Supporting Information

Palladium-catalysed synthesis of 1-isoindolecarboxylic acid esters and sequential Diels-Alder reactions: access to bridged- and fused-ring heterocycles

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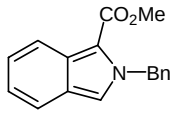
General Methods. All commercially available reagents were used without further purification. Unless otherwise noted ^{1}H- and ^{13}C NMR spectra were recorded in $CDCl_3$ solution, using Me_4Si as the internal standard, with a Varian Gemini 300 or a Varian Mercury 400 instrument. Chemical shifts are reported in ppm downfield (δ) from Me_4Si. TLC was carried out on SiO_2 (silica gel 60 F_{254}), and the spots were located with UV light, iodoplatinate reagent or 1% aqueous $KMnO_4$. Flash chromatography was carried out on SiO_2 (silica gel 60, 230-400 mesh ASTM). Drying of organic extracts during workup of reactions was performed over anhydrous $MgSO_4$. Evaporation of solvents was accomplished with a rotatory evaporator.

Experimental procedure and characterization data for new compounds of Schemes 1-3 and Table 1

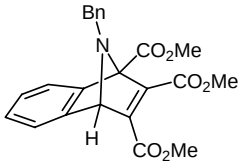
General Procedure: A mixture of ester **1b** (75 mg, 0.18 mmol), K_3PO_4 (115 mg, 0.54 mmol), phenol (6 mg, 0.06 mmol), and $Pd(PPh_3)_4$ (21 mg, 0.018 mmol) in DMF (3 mL) was stirred at 90 ºC for 24 h. The reaction mixture was poured into water and extracted with Et_2O. The organic extracts were washed with brine and 1N NaOH solution, dried, and concentrated.

The crude reaction mixture was dissolved in CH_2Cl_2 (10 mL) and DMAD (0.05 mL, 0.38 mmol) was added. The mixture was stirred at reflux for 12 h. The solvent was removed *in vacuo* and the residue was purified by flash chromatography (SiO_2, from hexanes to 1:1 hexanes-EtOAc) to give cycloadduct **3b** (40 mg, 51%).



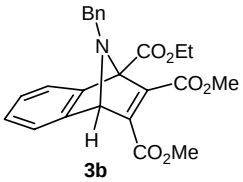
2a

Methyl 2-benzylisoindole-1-carboxylate (2a). Flash chromatography of the α-arylation reaction mixture (SiO_2, CH_2Cl_2) gave isoindole **2a** (39 mg, 76%). ^{1}H NMR (C_6D_6, 300 MHz) δ 3.53 (s, 3H), 5.55 (s, 2H), 6.75 (s, 1H), 6.91-7.10 (m, 6H), 7.25 (ddd, J = 8.7, 6.6, and 1.2 Hz, 1H), 7.48 (dt, J = 8.7 and 1.2 Hz, 1H), 8.40 (dm, J = 8.7 Hz, 1H). ^{13}C NMR (C_6D_6, 75.5 MHz) δ 50.5 (CH_3), 53.6 (CH_2), 111.6 (C), 120.5 (CH), 122.0 (CH), 122.2 (CH), 122.4 (CH), 124.6 (C), 125.6 (CH), 127.4 (CH), 127.7 (CH), 128.8 (CH), 129.9 (C), 138.0 (C), 162.0 (C). HRMS (ESI-TOF) cald for $C_{17}H_{15}NO_2$: 266.1176 $[M+H]^+$; found: 266.1172.



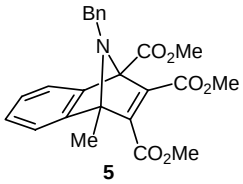
3a

Diels-Alder adduct 3a. ^{1}H NMR ($CDCl_3$, 400 MHz, 50 ºC) δ 3.50 (broad, 1H), 3.67 (broad d, *J* = 12.8 Hz, 1H), 3.73 (s, 3H), 3.76 (s, 3H), 3.78 (s, 3H), 4.84 (broad s, 1H), 7.10 (m, 2H), 7.21-7.33 (m, 6H), 7.63 (broad, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz, 50 ºC) δ 52.0 (CH_3), 52.1 (CH_2), 52.2 (CH_3), 52.5 (CH_3), 71.3 (CH), 84.1 (broad C), 122.8 (broad CH), 124.0 (broad CH), 126.0 (CH), 126.2 (CH), 127.4 (CH), 128.3 (CH), 129.3 (CH), 136.9 (C), 144.7 (broad C), 146.2 (C), 148.5 (broad C), 151.2 (broad C), 162.9 (C), 163.9 (broad C), 166.8 (C). IR (neat) ν 1743, 1717, 1640, 1454, 1435, 1309, 1257, 1198, 1148, 1110 cm^{-1}. HRMS (ESI-TOF) cald for $C_{23}H_{22}NO_6$: 408.1442 $[M+H]^+$; found: 408.1447.



3b

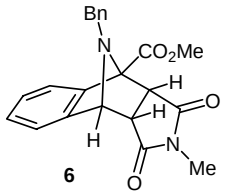
Diels-Alder adduct 3b. ^{1}H NMR ($CDCl_3$, 400 MHz, 50 ºC) δ 1.32 (t, *J* = 7.2 Hz, 3H), 3.50 (broad, 1H), 3.68 (d, *J* = 12.8 Hz, 1H), 3.72 (s, 3H), 3.79 (s, 3H), 4.18-4.33 (m, 2H), 4.83 (s, 1H), 7.07 (td, *J* = 7.6 and 0.8 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 7.21-7.32 (m, 6H), 7.63 (broad, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz, 50 ºC) δ 14.1 (CH_3), 51.9 (CH_2), 52.1 (CH_3), 52.2 (CH_3), 61.8 (CH_2), 71.1 (CH), 84.1 (C), 122.7 (broad CH), 124.3 (CH), 126.0 (CH), 126.2 (CH), 127.4 (CH), 128.3 (CH), 129.3 (CH), 137.0 (C), 144.9 (broad C), 146.3 (C), 151.5 (broad C), 162.9 (broad C), 164.3 (C), 164.1 (broad C), 166.2 (C). IR (neat) ν 1719, 1632, 1435, 1308, 1254, 1255, 1107, 747 cm^{-1}. HRMS (ESI-TOF) cald for $C_{24}H_{24}NO_6$: 422.1598 $[M+H]^+$; found: 422.1599.



5

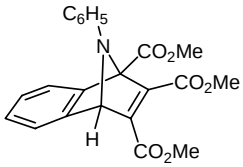
Diels-Alder adduct 5. ^{1}H NMR ($CDCl_3$, 400 MHz, 50 ºC) δ 1.69 (s, 3H), 3.40 (s, 3H), 3.47 (broad d, *J* = 14 Hz, 1H), 3.60 (broad d, *J* = 14 Hz, 1H), 3.71 (s, 3H), 3.76 (s, 3H), 7.09-7.33 (m, 8H), 7.69 (broad, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz, 50 ºC) δ 12.1 (CH_3), 50.1 (CH_3), 51.9 (CH_2), 52.0 (2 CH_3), 78.8 (C), 81.8 (C), 121.2 (broad CH), 124.2 (broad CH), 126.0 (CH), 126.1 (CH), 126.9 (CH), 128.0 (CH), 129.0 (CH), 138.3 (C), 145.5 (C), 149.0 (C), 149.4 (C), 153.4

(broad C), 163.4 (broad C), 164.3 (C), 167.1 (C). IR (CH_2Cl_2) ν 1743, 1721, 1634, 1451, 1305, 1257, 1197, 1157, 1128, 1028, 757 cm^{-1}. HRMS (ESI-TOF) cald for $C_{24}H_{24}NO_6$: 422.1598 $[M+H]^+$; found: 422.1599.



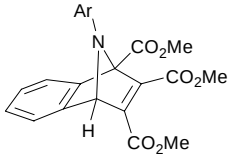
6

Diels-Alder adduct 6. ^1H NMR ($CDCl_3$, 300 MHz, only the most significant resonances from a mixture with 20% of the retro products are reported) δ 2.26 (s, 3H), 3.15 (d, *J* = 12.9 Hz, 1H), 3.54 (d, *J* = 12.9 Hz, 1H), 3.72 (dd, *J* = 8.1 and 5.4 Hz, 1H), 3.86 (s, 3H), 4.12 (d, *J* = 8.1 Hz, 1H), 4.55 (d, *J* = 5.4 Hz, 1H), 7.63 (dd, *J* = 6 Hz and 1.8 Hz, 1H).



8a

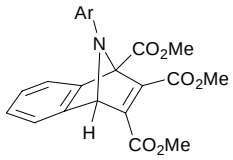
Diels-Alder adduct 8a. ^1H NMR ($CDCl_3$, 400 MHz) δ 3.71 (s, 3H), 3.84 (s, 3H), 3.88 (s, 3H), 5.62 (s, 1H), 6.89 (d, *J* = 7.2 Hz, 2H), 7.00 (t, *J* = 7.2 Hz, 2H), 7.10 (m, 2H), 7.18-7.24 (m, 2H), 7.39 (dd, *J* = 5.6 and 2 Hz, 1H), 7.57 (dd, *J* = 5.6 and 2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 52.4 (CH_3), 52.5 (CH_3), 53.1 (CH_3), 74.2 (CH), 82.6 (C), 121.0 (CH), 122.1 (CH), 122.4 (CH), 123.6 (CH), 126.1 (CH), 126.6 (CH), 129.0 (CH), 144.4 (C), 145.3 (C), 145.6 (C), 146.1 (C), 151.7 (C), 162.5 (C), 164.3 (C), 167.2 (C). IR (CH_2Cl_2) ν 1743, 1720, 1639, 1596, 1493, 1435, 1252, 1200, 1146, 1113, 757 cm^{-1}. HRMS (ESI-TOF) cald for $C_{22}H_{20}NO_6$: 394.1285 $[M+H]^+$; found: 394.1291.



Ar : *p*-FC_6H_4, **8b**

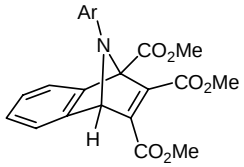
Diels-Alder adduct 8b. ^1H NMR ($CDCl_3$, 400 MHz) δ 3.73 (s, 3H), 3.84 (s, 3H), 3.88 (s, 3H), 5.54 (s, 1H), 6.84-6.94 (m, 4H), 7.11 (m, 2H), 7.40 (m, 1H), 7.57 (m, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 52.5 (CH_3), 52.6 (CH_3), 53.1 (CH_3), 74.5 (CH), 82.9 (C), 115.8 (d, *J* = 22.5 Hz, CH), 122.2 (CH), 122.5 (CH), 122.6 (d, *J* = 7.7 Hz, CH), 126.2 (CH), 126.7 (CH), 140.5 (d, *J* = 2.3 Hz, C), 145.0 (C), 145.5 (C), 146.2 (C), 151.5 (C), 159.4 (d, *J* = 242.7 Hz, C), 162.4 (C),

164.2 (C), 167.0 (C). IR (CH_2Cl_2) ν 1743, 1720, 1639, 1596, 1493, 1435, 1252, 1200, 1146, 1113, 757 cm^{-1}. HRMS (ESI-TOF) cald for $C_{22}H_{19}FNO_6$: 412.1191 $[M+H]^+$; found: 412.1186.



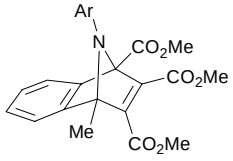
Ar : *p*-$MeO_2CC_6H_4$, **8c**

Diels-Alder adduct 8c. ^1H NMR ($CDCl_3$, 300 MHz) δ 3.74 (s, 3H), 3.84 (s, 3H), 3.85 (s, 3H), 3.90 (s, 3H), 5.70 (s, 1H), 6.90 (d, *J* = 8.7 Hz, 2H), 7.11 (m, 2H), 7.40 (m, 1H), 7.62 (m, 1H), 7.89 (d, *J* = 8.7 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 51.9 (CH_3), 52.5 (CH_3), 52.7 (CH_3), 53.2 (CH_3), 73.8 (CH), 82.1 (C), 120.2 (CH), 122.2 (CH), 122.7 (CH), 125.1 (C), 126.3 (CH), 126.7 (CH), 130.7 (CH), 144.6 (C), 145.1 (C), 146.4 (C), 148.5 (C), 151.8 (C), 162.2 (C), 163.9 (C), 166.5 (C), 166.9 (C). IR (neat) ν 1719, 1606, 1436, 1281, 1191, 1145, 1112, 1014, 771 cm^{-1}. HRMS (ESI-TOF) cald for $C_{24}H_{22}NO_8$: 452.1340 $[M+H]^+$; found: 452.1335.



Ar : *p*-$MeOC_6H_4$, **8d**

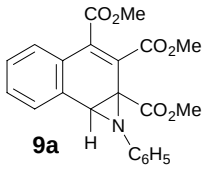
Diels-Alder adduct 8d. ^1H NMR ($CDCl_3$, 300 MHz) δ 3.72 (s, 3H), 3.73 (s, 3H), 3.85 (s, 3H), 3.87 (s, 3H), 5.53 (s, 1H), 6.76 (m, 2H), 6.85 (m, 2H), 7.10 (m, 2H), 7.40 (m, 1H), 7.54 (m, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz, from a mixture with small amounts of aziridine **9d**) δ 52.4 (CH_3), 52.5 (CH_3), 53.1 (CH_3), 55.3 (CH_3), 74.5 (CH), 83.2 (C), 114.3 (CH), 122.1 (CH), 122.3 (CH), 122.4 (CH), 126.1 (CH), 126.5 (CH), 137.6 (C), 145.4 (C), 145.8 (C), 146.2 (C), 151.5 (C), 156.1 (C), 162.6 (C), 164.5 (C), 167.2 (C). IR (neat) ν 1719, 1606, 1436, 1281, 1191, 1145, 1112, 1014, 771 cm^{-1}. HRMS (ESI-TOF) cald for $C_{23}H_{21}NO_7$: 424.1391 $[M+H]^+$; found: 424.1389.



Ar : *p*-$MeOC_6H_4$, **8e**

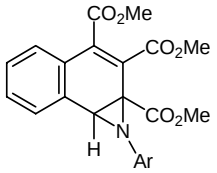
Diels-Alder adduct 8e. ^1H NMR ($CDCl_3$, 300 MHz) δ 1.84 (s, 3H), 3.72 (s, 3H), 3.74 (s, 3H), 3.77 (s, 3H), 3.80 (s, 3H), 6.73 (m, 2H), 6.90 (m, 2H), 7.11 (m, 2H), 7.23 (m, 1H), 7.68 (m, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 12.5 (CH_3), 52.2 (CH_3), 52.4 (CH_3), 52.8 (CH_3), 55.2 (CH_3), 79.3 (C), 80.9 (C), 113.7 (CH), 120.8 (CH), 122.3 (CH), 126.0 (2 CH), 126.3 (CH), 134.9 (C),

146.3 (C), 148.8 (C), 150.5 (C), 151.4 (broad C), 157.3 (C), 163.6 (C), 163.8 (C), 167.2 (C). IR (neat) ν 1736, 1717, 1508, 1435, 1303, 1228, 1197, 1154, 1032, 729 cm^{-1}. HRMS (ESI-TOF) cald for $C_{24}H_{24}NO_7$: 438.1547 $[M+H]^+$; found: 438.1542.



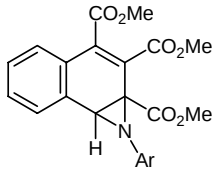
9a

Aziridine 9a. ^1H NMR ($CDCl_3$, 400 MHz) δ 3.81 (s, 3H), 3.84 (s, 3H), 3.93 (s, 3H), 4.42 (s, 1H), 6.61 (d, *J* = 7.2 Hz, 2H), 6.78 (t, *J* = 7.2 Hz, 1H), 6.94 (t, *J* = 7.2 Hz, 2H), 7.17 (d, *J* = 8 Hz, 1H), 7.34 (td, *J* = 8 and 1.2 Hz, 1H), 7.54 (td, *J* = 8 and 1.2 Hz, 1H), 7.78 (dd, *J* = 8 and 1.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 46.6 (C), 50.3 (CH), 52.7 (CH_3), 52.9 (CH_3), 53.1 (CH_3), 121.7 (CH), 122.5 (CH), 127.7 (CH), 127.8 (C), 127.9 (CH), 129.0 (CH), 129.3 (C), 130.6 (CH), 130.9 (CH), 139.5 (C), 142.3 (C), 165.7 (C), 167.0 (C), 168.8 (C). One C was not observed. IR (CH_2Cl_2) ν 1728, 1591, 1487, 1437, 1229, 1206 cm^{-1}. HRMS (ESI-TOF) cald for $C_{22}H_{20}NO_6$: 394.1285 $[M+H]^+$; found: 394.1282.



