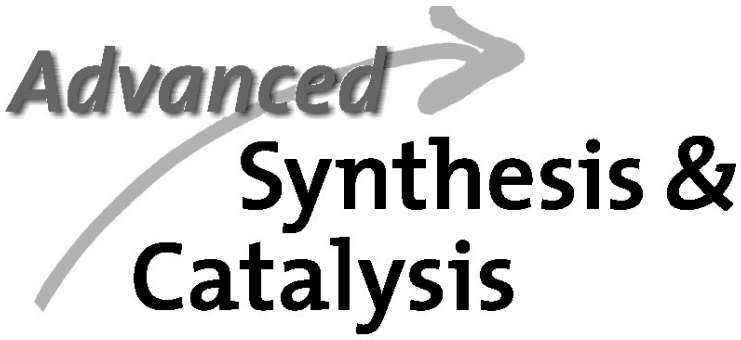

Supporting Information

Mild Conditions for Copper-Catalyzed Coupling Reaction of Phenols and Aryl Iodides and Bromides

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Supporting Information

General Experimental Procedures

All reactions were carried out in 35 mL Schlenk tubes or in Carousel "reaction stations RR98030" Radley tubes, under a pure and dry nitrogen atmosphere. Acetonitrile was distilled from P_4O_{10} and was stored on 4 Å activated molecular sieves under a nitrogen atmosphere. Cesium carbonate (Aldrich) and tri-potassium phosphate (Riedel-de Haën) were ground to a fine powder and stored under vacuum in the presence of P_4O_{10}. All other solid materials were stored in the presence of P_4O_{10} in a bench-top desiccator under vacuum at room temperature and weighed in the air. Copper (I) iodide was purified according to literature procedures[1] and stored protected from light. The synthesis of ligand **1** was reported in our previous papers.[2] Iodobenzene, aryl bromides and phenols were purchased from commercial sources (Aldrich, Acros, Avocado, Fluka, Lancaster). If solids, they were recrystallized in an appropriate solvent.[3] If liquids, they were distilled under vacuum and stored under an atmosphere of nitrogen. Special care was taken with liquid iodobenzene which was regularly distilled and stored protected from light. All phenols were also stored protected from light.
Column chromatography was performed with SDS 60 A C.C silica gel (35-70 μm). Thin layer chromatography was carried out using Merck silica gel 60 F_{254} plates. All products were characterized by their NMR, GC/MS and IR spectra. NMR spectra were recorded at 20°C on a Bruker AC 200 MHz or on a DRX-250 spectrometer working respectively at 200.13 and 250.13 MHz for ^1H, at 50.32 and 62.90 MHz for ^{13}C and at 188.31 and 236.36 for ^{19}F. Coupling constants are reported in Hz and chemical shifts in ppm/TMS for ^1H and $\{^1H\}^{13}C$ (δ 77.00 for $\underline{C}DCl_3$ signal) and in ppm/$CFCl_3$ for $\{^1H\}^{19}F$. The first-order peak patterns are indicated as s (singulet), d (doublet), t (triplet), q (quadruplet). Complex non-first-order signals are indicated as m (multiplet) and broad signals as br. Gas chromatography - mass spectra (GC/MS) were recorded on an Agilent Technologies 6890 N instrument with an Agilent 5973 N mass detector (EI) and a HP5-MS 30 m x 0.25 mm capillary apolar column (Stationary phase: 5 % diphenyldimethylpolysiloxane film, 0.25 μm). GC/MS method: Initial temperature: 45 °C; Initial time: 2 min; Ramp: 2 °C/min until 50 °C then 10 °C/min; Final temperature: 250 °C; Final time: 10 min. IR spectra were recorded on a Nicolet 210 FT-IR instrument (neat, thin film for liquid products and KBr pellet or in carbon tetrachloride solution for solid products). FAB+ mass spectra and HRMS were recorded on a JEOL JMS-DX300 spectrometer (3 keV, xenon) in a *m*-nitrobenzylalcohol matrix. Melting points were determined using a Büchi B-540 apparatus and are uncorrected.

General Procedure for Copper-Catalyzed Coupling Reaction of Phenols and Aryl Bromides (2 mmol scale)

After standard cycles of evacuation and back-filling with dry and pure nitrogen, an oven-dried Radley tube (Carousel "reaction stations RR98030") equipped with a magnetic stirring bar was charged with CuI (38.1 mg, 0.2 mmol), ligand **1** (29.2 mg, 0.1 mmol), the phenol (3.0 mmol), K_3PO_4 (848 mg, 4.0 mmol) and the aryl bromide (2.0 mmol), if a solid. The tube was evacuated, back-filled with nitrogen. If a liquid, aryl bromide was added under a stream of nitrogen by syringe at room temperature, followed by anhydrous and degassed acetonitrile (1.2 mL). The tube was sealed under a positive pressure of nitrogen, stirred and heated to 60 or 80 °C for the required time period. After cooling to room temperature, the mixture was diluted with dichloromethane (~ 20 mL) and filtered through a plug of celite®, the filter cake being further washed with dichloromethane (~ 5 mL). The filtrate was washed twice with water (~ 10 mL x 2). Gathered aqueous phases were twice extracted with dichloromethane (~ 10 mL). Organic layers were gathered, dried on $MgSO_4$, filtered and concentrated in vacuo to yield the crude product obtained was purified by silica gel chromatography with an eluent of hexanes and dichloromethane. The products were characterized by NMR, IR and mass spectra with those of authentic samples.

General Procedure for reactivity comparison of Phenols and Aryl Bromides (0.5 mmol scale)

After standard cycles of evacuation and back-filling with dry and pure nitrogen, an oven-dried Radley tube (Carousel "reaction stations RR98030") equipped with a magnetic stirring bar (12 × 4.5 mm) was charged with CuI (9.5 mg, 0.05 mmol), ligand **1** (7.3 mg, 0. 025 mmol), the phenol (0.75 mmol), K_3PO_4 (212 mg, 1.0 mmol) and the aryl bromide (0.5 mmol), if a solid. The tube was evacuated, back-filled with nitrogen. If a liquid, aryl bromide was added under a stream of nitrogen by syringe at room temperature, followed by anhydrous and degassed acetonitrile (300 µL). The tube was sealed under a positive pressure of nitrogen, stirred and heated to 60 or 80 °C for 24 hours. The reaction mixture was allowed to cool to room temperature and diluted with dichloromethane (5 mL). 65 µL of 1,3-dimethoxybenzene (internal standard) were added. A small sample of the reaction mixture was taken and filtered through a plug of celite®, the filter cake being further washed with dichloromethane. The filtrate was washed three times with water and analyzed by gas chromatography. The GC yields were determined by obtaining the correction factors using authentic samples of the expected products.

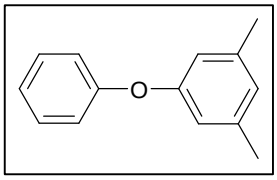
Experimental procedures and characterization data

3,5-dimethyldiphenylether 2a

Experimental procedure

Following the general procedure (80 °C, 24 hours), 3,5-dimethylphenol (366 mg, 3.0 mmol) was coupled with bromobenzene (216 µL, 2.0 mmol). The crude brown oil was purified by flash chromatography on silica gel (eluent : hexanes) to provide 316 mg (80 % yield) of the desired product as a colorless oil.

Identification



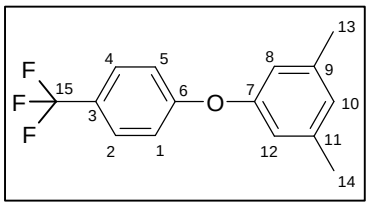
^{1}H NMR (200 MHz, $CDCl_3$): δ[4] 7.28-7.42 (m, 2H), 7.12-7.17 (m, 1H), 7.03-7.14 (m, 2H), 6.79 (m, 1H), 6.69 (m, 2H), 2.33 (s, 6H, CH_3).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ[4] 157.50 (Cq), 157.22 (Cq), 139.61 (2 Cq), 129.70 (2 CH), 125.04 (CH), 123.02 (CH), 118.89 (2 CH), 116.67 (2 CH), 21.35 (2 CH_3).
GC/MS: rt = 51.34 min, M/Z = 198.
Rf: 0.22 (eluent : hexanes).

3',5'-dimethyl-4-trifluoromethyldiphenylether 2b

Experimental procedure

Following the general procedure (80 °C, 24 hours), 3,5-dimethylphenol (366 mg, 3.0 mmol) was coupled with 1-bromo-4-trifluoromethylbenzene (280 µL, 2.0 mmol). The crude product was purified by flash chromatography on silica gel (eluent : hexanes) to provide 442 mg (83 % yield) of the desired product as an orange oil.

