Supporting Information

Intramolecular palladium-catalysed enolate arylation of 2- and 3-iodoindole derivatives for the synthesis of β-carbolines, γ-carbolines, and pyrrolo[3,4-*b*]indoles

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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.

PREPARATION OF THE STARTING MATERIALS

2-Iodo-1-methyl-1*H*-indole-3-carbaldehyde (2)

$POCl_3$ (1.3 mL, 13.95 mmol) was added slowly to ice-cooled dry DMF (14 mL). The solution was stirred, under Argon, for 15 min at room temperature and then cooled to 0 ºC. A solution of 2-iodo-1-methylindole[1] (**1**, 3.36 g, 13.07 mmol) in DMF (14 mL) was then added dropwise. The mixture was stirred at room temperature for 3 h, poured into ice water and made alkaline with 2M NaOH. The resulting mixture was extracted with ether. The organic layer was washed with brine, dried and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to 9:1 hexanes-EtOAc) to give **2** (3.20 g, 86%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.81 (s, 3H), 7.23-7.34 (m, 3H), 8.31 (m, 1H), 9.83 (s, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 34.6 (CH_3), 100.6 (C), 109.9 (CH), 119.1 (C), 120.7 (CH), 122.8 (CH), 124.0 (CH), 126.2 (C), 139.2 (C), 187.6 (CH). ESI-HRMS $[M+H]^+$ calcd for $C_{10}H_9INO$ 285.9723, found 285.9725.

N-[(2-Iodo-1-methyl-1*H*-indol-3-yl)methyl]-*N*-methylamine (3)

To a solution of aldehyde **2** (1 g, 3.5 mmol) in CH_2Cl_2 (5 mL) methylamine (4.4 mL of 8M solution in EtOH, 35.2 mmol) and AcOH (0.2 mL, 3.5 mmol) were added. The mixture was stirred at room temperature for 6 h. The solvent was removed *in vacuo*, the residue was dissolved in MeOH (10 mL), and $NaBH_4$ (0.4 g, 10.5 mmol) was slowly added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo* and the residue was partitioned between CH_2Cl_2 and water. The organic layer was dried and concentrated. The residue was purified by chromatography

[1] J. Bergman and N. Eklund, *Tetrahedron* 1980, **36**, 1439.

(SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 10%) to give amine **3** (0.95 g, 90%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.42 (broad s, 1H), 2.46 (s, 3H), 3.76 (s, 3H), 3.93 (s, 2H), 7.09 (ddd, *J* = 7.6, 7.2, and 0.8 Hz, 1H), 7.17 (ddd, *J* = 8, 7.2, and 1.2 Hz, 1H), 7.30 (d, *J* = 8 Hz, 1H), 7.65 (d, *J* = 7.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 34.2 (CH_3), 35.6 (CH_3), 47.9 (CH_2), 88.8 (C), 109.6 (CH), 118.1 (CH), 118.6 (C), 119.6 (CH), 122.0 (CH), 127.8 (C), 138.5 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{11}H_{14}IN_2$ 301.0202, found 301.0205.

4-{*N*-[(2-Iodo-1-methyl-1*H*-indol-3-yl)methyl]-*N*-methylamino}-2-butanone (4)

To a solution of amine **3** (560 mg, 1.87 mmol) in MeOH (16 mL), MVK (0.53 mL, 6.55 mmol) and Et_3N (0.65 mL, 4.68 mmol) were added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo* and the residue was partitioned between CH_2Cl_2 and water. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 1%) to give ketone **4** (450 mg, 65%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.10 (s, 3H), 2.22 (s, 3H), 2.66 (m, 2H), 2.77 (m, 2H), 3.64 (s, 2H), 3.76 (s, 3H), 7.07 (ddd, *J* = 8.1, 6.9, and 1.2 Hz, 1H), 7.16 (ddd, *J* = 8.4, 6.9, and 1.2 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 29.9 (CH_3), 34.3 (CH_3), 41.9 (CH_3), 42.0 (CH_2), 52.1 (CH_2), 55.0 (CH_2), 89.6 (C), 109.4 (CH), 117.4 (C), 118.8 (CH), 119.4 (CH), 121.9 (CH), 128.2 (C), 138.6 (C), 208.4 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{15}H_{20}IN_2O$ 371.0614, found 371.0615.