Ar : *p*-FC_6H_4, **9b**

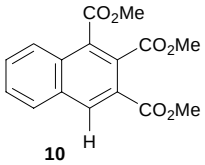
Aziridine 9b. ^1H NMR ($CDCl_3$, 400 MHz) δ 3.82 (s, 3H), 3.84 (s, 3H), 3.93 (s, 3H), 4.42 (s, 1H), 6.56 (m, 2H), 6.63 (m, 2H), 7.19 (d, *J* = 8 Hz, 1H), 7.36 (td, *J* = 8 and 1.2 Hz, 1H), 7.54 (td, *J* = 8 and 0.8 Hz, 1H), 7.77 (dd, *J* = 8 and 1.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 46.9 (C), 50.5 (CH), 52.8 (CH_3), 52.9 (CH_3), 53.1 (CH_3), 114.8 (d, *J* = 22.5 Hz, CH), 122.4 (C), 122.9 (d, *J* = 7.8 Hz, CH), 126.4 (C), 127.8 (CH), 128.9 (C), 129.1 (CH), 130.7 (CH), 131.1 (CH), 138.2 (d, *J* = 3.1Hz, C), 139.4 (C), 158.3 (d, *J* = 241.2 Hz, C), 165.8 (C), 166.8 (C), 168.6 (C). IR (CH_2Cl_2) ν 1732, 1615, 1505, 1436, 1212, 770 cm^{-1}. HRMS (ESI-TOF) cald for $C_{22}H_{19}FNO_6$: 412.1191 $[M+H]^+$; found: 412.1182.



Ar : *p*-$MeOC_6H_4$, **9d**

Aziridine 9d. ^1H NMR ($CDCl_3$, 400 MHz) δ 3.62 (s, 3H), 3.82 (s, 3H), 3.84 (s, 3H), 3.92 (s, 3H), 4.39 (s, 1H), 6.47 (d, *J* = 9 Hz, 2H), 6.53 (d, *J* = 9 Hz, 2H), 7.19 (d, *J* = 8 Hz, 1H), 7.35 (td,

J = 8 and 1.2 Hz, 1H), 7.52 (td, J = 8 and 0.8 Hz, 1H), 7.77 (dd, J = 8 and 1.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 46.9 (C), 50.6 (CH), 52.7 (CH_3), 52.9 (CH_3), 53.1 (CH_3), 55.2 (CH_3), 113.3 (CH), 122.6 (C), 122.7 (CH), 127.7 (CH), 127.9 (C), 128.9 (CH), 129.4 (C), 130.6 (CH), 130.8 (CH), 135.4 (C), 139.4 (C), 154.9 (C), 165.9 (C), 167.0 (C), 168.9 (C). IR (CH_2Cl_2) ν 1734, 1615, 1564, 1508, 1437, 1289, 1237, 753 cm^{-1}. HRMS (ESI-TOF) cald for $C_{23}H_{21}NO_7$: 424.1391 $[M+H]^+$; found: 424.1391.

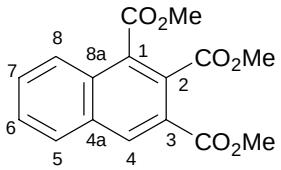


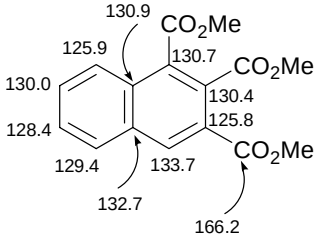
10

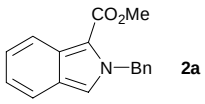
Trimethyl 1,2,3-naphthalenetricarboxylate (10). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.95 (s, 3H), 3.96 (s, 3H), 4.01 (s, 3H), 7.65 (ddd, J = 8, 7.2, and 0.9 Hz, 1H), 7.71 (ddd, J = 8, 7.2, and 0.9 Hz, 1H), 7.97 (dd, J = 8 and 0.9 Hz, 1H), 8.16 (dd, J = 8 and 0.9 Hz, 1H), 8.56 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 52.8 (CH_3), 52.9 (CH_3), 52.9 (CH_3), 125.8 (C), 125.9 (CH), 128.4 (CH), 129.4 (CH), 130.0 (CH), 130.4 (C), 130.7 (C), 130.9 (C), 132.7 (C), 133.7 (CH), 166.2 (C), 167.3 (C), 168.1 (C). IR (neat) ν 2946, 1722, 1608, 1521, 1444, 1306, 1276, 1210, 1135, 1013, 971 cm^{-1}. HRMS (ESI-TOF) cald for $C_{16}H_{15}O_6$: 303.0863 $[M+H]^+$; found: 303.0864.

Structure determination on compound 10

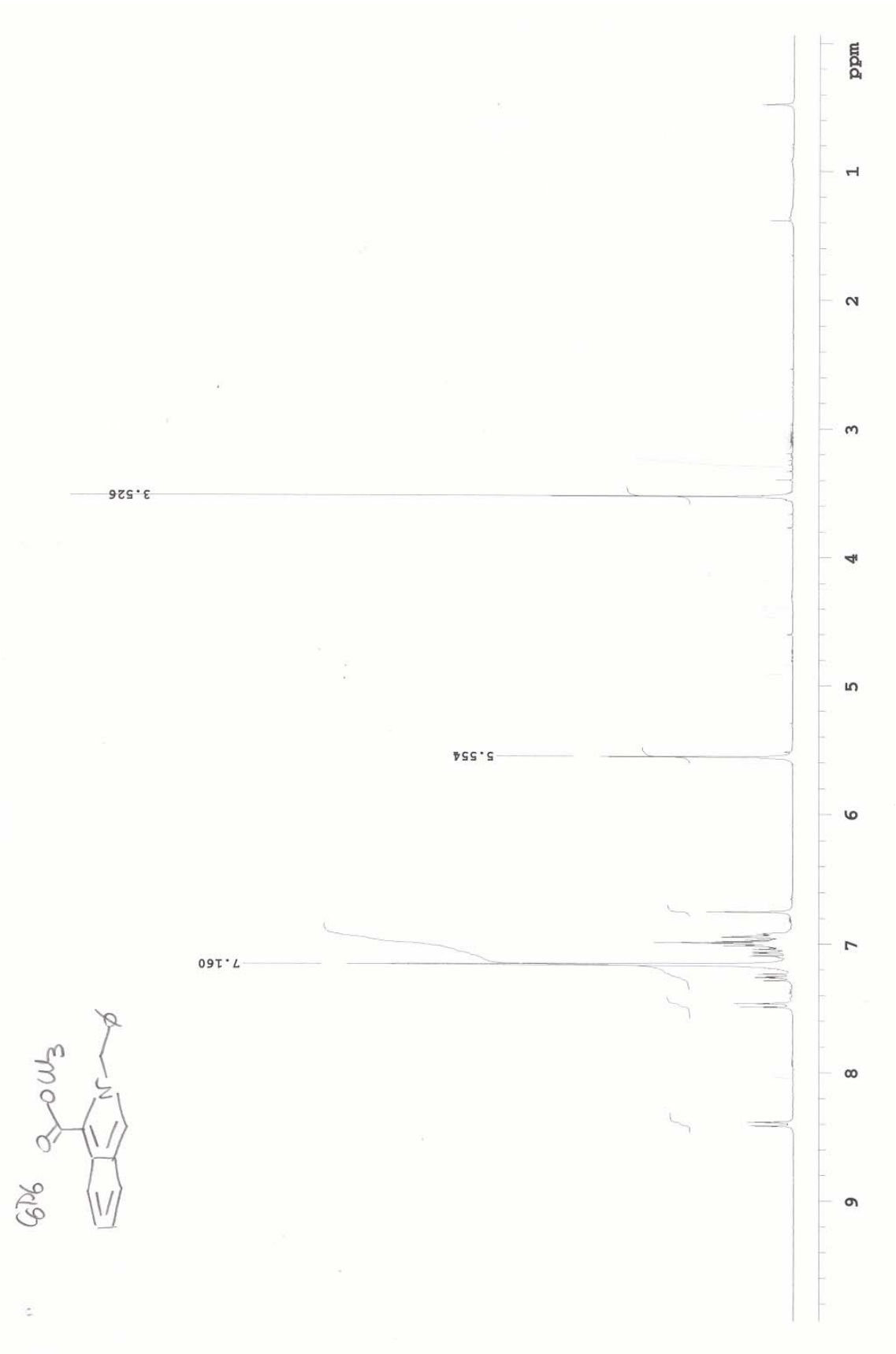
proton	δ	HMBC correlations
H-4	8.56	166.2 (COO) 130.9 (C-8a) 130.4 (C-2) 129.4 (C-5)
H-5	7.98	133.7 (C-4) 130.9 (C-8a) 130.0 (C-7)
H-6	7.65	132.7 (C-4a) 125.9 (C-8)
H-7	7.71	130.9 (C-8a) 129.4 (C-5)
H-8	8.16	132.7 (C-4a) 130.7 (C-1) 128.4 (C-6)
OCH_3	3.96	166.2 (COO)

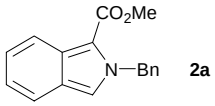




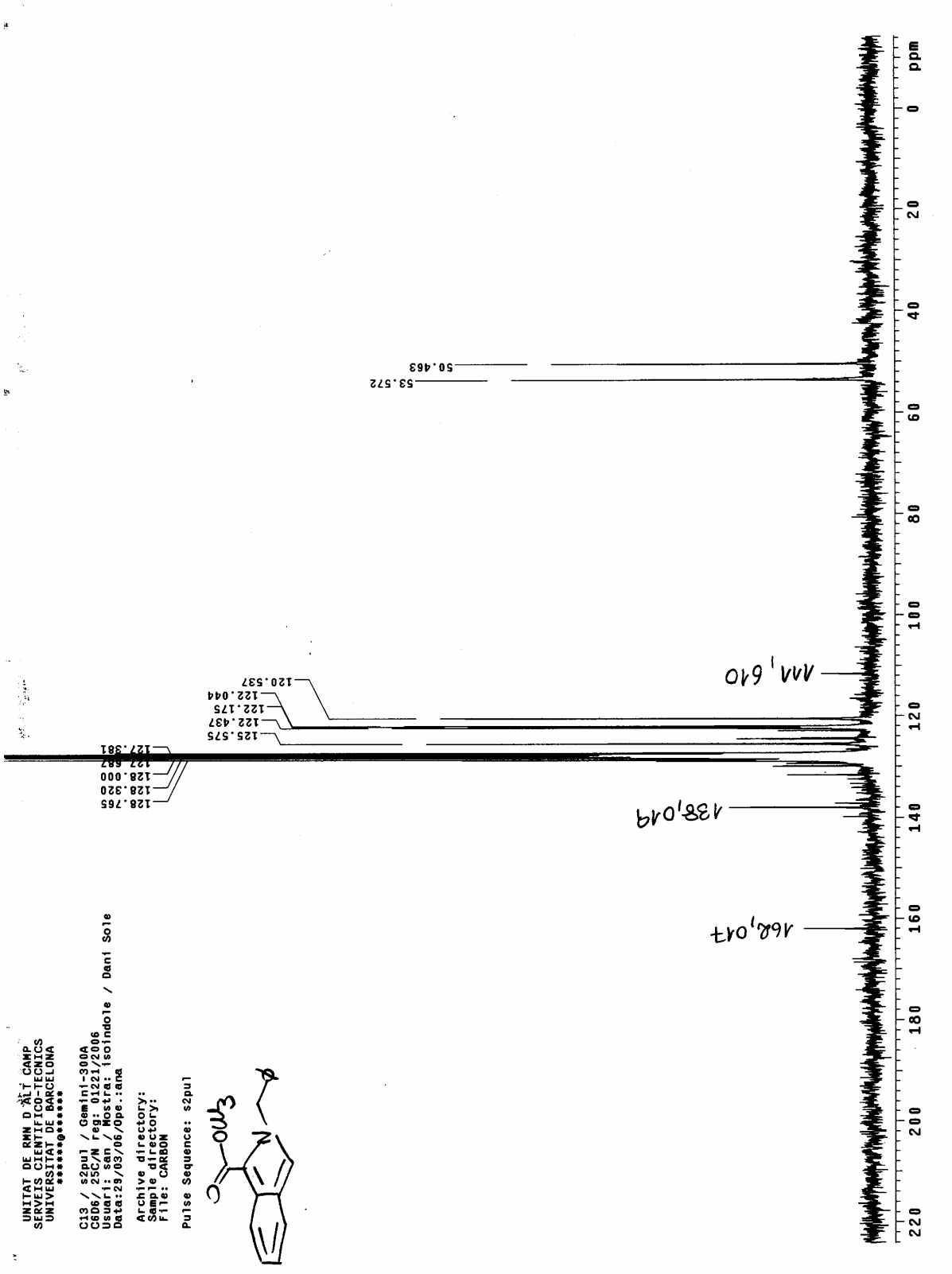


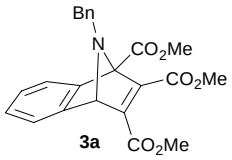
2a



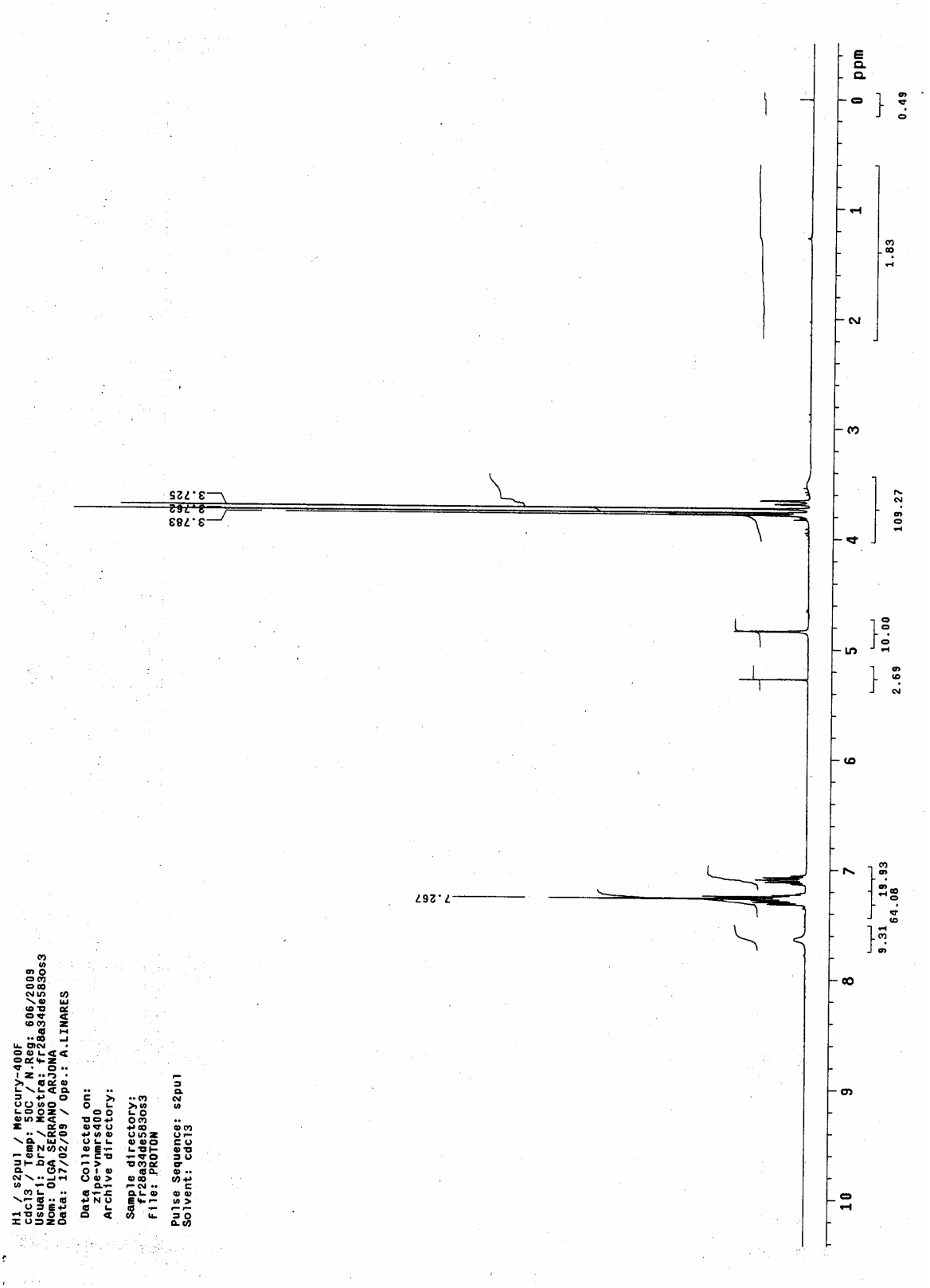

CO2Me
N—Bn

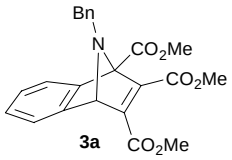
2a


UNITAT DE RMN D'ALT CAMP
SERVEIS CIENTIFICO-TECNICS
UNIVERSITAT DE BARCELONA
C13 / s2pul / Gemini-300A
C6D6/ 25C/N reg: 01221/2006
Usuari: san / Mostra: isoindole / Dani Sole
Data:29/03/06/Ope.:ana
Archive directory:
Sample directory:
File: CARBON
Pulse Sequence: s2pul
50.463
53.572
120.537
122.044
122.175
122.437
125.575
127.381
128.765
128.320
128.000
138.049
168.021
ppm

