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Supporting Information

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First Organocatalytic Enantioselective Protonation of Silyl Enolates Mediated by Cinchona Alkaloids and a Latent Source of Hydrogen Fluoride.

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1- General Information

CH_2Cl_2, $CHCl_3$, toluene, CH_3CN, DMF, NMP, DMSO, benzoyl chloride, chlorotrimethylsilane, triethylamine and $(i\text{Pr})_2NH$ was distilled from CaH_2. THF, dioxane, and Et_2O was distilled from Na/benzophenone. EtOH was carrefully distilled from Na. Acetic anhydride was distilled from quinoline prior to use. Benzoic anhydride was recrystallized from Et_2O prior to use. *n*-BuLi (Acros, 2.5 M in hexane) was titrated with menthol and phenantroline as indicator prior to use.

2-methyltetralone, α-tetralone, indanone, 2-methylindanone, diethyl carbonate, NaH as a 60% dispersion in mineral oil, MeI, N-methylpyrrolidine, DMAP and benzyl bromide were purchased from Acros and used as received.

$(DHQ)_2PHAL$, $(DHQ)_2Pyr$, $(DHQ)_2AQN$, hydroquinidine 9-phenanthryl ether, hydroquinidine, 4-methyl-2-quinolyl ether, hydroquinidine 4-chlorobenzoate, 2-ethylindanone, 2,2,6-trimethylcyclohexanone, quinuclidine, 3-(chloromethyl)pyridine hydrochloride, NaI and 5-methoxytetralone were purchased from Aldrich and used as received.

Benzoyl fluoride was purchased from Lancaster and used as received.

The NMR spectra were recorded on a Bruker AVANCE 300, ^1H at 300 MHz and ^{13}C at 75 MHz using $CDCl_3$ as solvent and the residual solvent (δ 7.26, ^1H ; δ 77.36, ^{13}C) as internal standard unless otherwise indicated.

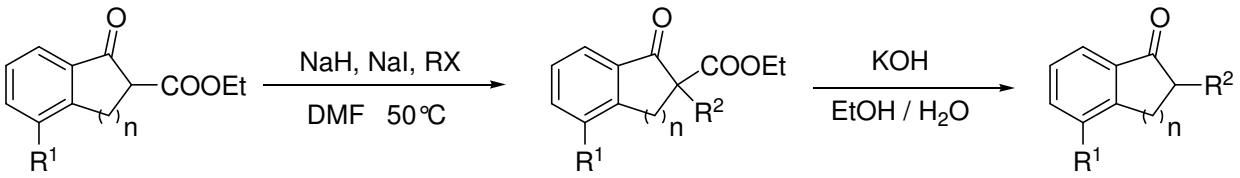
Melting point are uncorrected, analytical thin layer chromatographies were performed on a QF-254 precoted silica on aluminium. Flash chromatographies were performed with silica gel (70 – 230 µm) unless otherwise indicated.

HPLC analysis were performed on a Thermo Fisher P1500 with Daicel Chiralpack® columns (4.6 mm × 25 cm) in heptane / *iso*propanol solvent mixtures with visualization at 254 nm (UV 1000 detector) unless otherwise stated.

Chiral gas chromatographies were performed on a Varian 3900 using a Chirasil-Dex-CB® columm.
Gas chromatography were performed on a Varian 3900 using a DB-5 columm (30 m × 0.25 mm × 25 µm).
GC / MS analysis were performed on a Thermoquest Finnigan using a VF 5-MS® column (30 m × 0.25 mm × 25 µm).

All experiments were conducted under nitrogen atmosphere in oven-dried glassware with magnetic stirring.

2- Ketones 3b, 3c, 3d, 3e, 3f, 3i



2.1 Representative Procedure

Representative Procedure for the Alkylation of Keto-esters

To a solution of 2-(ethoxycarbonyl)-1-tetralone[1] (1.010 g, 4.6 mmol) in DMF (12 mL) was added NaH (60% in oil, 0.400 g, 10 mmol), the solution was stirred for 30 min (green solution) and ethyl iodide (1.12 mL, 13.9 mmol) was added. The reaction was stirred for 3h at 50°C. The reaction was cooled, quenched with a saturated NH_4Cl aqueous solution (15 mL) and the product was extracted with EtOAc (4 × 20 mL). The combined organic layers were washed with brine (5 × 50 mL), dried ($MgSO_4$) and concentrated under vacuum. The residue was used in the next step without further purification.

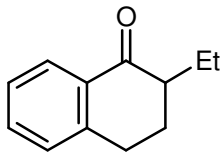
Representative Procedure Procedure for the Decarboxylation

The crude 2-ethyl-2-(ethoxycarbonyl)-1-tetralone was dissolved in EtOH / H_2O (5/1, 22 mL). After adding KOH (0.650 g, 11.5 mmol), the reaction mixture was refluxed for 2 hours. The residue was neutralized with a saturated NH_4Cl aqueous solution (10 mL), the volatiles were removed under vacuum, and the resulting aqueous layer was extracted with EtOAc (3 × 20 mL). The combined organic layers were washed with brine (50 mL), dried over $MgSO_4$ and concentrated. The residue was purified by flash chromatography (SiO_2, 5% Et_2O in cyclohexane, Rf = 0.28) to afford 2-ethyl-tetralone in 25% overall yield.

[1] Chini. M., Croti. P., Macchia. F.; *J. Org. Chem.,* **1989**, 3930.

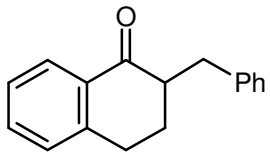
2.2 Spectral Data for Ketones 3b, 3c, 3d, 3e, 3f, 3i

2-Ethyl-tetralone 3b :



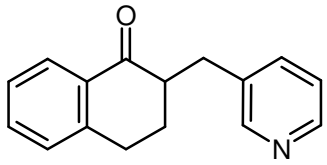
Aspect : Pale yellow oil.
Yield : 25%
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.28
^{1}H NMR (300 MHz, $CDCl_3$) : 1.00 (t, 3H, *J* = 7.5 Hz), 1.50-1.65 (m, 1H), 1.84-2.05 (m, 2H), 2.19-2.29 (m, 1H), 2.37-2.46 (m, 1H), 2.98-3.02 (m, 2H), 7.22-7.32 (m, 2H), 7.46 (t, 1H, *J* = 7.3 Hz), 8.03 (d, 1H, *J* = 7.9 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 11.76, 22.73, 28.02, 28.69, 49.20, 126.84, 127.72, 128.97, 132.88, 133.37, 144.32, 200.64.

2-Benzyl-tetralone 3c :



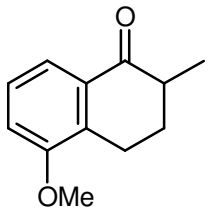
Aspect : White solid.
Yield : 36%
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.23
^{1}H NMR (300 MHz, $CDCl_3$) : 1.82 (m, 1H), 2.11 (m, 1H), 2.69 (m, 2H), 2.95 (m, 2H), 3.50 (dd, 1H, *J* = 13.3 Hz, *J* = 3.4 Hz), 7.30 (m, 8H), 8.10 (d, 1H, *J* = 7.7 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 27.98, 28.96, 35.99, 49.79, 126.47, 126.96, 127.88, 128.74, 129.05, 129.61, 132.79, 133.61, 140.38, 144.37, 199.76.
Mp : 51-52°C

2-(3-Picolyl)-tetralone 3d :



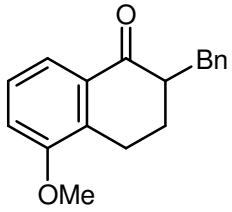
Aspect : Red oil.
Yield : 35%
Purification : SiO_2, 50% EtOAc in cyclohexane, Rf = 0.23
^{1}H NMR (300 MHz, $CDCl_3$) : 1.80 (m, 1H), 2.09 (m, 1H), 2.72 (m, 2H), 2.97 (q, 2H, *J* = 4.3 Hz), 3.46 (m, 1H), 7.24 (m, 3H), 7.32 (m, 1H), 7.48 (m, 1H), 7.57 (d, 1H, *J* = 7.9 Hz), 8.05 (d, 1H, *J* = 7.9 Hz), 8.50 (m, 1H).
^{13}C NMR (75 MHz, $CDCl_3$) : 28.18, 29.10, 33.22, 49.44, 123.72, 127.08, 127.90, 129.10, 132.64, 133.82, 135.74, 137.16, 144.20, 148.07, 150.98, 199.10.
IR (KBr, cm^{-1}) : 743, 1598, 1681, 2927.
HRMS (Cl^+) : *m/z* calcd for $C_{16}H_{15}NO$ (MH^+) : 238.1232 Found : 238.1223 (- 3.9 ppm).

2-Methyl-5-methoxy-tetralone 3e :



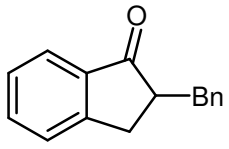
Aspect : Yellow oil.
Yield : 83%
Purification : SiO_2, 20% Et_2O in cyclohexane, Rf = 0.47
^{1}H NMR (300 MHz, $CDCl_3$) : 1.25 (d, 3H, *J* = 6.8 Hz), 1.75-1.89 (m, 1H), 2.16-2.25 (m,1H), 2.52-2.64 (m, 1H), 2.69-2.80 (m,1H), 3.04-3.13 (m, 1H), 3.86 (s, 3H), 7.00 (d, 1H, *J* = 8.1 Hz), 7.26 (t, 1H, *J* = 8.2 Hz), 7.65 (d, 1H, *J* = 7.8 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 15.61, 22.58, 30.92, 42.42, 55.97, 114.21, 119.28, 127.08, 133.48, 133.77, 157.06, 201.44.

2-Benzyl-5-methoxy-tetralone 3f :



Aspect : White Solid.
Yield : 63%
Purification : SiO_2, 10% Et_2O in cyclohexane, Rf = 0.26
^{1}H NMR (300 MHz, $CDCl_3$) : 1.67-1.80 (m, 1H), 2.07-2.16 (m, 1h), 2.58-2.79 (m, 3H), 3.01-3.10 (m, 1H), 3.45 (dd, 1H, *J* = 13.57 Hz, *J* = 3.9 Hz), 3.85 (s, 3H), 7.01 (d, 1H, *J* = 7.9 Hz), 7.19-7.33 (m, 6H), 7.68 (d, 1H, *J* = 7.7 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 22.27, 27.15, 35.86, 49.22, 55.97, 114.38, 119.40, 126.44, 127.18, 128.72, 129.61, 133.30, 133.80, 140.41, 157.06, 200.10.
IR (KBr, cm^{-1}) : 739, 1261, 1681.
HRMS (CI$^+$): *m/z* calcd for $C_{18}H_{18}O_2$ (MH^+) : 267.1385. Found : 267.1379 (-2.4 ppm).
Mp : 68-69°C

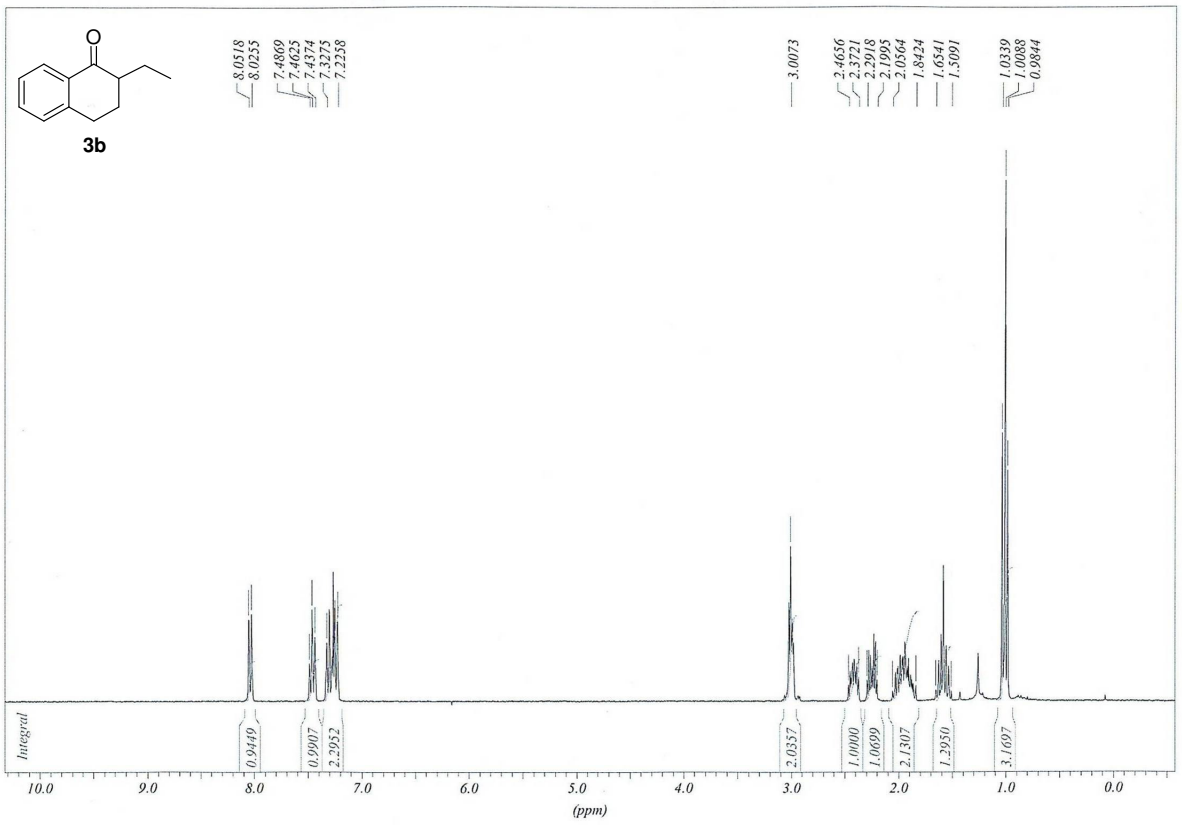
2-Benzyl-indanone 3i :

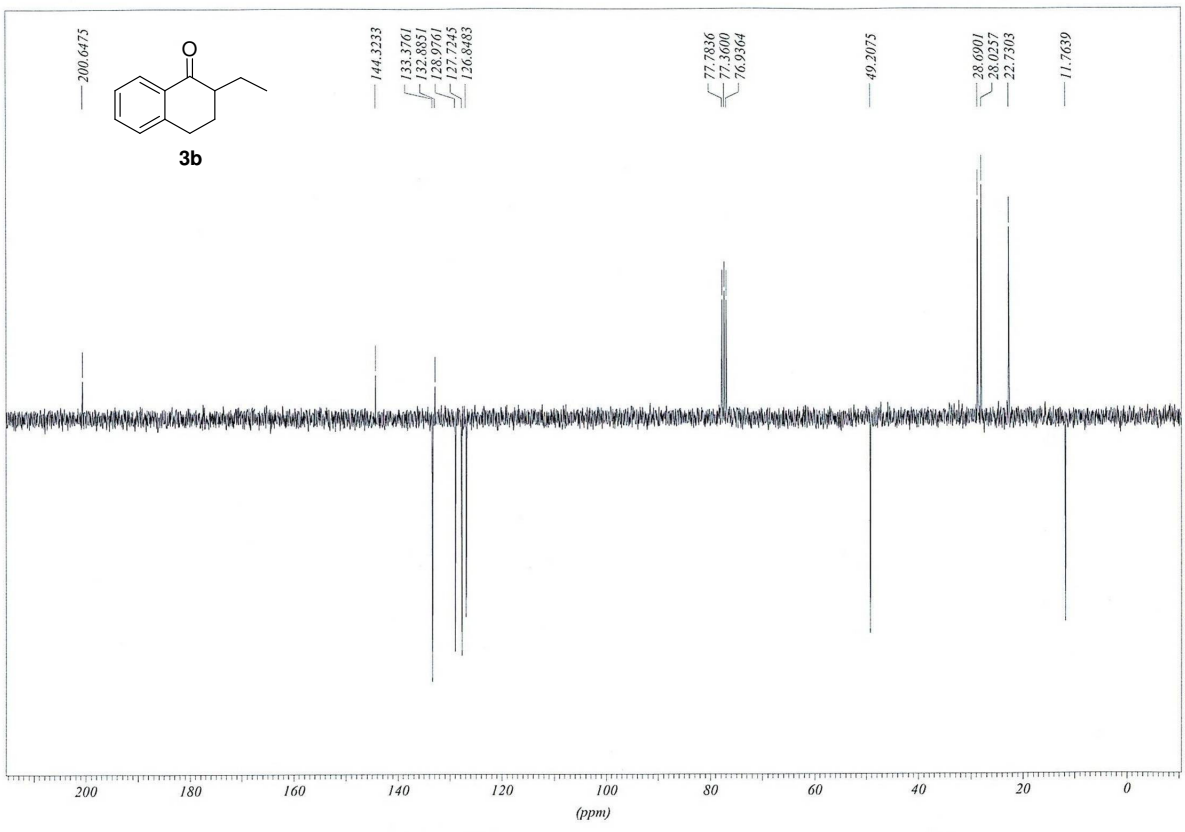


Aspect : Pale yellow oil.
Yield : 57%
Purification : SiO_2, 5% EtOAc in cyclohexane, Rf = 0.21
^{1}H NMR (300 MHz, $CDCl_3$) : 2.66 (dd, 1H, *J* = 10.3 Hz, *J* = 13.9 Hz), 2.87 (dd, 1H, *J* = 3.9 Hz, *J* = 17.1 Hz), 3.00 (m, 1H), 3.17 (dd, 1H, *J* = 7.5 Hz, *J* = 16.9 Hz), 3.41 (dd, 1H, *J* = 4.1 Hz, *J* = 13.9 Hz), 7.18-7.40 (m, 7H), 7.54-7.59 (t, 1H, *J* = 7.1 Hz), 7.77 (d, 1H, *J* = 7.5 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 32.50, 37.31, 49.25, 124.34, 126.67, 126.91, 127.75, 128.85, 129.22, 135.14, 136.86, 139.97, 153.97, 208.17.

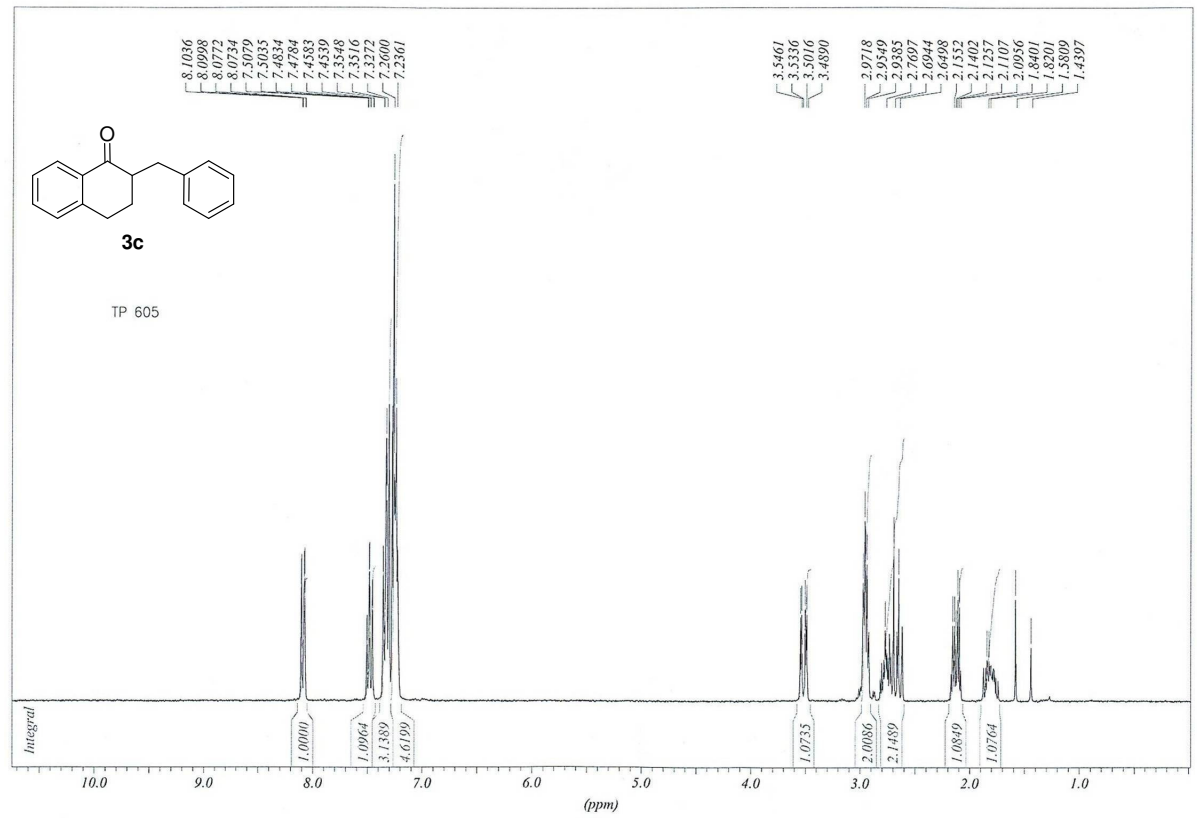
2.3 Copies of the ^{1}H and ^{13}C NMR Spectra of Ketones 3b, 3c, 3d, 3e, 3f, 3i

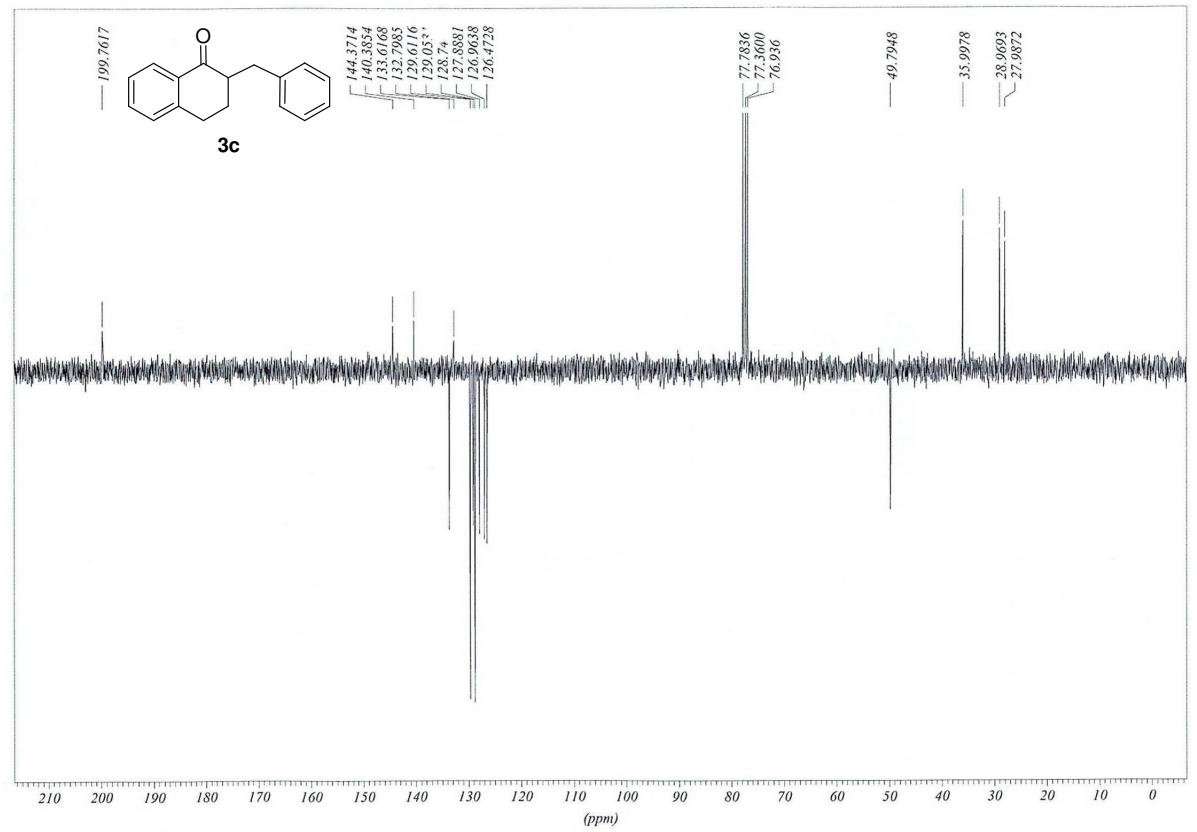
2-ethyl-tetralone 3b :



