Electronic Supplementary Information

Efficient Methylation of Anilines with Methanol Catalysed by Cyclometalated Ruthenium Complexes

Patrick Piehl[a], Roberta Amuso[a,b], Anke Spannenberg[a], Bartolo Gabriele[b], Helfried Neumann[a] and Matthias Beller*[a]

[a.] *Leibniz-Institut für Katalyse e.V.*
Albert-Einstein-Straße 29a, 18059 Rostock, Germany
E-Mail: Matthias.Beller@catalysis.de

[b.] *Laboratory of Industrial and Synthetic Organic Chemistry (LISOC), Department of Chemistry and Chemical Technologies, University of Calabria, Via Pietro Bucci 12/C, 87036 Arcavacata di Rende (CS), Italy*

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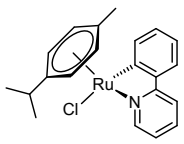
General Information

Unless otherwise stated, reactions were carried out under an argon atmosphere with exclusion of air and moisture by standard techniques for the manipulation air sensitive compounds. Reaction temperatures refer to external bath temperatures. TLC was performed on silica gel 60 F254 (layer thickness 0.2 mm) and spots were located using UV light and/or by treating the plates with phosphomolybdic acid reagent followed by heating. Column chromatography was performed on silica gel (230–400 mesh) using ethyl acetate/heptane as eluent. NMR spectra were performed on a Bruker Avance 400 spectrometer using the residual solvent signal as internal standard [chloroform: 7.26 ppm (1H), 77.0 ppm (13C)]. All measurements were carried out at room temperature unless otherwise stated. Mass spectra were in general recorded on a MAT 95XP or a HP 5973N mass selective detector. Gas chromatography was performed on a HP 6890N chromatographer with a HP5 column. Commercial reagents were used as received without purification.

General procedure for the synthesis of cyclometalated ruthenium complexes [Ru(C^N)(p-cym)Cl]

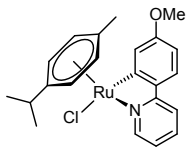
According to literature procedures,[1] $[Ru(p\text{-cym})Cl_2]_2$ (1.0 mmol), the aryl-substituted heterocycle (2.0 mmol) and KOAc (4.0 mmol) were dissolved in 50 ml of methanol and the resulting suspension stirred at room temperature for 4 days. Evaporation of the solvent provided the crude product as an orange solid which was purified either by column chromatography or washing with a suitable solvent mixture to remove impurities.



[Ru(2-phenyl pyridine)(*p*-cym)Cl], 4:[1] Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with 2-phenyl pyridine provided [Ru(2-phenyl pyridine)(*p*-cym)Cl] as an orange solid. The crude product was washed with a solution of heptane/ethyl acetate (1:1, 3 x 10 ml). The remaining solid was then dissolved in CH_2Cl_2 and the resulting solution filtered to remove insoluble impurities. After evaporation of the solvent, the pure product was obtained as an orange solid (742.4 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 0.88 (d, *J* = 6.9 Hz, 3H), 0.98 (d, *J* = 6.9 Hz, 3H), 2.04 (s, 3H), 2.43 (hept, *J* = 6.9 Hz, 1H), 4.98 (dd, *J* = 5.9, 1.3 Hz, 1H), 5.17 (dd, *J* = 5.9, 1.2 Hz, 1H), 5.57 (ddd, *J* = 10.4, 6.0, 1.2 Hz, 2H), 6.98 – 7.08 (m, 2H), 7.17 (td, *J* = 7.4, 1.4 Hz, 1H), 7.57 – 7.74 (m, 3H), 8.15 (ddd, *J* = 7.5, 1.2, 0.5 Hz, 1H), 9.23 (ddd, *J* = 5.7, 1.6, 0.8 Hz, 1H) ppm.

^{13}C NMR (101 MHz, $CDCl_3$) δ = 18.9 (CH_3), 21.9 (CH_3), 22.7 (CH_3), 30.9 (CH), 82.3 (CH), 84.2 (CH), 89.8 (CH), 90.9 (CH), 100.6 (C), 100.8 (C), 118.9 (CH), 121.5 (CH), 122.6, (CH) 124.0 (CH), 129.6 (CH), 136.7 (CH), 139.7 (CH), 143.4 (C), 154.7 (CH), 165.5 (C), 181.5 (Ru-C) ppm.



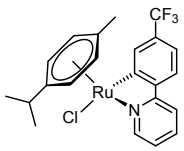
[Ru(2-(4-methoxy phenyl) pyridine)(p-cym)Cl], 5: Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with 2-(4-methoxy phenyl) pyridine provided [Ru(2-(4-methoxy phenyl) pyridine)(p-cym)Cl] as an orange solid. The crude product was washed with MeOH (3 x 5 ml). The remaining solid was then dissolved in CH_2Cl_2 and the resulting

[1] B. Li, T. Roisnel, C. Darcel, P. H. Dixneuf, *Dalton Trans.* **2012**, *41*, 10934-10937.

solution filtered to remove insoluble impurities. After evaporation of the solvent, the pure product was obtained as an orange solid (612.1 mg, 67%).

^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.88 (d, *J*=6.9, 3H), 0.98 (d, *J*=6.9, 3H), 2.04 (s, 3H), 2.44 (hept, *J*=7.0, 1H), 3.90 (s, 3H), 4.97 (dd, *J*=6.0, 1.2, 1H), 5.17 (dd, *J*=6.0, 1.2, 1H), 5.50 – 5.59 (m, 2H), 6.58 (dd, *J*=8.5, 2.5, 1H), 6.95 (ddd, *J*=6.2, 5.7, 2.3, 1H), 7.52 – 7.63 (m, 3H), 7.70 (d, *J*=2.5, 1H), 9.10 – 9.19 (m, 1H) ppm;

^{13}C NMR (75 MHz, $CDCl_3$) δ = 18.9 (CH_3), 21.8 (CH_3), 22.7 (CH_3), 30.9 (CH), 55.3 (CH_3), 82.4 (CH), 84.4 (CH), 89.6 (CH), 90.6 (CH), 100.4 (C), 100.6 (C), 108.8 (CH), 118.2 (CH), 120.4 (CH), 124.0 (CH), 125.0 (CH), 136.5 (CH), 136.7 (C), 154.5 (CH), 159.7 (C), 165.0 (C), 183.4 (C-Ru) ppm.

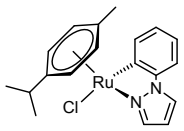


[Ru(2-(4-trifluoromethyl phenyl) pyridine)(*p*-cym)Cl], 6: Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with 2-(4-trifluoromethyl phenyl) pyridine provided [Ru(2-(4-trifluoromethyl phenyl) pyridine)(*p*-cym)Cl] as an orange solid. The crude product was washed with MeOH (3 x 5 ml). The remaining solid was then dissolved in CH_2Cl_2 and the resulting solution filtered to remove insoluble impurities. After evaporation of the solvent, the pure product was obtained as an orange solid (731.2 mg, 74%).

^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.86 (d, *J*=6.9, 3H), 0.97 (d, *J*=6.9, 3H), 2.06 (s, 3H), 2.41 (hept, *J*=6.9, 1H), 5.01 (dd, *J*=6.0, 1.2, 1H), 5.20 (dd, *J*=6.0, 1.1, 1H), 5.56 – 5.63 (m, 2H), 7.12 (ddd, *J*=7.3, 5.7, 1.7, 1H), 7.24 (ddd, *J*=8.1, 1.8, 0.7, 1H), 7.62 – 7.72 (m, 2H), 7.75 (ddd, *J*=8.3, 1.7, 0.8, 1H), 8.38 (dd, *J*=1.8, 0.9, 1H), 9.25 (ddd, *J*=5.7, 1.5, 0.8, 1H) ppm;

^{13}C NMR (75 MHz, $CDCl_3$) δ = 18.9 (CH), 21.8 (CH_3), 22.6 (CH_3), 31.0 (CH), 82.2 (CH), 84.6 (CH), 89.9 (CH), 91.1 (CH), 101.4 (C), 101.7 (C), 119.6 (q, *J*=4.0, CH), 119.7 (CH), 122.6 (CH), 123.5 (CH), 130.0 (q, *J*=30.3, *C*CF_3), 135.6 (q, *J*=3.5, CH), 137.1 (CH), 146.7 (q, *J*=1.6, C), 154.9 (CH), 164.1 (C), 181.4 (Ru-C) ppm, CF_3 not detectable;

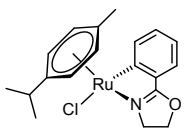
^{19}F NMR (282 MHz, $CDCl_3$) δ = -62.0 ppm.



[Ru(1-phenyl pyrazole)(*p*-cym)Cl], 7:[1] Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with 1-phenyl pyrazole provided [Ru(1-phenyl pyrazole)(*p*-cym)Cl] as a yellowish orange solid. The crude product was washed with a solution of heptane/ethyl acetate (1:1, 3 x 10 ml). The remaining solid was then dissolved in CH_2Cl_2 and the resulting solution filtered to remove insoluble impurities. After evaporation of the solvent, the pure product was obtained as a yellowish orange solid (556.6 mg, 67%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 0.94 (dd, *J*=14.2, 6.9, 6H), 2.04 (s, 3H), 2.43 (hept, *J*=6.9, 1H), 5.05 – 5.10 (m, 1H), 5.26 – 5.30 (m, 1H), 5.55 (dd, *J*=5.8, 1.2, 2H), 6.46 (dd, *J*=2.8, 2.2, 1H), 6.99 – 7.05 (m, 1H), 7.10 (td, *J*=7.3, 1.4, 1H), 7.16 (dd, *J*=7.7, 1.3, 1H), 7.90 (dd, *J*=2.8, 0.7, 1H), 8.05 (dd, *J*=2.2, 0.7, 1H), 8.13 (dd, *J*=7.3, 1.3, 1H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) δ = 18.9 (CH_3), 22.0 (CH_3), 22.5 (CH_3), 30.8 (CH), 82.3 (CH), 84.2 (CH), 88.2 (CH), 88.7 (CH), 100.1 (C), 100.1 (C), 108.4 (CH), 111.5 (CH), 123.2 (CH), 125.0 (CH), 126.1 (CH), 140.2 (CH), 141.9 (C), 142.2 (CH), 161.9 (Ru-C) ppm.

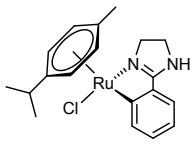


[Ru(2-phenyl-2-oxazoline)(*p*-cym)Cl], 8:[1] Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with 2-phenyl-2-oxazoline provided [Ru(2-phenyl-2-oxazoline)(p-cym)Cl] as an orange solid. The crude product was purified by column chromatography under an argon atmosphere using heptane/ethyl acetate (1:1) with 1% NEt_3 as eluent (412.2 mg, 49%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 0.97 (d, *J*=6.8, 3H), 1.08 (d, *J*=6.9, 3H), 2.03 (s, 3H), 2.53 (hept, *J*=6.9, 1H), 4.02 – 4.24 (m, 2H), 4.58 – 4.78 (m, 2H), 4.96 (dd, *J*=5.8, 1.2, 1H), 5.17 (dd, *J*=5.9, 1.2, 1H), 5.43 (dd, *J*=5.9,

1.2, 1H), 5.53 (dd, *J*=5.8, 1.2, 1H), 6.96 (td, *J*=7.4, 1.1, 1H), 7.19 (td, *J*=7.4, 1.5, 1H), 7.33 (ddd, *J*=7.5, 1.5, 0.6, 1H), 8.13 (ddd, *J*=7.6, 1.1, 0.5, 1H) ppm;

^{13}C NMR (101 MHz, $CDCl_3$) δ = 19.0 (CH_3), 22.1 (CH_3), 22.7 (CH_3), 31.1 (CH), 54.4 (CH_2), 70.7 (CH_2), 80.9 (CH), 81.7 (CH), 87.5 (CH), 88.0 (CH), 99.2 (C), 101.3 (C), 122.3 (CH), 126.4 (CH), 130.6 (CH), 130.8 (C), 139.3 (CH), 174.2 (CH), 182.7 (CH) ppm.



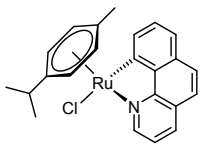
[Ru(2-phenyl imidazoline)(p-cym)Cl], 9: Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with 2-phenyl imidazoline provided [Ru(2-phenyl imidazoline)(p-cym)Cl] as an orange solid. The crude product was washed with MeOH (3 x 5 ml). The remaining solid was then dissolved in CH_2Cl_2 and the resulting solution filtered to remove insoluble impurities. After evaporation of the solvent, the product was obtained as an orange solid (300.3 mg, 72%). Crystals suitable for x-ray crystal structure analysis were obtained by slow evaporation of a solution of **9** in THF.

^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.95 (dd, J=18.4, 6.9, 6H), 2.00 (s, 3H), 2.29 – 2.47 (m, 2H), 3.26 (dt, J=10.9, 9.0, 1H), 3.76 – 3.98 (m, 2H), 4.91 (dd, J=5.7, 1.2, 1H), 5.14 (dd, J=5.9, 1.2, 1H), 5.37 (dd, J=5.8, 1.2, 1H), 5.43 (dd, J=5.7, 1.2, 1H), 6.19 (d, J=2.9, 1H), 6.93 (td, J=7.4, 1.2, 1H), 7.12 (td, J=7.4, 1.5, 1H), 7.32 (ddd, J=7.4, 1.5, 0.5, 1H), 8.16 (ddd, J=7.5, 1.2, 0.5, 1H) ppm;

^{13}C NMR (75 MHz, $CDCl_3$) δ = 19.0 (CH3), 22.3 (CH3), 22.6 (CH3), 31.1 (CH), 44.3 (CH2), 56.0 (CH2), 80.6 (CH), 82.5 (CH), 87.3 (CH), 87.9 (CH), 99.1 (C), 99.3 (C), 121.7 (CH), 125.4 (CH), 129.4 (CH), 135.1 (C), 139.4 (CH), 172.3 (C), 182.6 (C-Ru) ppm;

Elemental Anal. calcd. for $C_{19}H_{23}ClN_2Ru$: C, 54.87; H, 5.57; Cl, 8.52; N, 6.74; Ru, 24.30; found: C, 44.6; H, 4.4; Cl, 15.5; N, 5.2; Ru, 20.1. According to this, the sample contains ca. 82% of compound **9** and 18% of KCl.

HRMS (ESI-TOF): *m/z*: calcd for $[M\text{-}Cl]^+$: 381.0905, found 381.0901.



[Ru(benzo[*h*]quinone)(*p*-cym)Cl], 10:[1] Following the general procedure, the reaction of $[Ru(p\text{-cym})Cl_2]_2$ with benzo[*h*]quinone provided [Ru(benzo[*h*]quinone)(*p*-cym)Cl] as a dark green solid. The crude product was purified by column chromatography under an argon atmosphere using heptane/ethyl acetate (1:1) with 1% NEt_3 as eluent (738.9 mg, 82%).

^{1}H NMR (400 MHz, CD_2Cl_2) δ = 0.80 (d, *J*=6.9, 3H), 0.93 (d, *J*=6.9, 3H), 2.00 (s, 3H), 2.44 (hept, *J*=6.9, 1H), 5.12 (dd, *J*=5.9, 1.2, 1H), 5.30 (dd, *J*=5.9, 1.2, 1H), 5.67 (dd, *J*=5.9, 1.2, 1H), 5.73 (dd, *J*=5.9, 1.3, 1H), 7.47 (dd, *J*=8.0, 5.3, 1H), 7.53 – 7.62 (m, 3H), 7.79 (d, *J*=8.7, 1H), 8.21 (dd, *J*=8.0, 1.3, 1H), 8.38 (dd, *J*=6.4, 1.7, 1H), 9.47 (dd, *J*=5.3, 1.3, 1H) ppm;

^{13}C NMR (101 MHz, CD_2Cl_2) δ = 19.0 (CH_3), 21.9 (CH_3), 22.7 (CH_3), 31.3 (CH), 82.6 (CH), 84.0 (CH), 89.7 (CH), 90.3 (CH), 100.3 (C), 101.6 (C), 120.9 (CH), 121.6 (CH), 123.3 (CH), 127.1 (C), 129.3 (CH), 129.7 (CH), 134.1 (C), 135.9 (CH), 136.9 (CH), 140.9 (C), 153.2 (CH), 155.5 (C), 179.3 (Ru-C) ppm.

Optimisation of the Ruthenium-catalysed *N*-Methylation of Anilines

In a glass pressure tube (25 mL) under an argon atmosphere, ruthenium complex, base, and aniline (91 µl, 1.0 mmol) were dissolved in methanol. Next, the pressure tube was closed, and the resulting mixture was stirred at the stated temperature in an aluminium block for 22 hours. After cooling down to room temperature, the mixture was quenched with ethyl acetate, 100 µl of hexadecane were added, filtrated, and analysed by GC.

Table S1. Ruthenium-catalysed *N*-methylation of aniline with methanol: Full variation of reaction conditions.