Identification



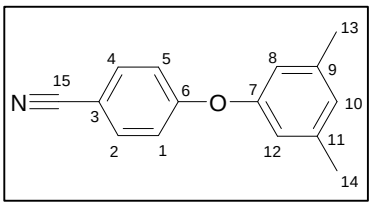
^{1}H NMR (200 MHz, $CDCl_3$): δ 7.59 (m, 2H, $H_{2,4}$), 7.06 (m, 2H, $H_{1,5}$), 6.87 (m, 1H, H_{10}), 6.71 (m, 2H, $H_{8,12}$), 2.35 (s, 6H, $H_{13,14}$).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 160.78 (C_6), 155.65 (C_7), 140.01 ($C_{9,11}$), 127.04 (q, $^{3}J_{CF}$ = 3.8 Hz, $C_{2,4}$), 126.25 (C_{10}), 124.59 (q, $^{2}J_{CF}$ = 32.7 Hz, C_3), 118.92 (q, $^{1}J_{CF}$ = 271.1 Hz, C_{15}), 117.78 ($C_{8,12}$), 117.63 ($C_{1,5}$), 21.26 ($C_{13,14}$).
^{19}F NMR {^{1}H} (235 MHz, $CDCl_3$): δ -62.11 (s).
GC/MS: rt = 50.90 min, M/Z = 266.
IR (CH_2Cl_2) : ν (cm^{-1}) = 3053, 2985, 1615, 1591, 1513, 1326 (CF_3), 1237 (C-O), 1169 (CF_3), 1123, 1066, 840.
Rf: 0.68 (eluent : hexanes).

3',5'-dimethyl-4-cyanodiphenylether 2c

Experimental procedure

Following the general procedure (80 °C, 24 hours), 3,5-dimethylphenol (366 mg, 3.0 mmol) was coupled with 4-bromobenzonitrile (364 mg, 2.0 mmol). The crude product was purified by flash chromatography on silica gel (eluent : hexanes / dichloromethane 100/0 à 50/50) to provide 366 mg (82 % yield) of the desired product as an orange oil.

Identification



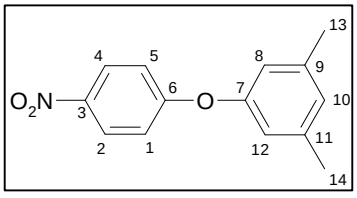
Mp: 58 °C (hexanes / dichloromethane) (Litt.[5]: 58 °C, hexanes / dichloromethane).
^{1}H NMR (200 MHz, $CDCl_3$): δ 7.53-7.60 (m, 2H), 6.95-7.00 (m, 2H), 6.86 (m, 1H), 6.68 (m, 2H), 2.32 (s, 6H, CH_3).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 161.90 (C_6), 154.76 (C_7), 140.17 ($C_{9,11}$), 134.07 ($C_{2,4}$), 126.86 (C_{10}), 118.92 (C_{15}), 118.03 (2 CH), 117.88 (2 CH), 105.55 (C_3), 21.28 ($C_{13,14}$).
GC/MS: rt = 20.54 min, M/Z = 223.
IR (KBr) : ν (cm^{-1}) = 3058, 3017, 2958, 2230 (CN), 1603, 1587, 1504, 1298, 1239 (C-O), 1166, 1134, 1024, 950, 859, 838.
Rf: 0.32 (eluent : hexanes / dichloromethane 50/50).

3',5'-dimethyl-4-nitrodiphenylether 2d

Experimental procedure

Following the general procedure (80 °C, 24 hours), phenol (366 mg, 3.0 mmol) was coupled with 4-bromonitrobenzene (404 mg, 2.0 mmol). The crude product was purified by flash chromatography on silica gel (eluent : hexanes) to provide 355 mg (73 % yield) of the desired product as a light yellow solid, that can be recrystallized in light petroleum / diethylether.

Identification



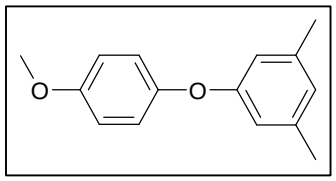
Mp: 59 °C (light petroleum / diethylether) (Litt.[6]: 60 °C, MeOH).
^{1}H NMR (200 MHz, $CDCl_3$): δ 8.1-8.25 (m, 2H), 7-7.05 (m, 2H), 6.91 (m, 1H), 6.73 (m, 2H), 2.29 (s, 6H, CH_3).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 163.62 (C_6), 154.61 (C_7), 142.64 (C_3), 140.27 ($C_{9,11}$), 127.08 ($C_{2,4}$), 125.88 (C_{10}), 118.10 ($C_{8,12}$), 117.01 ($C_{1,5}$), 21.27 ($C_{13,14}$).
GC/MS: rt = 62.89 min, M/Z = 243.
Rf: 0.37 (eluent : hexanes).

3',5'-dimethyl-4-methoxydiphenylether 2e

Experimental procedure

Following the general procedure (80 °C, 24 hours), 3,5-dimethylphenol (366 mg, 3.0 mmol) was coupled with 4-bromoanisole (252 µL, 2.0 mmol). The crude product was purified by flash chromatography on silica gel (eluent : hexanes) to provide 310 mg (68 % yield) of the desired product as a colorless solid that can be recrystallized in light petroleum.

Identification



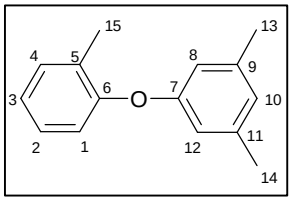
Mp: 67 °C (light petroleum) (Litt.[7]: 67 °C).
^{1}H NMR (200 MHz, $CDCl_3$): δ 6.94-7.00 (m, 2H), 6.86-6.92 (m, 2H), 6.70 (m, 1H), 6.57 (m, 2H), 3.81 (s, 3H, MeO), 2.27 (s, 6H, Me).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 158.52 (Cq), 155.79 (Cq), 150.35 (Cq), 139.45 (2 Cq), 124.24 (CH), 120.82 (2 CH), 115.36 (2 CH), 114.82 (2 CH), 55.64 (CH_3), 21.39 (2 CH_3).
GC/MS: rt = 60.04 min, M/Z = 228.
Rf: 0.61 (eluent : hexanes).

2,3',5'-trimethyldiphenylether 2f

Experimental procedure

Following the general procedure (80 °C, 85 hours), 3,5-dimethylphenol (366 mg, 3.0 mmol) was coupled with 2-bromotoluene (240 µL, 2.0 mmol). The crude oily residue was purified by flash chromatography on silica gel (eluent : hexanes) to provide 233 mg (55 % yield) of the desired product as an orange oil.

Identification



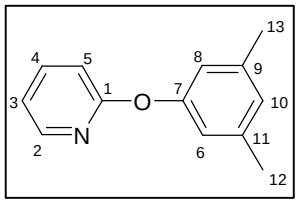
^{1}H NMR (200 MHz, $CDCl_3$): δ[8] 7.08-7.33 (m, 3H), 6.95-6.99 (m, 1H), 6.76 (m, 1H), 6.61 (m, 2H, $H_{8,12}$), 2.33 (s, 6H, $H_{13,14}$), 2.32 (s, 3H, H_{15}).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ[8] 157.94 (Cq), 154.69 (Cq), 139.55 ($C_{9,11}$), 131.41 (CH), 130.02 (C_5), 127.14 (CH), 124.22 (CH), 123.83 (CH), 119.81 (CH), 115.11 ($C_{8,12}$), 21.42 ($C_{13,14}$), 16.30 (C_{15}).
GC/MS: rt = 53.97 min, M/Z = 212.
Rf: 0.25 (eluent : hexanes).

2-(3,5-dimethylphenoxy)-pyridine 2g

Experimental procedure

Following the general procedure (80 °C, 24 hours), 3,5-dimethylphenol (366 mg, 3.0 mmol) was coupled with 2-bromopyridine (194 µL, 2.0 mmol). The crude oily residue was purified by flash chromatography on silica gel (eluent : hexanes / dichloromethane 100/0 à 85/15) to provide 386 mg (97 % yield) of the desired product as a yellow oil.