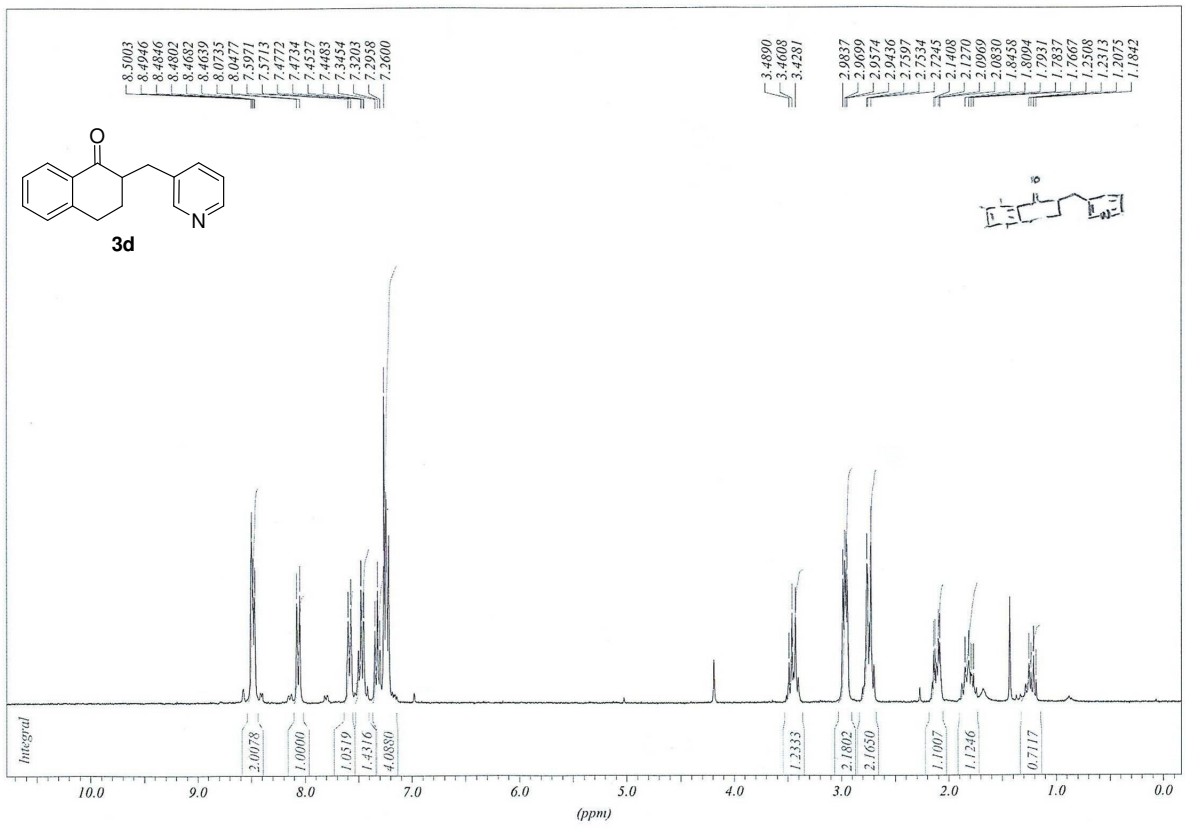


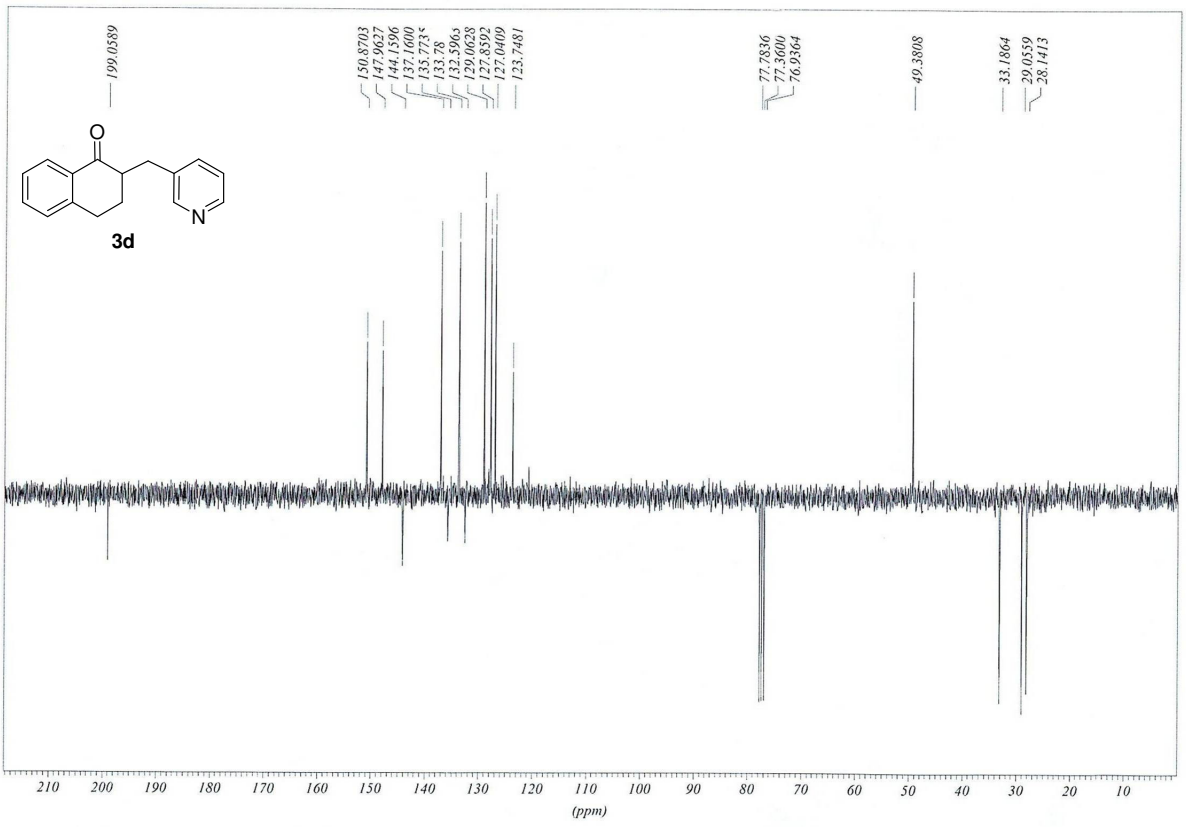
2-benzyl-tetralone 3c :



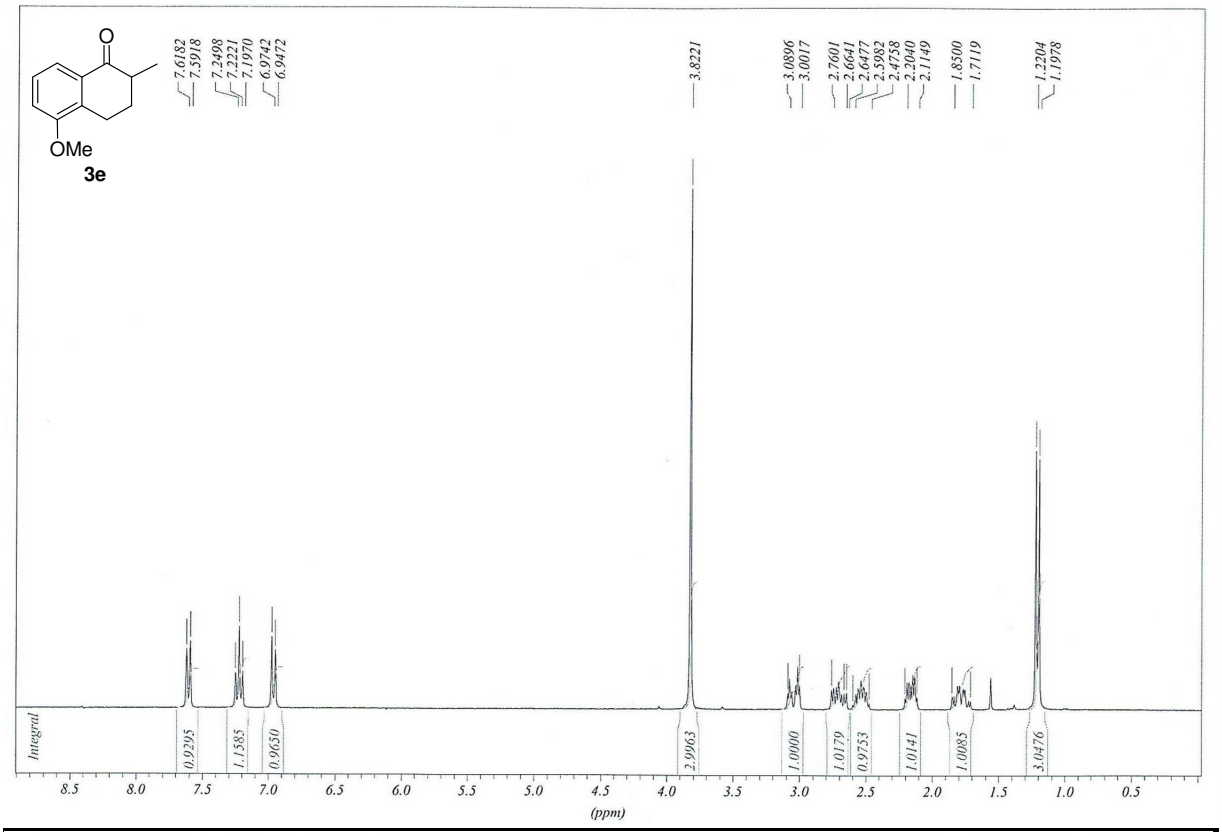


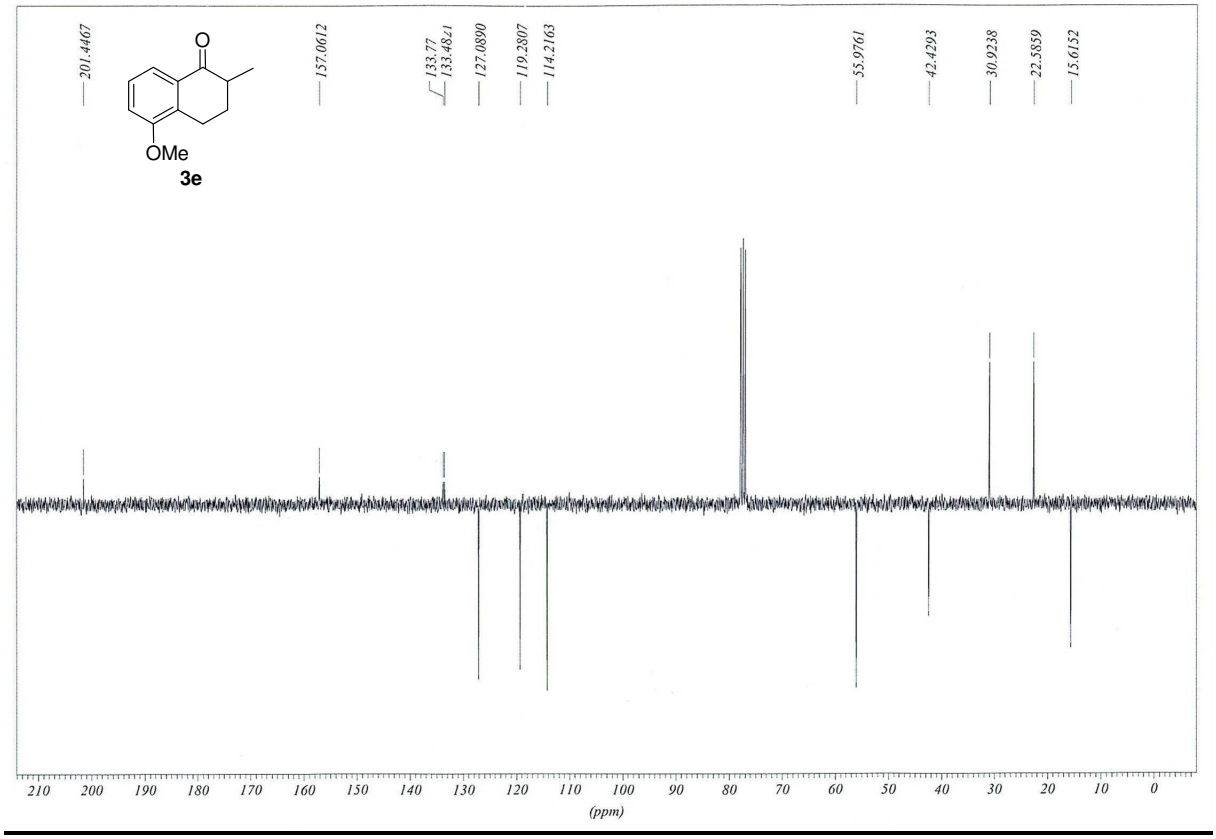
2-(3-picolyl)-tetralone 3d :



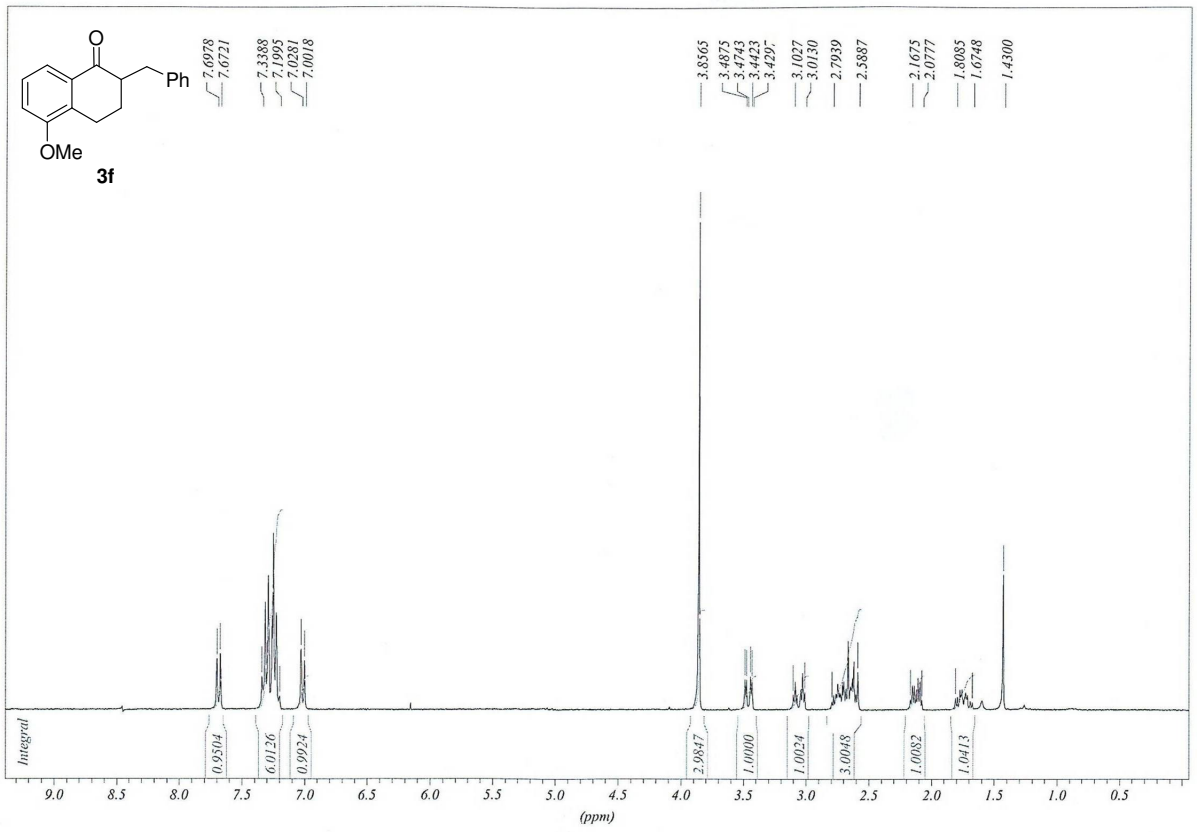


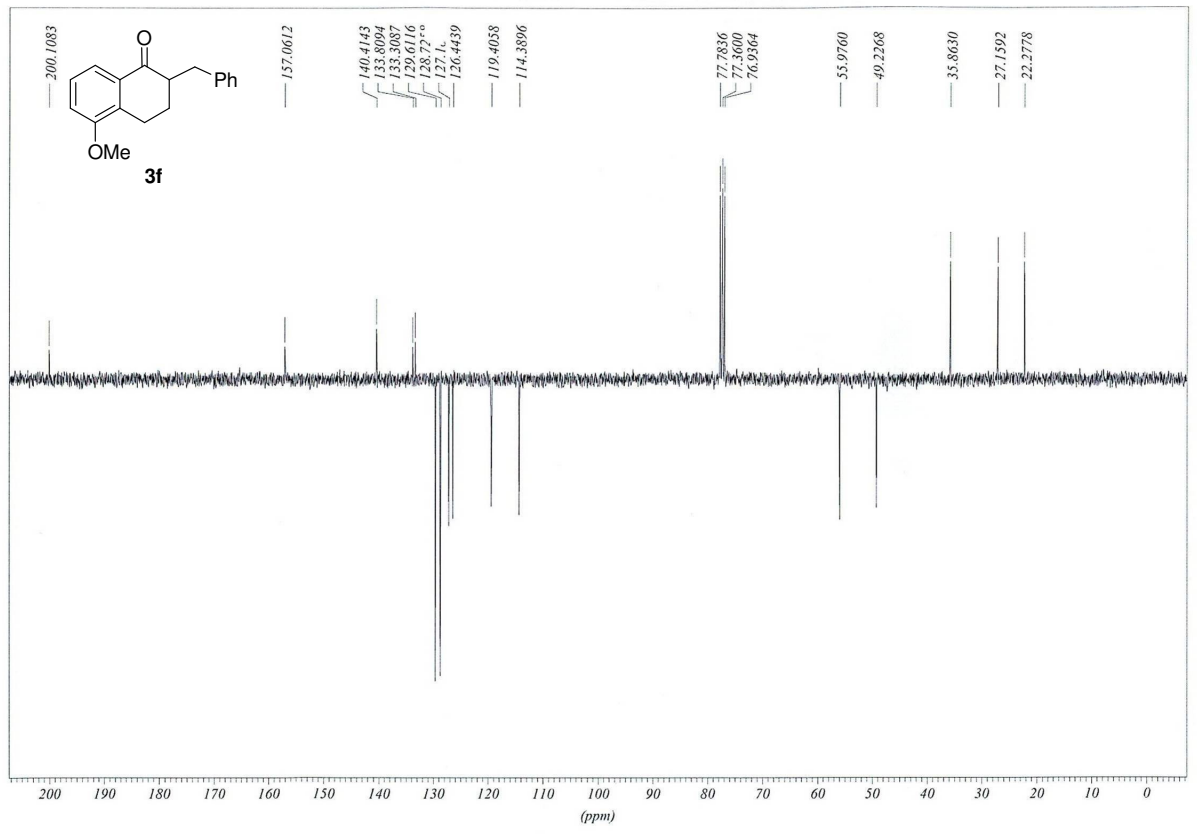
2-methyl-5-methoxy-tetralone 3e :



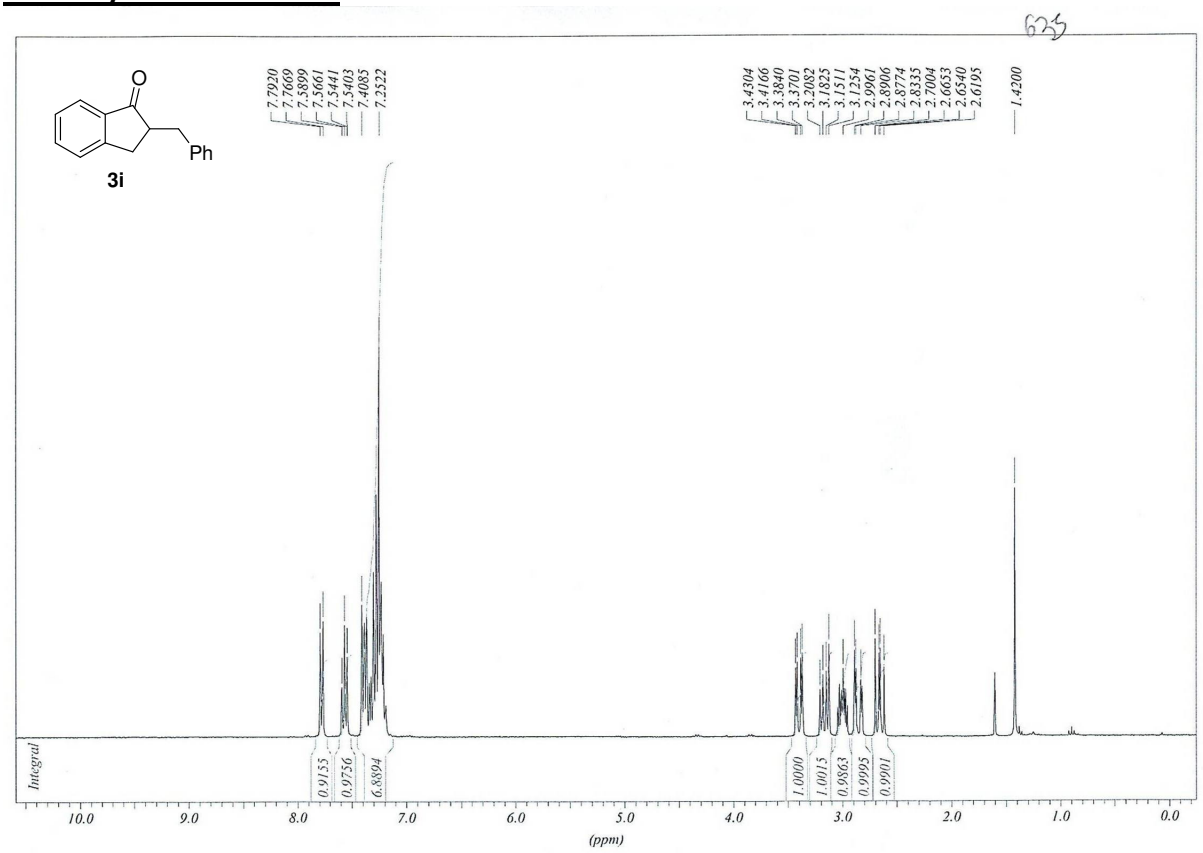


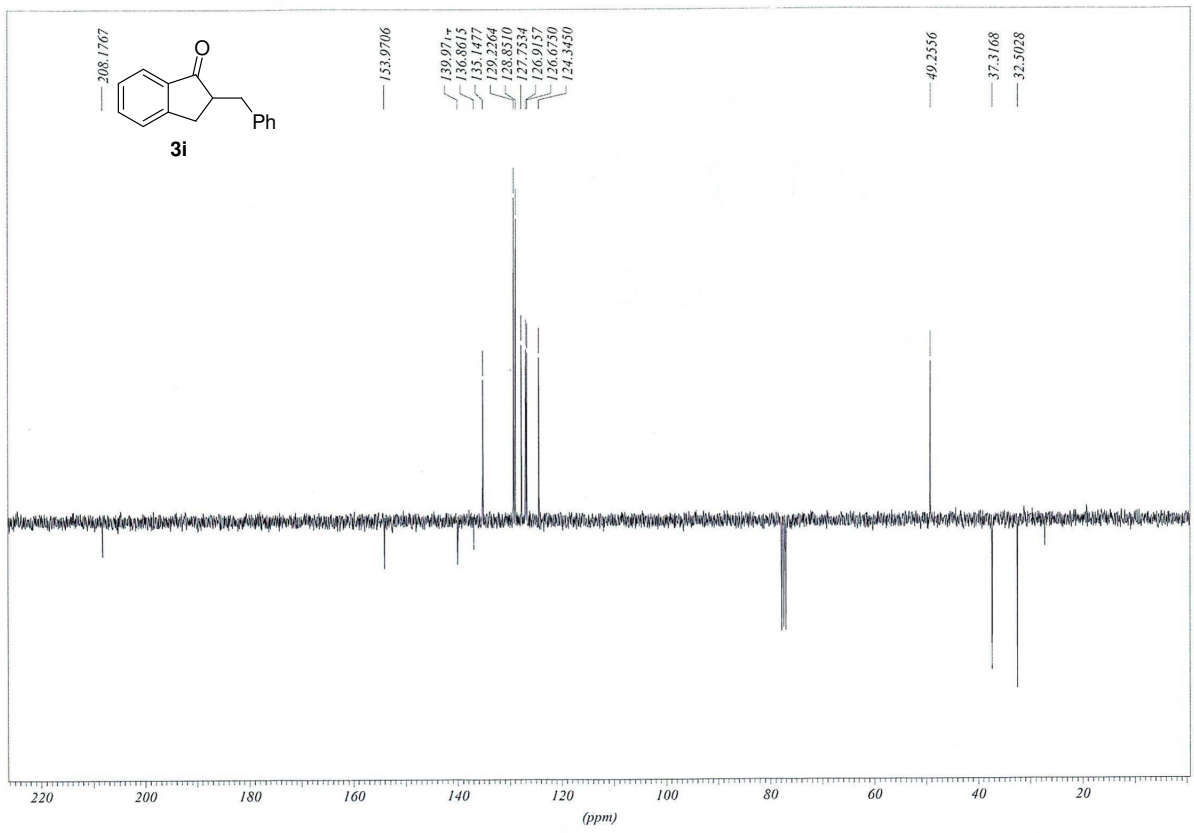
2-benzyl-5-methoxy-tetralone 3f :





2-benzyl-indanone 3i :





3- Silyl Enol Ethers 2a – j

3.1 General Procedures

Procedure I :

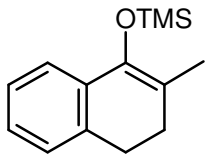
To a solution of $(i\text{Pr})_2\text{NH}$ (1.19 mL, 8.4 mmol) at -78°C in dry THF (50 mL) was added *n*-BuLi (2.5 M solution in hexane, 3.22 mL, 8.05 mmol), the whole was stirred for 1h at this temperature, after which the ketone (7 mmol) was slowly added. After 1h at -78°C freshly distilled TMSCl (0.98 mL, 7.7 mmol) was added and the solution was allowed to reach room temperature before being stirred for 2h. A solution of $NaHCO_3$ (20 mL) was added and the mixture was extracted with Et_2O (3 × 50mL). The combined organic layers were washed with brine and dried ($MgSO_4$). The solvent was removed under vacumm and the residue was purified by flash chromatography affording the pure silyl enol ethers **2**.