#	catalyst loading	base	base loading	temperature [°C]	MeOH [ml]	yield [%]
1	2 mol%	KOtBu	100 mol%	70	1.5	98
2	2 mol%	KOtBu	75 mol%	70	1.5	92
3	2 mol%	KOtBu	50 mol%	70	1.5	92
4	2 mol%	KOtBu	25 mol%	70	1.5	92
5	2 mol%	KOtBu	20 mol%	70	1.5	91
6	2 mol%	KOtBu	10 mol%	70	1.5	91
7	2 mol%	KOtBu	5 mol%	70	1.5	85
8	2 mol%	KOtBu	2 mol%	70	1.5	74
9	2 mol%	NaOtBu	5 mol%	70	1.5	82
10	2 mol%	NaOH	5 mol%	70	1.5	81
11	2 mol%	K_2CO_3	5 mol%	70	1.5	84
12	2 mol%	Cs_2CO_3	5 mol%	70	1.5	82
13	2 mol%	Cs_2CO_3	10 mol%	70	1.5	88
14	2 mol%	NaOH	10 mol%	70	1.5	87
15	2 mol%	NaOtBu	10 mol%	70	1.5	87
16	2 mol%	K_2CO_3	10 mol%	70	1.5	84
17	2 mol%	NEt3	10 mol%	70	1.5	-
18	3 mol%	NaOH	10 mol%	70	1.5	87
19	1 mol%	NaOH	10 mol%	70	1.5	75
20	2 mol%	NaOH	10 mol%	70	0.5	88
21	2 mol%	NaOH	10 mol%	70	2.0	68
22	2 mol%	NaOH	10 mol%	70	0.5 (techn.)	91
23	2 mol%	NaOH	10 mol%	60	0.5 (techn.)	96
24	2 mol%	NaOH	10 mol%	50	0.5 (techn.)	51
25	2 mol%	NaOH	10 mol%	40	0.5 (techn.)	14
26	-	NaOH	10 mol%	60	0.5 (techn.)	-
27	2 mol%	-	-	60	0.5 (techn.)	-
28	[Ru-5]	NaOH	10 mol%	60	0.5 (techn.)	79

Control Experiments for the Ruthenium-catalysed *N*-Methylation of Anilines

In a glass pressure tube (25 mL) under an argon atmosphere, ruthenium complex **9** (8.3 mg, 0.02 mmol), NaOH (4.0 mg, 0.1 mmol), aniline (91µl, 1.0 mmol), and the stated additive were dissolved in methanol (0.5 mL). Next, the pressure tube was closed, and the resulting mixture was stirred at 60 °C in an aluminium block for 22 hours. After cooling down to room temperature, the mixture was quenched with ethyl acetate, 100 µl of hexadecane were added, filtrated, and analysed by GC.

Table S2. Control experiments with different additives.

#	additive	amount	yield [%]
1	PPh_3	0.01 mmol	78
2	PPh_3	0.04 mmol	-
3	1,10-phenanthroline	0.01 mmol	68
4	1,10-phenanthroline	0.04 mmol	-
5	mercury	1 drop	98
6	phenyl imidazoline	0.1 mmol	79
7	*p*-cymene	0.1 mmol	99

Experiments to Determine the Deuteration Ratio of *N*-Methylaniline with Differently Deuterated Methanol

In a glass pressure tube (25 mL) under an argon atmosphere, ruthenium complex **9** (8.3 mg, 0.02 mmol), NaOH (4.0 mg, 0.1 mmol), and aniline (91µl, 1.0 mmol) were dissolved in methanol with the stated deuteration pattern (0.5 mL). Next, the pressure tube was closed, and the resulting mixture was stirred at 60 °C in an aluminium block for 22 hours. After cooling down to room temperature, the mixture was quenched with ethyl acetate, 100 µl of hexadecane were added, filtrated, and analysed by GC-MS. Additionally, the crude was purified by flash chromatography on silica gel to afford, after concentration and drying, the corresponding products, which were analysed by ^{1}H NMR spectroscopy.

Table S3. Product deuteration with differently deuterated methanol derivatives.

#	alcohol	deuteration ratio NH [%]	deuteration ratio Me [%]

1	methanol-d4	35	99
2	methanol-d3	10	98
3	methanol-OD	42	0

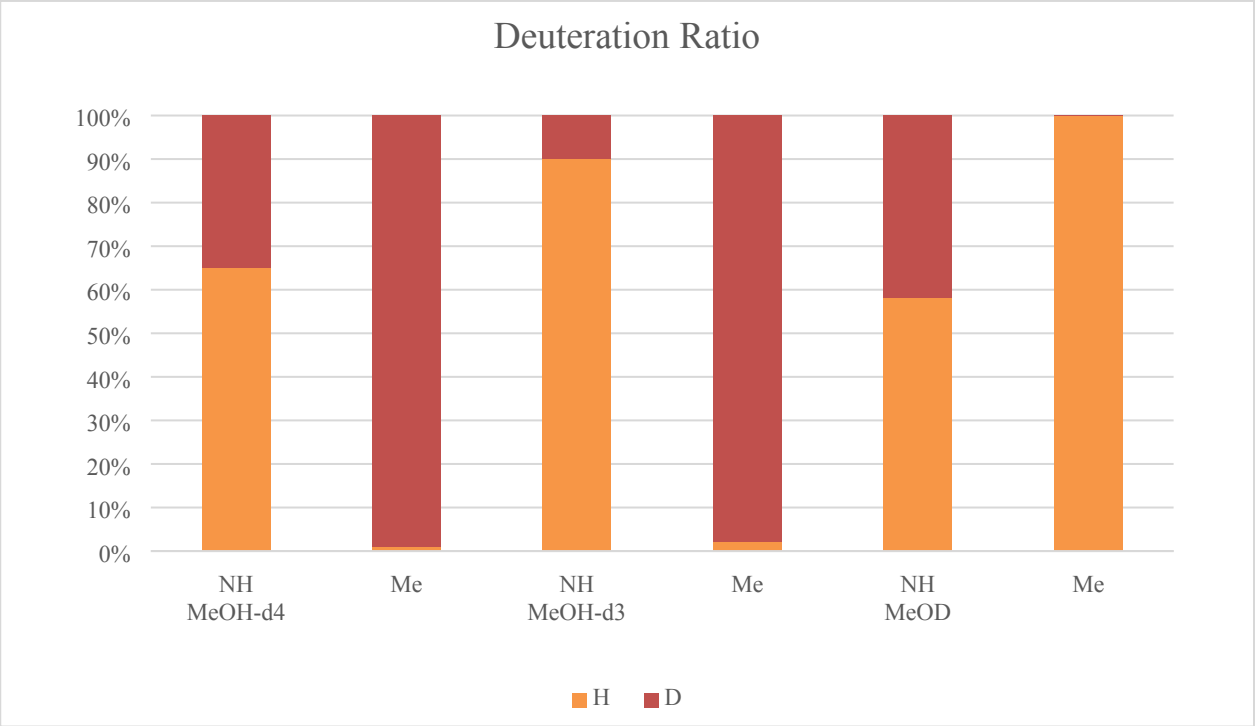
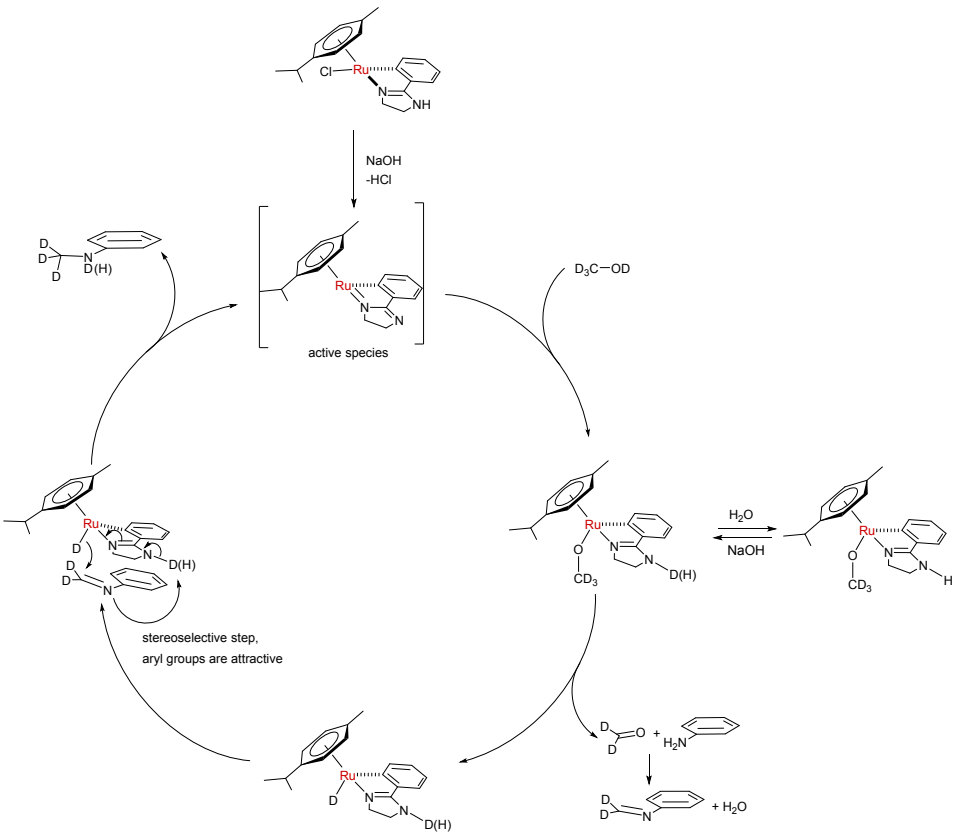


Figure S1. Deuteration of different positions in N-methyl aniline when deuterated methanol derivatives are used.



Scheme S1. Mechanistic proposal for a catalytic cycle.

Yield Time Plot of the Model Reaction

In a glass pressure tube (25 mL) under an argon atmosphere, ruthenium complex **9** (8.3 mg, 0.02 mmol), NaOH (4.0 mg, 0.1 mmol), and aniline (91µl, 1.0 mmol) were dissolved in methanol with the stated deuteration pattern (0.5 mL). Next, the pressure tube was closed, and the resulting mixture was stirred at 60 °C in an aluminium block for the stated time hours. After cooling down to room temperature, the mixture was quenched with ethyl acetate, 100 µl of hexadecane were added, filtrated, and analysed by GC.

Table S4. Ruthenium-catalysed *N*-methylation of aniline with methanol after different times with differently deuterated methanol derivatives.

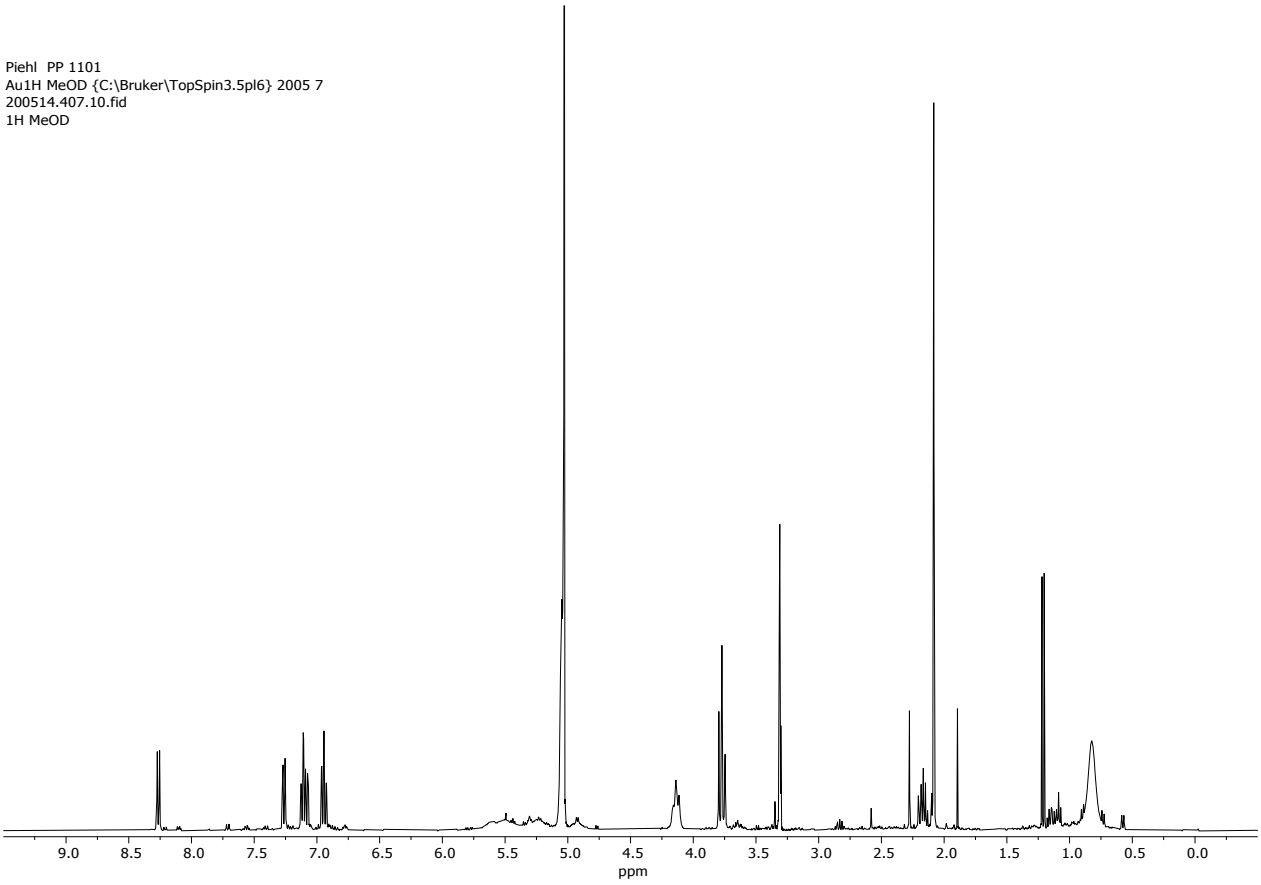
#	Alcohol	time [h]	yield [%]
1.1	methanol	1	13
1.2	methanol	3	39
1.3	methanol	5	71
2.1	methanol-OD	1	16
2.2	methanol-OD	3	47
2.3	methanol-OD	5	73
3.1	methanol-d3	1	5
3.2	methanol-d3	3	19
3.3	methanol-d3	5	41
4.1	methanol-d4	1	7

4.2	methanol-d4	3	22
4.3	methanol-d4	5	45

Control Experiments for the Ruthenium-catalysed *N*-Methylation of Anilines

In a J.-Young-NMR tube, ruthenium complex **9** (8.3 mg, 0.02 mmol) and NaOH (4.0 mg, 0.1 mmol) were suspended in 0.7 mL of MeOH-d4. After the mixture formed a clear orange solution ^{1}H NMR of it was recorded.

^{1}H NMR (400 MHz, MeOD) δ = 8.32 – 8.23 (m, 1H), 7.26 (dd, *J*=7.5, 1.4, 1H), 7.11 (td, *J*=7.4, 1.4, 1H), 6.94 (td, *J*=7.4, 1.1, 1H), 5.46 (br. m, 2H), 5.35 – 5.17 (br. m, 1H), 4.92 (br. m, 1H), 4.14 (t, *J*=9.8, 2H), 3.82 – 3.72 (m, 2H), 2.22 – 2.12 (m, 1H), 2.08 (s, 3H), 0.82 (s, 6H) ppm.



General Procedure for the Ruthenium-catalysed *N*-Methylation of Anilines

In a glass pressure tube (25 mL) under an argon atmosphere, ruthenium complex **9** (8.3 mg, 0.02 mmol), NaOH (4.0 mg, 0.1 mmol), and the aniline derivative (1.0 mmol) were dissolved the respective alcohol (0.5 mL). Next, the pressure tube was closed, and the resulting mixture was stirred at 60 °C in an aluminium block for 22 hours. After cooling down to room temperature, the crude was directly purified by flash

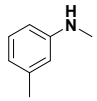
chromatography on silica gel to afford, after concentration and drying, the corresponding products were obtained in the reported yields.

N-methylaniline (1a): Following the general procedure, N-methylaniline was obtained as a colourless liquid (102.5 mg, 96%).

^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.86 (s, 3H), 3.69 (s, 1H), 6.59 – 6.69 (m, 2H), 6.74 (tt, *J*=7.5, 1.1, 1H), 7.17 – 7.27 (m, 2H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 30.8 (CH3), 112.5 (2 x CH), 117.4 (CH), 129.3 (2 x CH), 149.5 (C) ppm; **GCMS** (EI): *m/z* (%): 51 (8), 65 (7), 77 (20), 78 (7), 79 (16), 106 (100), 107 (79) [M^+], 108 (7); **HRMS** (ESI-TOF): *m/z*: calcd for C_7H_9N: 108.0813 [M+H]$^+$; found: 108.0811.

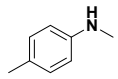
N,2-dimethylaniline (1b): Following the general procedure except using 30 mol% NaOH and a reaction temperature of 100 °C, N,2-dimethylaniline was obtained as a colourless liquid (42.8 mg, 35%, determined by GC using hexadecane as internal standard).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.16 (s, 3H), 2.92 (s, 3H), 3.58 (s, 1H), 6.64 (dd, *J*=8.1, 1.2, 1H), 6.70 (td, *J*=7.4, 1.2, 1H), 7.08 (ddq, *J*=7.3, 1.7, 0.8, 1H), 7.19 (dddd, *J*=8.0, 7.4, 1.6, 0.6, 1H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 17.5 (CH_3), 30.9 (CH_3), 109.2 (CH), 117.0 (CH), 122.0 (C), 127.3 (CH), 130.0 (CH), 147.3 (CH) ppm; **GCMS** (EI): *m/z* (%): 77 (12), 91 (26), 106 (79), 120 (63), 121 (100) [M^+]; **HRMS** (ESI-TOF): *m/z*: calcd for $C_8H_{11}N$: 122.0969 [M+H]$^+$; found: 122.0972.