Identification



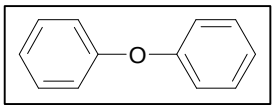
^{1}H NMR (250 MHz, $CDCl_3$): δ 8.21 (ddd, 1H, $^3J_{HH}$ = 5.0 Hz, $^4J_{HH}$ = 2.0 Hz, $^5J_{HH}$ = 0.7 Hz, H_2), 7.66 (ddd, 1H, $^3J_{HH}$ = 8.2 Hz, $^3J_{HH}$ = 7.2 Hz, $^4J_{HH}$ = 2.0 Hz, H_4), 6.97 (ddd, 1H, $^3J_{HH}$ = 7.2 Hz, $^3J_{HH}$ = 5.0 Hz, $^4J_{HH}$ = 0.9 Hz, H_3), 6.88 (ddd, 1H, $^3J_{HH}$ = 8.2 Hz, $^4J_{HH}$ = 0.9 Hz, $^5J_{HH}$ = 0.7 Hz, H_5), 6.84 (m, 1H, H_{10}), 6.76 (m, 2H, $H_{6,8}$), 2.32 (s, 6H, $H_{12,13}$).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 163.78 (C_1), 154.21 (C_7), 147.79 (C_2), 139.40 ($C_{9,11}$), 129.68 (C_4), 124.65 (C_{10}), 121.16 ($C_{6,8}$), 118.46 (C_5), 111.53 (C_3), 21.34 ($C_{12,13}$).
GC/MS: rt = 54.75 min, M/Z = 199.
IR (KBr) : ν (cm^{-1}) = 3057, 3051, 3013, 2918, 2856, 1616, 1583, 1572, 1467, 1429, 1296, 1246 (C-O), 1138, 856, 779.
Rf: 0.22 (eluent : hexanes / dichloromethane 75/25).

Diphenylether 2h

Experimental procedure

Following the general procedure (80 °C, 24 hours), phenol (282 mg, 3.0 mmol) was coupled with bromobenzene (216 µL, 2.0 mmol). The crude oily residue was purified by flash chromatography on silica gel (eluent : hexanes) to provide 299 mg (88% yield) of the desired product as a colorless oil, that can be crystallized in a few hours if left at -5 °C (colorless crystals).

Identification



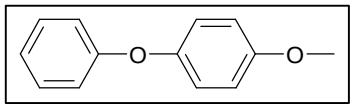
Mp: 26 °C (Litt.[9] : 26.85 °C).
^{1}H NMR (200 MHz, $CDCl_3$): δ 7.37-7.47 (m, 4H), 7.10-7.23 (m, 6H).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 157.38 (2 Cq), 129.88 (4 CH), 123.35 (2 CH), 119.02 (4 CH).
GC/MS: rt = 47.84 min, M/Z = 170.
Rf: 0.35 (eluent : hexanes).

4-methoxydiphenylether 2i

Experimental procedure

Following the general procedure (80 °C, 24 hours), 4-hydroxyanisole (372 mg, 3.0 mmol) was coupled with bromobenzene (216 µL, 2.0 mmol). The crude oil was purified by flash chromatography on silica gel (eluent : hexanes / dichloromethane 100/0 to 95/5) to provide 352 mg (88 % yield) of the desired product as a colorless oil.

Identification



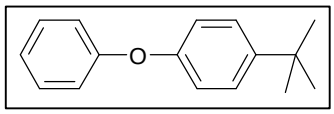
^{1}H NMR (200 MHz, $CDCl_3$): δ 7.30-7.39 (m, 2H), 6.89-7.09 (m, 7H), 3.84 (s, 3H).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 158.60 (Cq), 155.97 (Cq), 150.18 (Cq), 129.69 (2 CH), 122.49 (CH), 120.91 (2 CH), 117.64 (2 CH), 114.92 (2 CH), 55.67 (CH_3).
GC/MS: rt = 55.04 min, M/Z = 200.
Rf: 0.25 (eluent : hexanes / dichloromethane 80/20).

4-*t*-butyldiphenylether 2j

Experimental procedure

Following the general procedure (80 °C, 24 hours), 4-*t*-butylphenol (450 mg, 3.0 mmol) was coupled with bromobenzene (216 µL, 2.0 mmol). The crude oily residue was purified by flash chromatography on silica gel (eluent : hexanes) to provide 316 mg (70 % yield) of the desired product as a colorless oil, that can be crystallized in a few hours if left at -5 °C (colorless crystals).

Identification



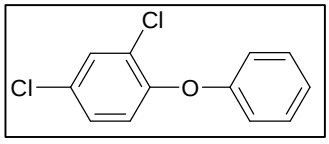
Mp: 52 °C (Litt.[10]: 53-54 °C).
^{1}H NMR (200 MHz, DMSO-d_6): δ 7.33-7.41 (m, 4H), 7.06-7.14 (m, 1H), 6.91-6.99 (m, 4H), 1.27 (s, 9H, Me).
^{13}C NMR {^{1}H} (50 MHz, DMSO-d_6): δ 156.94 (Cq), 154.09 (Cq), 145.73 (Cq), 129.88 (2 CH), 126.61 (2 CH), 123.05 (CH), 118.21 (4 CH), 33.96 (Cq), 31.18 (3 CH_3).
GC/MS: rt = 57.50 min, M/Z = 226.
Rf: 0.35 (eluent : hexanes).

2,4-dichlorodiphenylether 2k

Experimental procedure

Following the general procedure (80 °C, 24 hours), 2,4-dichlorophenol (489 mg, 3.0 mmol) was coupled with bromobenzene (216 µL, 2.0 mmol). The crude product was purified by flash chromatography on silica gel (eluent : hexanes) to provide 295 mg (62 % yield) of the desired product as a colorless oil.

Identification



^{1}H NMR (200 MHz, $CDCl_3$): δ 7.50 (dd, 1H, $^{4}J_{HH}$ = 2.3 Hz, $^{5}J_{HH}$ = 0.6 Hz, H_4), 7.34-7.42 (m, 2H), 7.15-7.19 (m, 2H), 6.90-7.02 (m, 3H).
^{13}C NMR {^{1}H} (50 MHz, $CDCl_3$): δ 156.66 (Cq), 151.49 (Cq), 130.52 (CH), 129.96 (2 CH), 129.16 (Cq), 128.08 (CH), 126.65 (Cq), 123.78 (CH), 121.39 (CH), 118.07 (2 CH).
GC/MS: rt = 57.31 min, M/Z = 238, 240 and 242.
Rf: 0.40 (eluent : hexanes).