Procedure II :

To a solution of $(i\text{Pr})_2\text{NH}$ (1.7 mL, 12 mmol) at -78°C in dry THF (70 mL) was added *n*-BuLi (2.5 M solution in hexane, 4.6 mL, 11.5 mmol), the whole was stirred 1h at this temperature, then the ketone (10 mmol) was slowly added. After 1h at -78°C freshly distilled TMSCl (1.4 mL, 11 mmol) was added and the solution was warmed to room temperature and stirred for 2h. After evaporation of the volatiles, the crude product was purified by distillation to give the pure silyl enol ethers **3**.

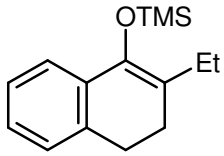
3.2 Spectral Data for Silyl Enol Ethers 2a –j

2-methyl-1-trimethylsilyloxy-tetral-1-ene 2a :



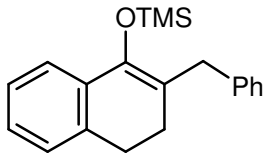
Procedure I
Aspect : Colorless oil.
Yield : 93%.
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.77.
^{1}H NMR (300 MHz, $CDCl_3$) : 0.19 (s, 9H), 1.81 (s, 3H), 2.25 (t, 2H, *J* = 7.7 Hz), 2.73 (t, 2H, *J* = 7.9 Hz), 7.09 (m, 2H), 7.11-7.19 (m, 1H), 7.32 (d, 1H, *J* = 7.5 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 0.88,17.63, 28.53, 29.37, 117.26, 121.76, 126.34, 126.44, 126.99, 134.63, 136.22, 142.66.

2-ethyl-1-trimethylsilyloxy-tetral-1-ene 2b:



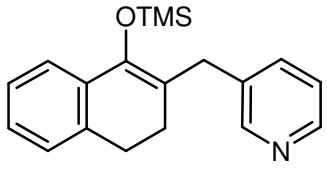
Procedure I
Aspect : Colorless oil.
Yield : 78%
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.87
^{1}H NMR (300 MHz, $CDCl_3$) : 0.19 (s, 9H), 1.03, (t, 3H, *J* = 7.5 Hz), 2.22-2.32 (m, 4H), 2.72 (t, 2H, *J* = 7.5 Hz), 7.08-7.20 (m, 3H), 7.32 (d, 1H, *J* = 7.3 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 0.81, 12.48, 23.78, 26.36, 28.72, 122.07, 122.96, 126.35, 126.48, 126.93, 134.83, 136.38, 141.87.

2-benzyl-1-trimethylsilyloxy-tetral-1-ene 2c :



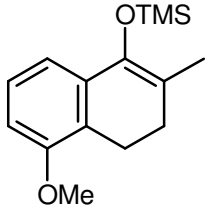
Procedure I
Aspect : Colorless oil.
Yield : 85%.
Purification : SiO_2, 4% Et_2O in cyclohexane, Rf = 0.75.
^{1}H NMR (300 MHz, $CDCl_3$) : 0.21 (s, 9H), 2.11 (t, 2H, *J* = 7.7 Hz), 2.71 (t, 2H, *J* = 7.7 Hz), 3.64 (s, 2H), 7.09 (m, 1H), 7.24 (m, 7H), 7.38 (d, 1H, *J* = 7.5 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 0.99, 26.97, 28.63, 37.08, 119.52, 122.42, 126.22, 126.40, 126.90, 127.09, 128.62, 129.16, 134.56, 136.62, 140.45, 143.78

2-(3-picolyl)-1-trimethylsilyloxy-tetral-1-ene 2d :



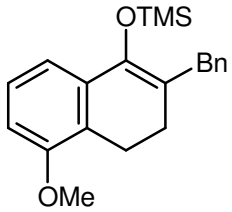
Procedure I
Aspect : Orange oil.
Yield : 68%.
Purification : SiO_2, 50% EtOAc in cyclohexane, Rf = 0.60.
^{1}H NMR (300 MHz, $CDCl_3$) : 0.21 (s, 9H), 2.09 (t, 2H, *J* = 7.9 Hz), 2.72 (t, 2H, *J* = 7.9 Hz), 3.62 (s, 2H), 7.20 (m, 4H), 7.38 (d, 1H, *J* = 7.5 Hz), 7.51 (d, 1H, *J* = 7.9 Hz), 8.42 (d, 1H, *J* = 4.7 Hz), 8.49 (s, 1H).
^{13}C NMR (75 MHz, $CDCl_3$) : -0.08, 25.89, 27.43, 33.25, 116.93, 121.40, 122.56, 125.36, 126.08, 126.10, 133.08, 134.76, 135.36, 135.38, 143.31, 146.77, 149.39.

2-methyl-5-methoxy-1-trimethylsilyloxy-tetral-1-ene 2e :



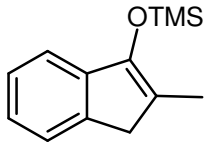
Procedure I
Aspect : Pale yellow oil.
Yield : 77%
Purification : SiO_2, 10% Et_2O in cyclohexane, Rf = 0.72
^{1}H NMR (300 MHz, $CDCl_3$) : 0.19 (s, 9H), 1.81 (s, 3H), 2.22 (t, 2H, *J* = 8.1 Hz), 2.73 (t, 2H, *J* = 8.0 Hz), 3.82 (s, 3H), 6.74 (d, 1H, *J* = 8.1 Hz), 6.99 (d, 1H, *J* = 7.7 Hz), 7.13 (t, 1H, *J* = 7.9 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 0.85, 17.63, 20.44, 28.88, 55.79, 109.22, 114.99, 117.28, 123.84, 126.45, 135.86, 142.49, 155.80.

2-benzyl-5-methoxy-1-trimethylsilyloxy-tetral-1-ene 2f :



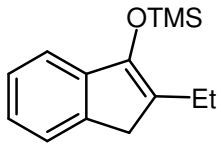
Procedure I
Aspect : Colorless oil.
Yield : 61%
Purification : SiO_2, 10% Et_2O in cyclohexane, Rf = 0.70
^{1}H NMR (300 MHz, $CDCl_3$) : 0.20 (s, 9H), 2.08 (t, 2H, *J* = 7.7 Hz), 2.71 (t, 2H, *J* = 7.7 Hz), 3.63 (s, 2H), 3.81 (s, 3H), 6.76 (d, 1H, *J* = 7.9 Hz), 7.07 (d, 1H, *J* = 7.5 Hz), 7.14-7.28 (m, 6H).
^{13}C NMR (75 MHz, $CDCl_3$) : 0.97, 20.53, 27.25, 37.09, 55.84, 109.67, 115.55, 119.47, 124.31, 126.19, 126.52, 128.60, 129.14, 135.80, 140.45, 143.65, 155.88.

2-methyl-1-trimethylsilyloxy-indan-1-ene 2g :



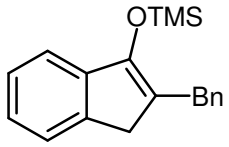
Procedure I
Aspect : Colorless oil.
Yield : 91%
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.77
^{1}H NMR (300 MHz, $CDCl_3$) : 0.27 (s, 9H), 1.97 (s, 3H), 7.09 (t, 2H, *J* = 8.4 Hz), 7.14 (m, 2H), 7.30 (d, 2H, *J* = 7.1 Hz)
^{13}C NMR (75 MHz, $CDCl_3$) : 1.06, 12.73, 38.73, 117.54, 120.45, 123.66, 124.30, 126.29, 141.19, 143.09, 147.84.

2-ethyl-1-trimethylsilyloxy-indan-1-ene 2h :



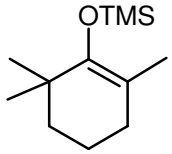
Procedure I
Aspect : Colorless oil.
Yield : 86%.
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.83.
^{1}H NMR (300 MHz, $CDCl_3$) : 0.26 (s, 9H), 1.13 (t, 3H, *J* = 7.5 Hz), 2.41 (q, 2H, *J* = 7.5 Hz), 3.21 (s, 2H), 7.12 (t, 1H, *J* = 7.1 Hz), 7.21-7.26 (m, 2H), 7.31 (d, 1H, *J* = 7.3 Hz).
^{13}C NMR (75 MHz, $CDCl_3$) : 1.02, 14.11, 20.08, 35.83, 117.72, 123.80, 124.33, 126.32, 126.64, 141.26, 143.08, 146.81.

2-benzyl-1-trimethylsilyloxy-indan-1-ene 2i :



Procedure I
Aspect : Pale yellow oil.
Yield : 74%.
Purification : SiO_2, 5% Et_2O in cyclohexane, Rf = 0.65.
^{1}H NMR (300 MHz, $CDCl_3$) : 0.31 (s, 9H), 3.10 (s, 2H), 3.75 (s, 2H), 7.10-7.35 (m, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) : 1.22, 33.36, 36.40, 118.06, 123.36, 123.93, 124.74, 126.28, 126.37, 128.74, 129.02, 141.06, 141.61, 142.63, 148.50.

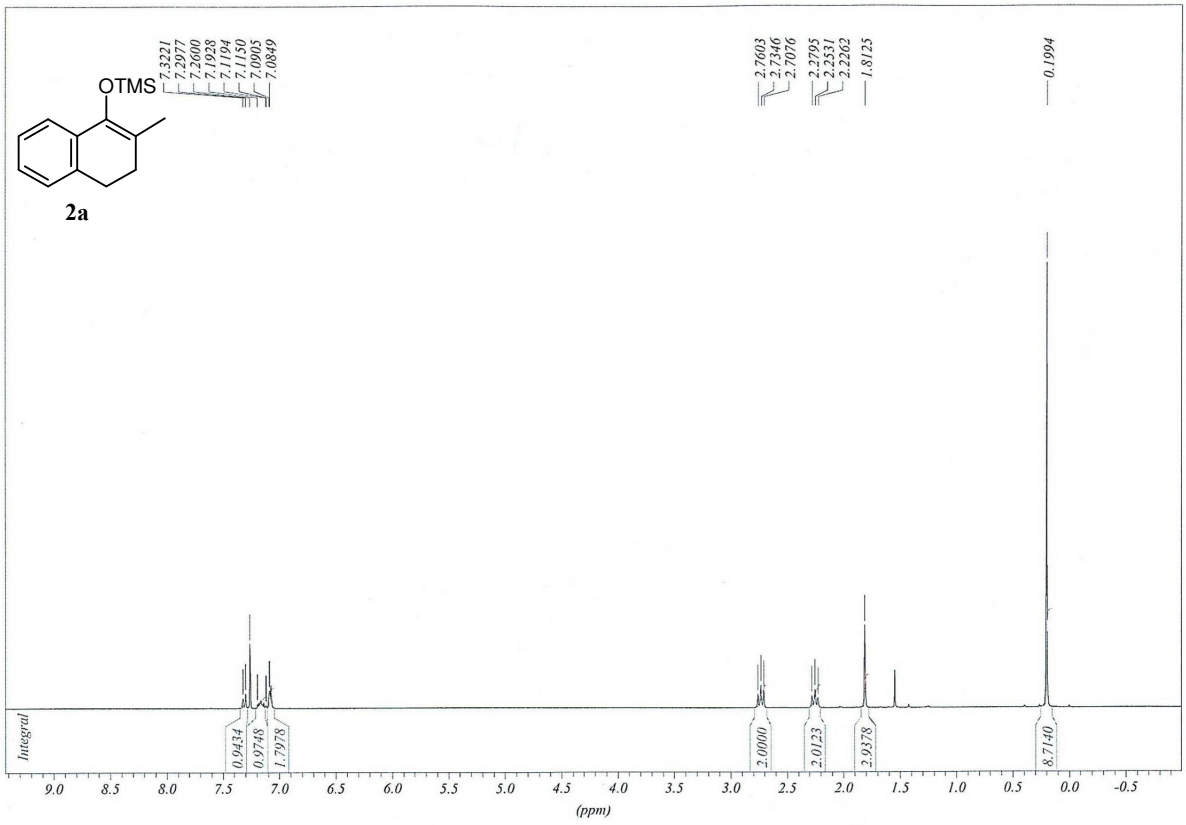
2,2,6-trimethyl-1-trimethylsilyloxy-hexene 2j :

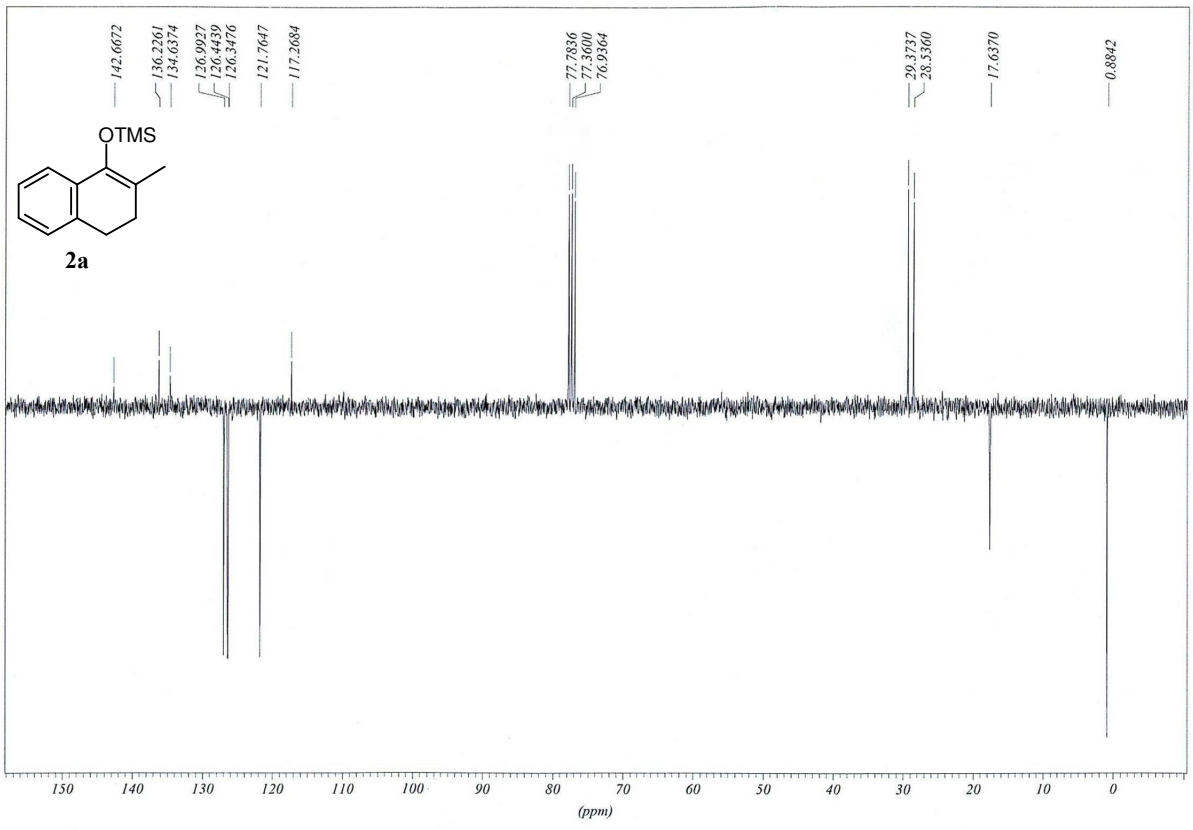


Procedure II
Aspect : Colorless oil.
Yield : 90%.
Purification : Distillation bp = 105°C / 10 mm Hg
^{1}H NMR (300 MHz, $CDCl_3$) : 0.21 (s, 9H), 1.00 (s, 6H), 1.54 (m, 7H), 1.95 (m, 2H).

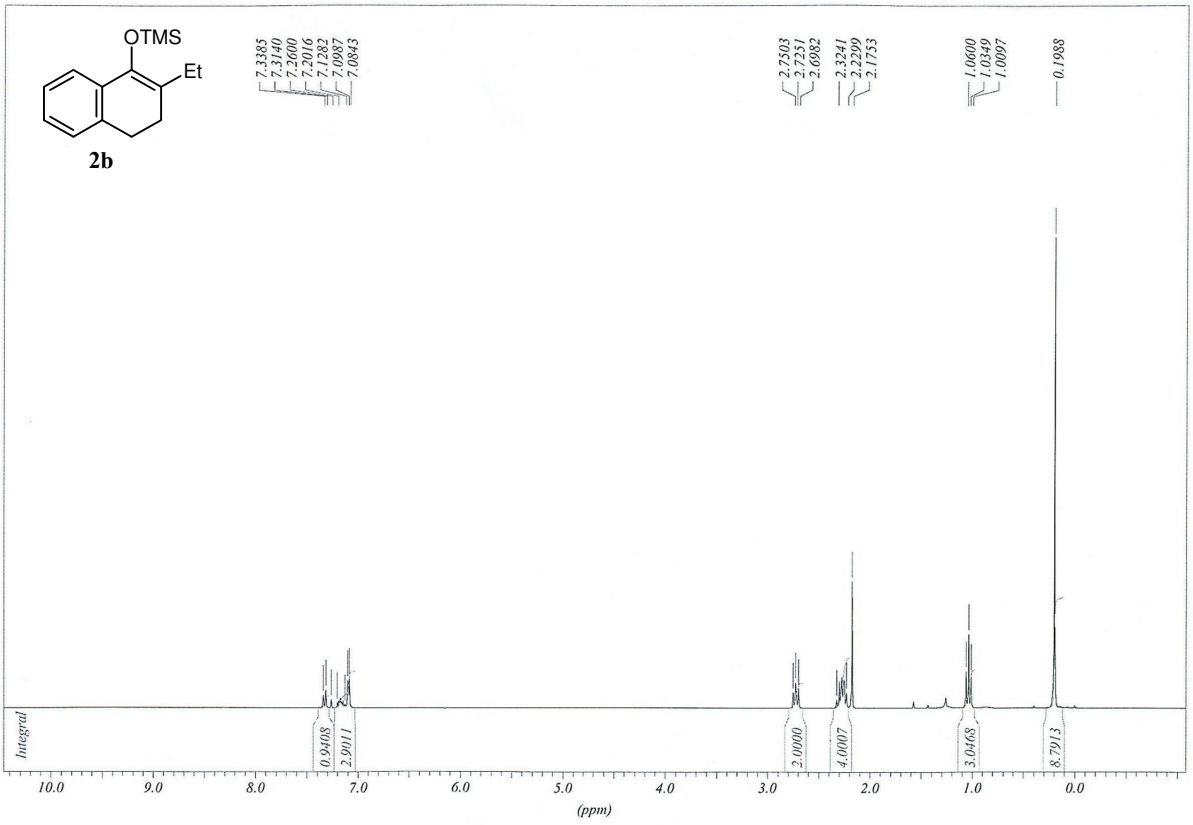
3.3 Copies of the ^{1}H and ^{13}C NMR spectra of Silyl Enol Ethers 2a –j

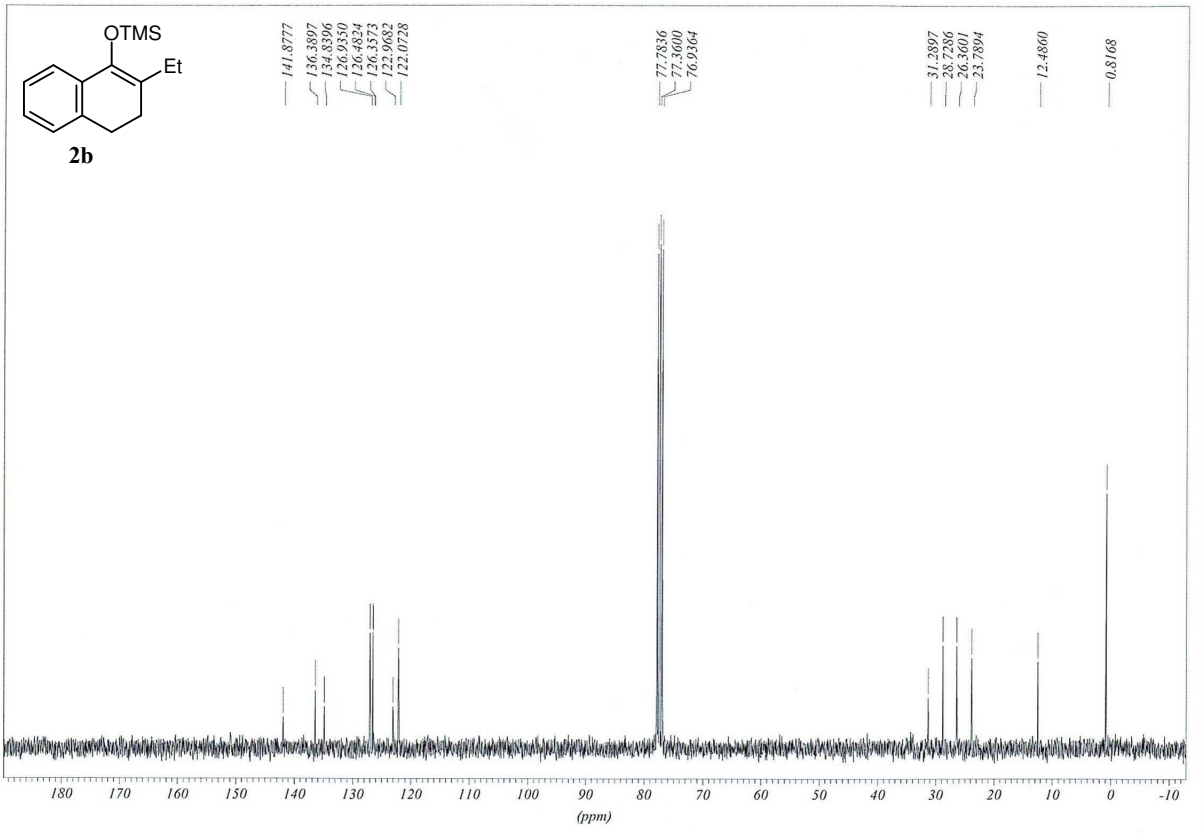
2-methyl-1-trimethylsilyloxy-tetral-1-ene 2a :



