Supporting Information for:

Structural Optimization of Enantiopure 2-Cyclialkylamino-2-aryl-1,1-diphenylethanols as Catalytic Ligands for Enantioselective Additions to Aldehydes

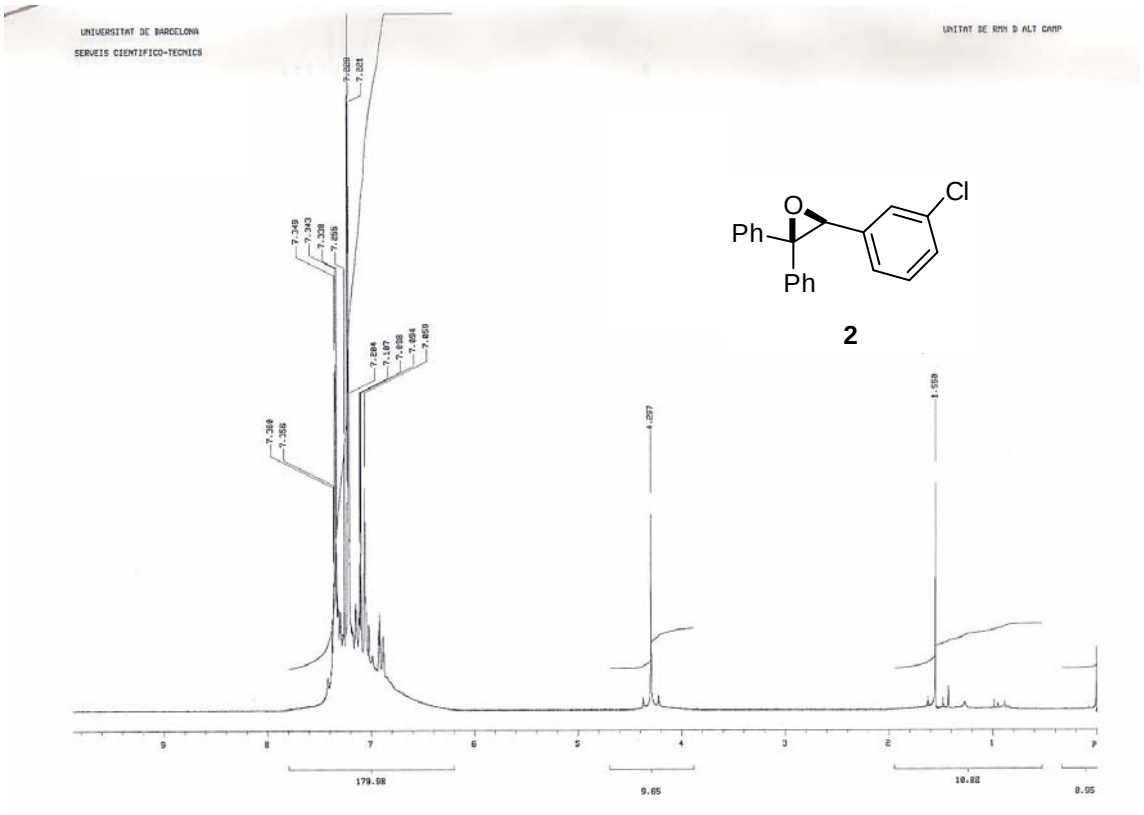
*Sergi Rodríguez-Escrich, Katamreddy Subba Reddy, Ciril Jimeno, Gisela Colet, Carles Rodríguez-Escrich, Lluís Solà, Anton Vidal-Ferran, and Miquel A. Pericàs**

Institute of Chemical Research of Catalonia (ICIQ). Av. Països Catalans 16, E-43007 Tarragona (Spain), Departament de Química Orgànica, Universitat de Barcelona. E-08028 Barcelona (Spain), and ICREA

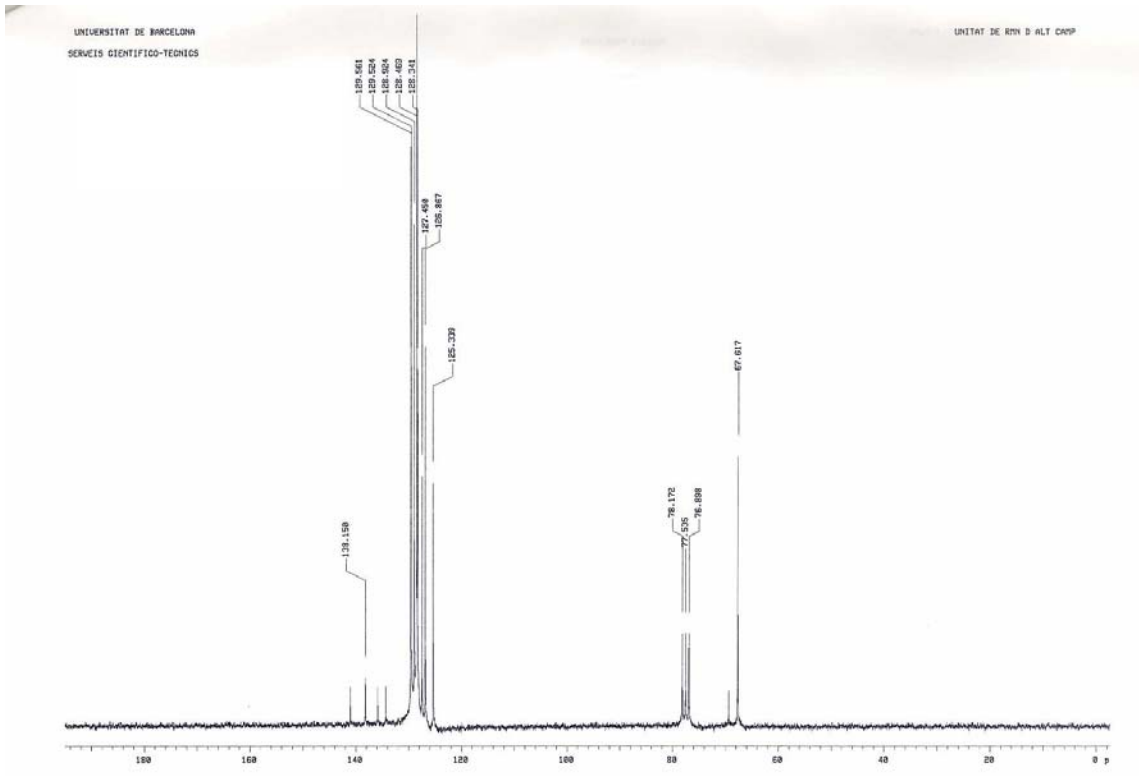
E-mail: mapericas@iciq.es

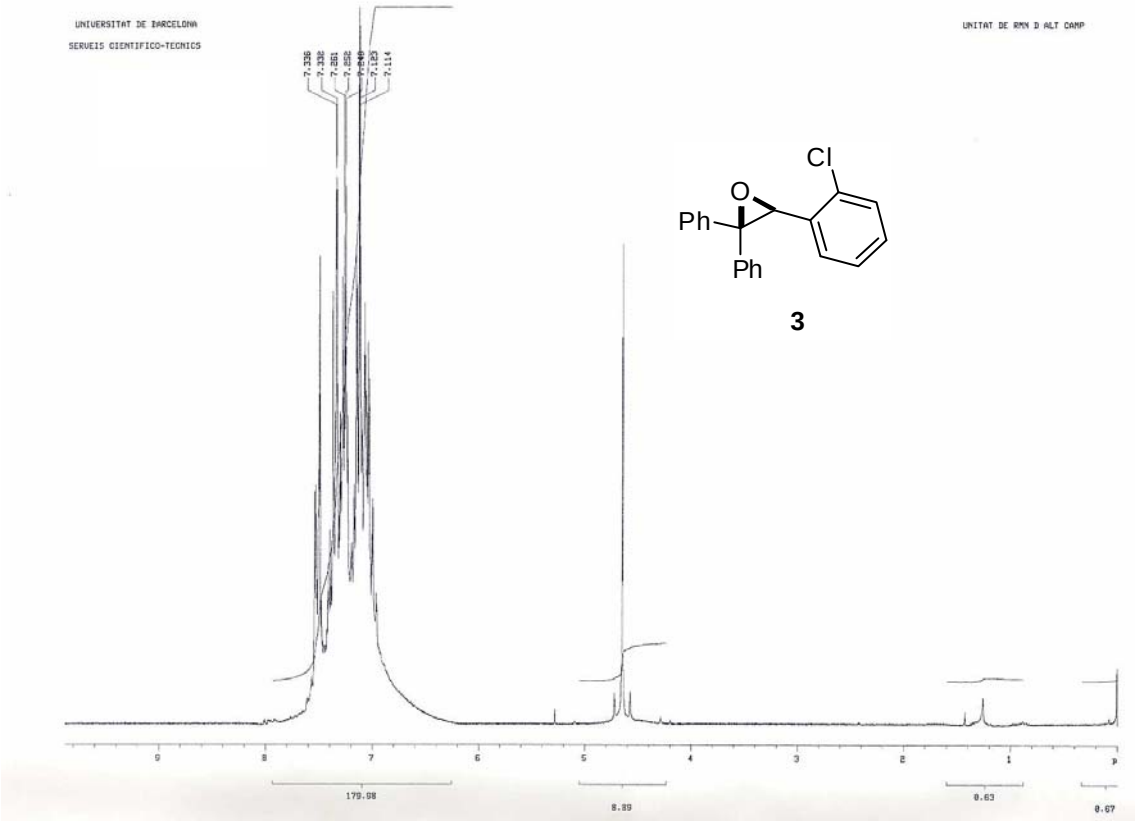
Copies of ^{1}H and ^{13}C NMR spectra for the following products:

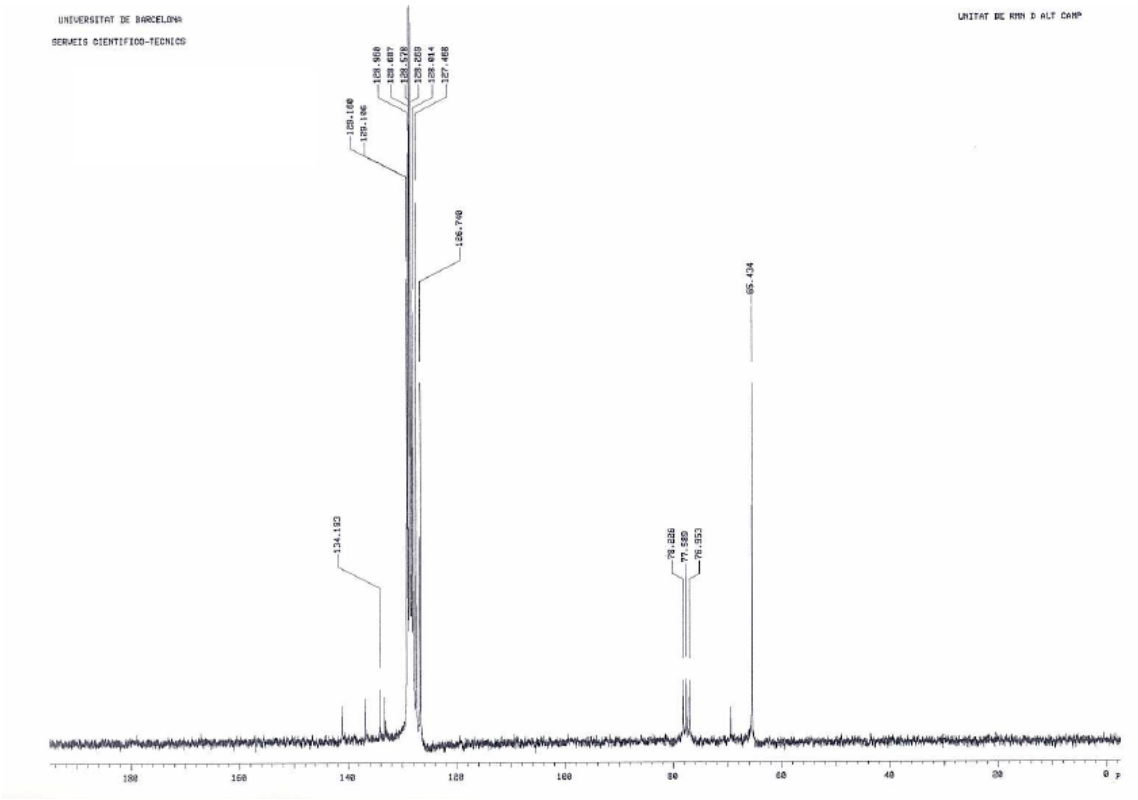
ANNEX: Synthetic details for the preparation of the starting olefins:

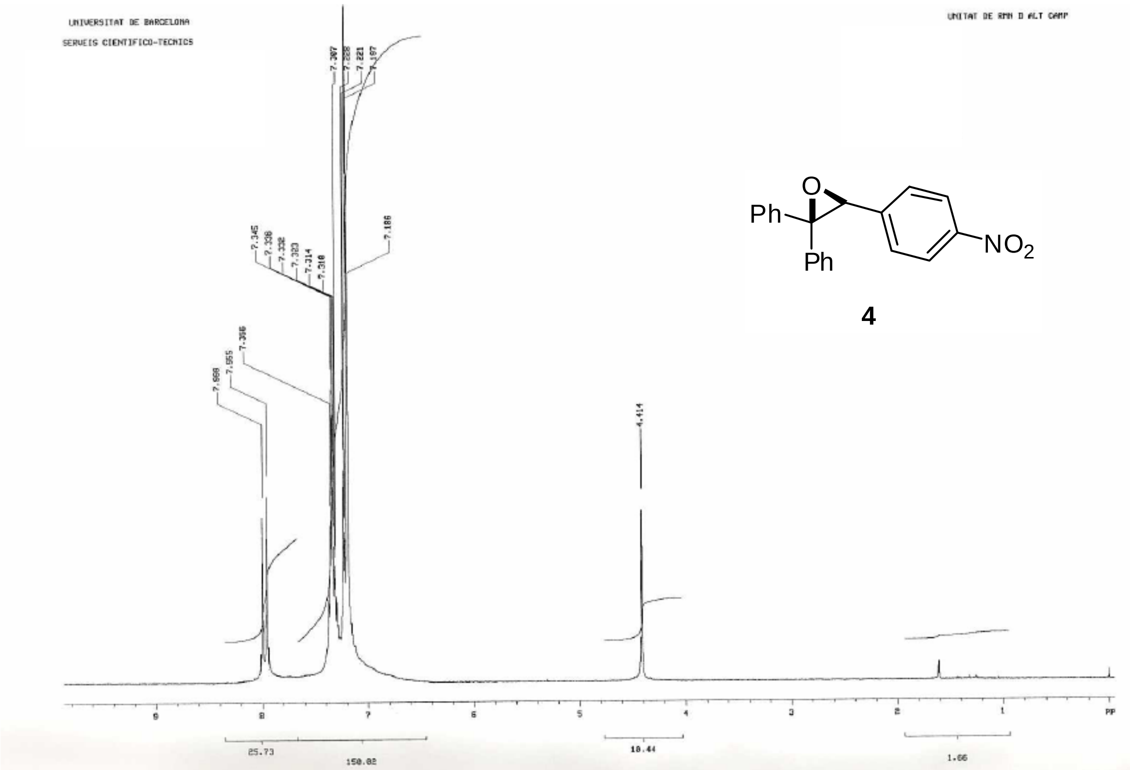

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
Ph
Ph
O
Cl
179.98
9.65
10.82
0.95

