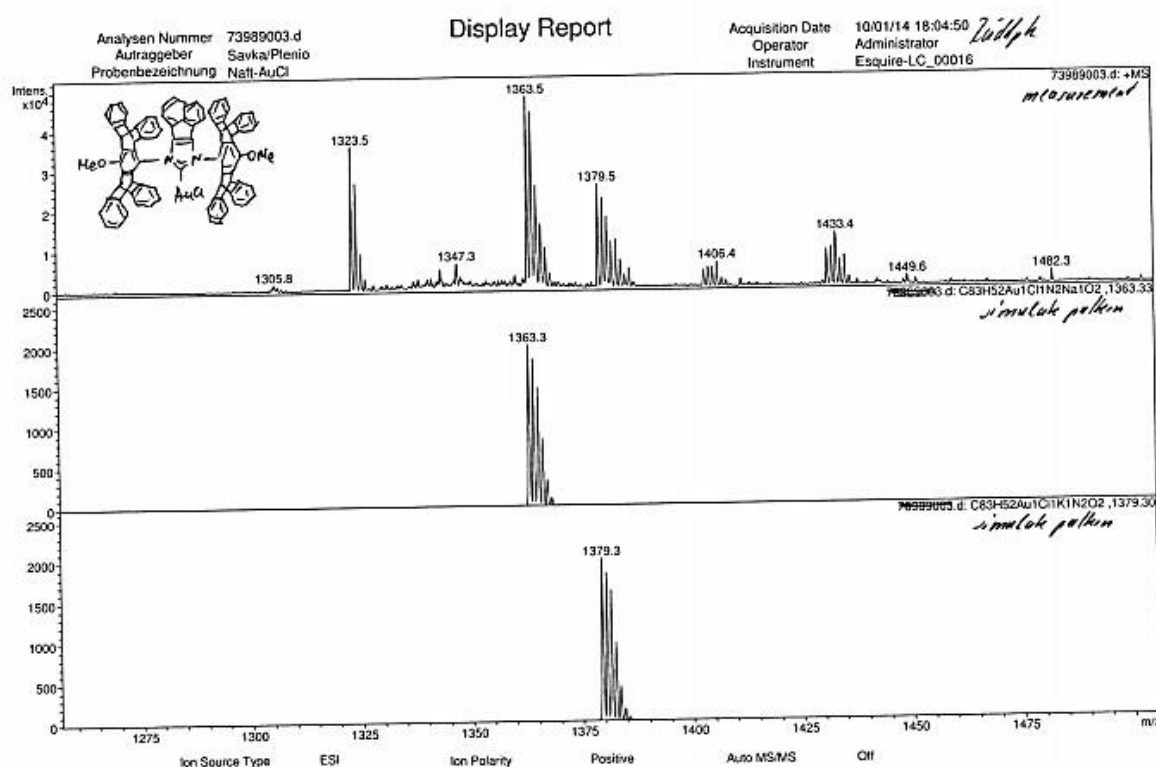
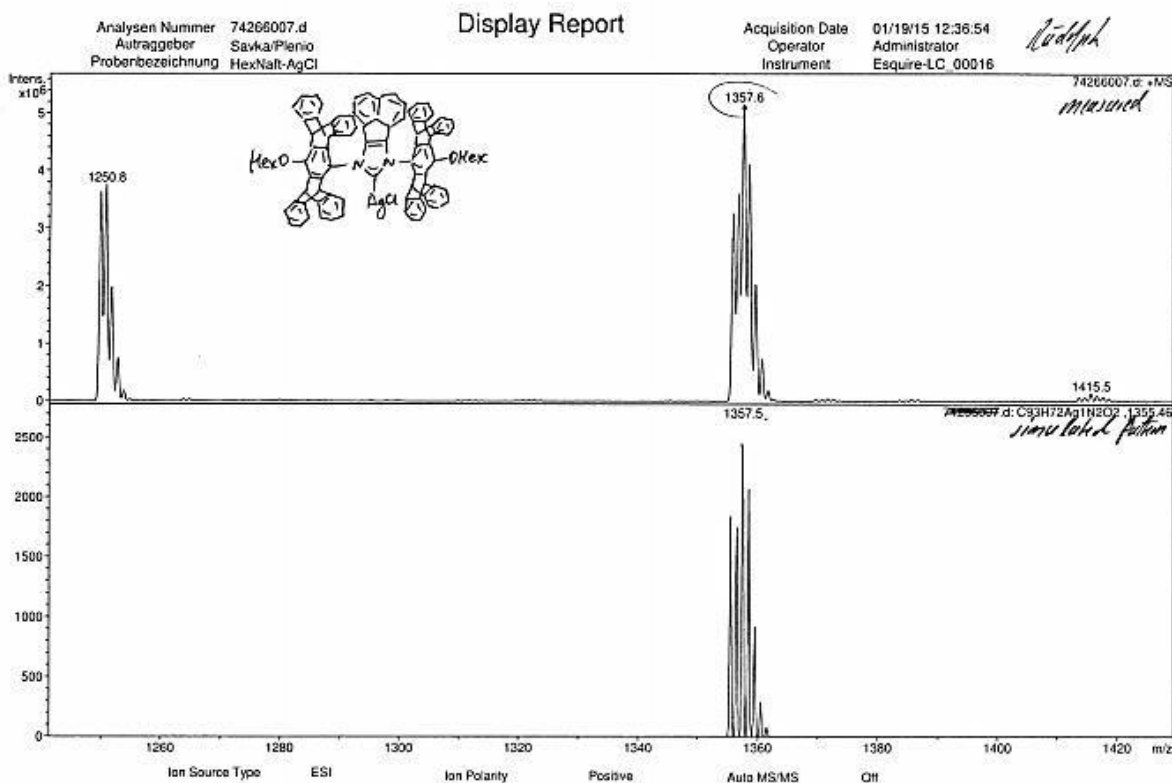


Savka/Plenio

74143A #2-26 RT: 0.11-2.44 AV: 25 NL: 3.50E6

T: + c ESI Full ms [80.00-2000.00]