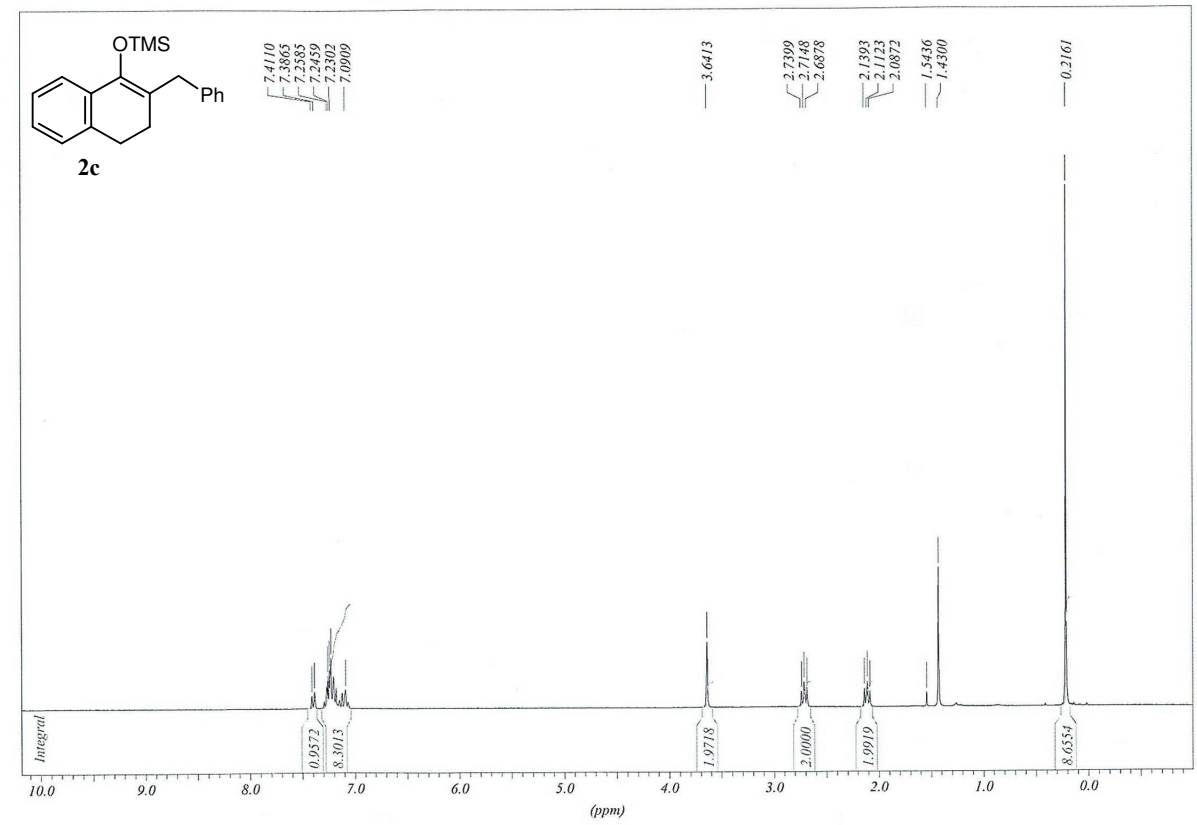


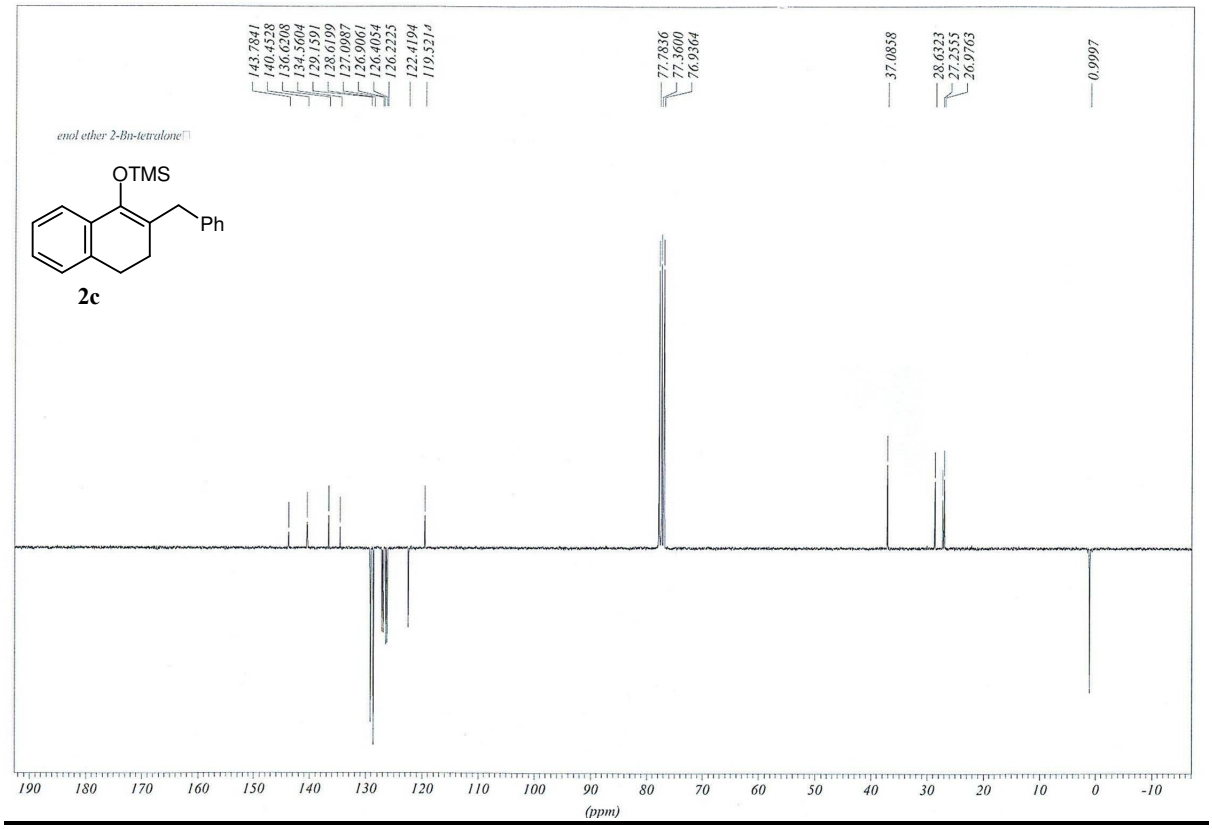
2-ethyl-1-trimethylsilyloxy-tetral-1-ene 2b:



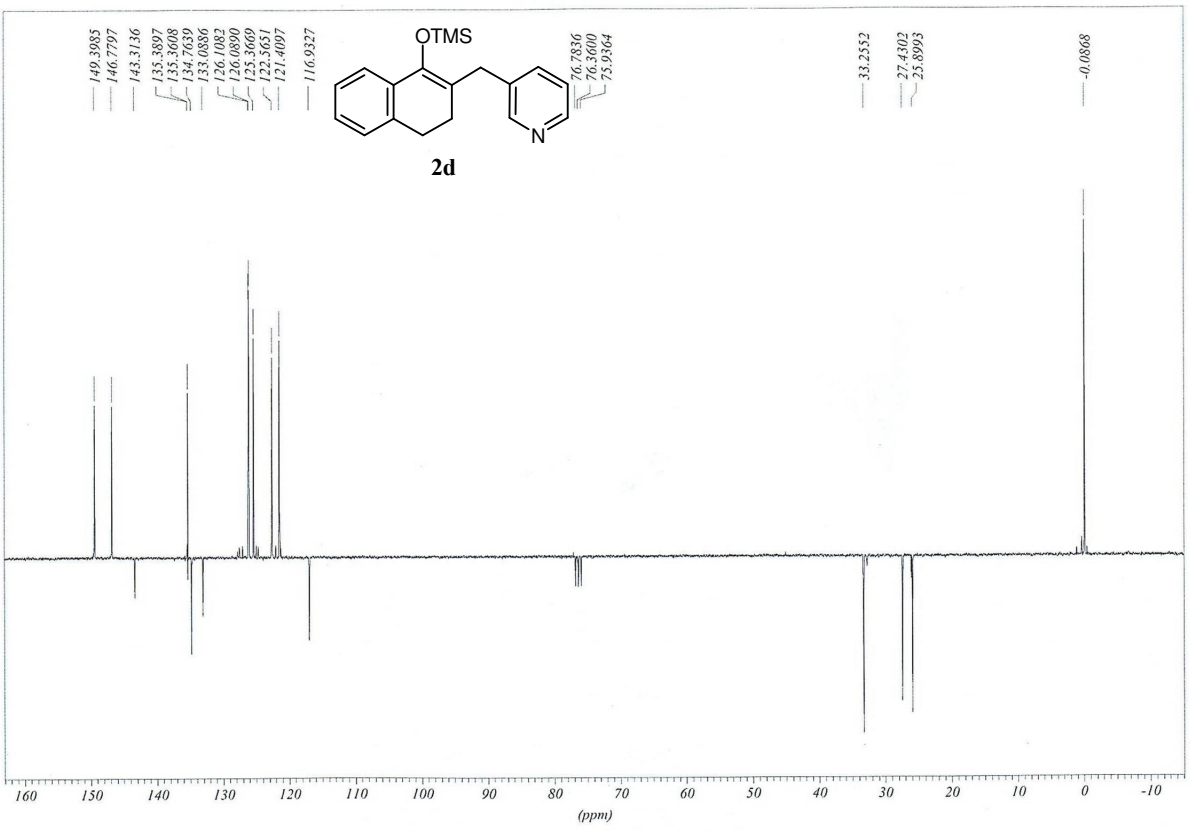


2-benzyl-1-trimethylsilyloxy-tetral-1-ene 2c :

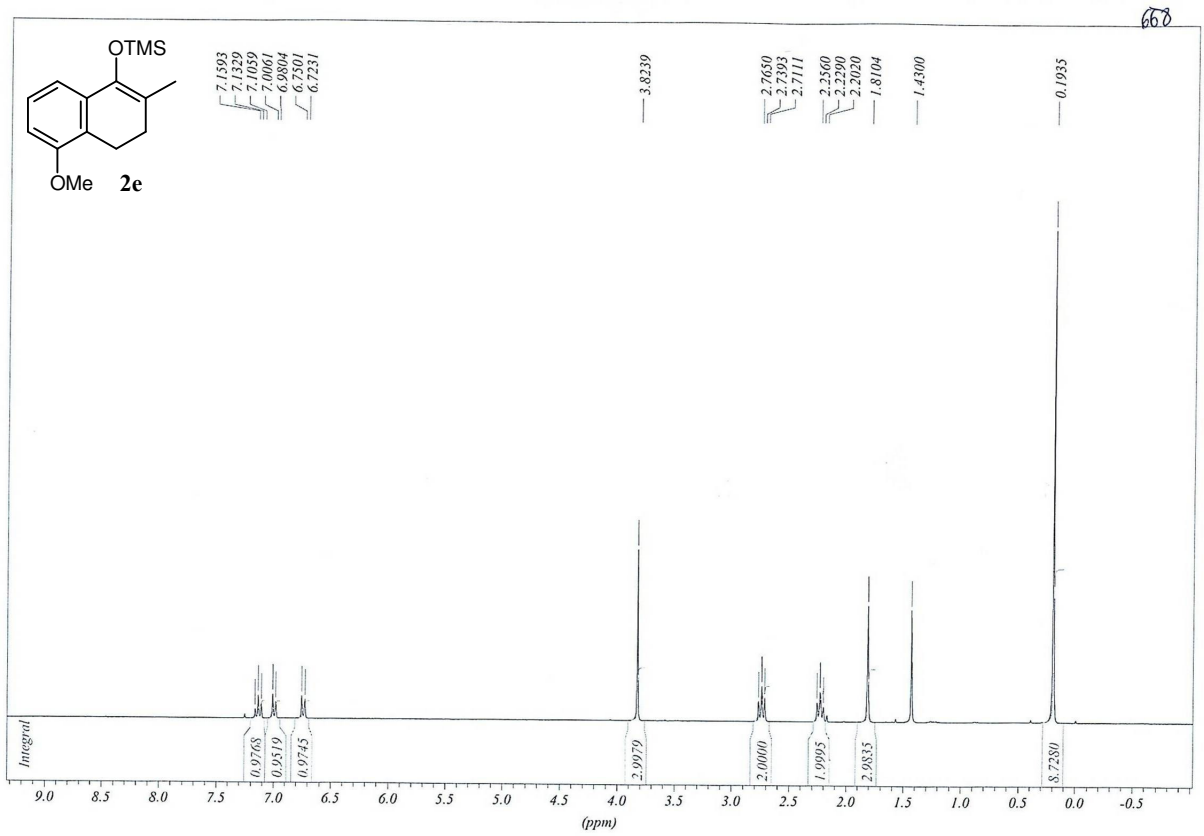


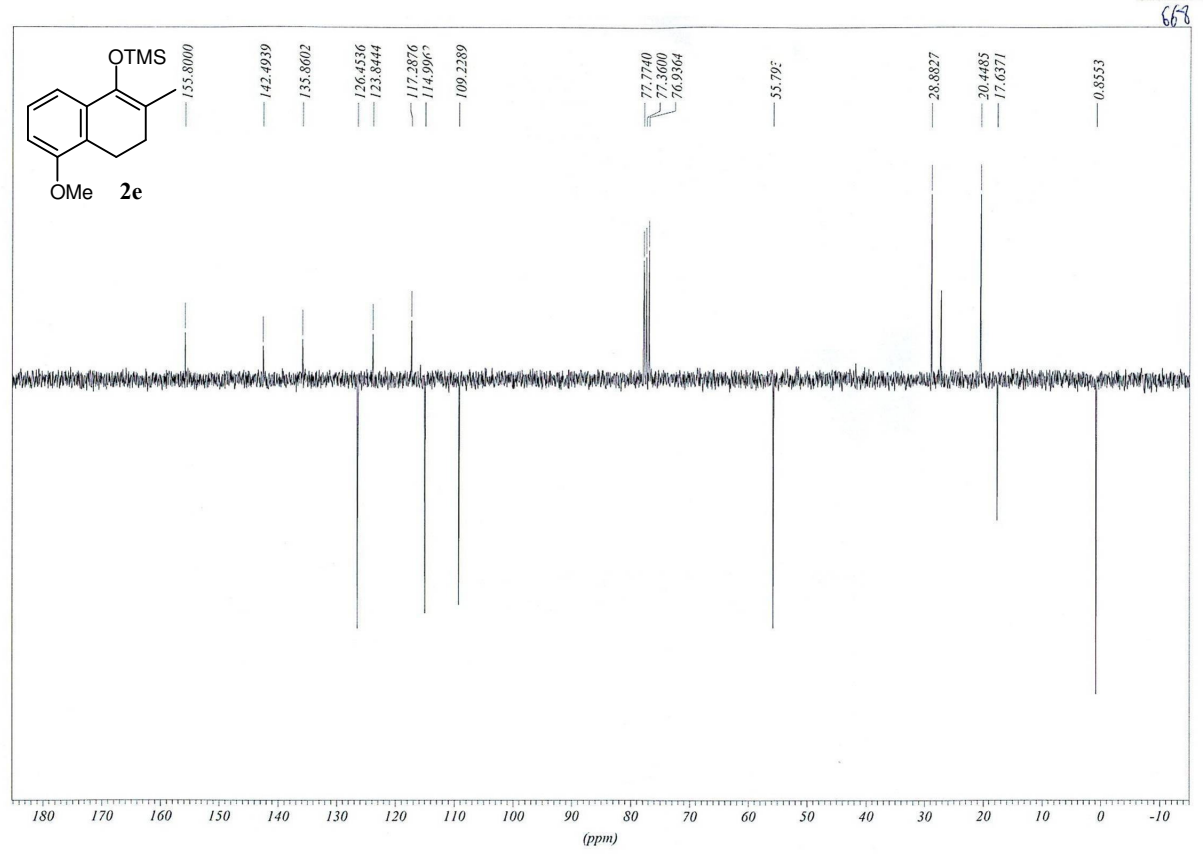


2-(3-picolyl)-1-trimethylsilyloxy-tetral-1-ene 2d :

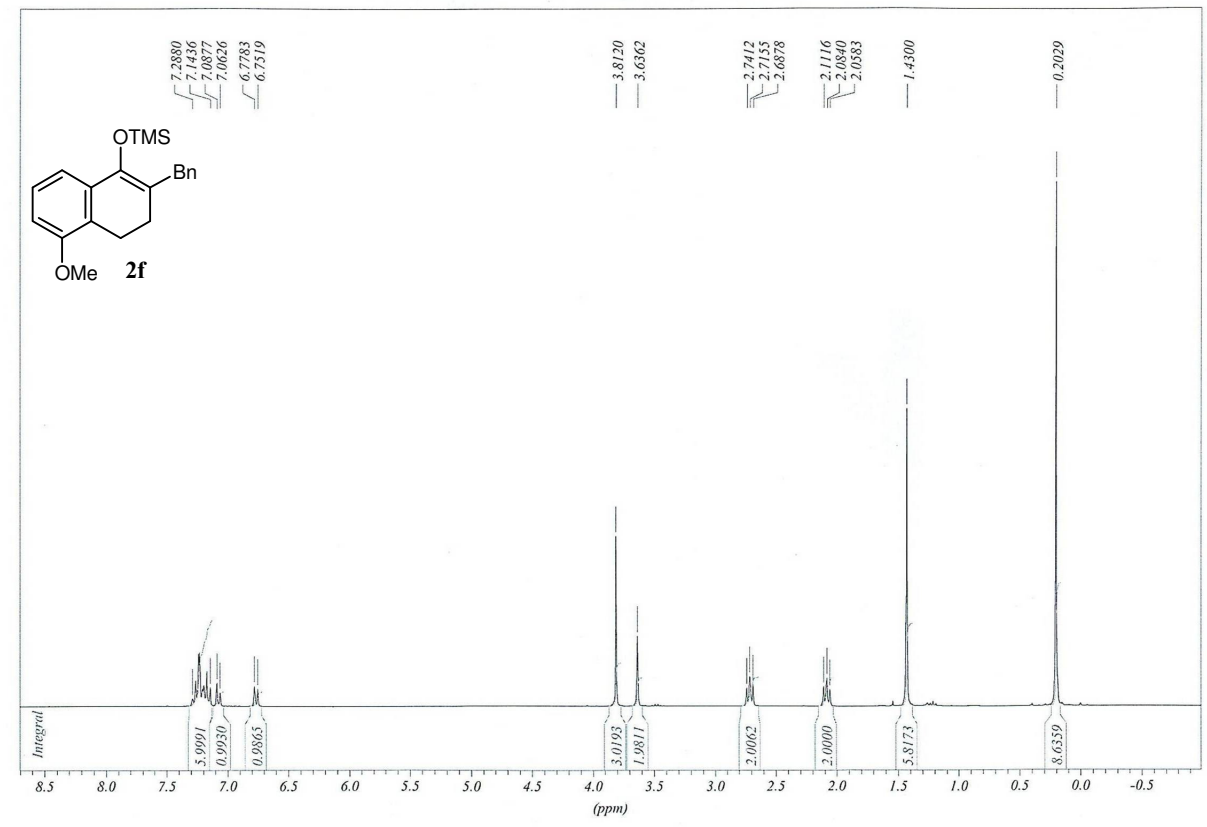


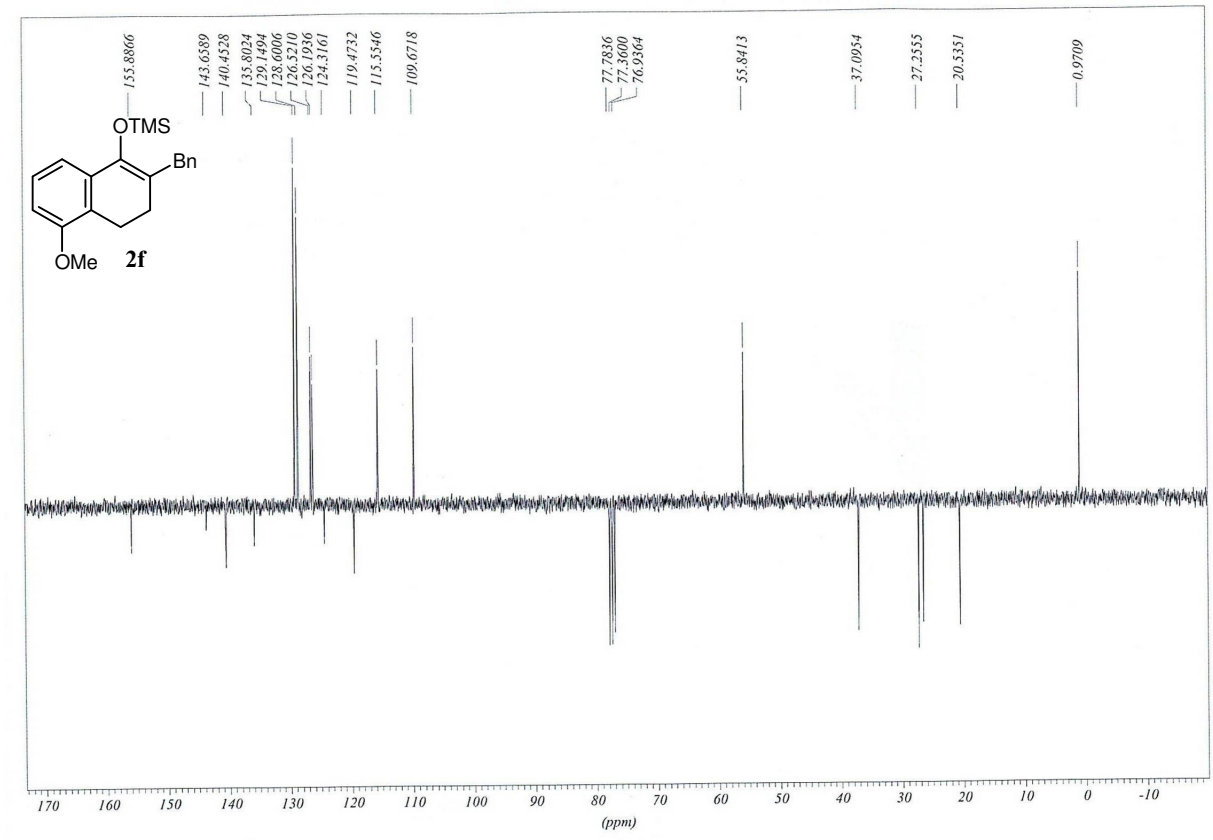
2-methyl-5-methoxy-1-trimethylsilyloxy-tetral-1-ene 2e :