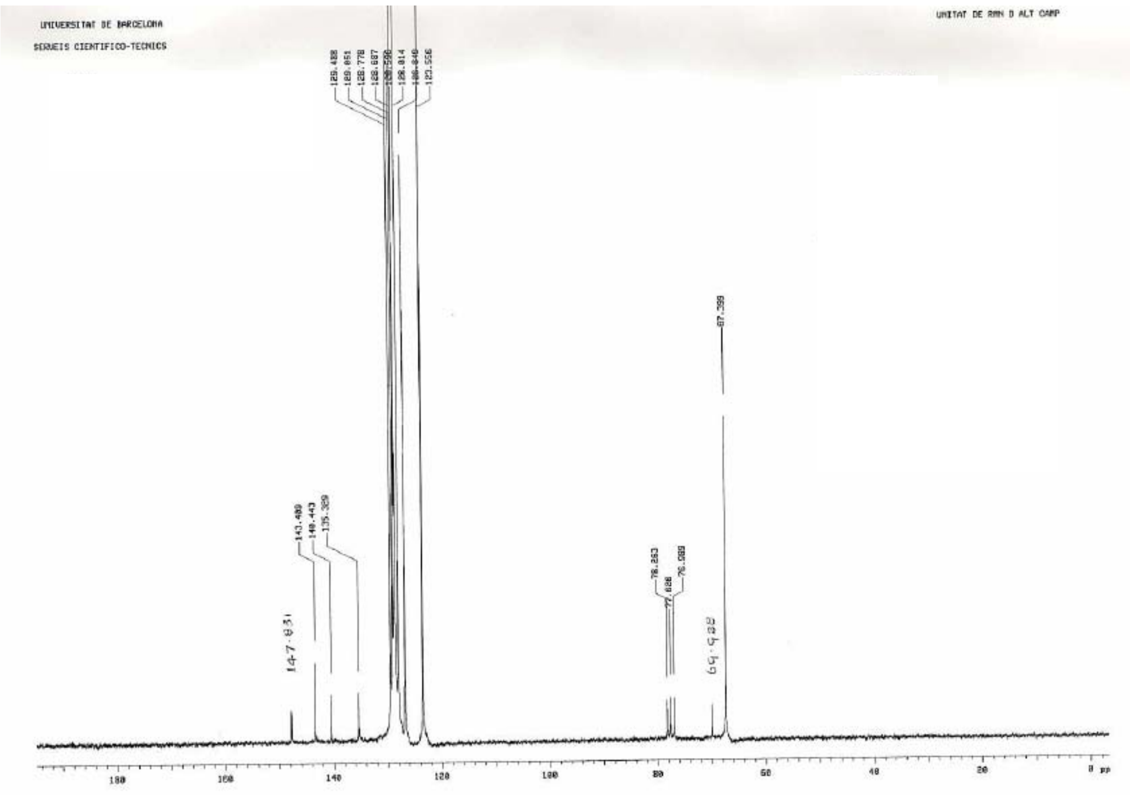
2

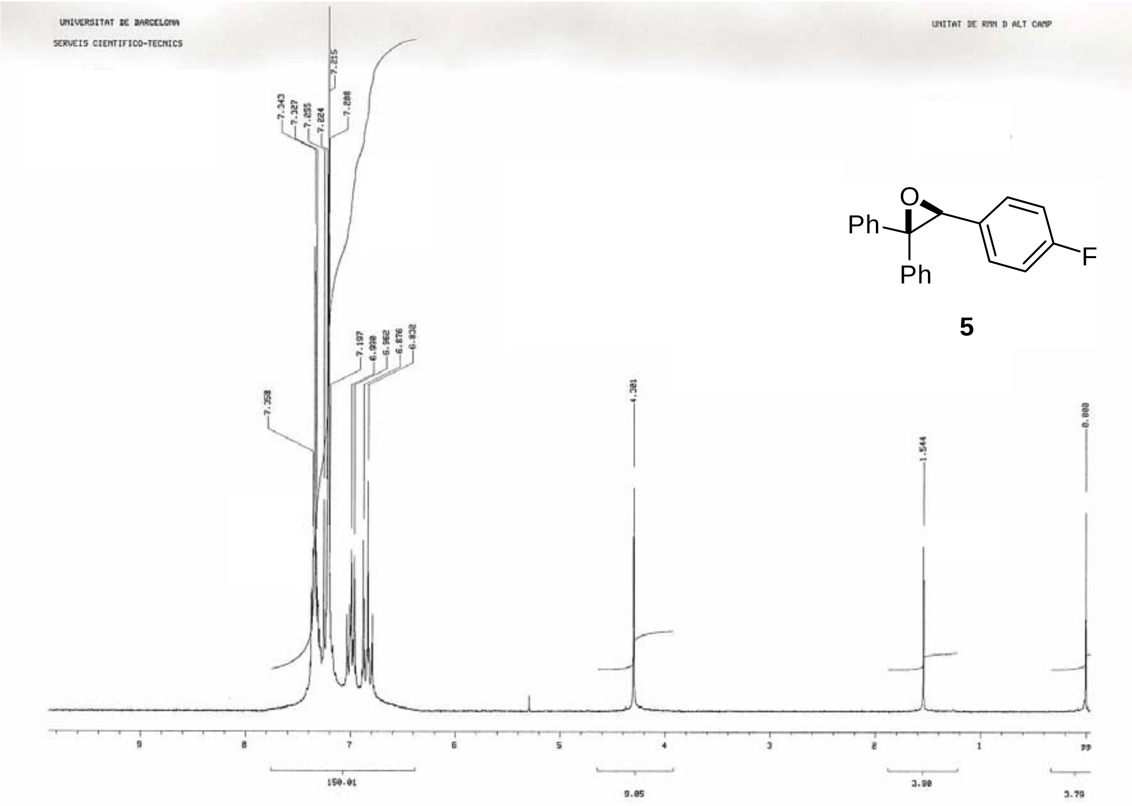

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP

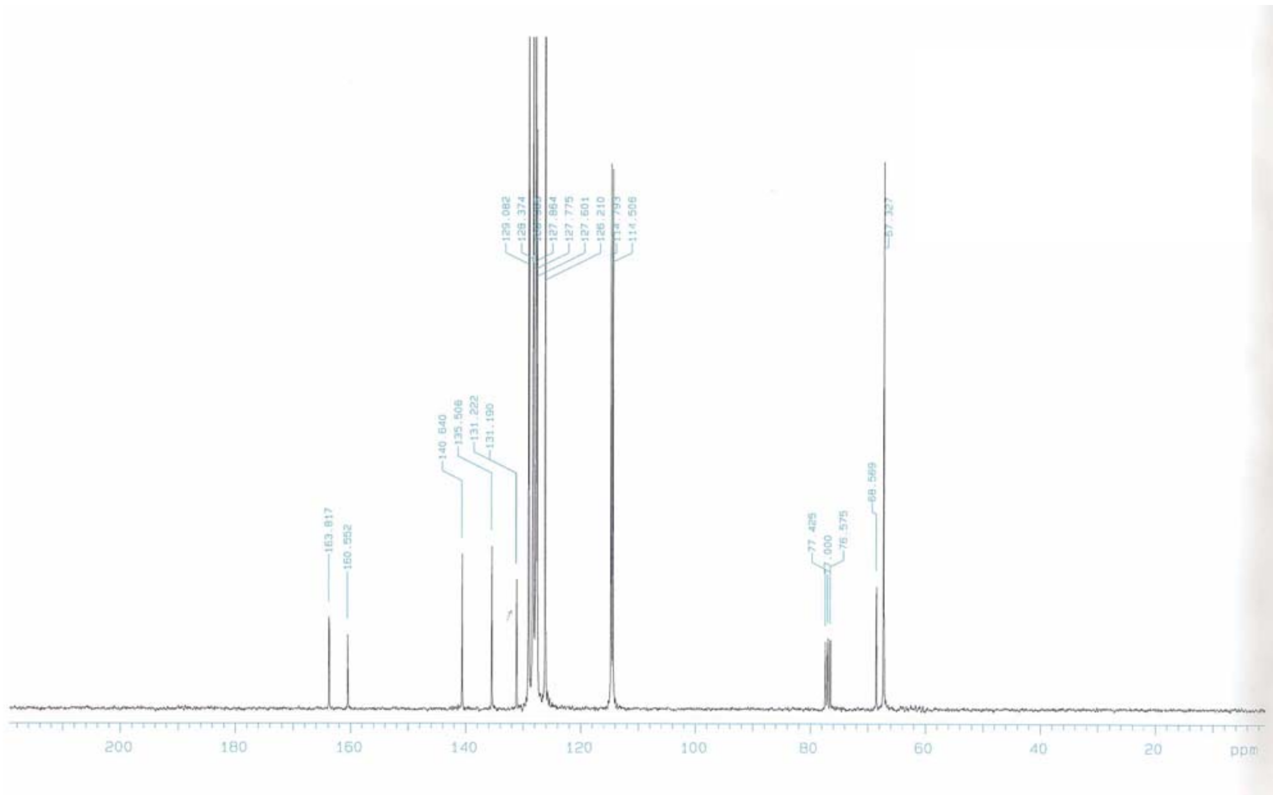

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
7.336
7.332
7.301
7.252
7.240
7.123
7.114
Cl
O
Ph
Ph
3
179.98
8.89
0.63
0.67

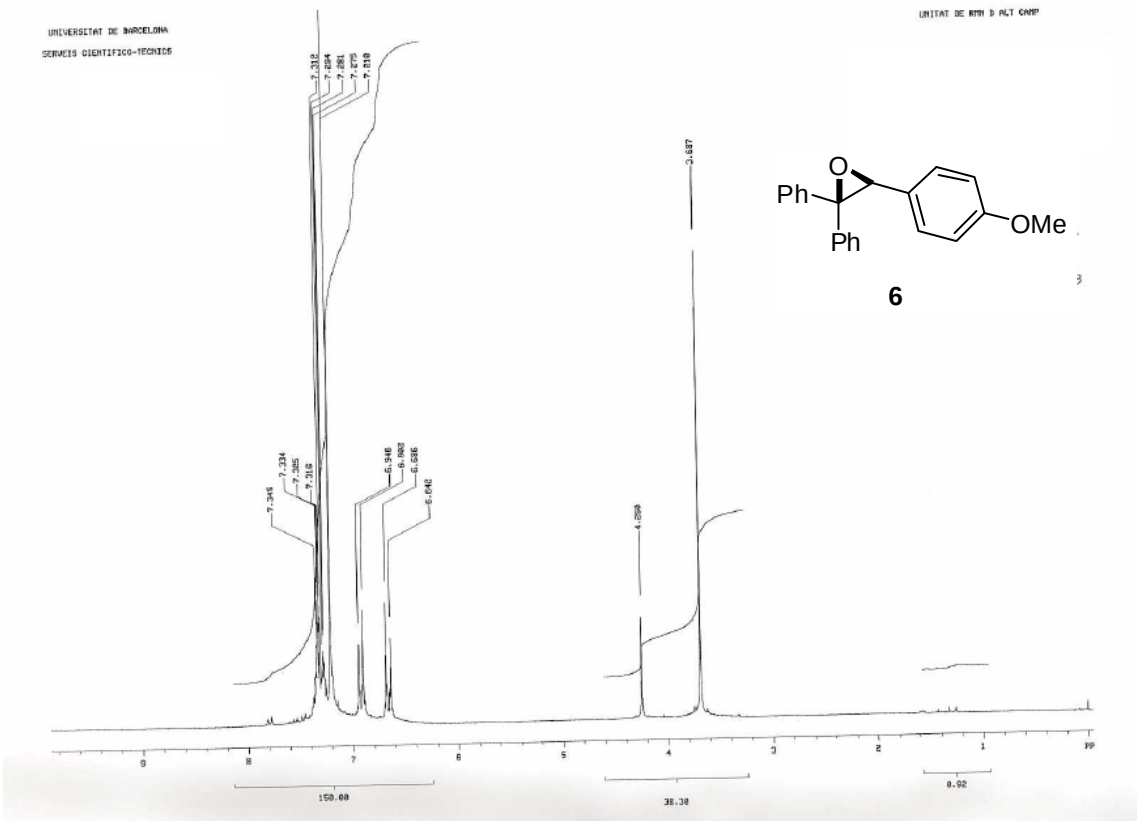

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
128.960
128.697
128.578
128.269
128.014
127.468
135.100
126.186
126.740
65.434
134.193
78.286
77.580
76.953


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
Ph
O
Ph
NO_2
4

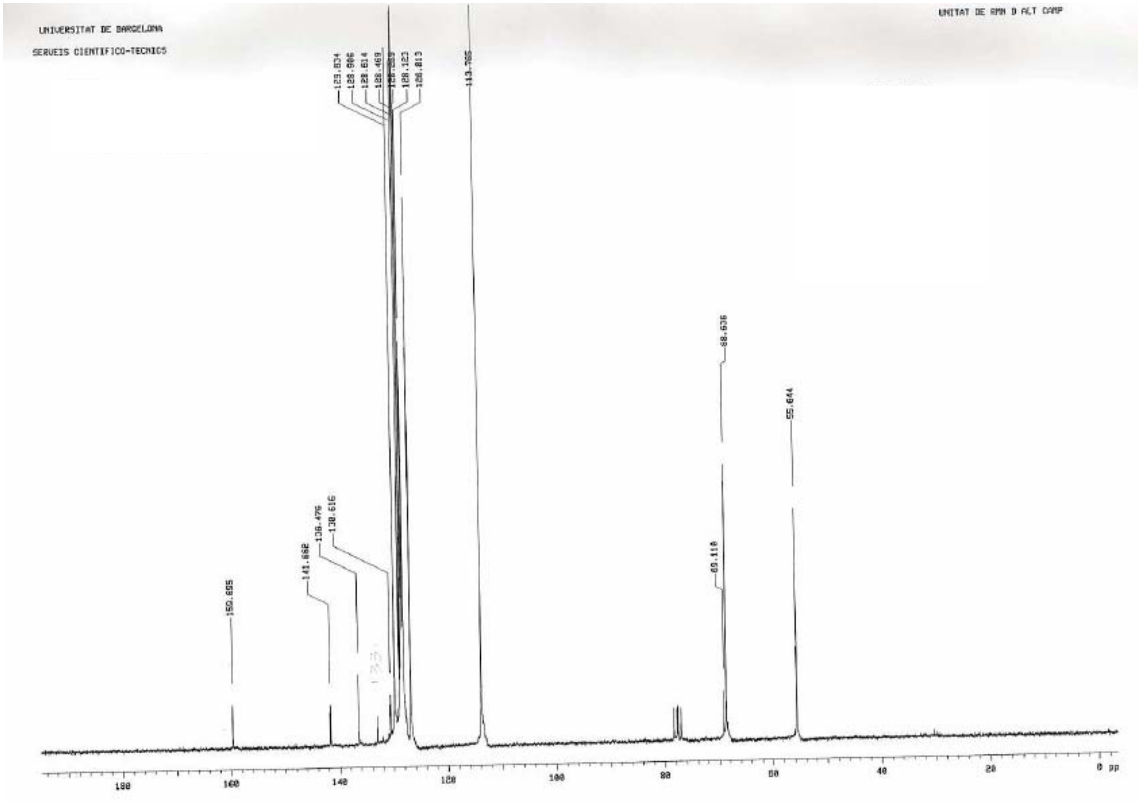

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
147.831
69.928

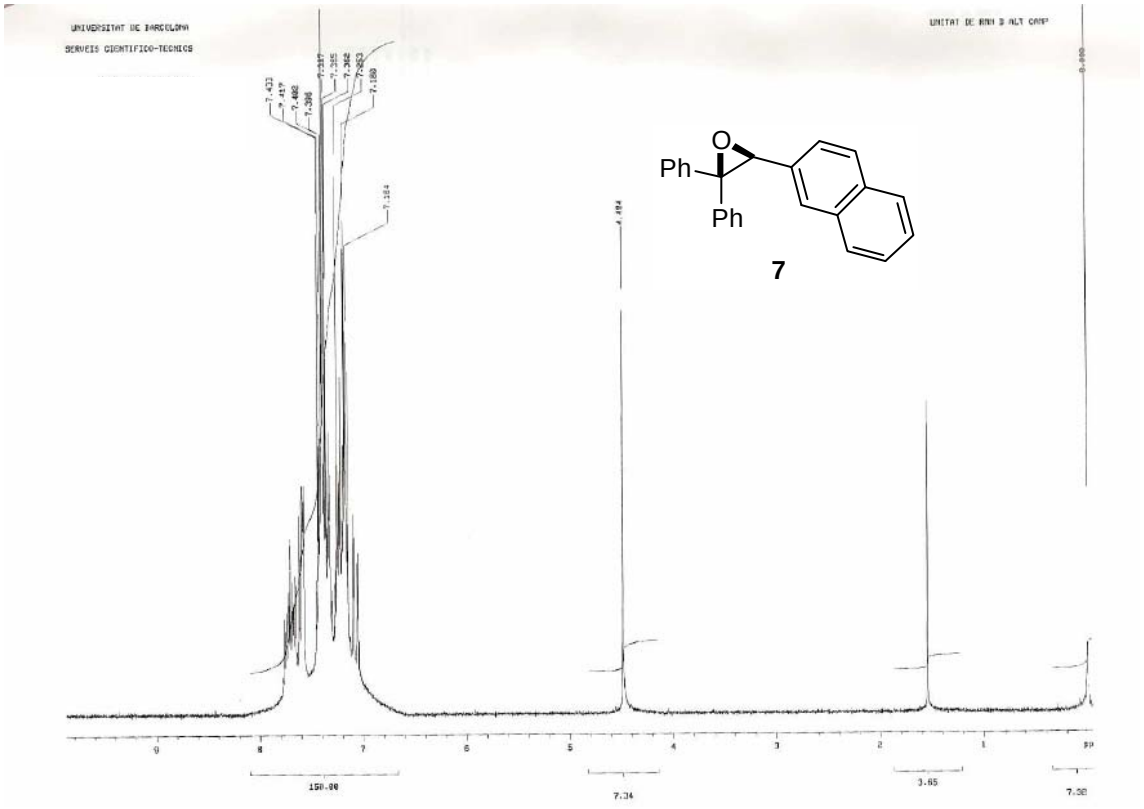

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
Ph
O
Ph
F
5
7.343
7.327
7.255
7.224
7.215
7.208
7.358
7.197
6.990
6.962
6.876
6.832
4.301
1.544
0.000
150.01
9.05
3.90
3.79

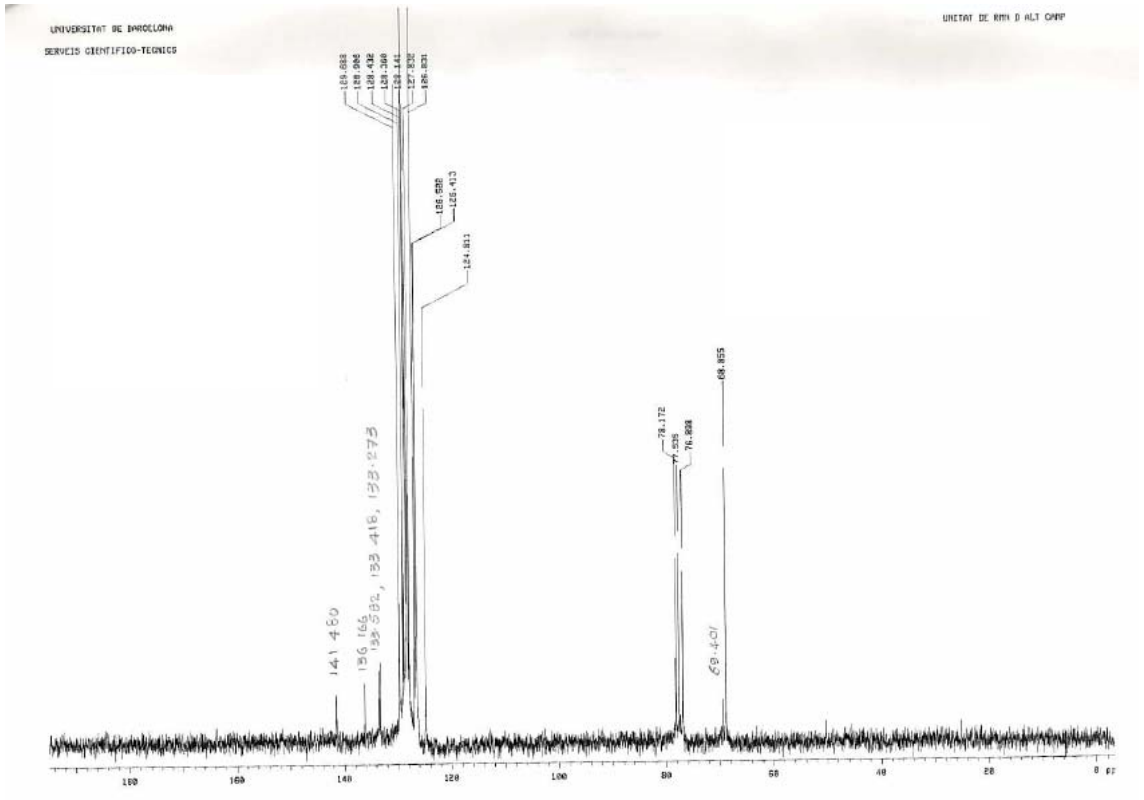

129.082
128.374
127.864
127.775
127.601
126.210
114.793
114.506
67.327
140.640
135.508
131.222
131.190
163.617
160.552
77.425
77.000
76.575
68.569
200
180
160
140
120
100
80
60
40
20
ppm