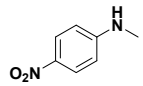
N,3-dimethylaniline (1c): Following the general procedure, N,3-dimethylaniline was obtained as a colourless liquid (120.2 mg, 99%, determined by GC using hexadecane as internal standard).

^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.31 (q, *J*=0.7, 3H), 2.84 (s, 3H), 3.60 (s, 1H), 6.40 – 6.50 (m, 2H), 6.51 – 6.61 (m, 1H), 7.04 – 7.16 (m, 1H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 21.8 (CH_3), 30.9 (CH_3), 109.8 (CH), 113.3 (CH), 118.3 (CH), 129.2 (CH), 139.1 (C), 149.5 (C) ppm; **GCMS** (EI): *m/z* (%): 65 (6), 77 (8), 91 (19), 92 (5), 106 (7), 120 (100), 121 (82) [M^+], 122 (7); **HRMS** (ESI-TOF): *m/z*: calcd for $C_8H_{11}N$: 122.0969 [M+H]$^+$; found: 122.0973.



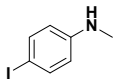
N,4-dimethylaniline (1d): Following the general procedure, N,4-dimethylaniline was obtained as a yellowish liquid (120.1 mg, 99%, determined by GC using hexadecane as internal standard).

^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.27 (d, *J*=0.9, 3H), 2.83 (s, 3H), 3.42 (s, 1H), 6.52 – 6.62 (m, 2H), 6.97 – 7.13 (m, 2H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 20.5 (CH_3), 31.2 (CH_3), 112.7 (2 x CH), 126.6 (C), 129.8 (2 x CH), 147.3 (C) ppm; **GCMS** (EI): *m/z* (%): 77 (7), 91 (14), 106 (7), 120 (100), 121 (73) [M^+], 122 (6); **HRMS** (ESI-TOF): *m/z*: calcd for $C_8H_{11}N$: 122.0969 [M+H]$^+$; found: 122.0970.



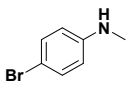
N-methyl-4-nitroaniline (1e): Following the general procedure except using 30 mol% NaOH and a reaction temperature of 80 °C, N-methyl-4-nitroaniline was obtained as a yellow solid (42.8 mg, 28%).

^{1}H NMR (300 MHz, DMSO) δ = 2.78 (d, *J*=5.0, 3H), 6.53 – 6.65 (m, 2H), 7.25 – 7.31 (m, 1H), 7.94 – 8.05 (m, 2H) ppm; **^{13}C NMR** (75 MHz, DMSO) δ = 29.3 (CH_3), 126.4 (4 x CH), 135.7 (C), 155.4 (C) ppm; **GCMS** (EI): *m/z* (%): 65 (19), 77 (30), 79 (18), 105 (13), 106 (17), 122 (45), 152 (100) [M^+]; **HRMS** (ESI-TOF): *m/z*: calcd for $C_7H_8N_2O_2$: 153.0664 [M+H]$^+$; found: 153.0668.



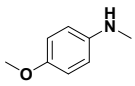
4-iodo-N-methylaniline (1f): Following the general procedure, 4-iodo-N-methylaniline was obtained as a violet oil (121.5 mg, 52%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.80 (s, 3H), 3.69 (s, 1H), 6.35 – 6.43 (m, 2H), 7.38 – 7.48 (m, 2H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ = 30.6 (CH_3), 77.8 (CI), 114.7 (2 x CH), 137.8 (2 x CH), 148.9 (C) ppm; **GCMS** (EI): *m/z* (%): 77 (6), 105 (6), 106 (6), 232 (31), 233 (100) [M^+], 234 (8); **HRMS** (ESI-TOF): *m/z*: calcd for C_7H_8NI: 233.9779 $[M+H]^+$; found: 233.9777.



4-bromo-N-methylaniline (1g): Following the general procedure, 4-bromo-N-methylaniline was obtained as a colourless liquid (177.0 mg, 95%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.81 (s, 3H), 3.73 (s, 1H), 6.44 – 6.53 (m, 2H), 7.22 – 7.31 (m, 2H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 30.8 (CH_3), 108.9 (C), 114.0 (2 x CH), 132.0 (2 x CH), 148.4 (C) ppm; **GCMS** (EI): *m/z* (%): 104 (12), 105 (17), 184 (80), 185 (100) [M^+], 186 (83), 187 (98); **HRMS** (ESI-TOF): *m/z*: calcd for C_7H_9BrN: 185.9918 $[M+H]^+$; found: 185.9919.

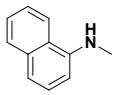


4-methoxy-N-methylaniline (1h): Following the general procedure, 4-methoxy-N-methylaniline was obtained as a yellowish liquid (116.7 mg, 85%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.82 (s, 3H), 3.38 (s, 1H), 3.77 (d, *J*=0.9, 3H), 6.56 – 6.65 (m, 2H), 6.79 – 6.85 (m, 2H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 31.7 (CH_3), 55.9 (CH_3), 113.7 (2 x CH), 115.0 (2 x CH), 143.8 (C), 152.1 (C) ppm; **GCMS** (EI): *m/z* (%): 65 (6), 94 (20), 122 (100), 123 (8), 137 (65) [M^+], 138 (6); **HRMS** (ESI-TOF): *m/z*: calcd for $C_8H_{11}NO$: 138.0919 $[M+H]^+$; found: 138.0921.

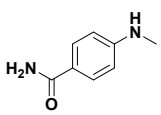
N-methylpyridin-2-amine (1i): Following the general procedure except using 30 mol% NaOH and a reaction temperature of 80 °C, N-methylpyridin-2-amine was obtained as a yellowish oil (16.3 mg, 15%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.90 (s, 3H), 4.61 (s, 1H), 6.37 (dt, *J*=8.4, 1.0, 1H), 6.56 (ddd, *J*=7.1, 5.0, 1.0, 1H), 7.42 (ddd, *J*=8.4, 7.1, 1.9, 1H), 8.08 (ddd, *J*=5.0, 1.9, 0.9, 1H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 29.2 (CH_3), 106.3 (CH), 112.8 (CH), 137.5 (CH), 148.3 (CH), 159.7 (C) ppm; **GCMS** (EI): *m/z* (%): 51 (11), 52 (19), 78 (34), 79 (76), 80 (51), 107 (57), 108 (100) [M^+]; **HRMS** (ESI-TOF): *m/z*: calcd for $C_6H_8N_2$: 109.0765 $[M+H]^+$; found: 109.0769.



N-methylnaphthalen-1-amine (1k): Following the general procedure except using 30 mol% NaOH and a reaction temperature of 80 °C, N-methylnaphthalen-1-amine was obtained as a reddish-brown liquid (102.8 mg, 65%).

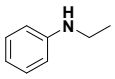
^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.04 (s, 3H), 4.43 (s, 1H), 6.64 (dd, *J*=7.6, 1.0, 1H), 7.26 – 7.33 (m, 1H), 7.39 – 7.54 (m, 3H), 7.75 – 7.90 (m, 2H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 31.1 (CH_3), 103.9 (CH), 117.4 (CH), 119.9 (CH), 123.5 (C), 124.8 (CH), 125.8 (CH), 126.8 (CH), 128.8 (CH), 134.3 (C), 144.6 (C) ppm; **GCMS** (EI): *m/z* (%): 115 (25), 127 (10), 128 (23), 129 (21), 156 (43), 157 (100) [M^+], 158 (12); **HRMS** (ESI-TOF): *m/z*: calcd for $C_{11}H_{11}N$: 158.0969 $[M+H]^+$; found: 158.0969.



4-(methylamino)benzamide (1l): Following the general procedure except using 30 mol% NaOH and a reaction temperature of 80 °C, 4-(methylamino)benzamide was obtained as a faintly brownish solid (144.0 mg, 96%).

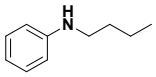
^{1}H NMR (300 MHz, DMSO) δ = 2.70 (d, *J*=5.0, 3H), 6.16 (q, *J*=5.0, 1H), 6.45 – 6.57 (m, 2H), 6.85 (s, 1H), 7.54 (s, 1H), 7.61 – 7.72 (m, 2H) ppm; **^{13}C NMR** (75 MHz, DMSO) δ = 29.3 (CH_3), 110.3 (2 x CH), 120.7 (C), 129.1

(2 x CH), 152.2 (C), 168.1 (C(O)NH_2) ppm; **GCMS** (EI): *m/z* (%): 65 (6), 77 (13), 79 (10), 106 (14), 131 (8), 132 (9), 134 (100), 135 (9), 149 (6), 150 (90) [M^+], 151 (9); **HRMS** (ESI-TOF): *m/z*: calcd for $C_8H_{10}N_2O$: 151.0871 $[M+H]^+$; found: 151.0870.



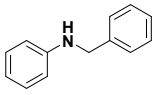
N-ethylaniline (3a): Following the general procedure, N-ethylaniline was obtained as a colorless liquid (82.1 mg, 68%, determined by GC using hexadecane as internal standard).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.27 (t, *J*=7.1, 3H), 3.17 (q, *J*=7.1, 2H), 3.50 (s, 1H), 6.58 – 6.66 (m, 2H), 6.67 – 6.76 (m, 1H), 7.14 – 7.24 (m, 2H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 15.0 (CH_3), 38.6 (CH_2), 112.9 (2 x CH), 117.3 (CH), 129.3 (2 x CH), 148.6 (C) ppm; **GCMS** (EI): *m/z* (%): 77 (16), 79 (8), 106 (100), 107 (8), 120 (6), 121 (43) [M^+]; **HRMS** (ESI-TOF): *m/z*: calcd for $C_8H_{11}N$: 122.0969 $[M+H]^+$; found: 122.0972.



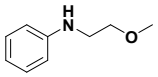
N-butylaniline (3b): Following the general procedure, N-butylaniline was obtained as a colourless liquid (63.4 mg, 43%).

^{1}H NMR (300 MHz, $CDCl_3$) δ = 0.99 (t, *J*=7.3, 3H), 1.37 – 1.57 (m, 2H), 1.53 – 1.71 (m, 2H), 3.08 – 3.20 (m, 2H), 3.59 (s, 1H), 6.57 – 6.67 (m, 2H), 6.71 (tt, *J*=7.4, 1.1, 1H), 7.13 – 7.26 (m, 2H) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 14.0 (CH_3), 20.4 (CH_2), 31.8 (CH_2), 43.8 (CH_2), 112.8 (2 x CH), 117.2 (CH), 129.3 (2 x CH), 148.7 (C) ppm; **GCMS** (EI): *m/z* (%): 77 (10), 106 (100), 107 (9), 149 (25) [M^+]; **HRMS** (ESI-TOF): *m/z*: calcd for $C_{10}H_{15}N$: 150.1283 $[M+H]^+$; found: 150.1284.



N-benzylaniline (3c): Following the general procedure, N-benzylaniline was obtained as a colourless oil (52.4 mg, 29%).

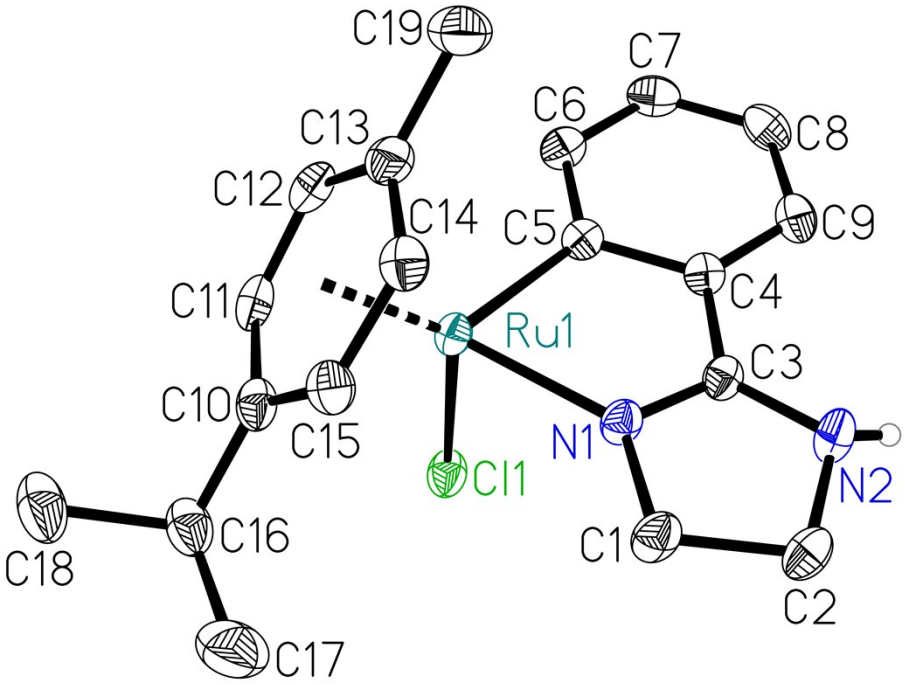
^{1}H NMR (400 MHz, $CDCl_3$) δ = 4.05 (s, 1H), 4.37 (s, 2H), 6.64 – 6.72 (m, 2H), 6.76 (tt, *J*=7.4, 1.1, 1H), 7.17 – 7.28 (m, 2H), 7.25 – 7.37 (m, 1H), 7.37 – 7.45 (m, 4H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 48.4 (CH3), 112.9 (2 x CH), 117.7 (CH), 127.3 (CH), 127.6 (2 x CH), 128.8 (2 x CH), 129.4 (2 x CH), 139.6 (C), 148.3 (C) ppm; **GCMS** (EI): *m/z* (%): 65 (11), 77 (13), 91 (83), 106 (18), 182 (37), 183 (100) [M^+], 184 (14); **HRMS** (ESI-TOF): *m/z*: calcd for $C_{13}H_{13}N$: 184.1126 $[M+H]^+$; found: 184.1122.



N-(2-methoxyethyl)aniline (3d): Following the general procedure except using 30 mol% NaOH and a reaction temperature of 80 °C, N-(2-methoxyethyl)aniline was obtained as a colourless liquid (86.9 mg, 57%).

^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.31 (dd, *J*=5.6, 4.8, 2H), 3.41 (s, 3H), 3.62 (dd, *J*=5.7, 4.8, 2H), 4.02 (s, 1H), 6.61 – 6.69 (m, 2H), 6.73 (tt, *J*=7.3, 1.1, 1H), 7.15 – 7.25 (m, 2H) ppm; **^{13}C NMR** (101 MHz, $CDCl_3$) δ = 43.6 (CH2), 58.9 (CH3), 71.1 (CH2), 113.2 (2 x CH), 117.7 (CH), 129.3 (2 x CH), 148.3 (C) ppm; **GCMS** (EI): *m/z* (%): 77 (13), 79 (6), 106 (100), 107 (8), 151 (25) [M^+]; **HRMS** (ESI-TOF): *m/z*: calcd for $C_9H_{13}NO$: 152.1075 $[M+H]^+$; found: 152.1075.

X-ray crystal structure analysis of ruthenium complex 9



Molecular structure of ruthenium complex 9 in the crystal. Displacement ellipsoids correspond to 50% probability. Carbon-bound hydrogen atoms are omitted for clarity.

Data were collected on a Bruker Kappa APEX II Duo diffractometer. The structure was solved by direct methods (SHELXS-97: Sheldrick, G. M. *Acta Cryst.* **2008**, *A64*, 112.) and refined by full-matrix least-squares procedures on F^2 (SHELXL-2018: Sheldrick, G. M. *Acta Cryst.* **2015**, *C71*, 3.). XP (Bruker AXS) was used for graphical representation.
CCDC 2038933 contains the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

Crystal data of ruthenium complex **9**: $C_{19}H_{23}ClN_2Ru$, M = 415.91, monoclinic, space group $P2_1/n$, a = 10.2420(8), b = 13.4921(11), c = 12.6747(10) Å, b = 94.3124(14)°, V = 1746.5(2) Å^3, T = 150(2) K, Z = 4, 42533 reflections measured, 4226 independent reflections (R_{int} = 0.0226), final R values ($I > 2\sigma(I)$): R_1 = 0.0197, wR_2 = 0.0497, final R values (all data): R_1 = 0.0217, wR_2 = 0.0515, 214 parameters.