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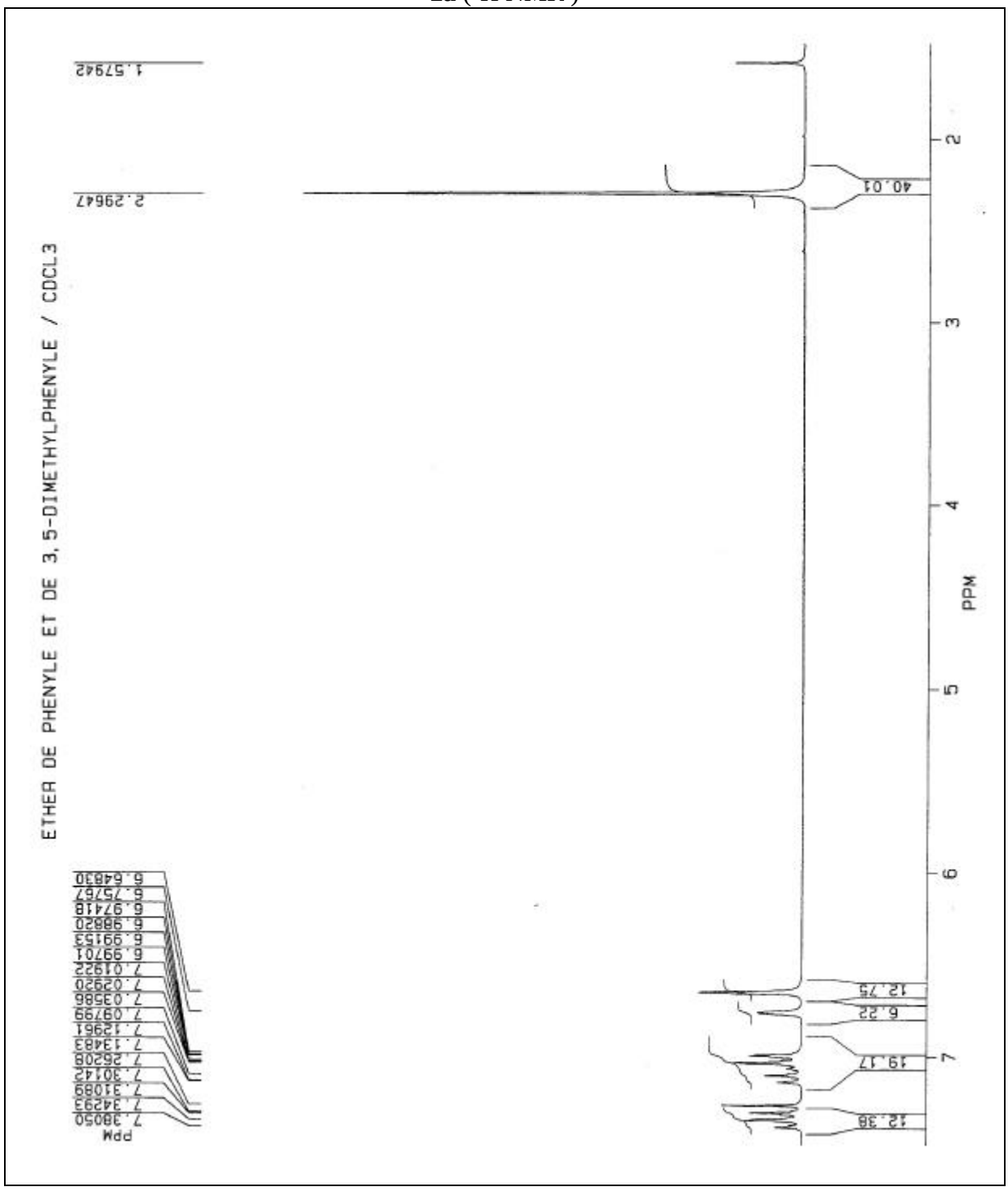
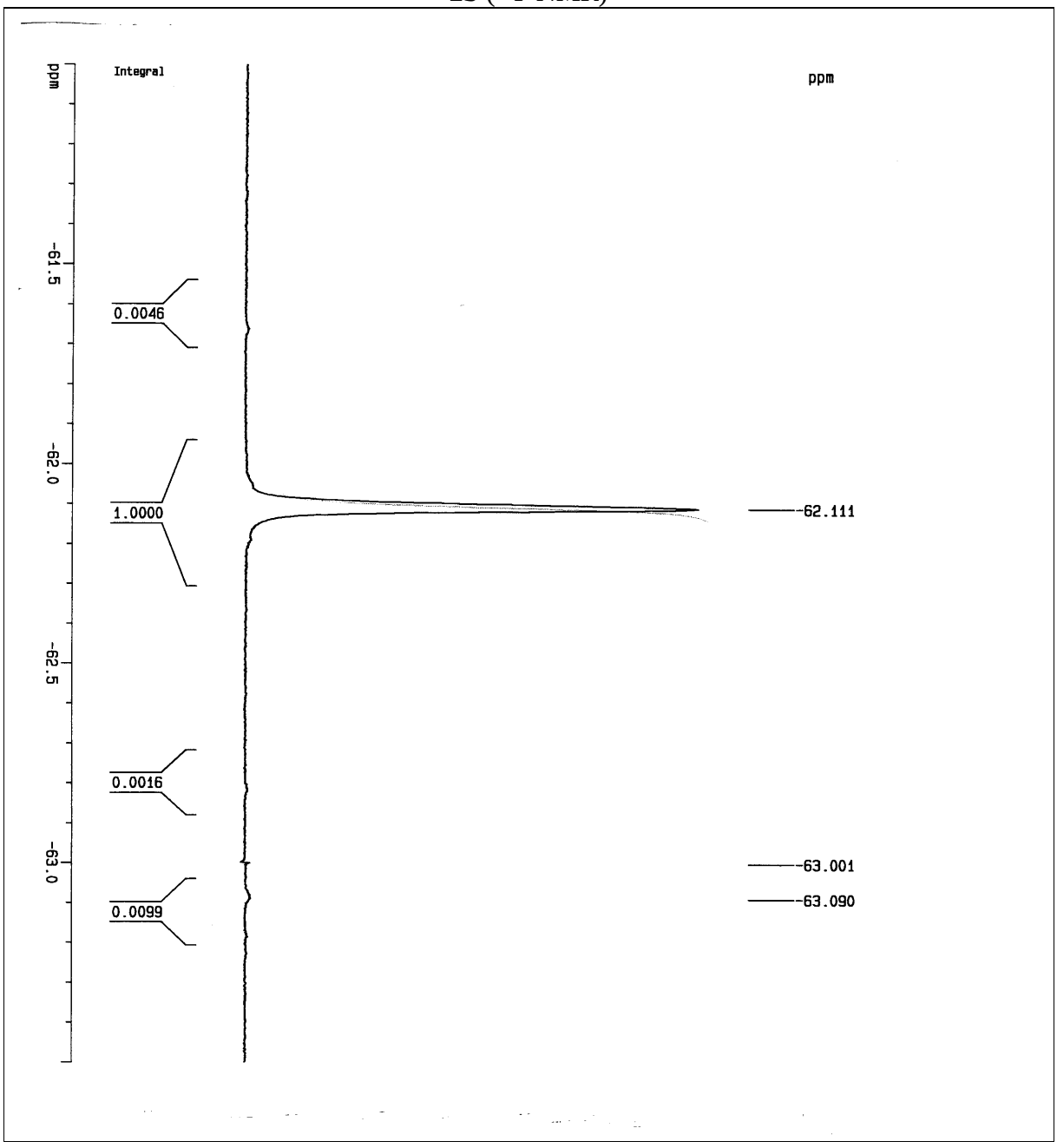
Some selected NMR spectra:

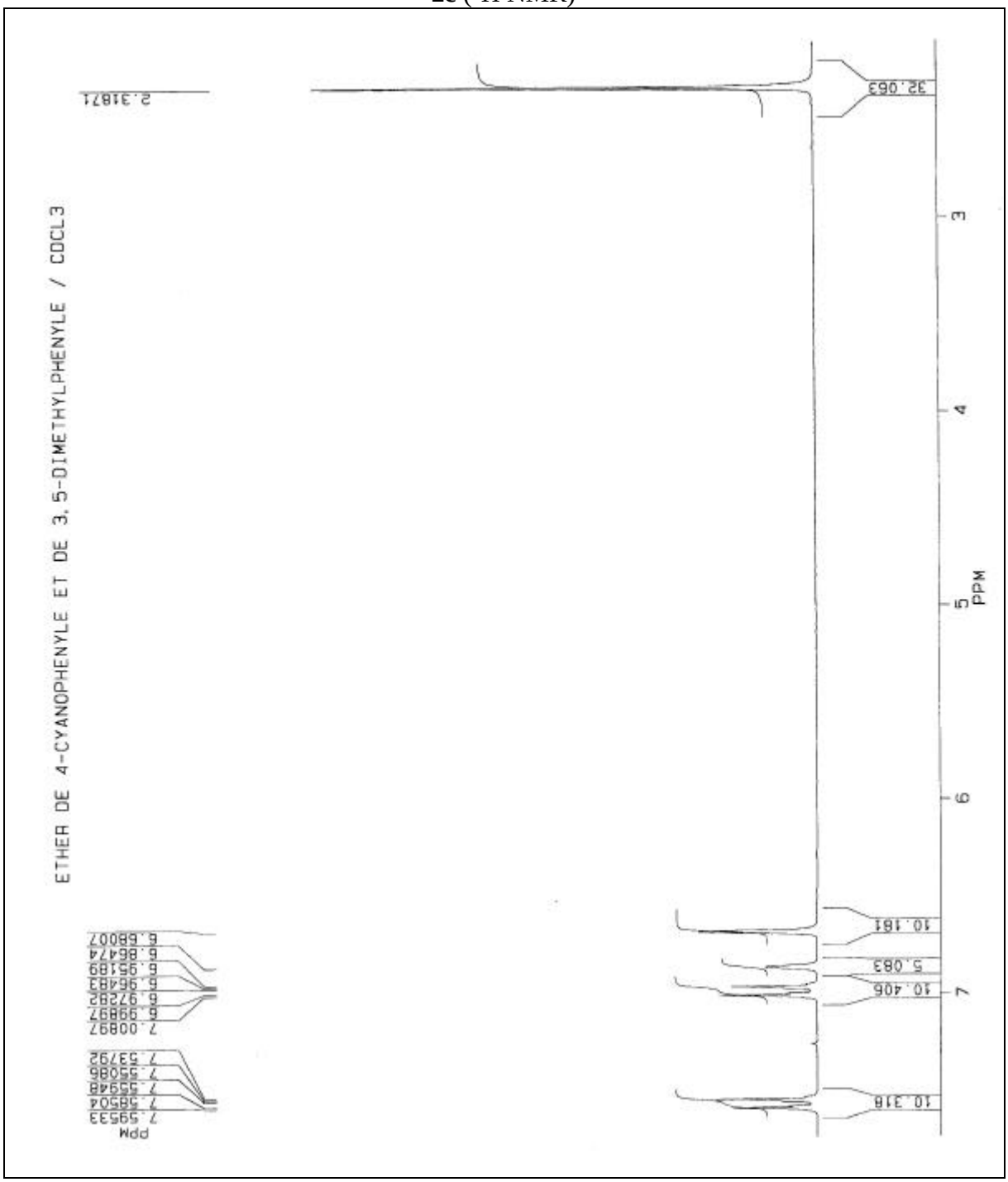
2a (^{1}H NMR)