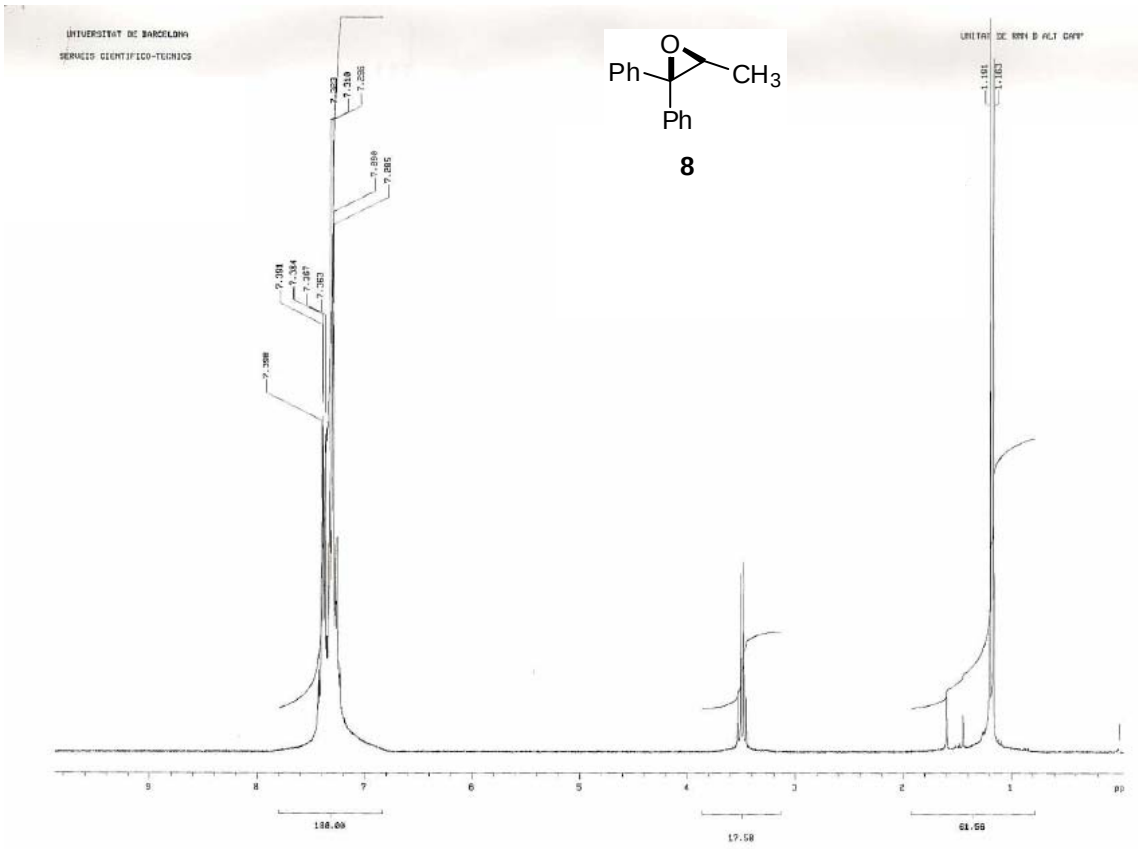

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
O
Ph
Ph
OMe

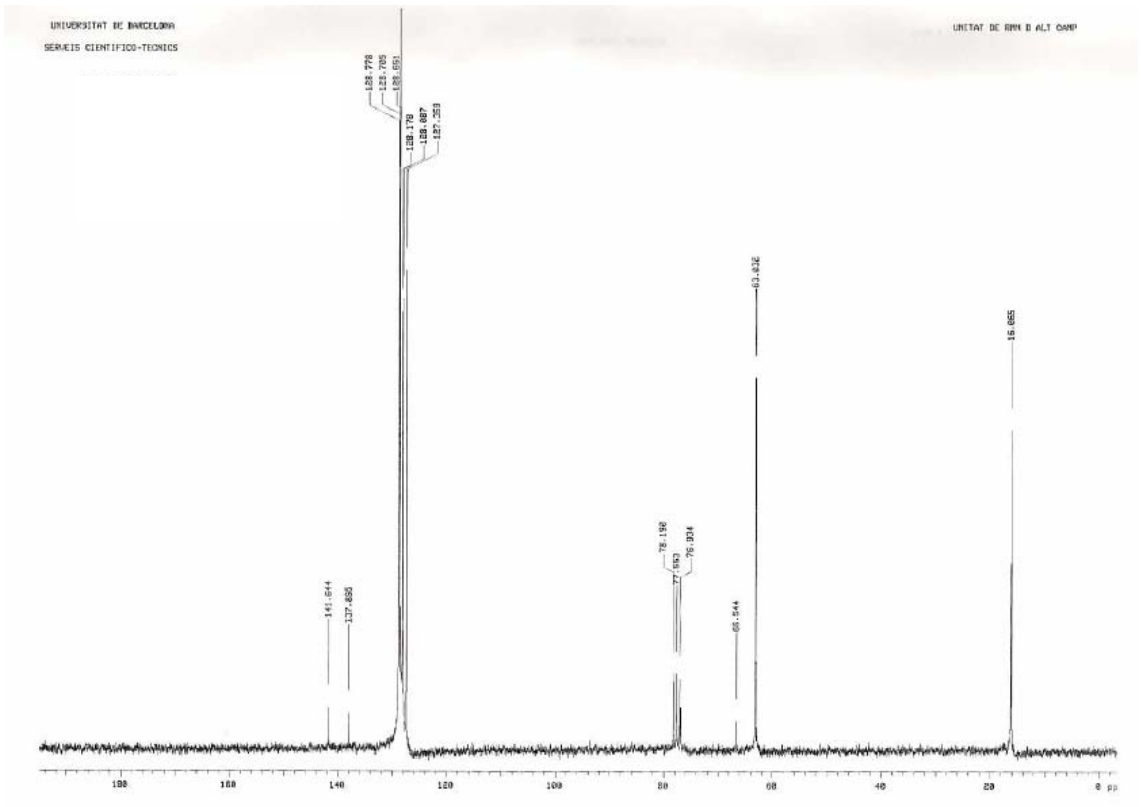
6

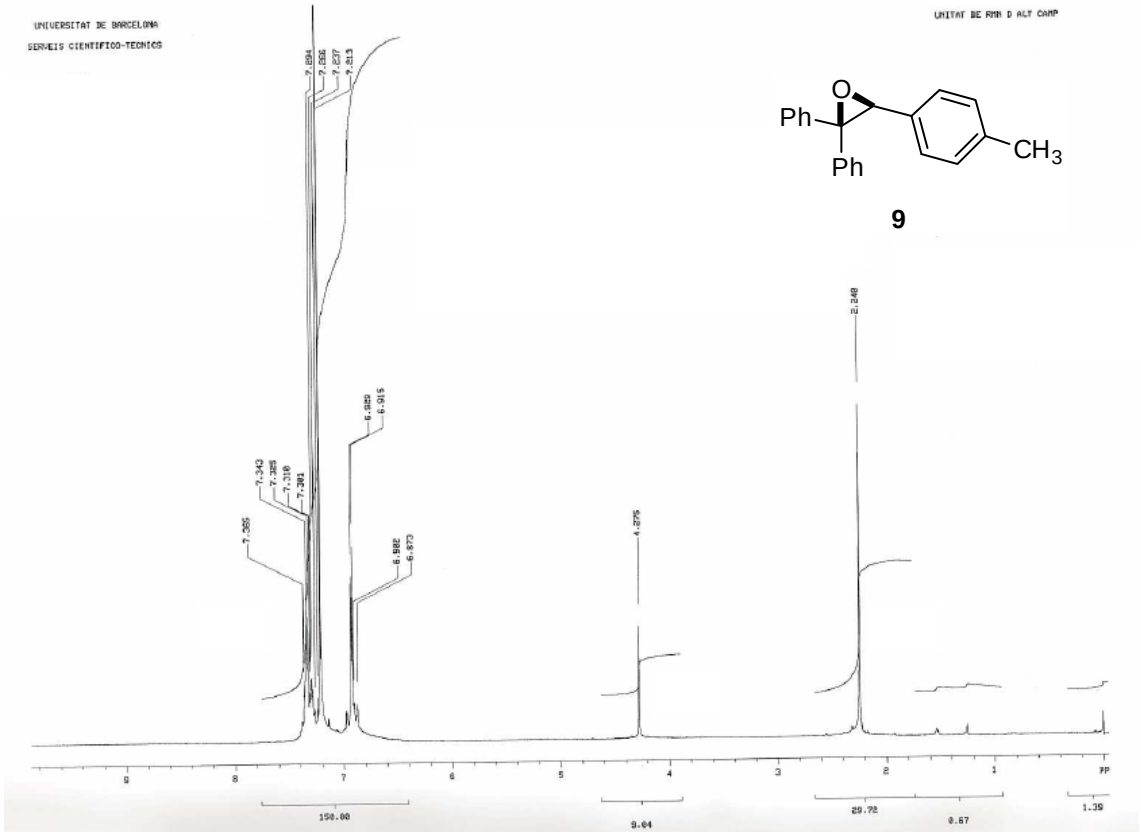

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
O
Ph
Ph
7
150.00
7.34
3.65
7.30

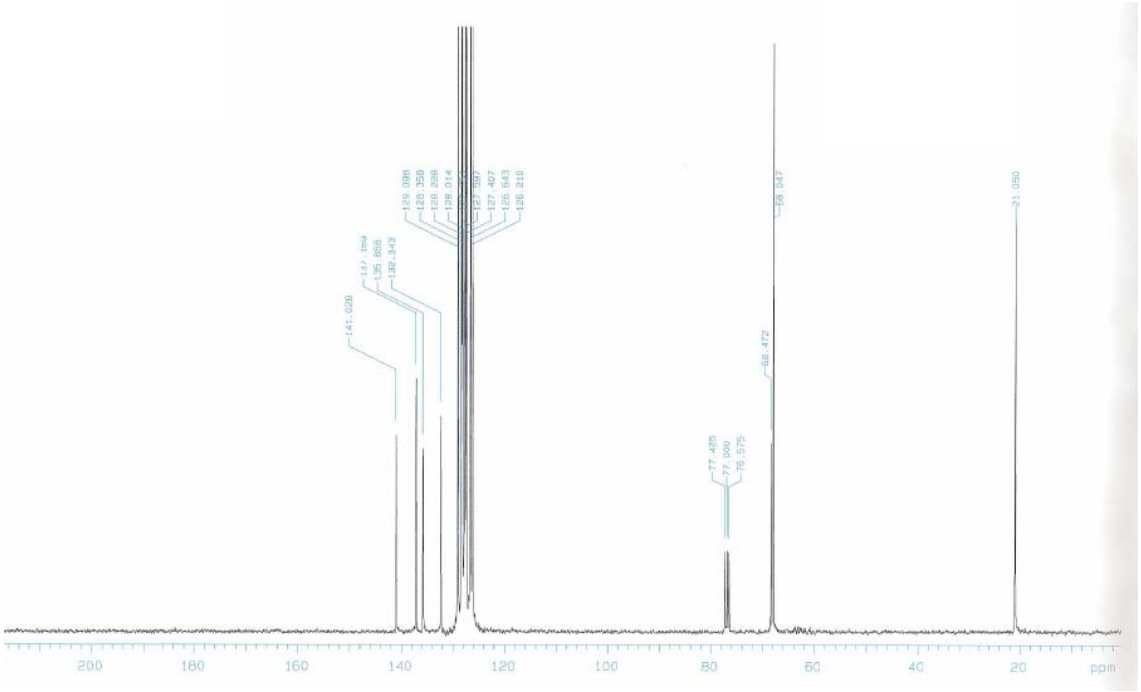

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
141.460
136.165
133.582, 133.416, 133.273
69.401

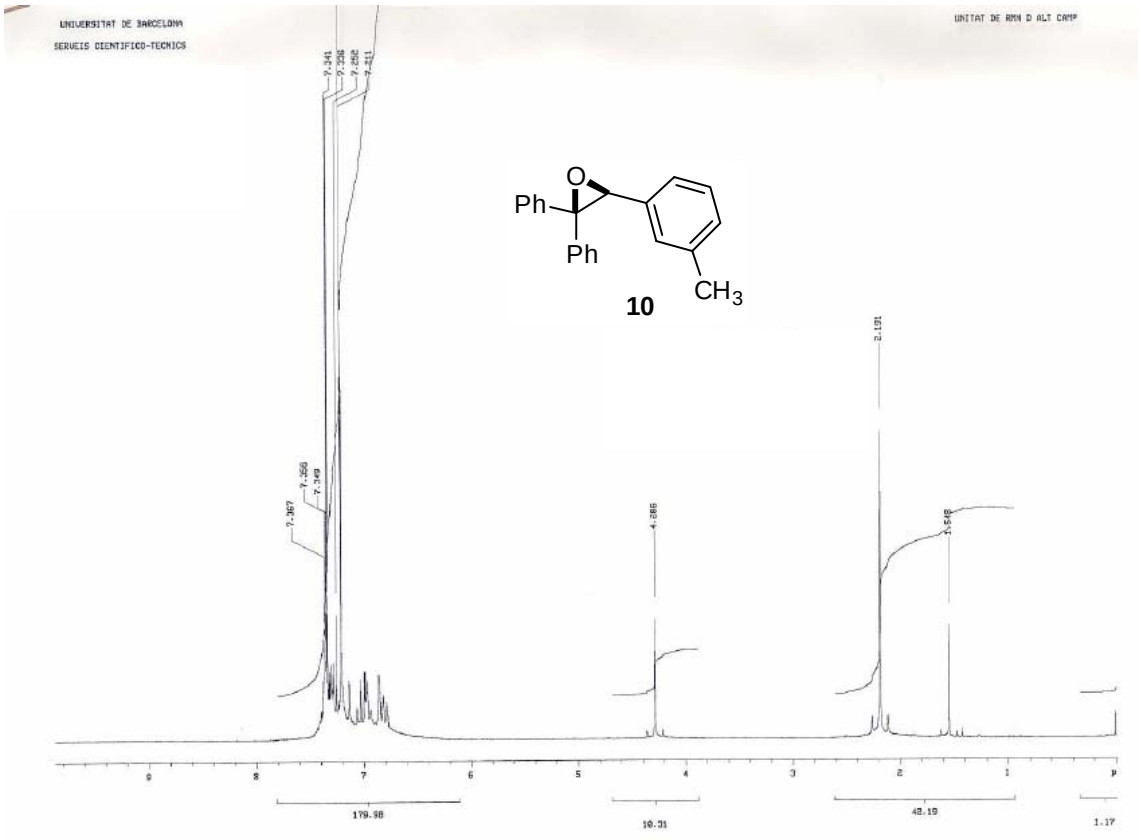

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
Ph
CH3
Ph
8
7.323
7.319
7.286
7.286
7.286
7.284
7.367
7.363
7.308
1.181
1.163
9
8
7
6
5
4
3
2
1
ppm
138.00
17.58
61.66

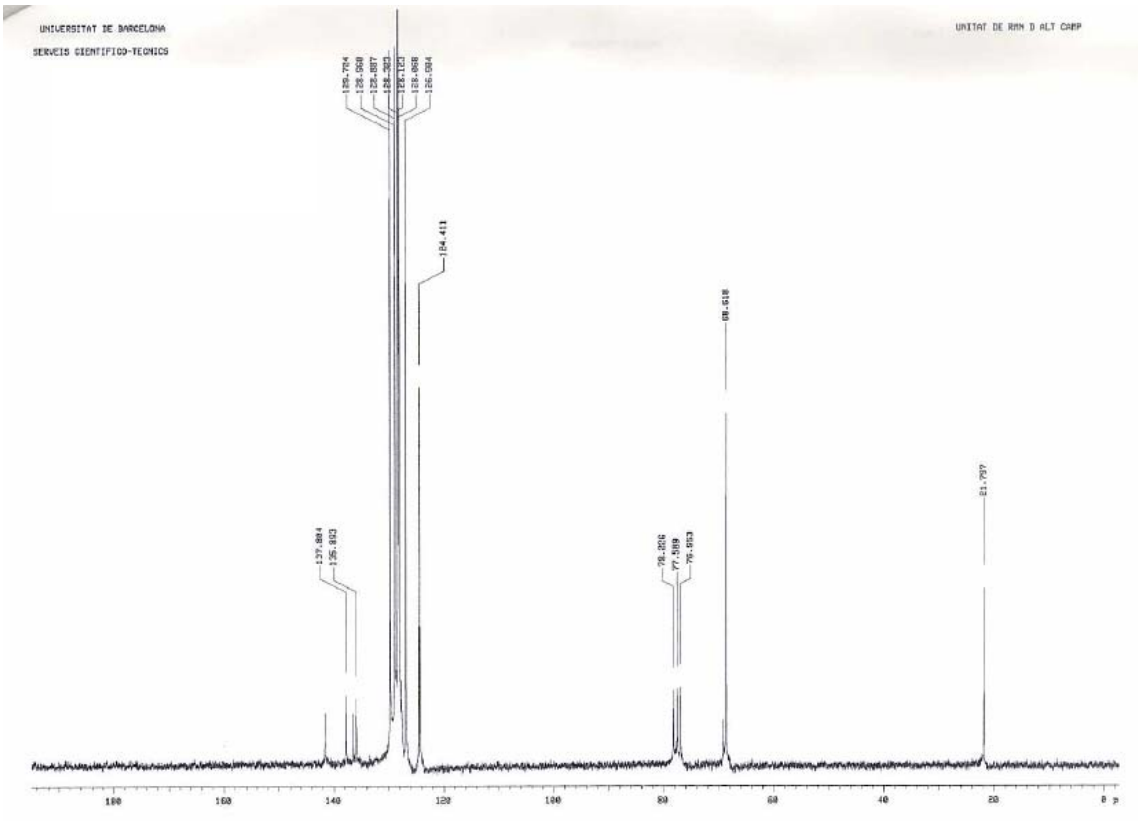

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
138.778
138.705
138.651
128.179
128.087
127.359
141.644
137.635
78.198
77.993
76.934
65.544
63.432
15.685
200
180
160
140
120
100
80
60
40
20
0 ppm

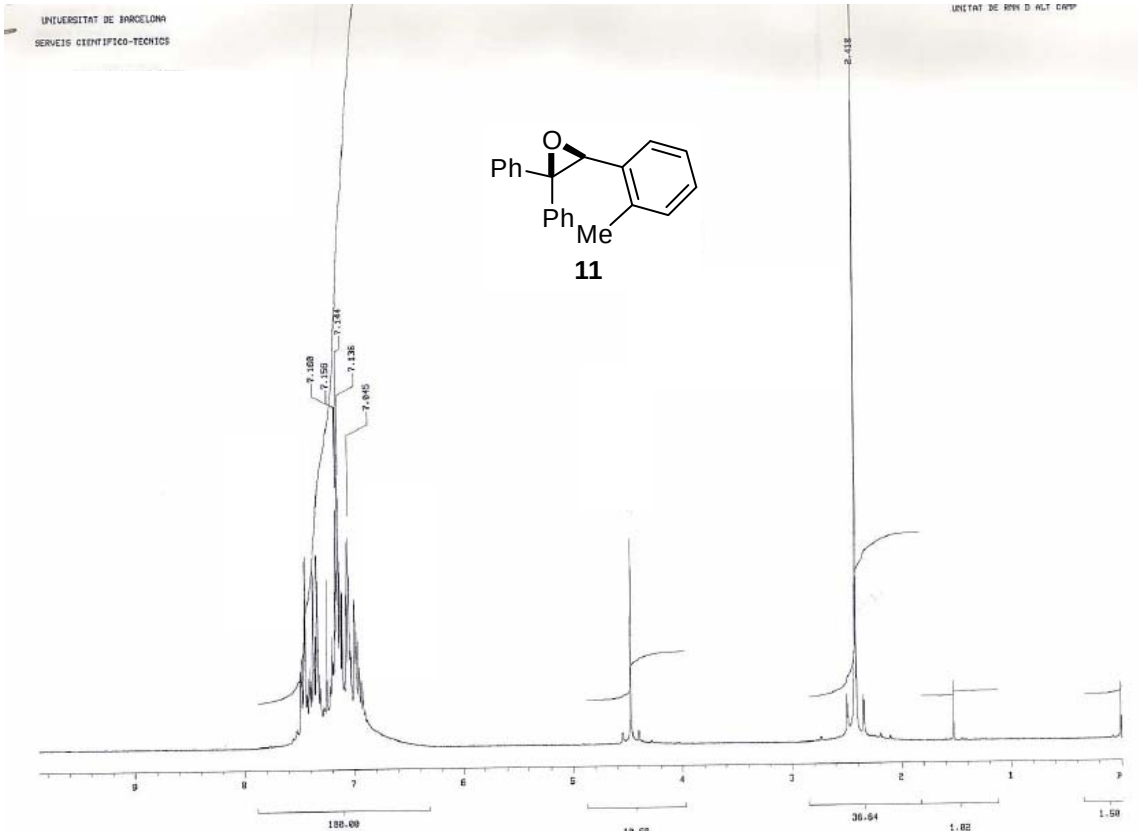

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
O
Ph
Ph
CH3
7.684
7.656
7.637
7.613
7.365
7.343
7.328
7.310
7.301
6.929
6.915
6.888
6.873
4.276
2.249
150.00
9.64
29.72
0.67
1.39