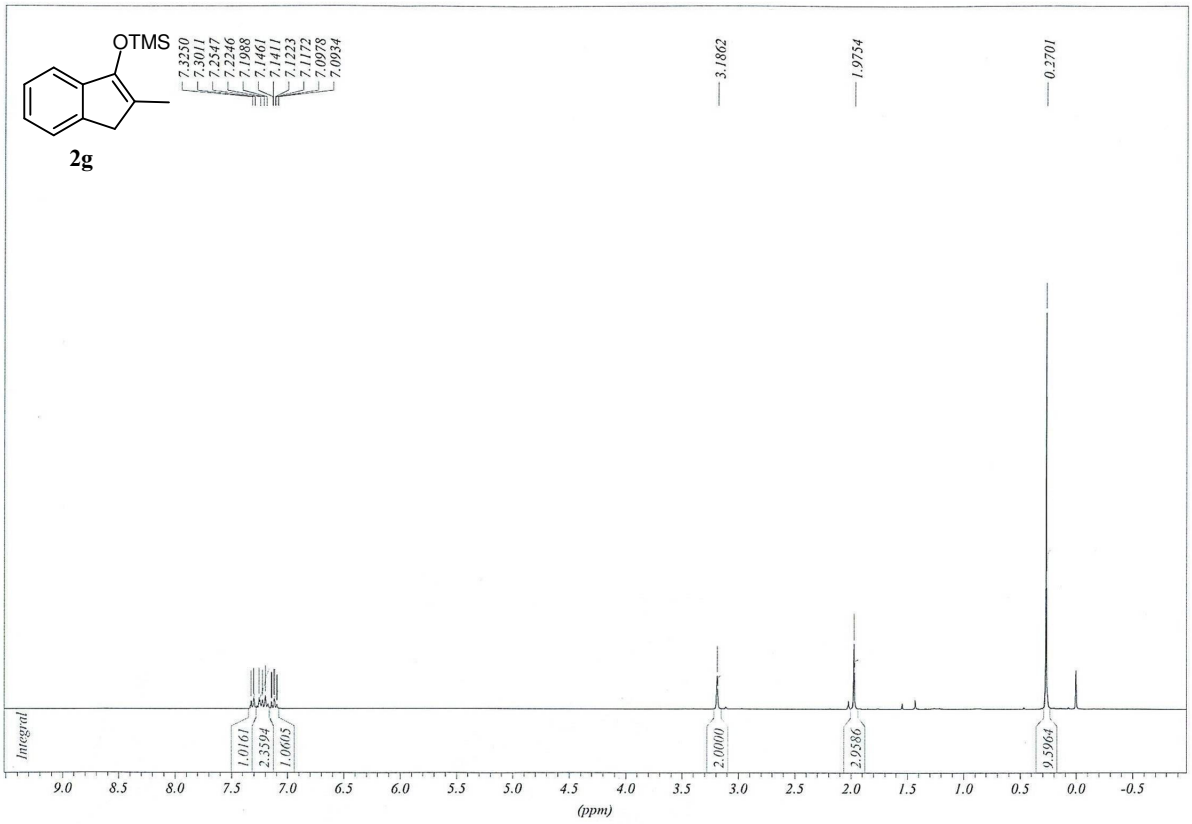


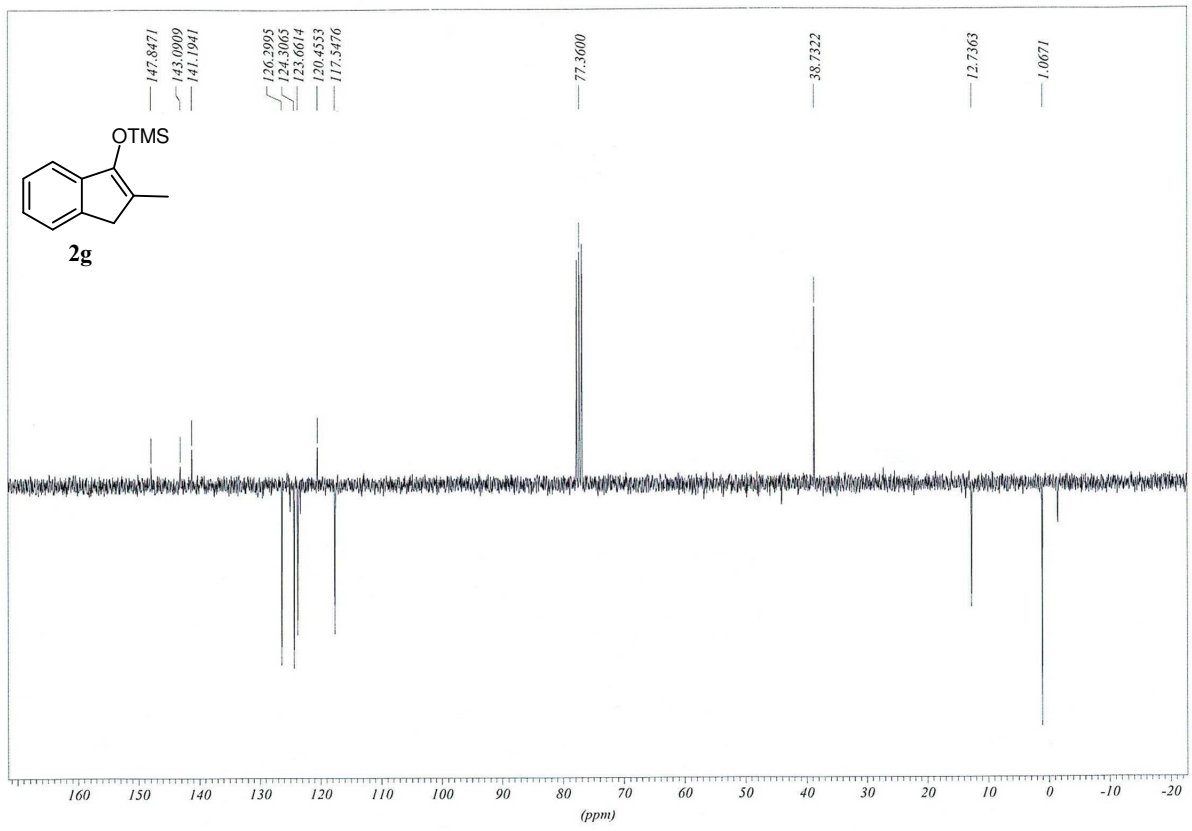
2-benzyl-5-methoxy-1-trimethylsilyloxy-tetral-1-ene 2f :



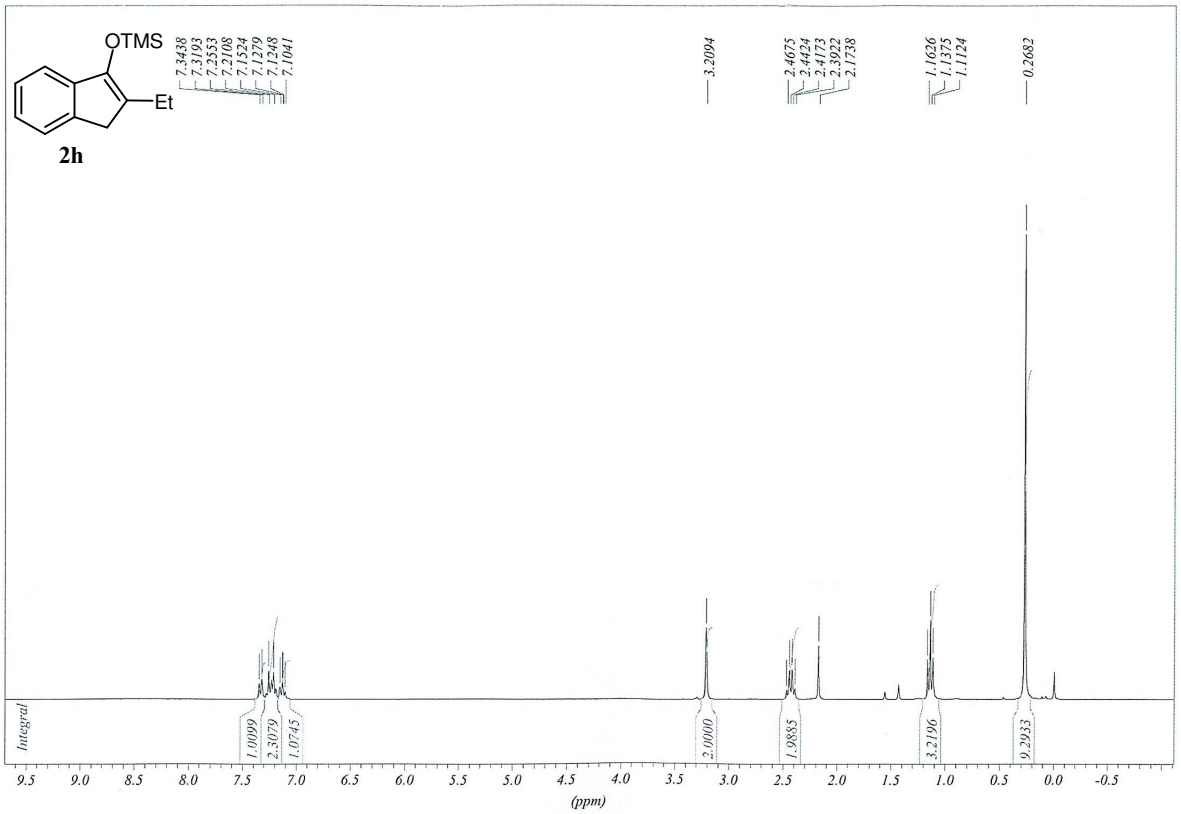


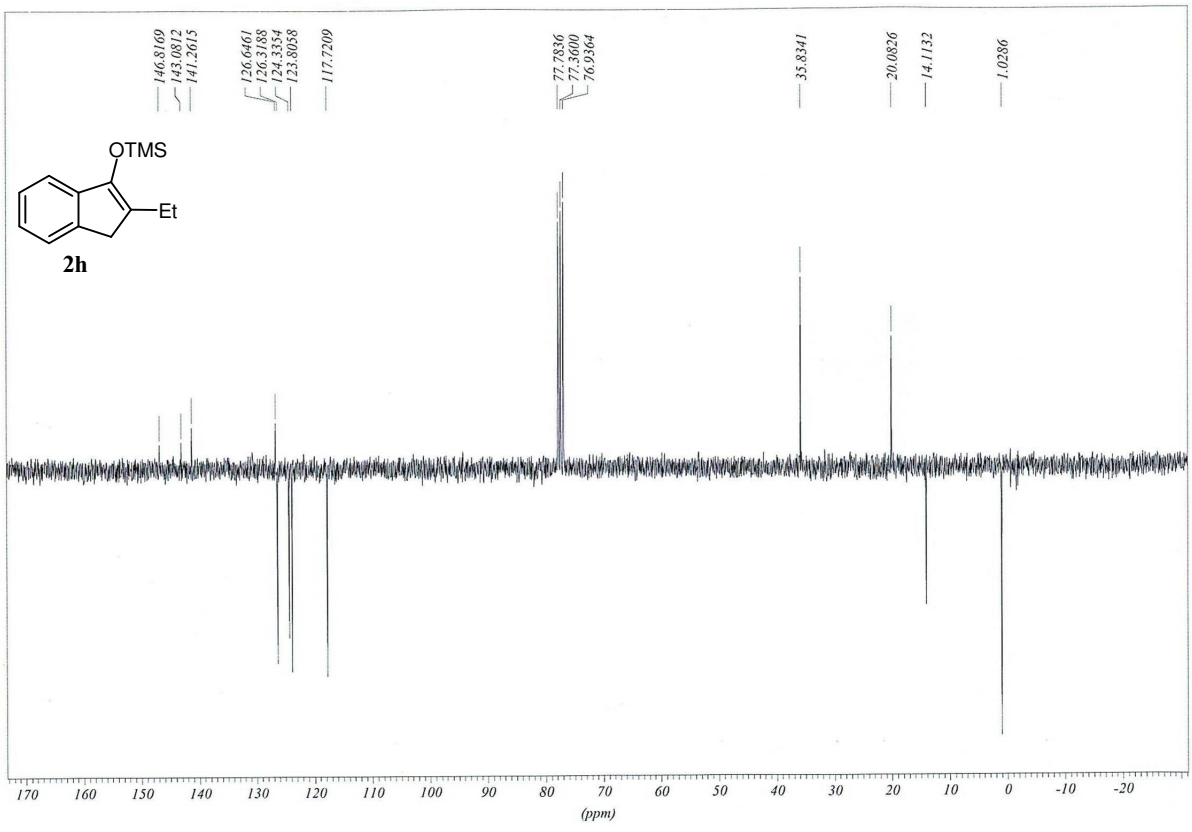
2-methyl-1-trimethylsilyloxy-indan-1-ene 2g :



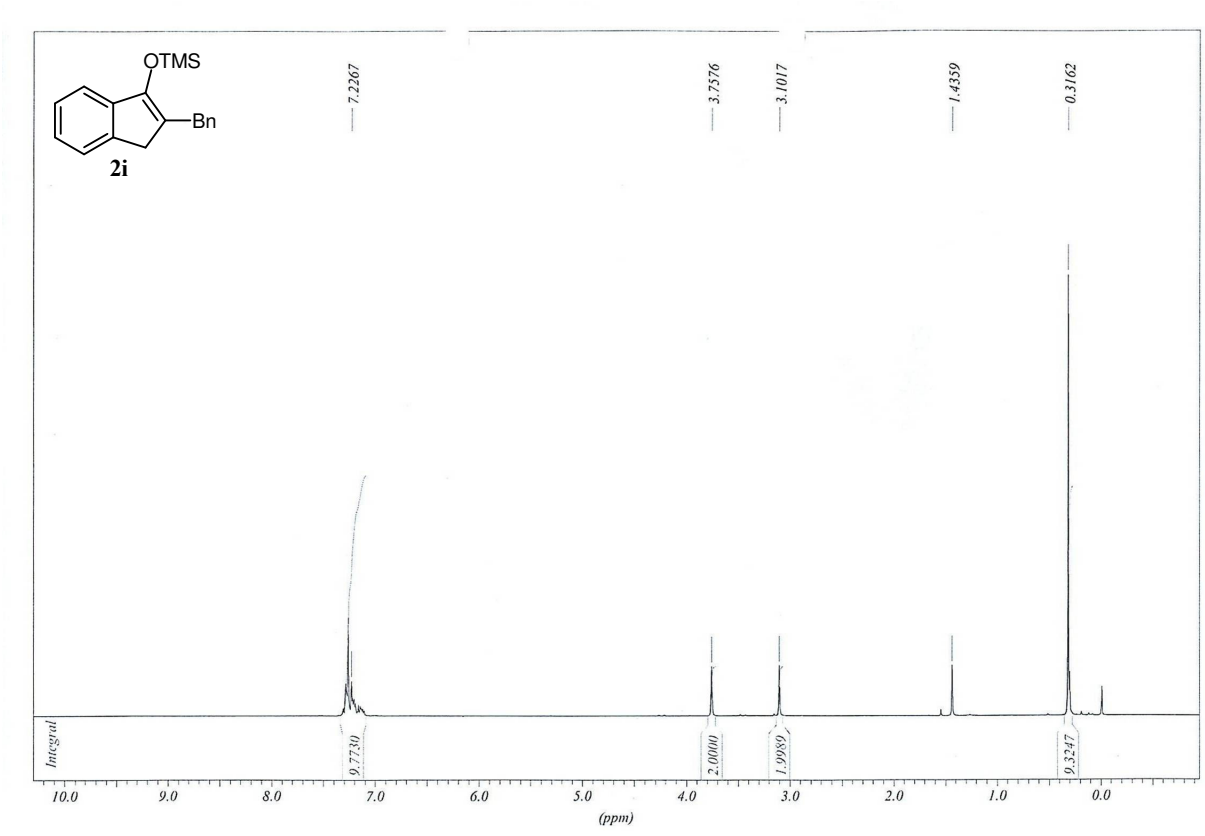


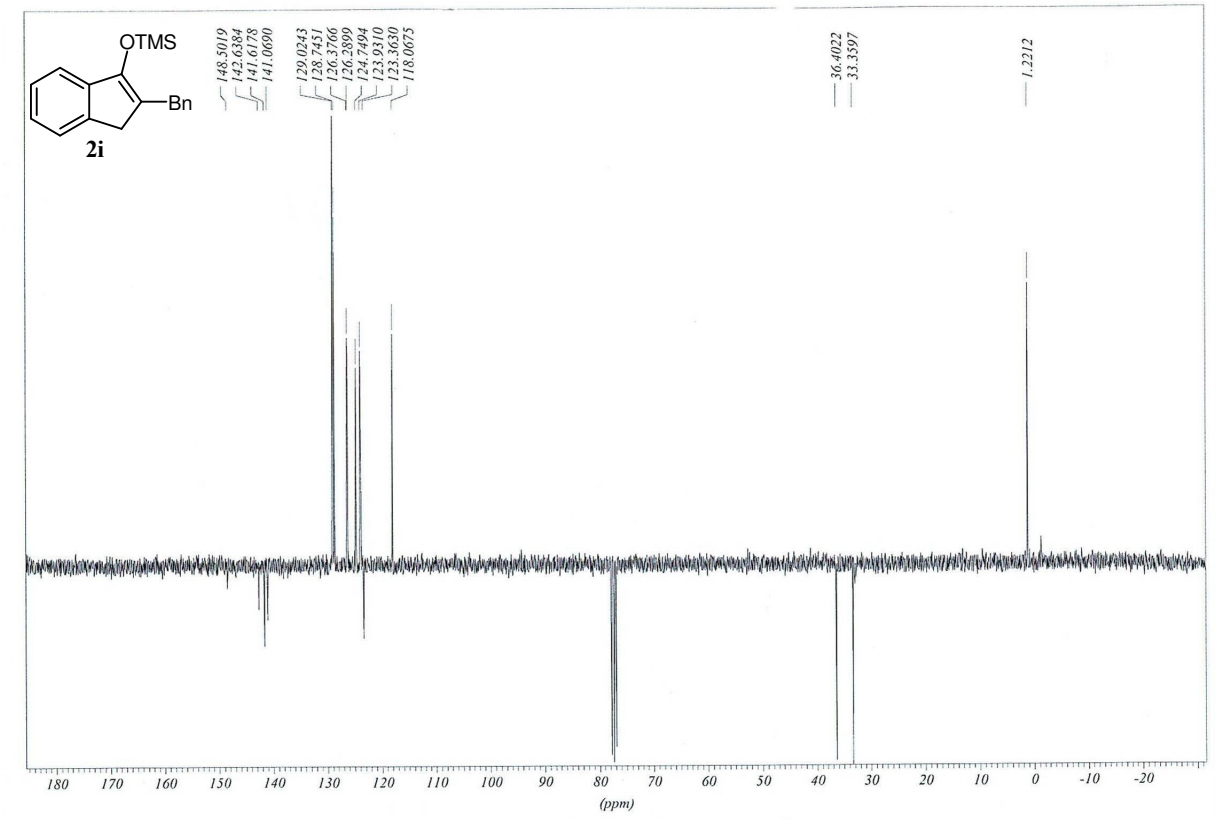
2-ethyl-1-trimethylsilyloxy-indan-1-ene 2h :



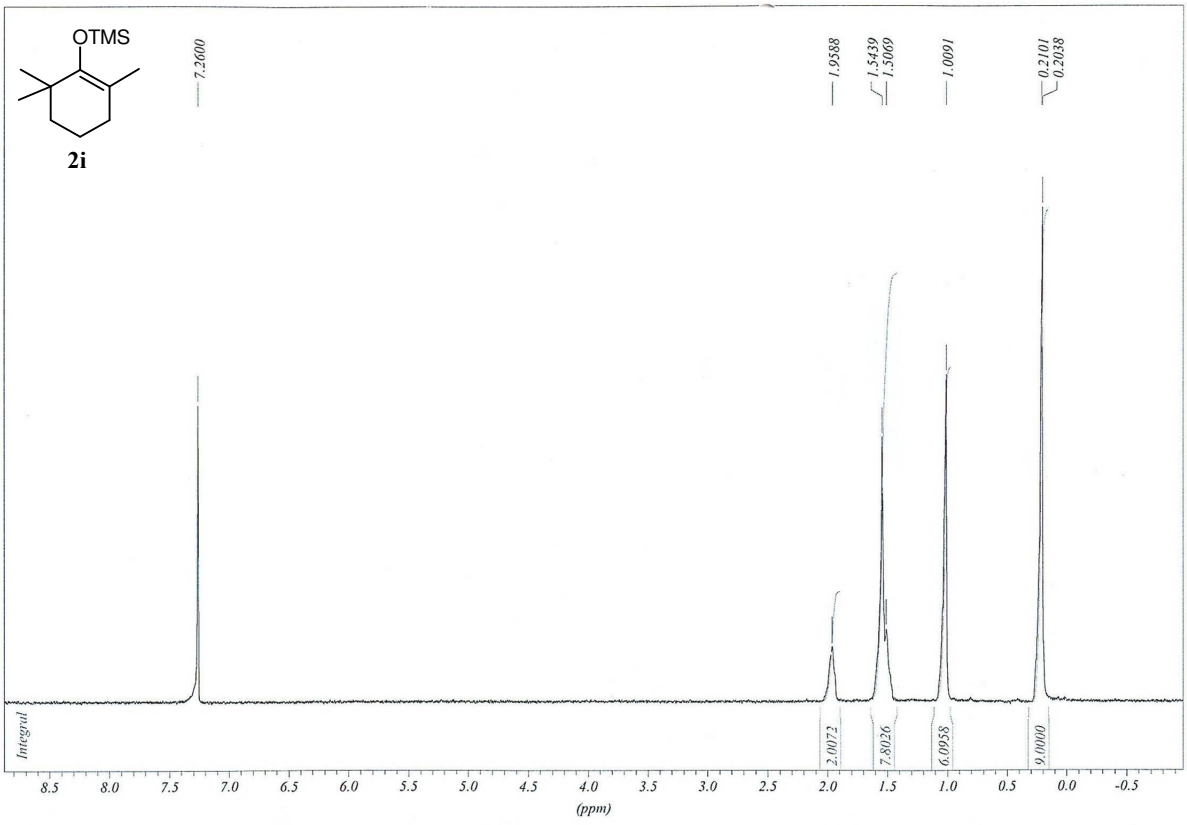


2-benzyl-1-trimethylsilyloxy-indan-1-ene 2i :





2,2,6-trimethyl-1-trimethylsilyloxy-hexene 2j :



4- General Procedure for Enantioselective Protonation (Table 4)

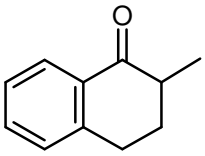
To a solution of silyl enol ether **2a** (0.197 g, 0.85 mmol) in dry DMF (1.2 mL) was added $(DHQ)_2AQN$ (73 mg, 0.085 mmol) as a solution in DMF (0.5 mL) followed by EtOH (0.05 mL, 0.89 mmol) and benzoyl fluoride (0.11 g, 0.89 mmol,). The solution was stirred until complete disappearance of the starting material (monitored by GC / MS). The solution was diluted with Et_2O, washed with saturated $NaHCO_3$ (10 mL) and the aqueous layer was extracted with Et_2O (3 × 10 mL). The combined organic layers were washed with brine (3 × 25 mL) and dried over $MgSO_4$. The solvent was removed under vacuum and the residue was purified by flash chromatography affording the pure ketone **3a** which was analyzed by chiral HPLC.

The catalyst could be recycled by simple filtration through a short plug of silica (Et_2O as eluent), followed by elution with 5% MeOH in DCM as eluent.

5- Data of Product Compounds

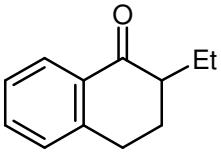
5.1 Methods for the Determination of Enantiomeric Excesses of Ketones 3a – j

(*S*)-2-Methyl-1-tetralone 3a :



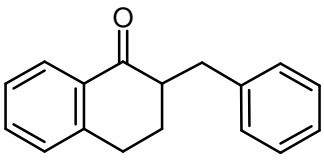
Yields : 88%
Purification : 2% to 5% Et_2O in cyclohexane, Rf = 0.3 (Et_2O / cyclohexane 5 : 95).
ee : 81%
HPLC : Daicel Chiralcel OD-H, heptane / *iso*propanol 99.6 : 0.4, flow rate 0.5 mL / min, 18°C, retention time of enantiomers : 17.05 min (*R*) minor, 18.77 min (*S*) major.
Absolute configuration assigned by comparison with literature data :
Mohr. J. T.; Nishimata. T.; Behema. D. C.; Stoltz. B. M. *J. Am. Chem. Soc.* **2006**, *128*, 11348.