Methyl 3-{*N*-[(2-iodo-1-methyl-1*H*-indol-3-yl)methyl]-*N*-methylamino}propanoate (5)

To a solution of amine **3** (430 mg, 1.43 mmol) in MeOH (5 mL), methyl acrylate (0.32 mL, 3.58 mmol) was added. The mixture was stirred at room temperature for 24 h. The solvent was removed *in vacuo* and the residue was purified by chromatography (SiO_2, from hexanes to 7:3 hexanes-EtOAc) to give ester **5** (310 mg, 56%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.25 (s, 3H), 2.58 (t, *J* = 7.2 Hz, 2H), 2.82 (t, *J* = 7.2 Hz, 2H), 3.64 (s, 3H), 3.67 (s, 2H), 3.76 (s, 3H), 7.08 (ddd, *J* = 8, 7.2, and 0.8 Hz, 1H), 7.17 (ddd, *J* = 8.4, 7.2, and 0.8 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 1H), 7.70 (dd, *J* = 8 and 0.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 32.9 (CH_2), 34.2 (CH_3), 41.8 (CH_3), 51.4 (CH_3), 52.9 (CH_2), 54.9 (CH_2), 89.3 (C), 109.4 (CH), 117.6 (C), 119.0 (CH), 119.3 (CH), 121.9 (CH),

128.3 (C), 138.6 (C), 173.1 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{15}H_{20}IN_2O_2$ 387.0564, found 387.0562.

1-{*N*-[(2-Iodo-1-methyl-1*H*-indol-3-yl)methyl]-*N*-methylamino}-2-propanone (6)

To a solution of amine **3** (260 mg, 0.87 mmol) in acetonitrile (5 mL), DIPEA (0.7 mL, 4.0 mmol) and chloroacetone (0.11 mL, 1.39 mmol) were added. After 2.5 h at reflux, the solvent was removed *in vacuo* and the residue was partitioned between dichloromethane and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 1%) to give ketone **6** (220 mg, 71%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.11 (s, 3H), 2.35 (s, 3H), 3.17 (s, 2H), 3.75 (s, 2H), 3.77 (s, 3H), 7.11 (ddd, *J* = 7.6, 7.2, and 0.9 Hz, 1H), 7.19 (ddd, *J* = 8.1, 7.2, and 1.5 Hz, 1H), 7.31 (dd, *J* = 8.1 and 0.9 Hz, 1H), 7.80 (dd, *J* = 7.6 and 1.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 27.5 (CH_3), 34.3 (CH_3), 43.1 (CH_3), 54.8 (CH_2), 66.8 (CH_2), 89.9 (C), 109.5 (CH), 117.1 (C), 118.9 (CH), 119.5 (CH), 122.0 (CH), 128.2 (C), 138.6 (C), 209.0 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{14}H_{18}IN_2O$ 357.0458, found 357.0460.

Methyl 2-{*N*-benzyl-*N*-[(2-iodo-1-methyl-1*H*-indol-3-yl)methyl]amino}acetate (7)

To a solution of aldehyde **2** (0.5 g, 1.75 mmol) in DCE (3 mL), glycine methyl ester hydrochloride (0.55 g, 4.38 mmol), AcOH (0.22 mL, 3.85 mmol), and Et_3N (0.54 mL, 3.85 mmol) were added. After 10 min at room temperature $NaBH(OAc)_3$ (0.89 g, 4.2 mmol) was slowly added. The resulting mixture was stirred at room temperature for 24 h, poured into 1N NaOH and extracted with CH_2Cl_2. The organic layer was dried and concentrated. The residue was dissolved in acetonitrile (10 mL) and benzyl bromide (0.25 mL, 2.1 mmol) and K_2CO_3 (0.48 g, 3.5 mmol) were added. The mixture was stirred at 55 ºC for 2.5 h. The solvent was removed *in vacuo* and the residue was partitioned between dichloromethane and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to 9:1 hexanes-EtOAc) to give ester **7** (0.45 g, 57%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.29 (s, 2H), 3.67 (s, 3H), 3.75 (s, 3H), 3.86 (s, 2H), 4.00 (s, 3H), 7.05-7.38 (m, 8H), 7.80 (dd, *J* = 8 and 1.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 34.2 (CH_3), 51.1 (CH_3), 51.4 (CH_2), 52.8 (CH_2), 57.6 (CH_2), 89.5 (C), 109.4 (CH), 117.5 (C), 119.3 (CH), 119.4 (CH), 122.0 (CH), 126.9 (CH), 128.2 (2 CH), 128.3 (C),

129.1 (2 CH), 138.7 (C), 139.4 (C), 172.1 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{20}H_{22}IN_2O_2$ 449.0720, found 449.0718.