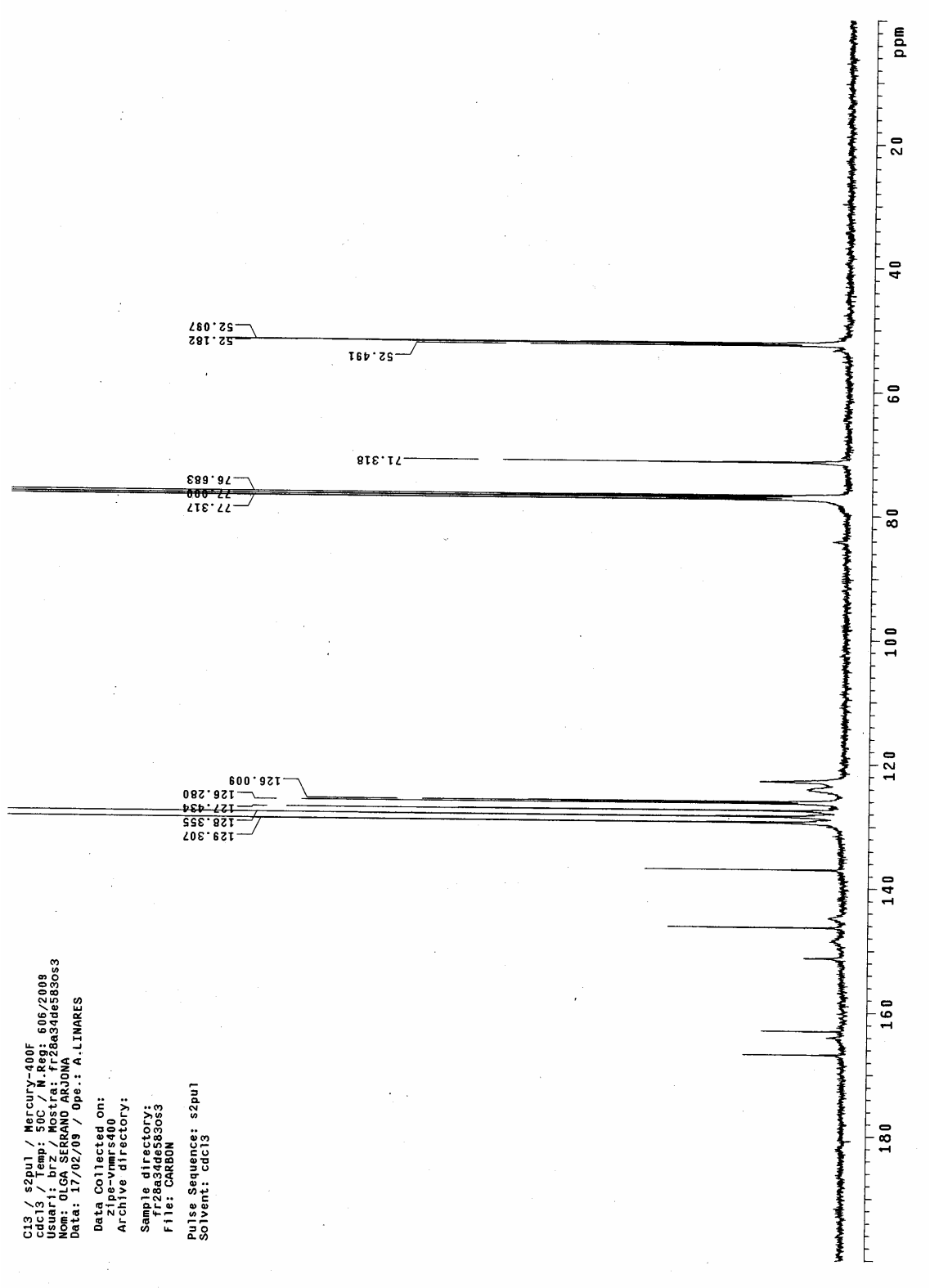

Bn
N
CO2Me
CO2Me
CO2Me

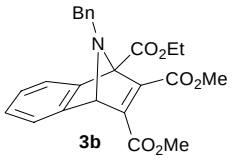
3a


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 50C / N.Reg: 606/2009
Usuari: brz / Mostra: fr28a34de583os3
Nom: OLGA SERRANO ARJONA
Data: 17/02/09 / Ope.: A.LINARES
Data Collected on:
zipe-vnmrs400
Archive directory:
Sample directory:
fr28a34de583os3
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
3.783
3.762
3.725
7.267
9.31
19.93
64.08
2.69
10.00
109.27
1.83
0.49
ppm

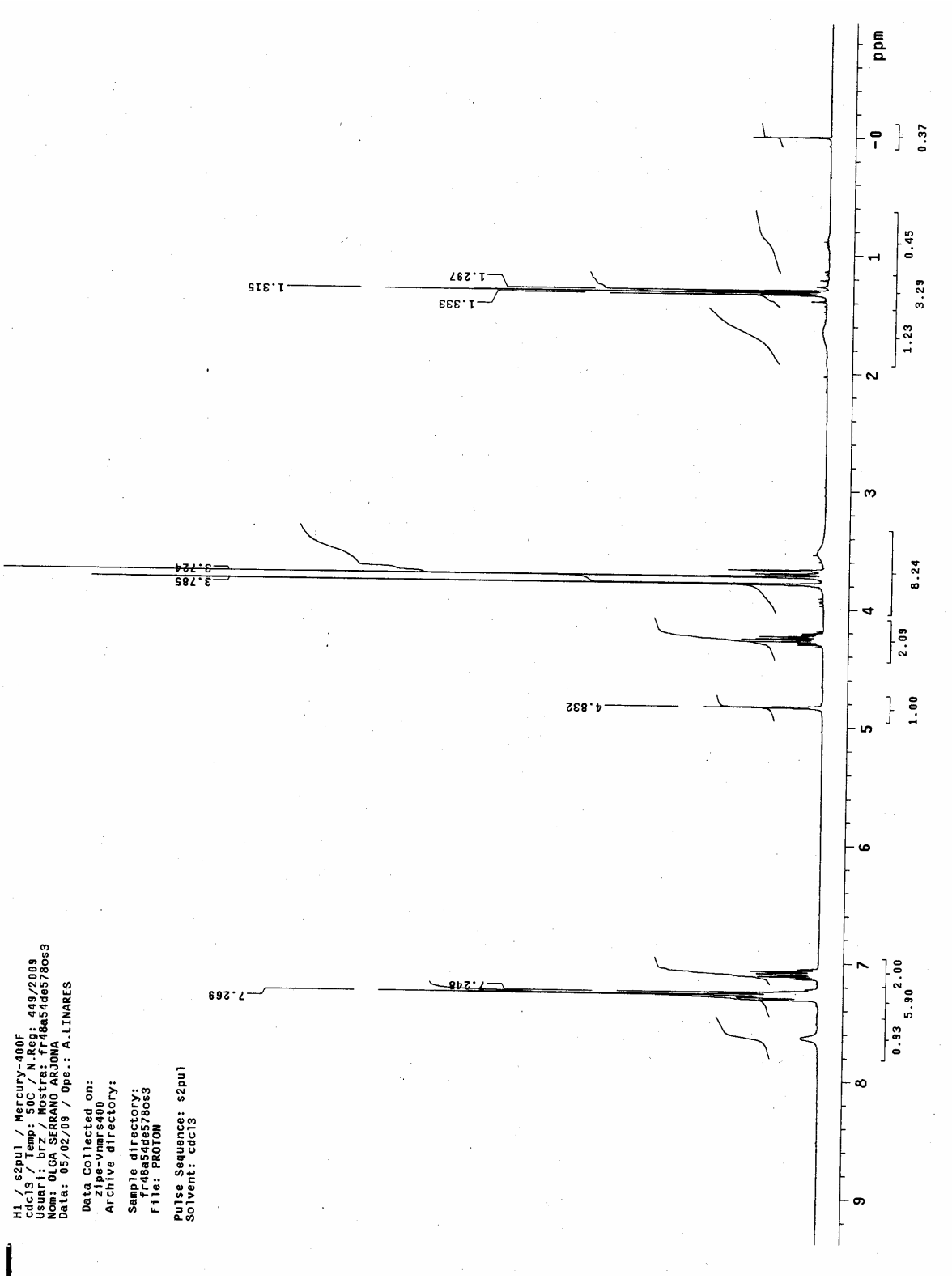

Bn
N
CO2Me
CO2Me
CO2Me

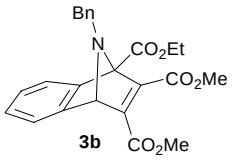
3a


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 50C / N.Reg: 606/2009
Usuari: brz / Mostra: fr28a34de583os3
Nom: OLGA SERRANO ARJONA
Data: 17/02/09 / Ope.: A.LINARES
Data Collected on:
zipe-vnmrs400
Archive directory:
Sample directory:
fr28a34de583os3
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
52.087
52.182
52.491
71.318
76.683
77.089
77.317
126.009
126.280
127.434
128.355
129.307
ppm
20
40
60
80
100
120
140
160
180

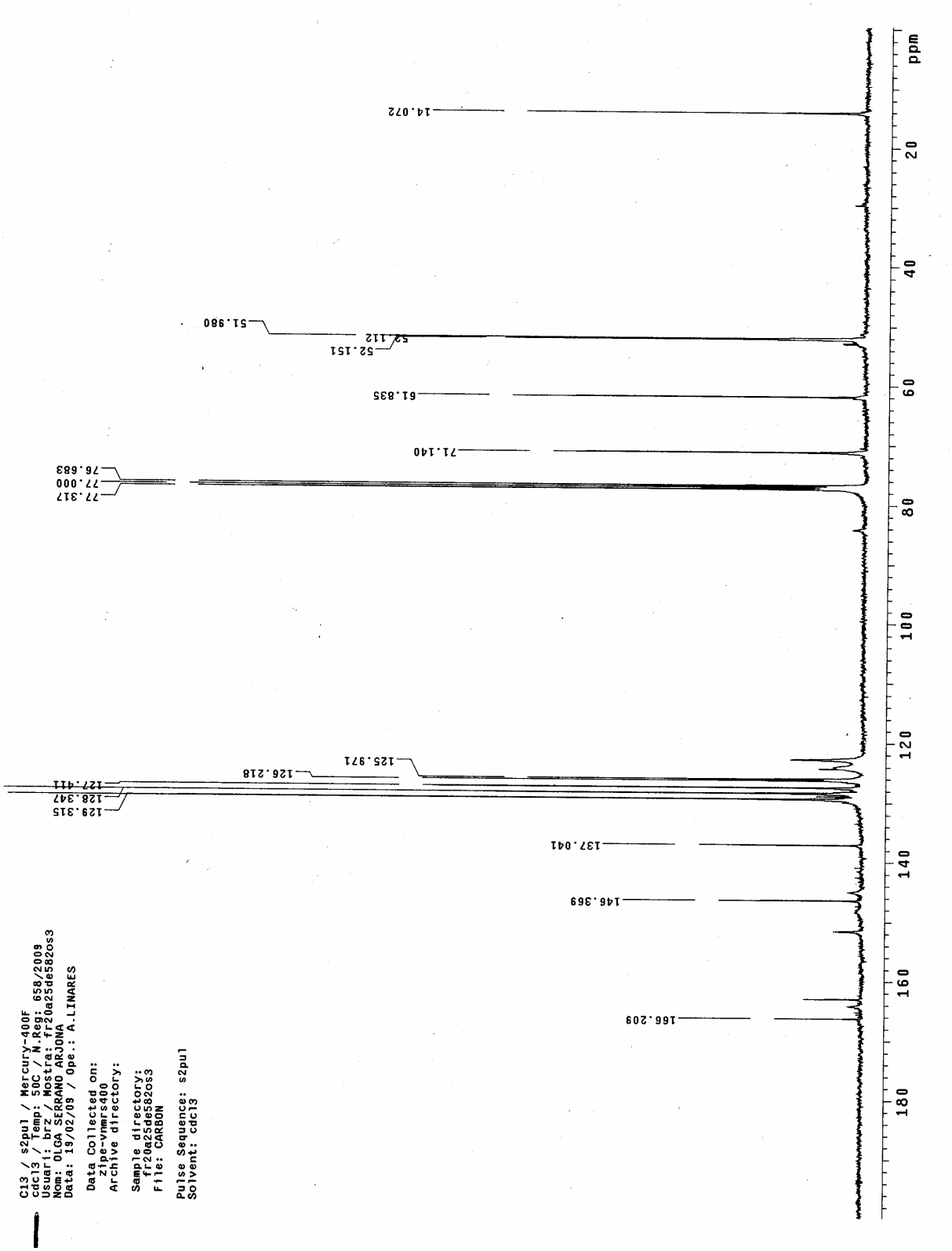

Bn
N
CO2Et
CO2Me
CO2Me

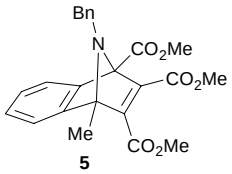
3b


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 50C / N.Reg: 449/2009
Usuari: brz / Mostra: fr48a54de578os3
Nom: OLGA SERRANO ARJONA
Data: 05/02/09 / Ope.: A.LINARES
Data Collected on:
z1pe-vmars400
Archive directory:
Sample directory:
fr48a54de578os3
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
7.269
7.248
4.832
3.785
3.764
1.315
1.297
1.333
ppm
0.37
0.45
3.29
1.23
8.24
2.09
1.00
2.00
5.90
0.93

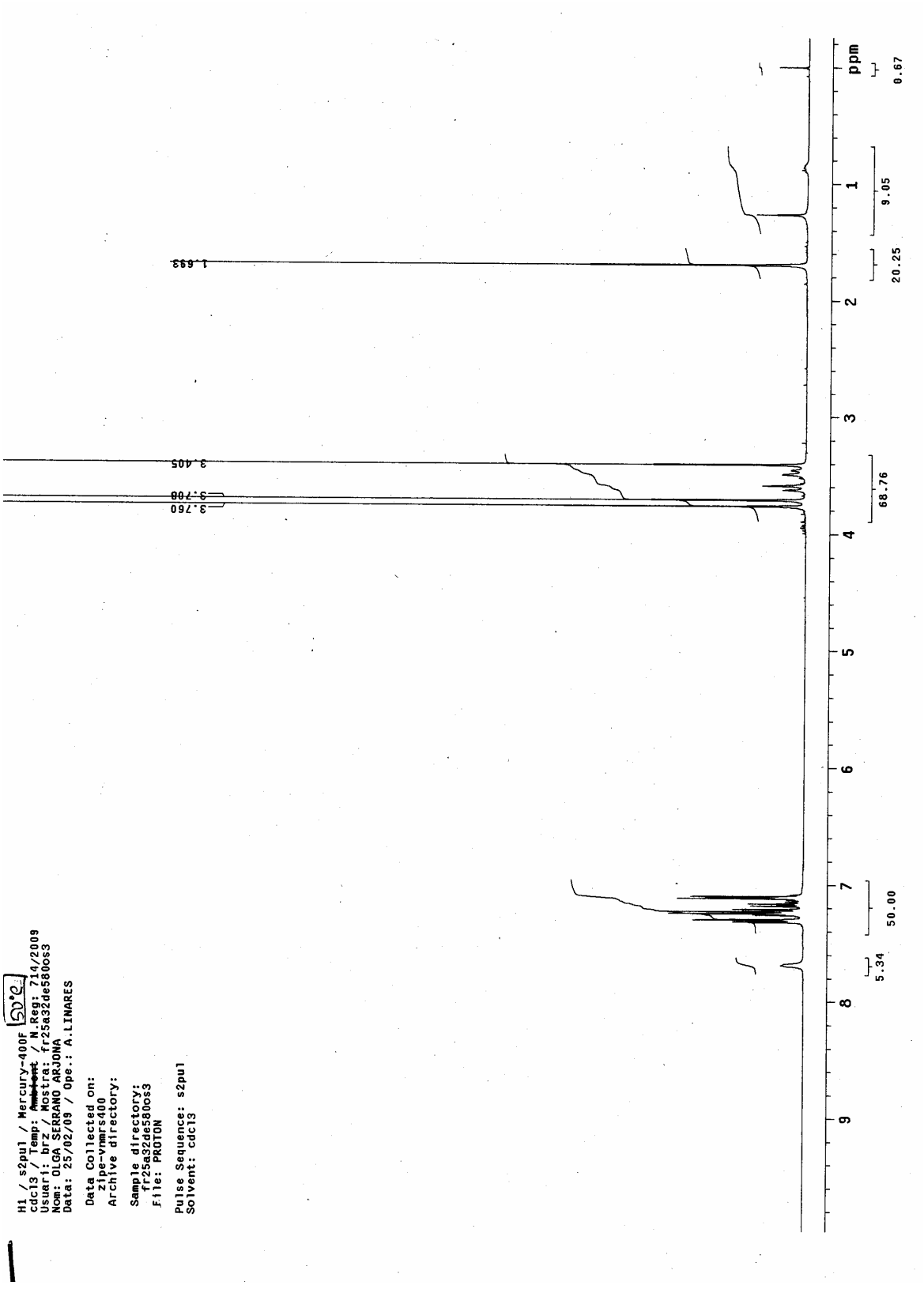

Bn
N
CO2Et
CO2Me
CO2Me

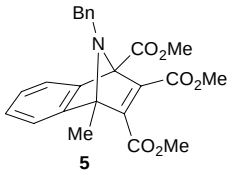
3b


14.072
51.980
52.112
52.151
61.835
71.140
76.683
77.000
77.317
125.971
126.218
127.411
128.347
129.315
137.041
146.369
166.209
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 50C / N.Reg: 658/2009
Usuari: brz / Mostra: fr20a25de582os3
Nom: OLGA SERRANO ARJONA
Data: 19/02/09 / Ope.: A.LINARES
Data Collected on:
zipe-vnmrs400
Archive directory:
Sample directory:
fr20a25de582os3
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3