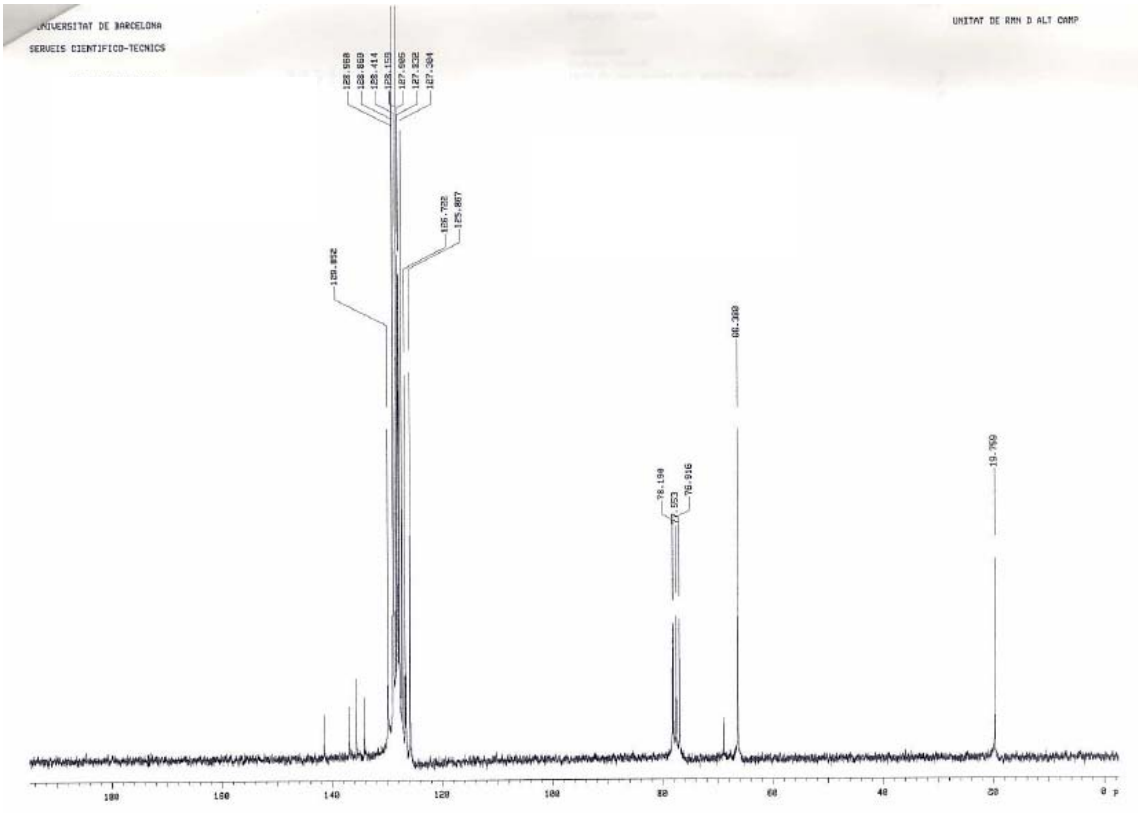
9

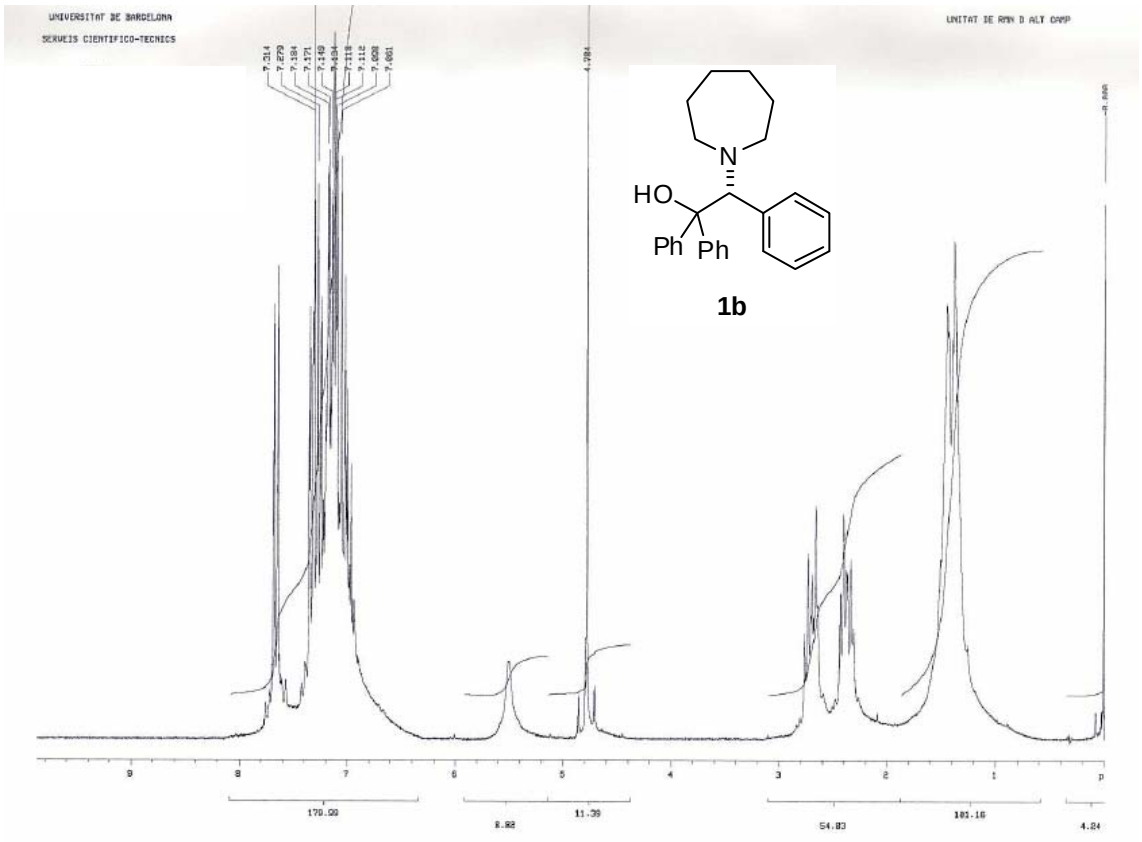

141.029
137.394
135.858
132.343
129.098
128.359
128.289
128.014
127.597
127.407
126.643
126.218
77.425
77.000
76.575
68.472
68.047
21.050
200
180
160
140
120
100
80
60
40
20
ppm

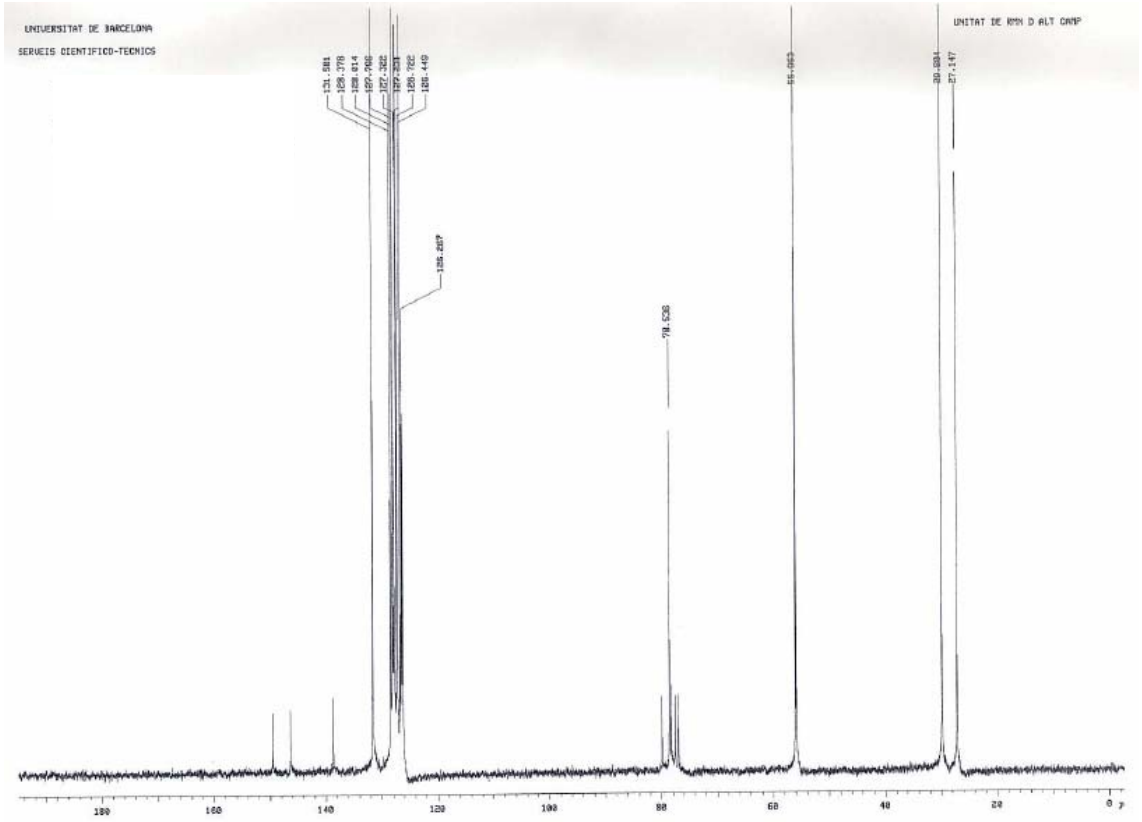

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
O
Ph
Ph
CH3
10
7.341
7.336
7.288
7.411
7.367
7.356
7.349
4.286
2.191
1.548
179.98
10.31
48.19
1.17

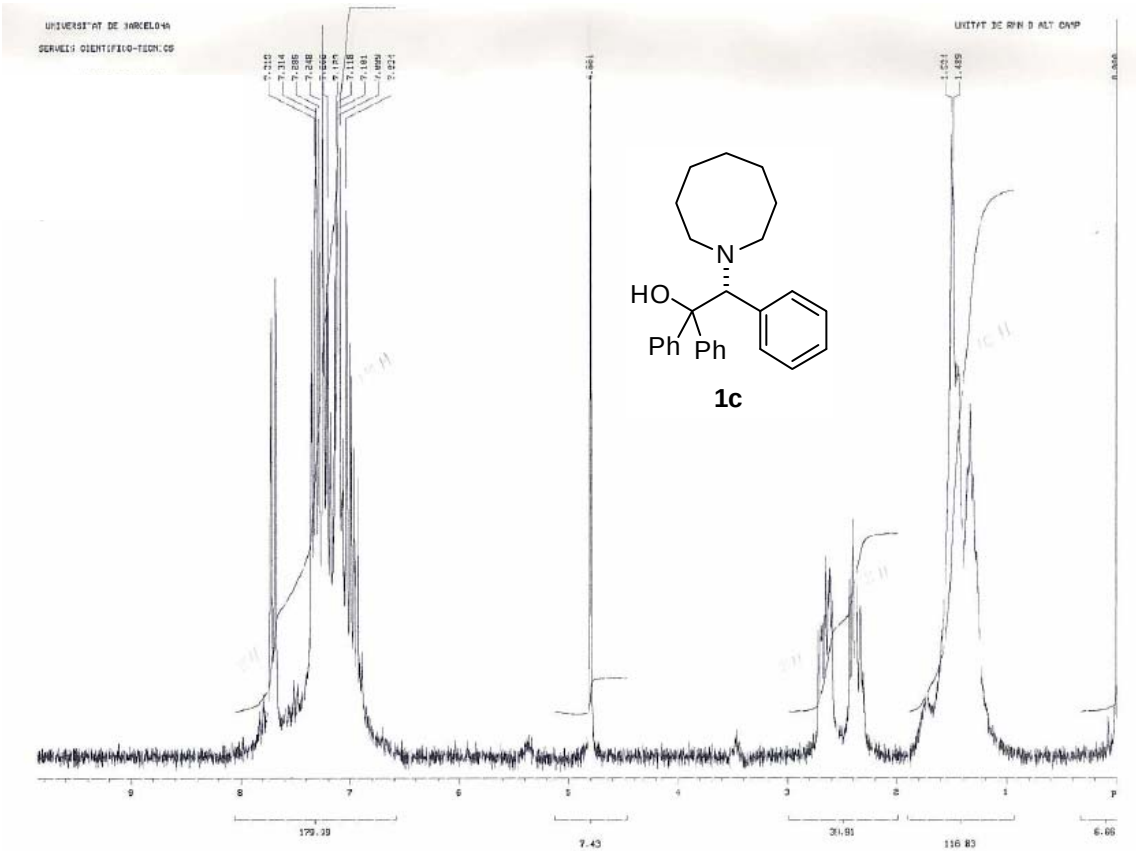

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
135.704
128.968
128.887
128.383
128.123
128.068
126.094
144.411
88.618
21.797
137.884
135.803
78.206
77.889
76.953

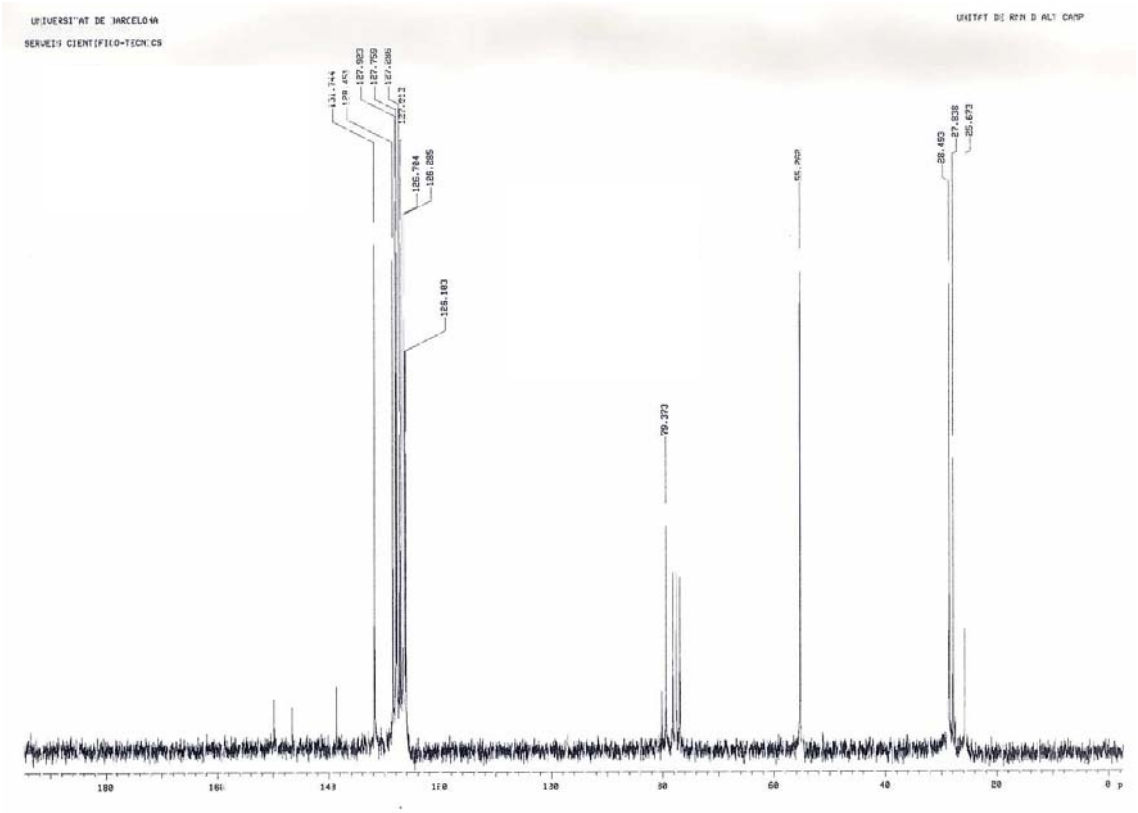

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
O
Ph
Ph
Me
11
7.168
7.150
7.134
7.136
7.045
2.418
180.00
36.64
1.82
1.50

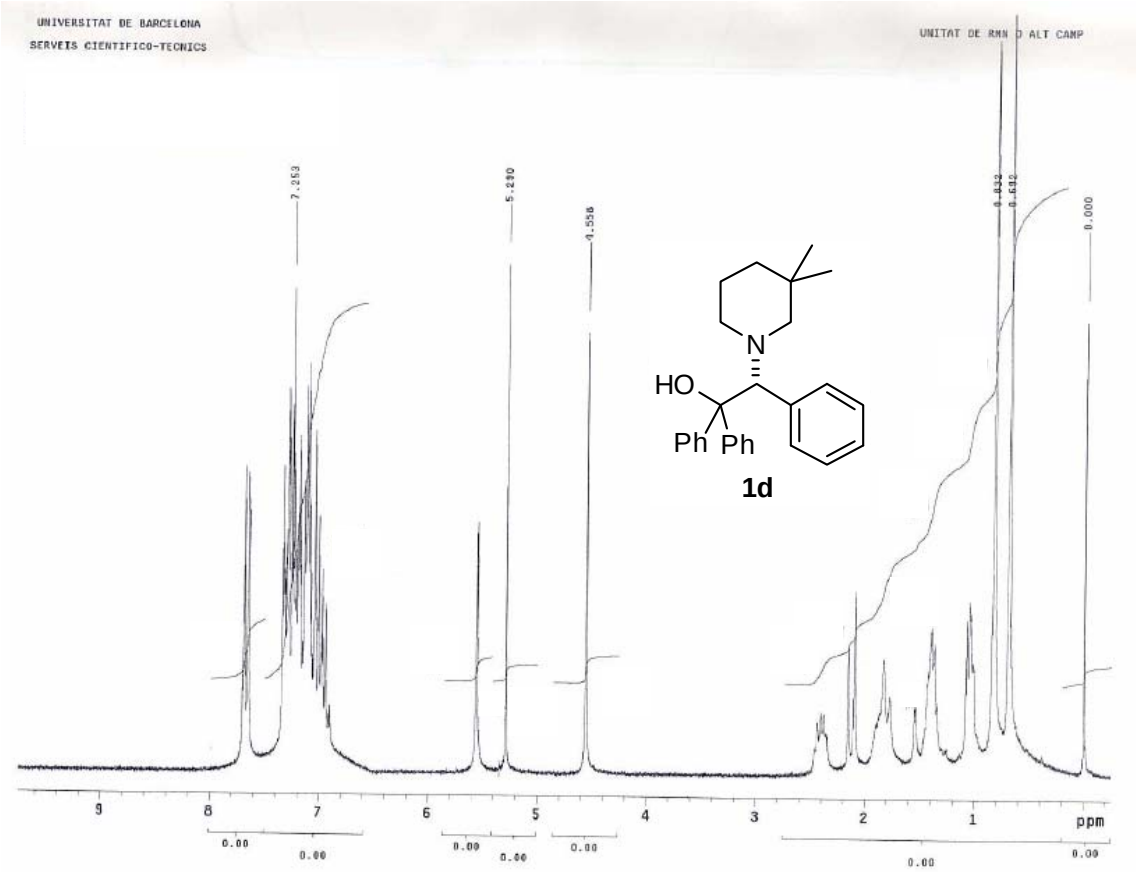

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
128.588
128.869
129.414
129.153
127.985
127.822
127.384
126.702
125.867
125.822
78.194
77.953
77.916
66.388
19.769