3-Iodo-1-methyl-1*H*-indole-2-carbaldehyde (9)

To a suspension of NaH (710 mg of 60% dispersion in mineral oil, 17.7 mmol) in THF (70 mL), cooled to 0 ºC, a solution of aldehyde **8**[2] (2.4 g, 8.85 mmol) in THF (30 mL) was added dropwise. The mixture was stirred at room temperature for 1 h and ICH_3 (1.4 mL, 22.13 mmol) was added. After 24 h at room temperature, the reaction mixture was poured into ice water, was extracted with EtOAc and washed with brine. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to hexanes-EtOAc 1:1) to give aldehyde **9** (2.1 g, 83%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 4.08 (s, 3H), 7.24 (ddd, *J* = 7.5, 6.9, and 1.2 Hz, 1H), 7.35 (dm, *J* = 7.5 Hz, 1H), 7.46 (ddd, *J* = 7.5, 6.9, and 1.2 Hz, 1H), 7.57 (dm, *J* = 7.5 Hz, 1H), 9.99 (s, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 31.8 (CH_3), 75.1 (C), 110.6 (CH), 121.7 (CH), 123.6 (CH), 128.0 (CH), 129.7 (C), 131.4 (C), 140.4 (C), 184.6 (CH). ESI-HRMS $[M+H]^+$ calcd for $C_{10}H_9INO$ 285.9723, found 285.9729.

***N*-[(3-Iodo-1-methyl-1*H*-indol-2-yl)methyl]-*N*-methylamine (10)**

To a solution of aldehyde **9** (1.1 g, 3.86 mmol) in CH_2Cl_2 (6 mL) methylamine (4.8 mL of 8M solution in EtOH, 38.5 mmol) and AcOH (0.22 mL, 3.86 mmol) were added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo*, the residue was dissolved in MeOH (10 mL) and $NaBH_4$ (0.4 g, 10.5 mmol) was slowly added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo* and the residue was partitioned between CH_2Cl_2 and water. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 10%) to give amine **10** (1.05 g, 91%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.49 (s, 3H), 3.85 (s, 3H), 3.98 (s, 2H), 7.17 (m, 1H), 7.26 (m, 2H), 7.41 (dm, *J* = 7.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 30.6 (CH_3), 36.1 (CH_3), 47.4 (CH_2), 59.6 (C), 109.4 (CH), 120.2 (CH), 121.1 (CH), 122.7 (CH), 129.6 (C), 137.8 (C), 138.3 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{11}H_{14}IN_2$ 301.0202, found 301.0204.

[2] T. Choshi, T. Sada, H. Fujimoto, C. Nagayama, E. Supino and S. Hibino, *J. Org. Chem.* 1997, **62**, 2535.

4-{*N*-[(3-Iodo-1-methyl-1*H*-indol-2-yl)methyl]-*N*-methylamino}-2-butanone (11)

To a solution of amine **10** (550 mg, 1.83 mmol) in MeOH (15 mL), MVK (0.53 mL, 6.55 mmol) and Et_3N (0.65 mL, 4.68 mmol) were added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo* and the residue was partitioned between CH_2Cl_2 and water. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 1%) to give ketone **11** (583 mg, 86%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.05 (s, 3H), 2.22 (s, 3H), 2.59 (t, *J* = 7.2 Hz, 2H), 2.79 (t, *J* = 7.2 Hz, 2H), 3.72 (s, 2H), 3.80 (s, 3H), 7.17 (m, 1H), 7.27 (m, 2H), 7.41 (d, *J* = 8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 29.8 (CH_3), 30.5 (CH_3), 41.4 (CH_3), 42.5 (CH_2), 51.9 (CH_2), 53.9 (CH_2), 61.0 (C), 109.3 (CH), 120.2 (CH), 121.2 (CH), 122.7 (CH), 129.6 (C), 136.6 (C), 138.1 (C), 207.9 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{15}H_{20}IN_2O$ 371.0614, found 371.0617.