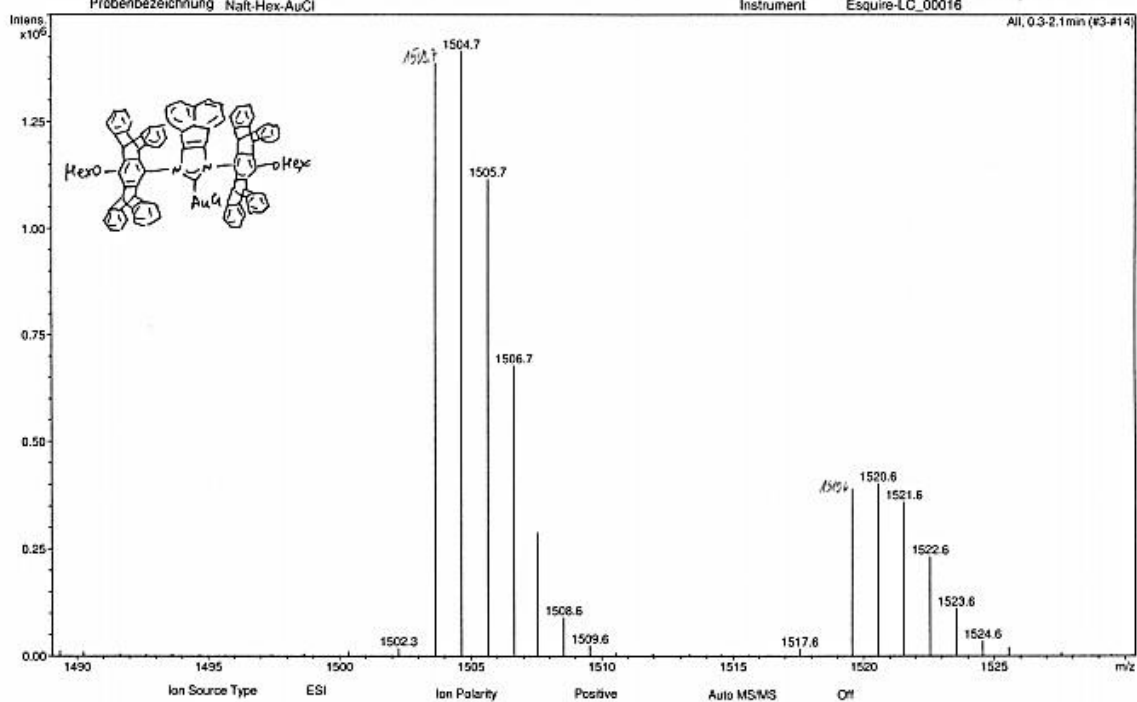


Analysen Nummer 74231001.d
 Auftraggeber Savka/Plenio
 Probenbezeichnung Nalt-Hex-AuCl

Display Report

Acquisition Date 12/16/14 16:04:45
 Operator Administrator
 Instrument Esquire-LC_00016

lidhph

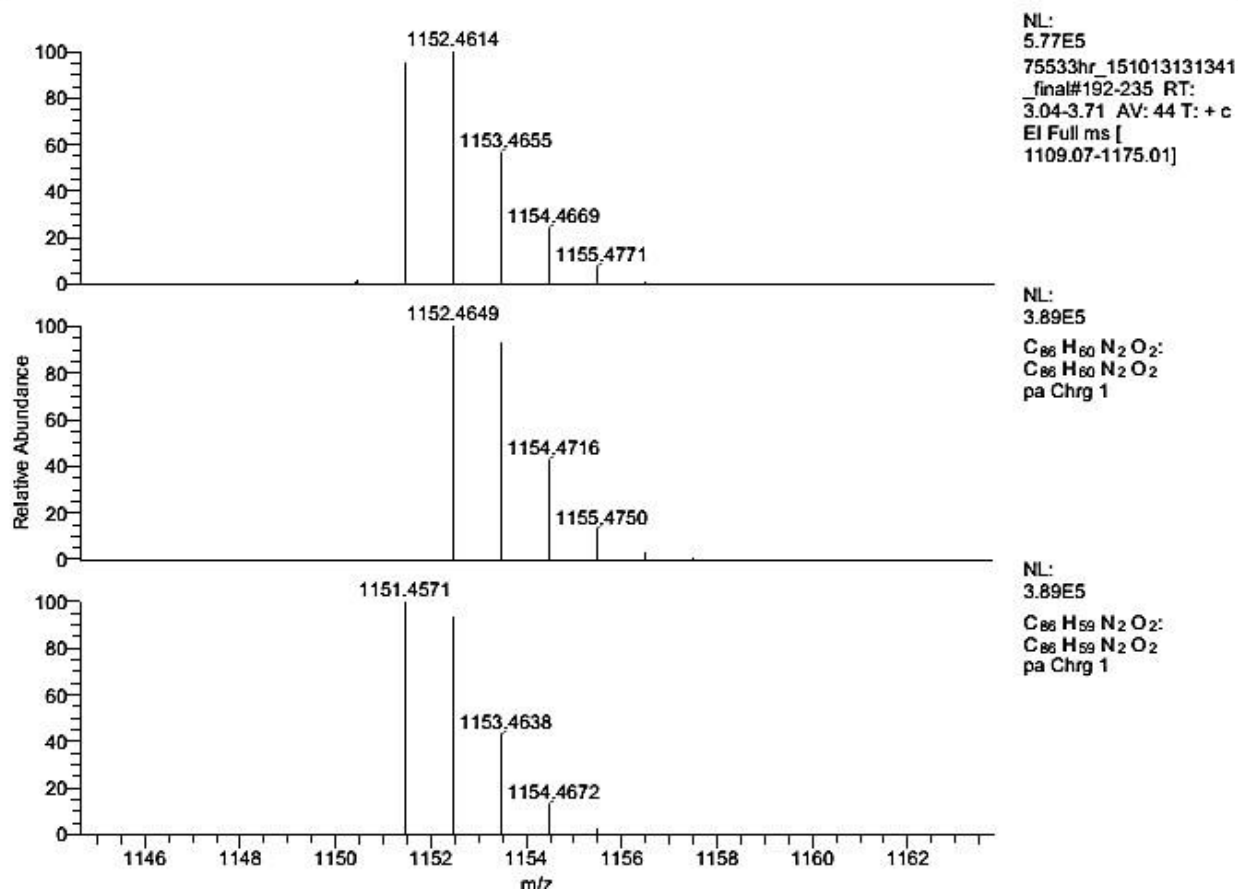


Compound 5b. HRMS (EI): m/z calcd for C₈₆H₅₉N₂O₂: 1151.4571 [M-C₆H₁₃]⁺; found: 1151.4561.