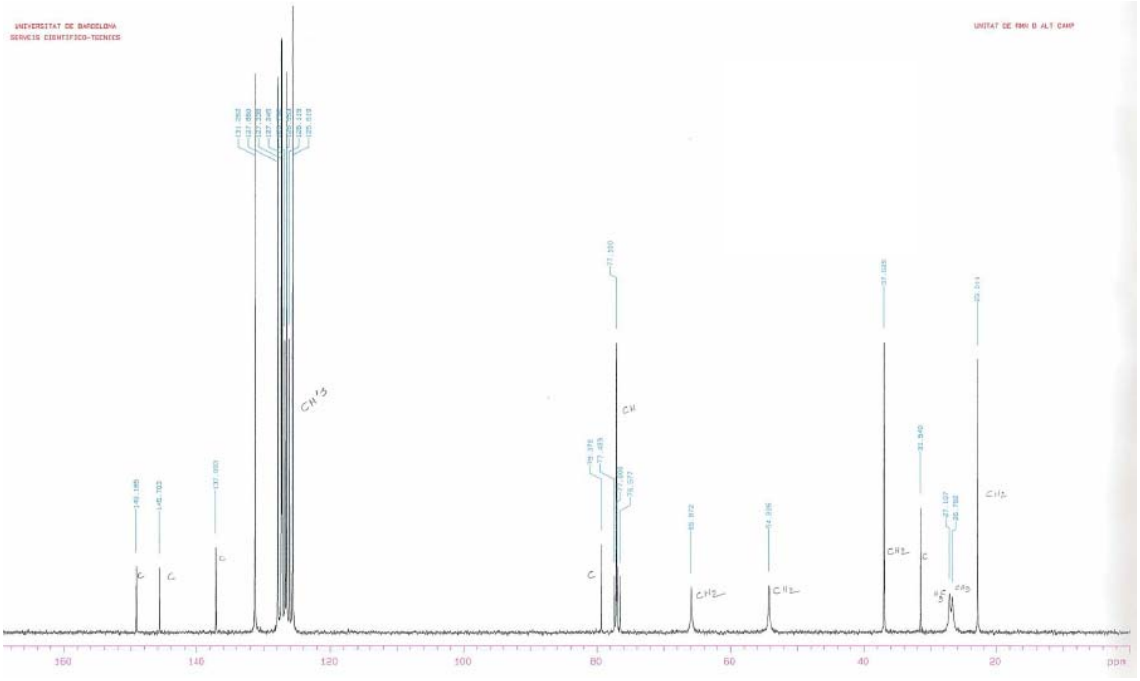

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
HO
N
Ph
Ph
1b

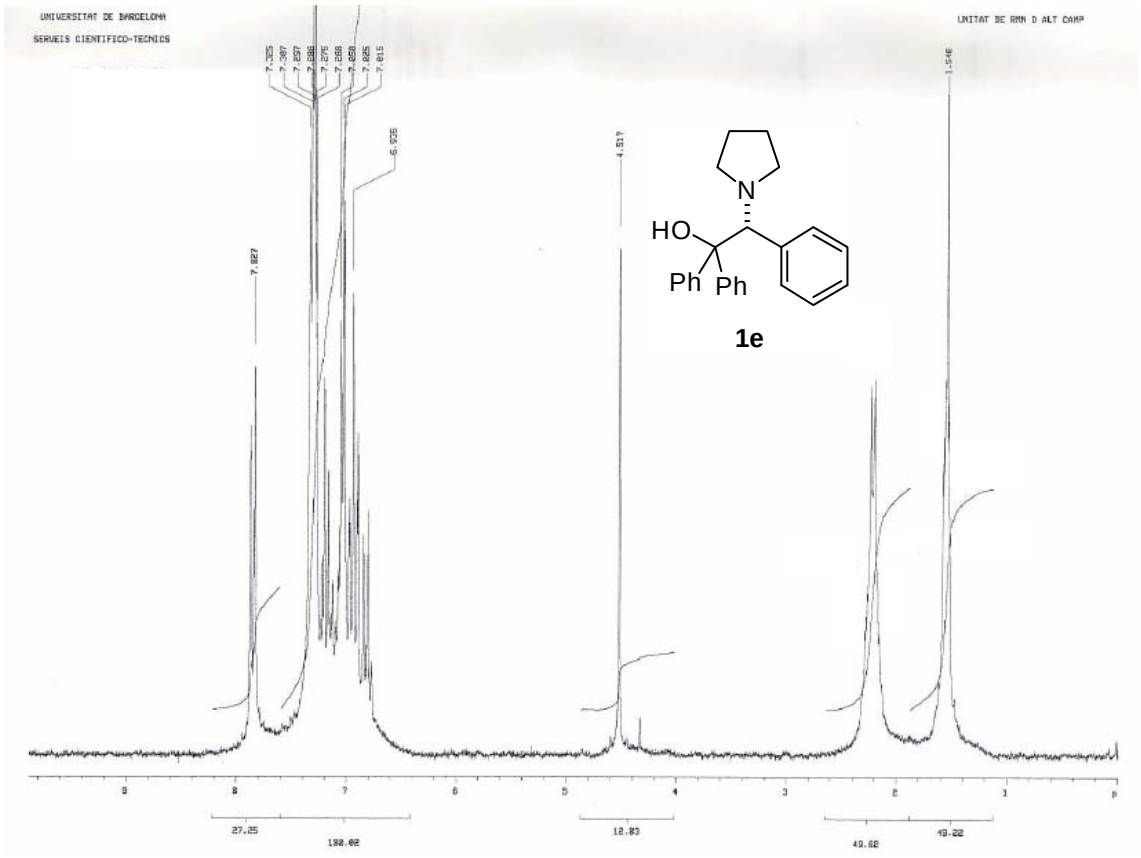

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP

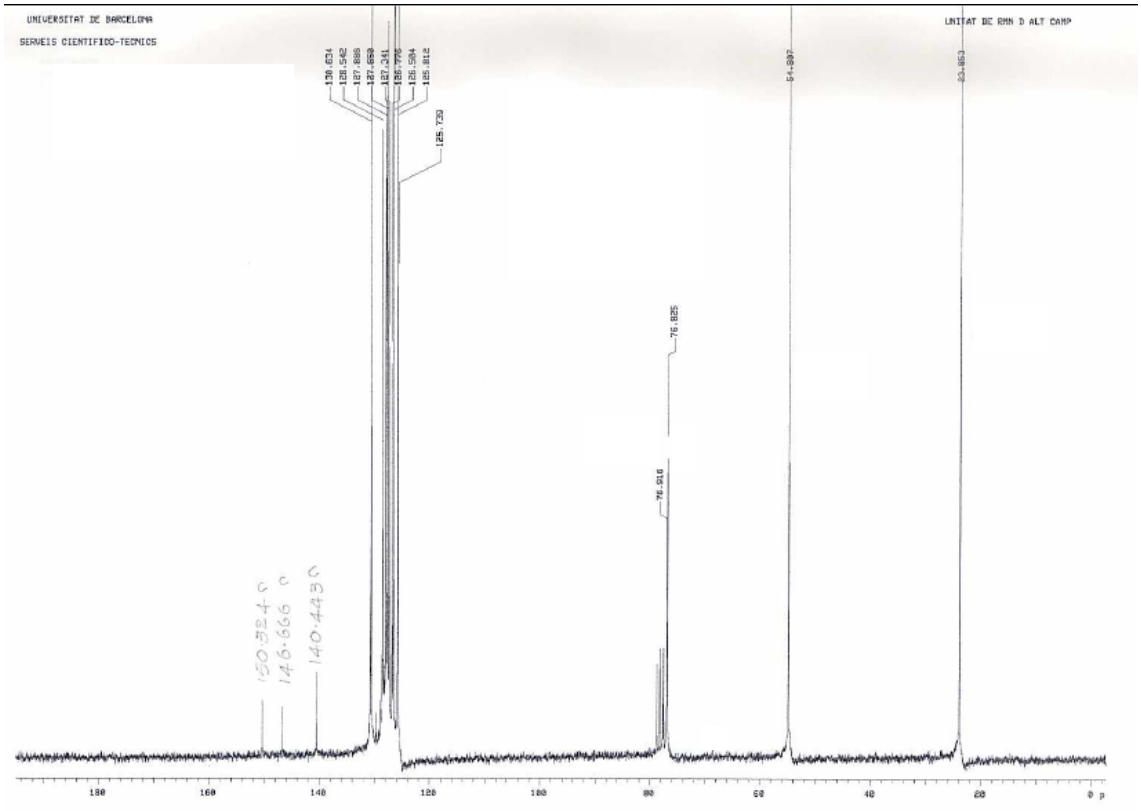

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
N
HO
Ph
Ph
1c

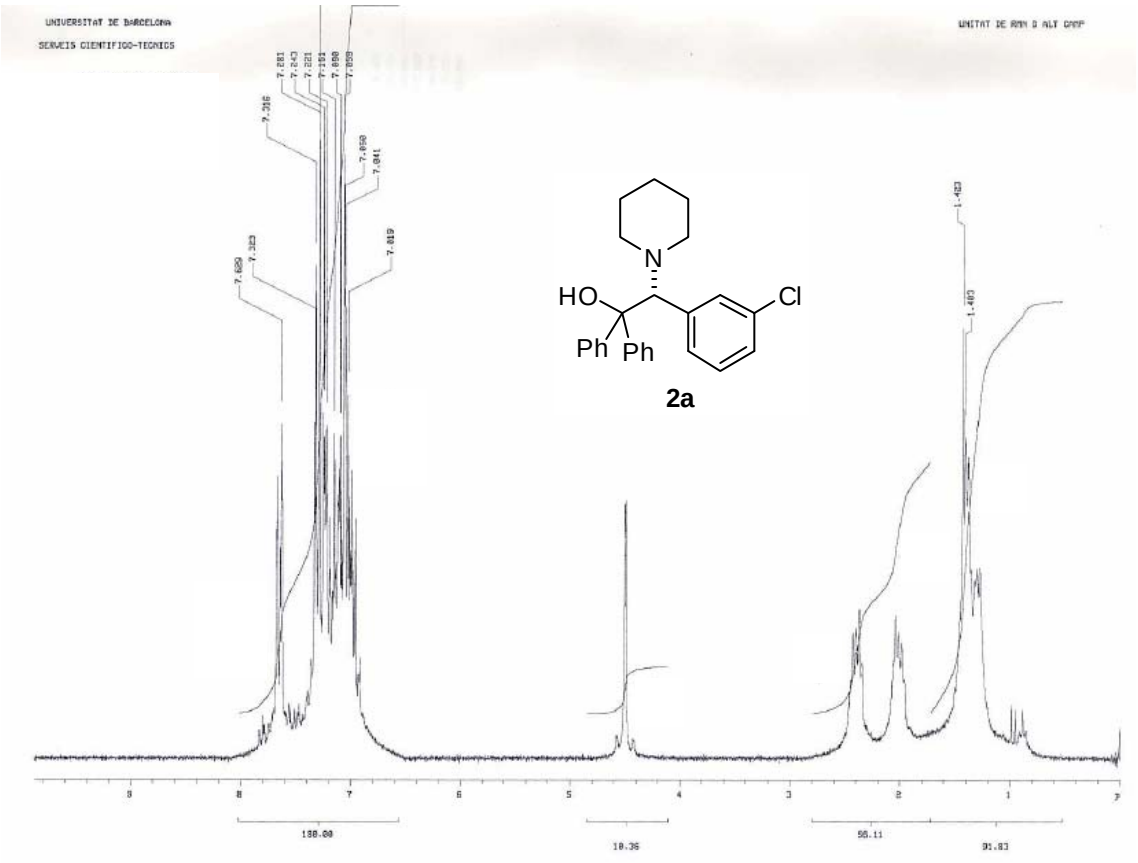

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP

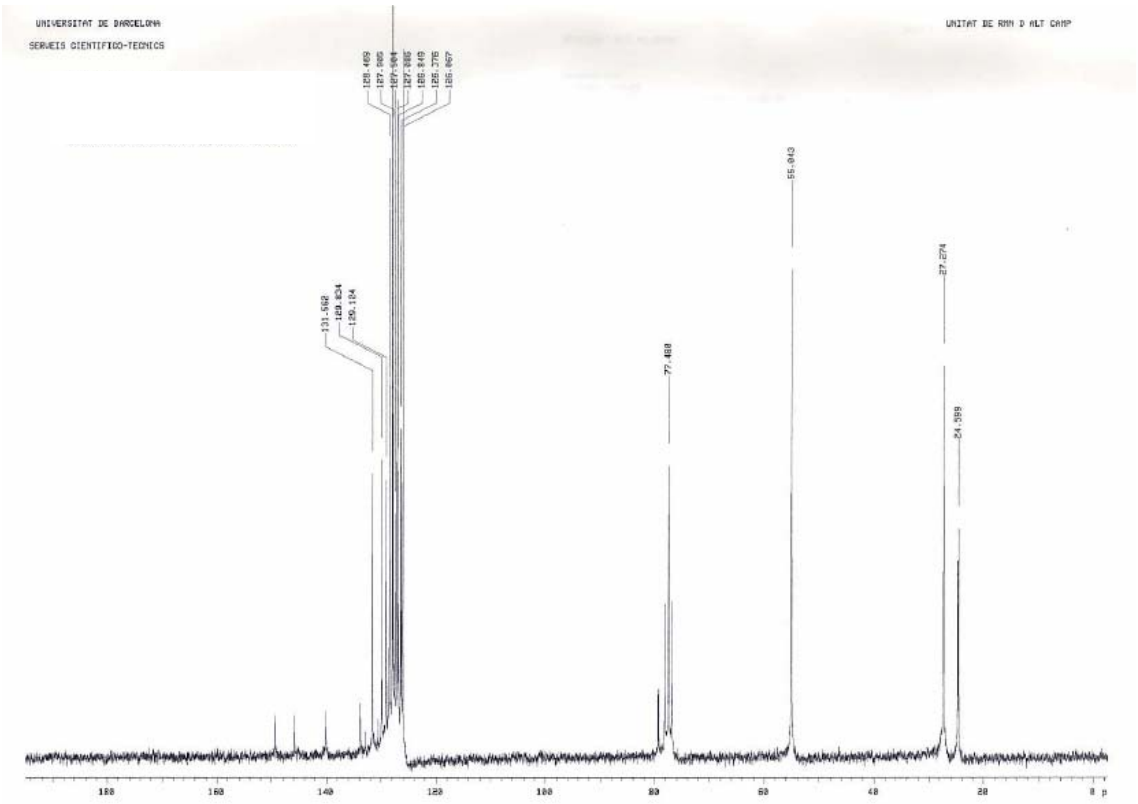

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
7.263
5.290
4.556
0.853
0.682
0.000
N
HO
Ph Ph
1d
9
8
7
6
5
4
3
2
1
ppm
0.00
0.00
0.00
0.00
0.00
0.00
0.00