Methyl 3-{*N*-[(3-iodo-1-methyl-1*H*-indol-2-yl)methyl]-*N*-methylamino}propanoate (12)

To a solution of amine **10** (480 mg, 1.6 mmol) in MeOH (5 mL), methyl acrylate (0.36 mL, 4.0 mmol) was added. The mixture was stirred at room temperature for 24 h. The solvent was removed *in vacuo* and the residue was purified by chromatography (SiO_2, from hexanes to 7:3 hexanes-EtOAc) to give ester **12** (435 mg, 70%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.21 (s, 3H), 2.49 (t, *J* = 6.8 Hz, 2H), 2.79 (t, *J* = 6.8 Hz, 2H), 3.60 (s, 3H), 3.73 (s, 2H), 3.79 (s, 3H), 7.16 (m, 1H), 7.25 (m, 2H), 7.41 (dm, *J* = 8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 30.4 (CH_3), 32.7 (CH_2), 41.2 (CH_3), 51.5 (CH_3), 52.7 (CH_2), 53.9 (CH_2), 60.9 (C), 109.2 (CH), 120.1 (CH), 121.2 (CH), 122.6 (CH), 129.6 (C), 136.6 (C), 138.0 (C), 172.8 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{15}H_{20}IN_2O_2$ 387.0564, found 387.0561.

1-{*N*-[(3-Iodo-1-methyl-1*H*-indol-2-yl)methyl]-*N*-methylamino}-2-propanone (13)

To a solution of amine **10** (475 mg, 1.58 mmol) in acetonitrile (10 mL), DIPEA (1.4 mL, 8.0 mmol) and chloroacetone (0.26 mL, 3.32 mmol) were added. After 2.5 h at reflux, the solvent was removed *in vacuo* and the residue was partitioned between dichloromethane and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 1%) to give ketone **13** (450 mg, 80%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ

2.05 (s, 3H), 2.36 (s, 3H), 3.24 (s, 2H), 3.82 (s, 2H), 3.94 (s, 3H), 7.18 (m, 1H), 7.28 (m, 2H), 7.42 (d, J = 7.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 27.5 (CH_3), 30.7 (CH_3), 43.0 (CH_3), 53.2 (CH_2), 61.5 (C), 66.0 (CH_2), 109.5 (CH), 120.3 (CH), 121.3 (CH), 122.9 (CH), 129.5 (C), 136.2 (C), 138.1 (C), 206.8 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{14}H_{18}IN_2O$ 357.0458, found 357.0457.

Methyl 2-{*N*-benzyl-*N*-[(3-iodo-1-methyl-1*H*-indol-2-yl)methyl]amino}acetate (14)

To a solution of aldehyde **9** (0.35 g, 1.23 mmol) in DCE (3 mL), glycine methyl ester hydrochloride (0.39 g, 3.1 mmol), AcOH (0.16 mL, 2.8 mmol), and Et_3N (0.4 mL, 2.8 mmol) were added. After 10 min at room temperature $NaBH(OAc)_3$ (0.63 g, 2.97 mmol) was slowly added. The resulting mixture was stirred at room temperature for 24 h, poured into 1N NaOH and extracted with CH_2Cl_2. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to 1:1 hexanes-EtOAc) to give methyl 2-{*N*-[(3-iodo-1-methyl-1*H*-indol-2-yl)methyl]amino}acetate (0.28 g, 64%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.41 (s, 2H), 3.71 (s, 3H), 3.87 (s, 3H), 4.07 (s, 2H), 7.16 (m, 1H), 7.26 (m, 1H), 7.40 (d, J = 8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 30.6 (CH_3), 44.7 (CH_2), 49.1 (CH_2), 51.8 (CH_3), 60.3 (C), 109.4 (CH), 120.3 (CH), 121.2 (CH), 122.8 (CH), 129.5 (C), 137.3 (C), 138.0 (C), 172.8 (C).

To a solution of methyl 2-{*N*-[(3-iodo-1-methyl-1*H*-indol-2-yl)methyl]amino}acetate (0.28 g, 0.78 mmol) in acetonitrile (10 mL), benzyl bromide (0.11 mL, 0.94 mmol) and K_2CO_3 (0.21 g, 1.56 mmol) were added. The mixture was stirred at 55 ºC for 2.5 h. The solvent was removed *in vacuo* and the residue was partitioned between dichloromethane and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to 9:1 hexanes-EtOAc) to give ester **14** (0.32 g, 91%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.28 (s, 2H), 3.64 (s, 3H), 3.80 (s, 2H), 3.84 (s, 3H), 4.03 (s, 2H), 7.12-7.42 (m, 9H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 30.7 (CH_3), 50.2 (CH_2), 51.4 (CH_3), 53.3 (CH_2), 58.2 (CH_2), 61.6 (C), 109.4 (CH), 120.2 (CH), 121.2 (CH), 122.8 (CH), 127.3 (CH), 128.3 (2 CH), 129.1 (2 CH), 129.6 (C), 136.3 (C), 138.2 (C), 138.4 (C), 171.5 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{20}H_{22}IN_2O_2$ 449.0720, found 449.0716.