(*S*)-2-Ethyl-1-tetralone 3b :



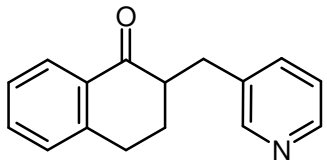
Yields : 68%
Purification : 2% Et_2O in cyclohexane, Rf = 0.28 (Et_2O / cyclohexane 5 : 95).
ee : 76.5%
HPLC : Daicel Chiralcel AD-H, heptane / *iso*propanol 99.96 : 0.04, flow rate 1.2 mL / min, 23.5 °C, retention time of enantiomers : 16.46 min (*S*) major, 17.89 min (*R*) minor.
Absolute configuration assigned by comparison with literature data :
Yanagisawa. A.; Touge. T.; Arai. T. *Angew. Chem. Int. Ed.* **2005**, *44*, 1546.

(*R*)-2-Benzyl-1-tetralone 3c :



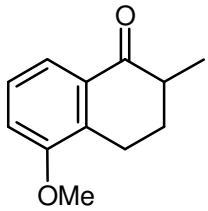
Yields : 84%
Purification : 2% to 5% Et_2O in cyclohexane, Rf = 0.33 (Et_2O / cyclohexane 5 : 95).
ee : 84 %
HPLC : Daicel Chiralcel OJ-H, heptane / *iso*propanol 90 : 10, flow rate 1 mL / min, retention time of enantiomers : 9.80 min (*R*) major, 12.14 min (*S*) minor.
Absolute configuration assigned by comparison with literature data :
Graf. C. D.; Malan. C.; Harms. K.; Knochel. P. *J. Org. Chem.* **1999**, *64*, 5581.

2-(3-picolyl)-1-tetralone 3d :



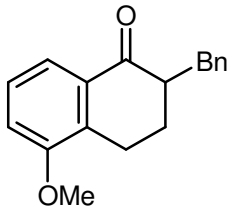
Yields : 91%
Purification : 50% EtOAc in cyclohexane, Rf = 0.23.
ee : 74%
HPLC : Daicel Chiralcel AD-H, heptane / *iso*propanol 90 : 10, flow rate 1 mL / min, retention time of enantiomers : 16.72 min (major), 18.64 min (minor).

2-methyl-5-methoxy-tetral-1-one 3e :



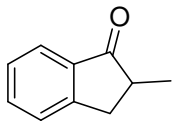
Yields : 98%
Purification : 2% to 5% Et_2O in cyclohexane, Rf = 0.20 (Et_2O / cyclohexane 5 :95).
ee : 81%
HPLC : Daicel Chiralcel AD-H, heptane / *iso*propanol 99.8 : 0.2, flow rate 1 mL / min, 16°C, retention time of enantiomers :19.24 min (major), 21.10 min (minor).

2-benzyl-5-methoxy-tetral-1-one 3f :



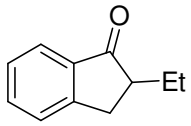
Yields : 86%
Purification : SiO_2, 2% to 5% Et_2O in cyclohexane, Rf = 0.26 (10% Et_2O in cyclohexane).
ee : 91%
HPLC : Daicel Chiralcel OB, heptane / *iso*propanol 99 : 1, flow rate 1 mL / min, 22°C, retention time of enantiomers : 20.38 min (major), 35.90 min (minor).

(*S*)-2-Methyl-1-indanone 3g :



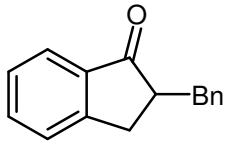
Yields : 78%
Purification : 5% to 10% Et_2O in cyclohexane, Rf = 0.16 (Et_2O / cyclohexane 5 :95).
ee : 71%
HPLC : Daicel Chiralcel OJ-H, heptane / *iso*propanol 99.6 : 0.4, flow rate 1 mL / min, 20°C, retention time of enantiomers : 9.29 min (*S*) major, 10.27 min (*R*) minor.
Absolute configuration assigned by comparison with literature data :
Mohr. J. T.; Nishimata. T.; Behema. D. C.; Stoltz. B. M. *J. Am. Chem. Soc.* **2006**, *128*, 11348.

(*S*)-2-Ethyl-1-indanone 3h :



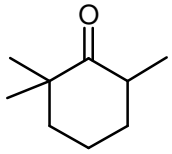
Yields : 76%
Purification : 5% Et_2O in cyclohexane, Rf = 0.22 (Et_2O / cyclohexane 5 :95).
ee : 74%
HPLC : Daicel Chiralcel OJ-H, heptane / *iso*propanol 99.6 : 0.4, flow rate 1 mL / min, retention time of enantiomers : 10.14 min (*S*) major, 12.09 min (*R*) minor.
Absolute configuration was determined by analogy with 2-methyl-indanone.

2-benzyl-1-indanone 3i :



Yields : 77%
Purification : 5% EtOAc in cyclohexane, Rf = 0.21 (EtOAc / cyclohexane 5 :95).
ee : 64%
HPLC : Daicel Chiralcel OJ-H, heptane / *iso*propanol 90 : 10, flow rate 1 mL / min, retention time of enantiomers : 11.30 min (major), 12.73 min (minor).

(*S*)-2,2,6-Trimethylcyclohexanone 3j :



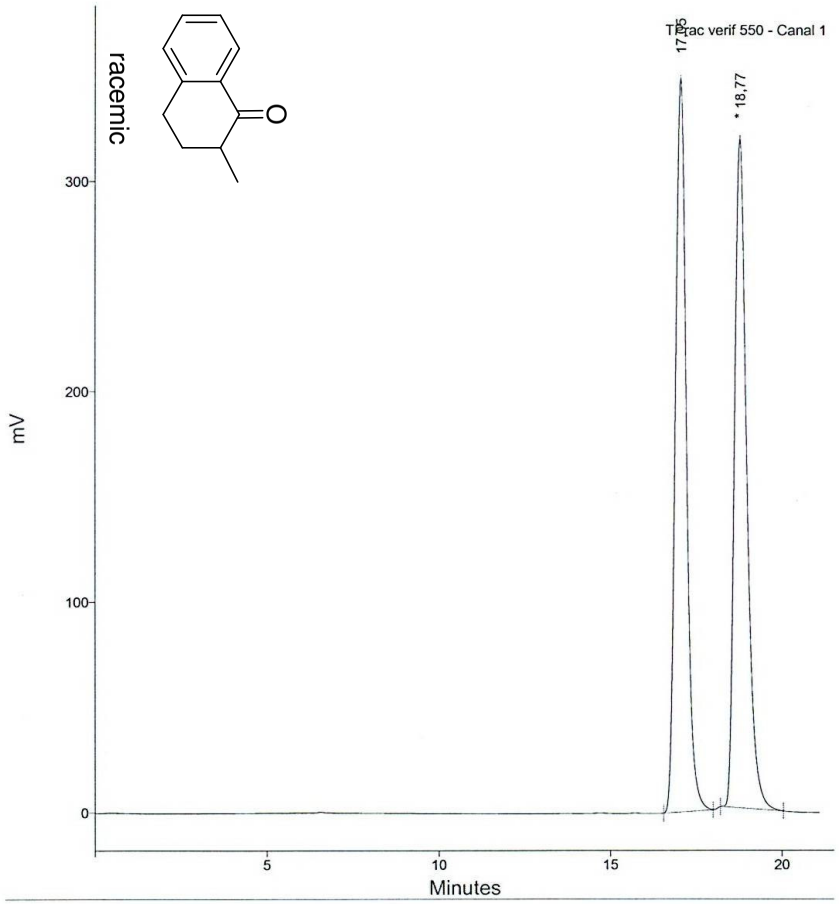
Yields : 98% (GC yield)
Purification : Filtration through a short pad of silice (Et_2O as eluant).
ee : 58%

GC : Chiraldex CB (25m × 0.25), isotherm 90°C, 1 mL.min^{-1} carrier gas flow, retention time of enantiomers : 12.25 min (*R*) minor, 13.63 min (*S*) major.
Absolute configuration was assigned by comparing the sign of optical rotation with literature data :
Eleveld. M. B.; Hogeveen. H. *Tetrahedron Lett.* **1986**, 631.

5.2 Copies of HPLC Chromatograms of Ketones 3a – j

2-methyl-tetralone 3a :

Analyse : TPrac verif 550



Informations sur l'acquisition

Date d'acquisition	02/12/2006 12:52:45 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	21,09 Minutes	Nb de points	12652
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		17,05	7827,41	49,93	0,00	0,00
2		18,77	7847,94	50,07	0,00	0,00
SOMME			**15675,35**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

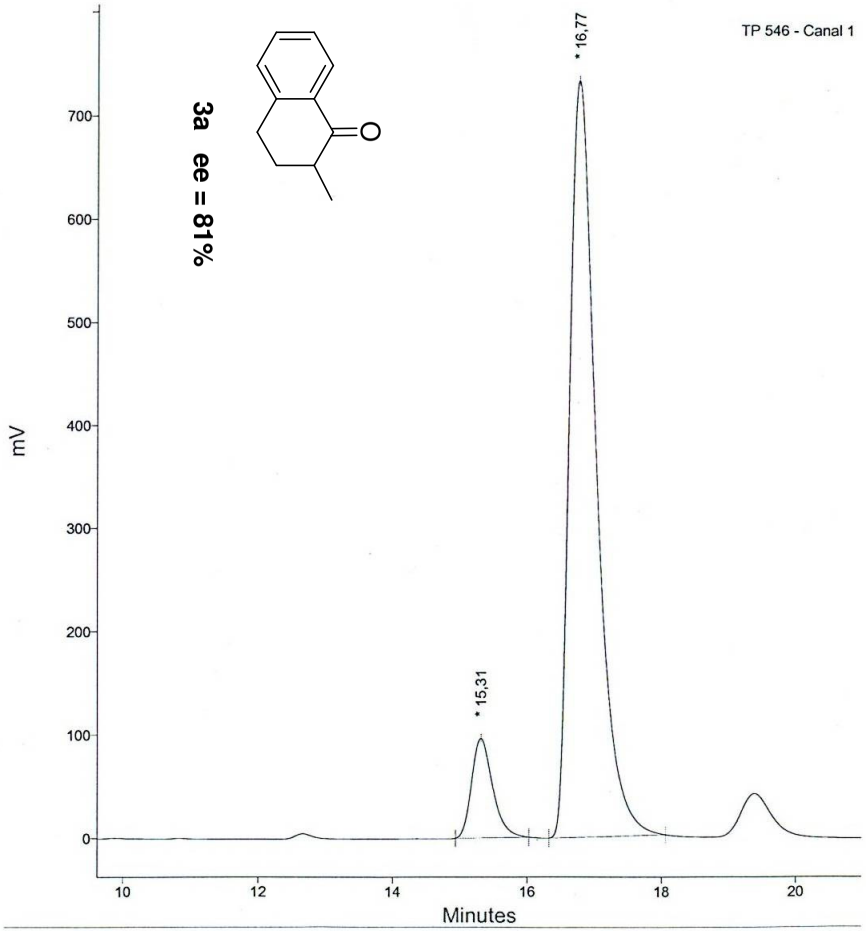
Nom	TPrac verif 550	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

OD-H heptane 99.6/0.4 0.5ml/min @254nm 18°C

Analyse : TP 546



Informations sur l'acquisition

Date d'acquisition	23/11/2006 11:54:55 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	21,67 Minutes	Nb de points	13005
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		15,31	2099,11	9,39	0,00	0,00
2		16,77	20253,20	90,61	0,00	0,00
SOMME			**22352,32**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	TP 546	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

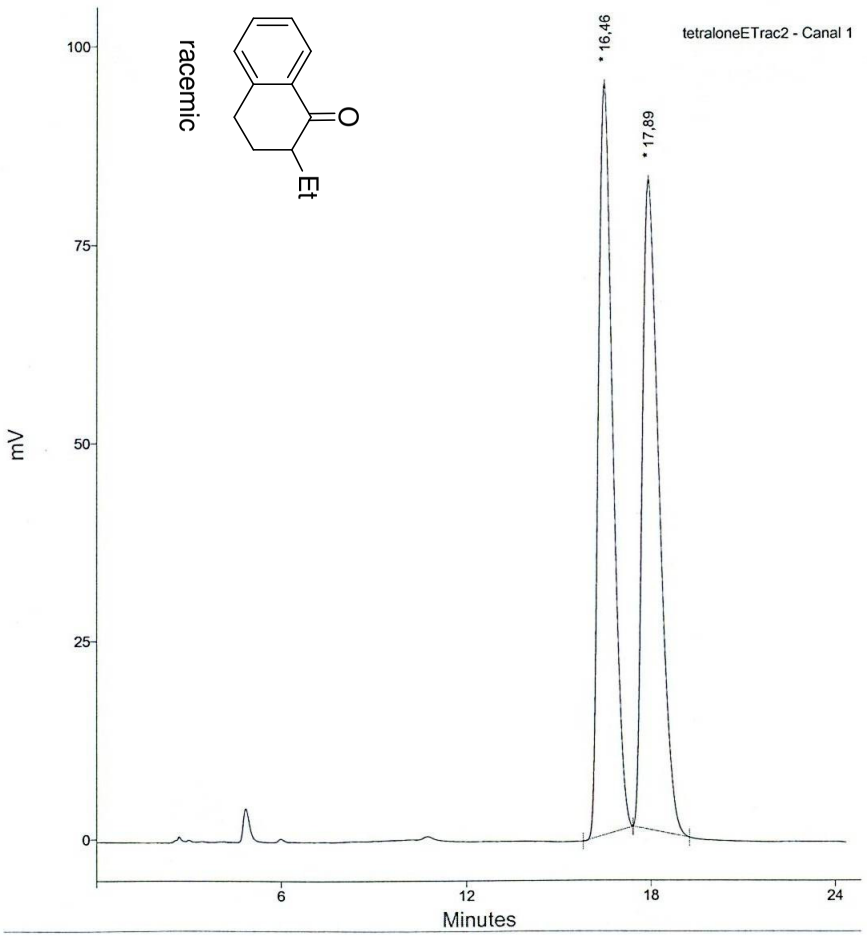
(* = valeur initiale modifiée)

Informations :

OD-H heptane/IPA: 99.6/0.4 0.5ml/min @254nm 22°C

2-ethyl-tetralone 3b :

Analyse : tetraloneETrac2



Informations sur l'acquisition

Date d'acquisition	20/03/2007 14:42:28 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	24,35 Minutes	Nb de points	14609
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		16,46	3078,42	49,72	0,00	0,00
2		17,89	3112,70	50,28	0,00	0,00
SOMME			**6191,12**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

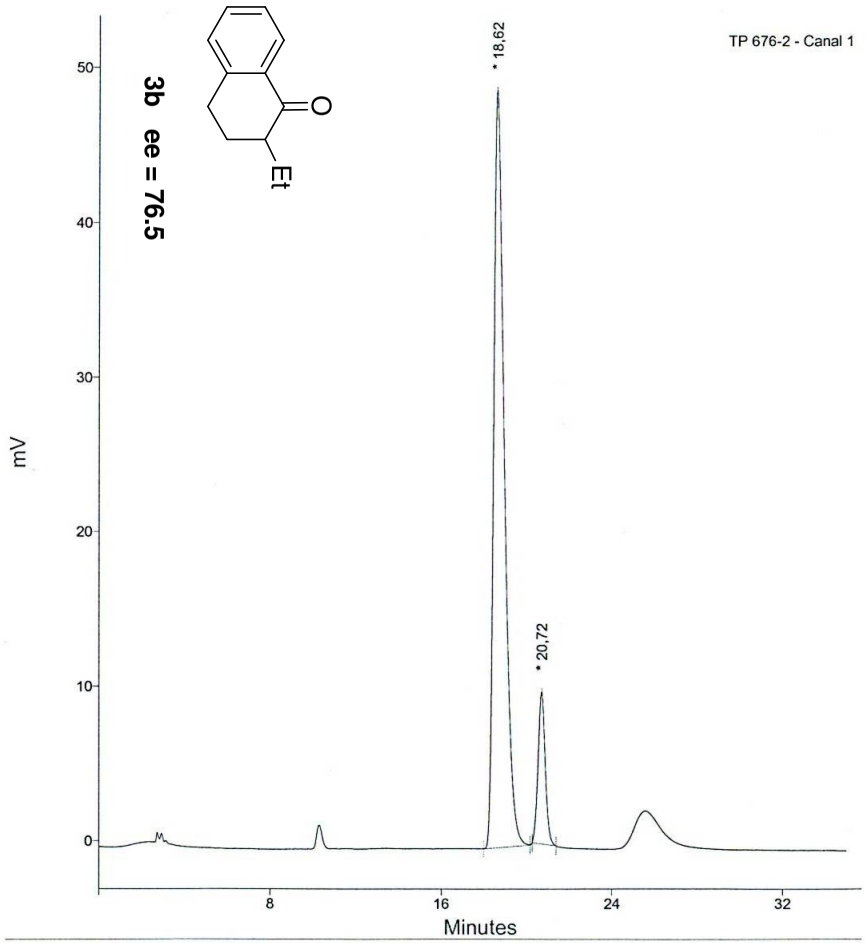
Nom	tetraloneETrac2	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

AD-H Heptane / iPrOH 99.96/0.04 1.2ml/min 254nm 23.5°C

Analyse : TP 676-2



Informations sur l'acquisition

Date d'acquisition	23/03/2007 10:56:47 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	35,00 Minutes	Nb de points	21001
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		18,62	1698,65	88,25	0,00	0,00
2		20,72	226,23	11,75	0,00	0,00
SOMME			**1924,88**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

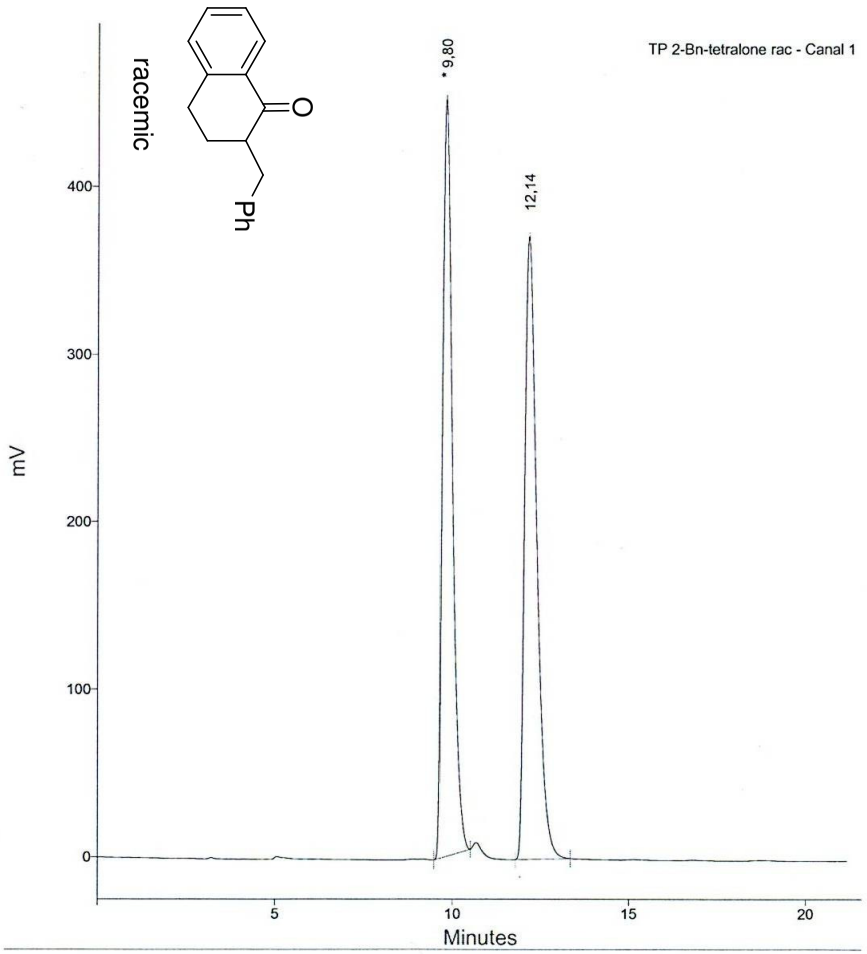
Nom	TP 676-2	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

AD-H Heptane / iPrOH 99.96/0.04 1ml/min 254nm 20°C

Analyse : TP 2-Bn-tetralone rac



Informations sur l'acquisition

Date d'acquisition	11/01/2007 15:01:35 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :	INT7Channel 1		
Durée	21,17 Minutes	Nb de points	12705
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		9,80	8696,39	49,32	0,00	0,00
2		12,14	8936,90	50,68	0,00	0,00
SOMME			**17633,29**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	TP 2-Bn-tetralone rac	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

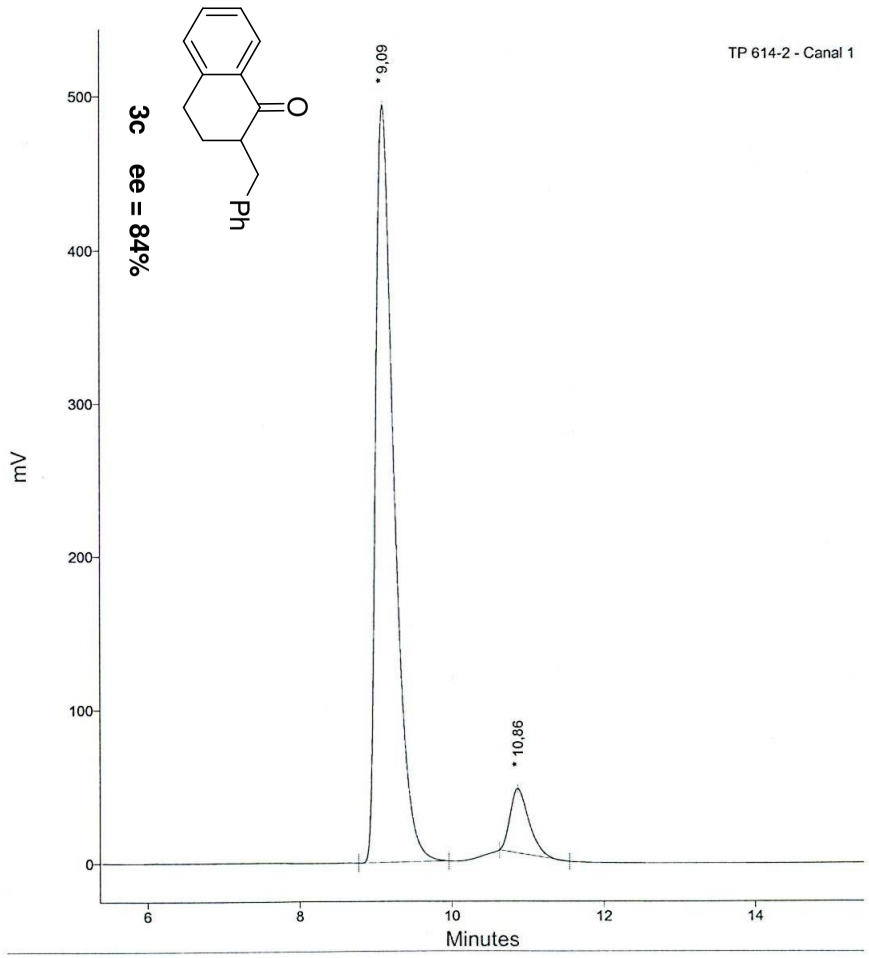
(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 90/10 1ml/min @254nm

2-benzyl-tetralone 3c :

Analyse : TP 614-2



Informations sur l'acquisition

Date d'acquisition	01/02/2007 16:18:57 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	16,38 Minutes	Nb de points	9826
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		9,09	8234,71	91,94	0,00	0,00
2		10,86	721,60	8,06	0,00	0,00
SOMME			8956,31	100,00	0,00	0,00