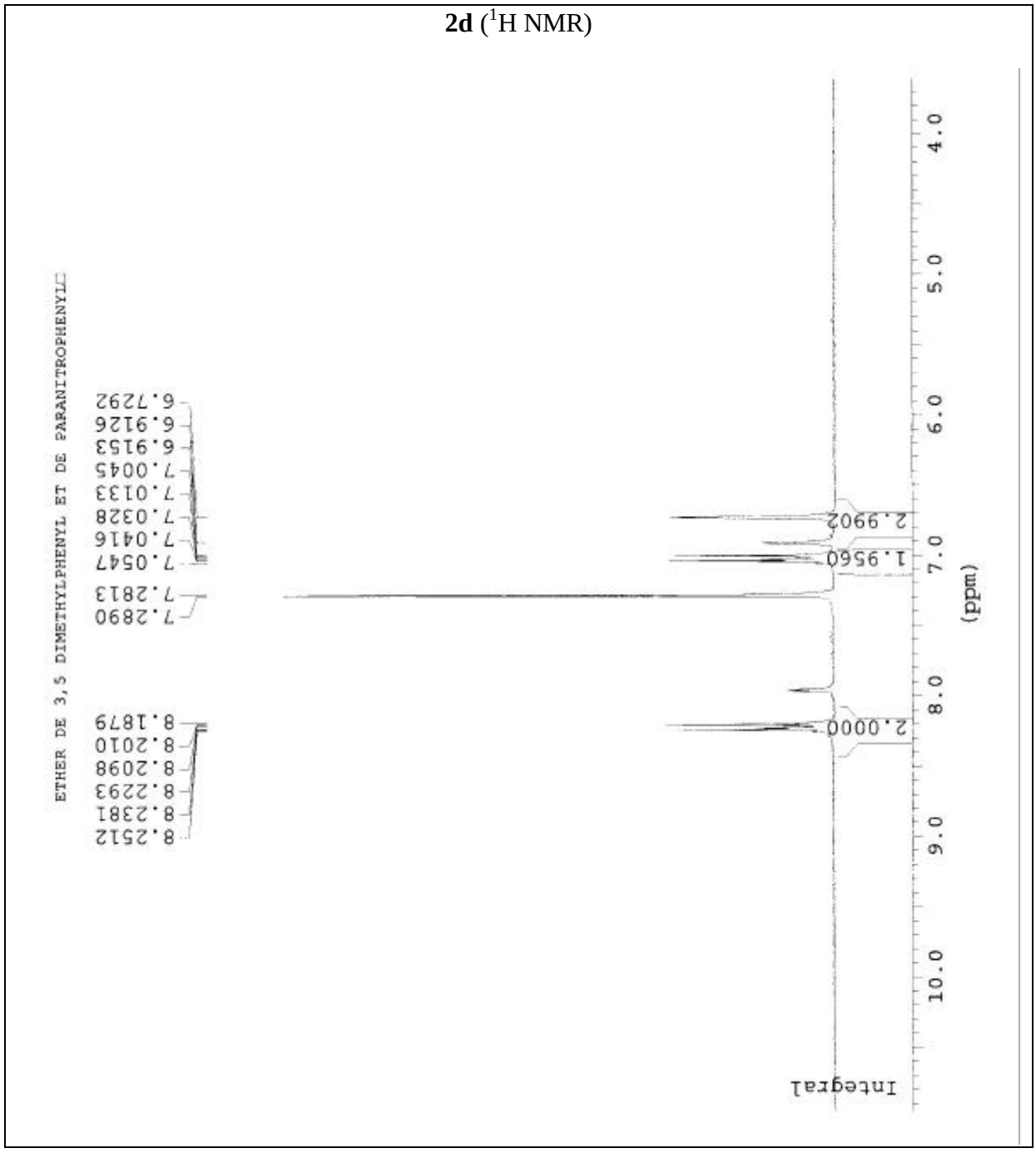
2b (^{19}F NMR)


Integral
ppm
ppm
-61.5
-62.0
-62.5
-63.0
0.0046
1.0000
0.0016
0.0099
-62.111
-63.001
-63.090

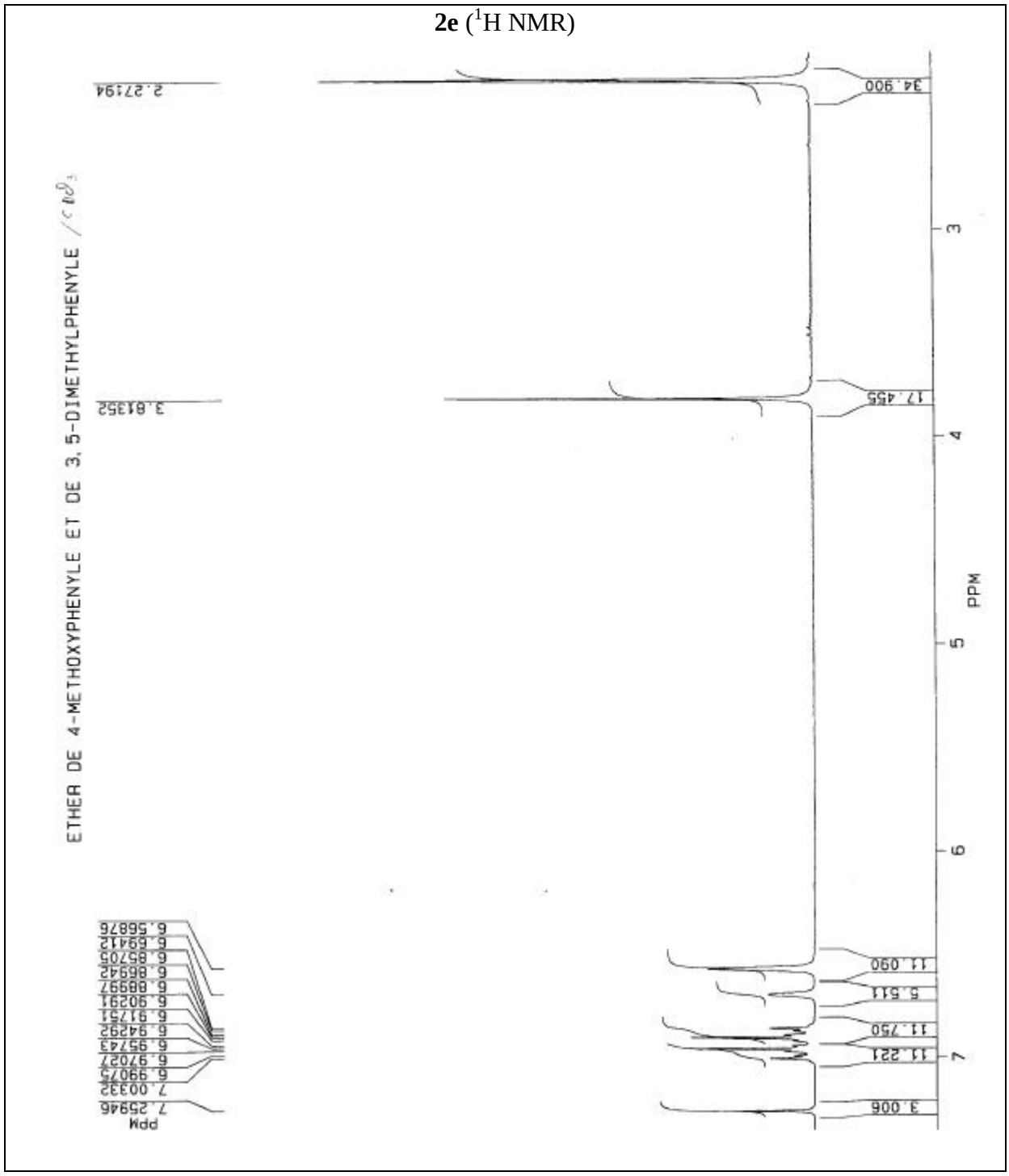
2c (^{1}H NMR)