***N*-[(3-Iodo-1-phenylsulfonyl-1*H*-indol-2-yl)methyl]-*N*-methylamine (16)**

To a solution of aldehyde **15**[2] (0.6 g, 1.46 mmol) in CH_2Cl_2 (5 mL), methylamine (1.9 mL of 8M solution in EtOH, 15.2 mmol) and AcOH (0.1 mL, 1.75 mmol) were added. The mixture was stirred overnight at room temperature. The solvent was removed *in vacuo*, the residue was dissolved in MeOH (10 mL) and $NaBH_4$ (0.22 g, 5.84 mmol) was slowly added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo* and the residue was partitioned between CH_2Cl_2 and water. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 10%) to give amine **16** (0.55 g, 88%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.43 (s, 4H), 4.27 (s, 2H), 7.30-7.44 (m, 5H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.881 (m, 2H), 8.14 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 34.6 (CH_3), 48.2 (CH_2), 77.2 (C), 114.9 (CH), 122.3 (CH), 124.5 (CH), 126.2 (CH), 126.5 (2 CH), 129.3 (2 CH), 131.4 (C), 134.1 (CH), 136.8 (C), 138.1 (C), 138.2 (C).

4-{*N*-[(3-Iodo-1-phenylsulfonyl-1*H*-indol-2-yl)methyl]-*N*-methylamino}-2-butanone (17)

To a solution of amine **16** (0.54 g, 1.27 mmol) in MeOH (15 mL), MVK (0.4 mL, 4.9 mmol) and Et_3N (0.5 mL, 3.6 mmol) were added. The mixture was stirred at room temperature overnight. The solvent was removed *in vacuo* and the residue was partitioned between CH_2Cl_2 and water. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 3%) to give ketone **17** (0.33 g, 52%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.10 (s, 3H), 2.17 (s, 3H), 2.55 (t, *J* = 7.2 Hz, 2H), 2.78 (t, *J* = 7.2 Hz, 2H), 4.03 (s, 2H), 7.28-7.44 (m, 5H), 7.52 (tt, *J* = 8 and 1.2 Hz, 1H), 8.03-8.10 (m, 3H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 30.0 (CH_3), 40.7 (CH_2), 41.0 (CH_3), 51.5 (CH_2), 53.4 (CH_2), 76.5 (C), 114.7 (CH), 122.3 (CH), 123.9 (CH), 126.0 (CH), 127.1 (2 CH), 128.8 (2 CH), 130.9 (C), 133.5 (CH), 136.6 (C), 136.9 (C), 139.4 (C), 208.0 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{20}H_{22}IN_2O_3S$ 497.0390, found 497.0388.

1-{*N*-[(3-Iodo-1-phenylsulfonyl-1*H*-indol-2-yl)methyl]-*N*-methylamino}-2-propanone (18)

To a solution of amine **16** (0.45 g, 1.06 mmol) in acetonitrile (5 mL), DIPEA (0.7 mL, 4.0 mmol) and chloroacetone (0.2 mL, 2.53 mmol) were added. After 2.5 h at reflux, the solvent was removed *in vacuo* and the residue was partitioned between dichloromethane

and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 2%) to give ketone **18** (0.41 g, 81%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.04 (s, 3H), 2.40 (s, 3H), 3.39 (s, 2H), 4.25 (s, 2H), 7.27-7.47 (m, 5H), 7.52 (tt, *J* = 8 and 1.2 Hz, 1H), 8.04 (dd, *J* = 8.4 and 0.8 Hz, 1H), 8.18 (m, 2H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 27.8 (CH_3), 41.5 (CH_3), 53.1 (CH_2), 65.8 (CH_2), 76.3 (C), 114.7 (CH), 122.3 (CH), 123.9 (CH), 126.0 (CH), 127.4 (2 CH), 128.9 (2 CH), 130.9 (C), 133.7 (CH), 136.5 (C), 136.9 (C), 138.9 (C), 206.9 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{19}H_{20}IN_2O_3S$ 483.0234, found 483.0232.