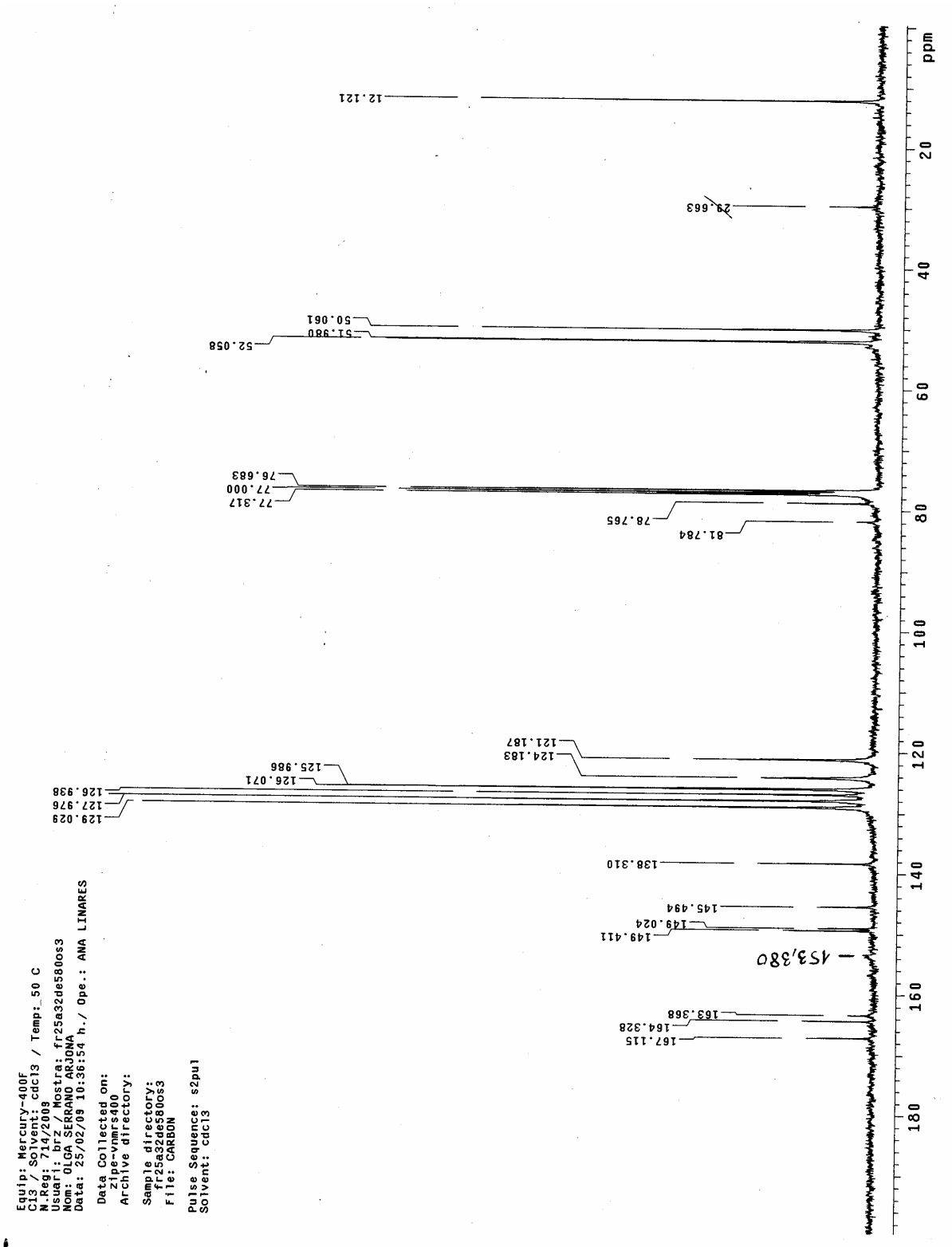

Bn
N
CO2Me
CO2Me
Me
CO2Me

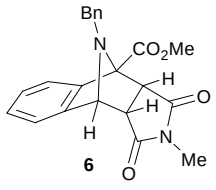
5


H1 / s2pul / Mercury-400f
cdcl3 / Temp: Ambient / N.Reg: 714/2009
Usuari: brz / Mostra: fr25a32de580os3
Nom: OLGA SERRANO ARJONA
Data: 25/02/09 / Ope.: A.LINARES
Data Collected on:
z1pe-vmrs400
Archive directory:
Sample directory:
fr25a32de580os3
File: PROTON
Pulse Sequence: s2pul
Solvent: cdcl3
1.693
3.405
3.708
3.760
ppm
0.67
9.05
20.25
68.76
50.00
5.34

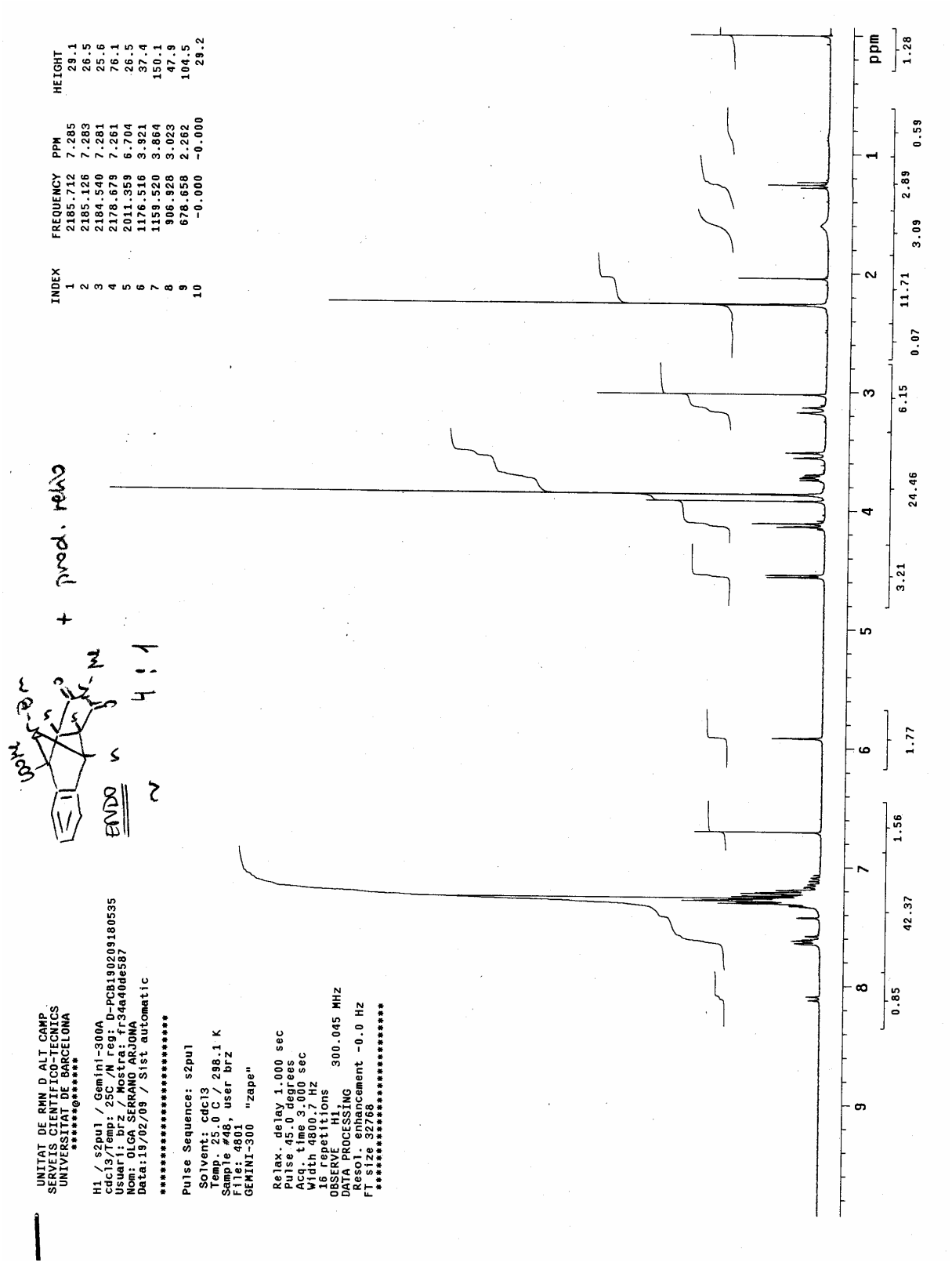

Bn
N
CO2Me
CO2Me
Me
CO2Me

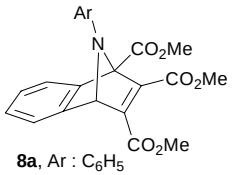
5


Equip: Mercury-400F
C13 / Solvent: cdcl3 / Temp: 50 C
N.Reg: 714/2009
Usuari: brz / Mostra: fr25a32de580os3
Nom: OLGA SERRANO ARJONA
Data: 25/02/09 10:36:54 h./ Ope.: ANA LINARES
Data Collected on:
zipe-vmmrs400
Archive directory:
Sample directory:
fr25a32de580os3
File: CARBON
Pulse Sequence: s2pul
Solvent: cdcl3
12.121
29.663
50.061
51.980
52.058
76.683
77.000
77.317
78.765
81.784
121.187
124.183
125.986
126.071
126.938
127.976
129.029
138.310
145.494
149.024
149.411
153.380
163.368
164.328
167.115
ppm

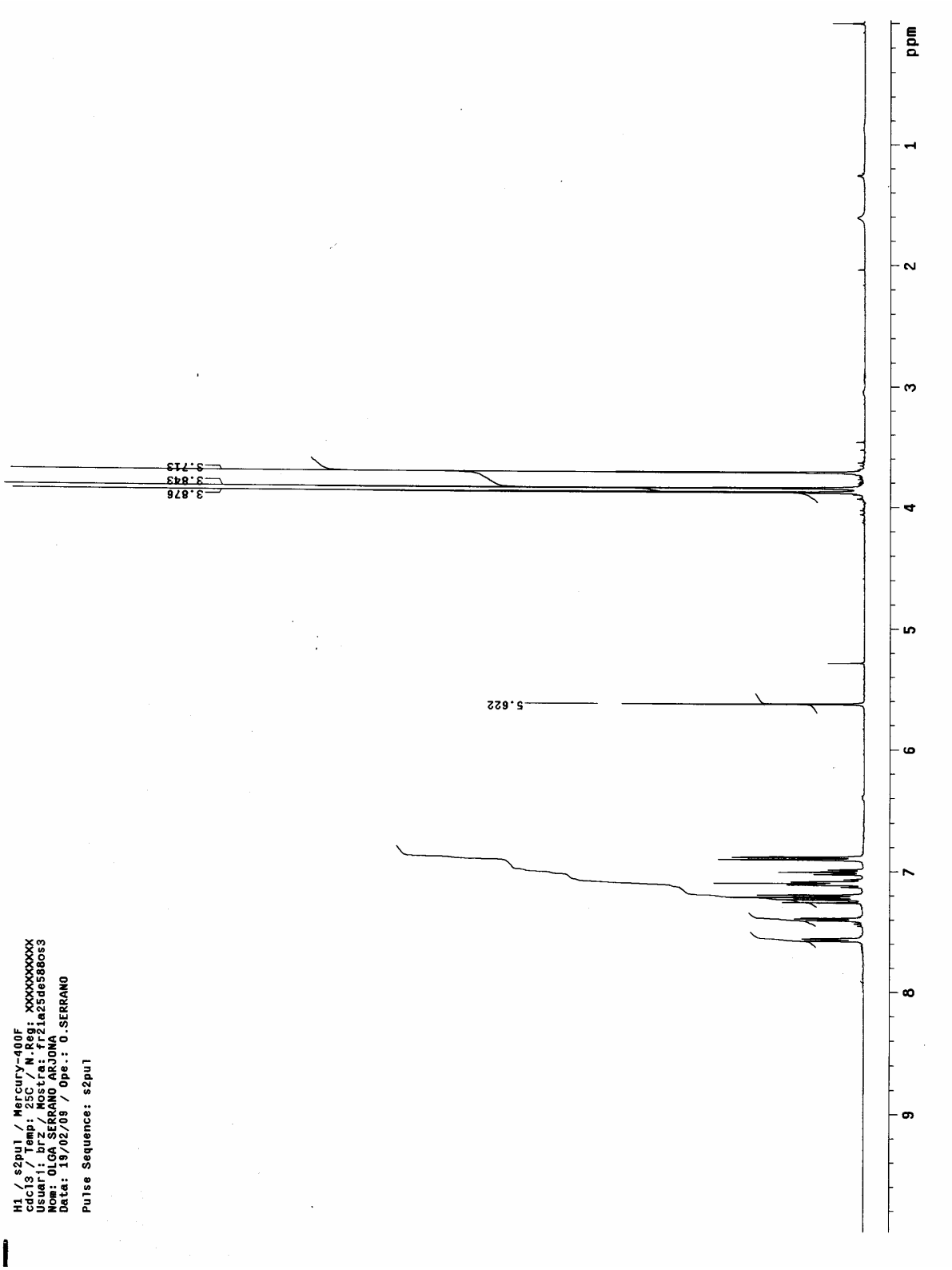

Bn
N
CO2Me
H
H
O
H
N
Me
O

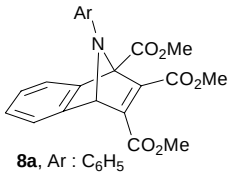
6


UNITAT DE RMN D ALT CAMP
SERVEIS CIENTIFICO-TECNICS
UNIVERSITAT DE BARCELONA
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N regr D-PCB19020918053S
Usuari: bz / Mostra: fr34a40de587
Nom: OLGA SERRANO ARJONA
Data:19/02/09 / Sist automatic
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #48, user brz
File: 4801
GEMINI-300 "zape"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 4800.7 Hz
16 repetitions
OBSERVE H1, 300.045 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 32768
INDEX FREQUENCY PPM HEIGHT
1 2185.712 7.285 29.1
2 2185.126 7.283 26.5
3 2184.540 7.281 25.6
4 2178.679 7.261 76.1
5 2011.359 6.704 26.5
6 1176.516 3.921 37.4
7 1159.520 3.864 150.1
8 906.928 3.023 47.9
9 678.658 2.262 104.5
10 -0.000 -0.000 29.2
ppm
3.21
24.46
6.15
0.07
11.71
3.09
2.89
0.59
1.28
0.85
42.37
1.56
1.77