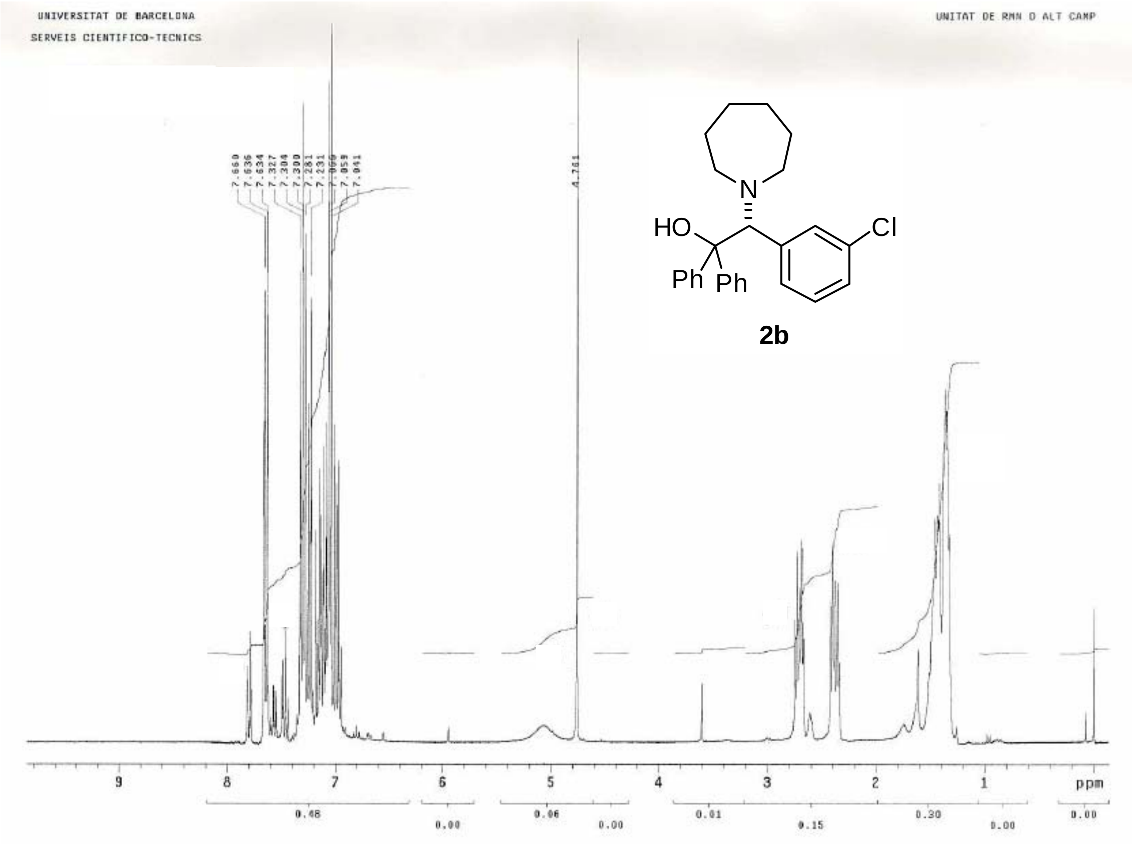

CH3
CH
CH2
CH2
CH2
CH2
160
140
120
100
80
60
40
20
ppm

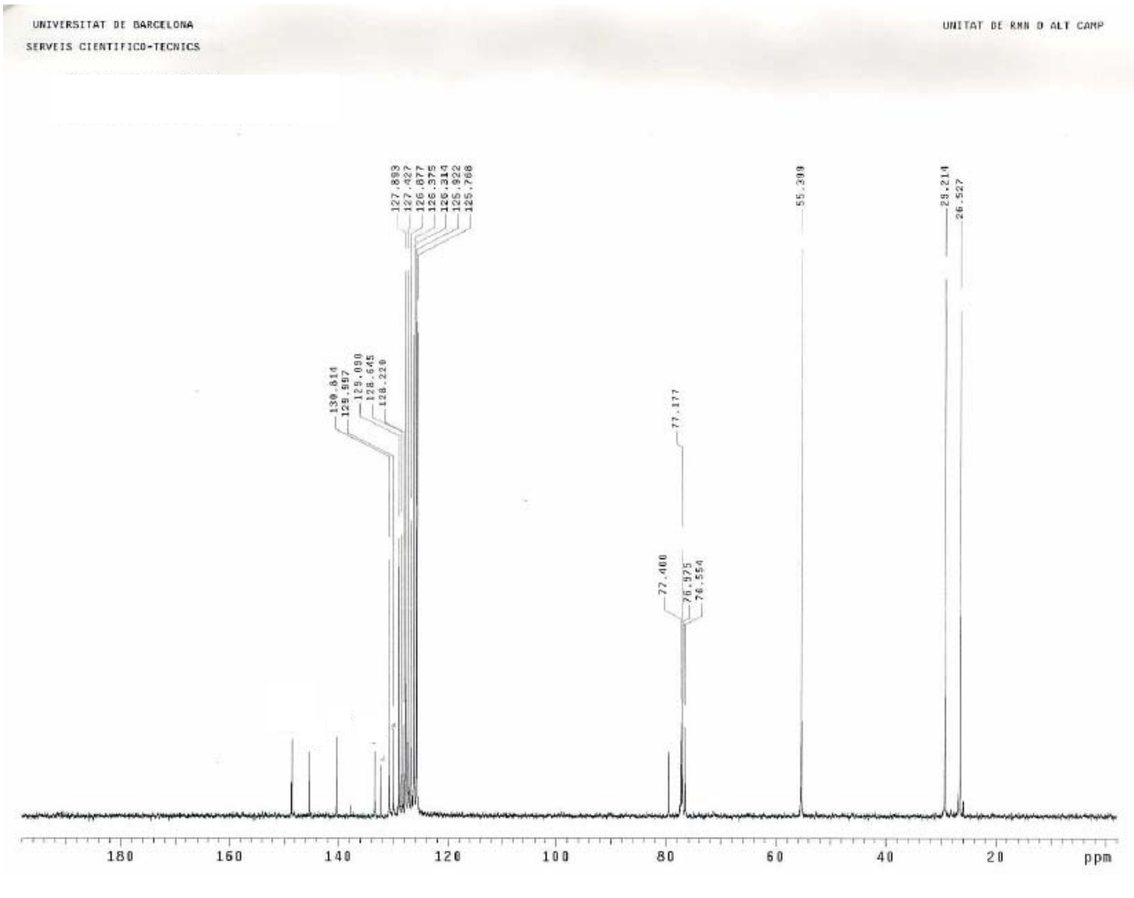

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
HO
N
Ph
Ph
1e

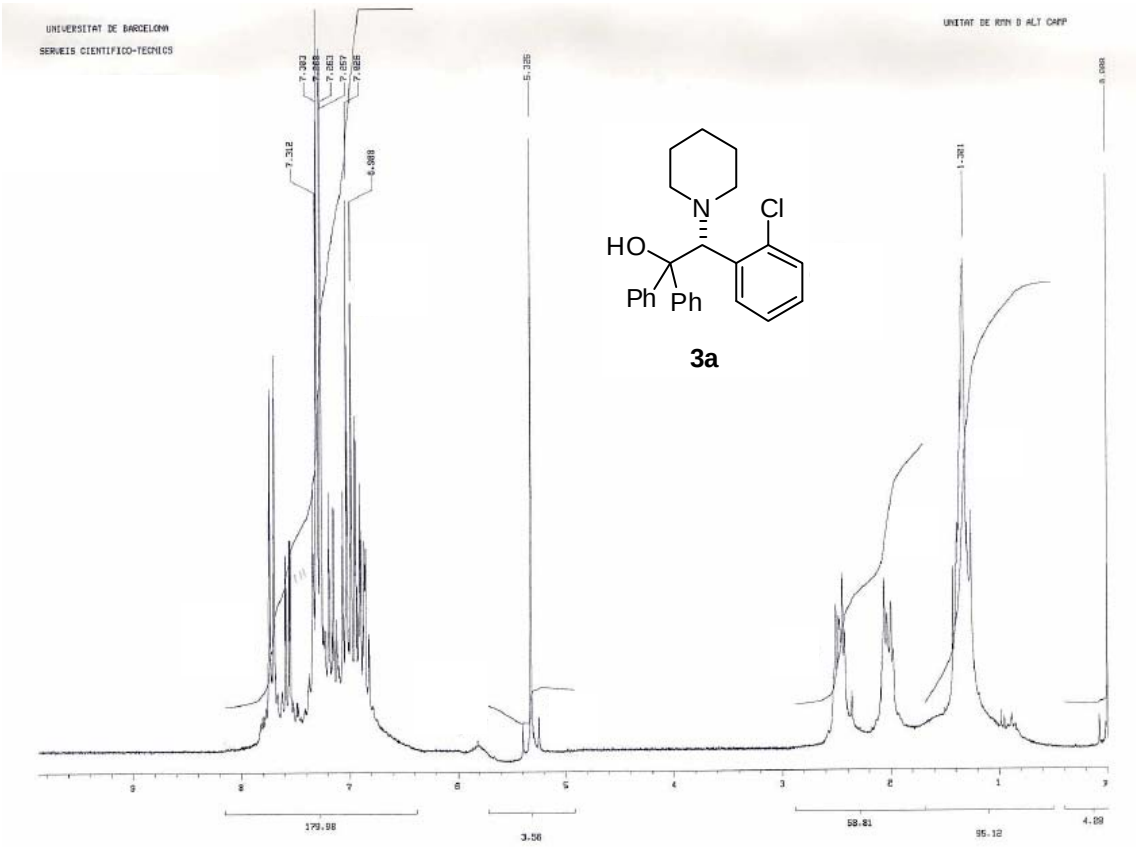

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
HO
N
Cl
Ph
Ph
2a

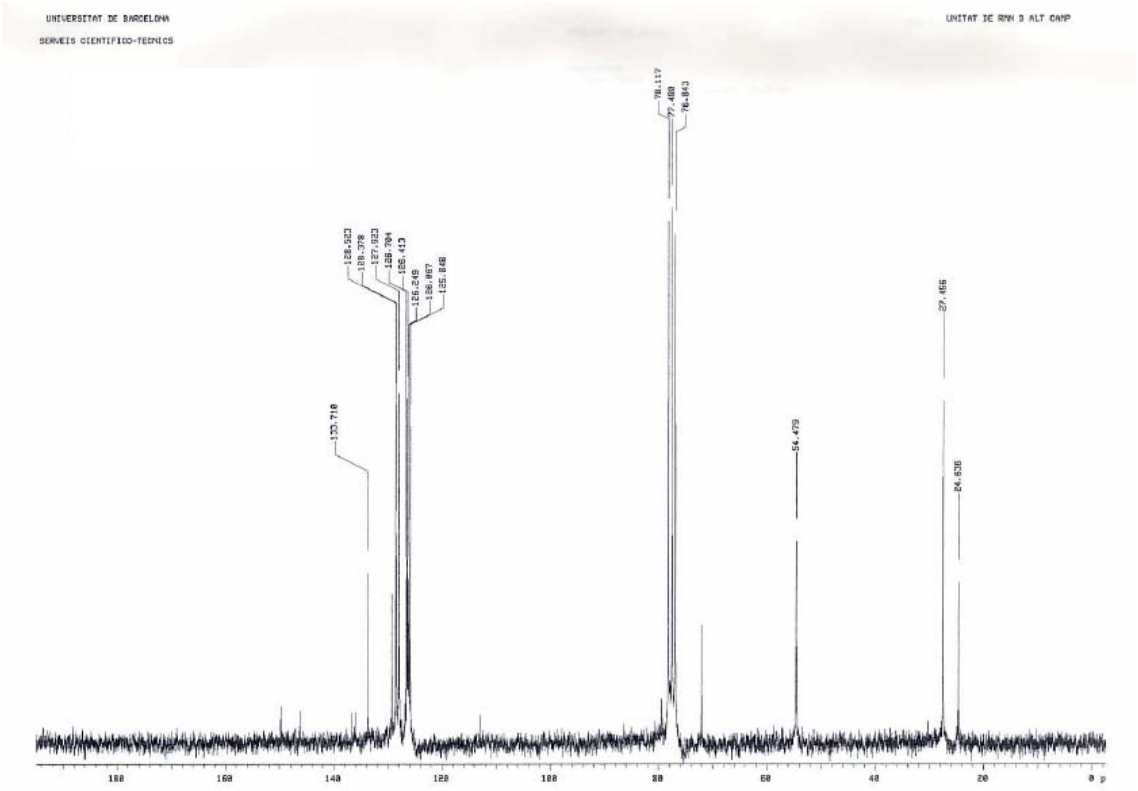

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP

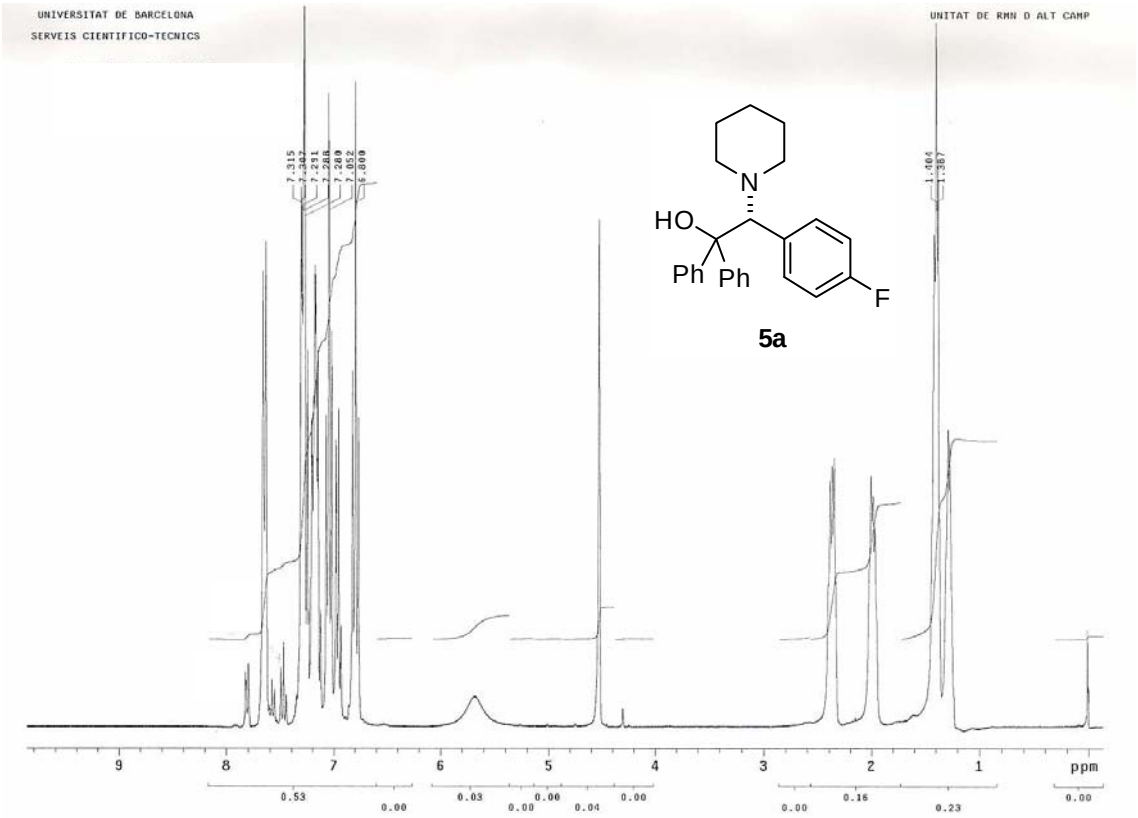

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
7.660
7.636
7.634
7.327
7.304
7.300
7.281
7.231
7.059
7.041
4.741
HO
N
Cl
Ph
Ph
2b
9
8
7
6
5
4
3
2
1
ppm
0.48
0.00
0.06
0.00
0.01
0.15
0.30
0.00
0.00

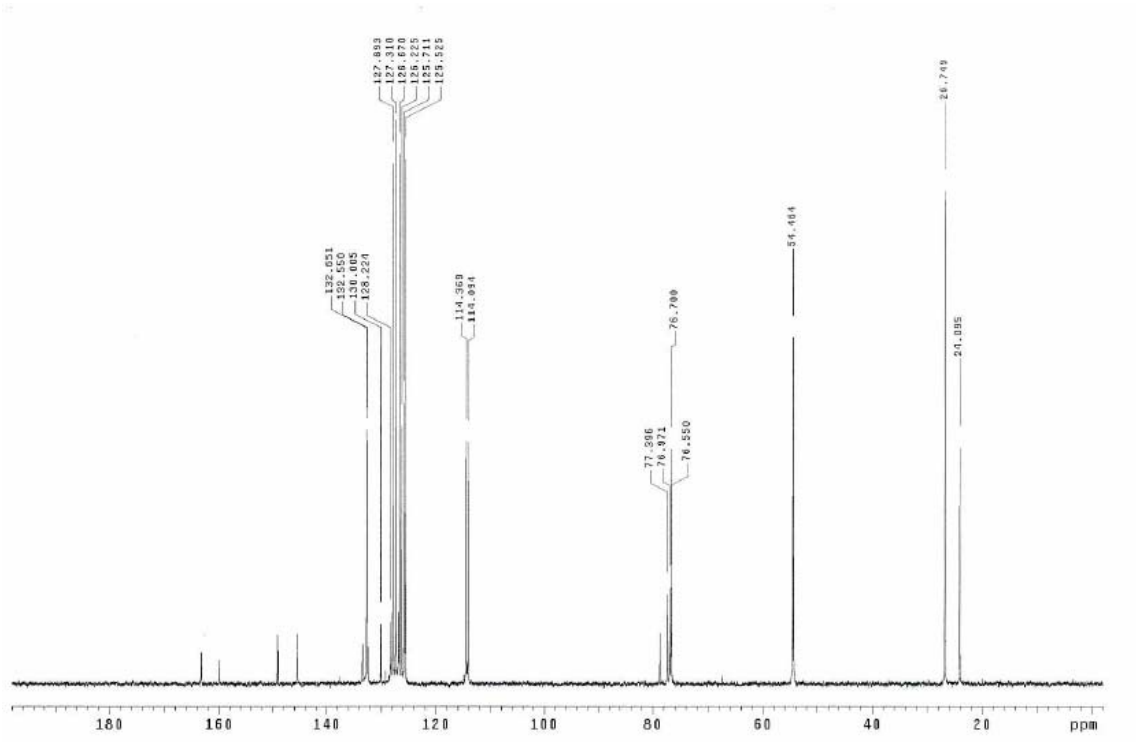

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
127.893
127.427
126.877
126.375
126.314
125.922
125.768
130.814
129.897
129.090
128.645
128.220
77.177
72.400
76.975
76.554
55.399
29.214
26.527
180
160
140
120
100
80
60
40
20
ppm

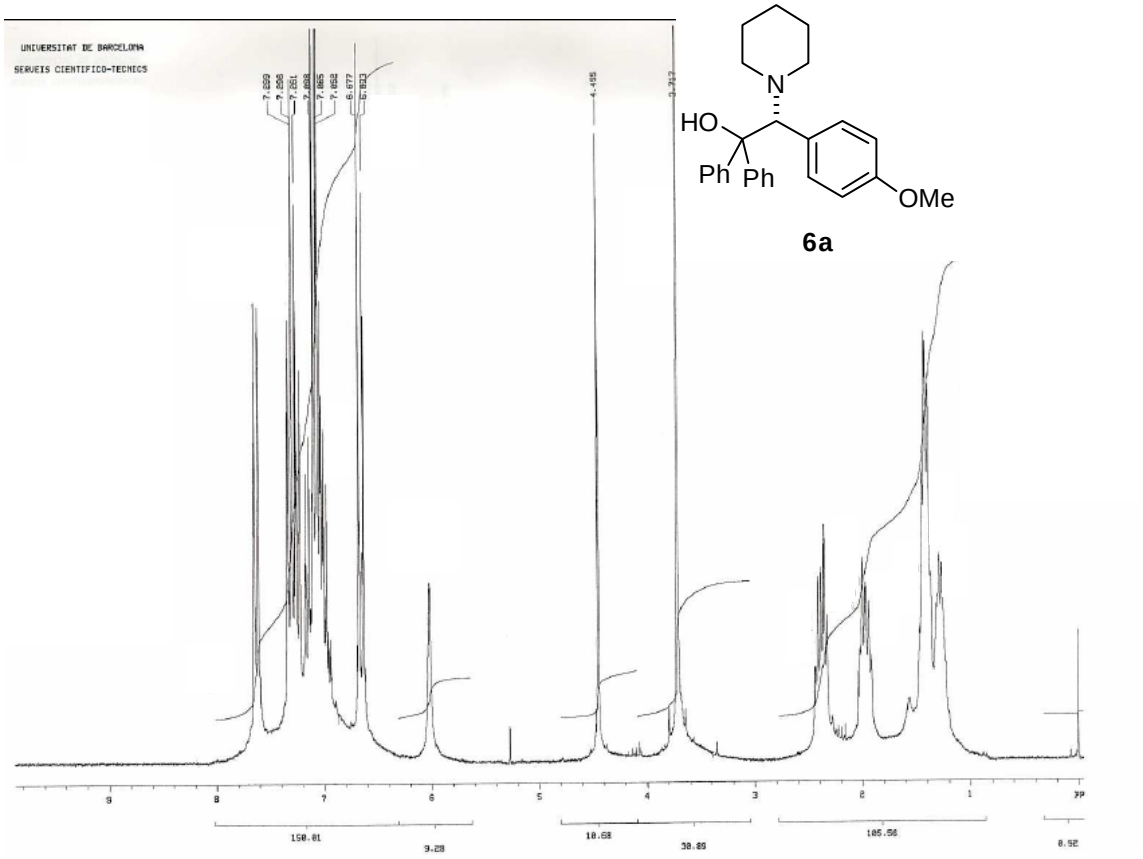

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
N
Cl
HO
Ph
Ph
179.98
3.56
58.81
95.12
4.09