NMR and IR Spectra

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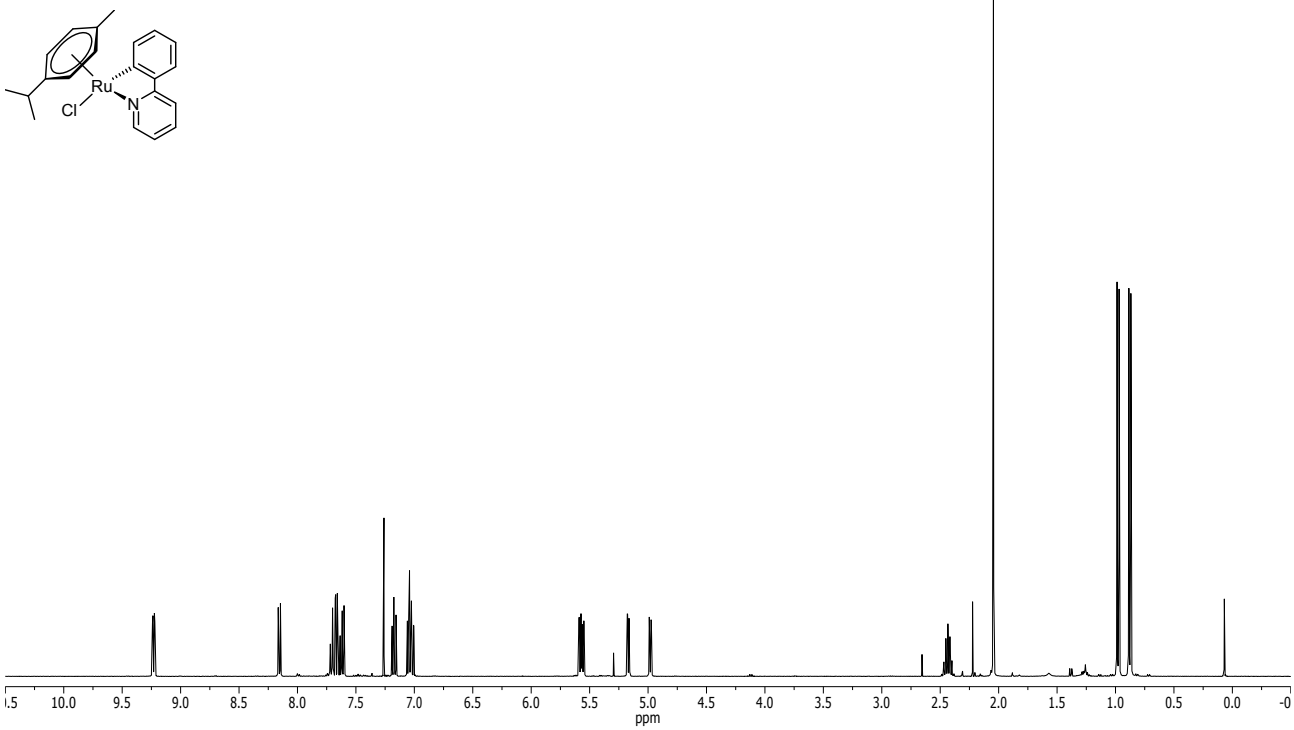
1H CDCl3



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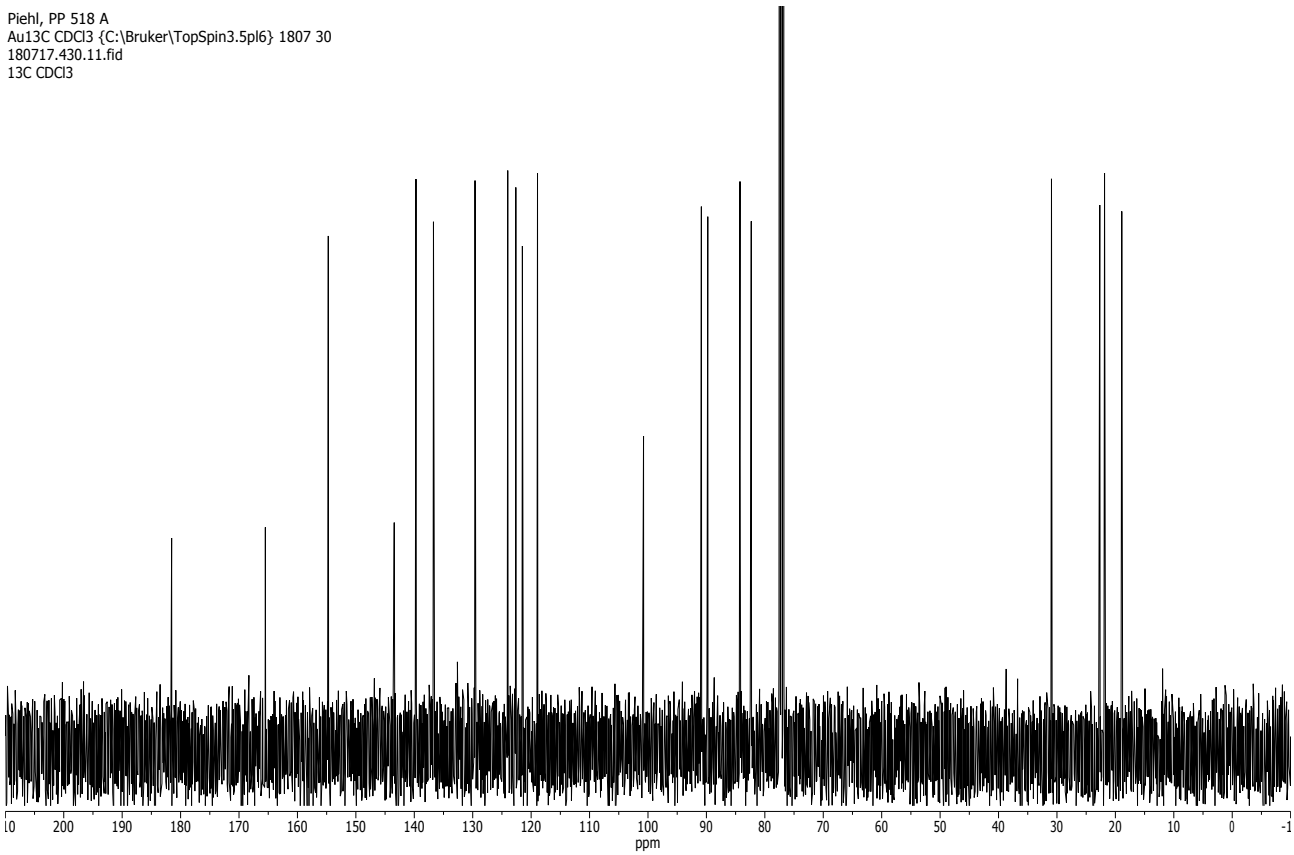

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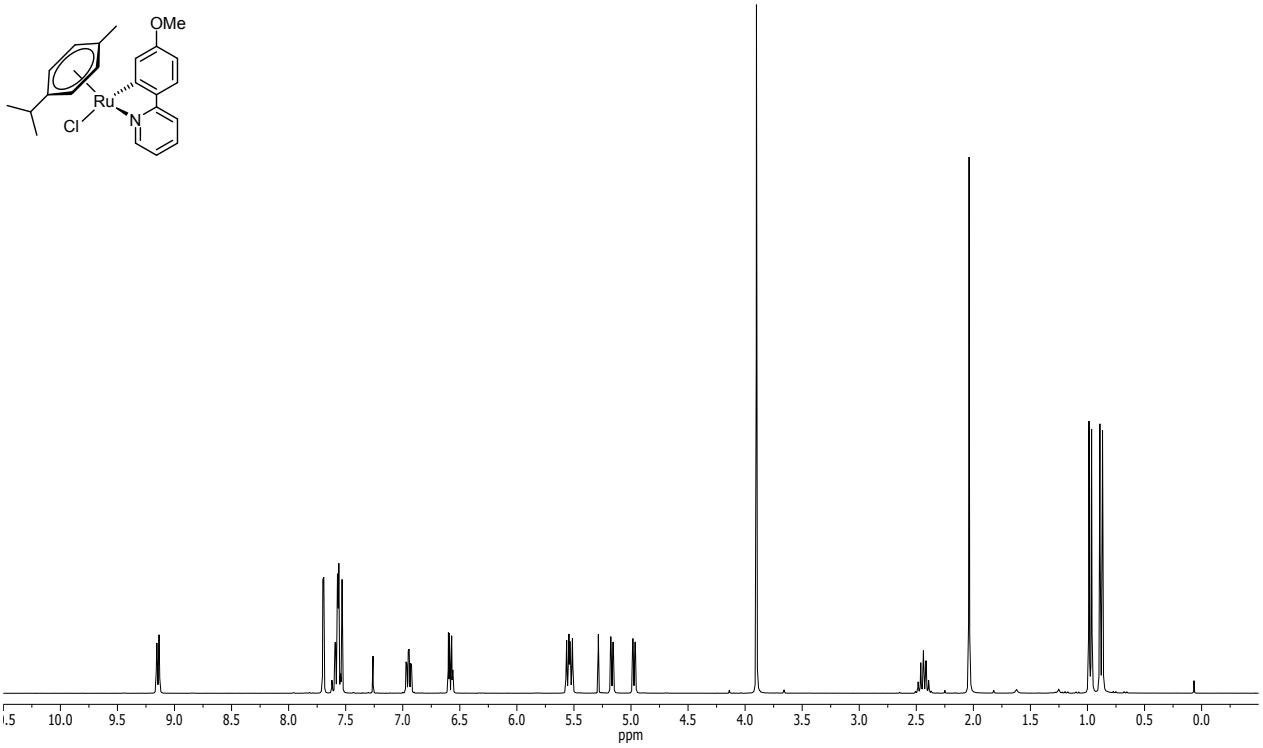

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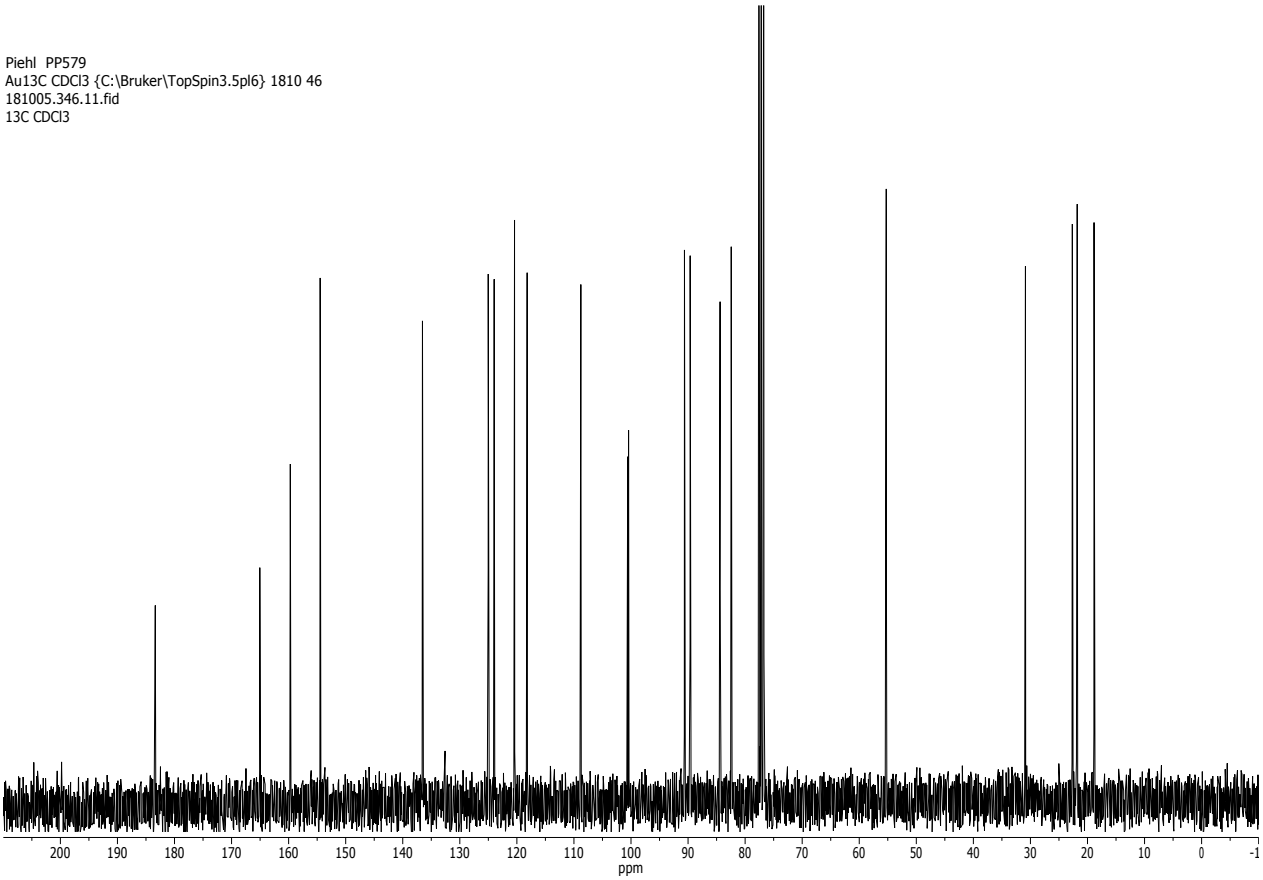

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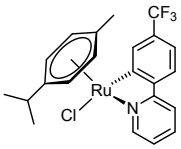

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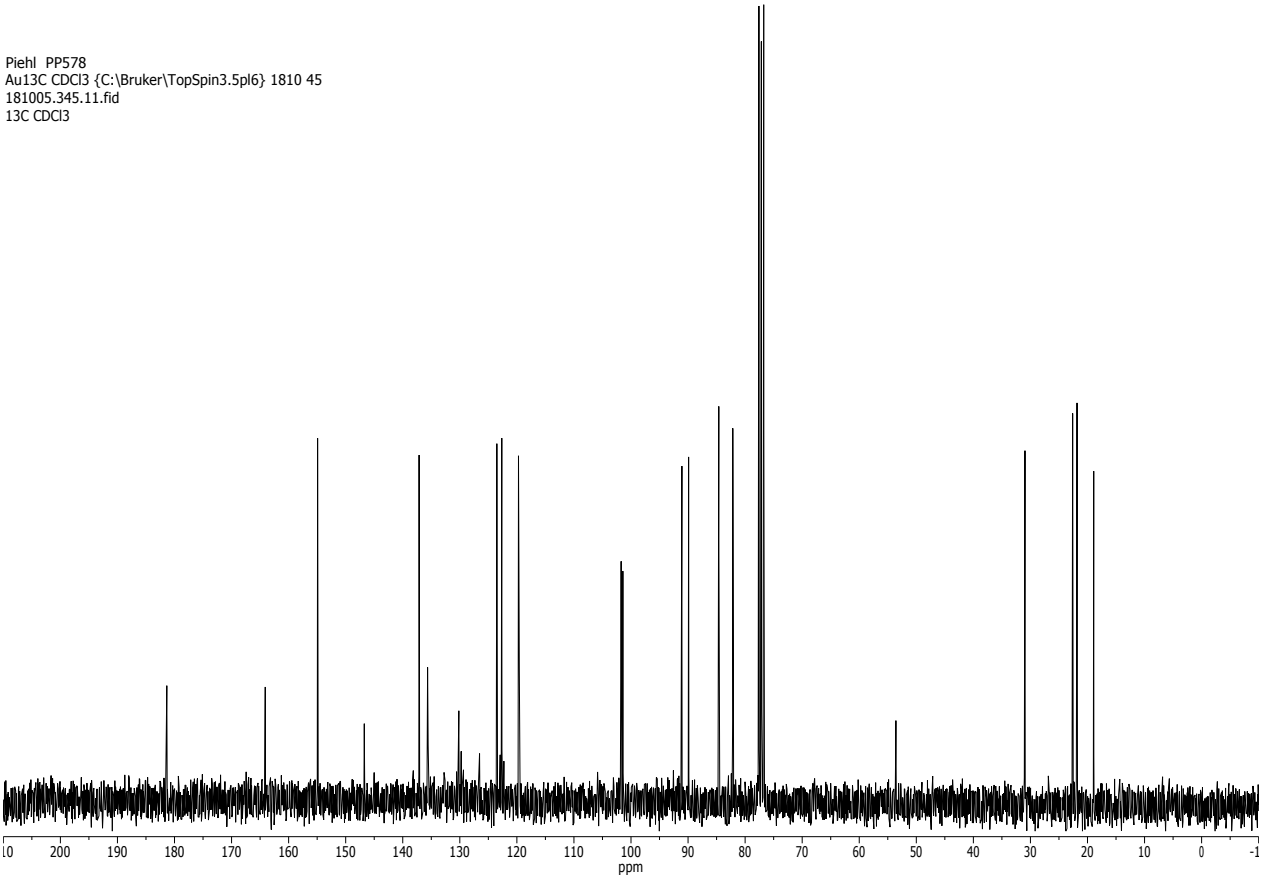
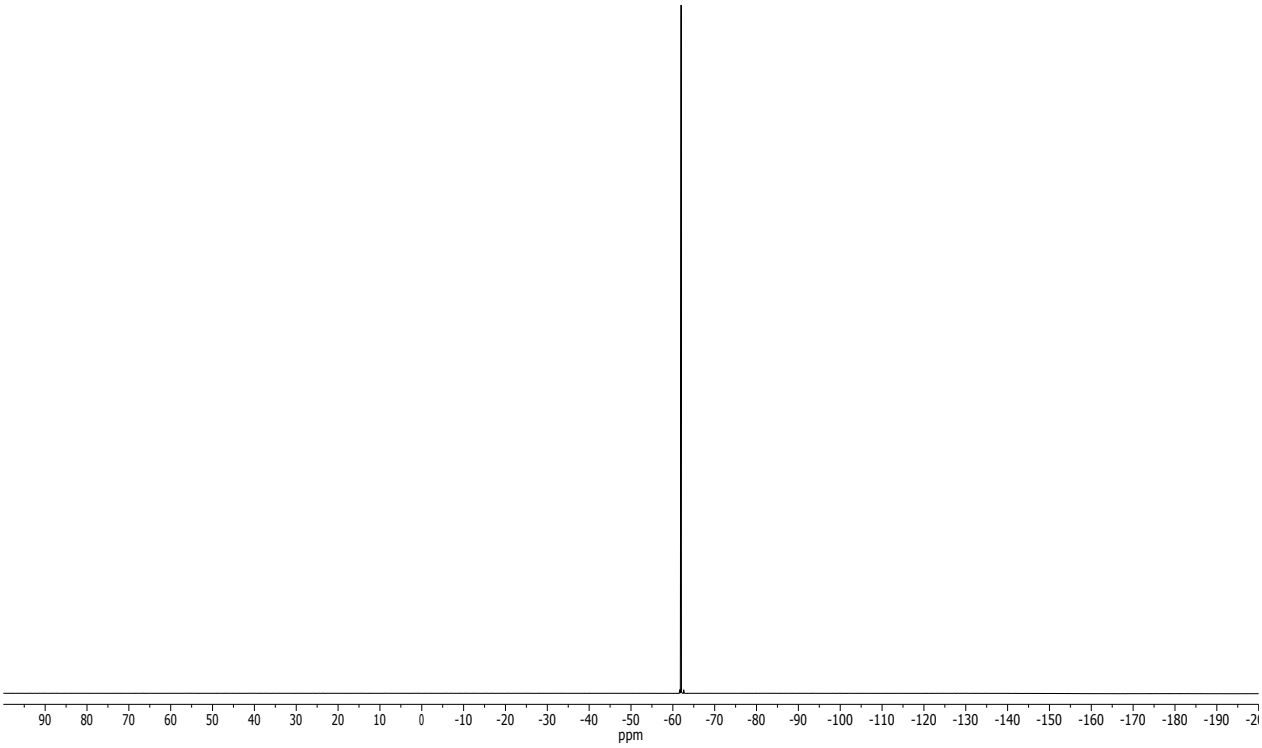
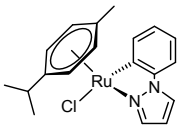