75533hr_151013131341_final
SAVKA/AK Plenio

10/14/2015 11:01:16 AM

P-5b



Elemental composition search on mass 1151.4561, P-5b, SAVKA/AK Plenio, 75533hr_151013131341_final

m/z= 1146.4561-1156.4561

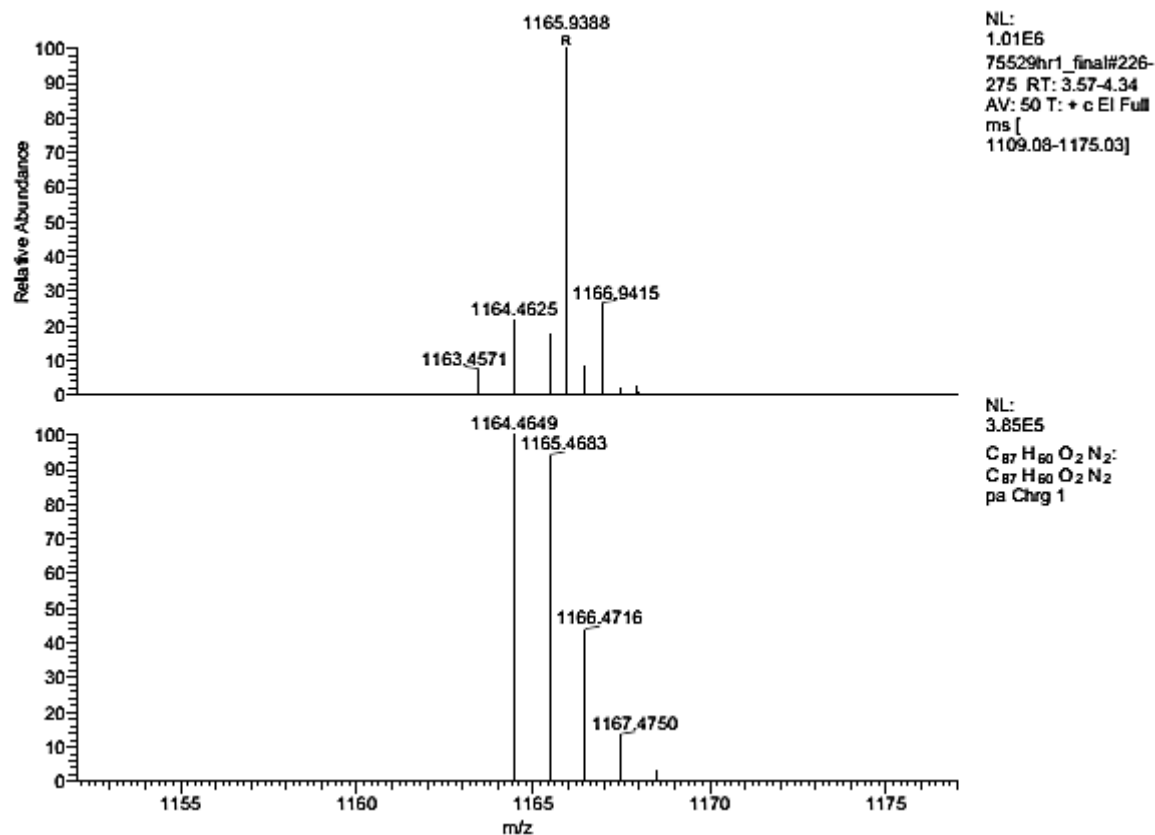
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1151.4561	1151.4571	-1.01	58.5	C ₈₆ H ₅₉ O ₂ N ₂

Compound 6b-HCl. HRMS (EI): m/z calcd for $C_{87}H_{60}N_2O_2$: 1164.4649 $[M-Cl-C_6H_{13}]^+$; found: 1164.4620.

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P-6b-HCL



NL:
1.01E6
75529hr1_final#226-
275 RT: 3.57-4.34
AV: 50 T: + c EI Full
ms [
1109.08-1175.03]

NL:
3.85E5
 $C_{87}H_{60}O_2N_2$:
 $C_{87}H_{60}O_2N_2$
pa Chrg 1

Elemental composition search on mass 1164.4620 , P-6b-HCL , SAVKA/AK Plenio , 75529hr1_final

m/z = 1159.4620-1169.4620

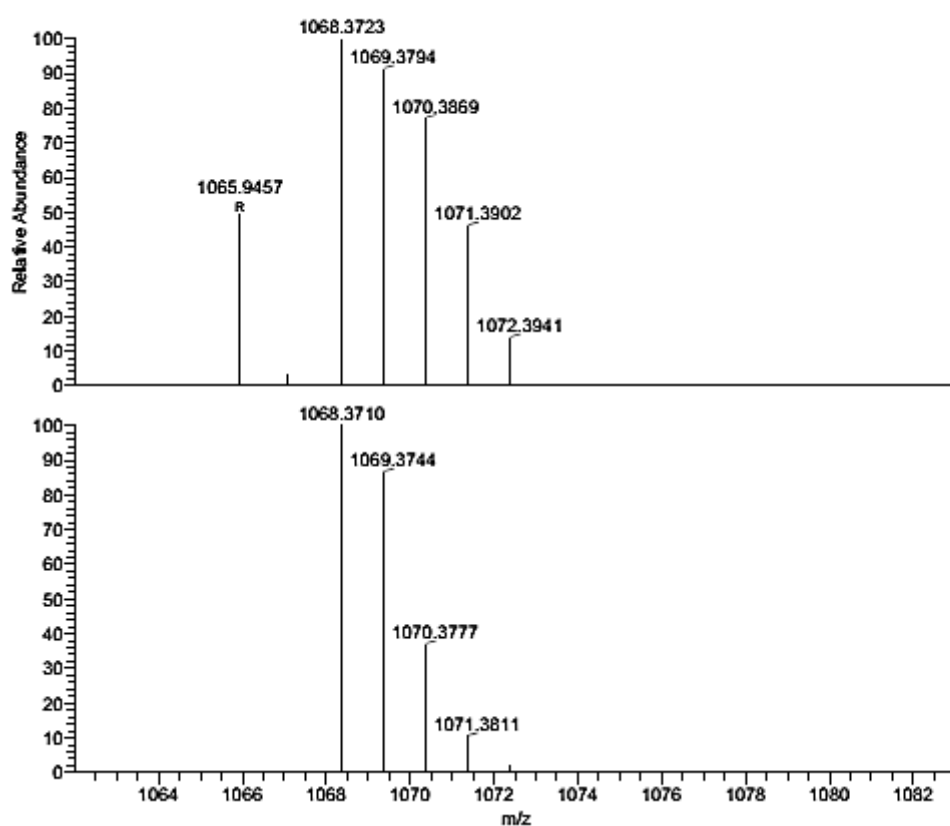
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1164.4620	1164.4649	-2.95	59.0	$C_{87}H_{60}O_2N_2$

Compound 4. HRMS (EI): m/z calcd for $C_{80}H_{48}N_2O_2$: 1068.3710 $[M]^+$; found: 1068.3723.