3a

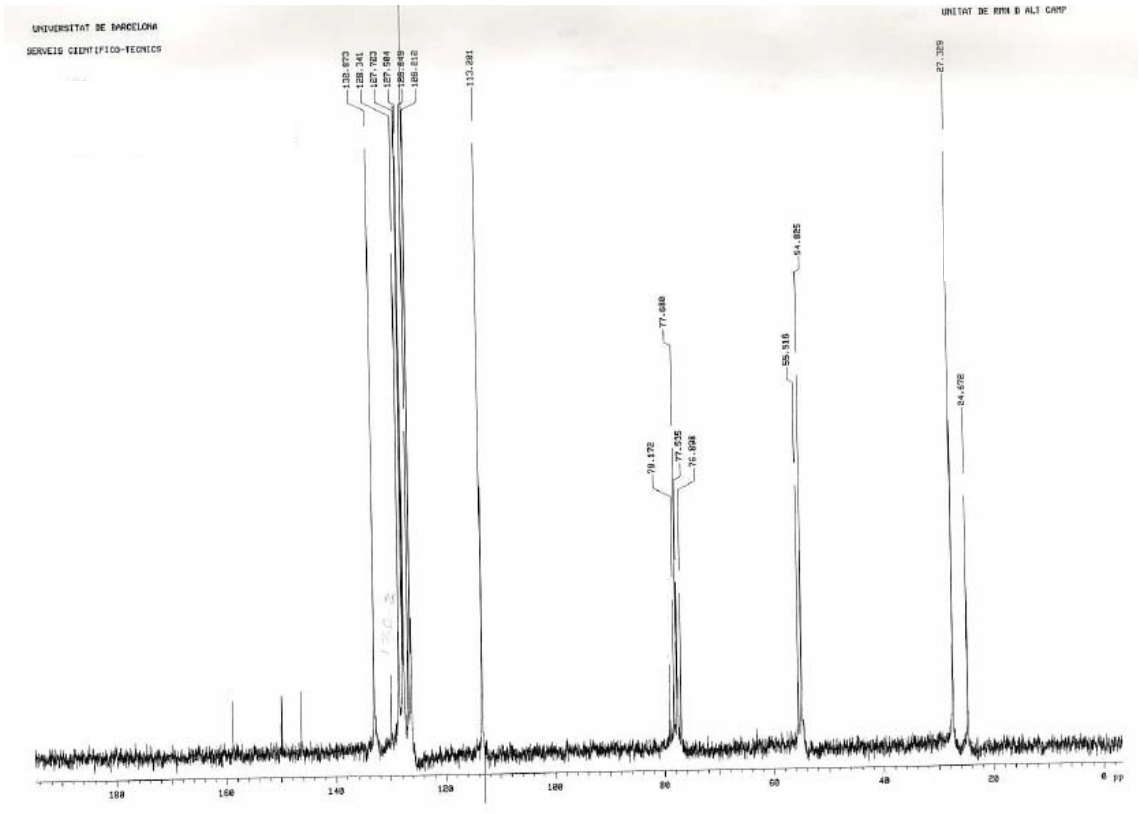

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP

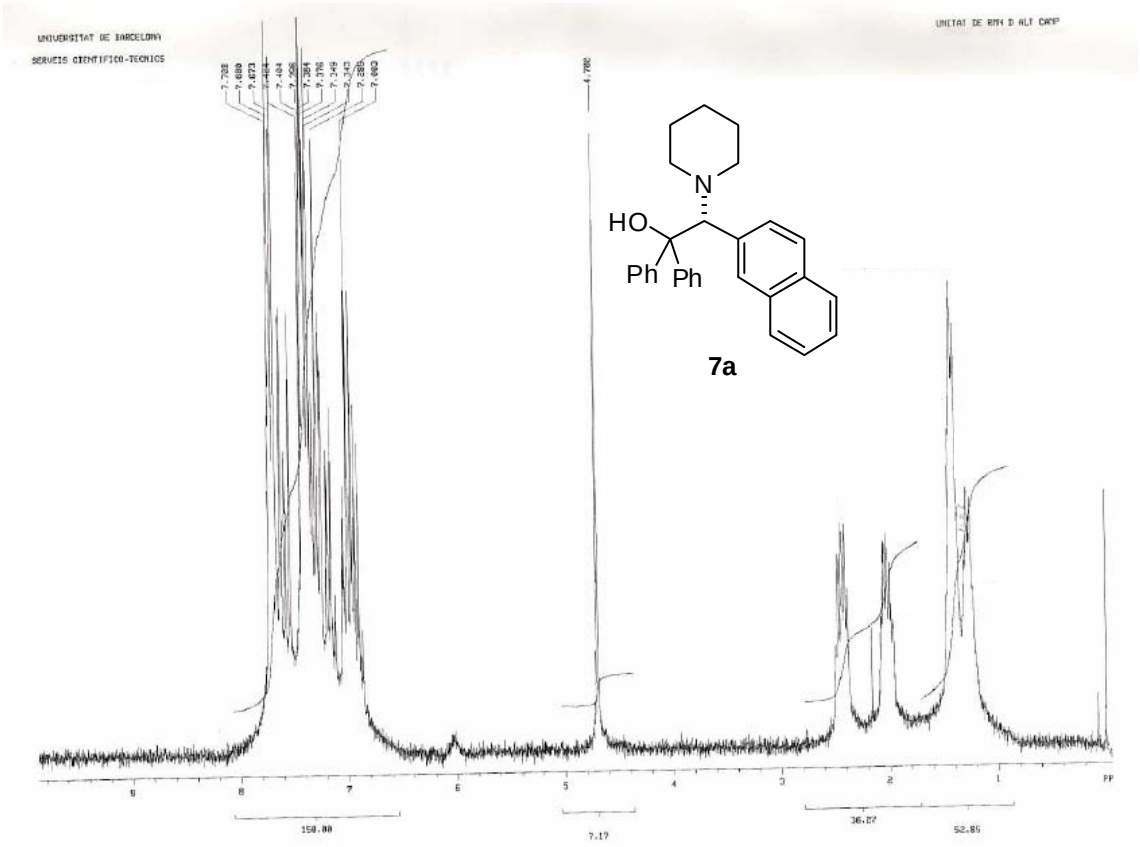

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
HO
N
Ph
Ph
F
5a
ppm

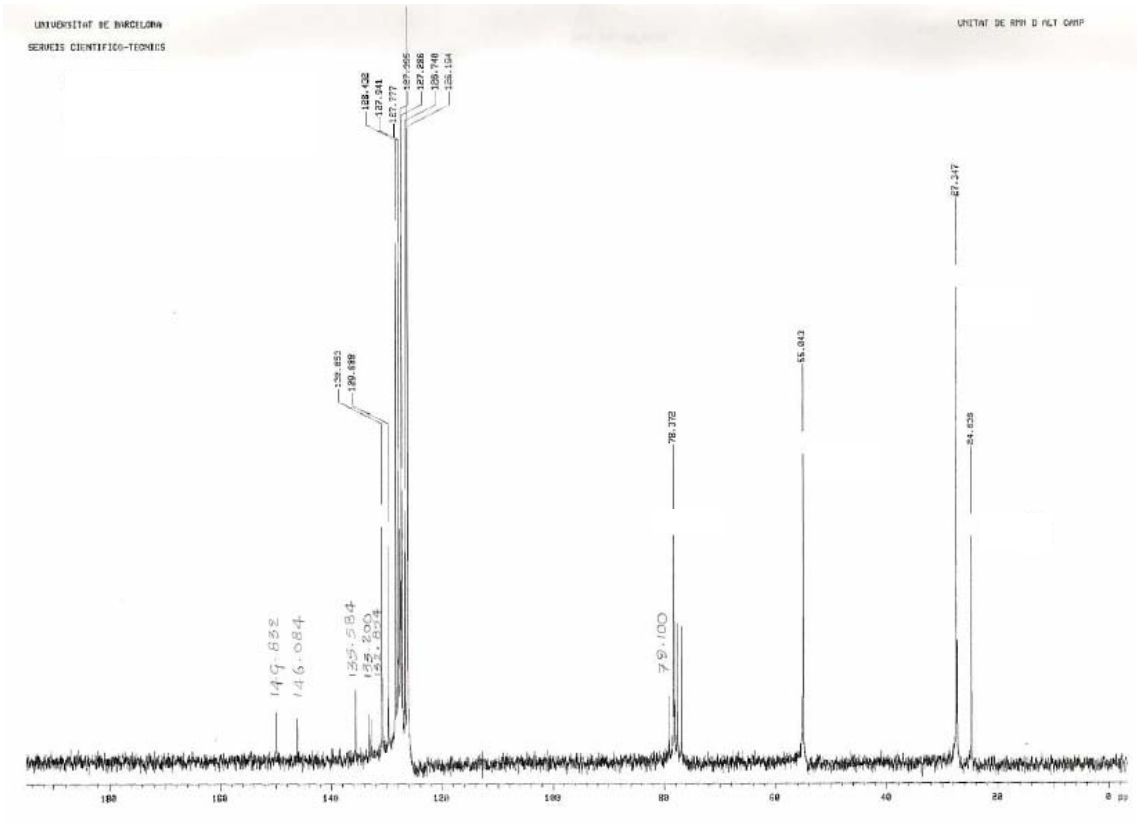

ppm

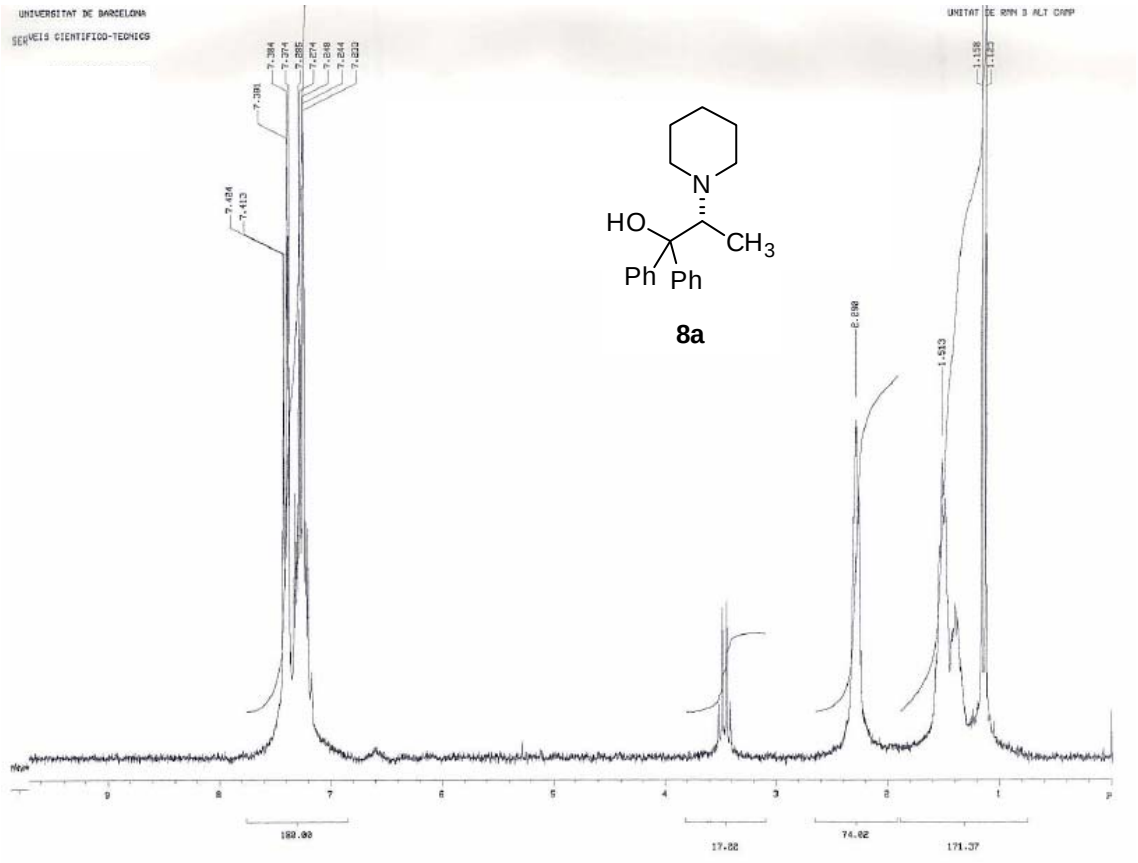

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
HO
N
Ph
Ph
OMe

6a

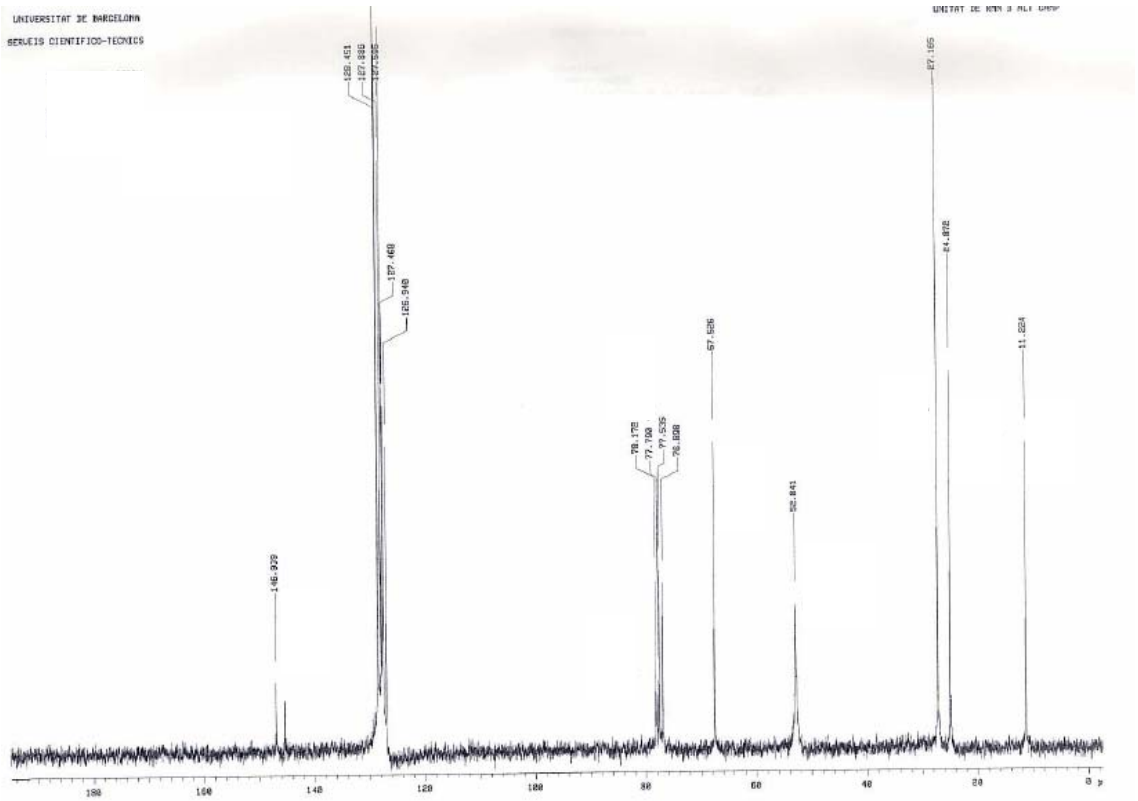

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS

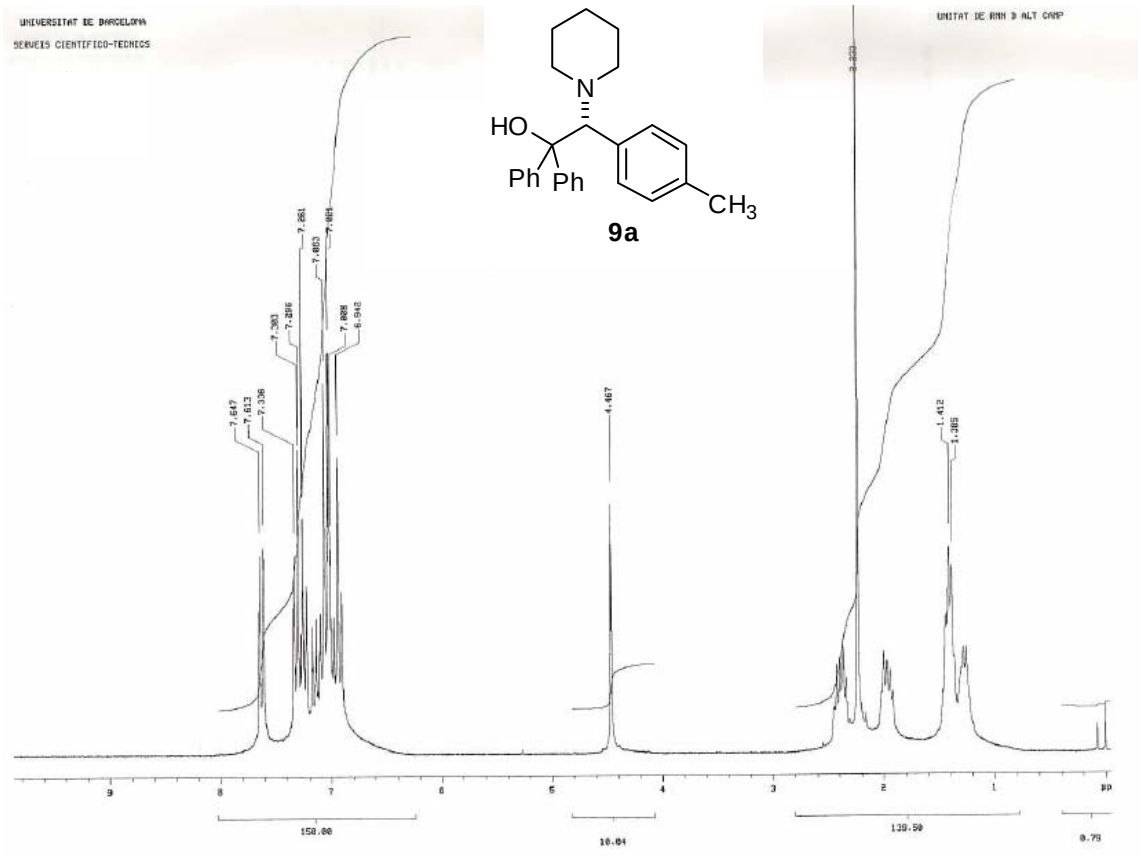

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
HO
N
Ph
Ph
7a

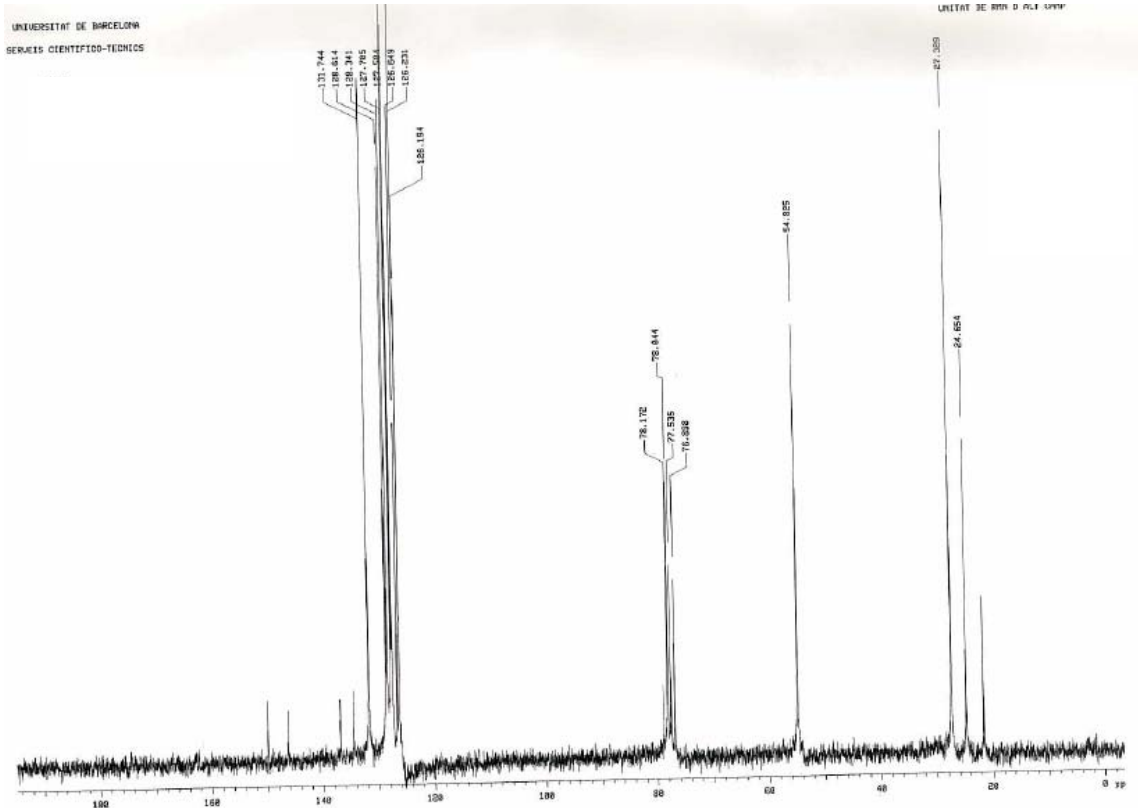

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
27.247
55.041
78.372
79.100
146.084
146.832