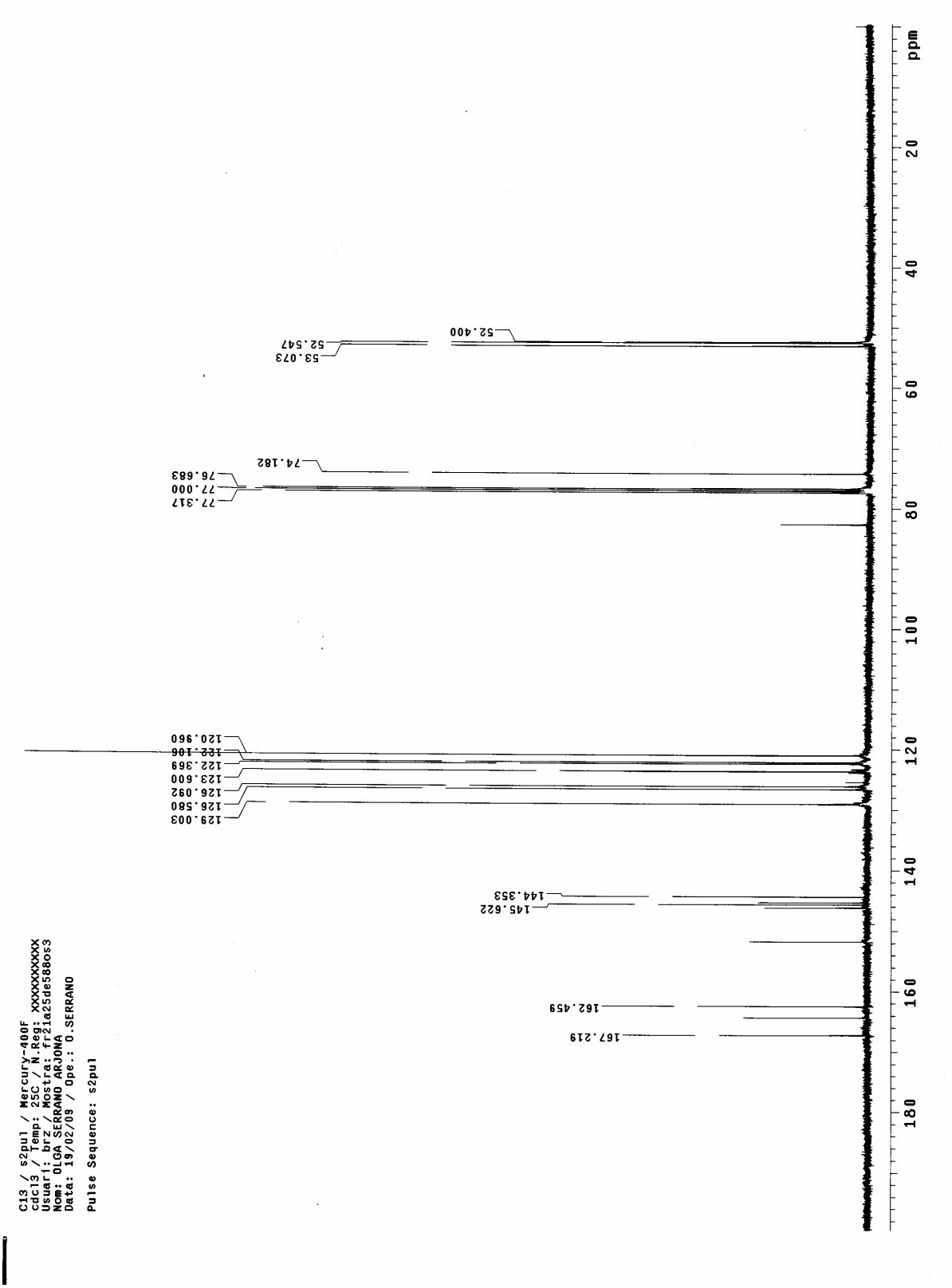

Ar
N
CO2Me
CO2Me
CO2Me

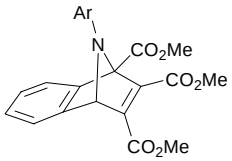
8a, Ar : C_6H_5


3.876
3.843
3.713
5.622
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr21a25de588os3
Nom: OLGA SERRANO ARJONA
Data: 19/02/08 / Ope.: O.SERRANO
Pulse Sequence: s2pul

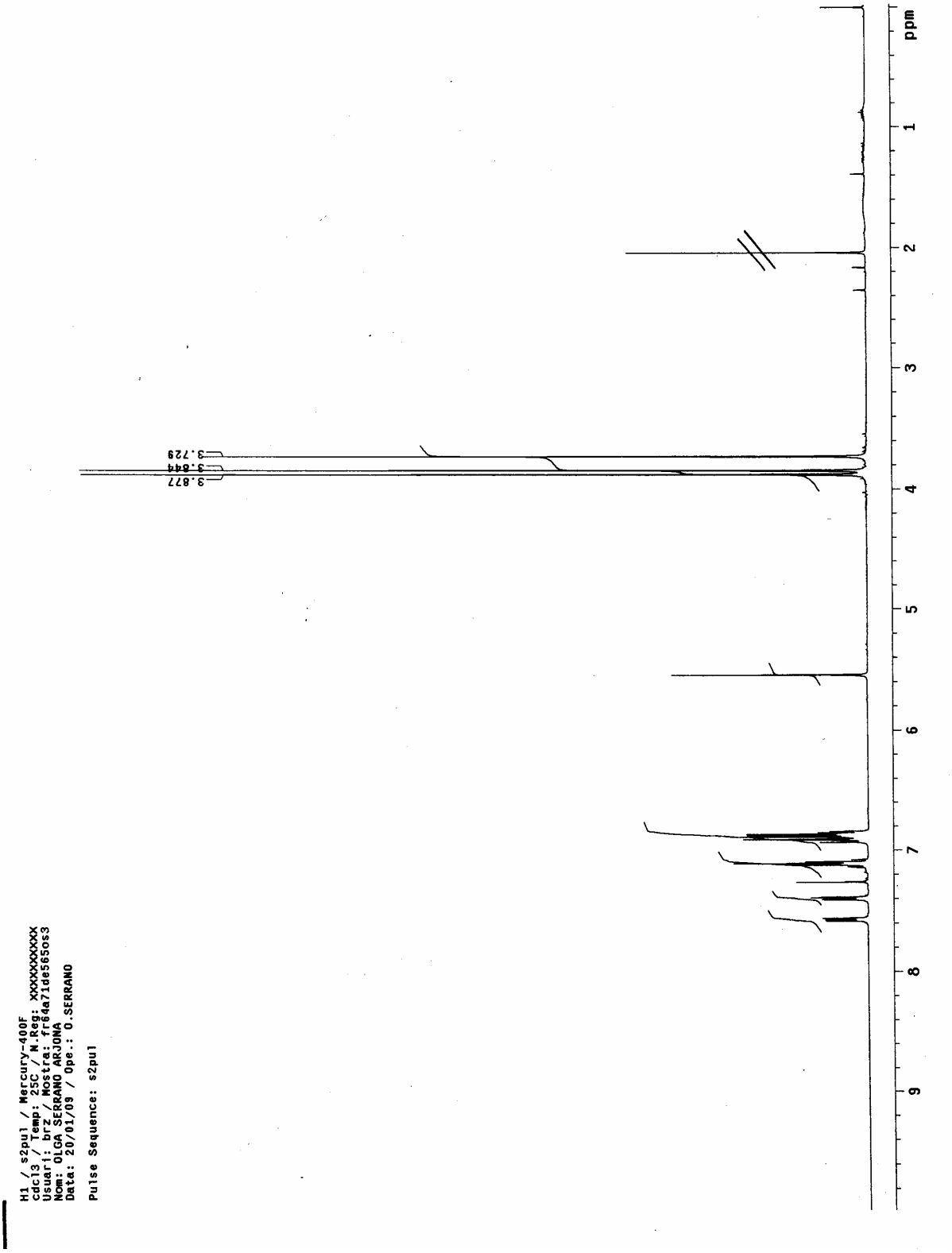

Ar
N
CO2Me
CO2Me
CO2Me

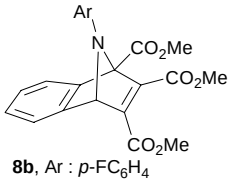
8a, Ar : C_6H_5


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr21a25de588os3
Nom: OLGA SERRANO ARJONA
Data: 19/02/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
167.219
162.459
145.622
144.353
129.003
126.580
126.092
123.600
122.369
122.106
120.960
77.317
77.000
76.683
74.182
53.073
52.547
52.400
ppm

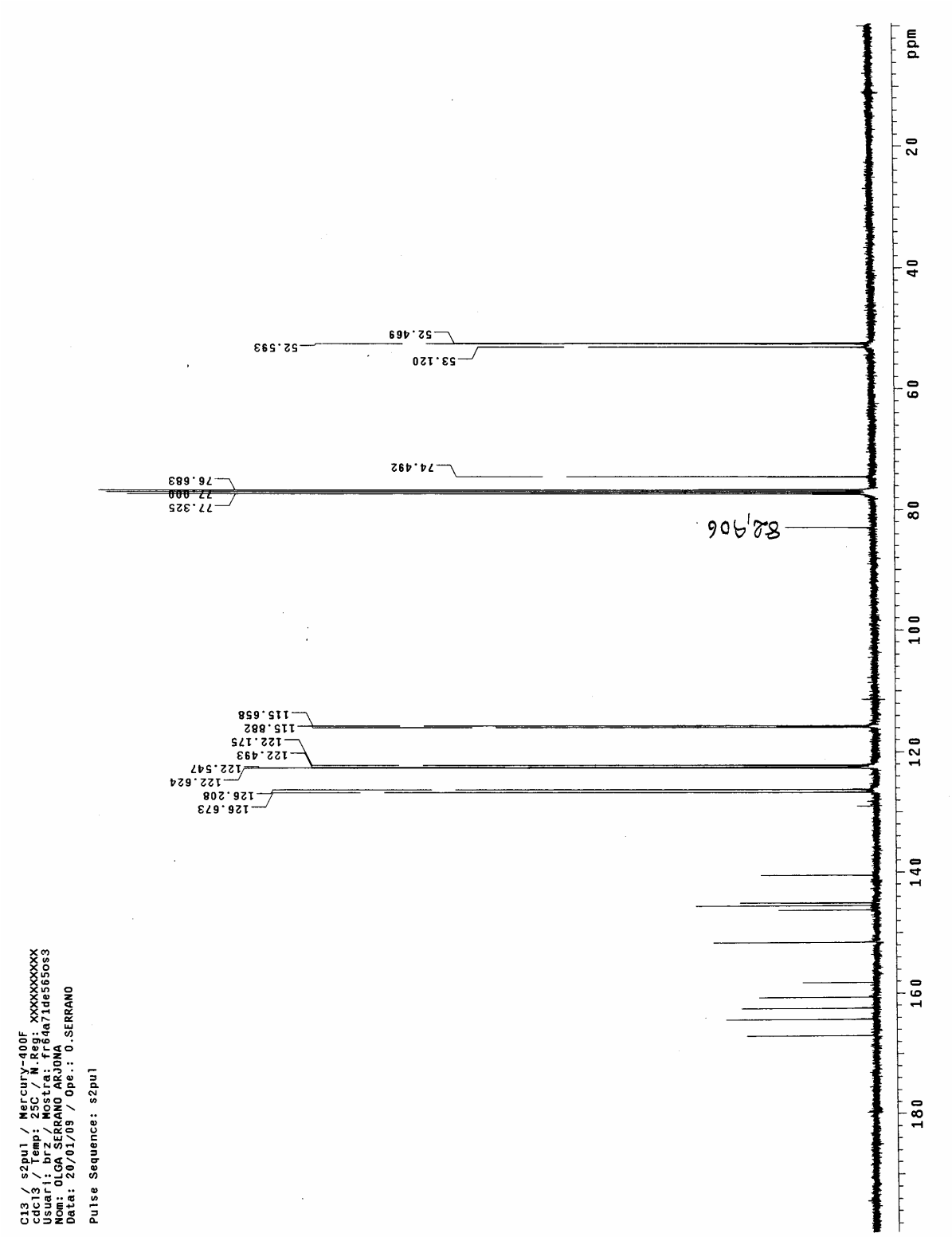

Ar
N
CO2Me
CO2Me
CO2Me

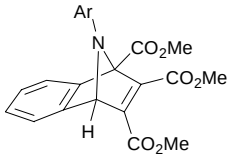
8b, Ar : *p*-FC_6H_4


3.877
3.844
3.729
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
Cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brZ / Mostra: fr64a71de565os3
Nom: OLGA SERRANO ARJONA
Data: 20/01/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul

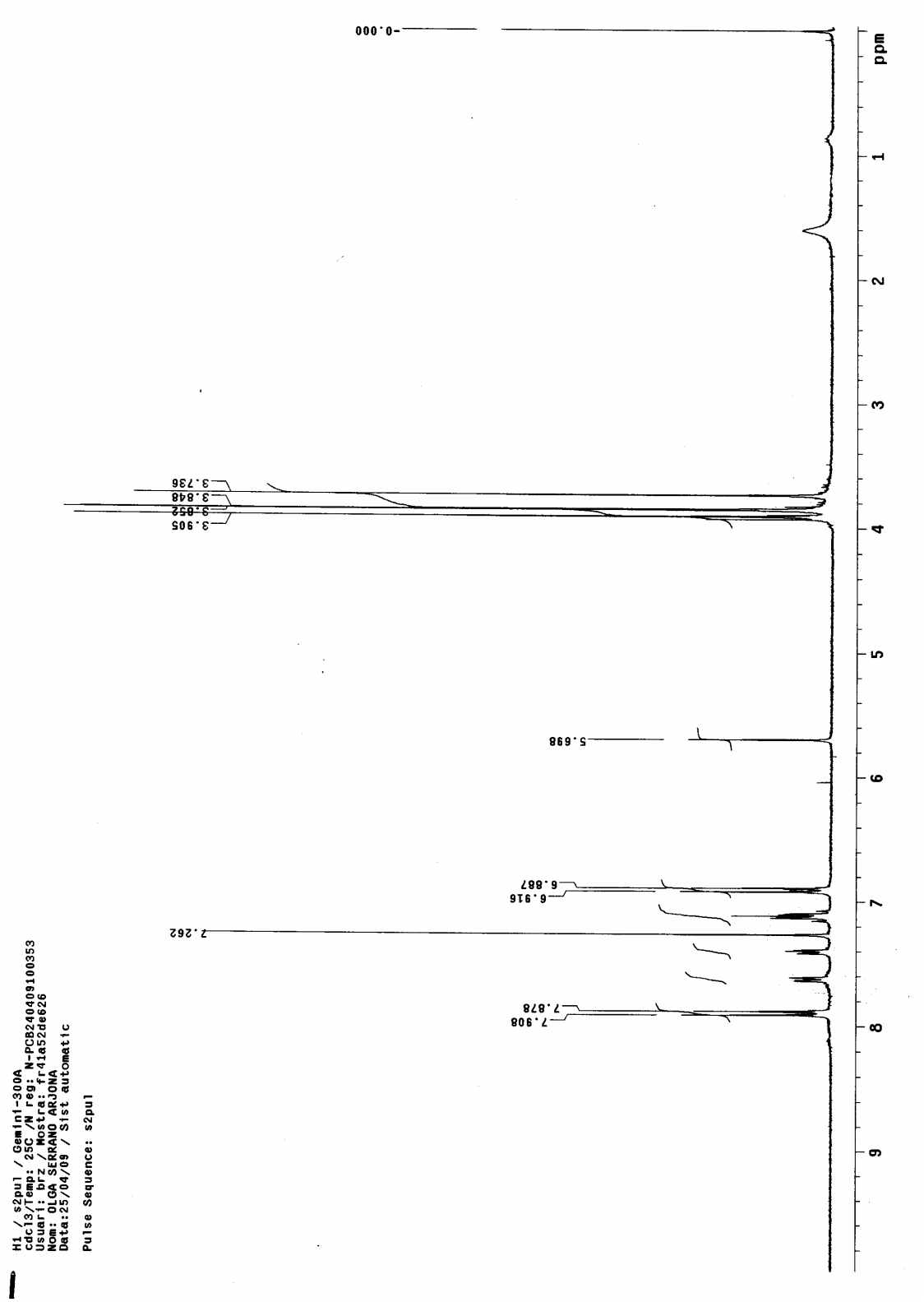

Ar
N
CO2Me
CO2Me
CO2Me

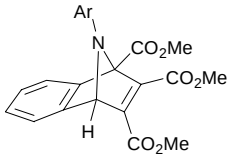
8b, Ar : *p*-FC_6H_4


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr64a/71de565os3
Nom: OLGA SERRANO ARJONA
Data: 20/01/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
126.673
126.208
122.624
122.547
122.493
122.175
115.882
115.658
77.325
76.683
74.492
53.120
52.469
52.593
ppm