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Natt-imine-OH



NL:
1.47E5
75534hr_final#287-
348 RT: 4.68-5.67
AV: 62 T: + c EI Full
ms [
1059.46-1125.46]

NL:
4.15E5
 $C_{80}H_{48}N_2O_2$:
 $C_{80}H_{48}N_2O_2$:
pa Chrg 1

Elemental composition search on mass 1068.3723 , Natt-imine-OH , SAVKA/AK Plenio , 75534hr_final

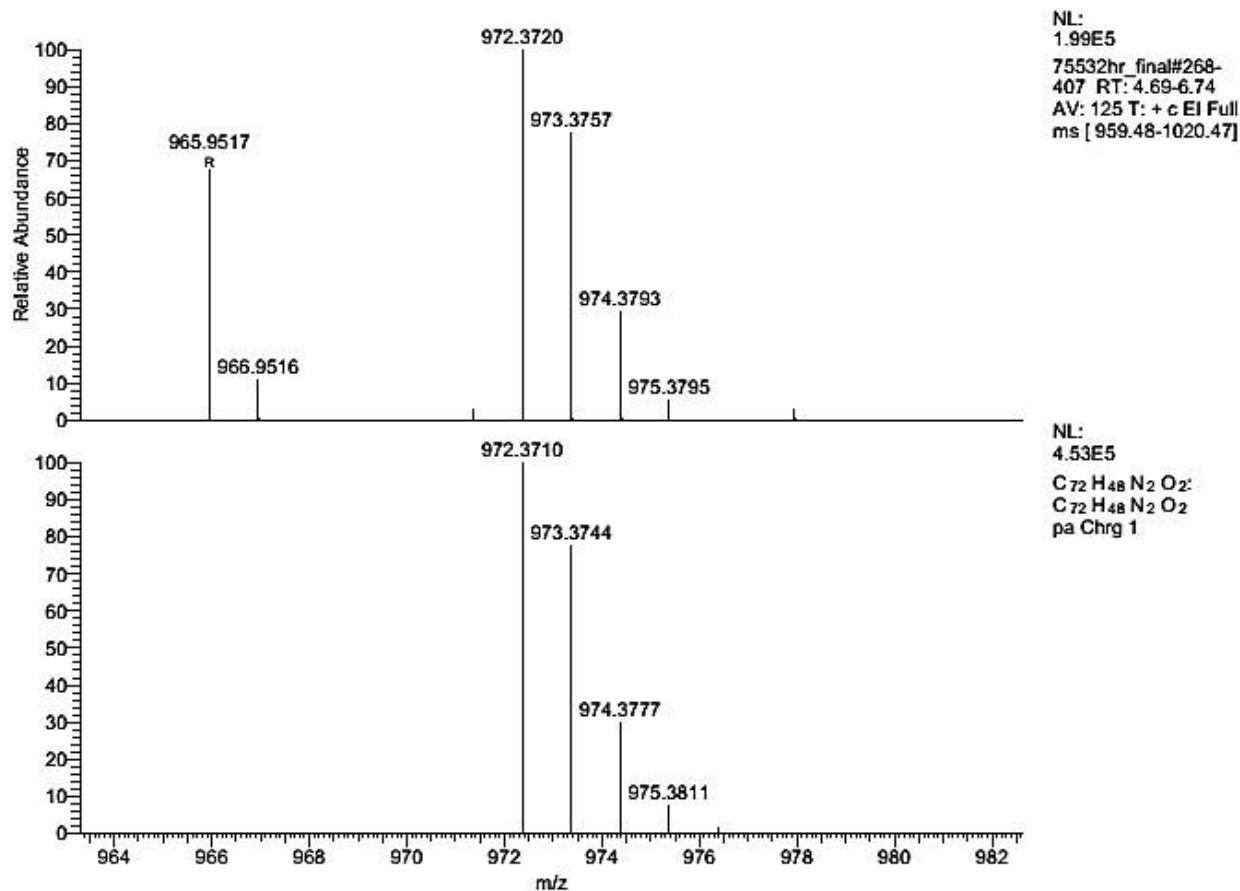
$m/z = 1063.3723-1073.3723$				
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1068.3723	1068.3710	1.31	58.0	$C_{80}H_{48}O_2N_2$
	1068.3751	-2.71	62.0	$C_{85}H_{48}$

Compound 7. HRMS (EI): m/z calcd for C₇₂H₄₈N₂O₂: 972.3710 [M]⁺; found: 972.3720.

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P-7



Elemental composition search on mass 972.3720 , P-7 , SAVKA/AK Plenio , 75532hr_final

m/z= 967.3720-977.3720

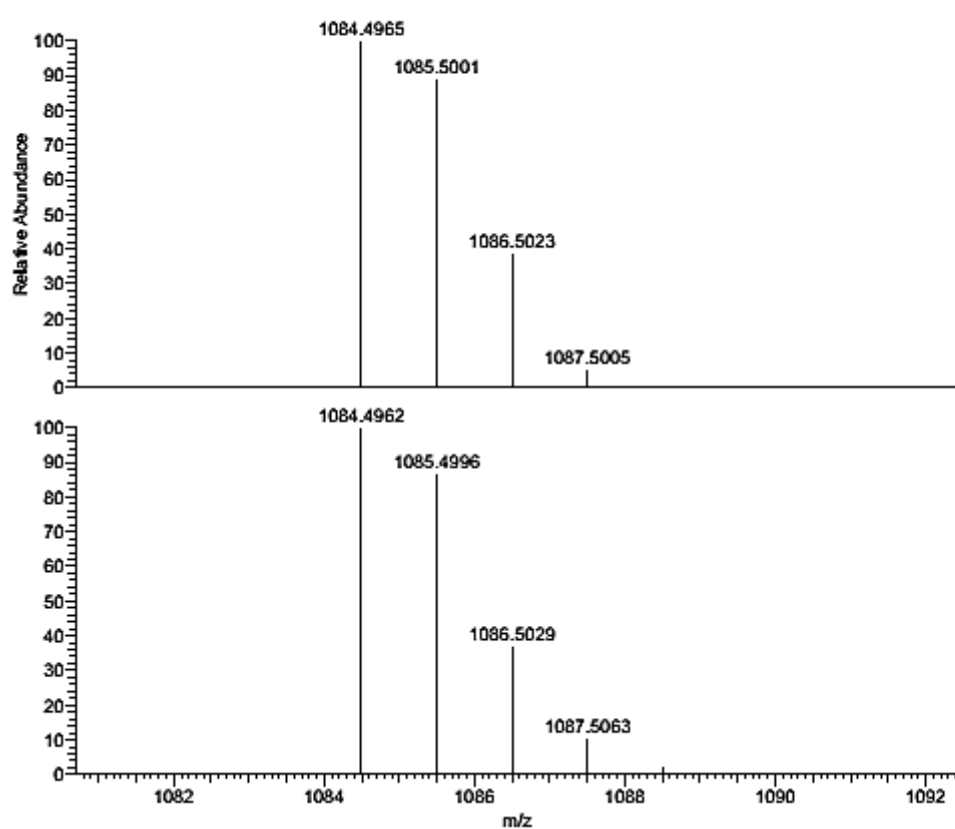
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
972.3720	972.3710	0.95	50.0	C ₇₂ H ₄₈ O ₂ N ₂
	972.3751	-3.07	54.0	C ₇₇ H ₄₈

Compound 8. HRMS (EI): m/z calcd for $C_{80}H_{64}N_2O_2$: 1084.4962 $[M]^+$; found: 1084.4965.