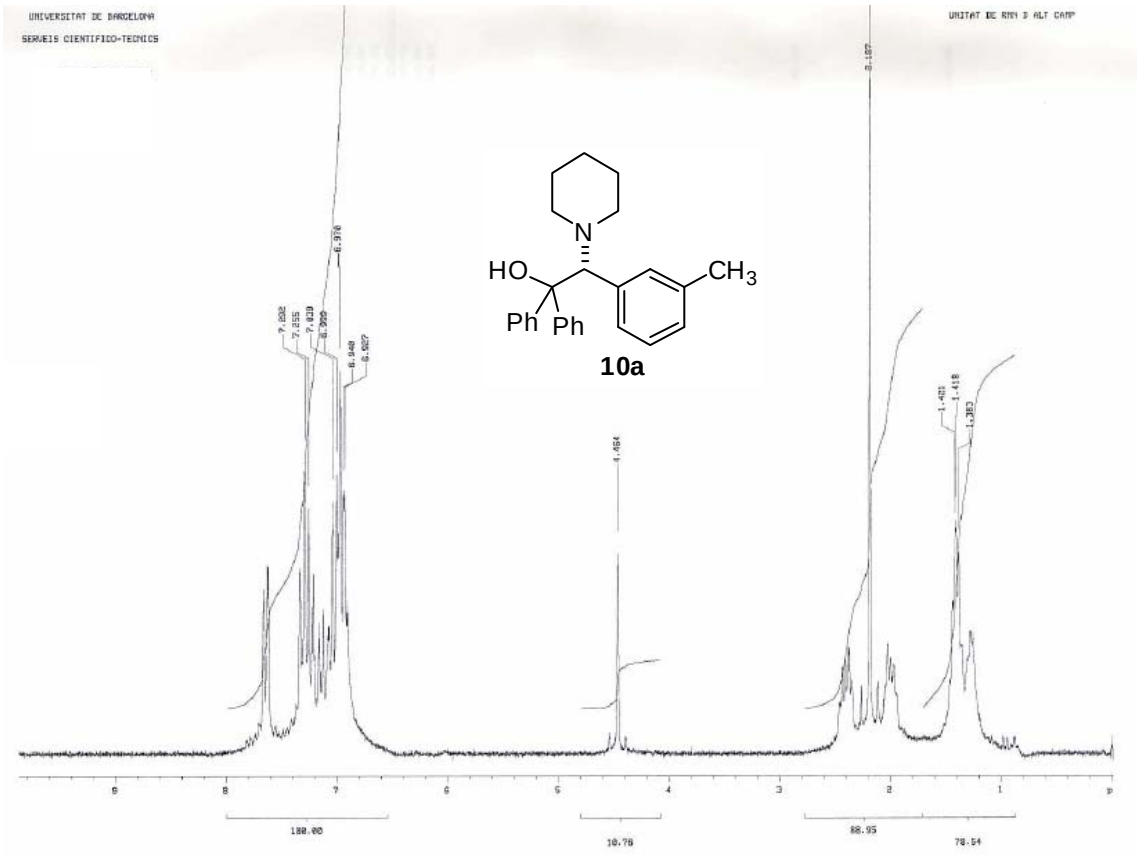

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
HO
CH3
Ph Ph
N

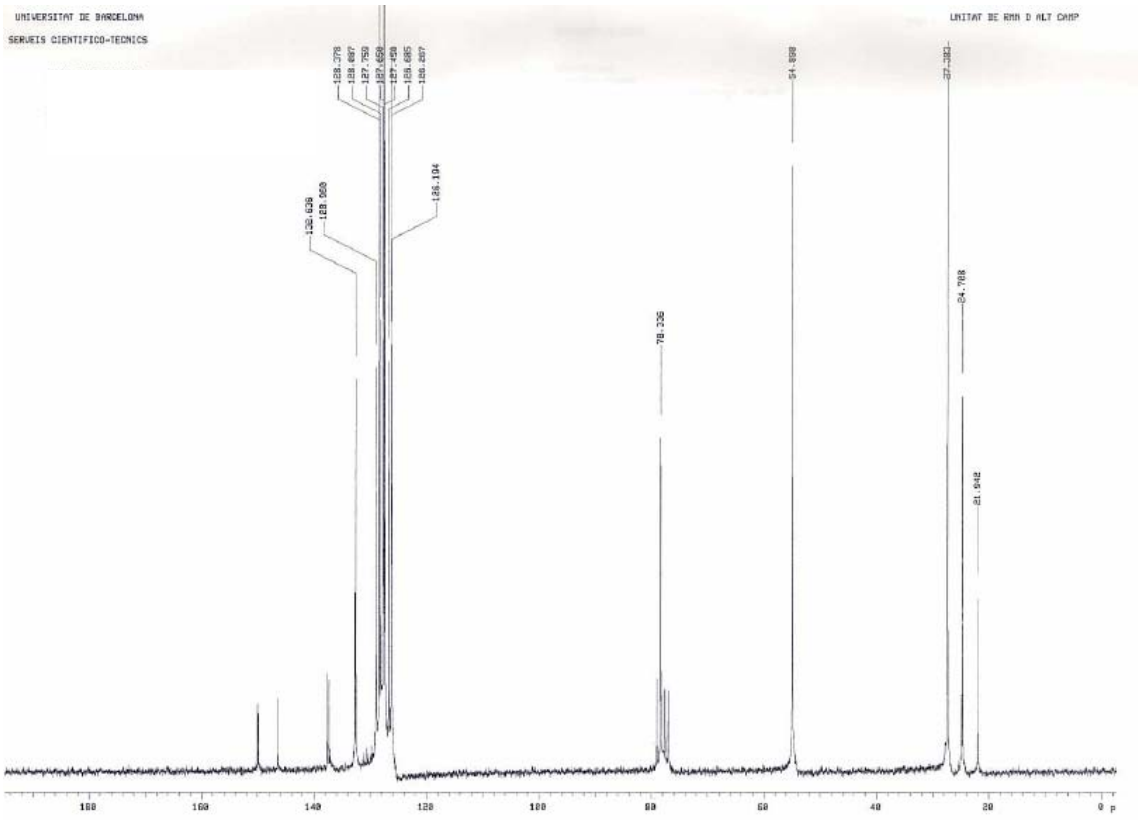
8a

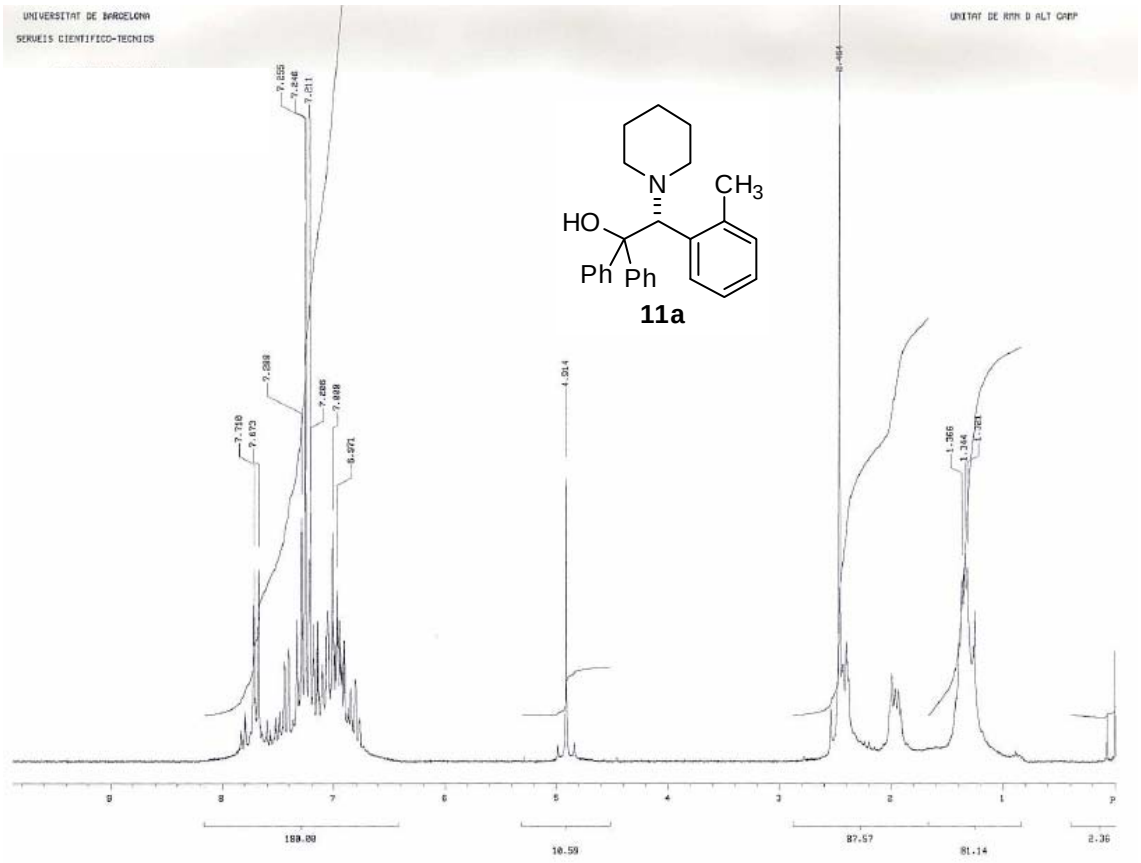

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS

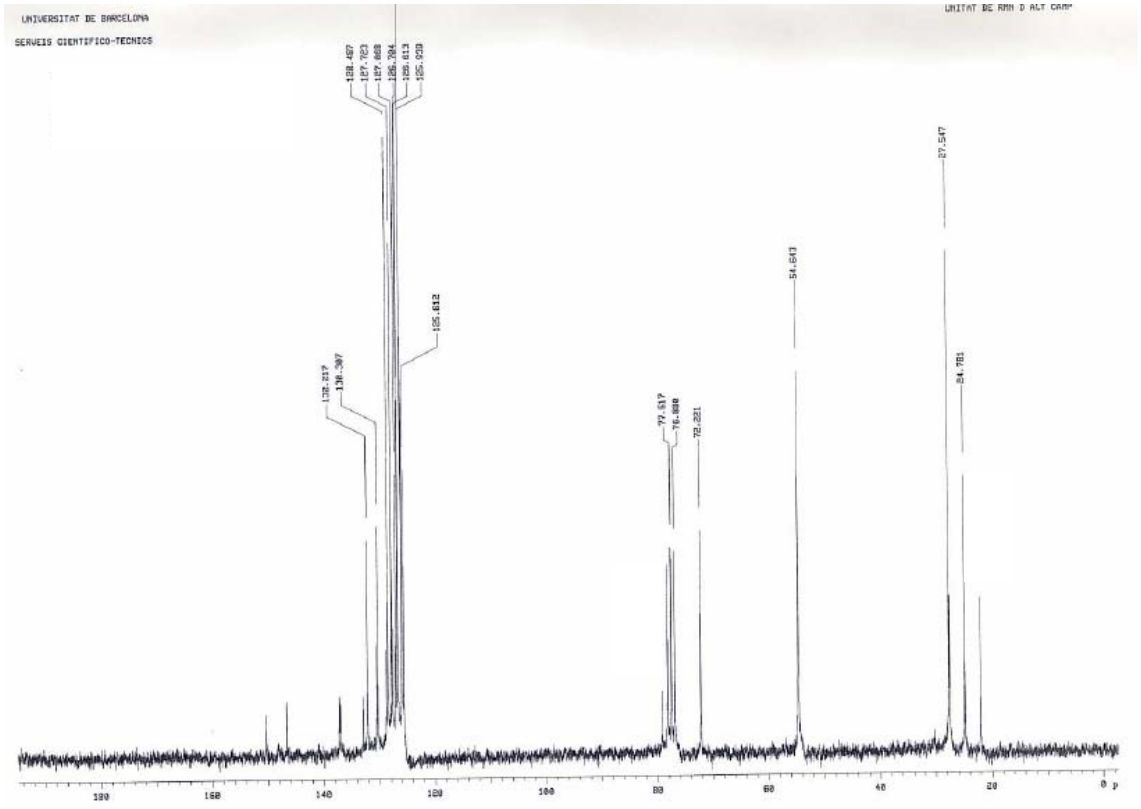

UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
HO
Ph
Ph
N
CH3
9a
7.647
7.613
7.336
7.303
7.286
7.261
7.063
7.024
7.008
6.948
4.467
3.220
1.412
1.385
150.00
10.04
139.50
0.79


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
131.744
130.614
129.341
127.795
126.649
126.231
108.194
78.844
78.172
77.535
76.898
54.825
27.389
24.654


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
HO
N
CH3
Ph
Ph
10a
188.00
10.78
88.95
78.54


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP
N
CH3
HO
Ph
Ph
11a


UNIVERSITAT DE BARCELONA
SERVEIS CIENTIFICO-TECNICS
UNITAT DE RMN D ALT CAMP

Synthetic details for the preparation of the starting olefins.

General Grignard procedure for the preparation of olefins (GP-G):

The corresponding substituted benzyl chloride (30.0 mmol) was added dropwise, over a period of 30 minutes, to a suspension of magnesium turnings (36.0 mmol, 875 mg) and iodine (0.3 mmol, 76 mg) in dry diethyl ether (3 mL at the beginning + 10 mL once the reaction is initiated). After two hours, the suspension was cooled to 0 ºC, and benzophenone (28.5 mmol, 4.7 mL) in anhydrous diethyl ether (7 mL) was slowly added. The reaction was allowed to warm and stirred at room temperature until TLC showed complete conversion. Saturated NH_4Cl aqueous solution was slowly added to the mixture, the aqueous phase was extracted with EtOAc (3 × 15 mL), and the combined organic phases were dried over $MgSO_4$ and concentrated under vacuum. The residual solid was dissolved in 12 mL of 20% solution of H_2SO_4 in AcOH and stirred at room temperature for 15 minutes. The reaction mass was poured into a stirred mixture of water (30 mL) and ethyl ether (45 mL). Phases were separated. The organic layer was washed with saturated solution of $NaHCO_3$ (2 × 25 mL) and water (2 × 25 mL), dried over Na_2SO_4 and concentrated under vacuum. The crude product was purified by chromatography to afford the corresponding olefin.

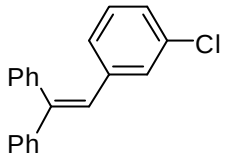
General Wittig procedure for the preparation of olefins (GP-W):

A suspension of potassium *tert*-butoxide (6.25 g, 56.0 mmol) in anhydrous THF (80 mL) was added to a suspension of benzhydryl-triphenylphosphonium bromide (25.0 g, 49.0 mmol) in anh. THF (100 mL) at room temperature under vigorous stirring. After 23 h, the resulting phosphorous ylide, which has a strong red color, was added to a solution of the corresponding aldehyde (33.0 mmol) in anh. toluene (60 mL). The mixture was kept at reflux until disappearance of the starting material, it was cooled to room temperature and solvents were eliminated under vacuum. The residual solid was purified by chromatography to afford the corresponding olefin.

General procedure for the alternative preparation of olefins (GP-A)

At 0 ºC, a 2.5 M solution of *n*-BuLi in hexanes (29.9 mmol, 12.0 mL) was added to a solution of diphenylmethane (29.9 mmol, 5.0 mL) in anhydrous THF (15 mL) under argon. After the addition, it was allowed to reach r.t. and remained at the same temperature for 2 h. Then it was cooled again to 0 ºC and the corresponding aldehyde (29.9 mmol) dissolved in anhydrous THF (15 mL) was slowly added. The mixture was stirred for 40 minutes before allowing it to reach r.t. The reaction was quenched by adding 20 mL of NH_4Cl sat. sol. The organic solvent was removed under vacuum and the resulting yellowish precipitate was filtered and washed with water. The solid was dissolved in CH_2Cl_2, dried over $MgSO_4$ and concentrated under vacuum. The crude product was redissolved in toluene (100 mL) and *p*-toluenesulfonic acid (2.9 mmol, 0.552 g), and a small amount of $MgSO_4$ were added. The mixture was heated to reflux for the corresponding time. At r.t. the solution was filtered off, the solvent removed under vacuum and the resulting brown solid was recrystallized from hexane affording the corresponding olefin.

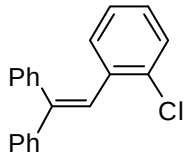
1,1-diphenyl-2-(3-chlorophenyl)ethene[1]



The general procedure (GP-W), was followed for *m*-chlorobenzaldehyde (33.0 mmol, 3.72 mL) for 24 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the desired olefin (9.12 g, 95%) as an oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40-6.75 (15H, m).
^{13}C NMR (50 MHz, $CDCl_3$): δ 143.3, 141.1, 140.3, 137.2, 130.2, 130.2, 128.5, 128.3, 128.2, 127.6, 127.5, 127.3, 127.2, 127.1, 126.5.

1,1-diphenyl-2-(2-chlorophenyl)ethene[1]

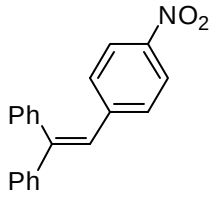


The general procedure (GP-W), was followed for *o*-chlorobenzaldehyde (33.0 mmol, 3.72 mL) for 12 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the desired compound (6.72 g, 70%) as an oil.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.42-6.73 (15H, m).
^{13}C NMR (50 MHz, $CDCl_3$): δ 143.1, 140.0, 139.4, 136.9, 130.1, 130.0, 128.4, 128.3, 128.1, 127.6, 127.4, 127.3, 127.2, 127.1, 126.3.

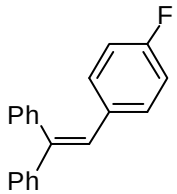
1,1-diphenyl-2-(4-nitrophenyl)ethene[2]

The general procedure (GP-W), was followed for *p*-nitrobenzaldehyde (33.0 mmol, 4.98 g) for 5 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the desired olefin (8.95 g, 90%) as a white solid. Spectroscopic data matched the literature.



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40-6.70 (15H, m).
^{13}C NMR (50 MHz, $CDCl_3$): δ 146.3, 143.9, 143.7, 137.0, 136.9, 130.1, 130.0, 129.3, 128.3, 128.2, 127.6, 127.4, 127.3, 127.2, 126.8, 124.4.

1,1-diphenyl-2-(4-fluorophenyl)ethene[3]

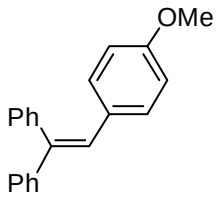


The general procedure (GP-W), was followed for *p*-fluorobenzaldehyde (33.0 mmol, 3.54 mL) for 12 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 98:2) gave the title compound (5.88 g, 65%) as an oil. Spectroscopic data matched the literature.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.36-6.78 (15H, m).
^{13}C NMR (50 MHz, $CDCl_3$): δ 161.3, 145.7, 140.0, 139.9, 134.7, 130.1, 130.0, 129.3, 128.3, 128.2, 127.6, 127.5, 127.3, 127.2, 126.9, 118.3.

[1] Tewari, R. S.; Suri, S. K.; Gupta, K. C. Z. *Naturforsch., B: Chem. Sci.* **1979**, *34*, 606.
[2] Jawdosiuk, M.; Uminski, M.; Kmiotek-Skarzynska, I.; Makosza, M. *Pol. J. Chem.* **1981**, *55*, 1309.
[3] Garcia-Delgado, N.; Reddy, K. S.; Solà, L.; Riera, A.; Pericas, M. A.; Verdaguer, X. *J. Org. Chem.* **2005**, *70*, 7426.

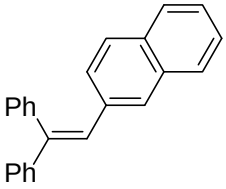
1,1-diphenyl-2-(4-methoxyphenyl)ethene[4]



The general procedure (GP-A), was followed for *p*-anisaldehyde (29.9 mmol, 3.64 mL) for 4 h, yielding the desired olefin (6.29 g, 75%) as a pale yellow solid (the Wittig procedure gave only 18% yield). Spectroscopic data matched the literature.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.30-7.18 (10H, m), 6.88 (2H, d, J = 8.6 Hz), 6.84 (s, 1H), 6.59 (2H, d, J = 8.6 Hz), 3.68 (3H, s).
^{13}C NMR (50 MHz, $CDCl_3$): δ 158.8, 144.0, 141.0, 140.9, 131.2, 130.8, 130.5, 129.1, 128.5, 128.0, 127.8, 127.7, 127.6, 113.8, 55.5.

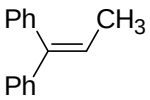
1,1-diphenyl-2-(2-naphtyl)ethene



The general procedure (GP-A), was followed for 2-naphthaldehyde (29.9 mmol, 4.67 g) for 6 h, yielding the title compound (5.77 g, 63%) as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.10-7.25 (17H, m), 6.87 (1H, s).
^{13}C NMR (75 MHz, $CDCl_3$): δ 142.1, 141.4, 140.9, 134.7, 134.0, 131.9, 131.5, 129.7, 128.4, 128.3, 128.1, 127.8, 127.4, 126.9, 126.2, 125.9, 125.7, 125.6, 125.3.

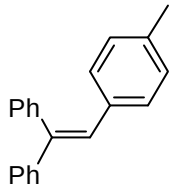
1,1-diphenyl-1-propene[5]



The general procedure (GP-W), was followed for acetaldehyde (33.0 mmol, 1.83 mL) for 12 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the title compound (6.15 g, 96%) as a white solid. Spectroscopic data matched the literature.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.36–7.16 (10H, m), 6.18 (1H, q, J = 7.2 Hz), 1.77 (3H, d, J = 7.2 Hz).
^{13}C NMR (75 MHz, $CDCl_3$): δ 143.0, 142.4, 140.0, 130.1, 128.2, 128.1, 127.2, 126.8, 126.7, 124.1, 15.7.

1,1-diphenyl-2-(4-methylphenyl)ethene[6]

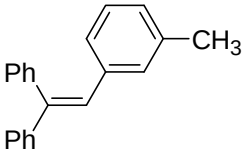


The general procedure (GP-W), was followed for *p*-tolualdehyde (33.0 mmol, 3.89 mL) for 72 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the title compound (4.91 g, 55%) as an oil. Spectroscopic data matched the literature.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25-7.15 (14H, m), 6.93 (1H, s), 2.31 (3H, s).
^{13}C NMR (50 MHz, $CDCl_3$): δ 143.1, 142.7, 142.4, 141.9, 135.5, 134.8, 132.1, 131.3, 128.9, 128.0, 21.3.

[4] Dunet, G.; Knochel, P. *Synlett* **2006**, 407.
[5] Firouzabadi, H.; Iranpoor, N.; Hazarkhani, H.; Karimi, B. *Synth. Commun.* **2003**, *33*, 3653.
[6] Corsico, E. F.; Rossi, R. A. *J. Org. Chem*. **2004**, *69*, 6427.

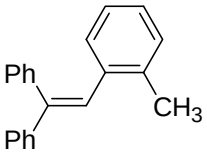
1,1-diphenyl-2-(3-methylphenyl)ethene[7]



The general procedure (GP-G), was followed for *m*-tolyl chloride (30 mmol, 6.8 mL) for 12 h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the title compound (8.28 g, 93%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40-7.15 (11H, m), 7.10-6.70 (4H, m), 2.18 (3H, s).
^{13}C NMR (50 MHz, $CDCl_3$): δ 143.5, 142.4, 140.5, 137.4, 137.3, 130.5, 130.4, 128.6, 128.3, 128.2, 127.8, 127.6, 127.5, 127.4, 127.3, 126.5), 21.3.
IR (KBr): 3024, 2921, 1602, 1492, 1445, 695 cm^{-1}.

1,1-diphenyl-2-(2-methylphenyl)ethene[7]



The general procedure (GP-G), was followed for *o*-tolyl chloride (30 mmol, 7.5 mL) for 2h. Purification of crude mixture by flash chromatography (hexane:EtOAc, from 100:0 to 99:1) gave the desired compound (8.29 g, 93%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.40-6.80 (15H, m); 2.31 (3H, s).
^{13}C NMR (75 MHz, $CDCl_3$): δ 143.6, 143.3, 140.2, 137.0, 130.7, 129.8, 128.2, 128.1, 127.6, 127.3, 127.2, 126.8, 125.3, 20.2.
IR (KBr): 3058, 3022, 1600, 1492, 1445, 731 cm^{-1}.

[7] Kitamura, T.; Kobayashi, S.; Taniguchi, H.; Rappoport, Z. *J. Org. Chem.* **1982**, *47*, 5003.