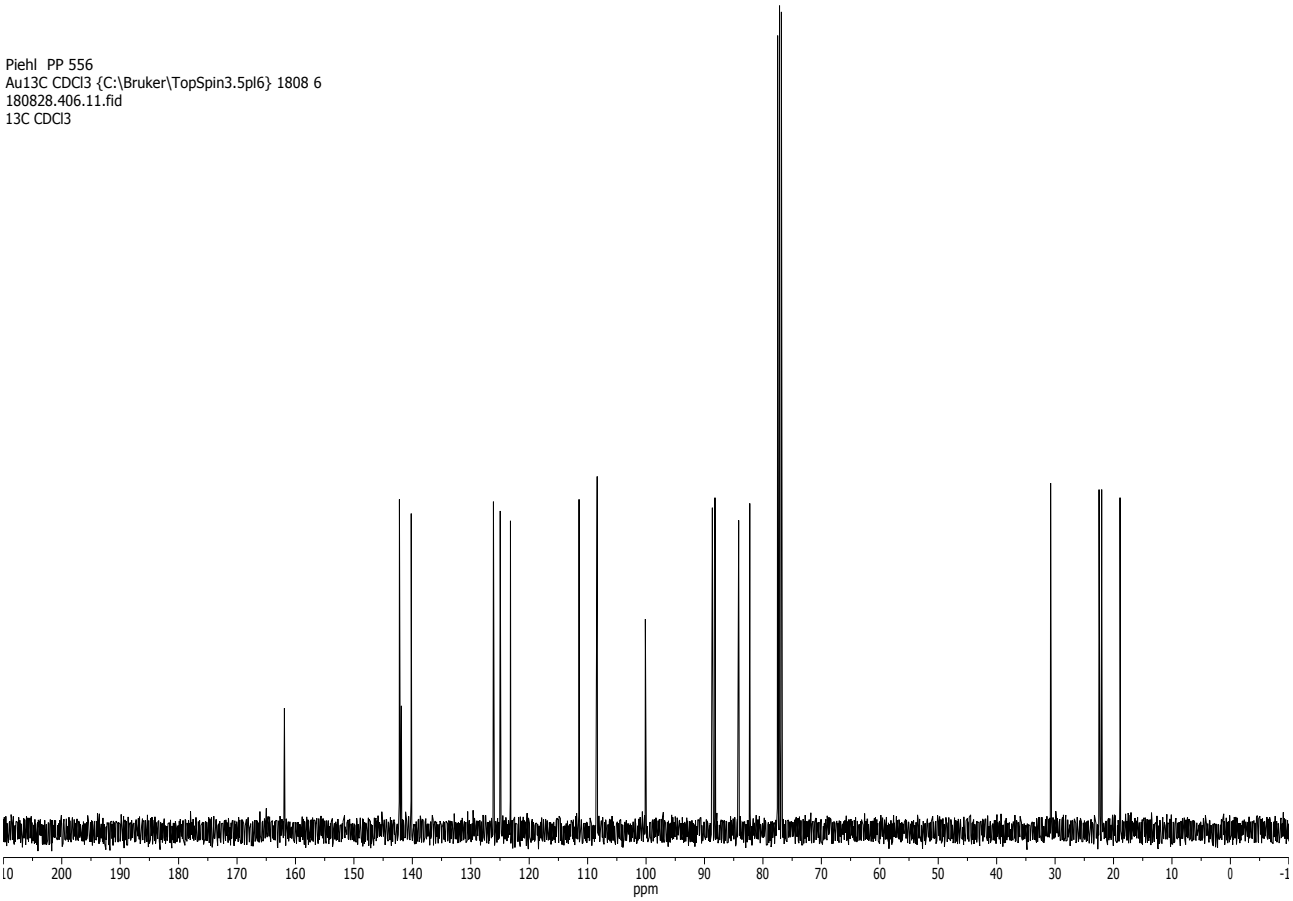
Piehl PP578
Au19F CDCl3 {C:\Bruker\TopSpin3.5pl6} 1810 45
181005.345.13.fid
19F CDCl3



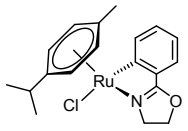
Piehl PP 556
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1808 6
180828.406.10.fid
1H CDCl3



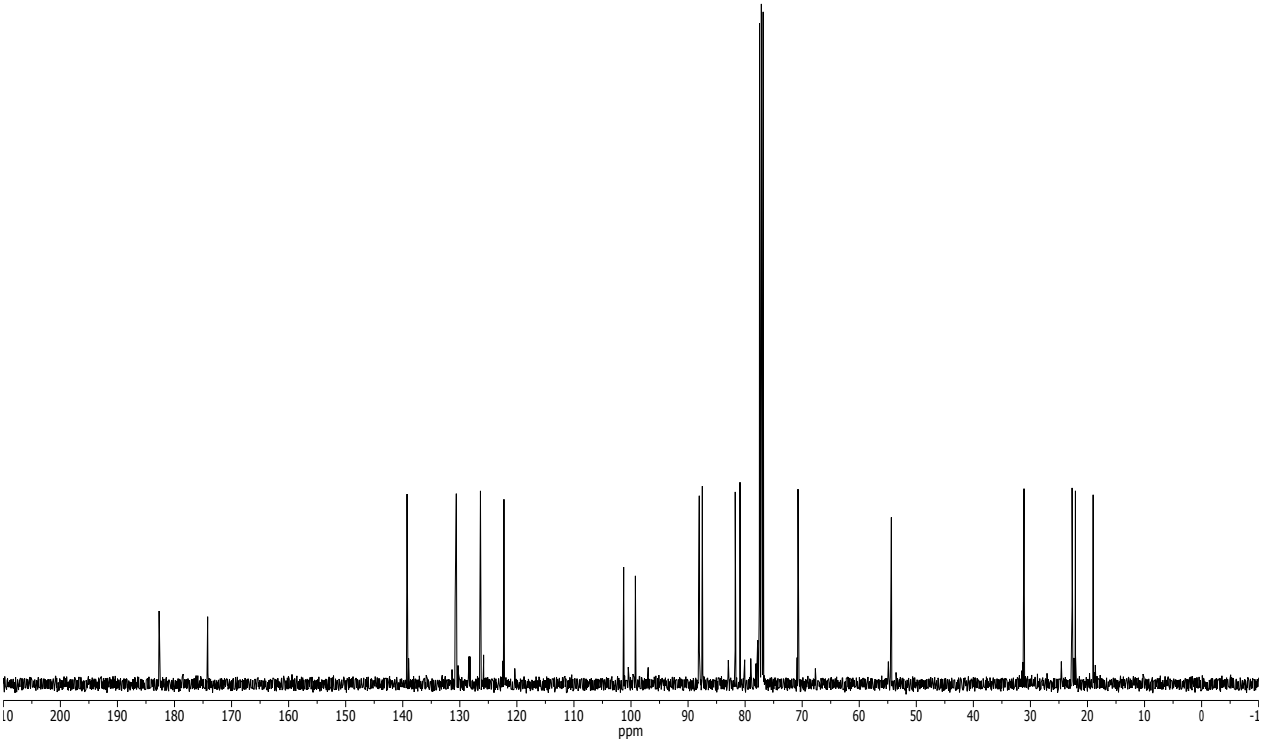
Piehl PP 556
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1808 6
180828.406.11.fid
13C CDCl3



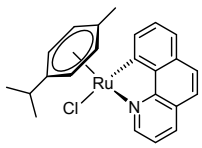
Piehl PP 538A
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1807 40
180730.440.10.fid
1H CDCl3



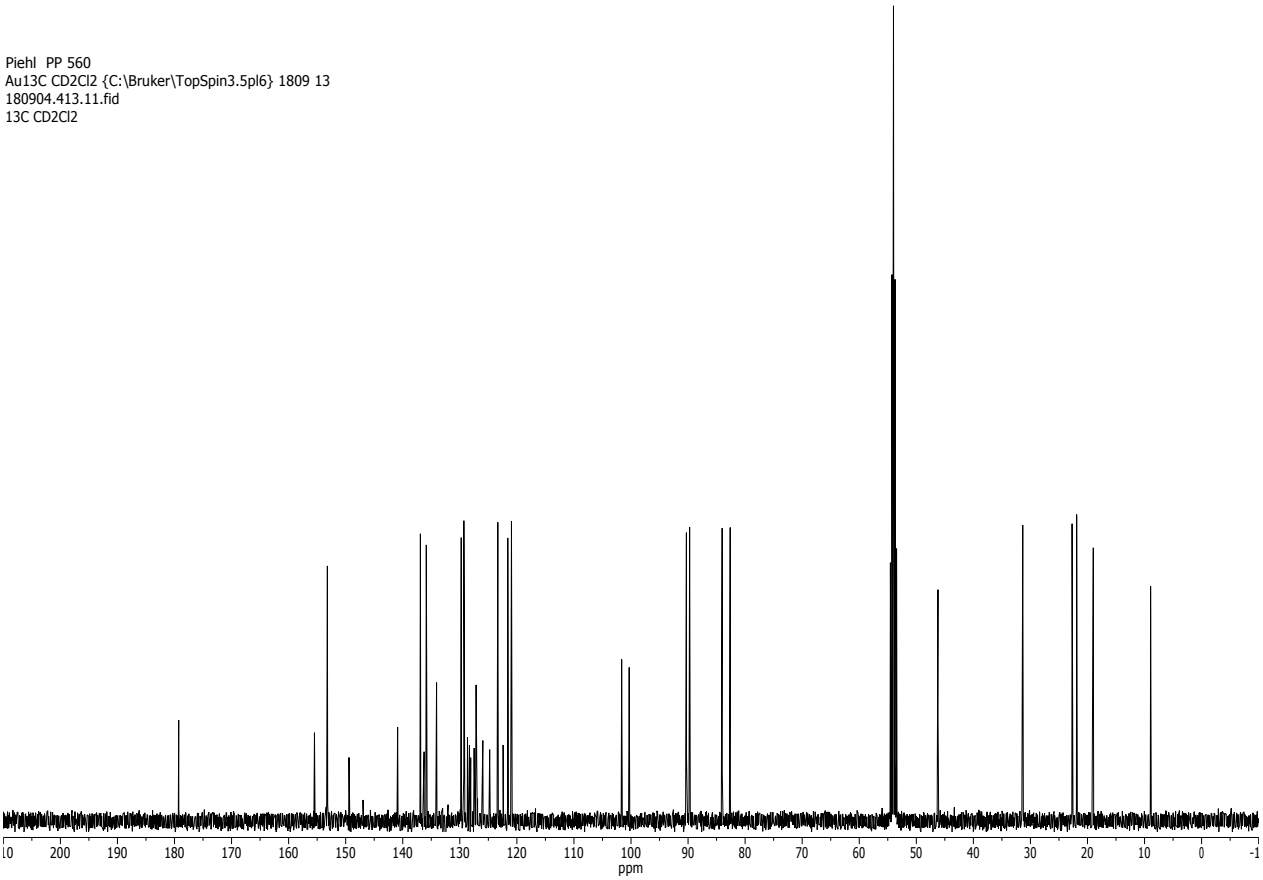
Piehl PP 538A
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1807 40
180730.440.11.fid
13C CDCl3



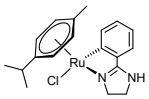
Piehl PP 560
Au1H CD2Cl2 {C:\Bruker\TopSpin3.5pl6} 1809 13
180904.413.10.fid
1H CD2Cl2



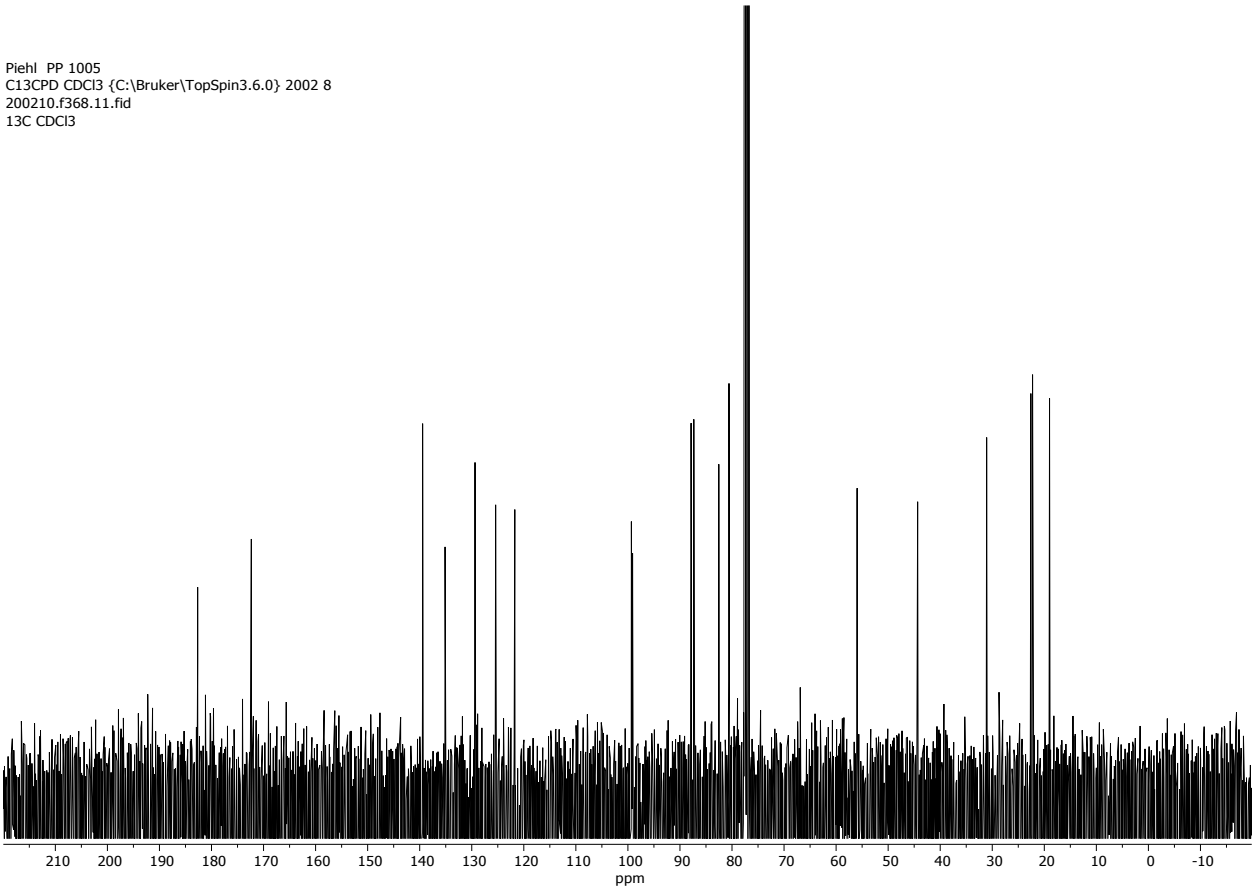
Piehl PP 560
Au13C CD2Cl2 {C:\Bruker\TopSpin3.5pl6} 1809 13
180904.413.11.fid
13C CD2Cl2