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SAVKA/AK Plenio

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P-8



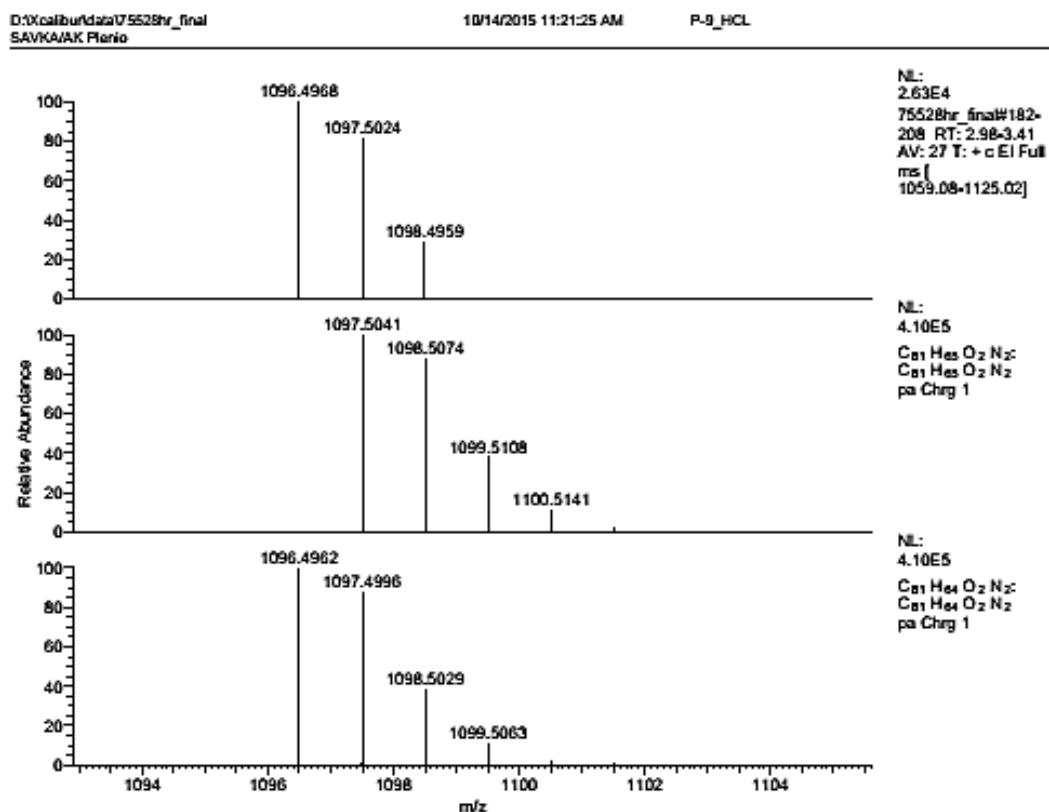
NL:
8.55E4
75531hr_final#304-
389 RT: 4.97-6.35
AV: 86 T: + c EI Full
ms [
1059.08-1125.02]

NL:
4.15E5
 $C_{80}H_{64}N_2O_2$
 $C_{80}H_{64}N_2O_2$
pa Chrg 1

Elemental composition search on mass 1084.4965 , P-8 , SAVKA/AK Plenio , 75531hr_final

m/z 1079.4965-1089.4965				
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1084.4965	1084.4962	0.30	50.0	$C_{80}H_{64}O_2N_2$

Compound 9·HCl. HRMS (EI): m/z calcd for $C_{81}H_{65}N_2O_2$: 1097.5041 $[M-Cl]^+$; found: 1097.5024.



Elemental composition search on mass 1097.5024 , P-9_HCL , SAVKA/AK Plenio , 75528hr_final

m/z - 1092.5024-1102.5024

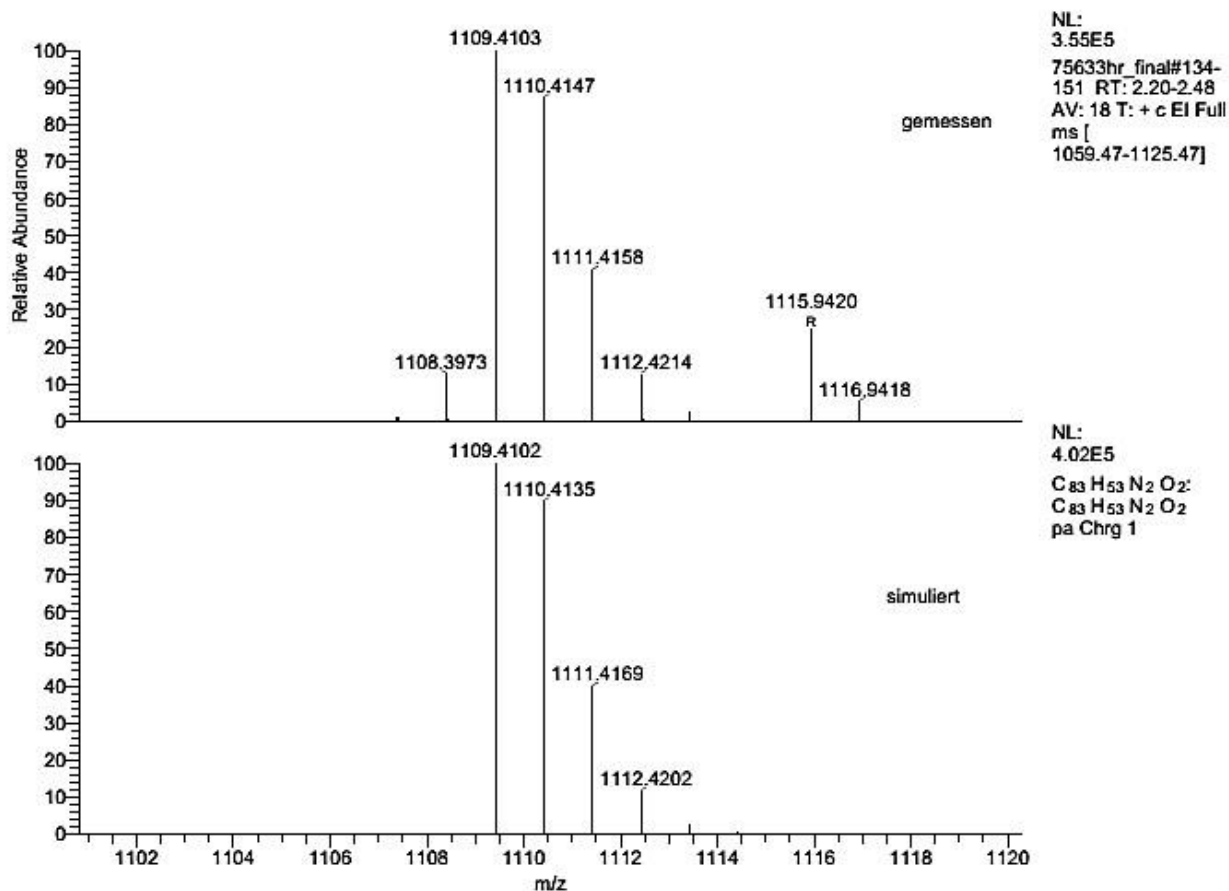
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1097.5024	1097.5041	-1.66	50.5	$C_{81}H_{65}O_2N_2$

Compound [(6a)IrCl(cod)]. HRMS (EI): m/z calcd for $C_{83}H_{53}N_2O_2$: 1109.4102 $[M+H-IrCl(cod)]^+$; found: 1109.4103.