ETHER DE 4-CYANOPHENYLE ET DE 3,5-DIMETHYLPHENYLE / CDCL3
2.31871
32.063
10.181
5.083
10.406
10.318
PPM
3
4
5
6
7
ppm

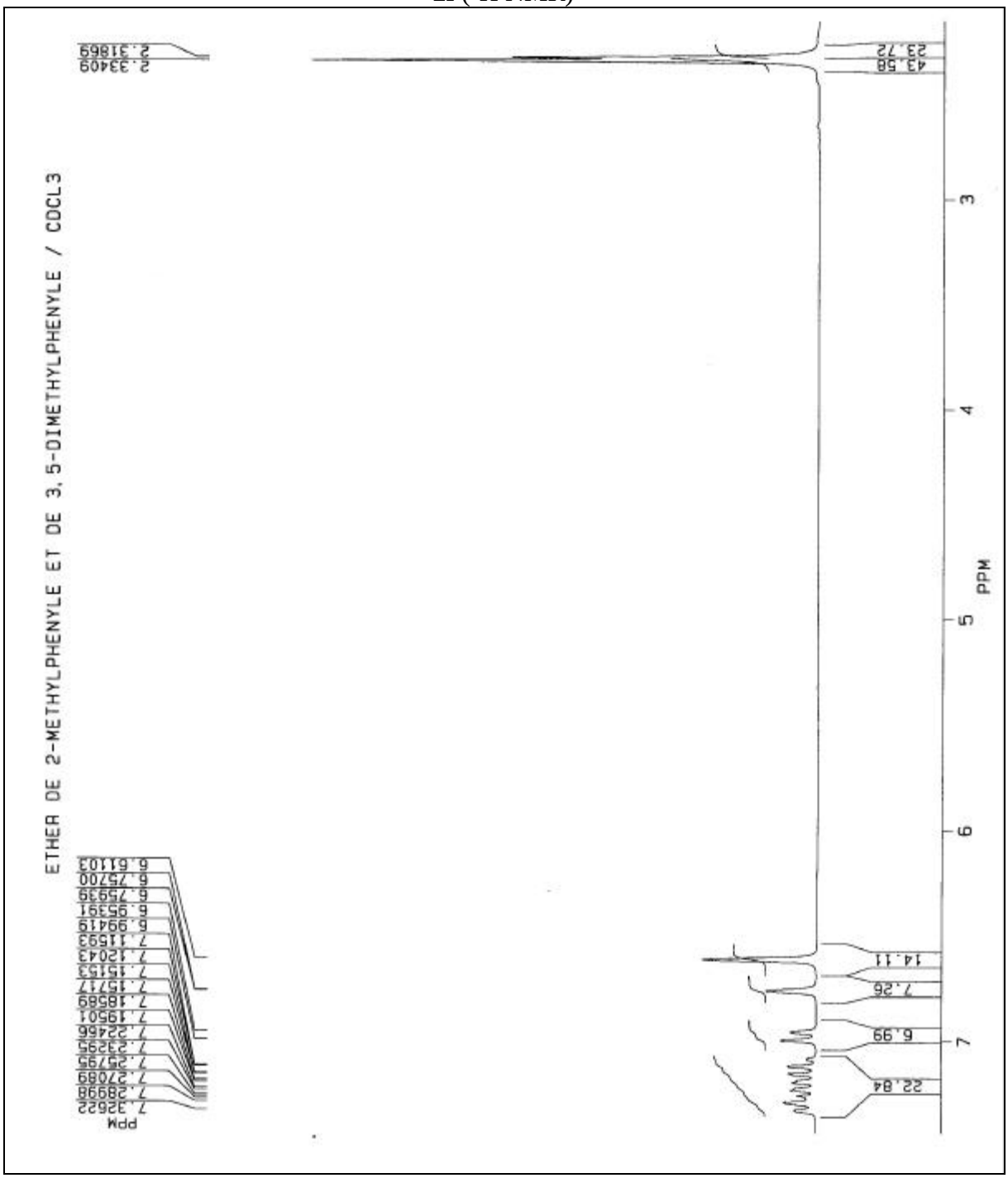
2d (^{1}H NMR)


ETHER DE 3,5 DIMETHYLPHENYL ET DE PARANITROPHENYL
8.2512
8.2381
8.2293
8.2098
8.2010
8.1879
7.2890
7.2813
7.0547
7.0416
7.0328
7.0133
7.0045
6.9153
6.9126
6.7292
2.9902
1.9560
2.0000
Integral
4.0
5.0
6.0
7.0
8.0
9.0
10.0
(ppm)

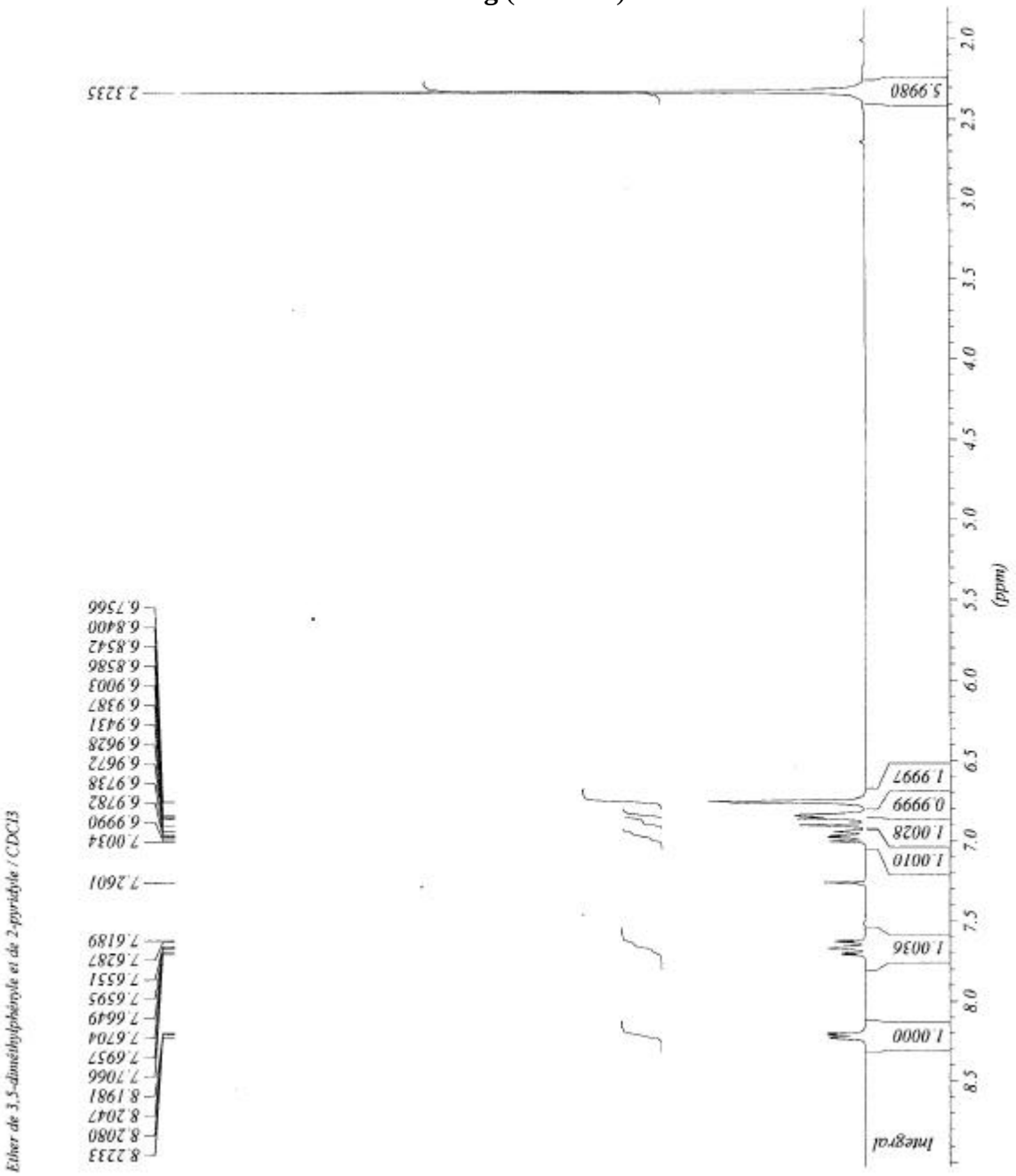
2e (^{1}H NMR)


ETHER DE 4-METHOXYPHENYLE ET DE 3,5-DIMETHYLPHENYLE
2.27194
3.81352
34.900
17.455
11.090
5.511
11.750
11.221
3.006
7.25946
7.00332
6.99075
6.97027
6.95743
6.94292
6.91751
6.90291
6.88997
6.86942
6.85705
6.69412
6.56876
PPM
3
4
5
6
7