Piehl PP 1005
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 8
200210.f368.10.fid
1H CDCl3



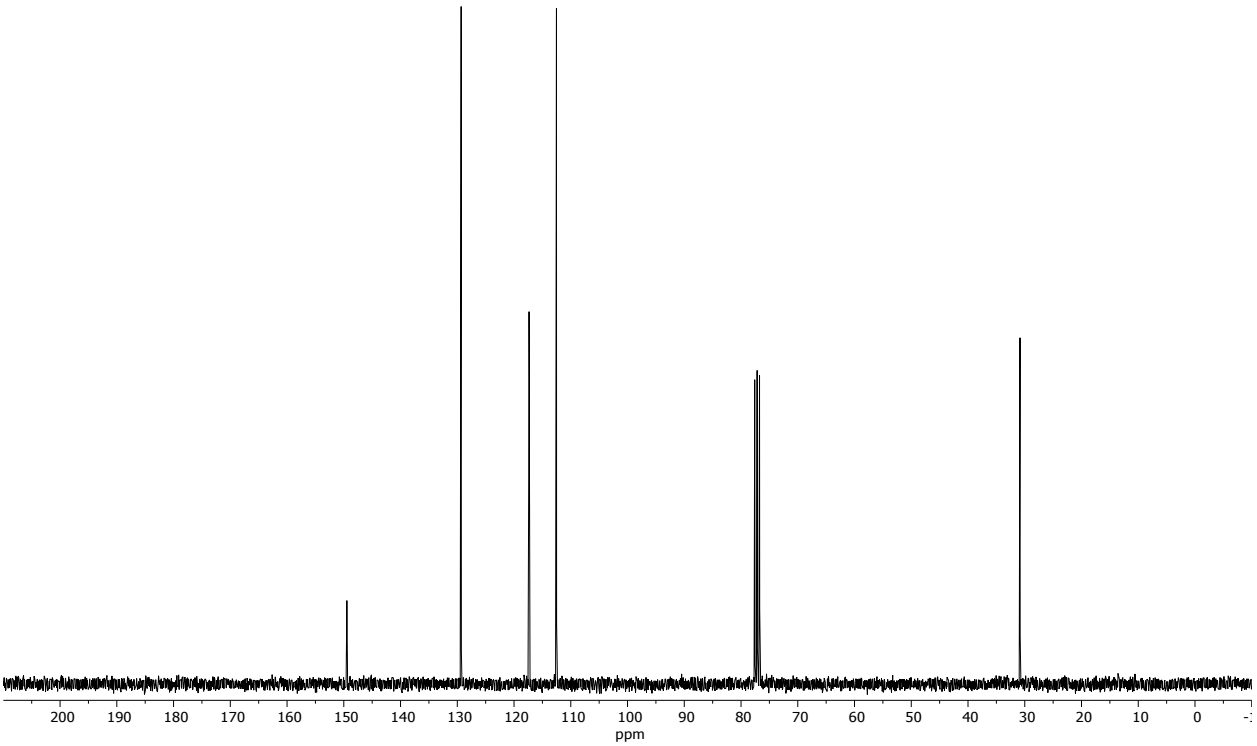
Piehl PP 1005
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 8
200210.f368.11.fid
13C CDCl3



Piehl PP 984B
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 40
200115.f340.10.fid
1H CDCl3

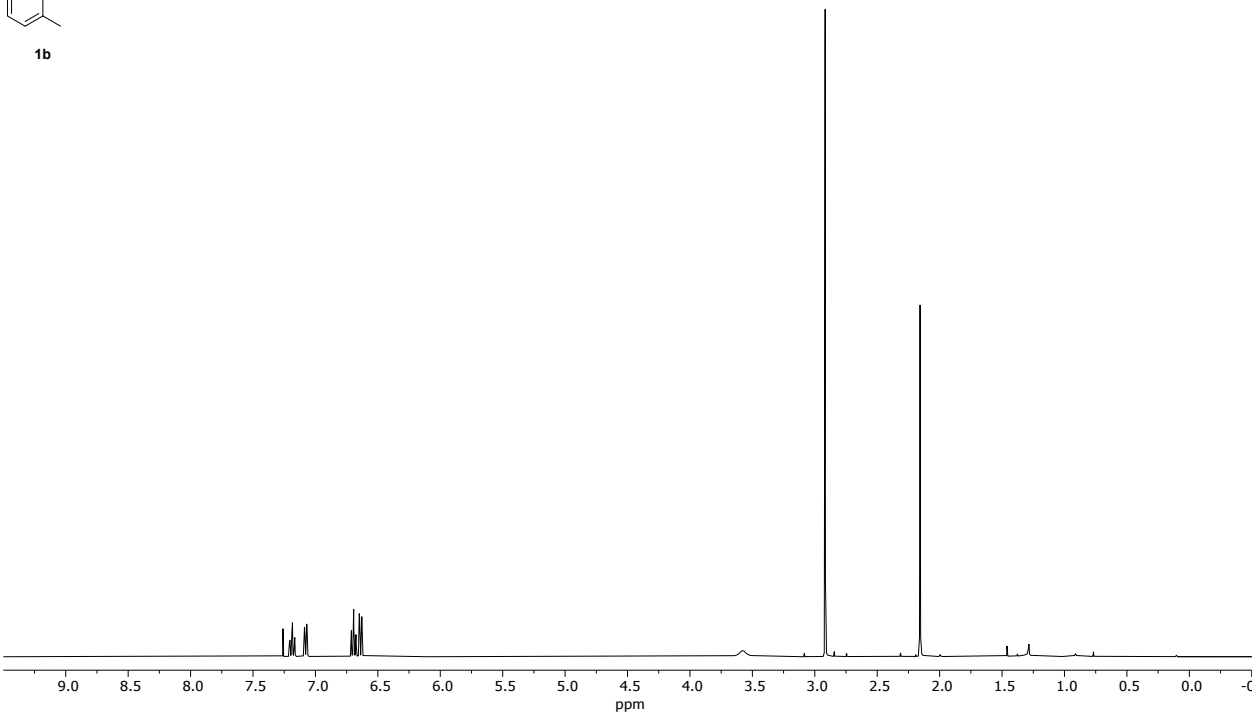
1a

Piehl PP 984B
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 40
200115.f340.11.fid
13C CDCl3

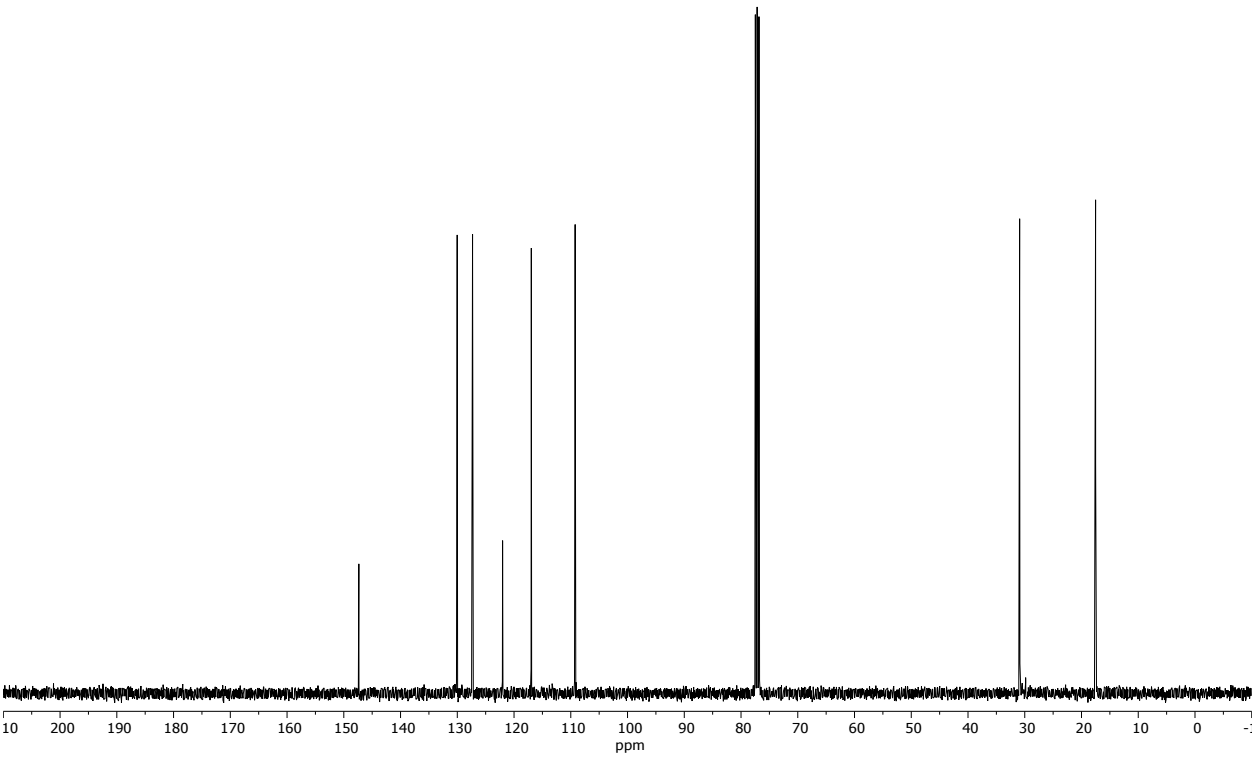


Piehl / PP1053B
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 38
200323.438.10.fid
1H CDCl3

1b



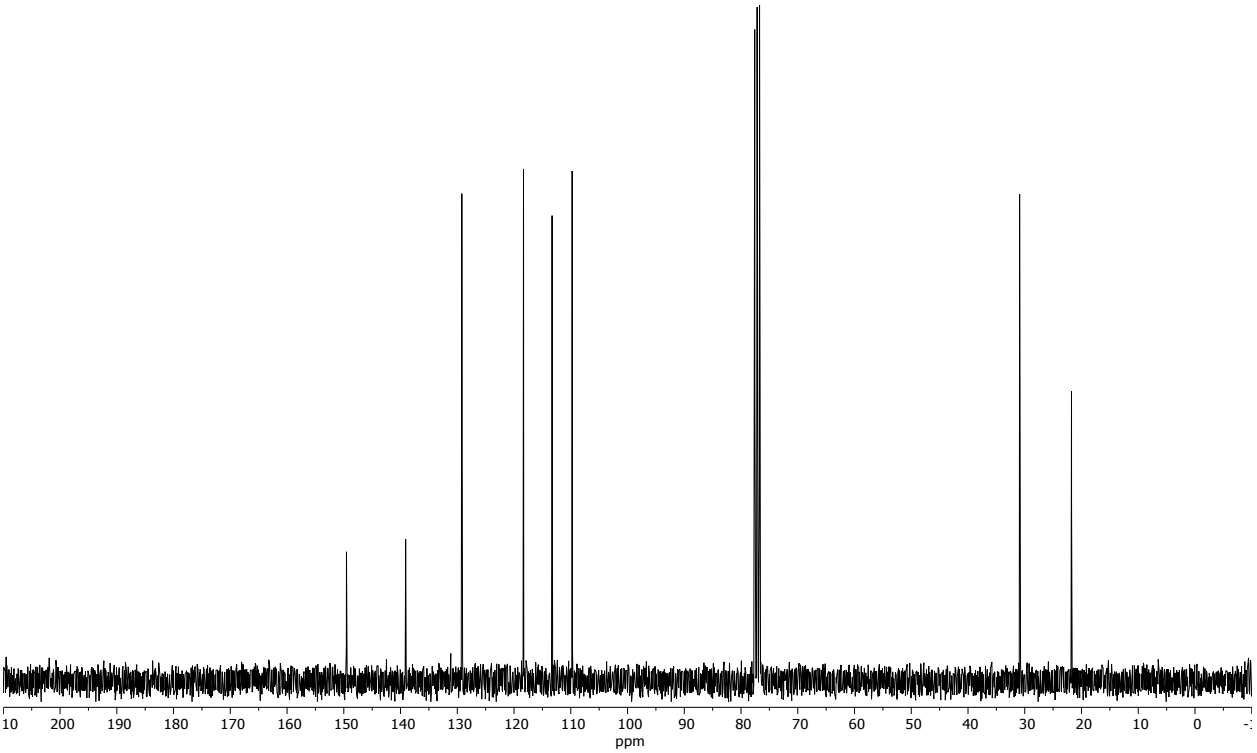
Piehl / PP1053B
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 38
200323.438.11.fid
13C CDCl3



Piehl PP 1001 B
Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 11
200211.311.10.fid
1H CDCl3

1c

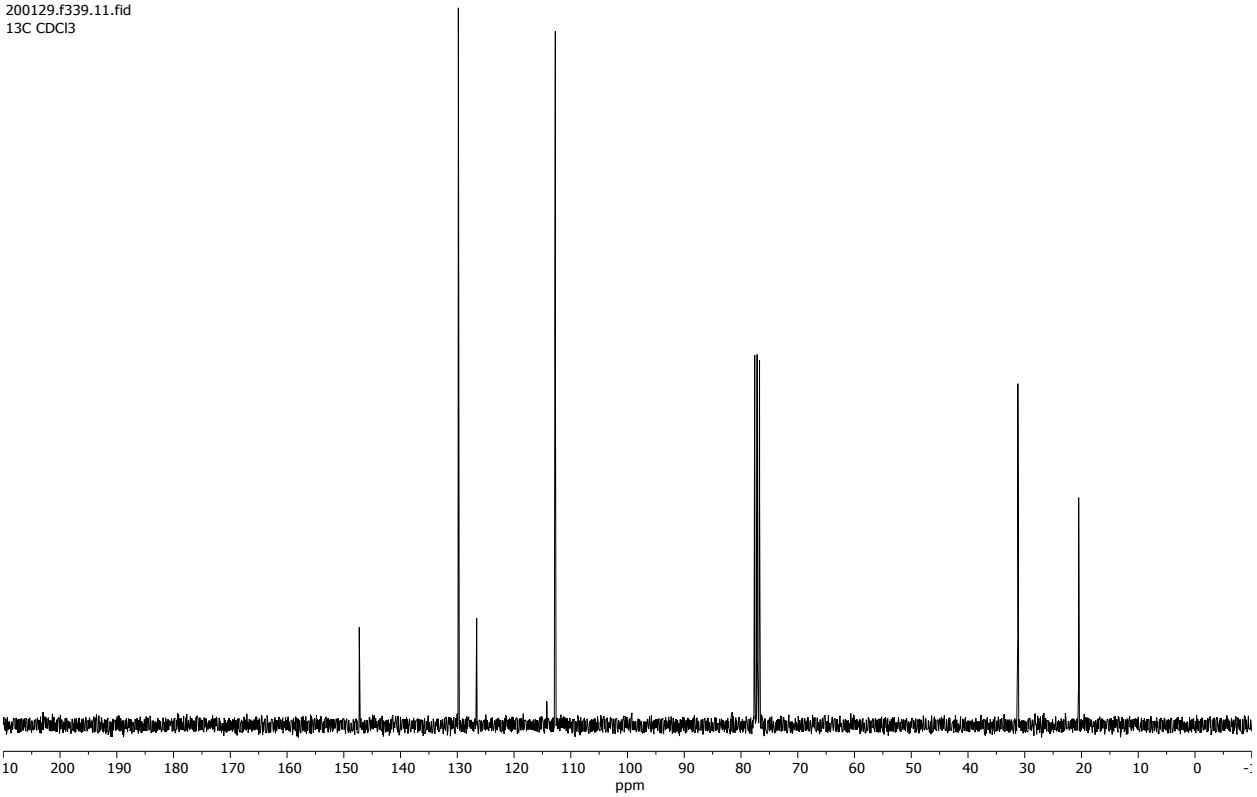
Piehl PP 1001 B
Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 11
200211.311.11.fid
13C CDCl3



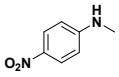
Piehl PP 994
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 39
200129.f339.10.fid
1H CDCl3

1d

Piehl PP 994
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 39
200129.f339.11.fid
13C CDCl3