2-(2-Iodo-1-phenylsulfonyl-1*H*-indol-3-yl)ethyl *p*-toluenesulfonate (20)

To a cooled (0 ºC) solution of iodotryptophol **19**[3] (0.71 g, 1.66 mmol) in CH_2Cl_2 (10 mL), TsCl (0.35 g, 1.83 mmol), DMAP (10 mg) and Et_3N (0.58 mL, 4.15 mmol) were added. The resulting mixture was stirred at room temperature for 24 h. The reaction mixture was diluted with CH_2Cl_2 and washed with 1N HCl and saturated Na_2CO_3 aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to 1:1 hexanes-EtOAc) to give **20** (0.8 g, 75%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.41 (s, 3H), 3.01 (t, *J* = 7.2 Hz, 2H), 4.17 (t, *J* = 7.2 Hz, 2H), 7.19-7.58 (m, 10H), 7.83 (m, 2H), 8.25 (d, *J* = 8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 21.6 (CH_3), 27.8 (CH_2), 68.2 (CH_2), 80.1 (C), 115.6 (CH), 118.5 (CH), 123.8 (CH), 125.1 (CH), 126.7 (C), 127.1 (2 CH), 127.4 (2 CH), 129.2 (2 CH), 129.6 (2 CH), 130.1 (C), 132.3 (C), 134.0 (CH), 138.1 (C), 138.8 (C), 144.8 (C). ESI-HRMS $[M+NH_4]^+$ calcd for $C_{23}H_{24}IN_2O_5S_2$ 599.0166, found 599.0165.

{*N*-[2-(2-Iodo-1-phenylsulfonyl-1*H*-indol-3-yl)ethyl]-*N*-methylamino}-2-propanone (21)

A mixture of **20** (0.7 g, 1.17 mmol), methylamine (3 mL of 8M solution in EtOH, 24 mmol), and K_2CO_3 (0.34 g, 2.46 mmol) in acetonitrile (7 mL) was stirred at 85 ºC in a sealed tube for 24 h. The solvent was removed *in vacuo* and the residue was partitioned between dichloromethane and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was dissolved in acetonitrile (10 mL), and DIPEA (1 mL, 5.7 mmol) and chloroacetone (0.2 mL, 2.46 mmol) were added. After 5

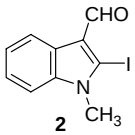
[3] J. T. Kuethe, A. Wong, I. W. Davies and P. J. Reider, *Tetrahedron Lett.* 2002, **43**, 3871.

h at reflux, the solvent was removed *in vacuo* and the residue was partitioned between dichloromethane and saturated $NaHCO_3$ aqueous solution. The organic layer was dried and concentrated. The residue was purified by chromatography (SiO_2, from CH_2Cl_2 to CH_2Cl_2-MeOH 1%) to give ketone **21** (270 mg, 46%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.05 (s, 3H), 2.39 (s, 3H), 2.53 (m, 2H), 2.86 (m, 2H), 3.24 (s, 2H), 7.21-7.32 (m, 2H), 7.36-7.46 (m, 3H), 7.53 (tt, *J* = 7.2 and 1.6 Hz, 1H), 7.85 (m, 2H), 8.30 (dm, *J* = 7.2 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 26.2 (CH_2), 27.5 (CH_3), 42.7 (CH_3), 56.3 (CH_2), 67.5 (CH_2), 79.3 (C), 115.9 (CH), 118.5 (CH), 123.8 (CH), 125.1 (CH), 127.1 (2 CH), 129.0 (2 CH), 130.3 (C), 130.4 (C), 133.9 (CH), 138.0 (C), 139.2 (C), 207.3 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{20}H_{22}IN_2O_3S$ 497.0396, found 497.0391.

2-{*N*-Benzyl-*N*-[(3-iodo-1-methyl-1*H*-indol-2-yl)methyl]amino}-*N*,*N*-dimethylacetamide (22)

A solution of dimethylamine hydrochloride (296 mg, 3.63 mmol) in toluene (6 mL) was cooled to -30 ºC under argon, and a 2M solution of trimethylaluminum in toluene (1.82 mL, 3.63 mmol) was added dropwise. The solution was stirred at room temperature for 1h.