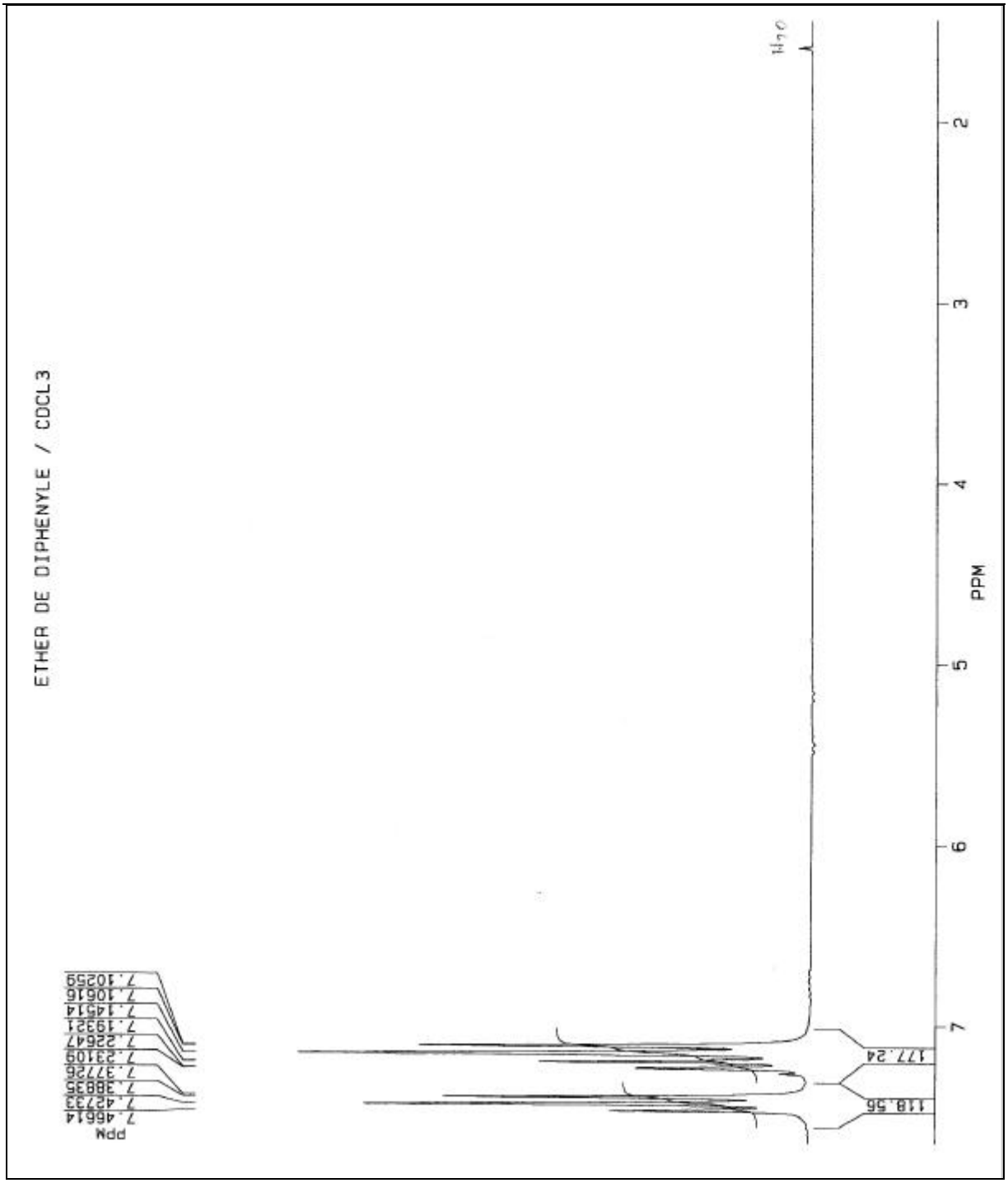
2f (^{1}H NMR)


ETHER DE 2-METHYLPHENYLE ET DE 3,5-DIMETHYLPHENYLE / CDCL3
2.33409
2.31869
43.58
23.72
14.11
7.26
6.99
22.84
7.32622
7.28998
7.27089
7.25795
7.23295
7.22466
7.19501
7.18589
7.15717
7.15153
7.12043
7.11593
6.99419
6.95391
6.79339
6.75700
6.61103
PPM
PPM

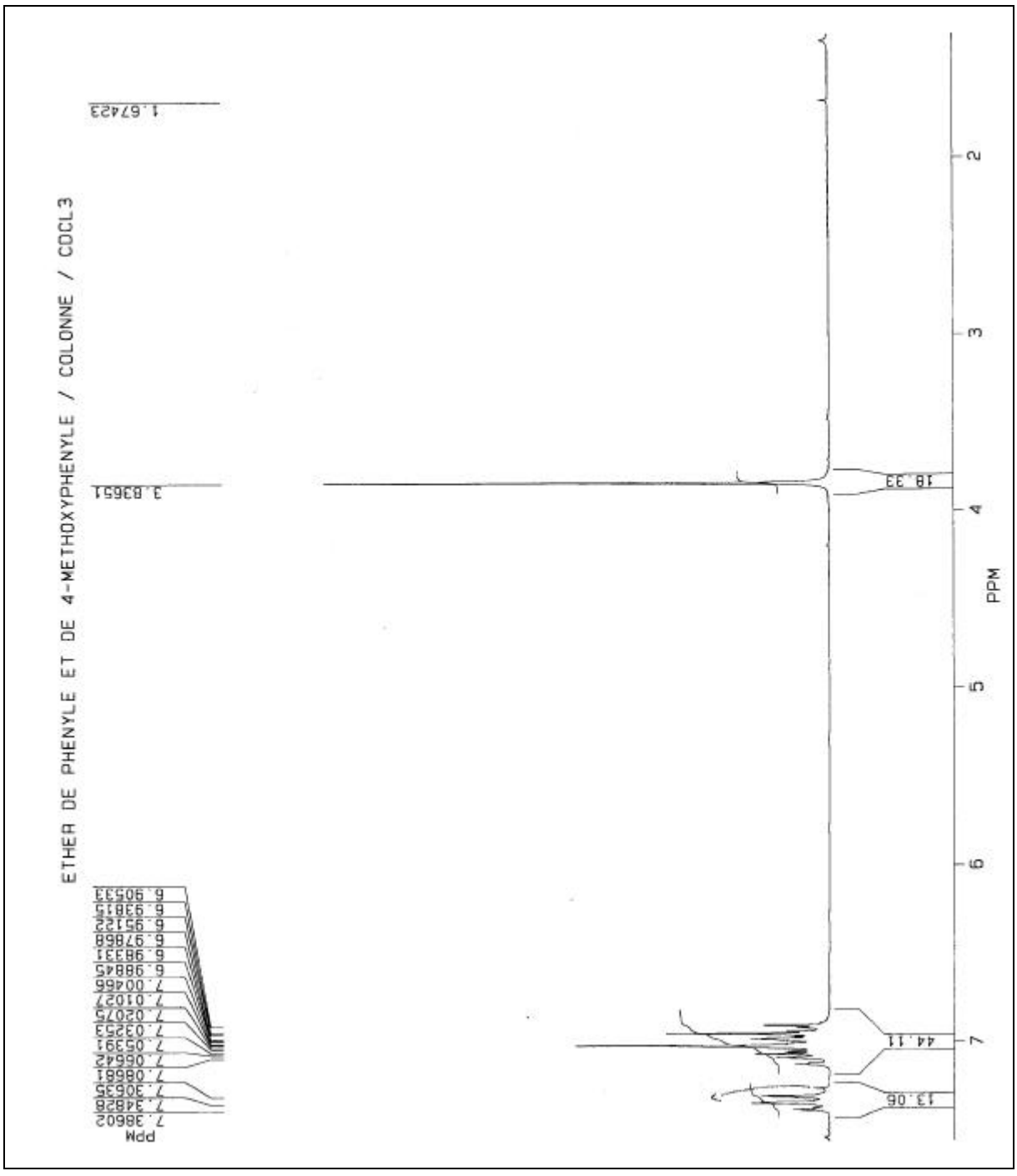
2g (^{1}H NMR)


Ether de 3,5-diméthylphényle et de 2-pyridyle / CDCl3
2.3235
5.9980
6.7566
6.8400
6.8542
6.8586
6.9003
6.9387
6.9431
6.9628
6.9672
6.9738
6.9782
6.9990
7.0034
7.2601
7.6189
7.6287
7.6551
7.6595
7.6649
7.6704
7.6957
7.7066
8.1981
8.2047
8.2080
8.2233
1.9997
0.9999
1.0028
1.0010
1.0036
1.0000
Integral
(ppm)