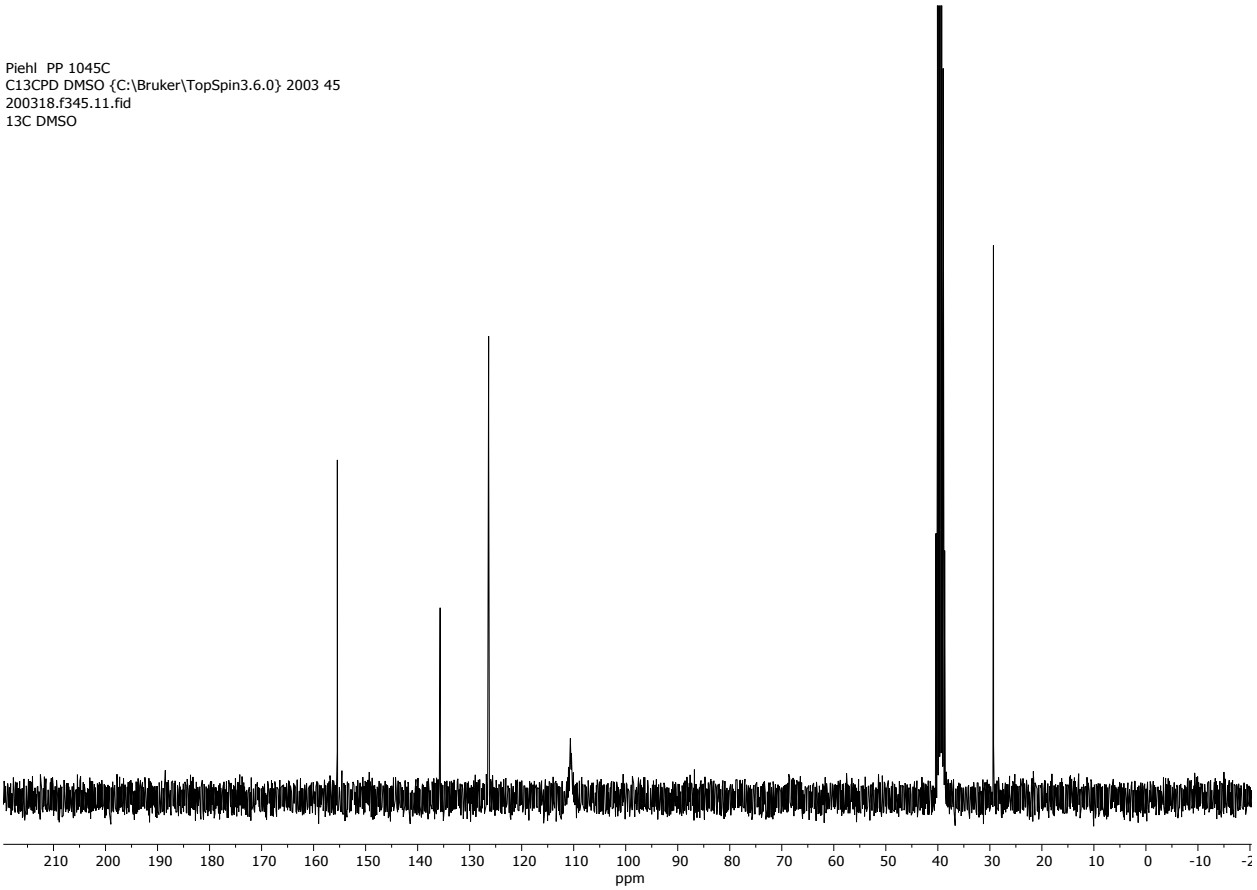


Piehl PP 1045C
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 45
200318.f345.10.fid
1H DMSO

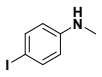


1e

Piehl PP 1045C
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 45
200318.f345.11.fid
13C DMSO

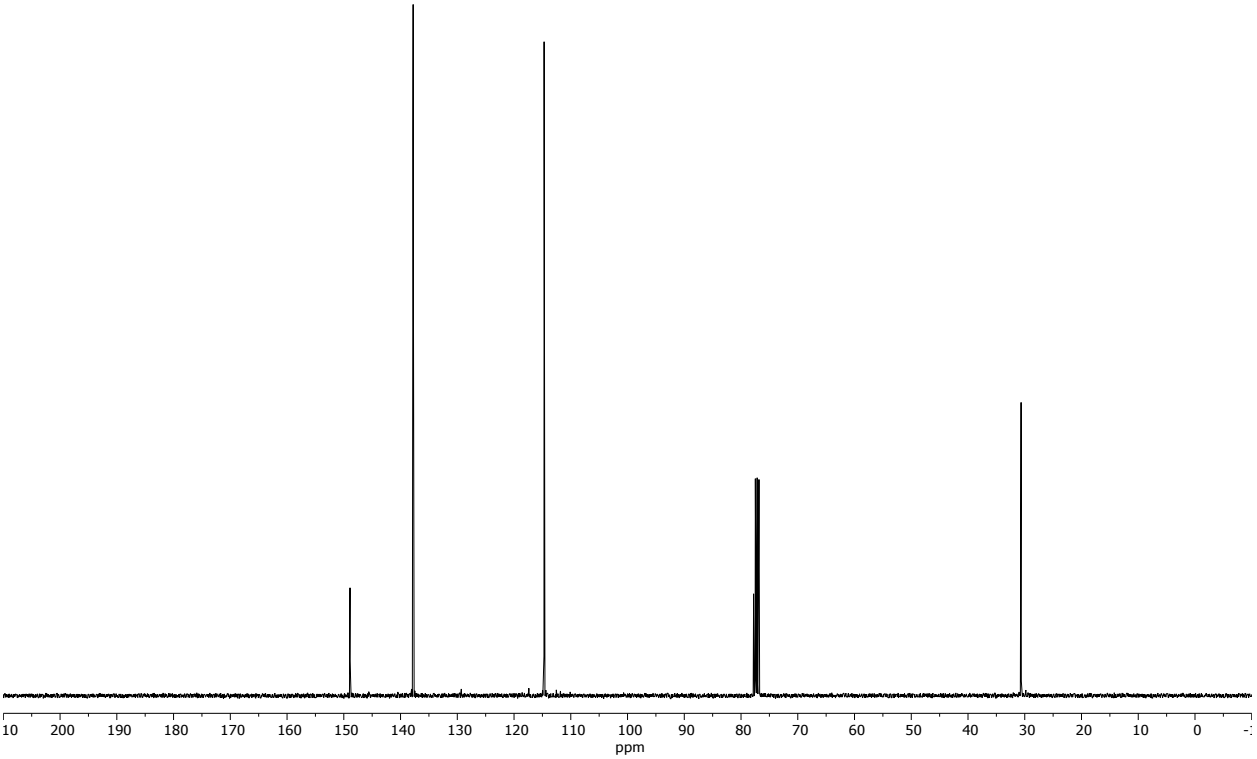


Piehl PP 1068
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 11
200406.411.10.fid
1H CDCl3

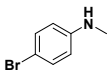


1f

Piehl PP 1068
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 11
200406.411.11.fid
13C CDCl3

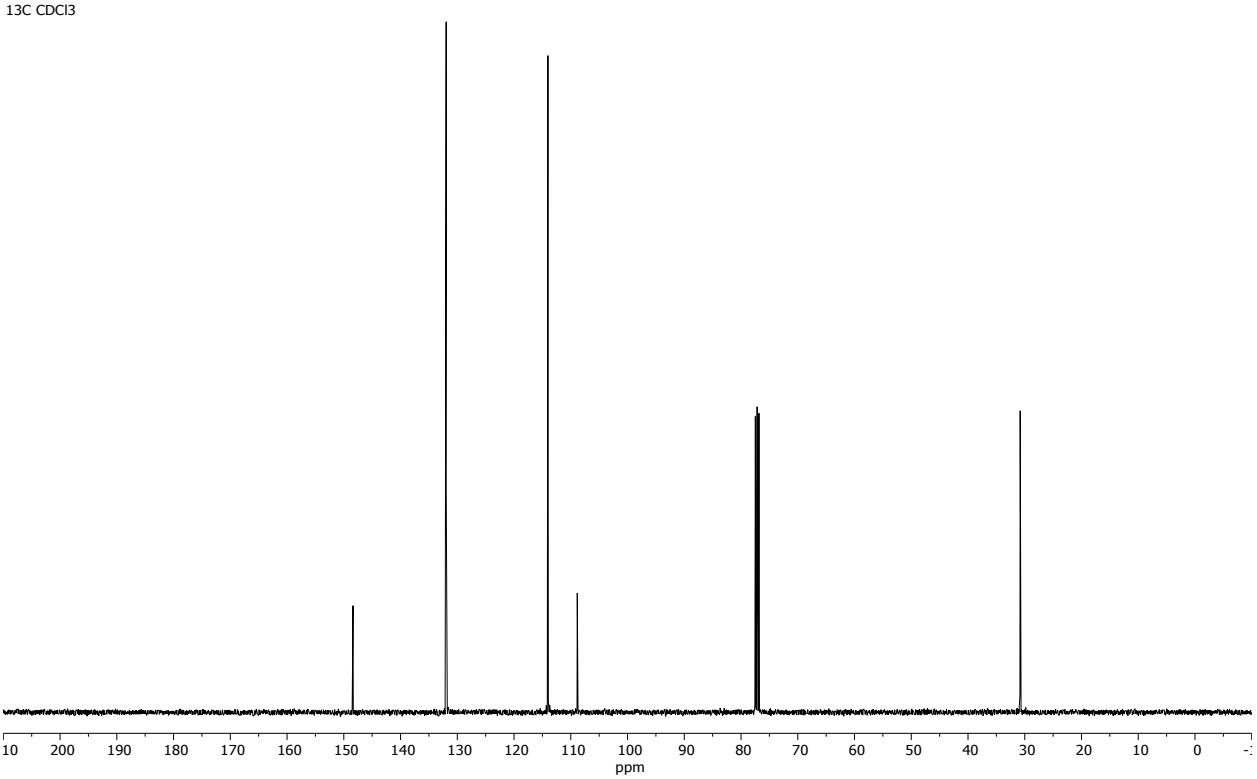


Piehl PP 995 B
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2002 4
200204.404.10.fid
1H CDCl3

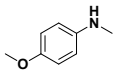


1g

Piehl PP 995 B
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2002 4
200204.404.11.fid
13C CDCl3

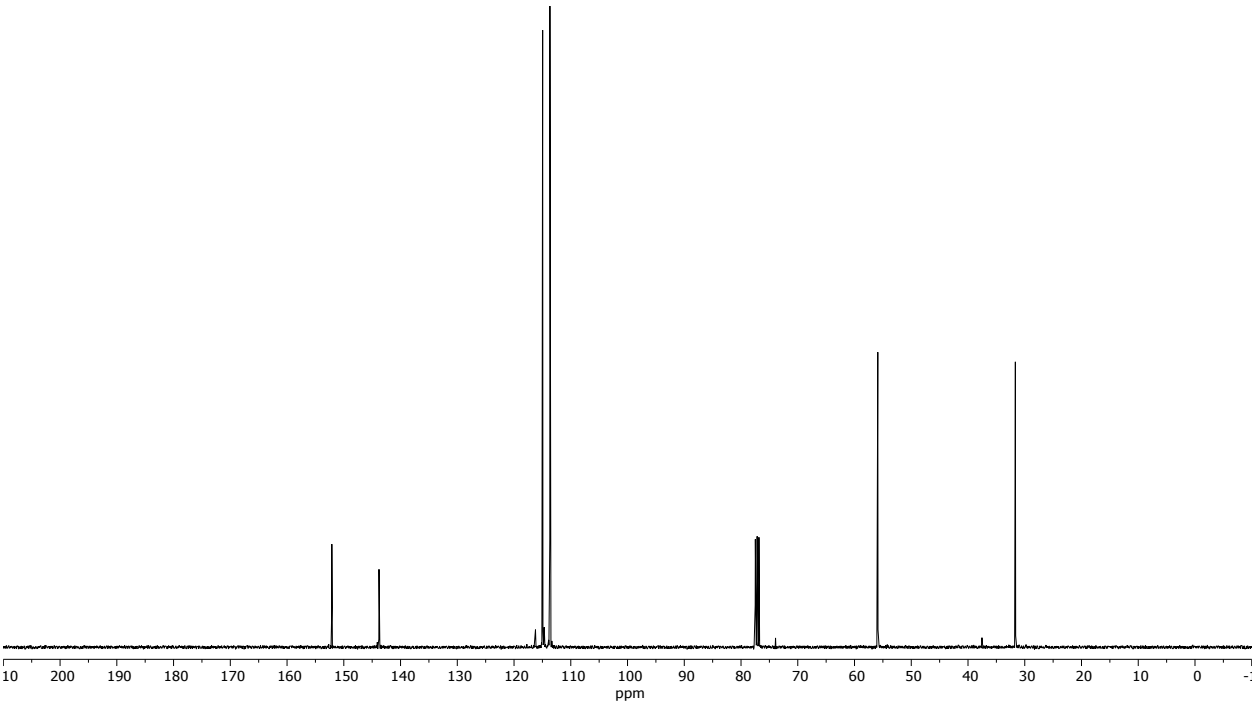


Piehl PP 996
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2002 3
200204.403.10.fid
1H CDCl3



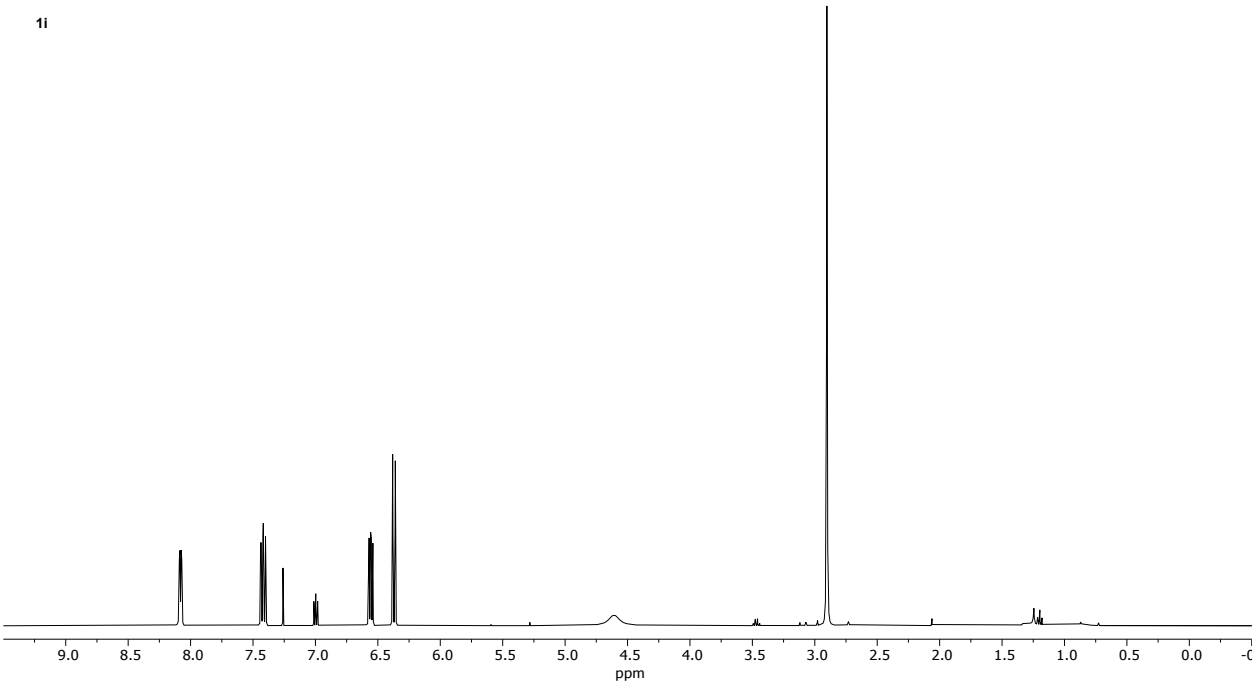
1h

Piehl PP 996
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2002 3
200204.403.11.fid
13C CDCl3

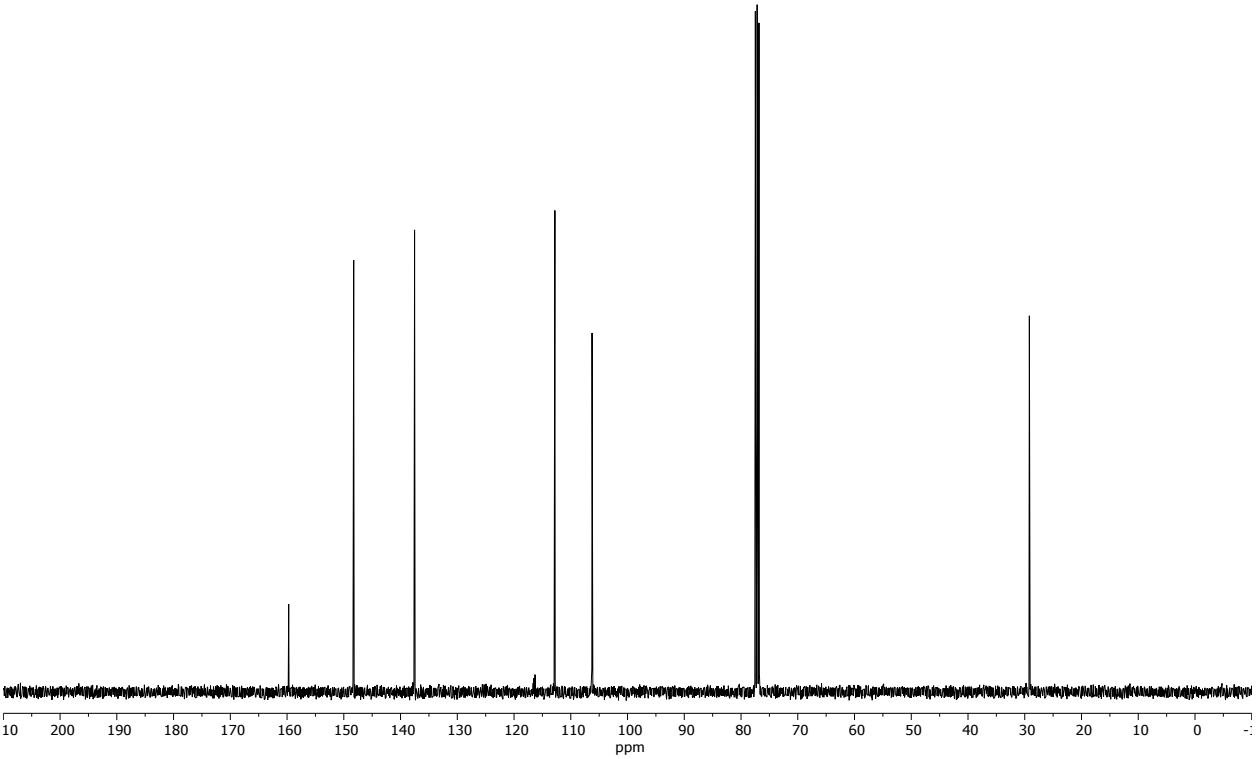


Piehl PP 1051
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 16
200319.416.10.fid
1H CDCl3