Informations sur l'échantillon

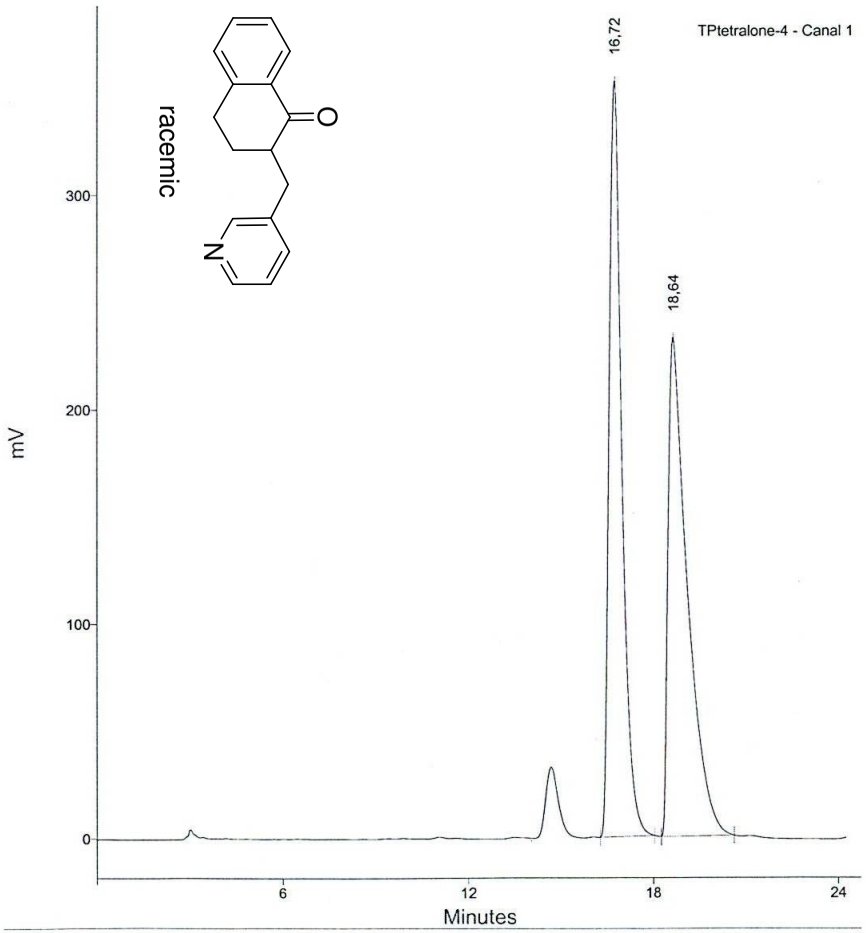
Nom	TP 614-2	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 90/10 1ml/min @210nm

Analyse : TPtetralone-4



Informations sur l'acquisition

Date d'acquisition	13/02/2007 10:48:43 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	24,26 Minutes	Nb de points	14555
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		16,72	10201,40	49,97	0,00	0,00
2		18,64	10213,52	50,03	0,00	0,00
SOMME			20414,92	100,00	0,00	0,00

Informations sur l'échantillon

Nom	TPtetralone-4	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

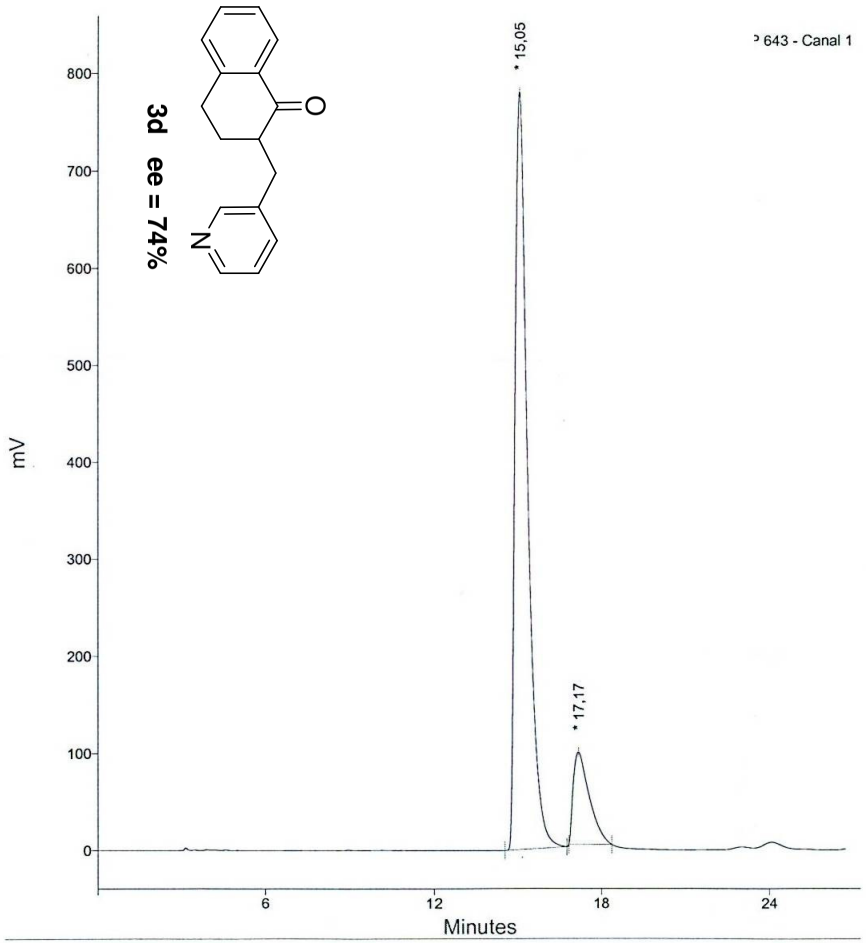
OJ-H heptane/IPA 90/10 1ml/min @254nm @19°C

AD

2-(3-picolyl)-1-tetralone 3d :

ee = 73,4%

Analyse : TP 643



3d ee = 74%

Informations sur l'acquisition

Date d'acquisition	19/02/2007 12:55:45 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	26,74 Minutes	Nb de points	16044
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		15,05	24618,02	86,71	0,00	0,00
2		17,17	3772,87	13,29	0,00	0,00
SOMME			**28390,88**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	TP 643	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

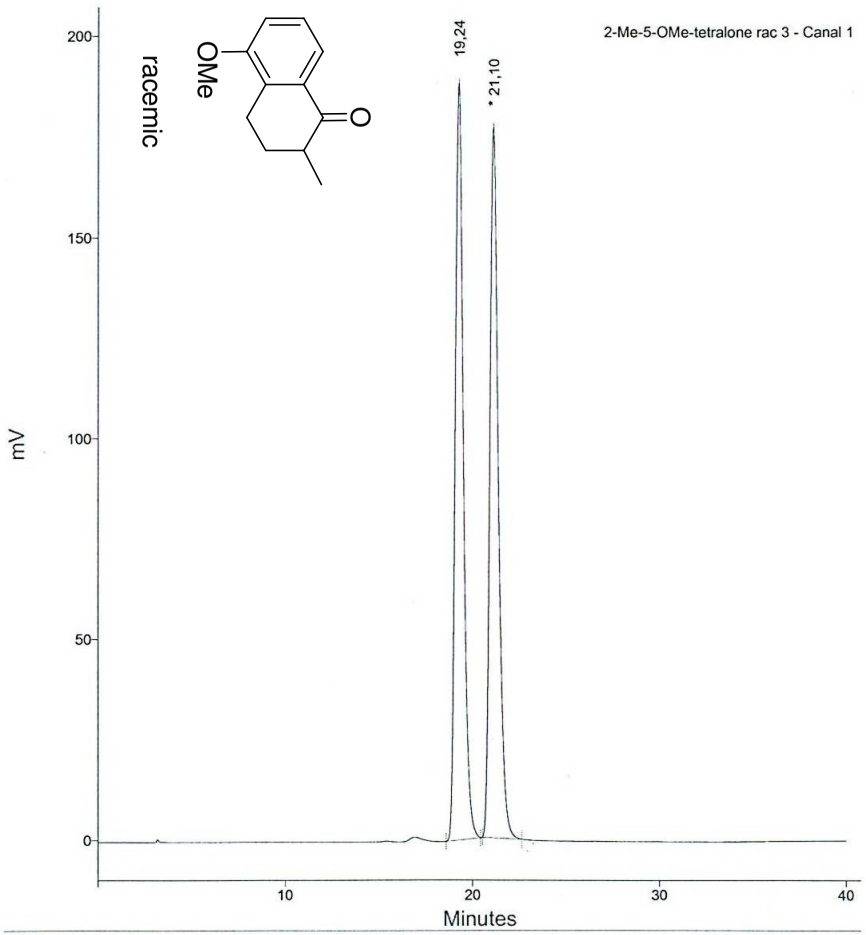
(* = valeur initiale modifiée)

Informations :

AD-H Heptane / iPrOH 90.10 1ml/min 254nm

2-methyl-5-methoxy-tetral-1-one 3e :

Analyse : 2-Me-5-OMe-tetralone rac 3



Informations sur l'acquisition

Date d'acquisition	15/03/2007 08:25:18 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	40,00 Minutes	Nb de points	24001
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		19,24	5646,53	49,98	0,00	0,00
2		21,10	5651,47	50,02	0,00	0,00
SOMME			11298,00	100,00	0,00	0,00

Informations sur l'échantillon

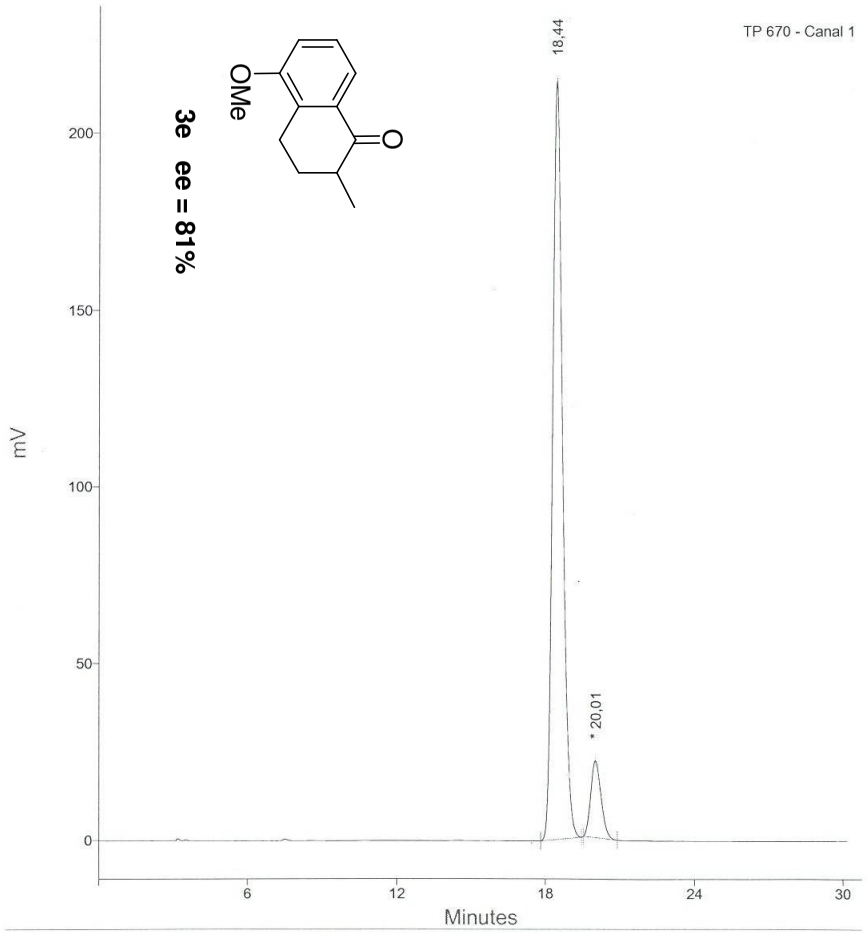
Nom	2-Me-5-OMe-tetralone rac	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

AD-H Heptane / iPrOH 99.8/0.2 1ml/min 254nm 16°C

Analyse : TP 670



Informations sur l'acquisition

Date d'acquisition	17/03/2007 15:57:15 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	30,16 Minutes	Nb de points	18097
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		18,44	5989,55	90,52	0,00	0,00
2		20,01	627,43	9,48	0,00	0,00
SOMME			**6616,97**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

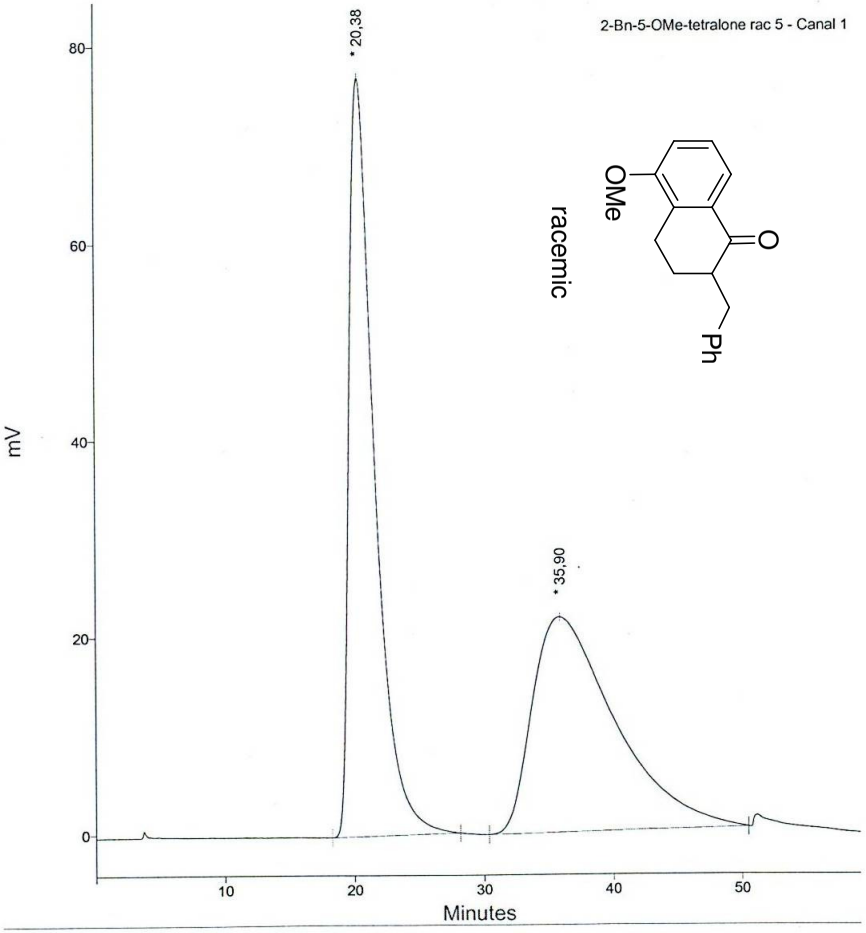
Nom	TP 670	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

AD-H Heptane / iPrOH 99.8/0.2 1ml/min 254nm 23.5°C

Analyse : 2-Bn-5-OMe-tetralone rac 5



racemic

Informations sur l'acquisition

Date d'acquisition	15/03/2007 13:29:15 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	59,17 Minutes	Nb de points	35504
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		20,38	10461,35	51,84	0,00	0,00
2		35,90	9719,35	48,16	0,00	0,00
SOMME			**20180,69**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	2-Bn-5-OMe-tetralone rac	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

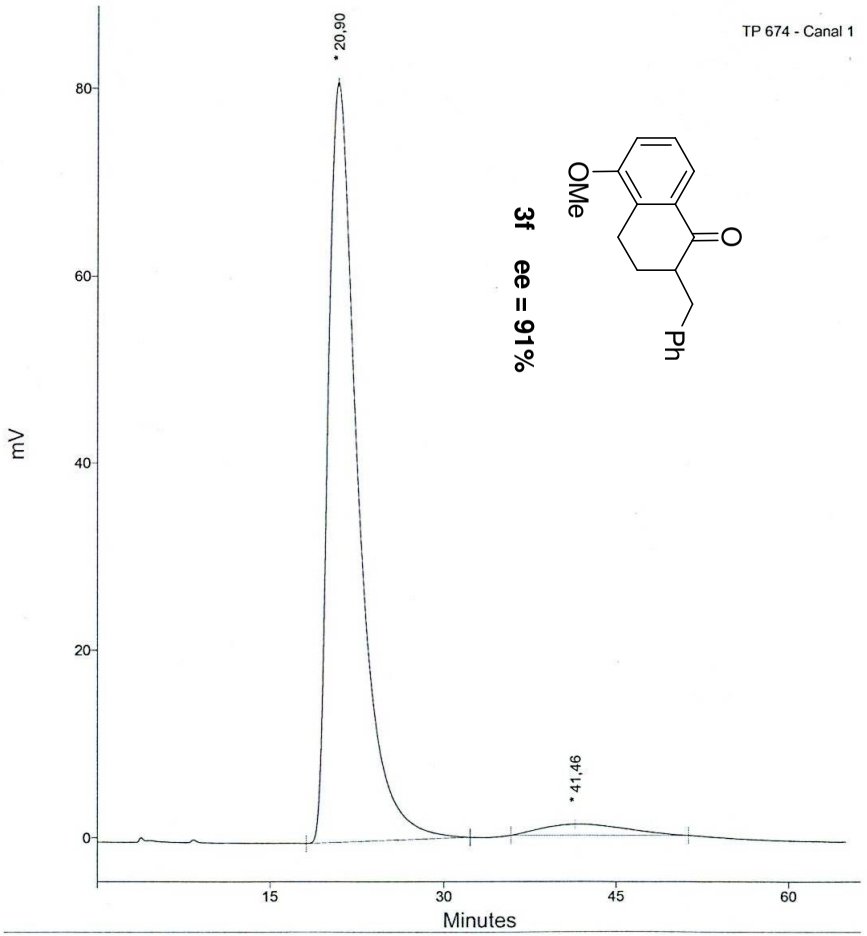
(* = valeur initiale modifiée)

Informations :

OB Heptane / iPrOH 99/1 1ml/min 254nm 22°C

2-benzyl-5-methoxy-tetral-1-one 3f :

Analyse : TP 674



3f ee = 91%

Informations sur l'acquisition

Date d'acquisition	21/03/2007 17:54:41 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	64,97 Minutes	Nb de points	38983
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		20,90	14499,90	96,03	0,00	0,00
2		41,46	599,48	3,97	0,00	0,00
SOMME			**15099,38**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

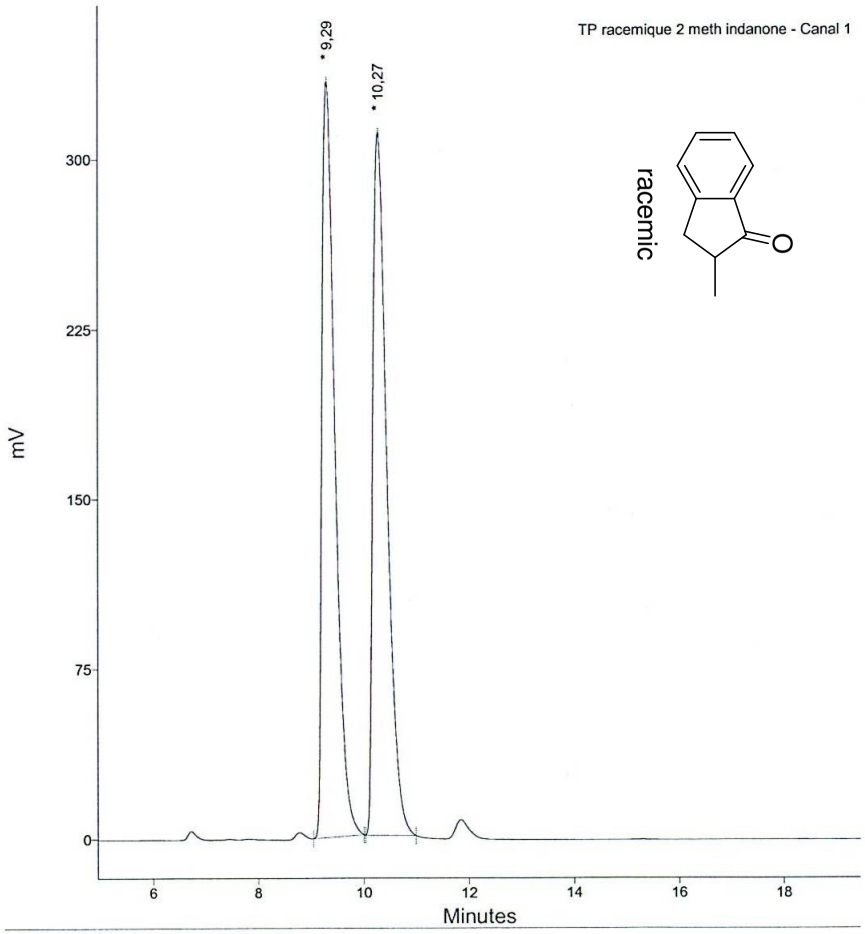
Nom	TP 674	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

OB Heptane / iPrOH 99/1 1ml/min 254nm 17°C

Analyse : TP racemique 2 meth indanone



Informations sur l'acquisition

Date d'acquisition	18/01/2007 18:08:24 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	30,00 Minutes	Nb de points	18001
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		9,29	5749,16	49,93	0,00	0,00
2		10,27	5765,35	50,07	0,00	0,00
SOMME			**11514,51**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	TP racemique 2 meth indanone	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

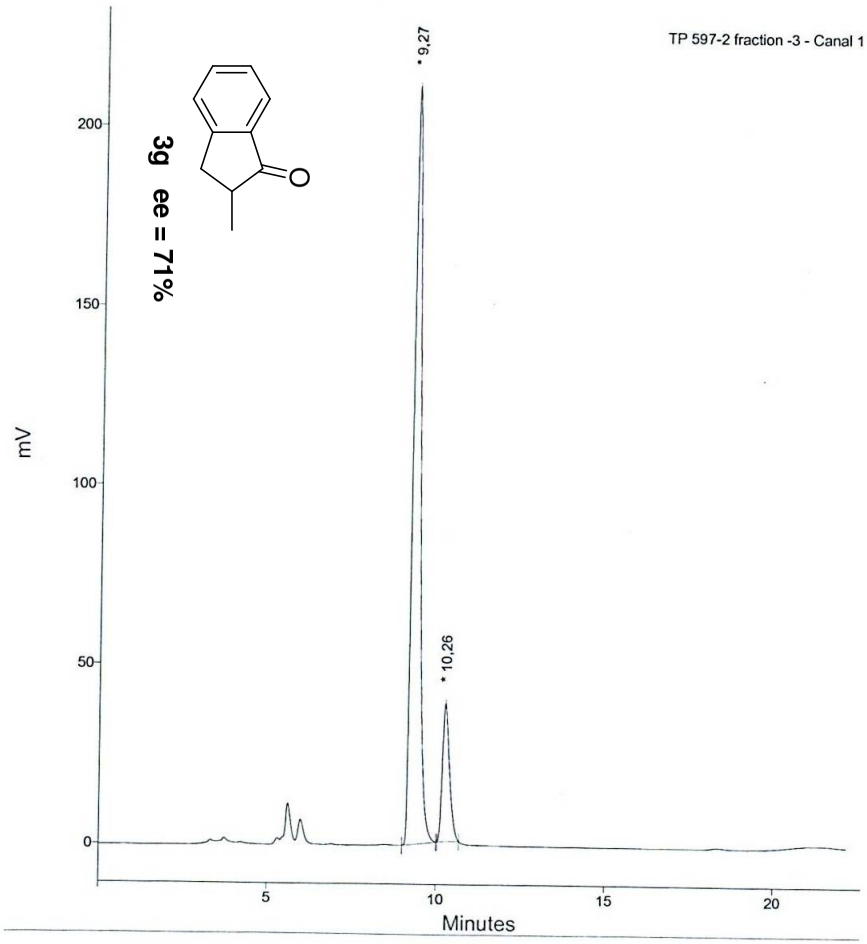
(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 99/1 1ml/min @254nm

2-Methyl-1-indanone 3q :