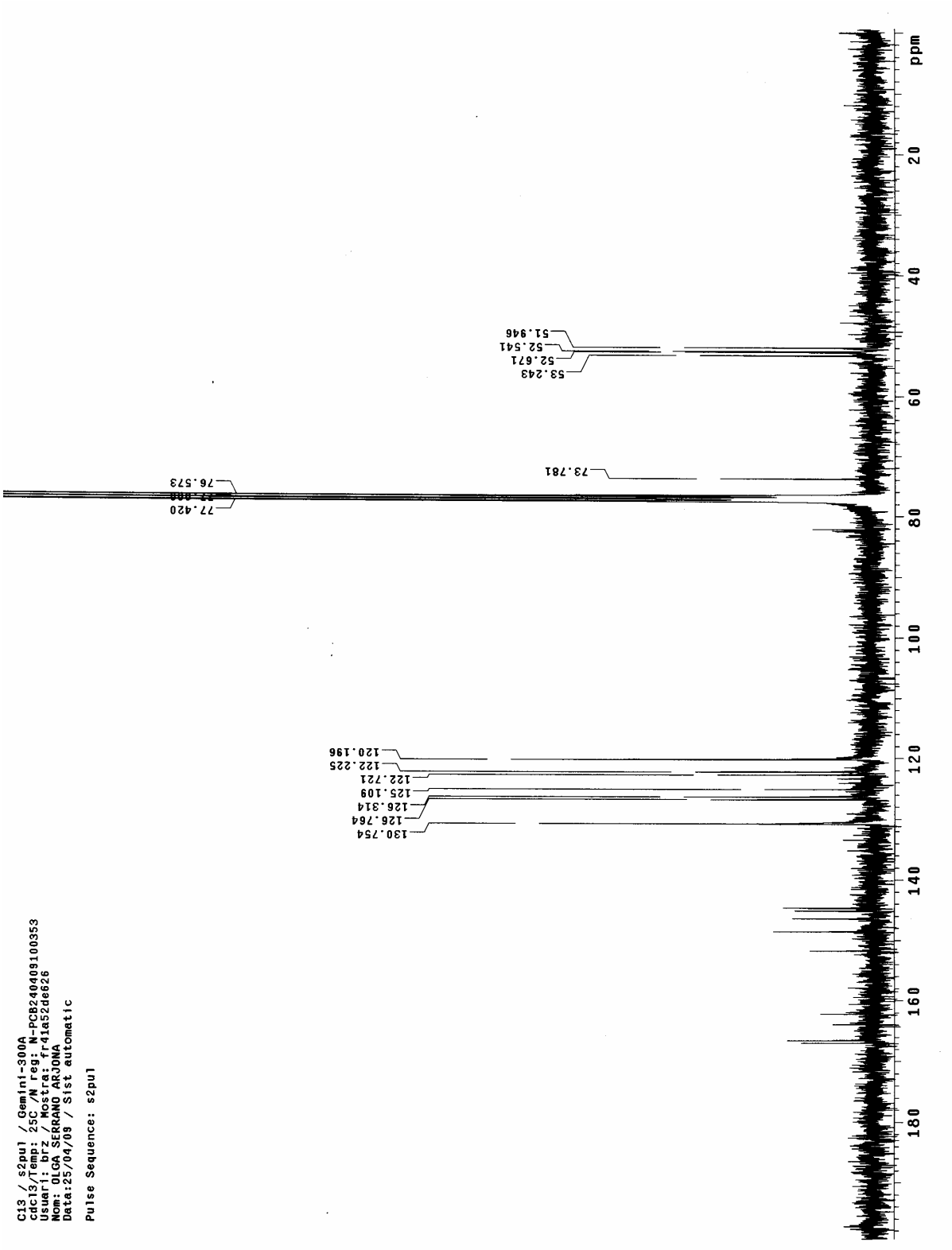


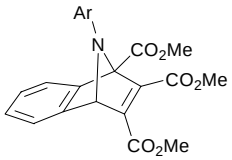
Ar : *p*-$MeO_2CC_6H_4$, **8c**



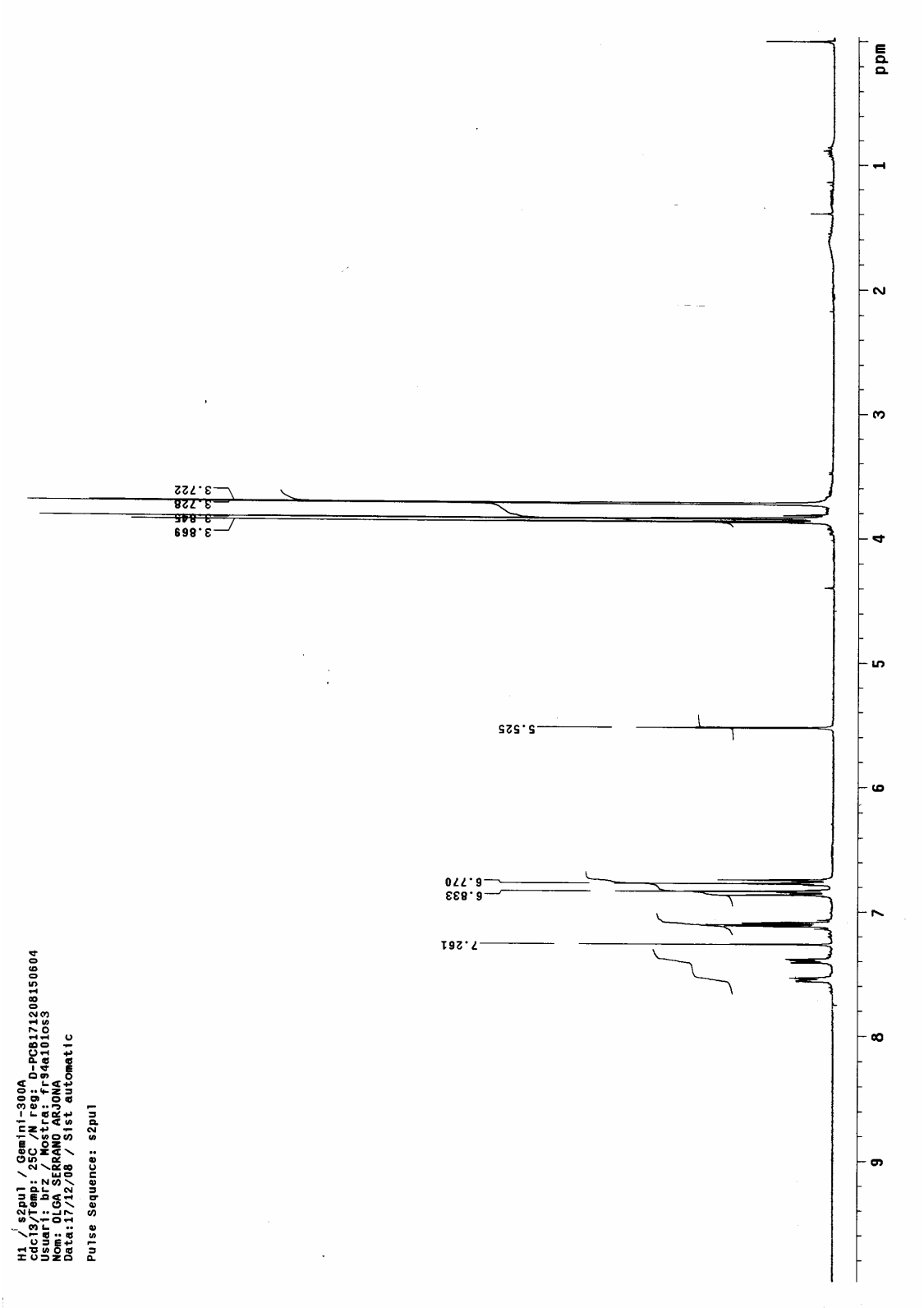


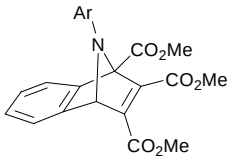
Ar : *p*-$MeO_2CC_6H_4$, **8c**



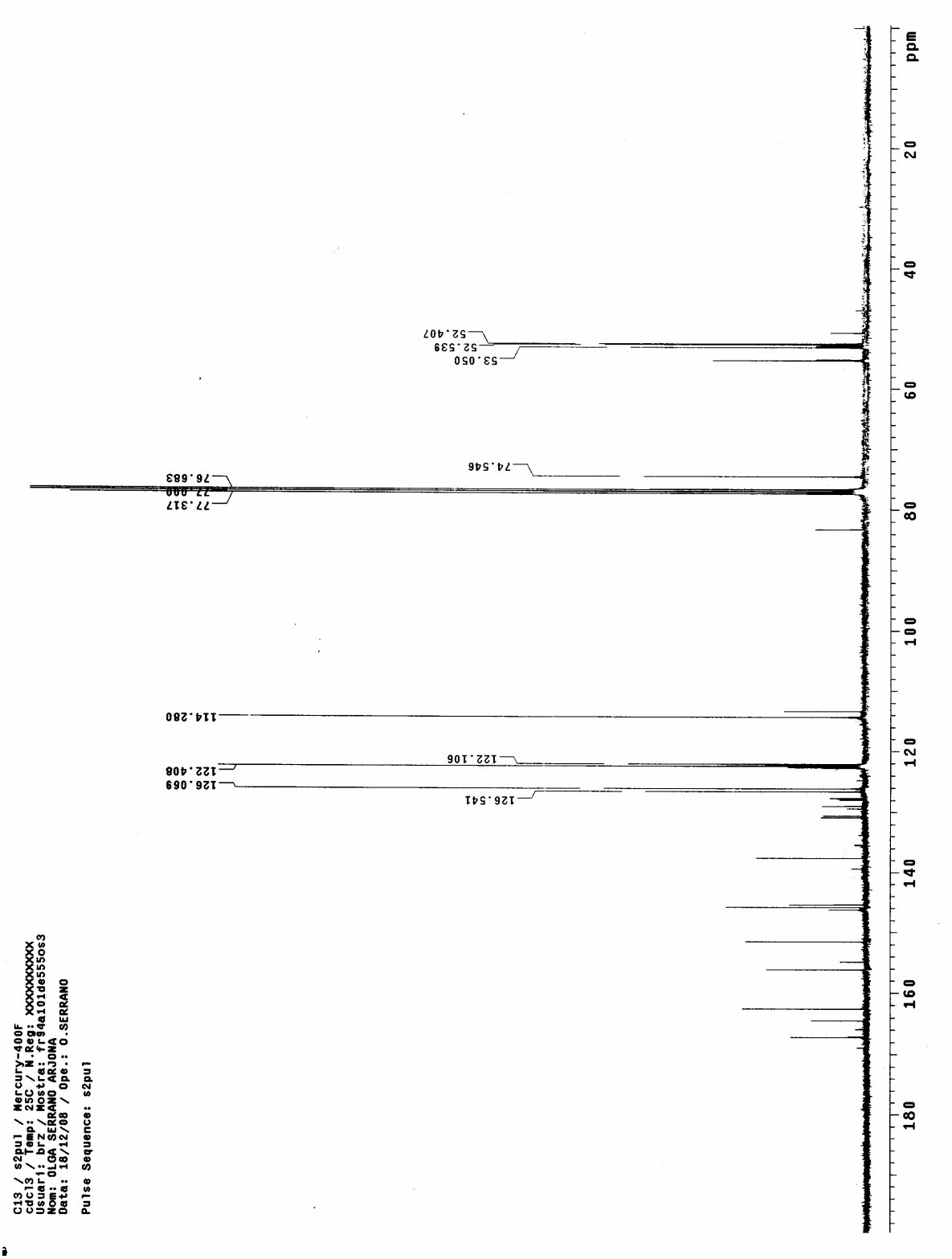


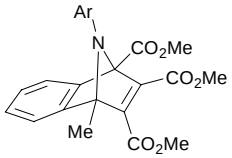
Ar : *p*-MeOC$_6$H$_4$, **8d**



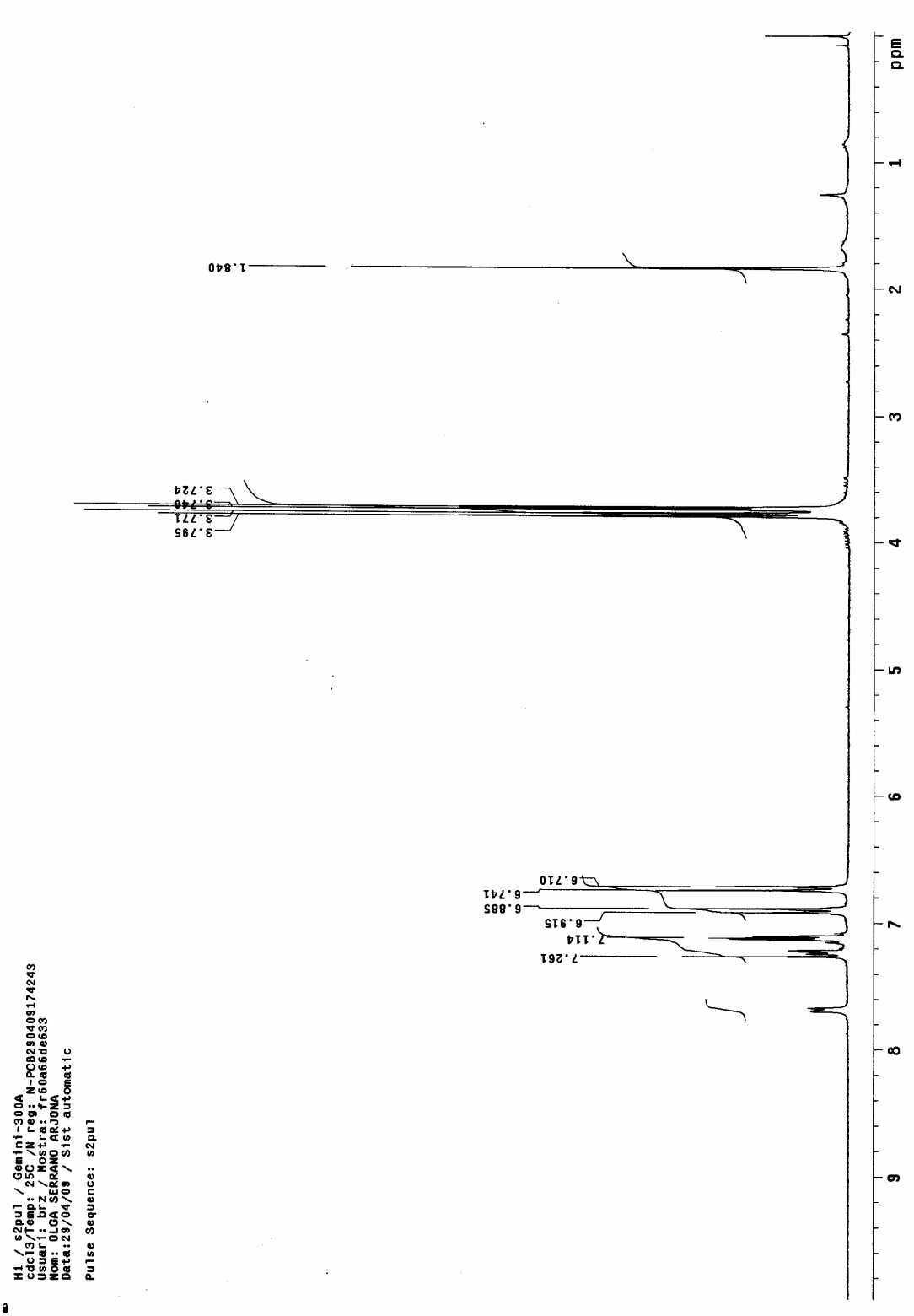


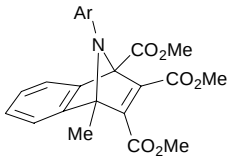
Ar : *p*-MeOC$_6$H$_4$, **8d**



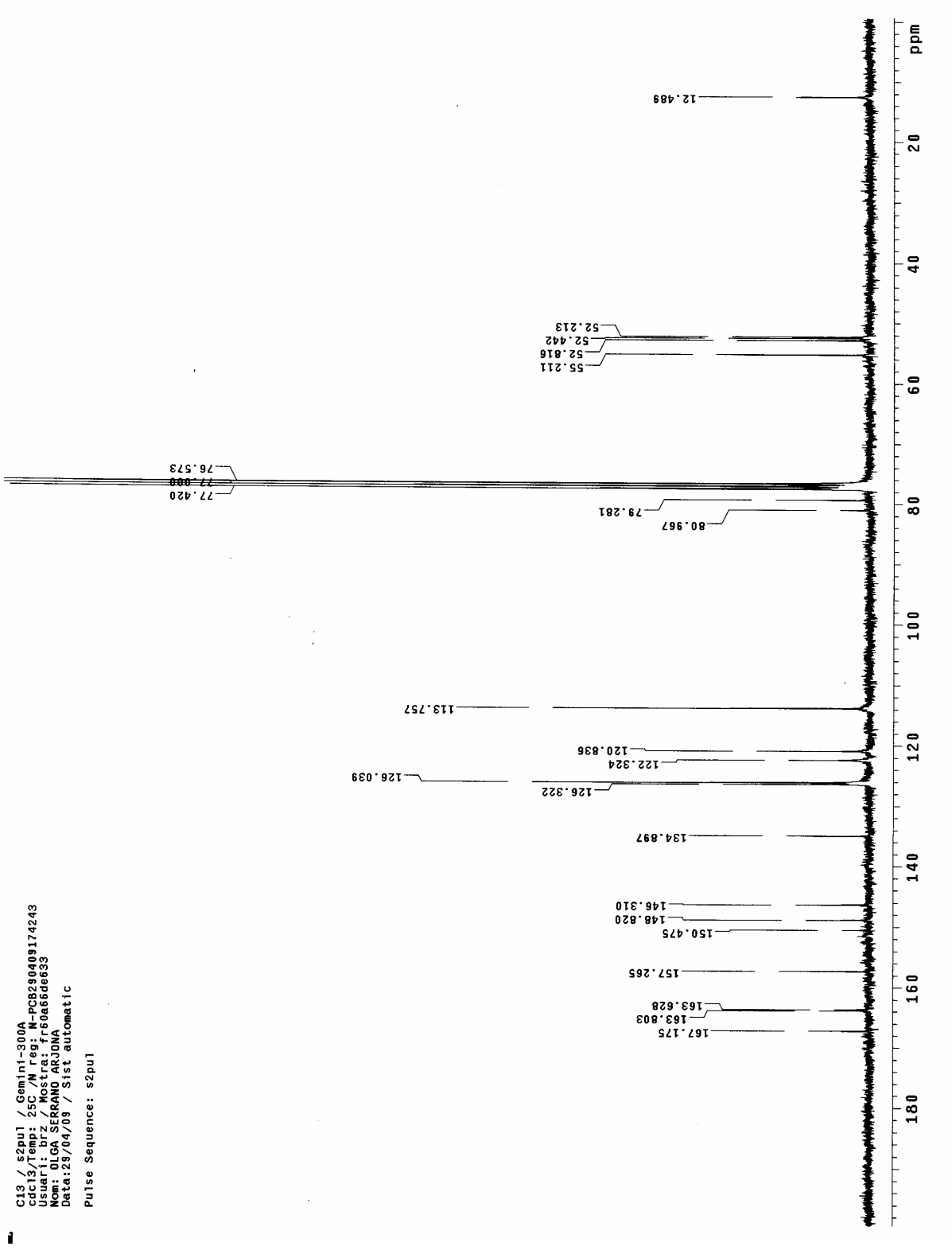


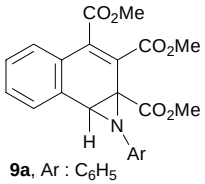
Ar : *p*-MeOC$_6$H$_4$, **8e**



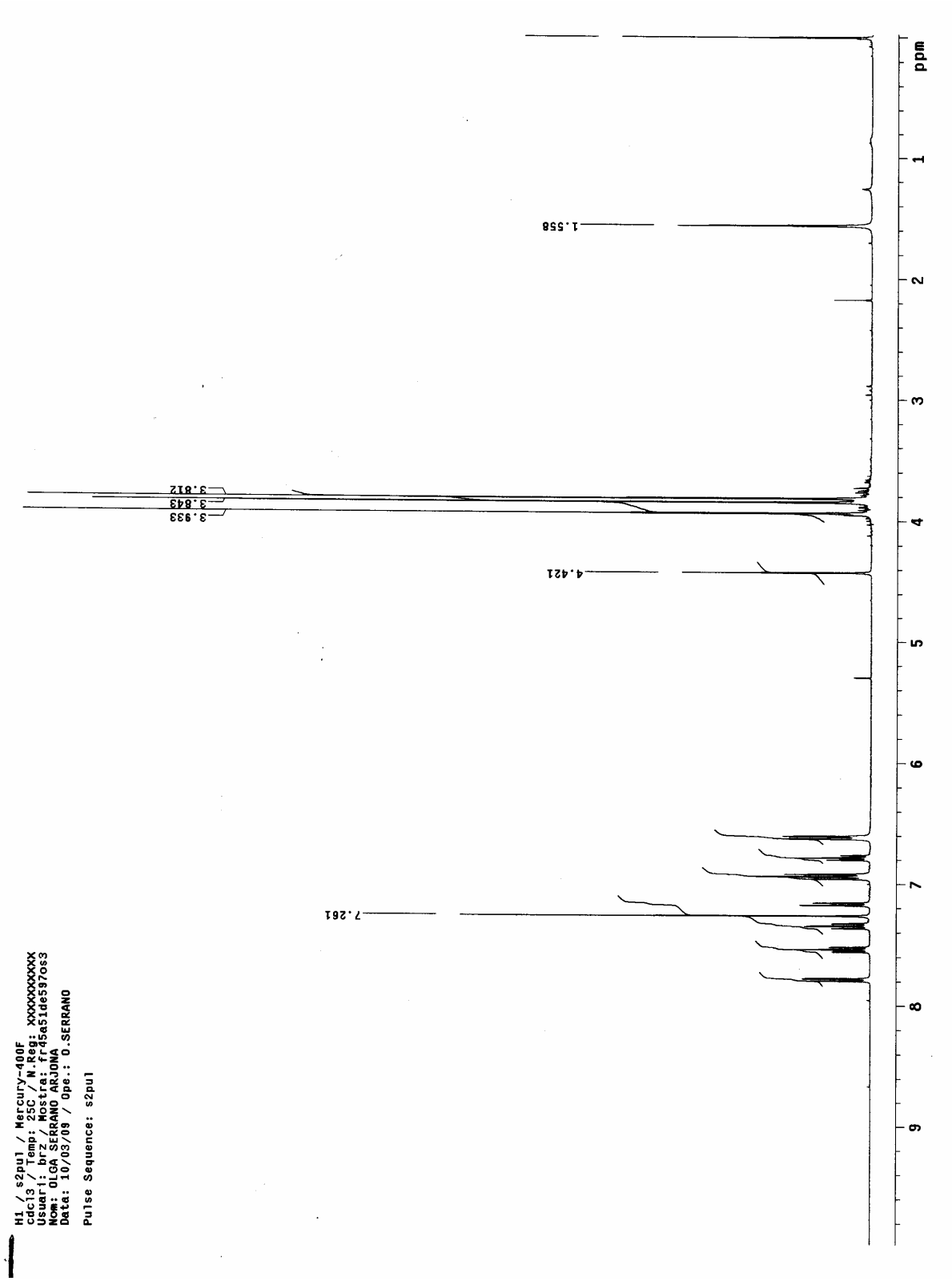


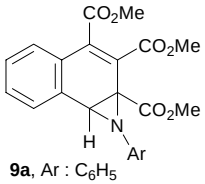
Ar : *p*-MeOC$_6$H$_4$, **8e**



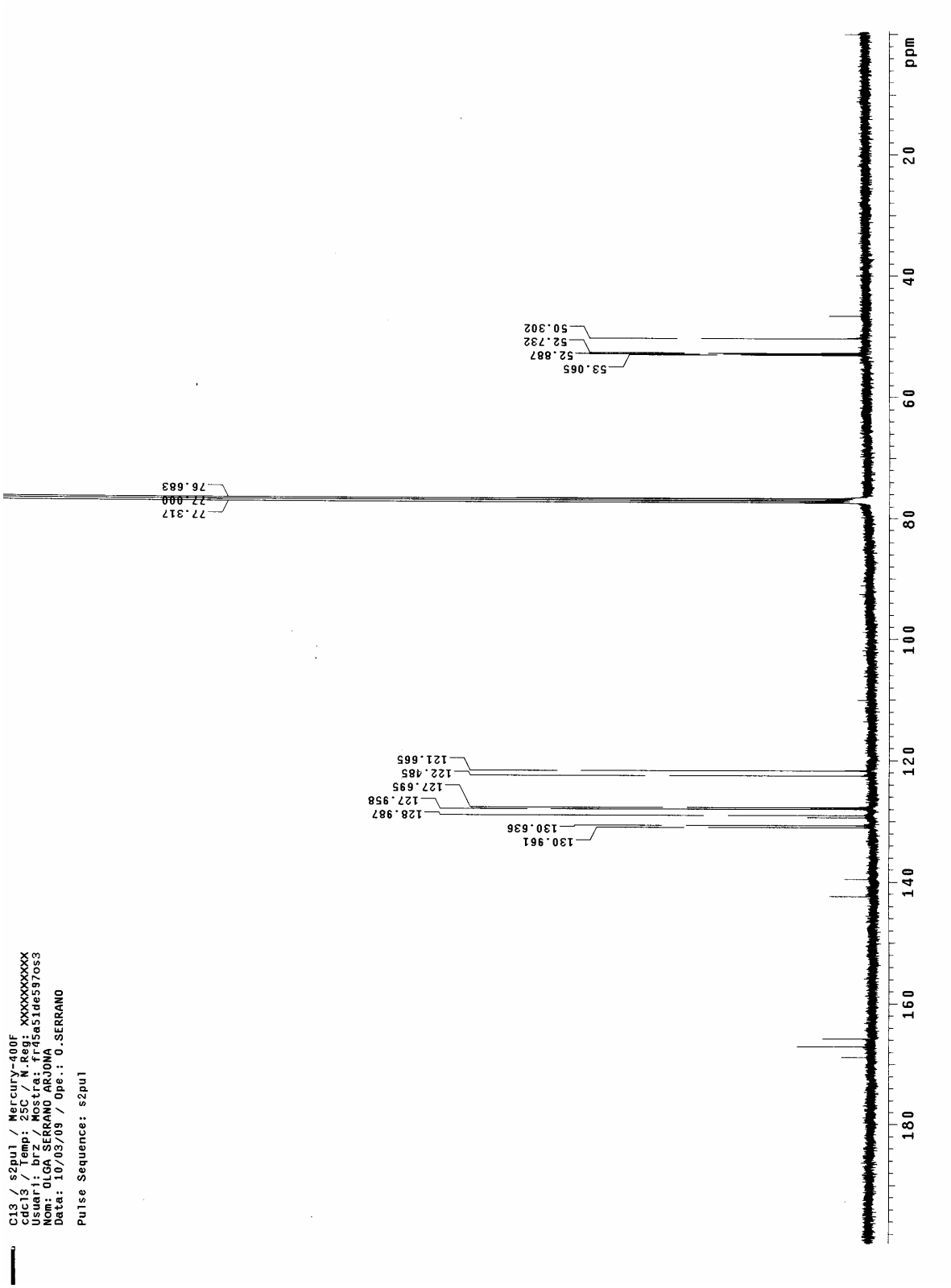

CO2Me
CO2Me
CO2Me
H
N
Ar

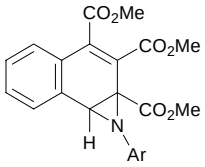
9a, Ar : C_6H_5


H1 / s2pul / Mercury-400F
Cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Nostra: fr45a51de597os3
Nom: OLGA SERRANO ARJONA
Data: 10/03/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
7.261
4.421
3.812
3.843
3.933
1.558
ppm

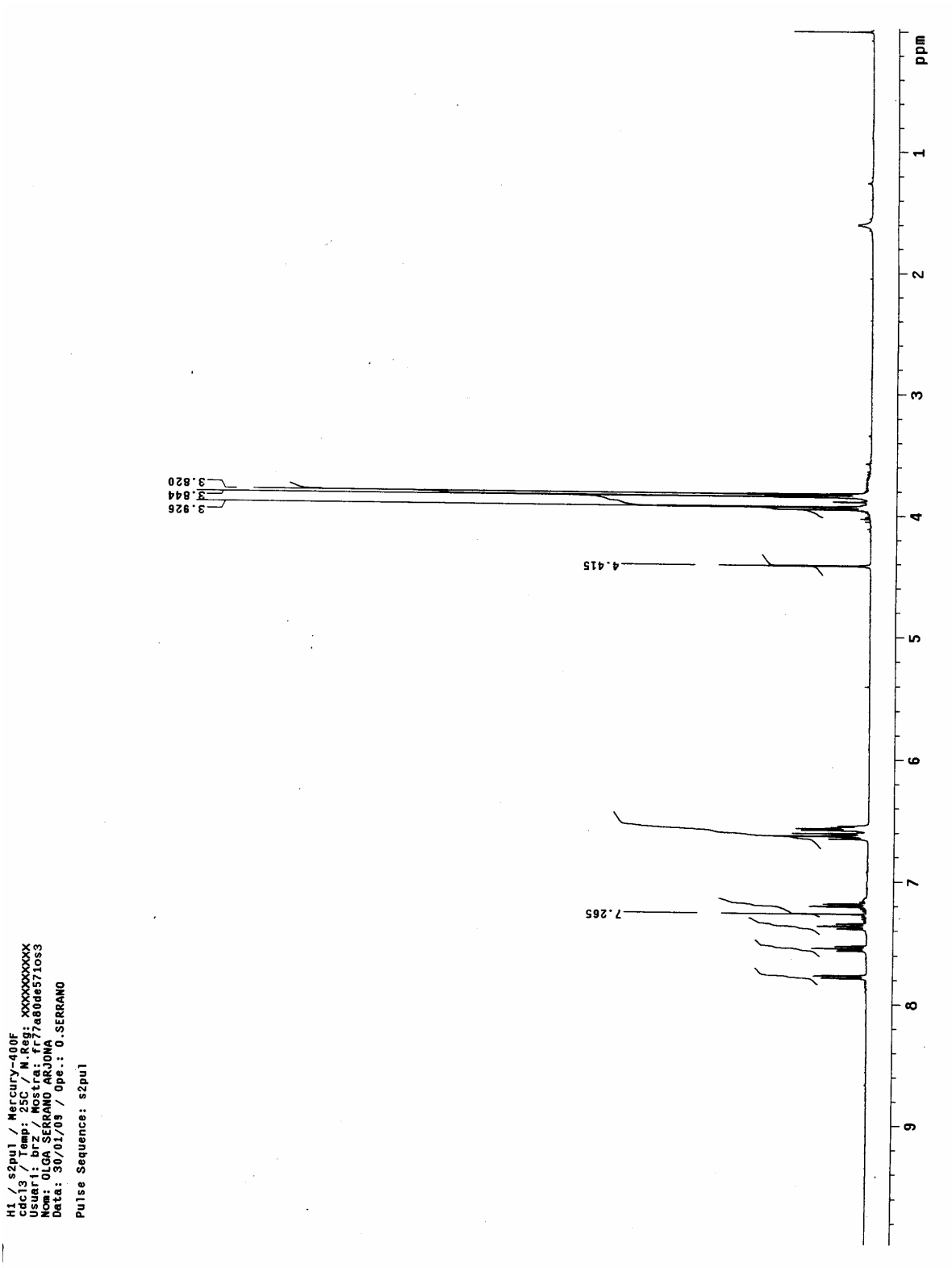

CO2Me
CO2Me
CO2Me
H
N
Ar

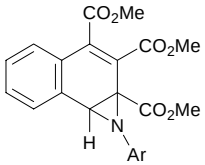
9a, Ar : C_6H_5


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr45a51de597os3
Nom: OLGA SERRANO ARJONA
Data: 10/03/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
50.302
52.732
52.887
53.065
76.683
77.000
77.317
121.665
122.485
127.695
127.958
128.987
130.636
130.961

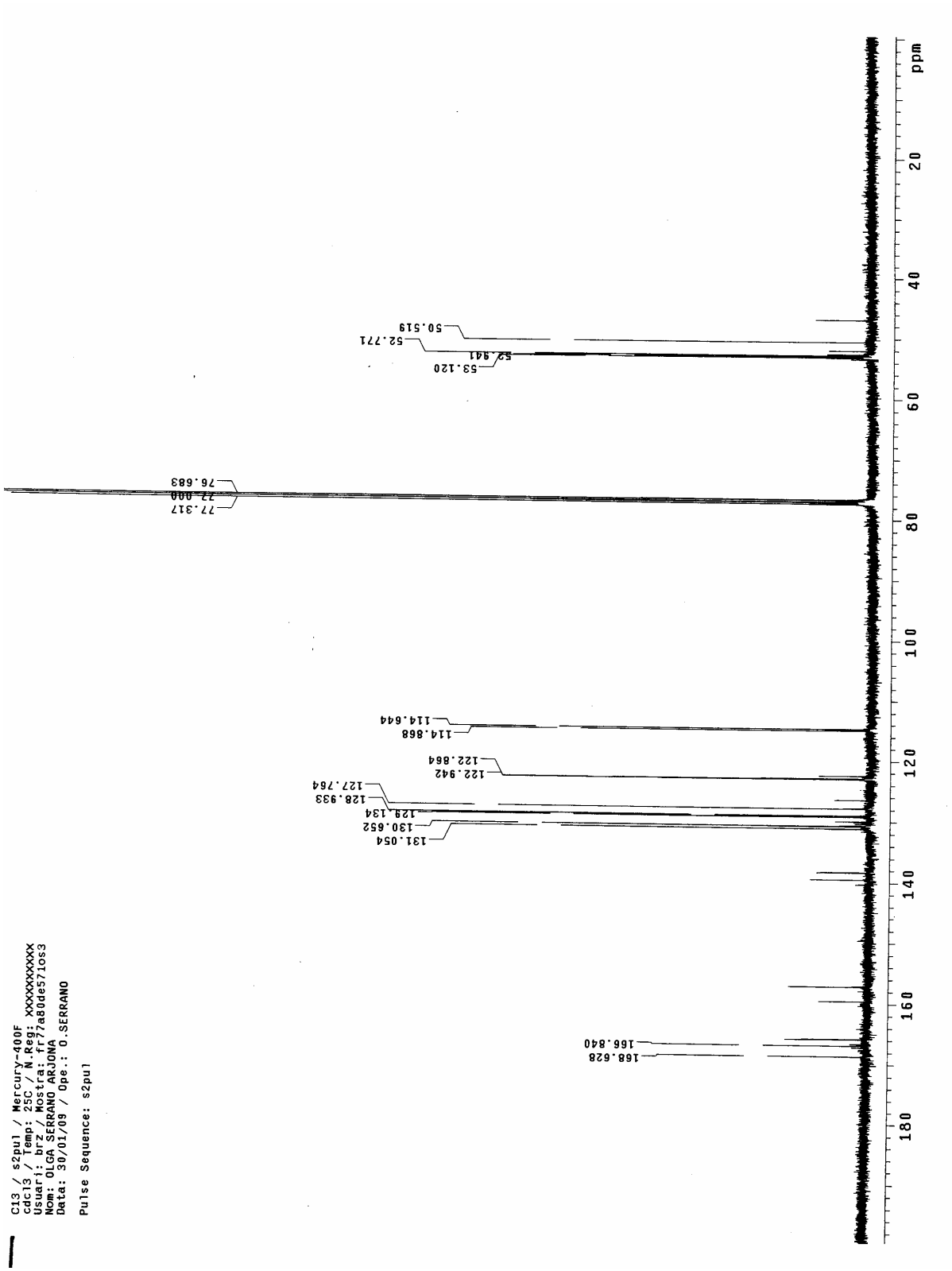

CO2Me
CO2Me
CO2Me
H
N
Ar

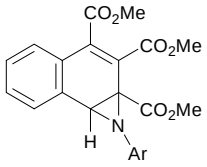
9b, Ar : *p*-FC_6H_4


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr77a80de571os3
Nom: OLGA SERRANO ARJONA
Data: 30/01/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
3.820
3.844
3.926
4.415
7.265
ppm