A solution of ester **14** (0.54 g, 1.20 mmol) in dichloromethane (12 mL) was cooled to 0 ºC. The solution of $AlMe_2$-NMe_2 complex prepared above was added dropwise to the ester solution, the temperature being kept below 5 ºC. The mixture was slowly warmed to room temperature. The solution was stirred at 50 ºC for 24 h. The reaction was quenched by slowly adding it to an ice-cold solution containing 10% aqueous KH_2PO_4 (40 mL), THF (30 mL), and EtOAc (30 mL). The mixture was warmed to 40 ºC and stirred for 1h. The organic layer was then separated, washed with brine, dried, filtered through Celite, and concentrated. The residue was purified by chromatography (SiO_2, from hexanes to hexanes-EtOAc 1:1) to give amide **22** (266 mg, 48%). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.74 (s, 3H), 2.90 (s, 3H), 3.29 (s, 2H), 3.77 (s, 3H), 3.79 (s, 2H), 3.98 (s, 2H), 7.11-7.42 (m, 9H). ^{13}C NMR ($CDCl_3$, 100.5 MHz) δ 30.6 (CH_3), 35.4 (CH_3), 36.6 (CH_3), 50.2 (CH_2), 54.5 (CH_2), 58.7 (CH_2), 61.7 (C), 109.4 (CH), 120.2 (CH), 121.1 (CH), 122.7 (CH), 127.2 (CH), 128.2 (2 CH), 129.4 (2 CH), 129.6 (C), 136.7 (C), 138.0 (C), 138.7 (C), 170.2 (C). ESI-HRMS $[M+H]^+$ calcd for $C_{21}H_{25}IN_3O$ 462.1037, found 462.1036.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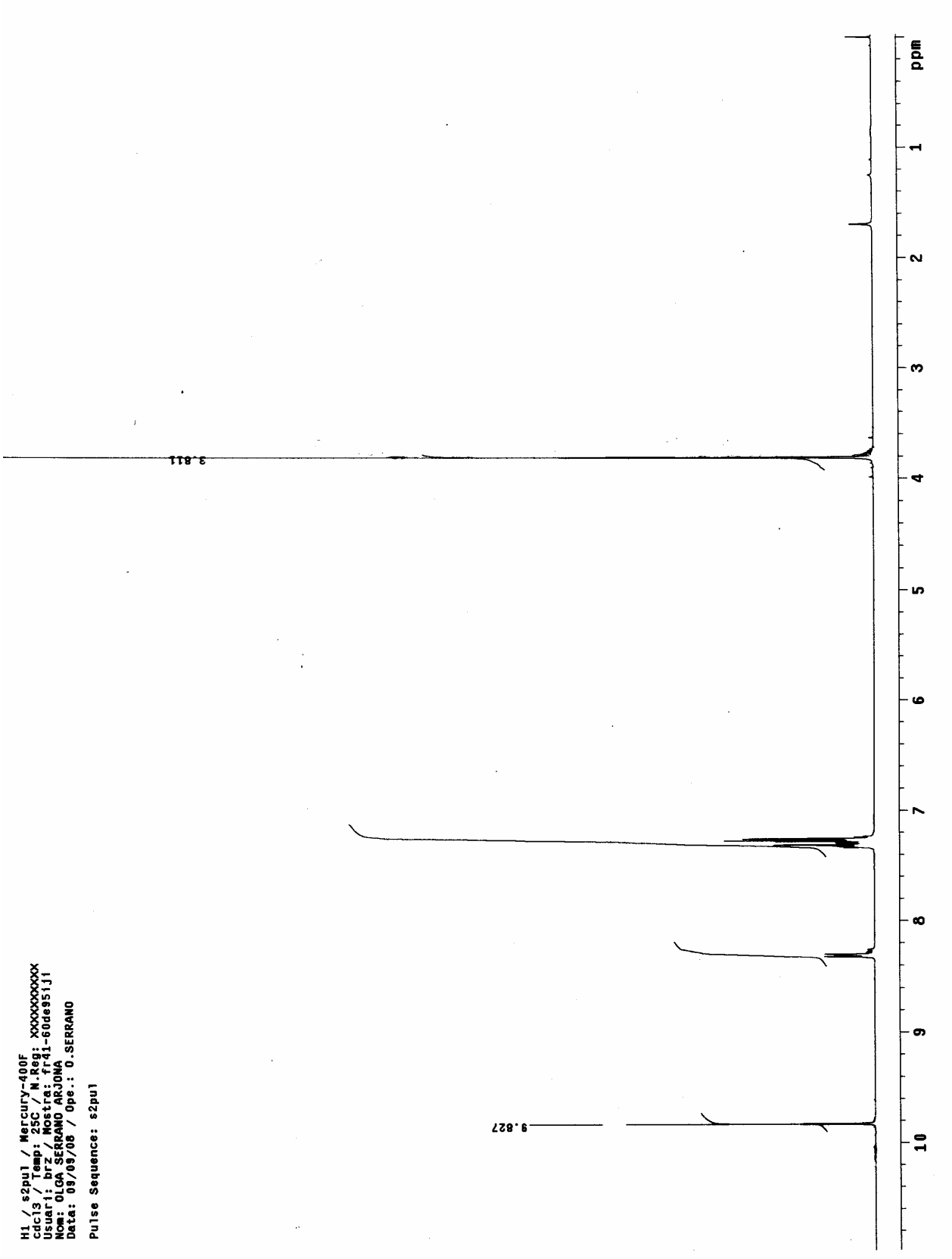
N
CH3