Analyse : TP 597-2 fraction -3



Informations sur l'acquisition

Date d'acquisition	18/01/2007 17:40:32 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	22,18 Minutes	Nb de points	13310
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		9,27	3359,62	85,55	0,00	0,00
2		10,26	567,37	14,45	0,00	0,00
SOMME			**3926,98**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

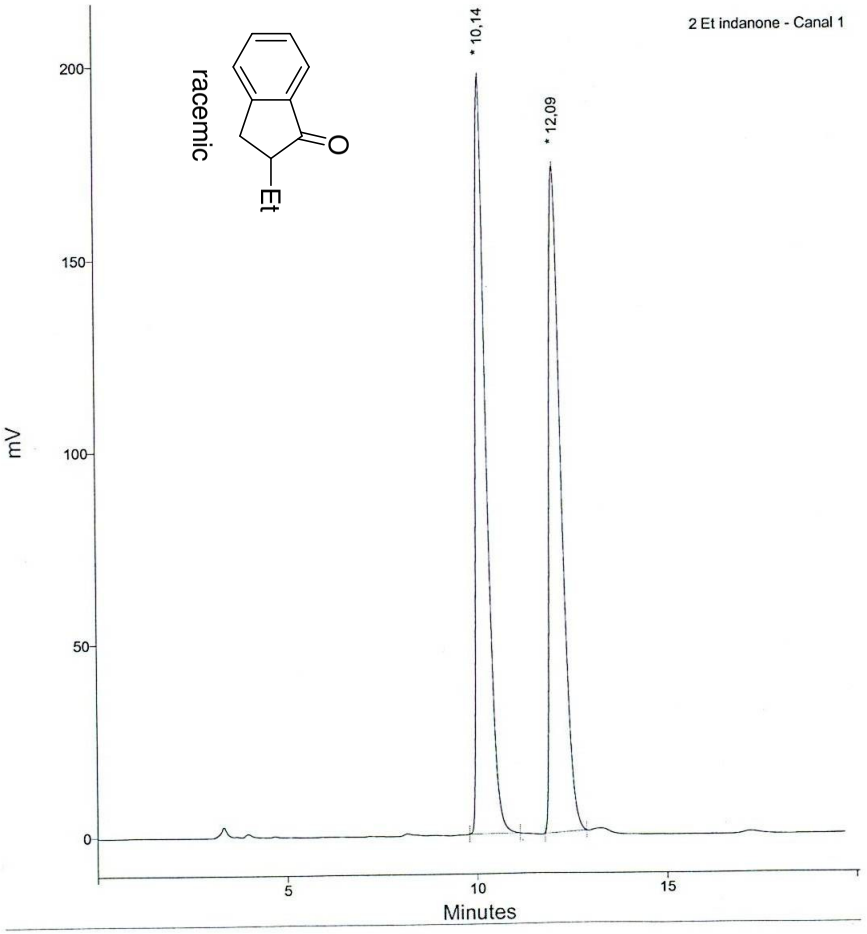
Nom	TP 597-2 fraction -3	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 99/1 1ml/min @254nm

Analyse : 2 Et indanone



Informations sur l'acquisition

Date d'acquisition	02/02/2007 10:11:45 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	19,68 Minutes	Nb de points	11809
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		10,14	3785,59	49,96	0,00	0,00
2		12,09	3792,17	50,04	0,00	0,00
SOMME			7577,76	100,00	0,00	0,00

Informations sur l'échantillon

Nom	2 Et indanone	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

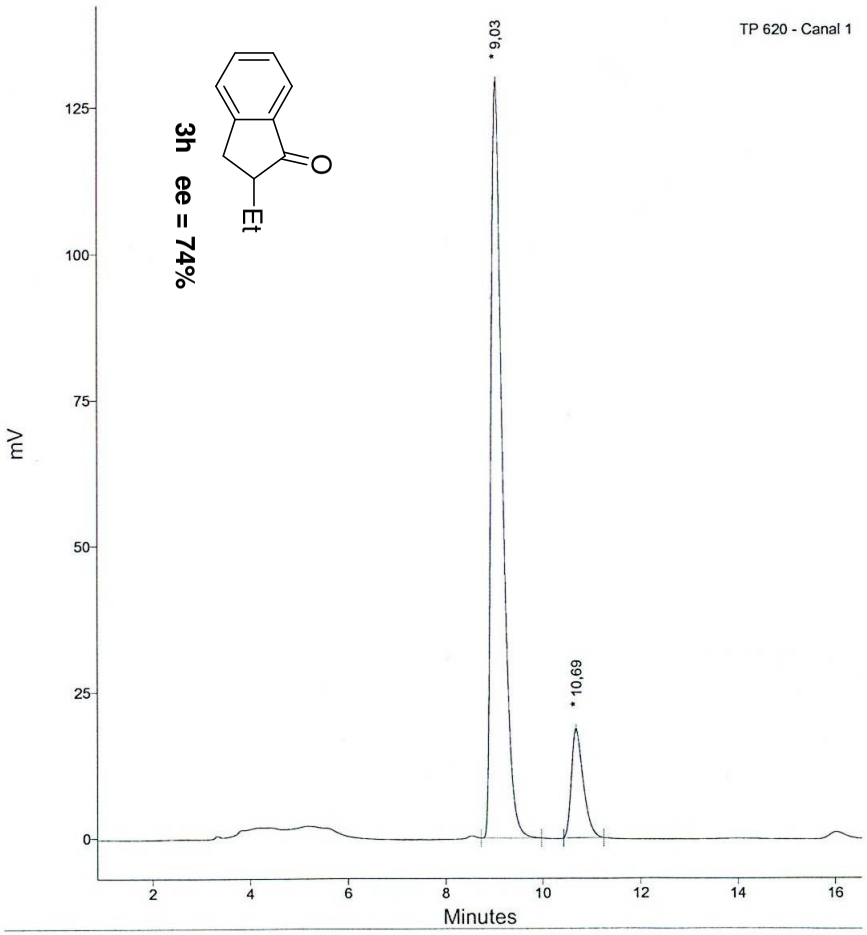
(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 99.6/0.4 1ml/min @254nm

2-Ethyl-1-indanone 3h :

Analyse : TP 620



Informations sur l'acquisition

Date d'acquisition	02/02/2007 13:25:34 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	22,75 Minutes	Nb de points	13651
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		9,03	2100,46	87,00	0,00	0,00
2		10,69	313,80	13,00	0,00	0,00
SOMME			**2414,26**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	TP 620	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

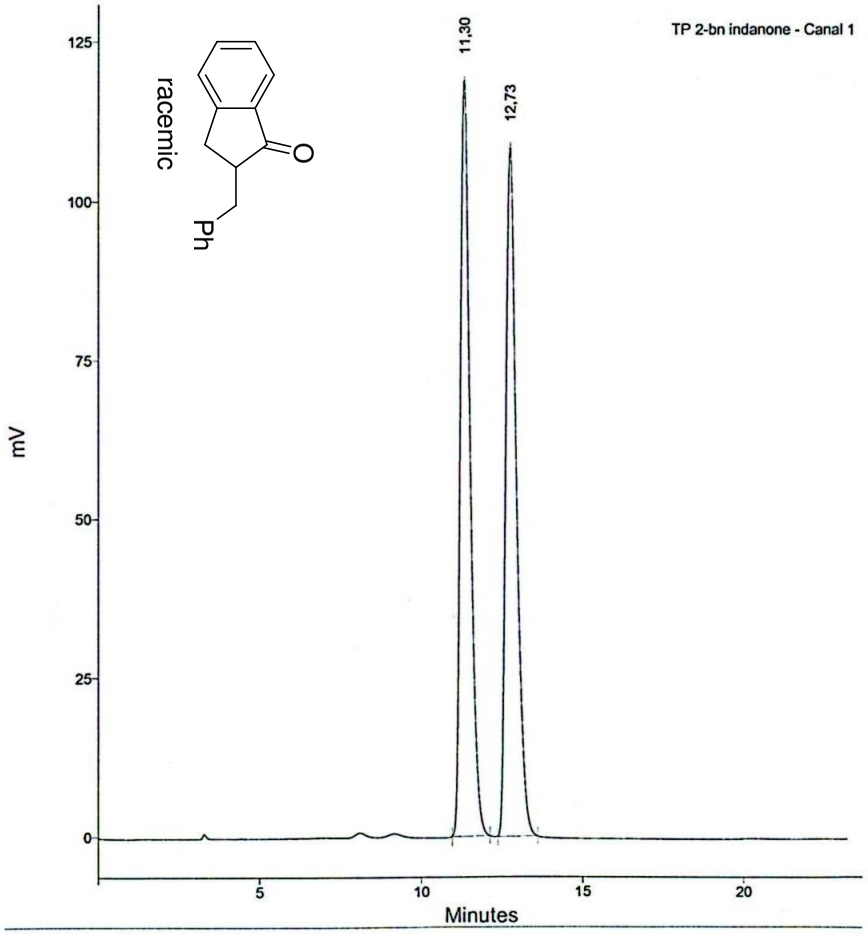
(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 99.6/0.4 1ml/min @254nm

2-benzyl-1-indanone 3i :

Analyse : TP 2-bn indanone



Informations sur l'acquisition

Date d'acquisition	13/02/2007 18:22:48 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	23,22 Minutes	Nb de points	13934
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		11,30	2511,54	49,99	0,00	0,00
2		12,73	2512,54	50,01	0,00	0,00
SOMME			5024,08	100,00	0,00	0,00

Informations sur l'échantillon

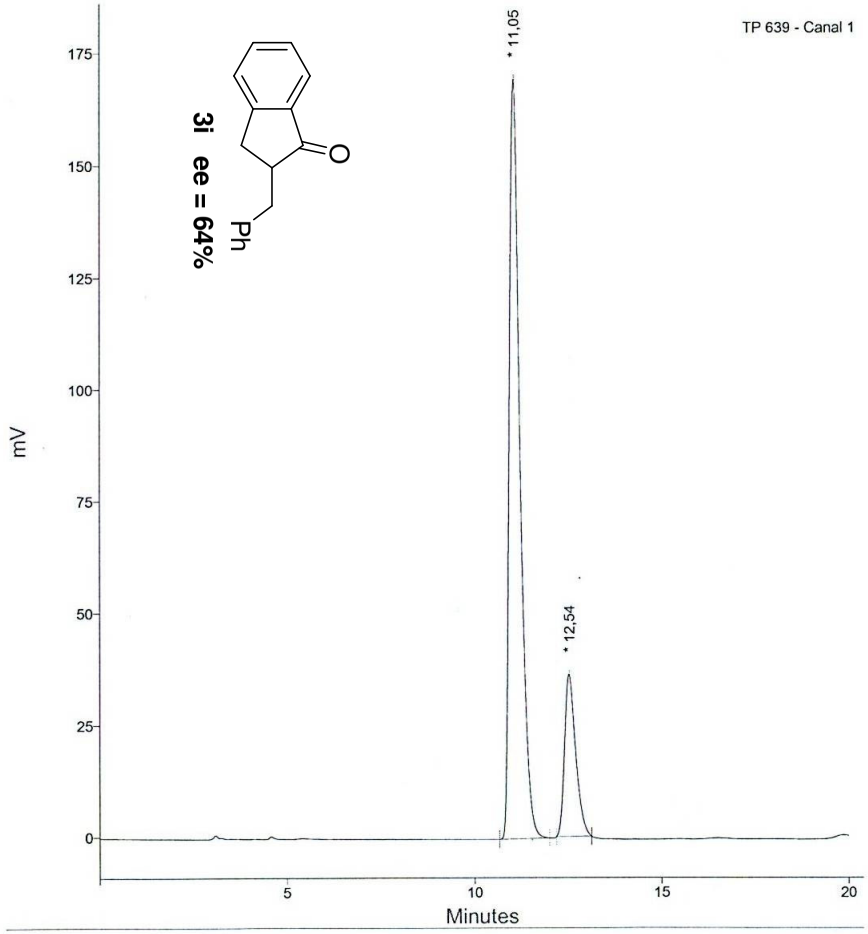
Nom	TP 2-bn indanone	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

(* = valeur initiale modifiée)

Informations :

OJ-H heptane/IPA 90/10 1ml/min @254nm @22°C

Analyse : TP 639



3i ee = 64%

Informations sur l'acquisition

Date d'acquisition	16/02/2007 13:35:37 (+01:00) (Azur 4.6.0.0)		
Source de l'acquisition :			
	INT7Channel 1		
Durée	20,00 Minutes	Nb de points	12001
Vitesse d'acquisition	10,00 points/seconde		

Résultats d'intégration

#	Nom du pic	Tr.	Aire	% Aire	Résultats	% Résultats
1		11,05	3404,31	81,90	0,00	0,00
2		12,54	752,14	18,10	0,00	0,00
SOMME			**4156,45**	**100,00**	**0,00**	**0,00**

Informations sur l'échantillon

Nom	TP 639	Type d'échantillon	Echantillon
N° Flacon	1		
Quantité	1,000000 mg	Volume d'injection	20,00 µl
Dilution	1	Diviseur	1

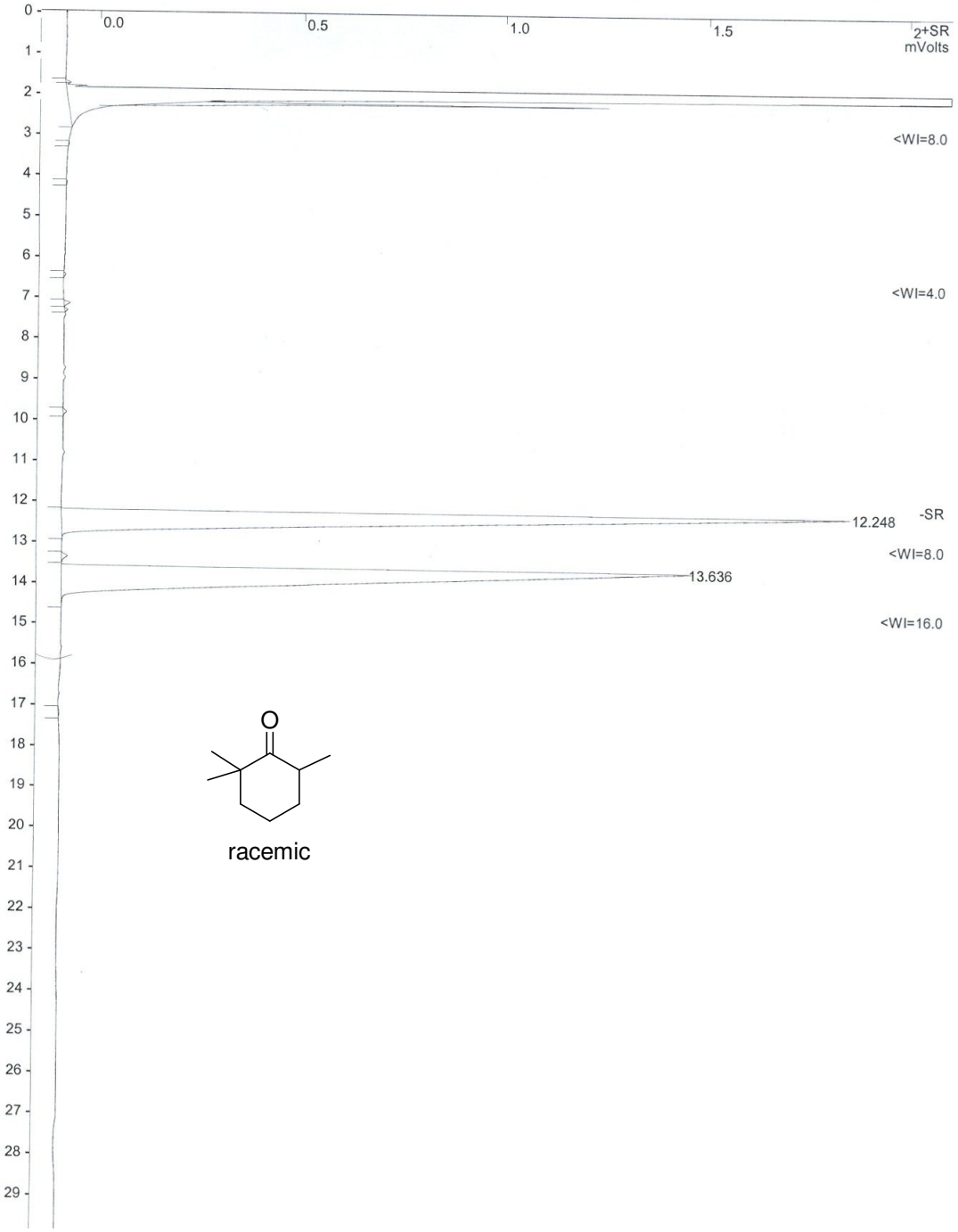
(* = valeur initiale modifiée)

Informations :

OJ-H Heptane / iPrOH 90.10 1ml/min 254nm

2,2,6-Trimethylcyclohexanone 3j :

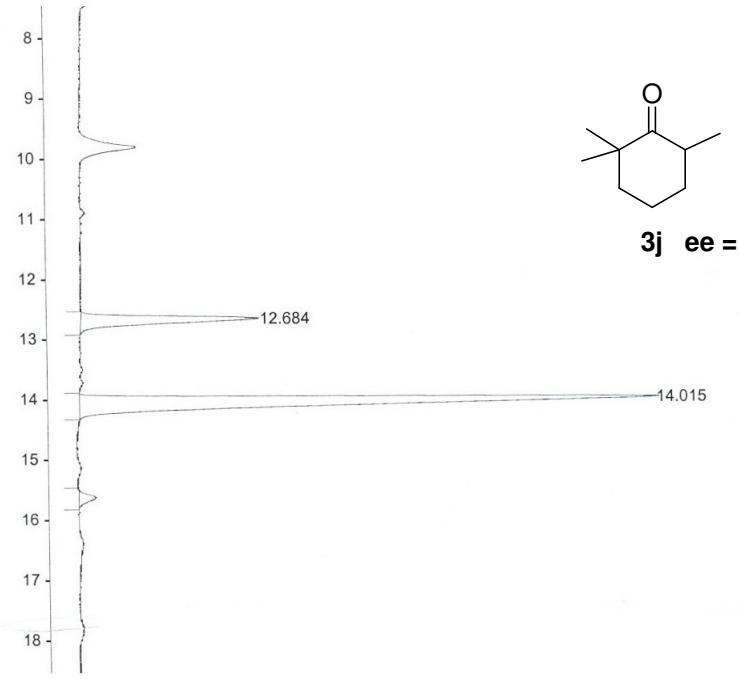
```
Title       :
Run File    : d:\poisson thomas\cyclo 90.run
Method File : D:\Methode GC 3900\METHODES sur 39XL1\MethodLB39XL1 iso90.mth
Sample ID   : cyclo 90

Injection Date: 05/02/2007 15:42     Calculation Date: 05/02/2007 16:12

Operator    :                        Detector Type: 39XL (10 Volts)
Workstation: Donnees                 Bus Address  : 44
Instrument : Varian 3900 (1)         Sample Rate  : 20.00 Hz
Channel    : Front = FID             Run Time     : 29.997 min

** Star Chromatography Workstation Version 5.52 ** 00155-5b08-e52-23b9 **

Chart Speed =    0.72 cm/min   Attenuation = 1            Zero Offset = 6%
Start Time  =    0.000   min    End Time    = 29.997  min   Min / Tick  = 1.00
```



racemic

```
Print Date: Mon Feb 05 16:26:17 2007            Page 1 of 1

Title       :
Run File    : d:\poisson thomas\cyclo 90.run
Method File : D:\Methode GC 3900\METHODES sur 39XL1\MethodLB39XL1 iso90.mth
Sample ID   : cyclo 90

Injection Date: 05/02/2007 15:42    Calculation Date: 05/02/2007 16:12

Operator    :                       Detector Type: 39XL (10 Volts)
Workstation: Donnees                Bus Address  : 44
Instrument : Varian 3900 (1)        Sample Rate  : 20.00 Hz
Channel    : Front = FID            Run Time     : 29.997 min

** Star Chromatography Workstation Version 5.52 ** 00155-5b08-e52-23b9 **

Run Mode        : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

                                 Ret.    Time                      Width
 Peak      Peak       Result     Time   Offset      Area    Sep.  1/2   Status
  No.      Name         ()      (min)   (min)     (counts) Code (sec)  Codes
 ---- ------------ ---------- ------- ------- ---------- ---- -----  ------
    1                 50.0713  12.248   0.000      31582   BB  15.8
    2                 49.9287  13.636   0.000      31492   VB  19.9
 ---- ------------ ========== ======= ======= ========== ---- -----  ------
      Totals:        100.0000           0.000      63074

Total Unidentified Counts :      63074 counts

Detected Peaks: 4           Rejected Peaks: 2      Identified Peaks: 0

Multiplier: 1          Divisor: 1          Unidentified Peak Factor: 0

Baseline Offset: -83 microVolts

Noise (used): 2 microVolts - monitored before this run

Manual injection

*******************************************************************************
```

```
Title       :
Run File    : d:\poisson thomas\tp 631.run
Method File : d:\methode gc 3900\methodes sur 39xl1\methodlb39xl1 iso90.mth
Sample ID   : TP 631

Injection Date: 13/02/2007 10:58    Calculation Date: 13/02/2007 11:21

Operator    :                       Detector Type: 39XL (10 Volts)
Workstation: Donnees                Bus Address  : 44
Instrument : Varian 3900 (1)        Sample Rate  : 20.00 Hz
Channel    : Front = FID            Run Time     : 22.747 min

** Star Chromatography Workstation Version 5.52 ** 00155-5b08-e52-23b9 **

Chart Speed =    0.95 cm/min   Attenuation = 1          Zero Offset = 4%
Start Time  =    0.000   min    End Time    = 22.747  min   Min / Tick  = 1.00
```



3j ee = 58%

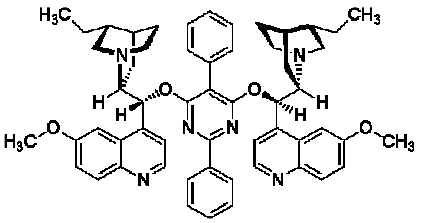
-II
+FP
-FP
+FP
-FP
+II

```
Print Date: Tue Feb 13 11:22:24 2007            Page 1 of 1

Title       :
Run File    : d:\poisson thomas\tp 631.run
Method File : d:\methode gc 3900\methodes sur 39xl1\methodlb39xl1 iso90.mth
Sample ID   : TP 631

Injection Date: 13/02/2007 10:58    Calculation Date: 13/02/2007 11:21

Operator    :                       Detector Type: 39XL (10 Volts)
Workstation: Donnees                Bus Address  : 44
Instrument : Varian 3900 (1)        Sample Rate  : 20.00 Hz
Channel    : Front = FID            Run Time     : 22.747 min

** Star Chromatography Workstation Version 5.52 ** 00155-5b08-e52-23b9 **

Run Mode        : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent
```

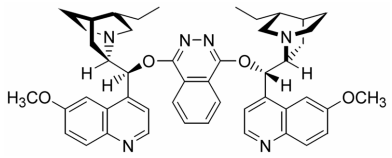
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		20.9281	12.684	0.000	856	BB	7.3	
2		79.0719	14.015	0.000	3234	BB	8.5	
	Totals:	100.0000		0.000	4090			

```
Total Unidentified Counts :        4090 counts

Detected Peaks: 3            Rejected Peaks: 1       Identified Peaks: 0

Multiplier: 1           Divisor: 1           Unidentified Peak Factor: 0

Baseline Offset: -69 microVolts

Noise (used): 3 microVolts - monitored before this run

Manual injection

********************************************************************************
```

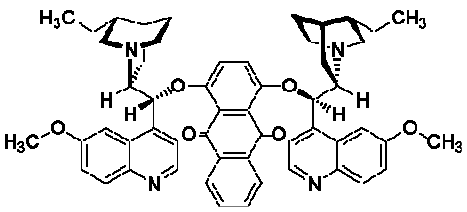
6-Chemical formula of catalysts 1d-f



1d : $(DHQ)_2Pyr$



1e: $(DHQD)_2PHAL$



1f: $(DHQ)_2AQN$