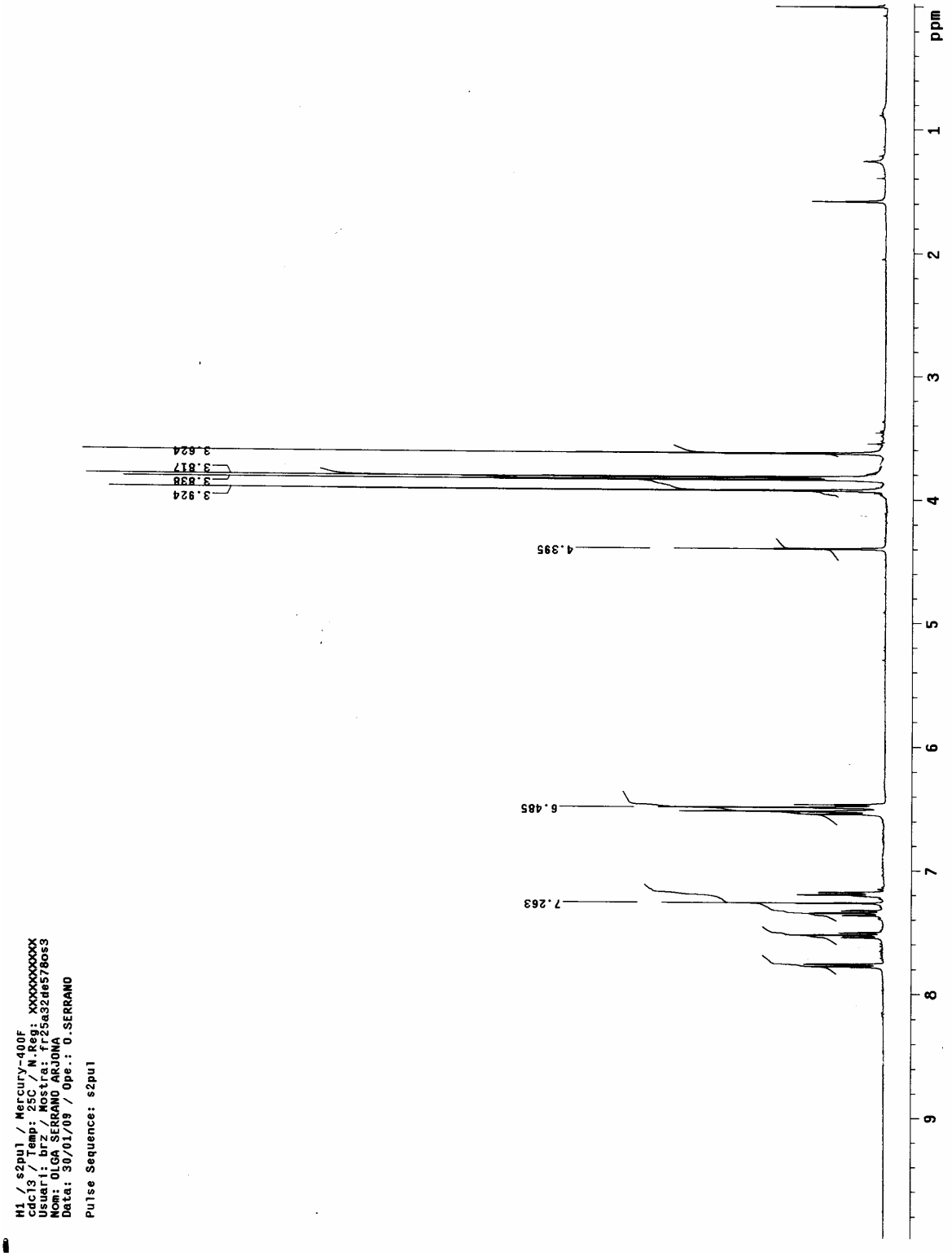

CO2Me
CO2Me
CO2Me
H
N
Ar

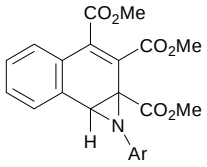
9b, Ar : *p*-FC_6H_4


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr77a80de571os3
Nom: OLGA SERRANO ARJONA
Data: 30/01/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
168.628
166.840
131.054
130.652
129.134
128.933
127.764
122.942
122.864
114.868
114.644
77.317
77.000
76.683
53.120
52.941
52.771
50.519
ppm
180
160
140
120
100
80
60
40
20

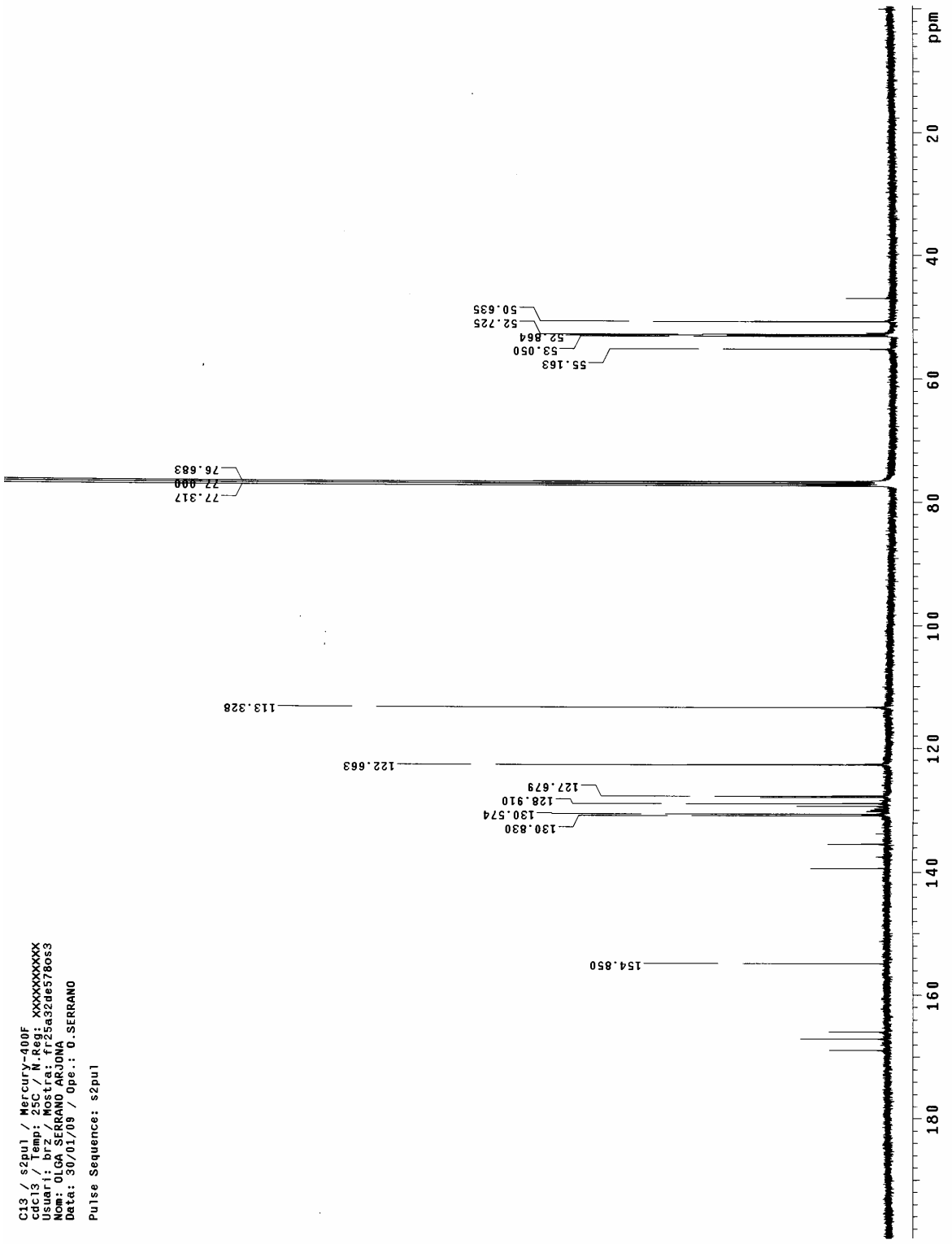


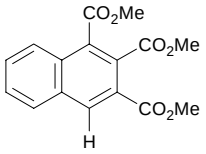
Ar : *p*-$MeOC_6H_4$, **9d**



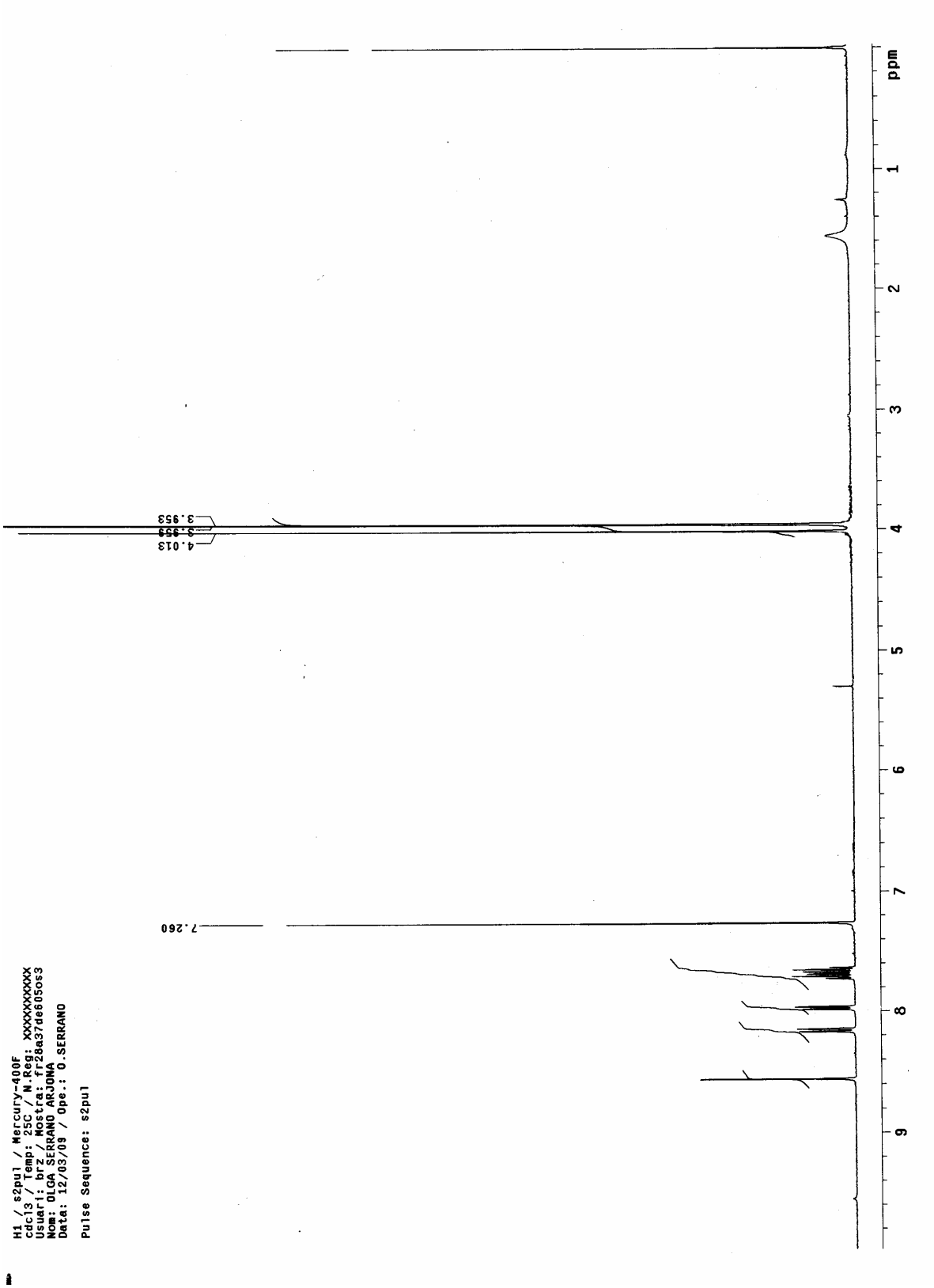


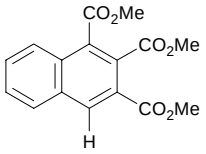
Ar : *p*-MeOC$_6$H$_4$, **9d**



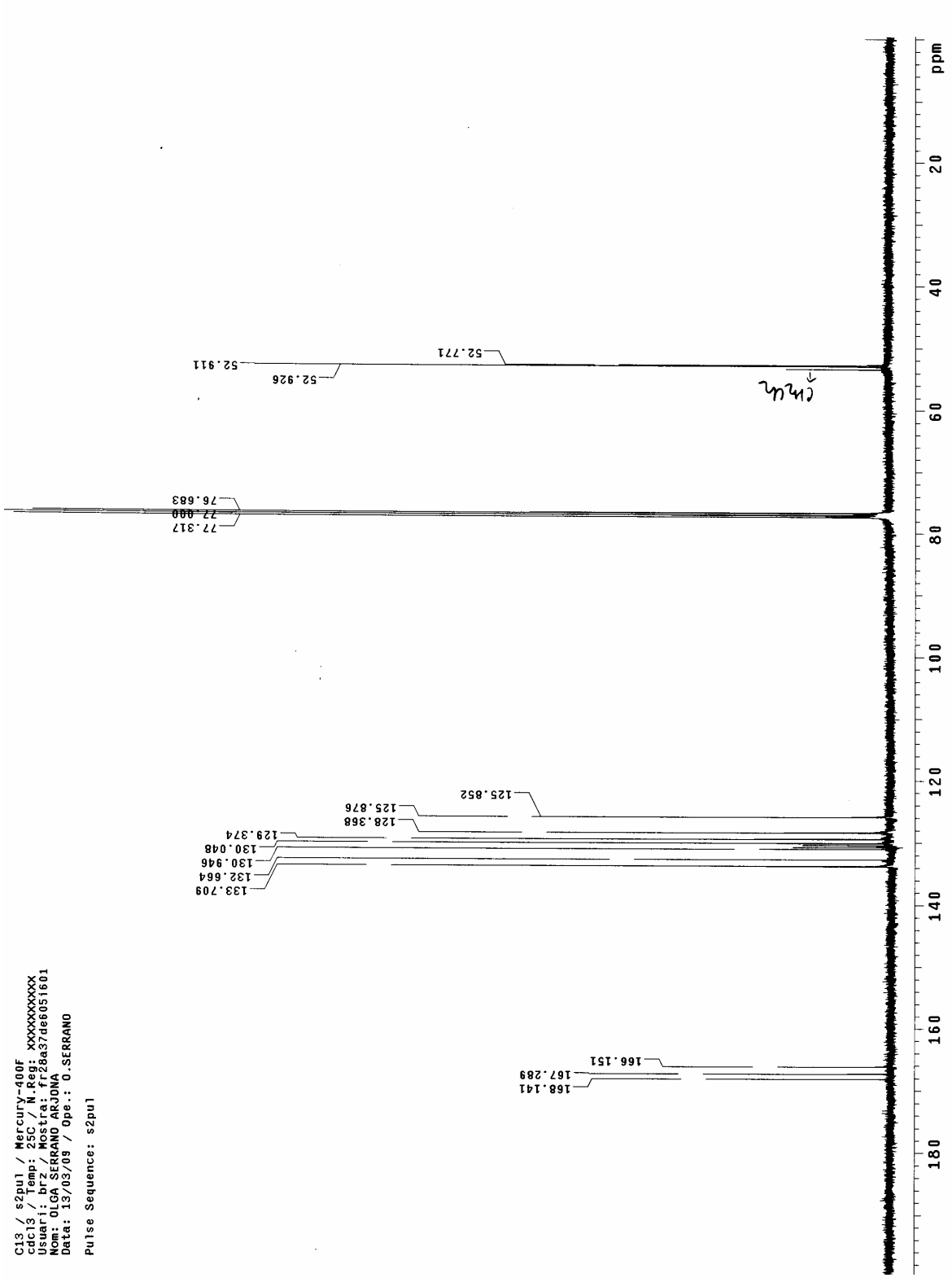

CO2Me
CO2Me
CO2Me
H

10


7.260
4.013
3.959
3.953
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr28a37de605os3
Nom: OLGA SERRANO ARJONA
Data: 12/03/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul


CO2Me
CO2Me
CO2Me
H

10


C13 / s2pul / Mercury-400F XXXXXXXXXX
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr28a37de6051601
Nom: OLGA SERRANO ARJONA
Data: 13/03/09 / Ope.: O.SERRANO
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
52.771
52.926
52.911
77.317
77.000
76.683
125.852
125.876
128.368
129.374
130.048
130.946
132.664
133.709
166.151
167.289
168.141