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SAVKA/AK Plenio

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6a-Ir-COd



Elemental composition search on mass 1109.4103 , 6a-Ir-COd , SAVKA/AK Plenio , 75633hr_final

m/z - 1104.4103-1114.4103

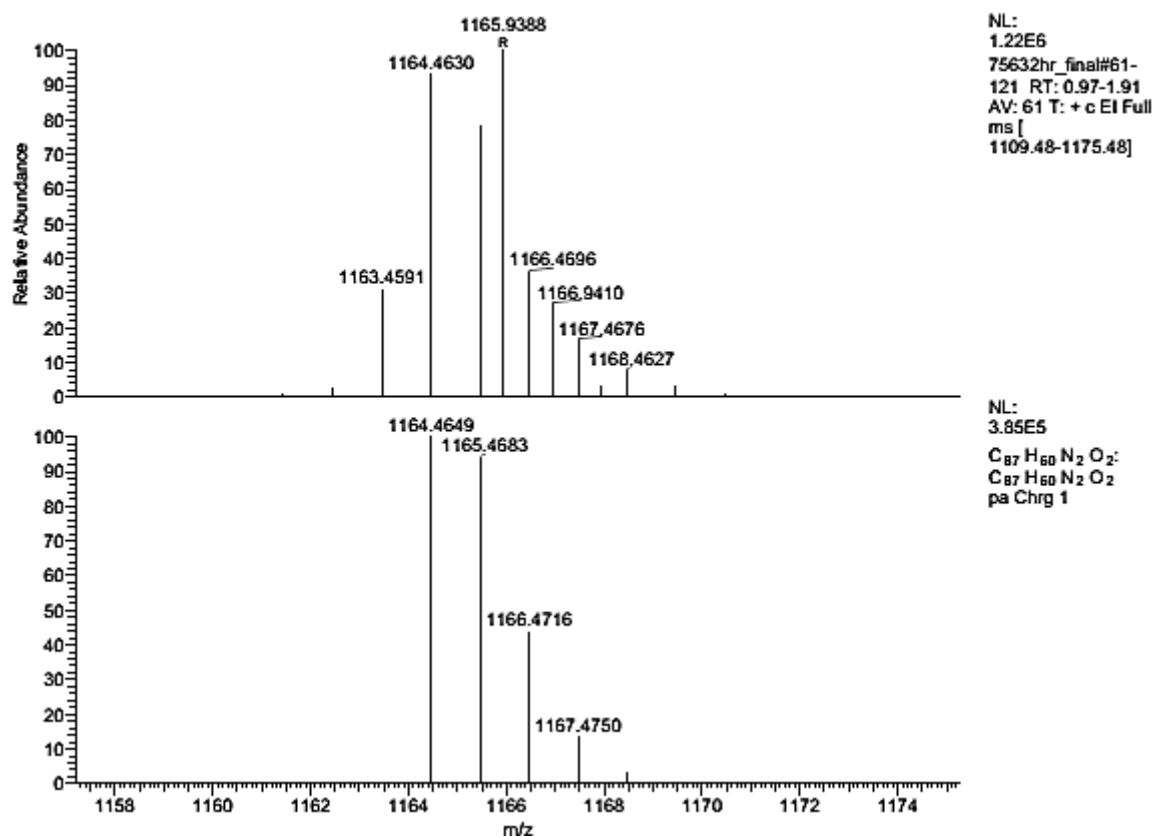
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1109.4103	1109.4102	0.14	58.5	$C_{83}H_{53}O_2N_2$
	1109.4120	-1.69	53.5	$C_{82}H_{58}O_2^{35}Cl_1$
	1109.4142	-3.88	62.5	$C_{83}H_{53}$
	1109.4142	-3.92	41.5	$C_{69}H_{58}O_1N_1^{193}Ir_1$
	1109.4147	-4.40	37.0	$C_{66}H_{61}N_2^{35}Cl_1^{193}Ir_1$

Compound [(6b)AgCl]. HRMS (EI): m/z calcd for $C_{87}H_{60}N_2O_2$: 1164.4649 $[M-AgCl-C_6H_{12}]^+$; found: 1164.4630.

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SAVKA/AK Plenio

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6b-AgCl



Elemental composition search on mass 1164.4630, 6b-AgCl, SAVKA/AK Plenio, 75632hr_final

m/z = 1159.4630-1169.4630

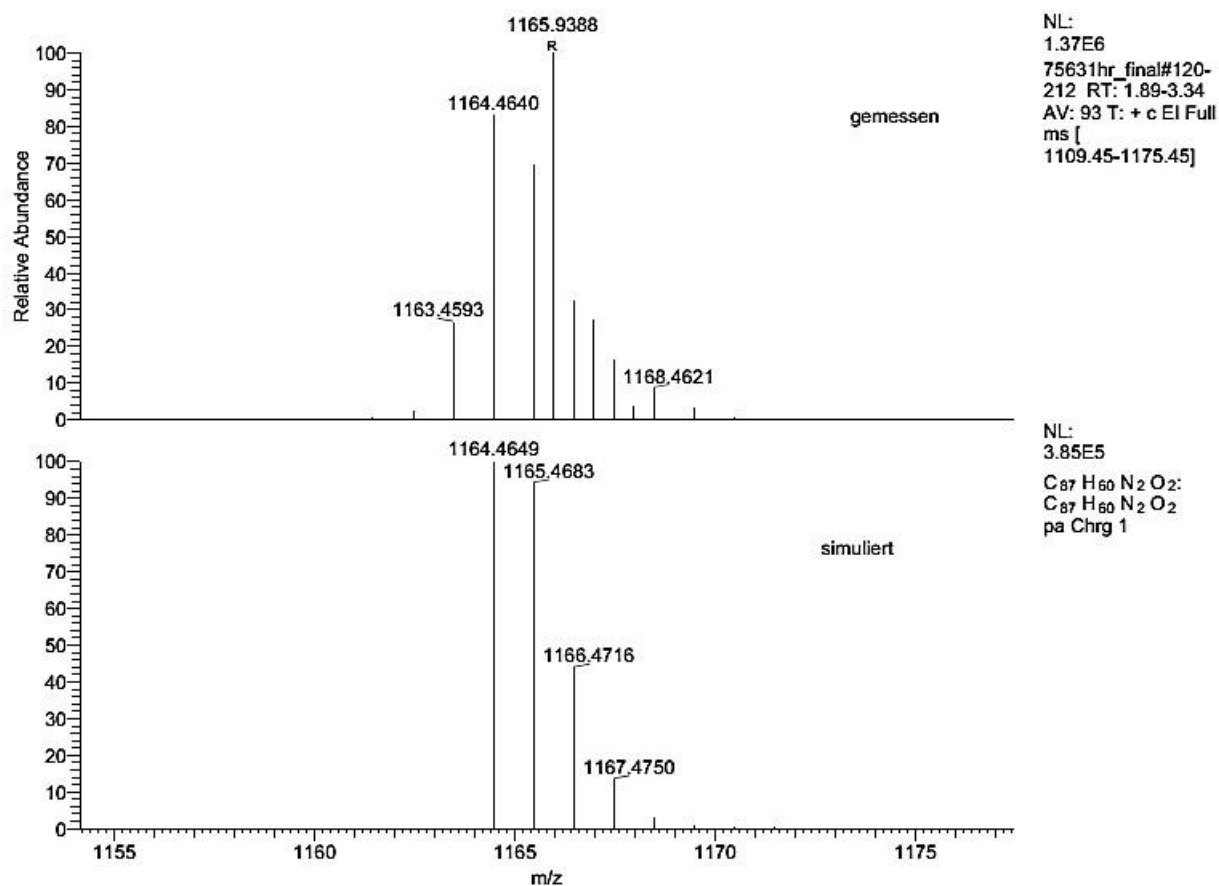
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1164.4630	1164.4632	-0.20	47.0	$C_{81}H_{71}N_1^{107}Ag_1$
	1164.4649	-1.93	59.0	$C_{87}H_{60}O_2N_2$
	1164.4610	2.00	38.0	$C_{75}H_{76}O_2N_1^{107}Ag_1^{35}Cl_1$
	1164.4668	-3.76	54.0	$C_{86}H_{65}O_2^{35}Cl_1$