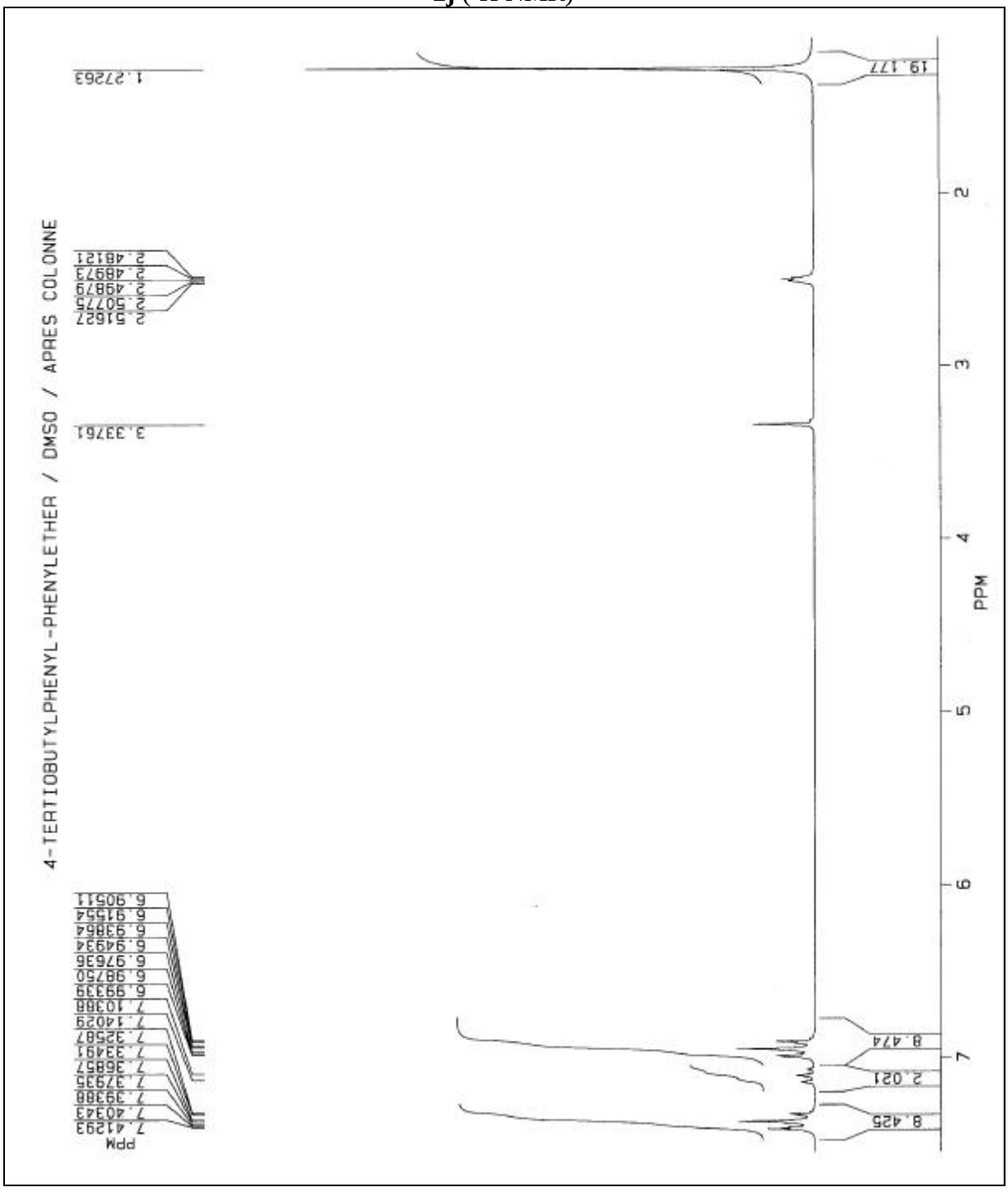
2h (^{1}H NMR)


ETHER DE DIPHENYLE / CDCL3
PPM
2
3
4
5
6
7
177.24
118.55
7.46614
7.42723
7.38836
7.37726
7.23109
7.22647
7.19921
7.14514
7.10816
7.10259
PPM

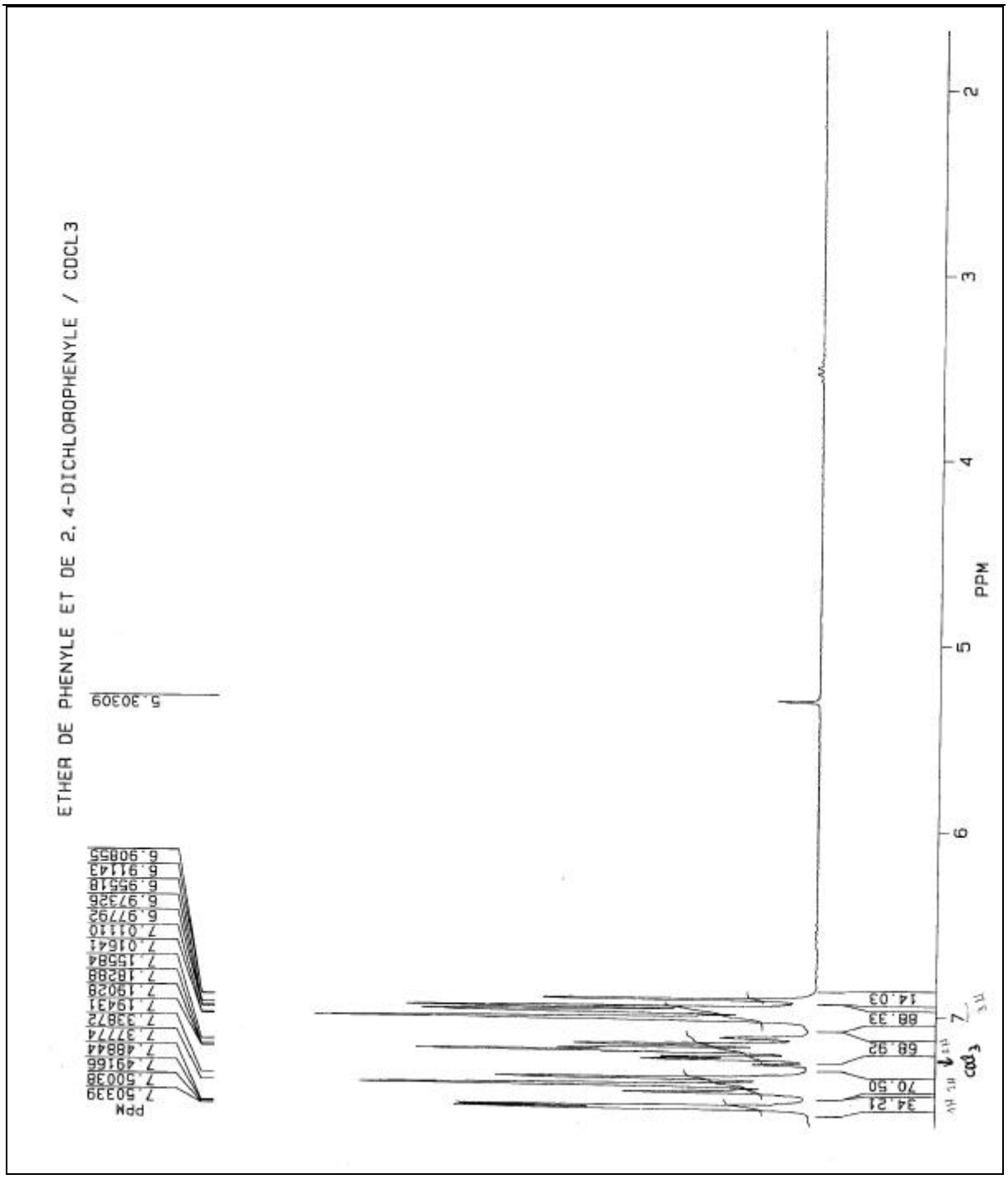
2i (^{1}H NMR)


ETHER DE PHENYLE ET DE 4-METHOXYPHENYLE / COLONNE / COCL3
PPM
18.33
44.11
13.06
3.83651
1.67423

2j (^{1}H NMR)



2k (^{1}H NMR)


ETHER DE PHENYLE ET DE 2,4-DICHLOROPHENYLE / CDCL3
5.30309
7.50339
7.50038
7.49166
7.48844
7.37774
7.33872
7.19431
7.19028
7.18288
7.15584
7.01641
7.01110
6.97792
6.97326
6.95518
6.91143
6.90855
PPM
14.03
68.33
68.92
70.50
34.21
CDCL3