1i



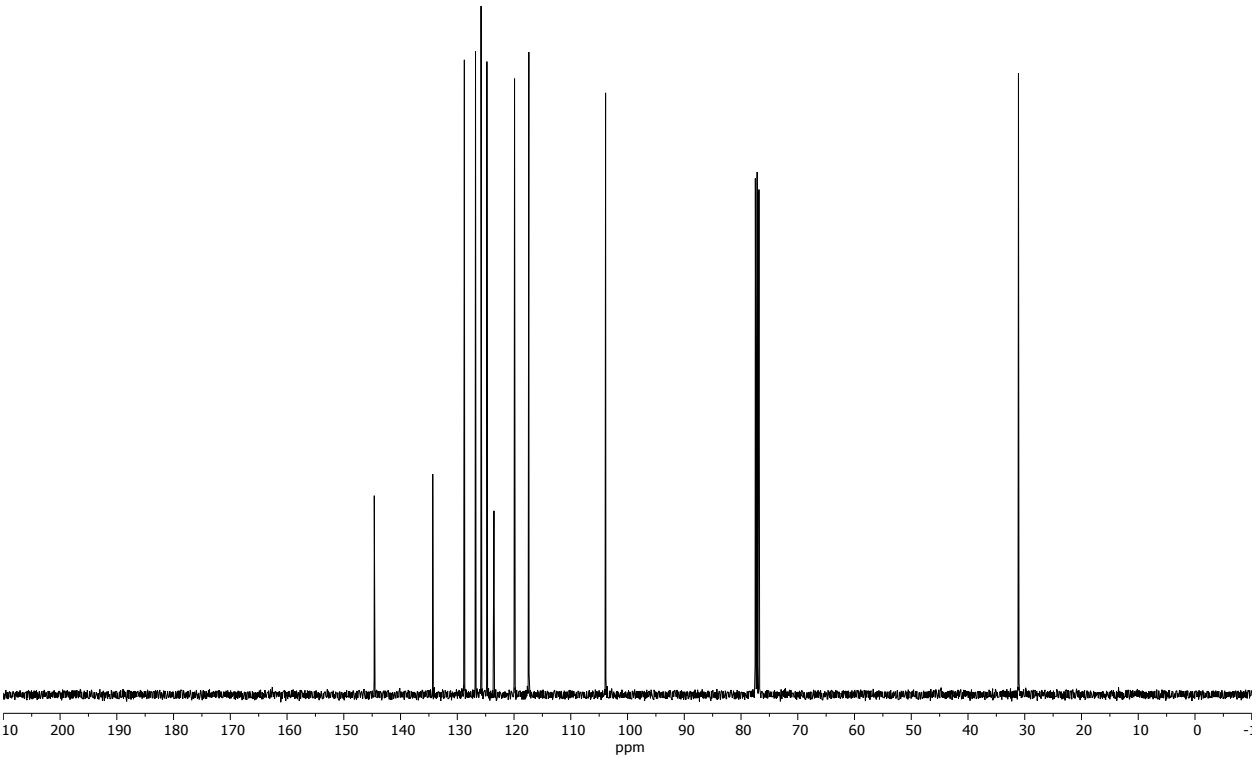
Piehl PP 1051
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 16
200319.416.11.fid
13C CDCl3



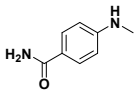
Piehl PP 1035
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 7
200306.407.10.fid
1H CDCl3

1k

Piehl PP 1035
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 7
200306.407.11.fid
13C CDCl3

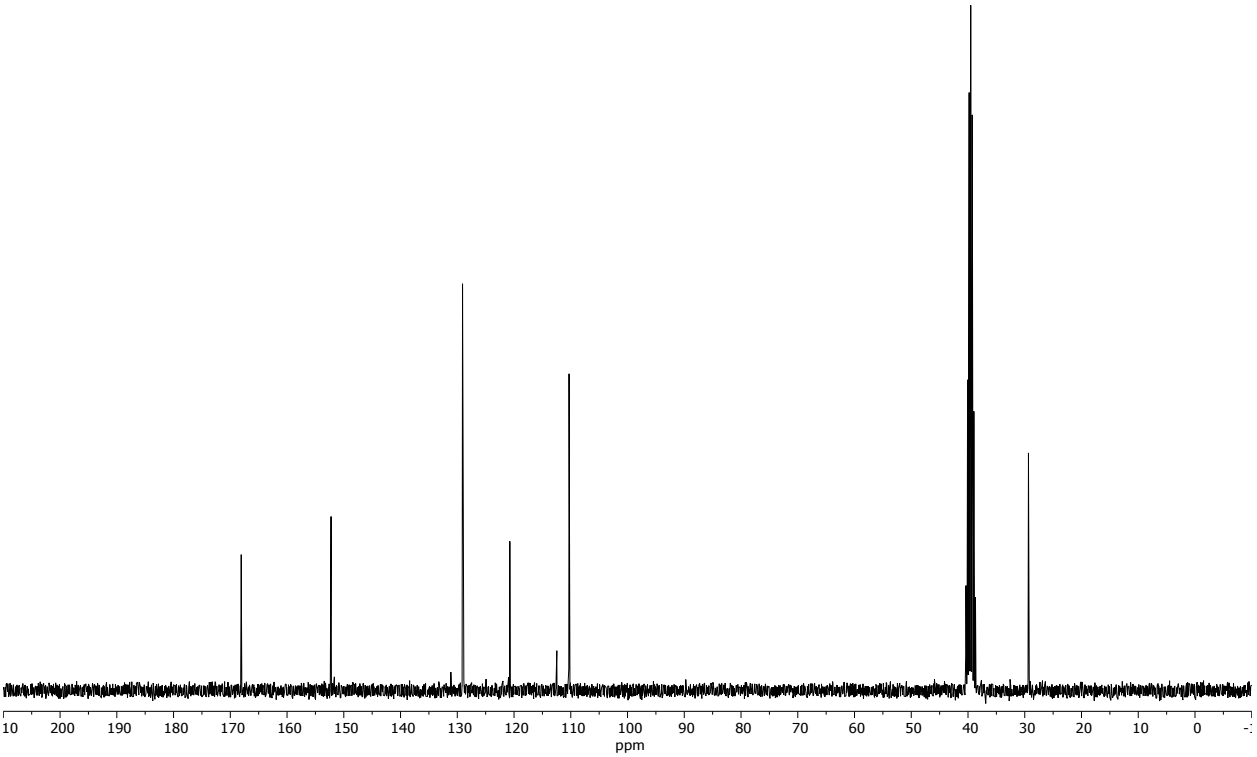


Piehl / PP1057
PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2003 53
200325.f353.10.fid
1H DMSO

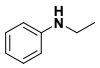


1l

Piehl / PP1057
C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2003 53
200325.f353.11.fid
13C DMSO

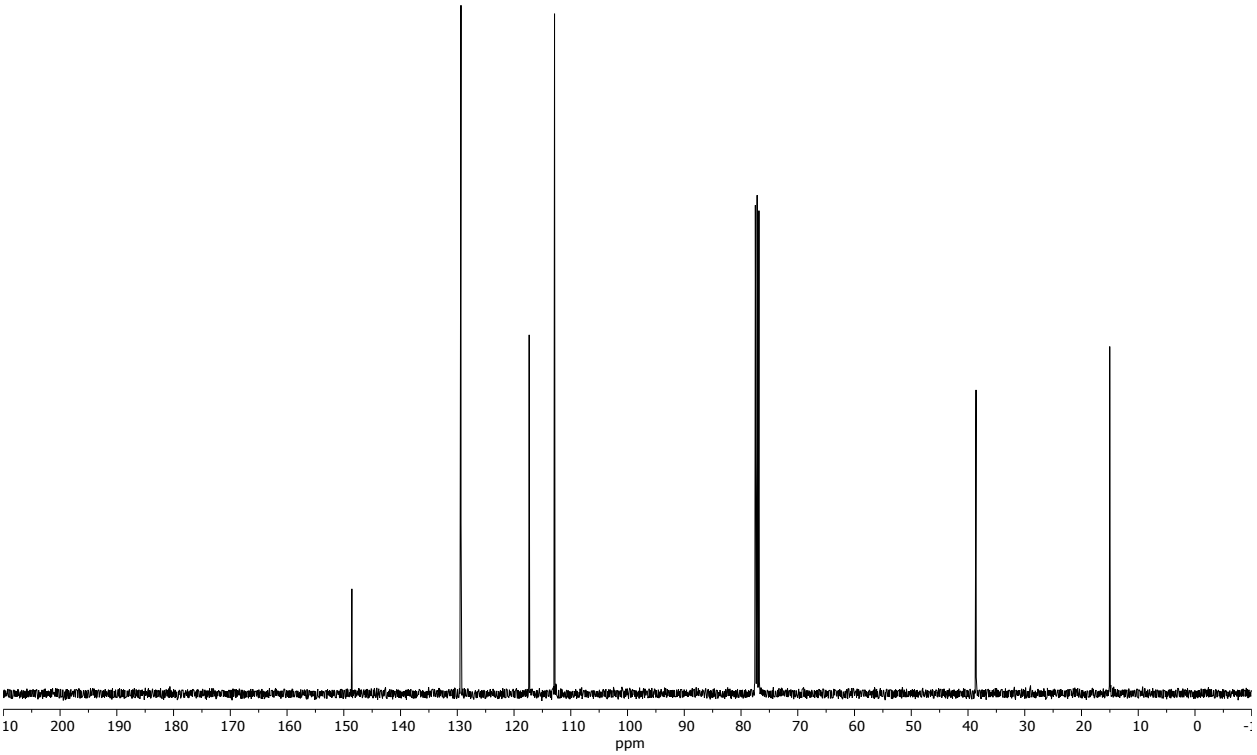


Piehl PP 1037
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 23
200310.423.10.fid
1H CDCl3

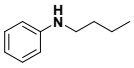


3a

Piehl PP 1037
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 23
200310.423.11.fid
13C CDCl3

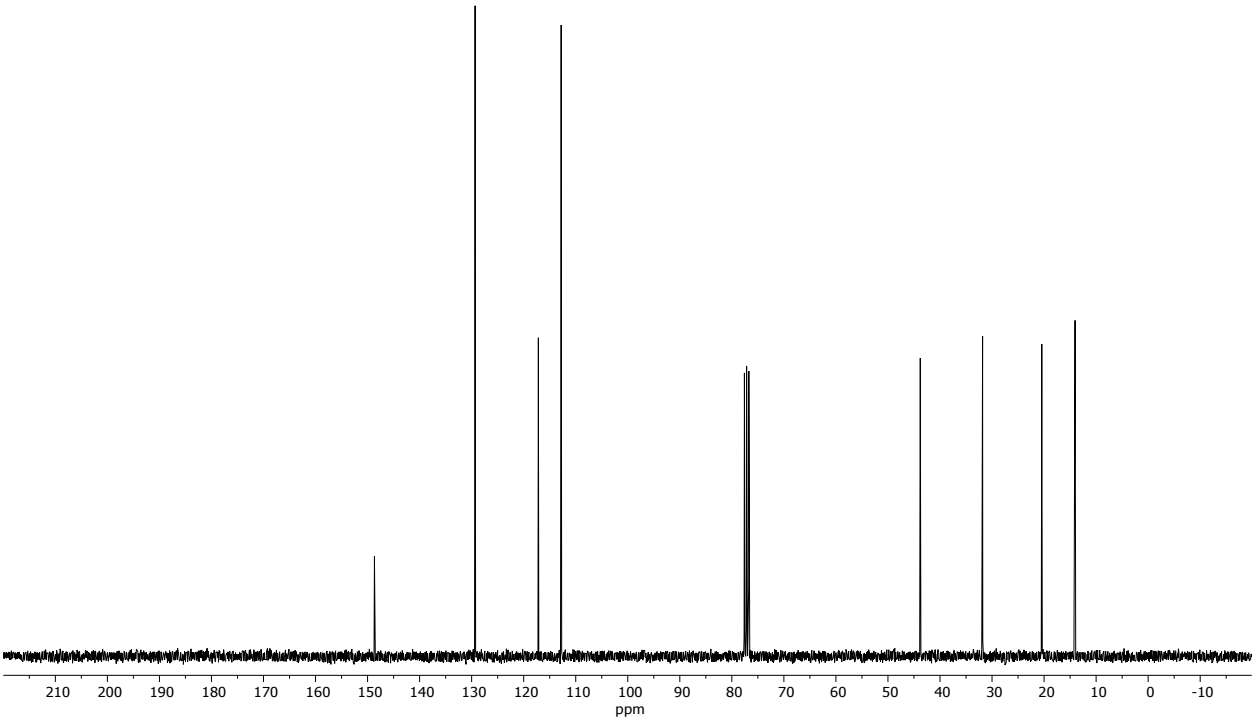


Piehl PP 1013
PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 37
200218.f337.10.fid
1H CDCl3

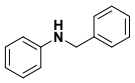


3b

Piehl PP 1013
C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 37
200218.f337.11.fid
13C CDCl3

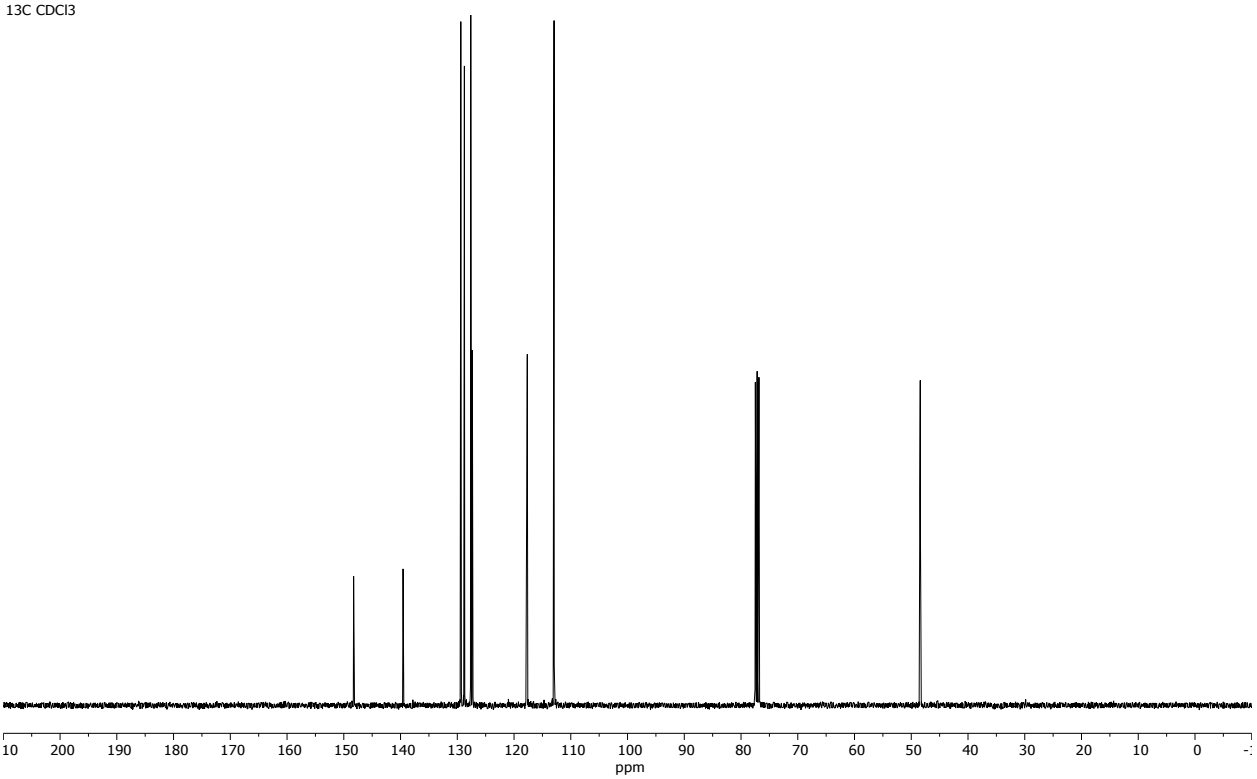


Piehl PP 1066
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 12
200406.412.10.fid
1H CDCl3

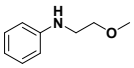


3c

Piehl PP 1066
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 12
200406.412.11.fid
13C CDCl3



Piehl PP 1040
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 24
200310.424.10.fid
1H CDCl3



3d

Piehl PP 1040
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 24
200310.424.11.fid
13C CDCl3