Compound [(6b)AuCl]. HRMS (EI): m/z calcd for $C_{87}H_{60}N_2O_2$: 1164.4649 $[M-AuCl-C_6H_{12}]^+$;
found: 1164.4640.

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SAVKA/AK Plenio

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6b-AuCl



Elemental composition search on mass 1164.4640 , 6b-AuCl , SAVKA/AK Plenio , 75631hr_final

$m/z = 1159.4640-1169.4640$

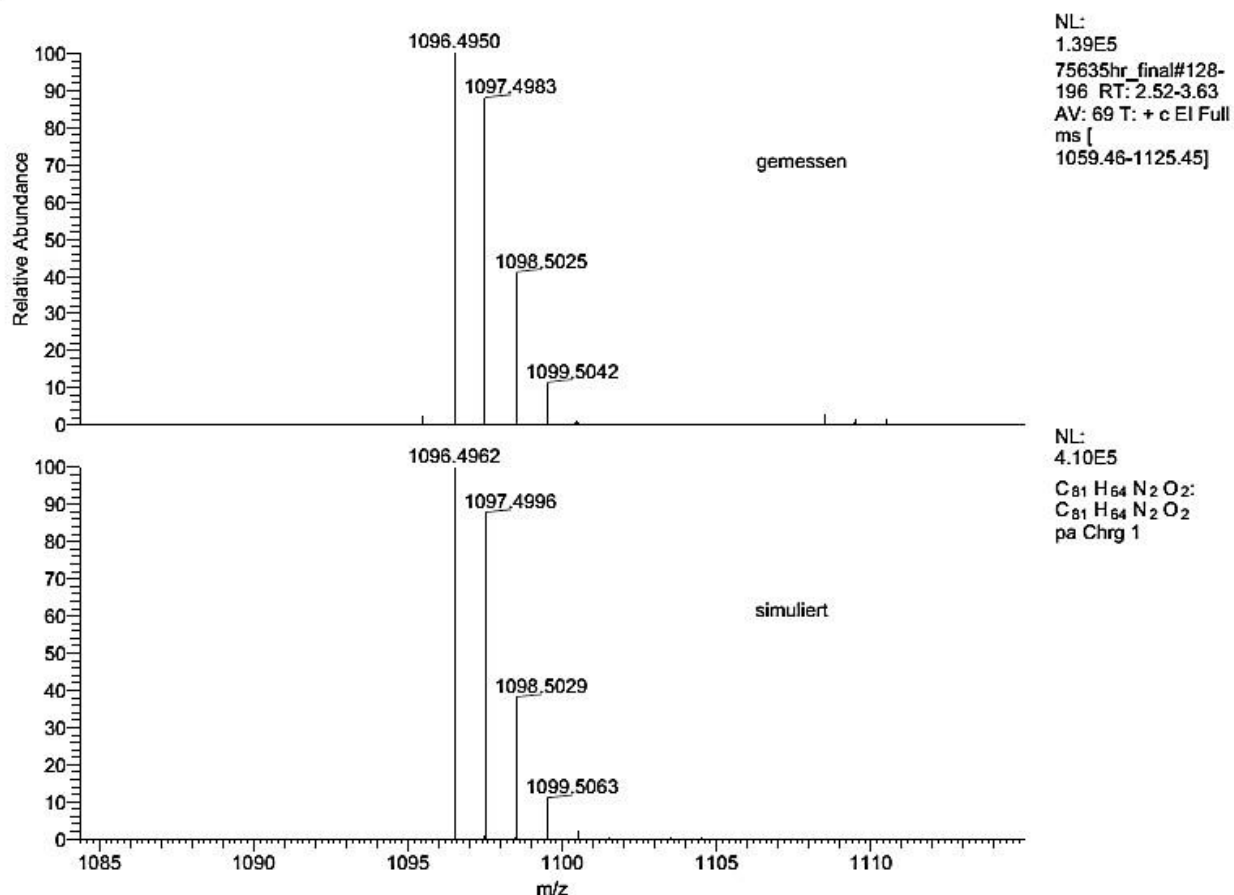
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1164.4640	1164.4649	-0.94	59.0	$C_{87}H_{60}O_2N_2$
	1164.4629	1.05	34.5	$C_{67}H_{68}O_2N_2^{197}Au_1^{35}Cl_1$
	1164.4651	-1.14	43.5	$C_{73}H_{63}N_2^{197}Au_1$
	1164.4668	-2.77	54.0	$C_{86}H_{65}O_2^{35}Cl_1$
	1164.4670	-2.97	38.5	$C_{72}H_{68}^{197}Au_1^{35}Cl_1$
	1164.4690	-4.96	63.0	$C_{92}H_{60}$

Compound [(9)AgCl]. HRMS (EI): m/z calcd for C₈₁H₆₄N₂O₂: 1096.4962 [M-AgCl]⁺; found: 1096.4950.

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9-AgCl



Elemental composition search on mass 1096.4950 , 9-AgCl , SAVKA/AK Plenio , 75635hr_final

m/z= 1091.4950-1101.4950

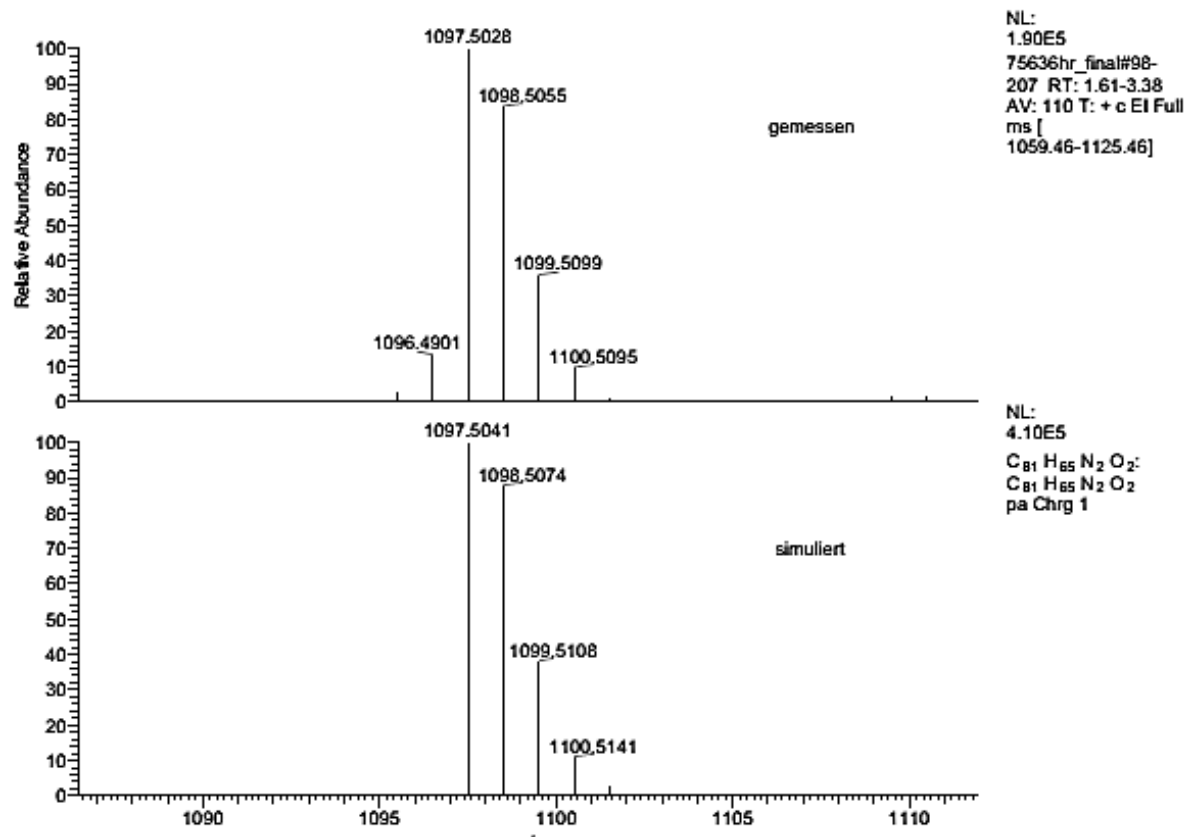
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1096.4950	1096.4962	-1.28	51.0	C ₈₁ H ₆₄ O ₂ N ₂
	1096.4981	-3.11	46.0	C ₈₀ H ₆₉ O ₂ ³⁵ Cl ₁

Compound [(9)IrCl(cod)]. HRMS (EI): m/z calcd for $C_{81}H_{65}N_2O_2$: 1097.5041 $[M+H-IrCl(cod)]^+$; found: 1097.5028.

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SAVKA/AK Plenio

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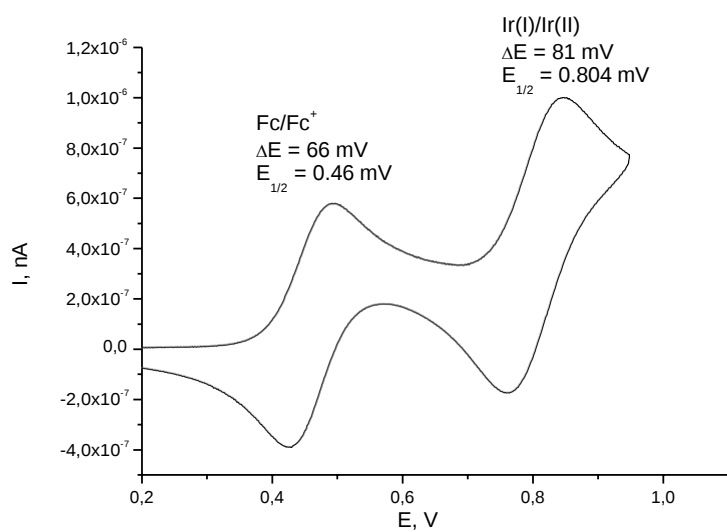
9-Ir-COd



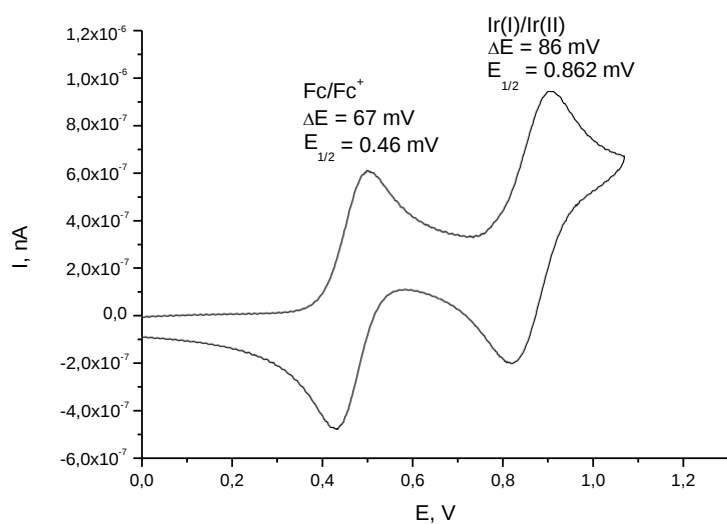
Elemental composition search on mass 1097.5028, 9-Ir-COd, SAVKA/AK Plenio, 75636hr_final

$m/z = 1092.5028-1102.5028$				
m/z	Theo. Mass	Delta (mmu)	RDB equiv.	Composition
1097.5028	1097.5041	-1.24	50.5	$C_{81}H_{65}O_2N_2$
	1097.5059	-3.07	45.5	$C_{80}H_{70}O_2^{35}Cl_1$

Cyclic voltammograms of iridium complexes



Cyclic voltammogram of complex [(**9**)Ir(cod)Cl].



Cyclic voltammogram of complex [(**6a**)Ir(cod)Cl].

IR spectra of iridium complexes