2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ppm
1
2
3
4
5
6
7
8
9
10
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
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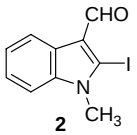
Nom: OLGA SERRANO ARJONA
Data: 09/05/08 / Ope.: O.SERRANO
Pulse Sequence: s2pul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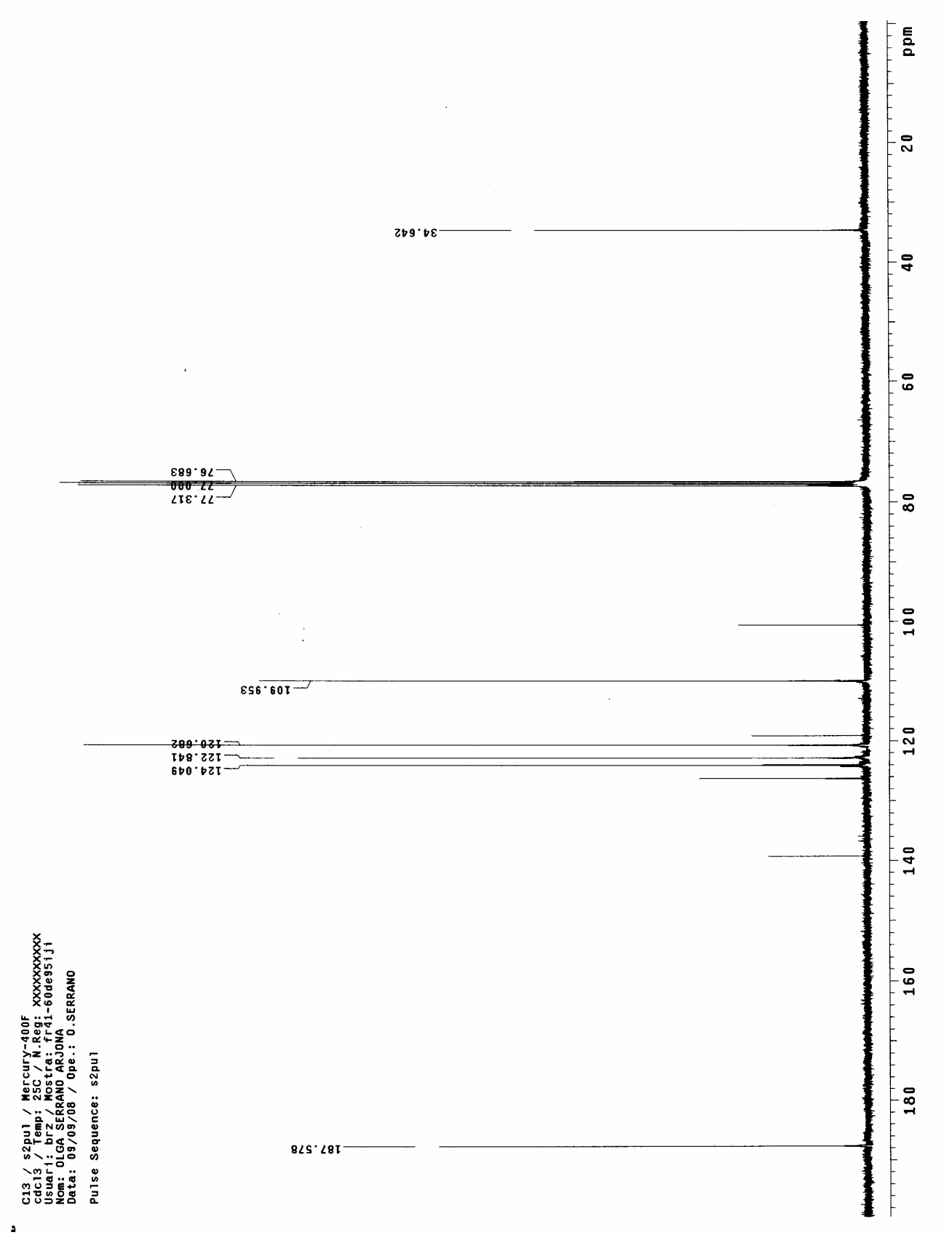
I
N
CH3

2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77.317
109.953
120.882
122.841
124.049
187.578
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
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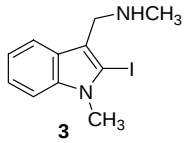
Data: 09/09/08 / Ope.: O.SERRANO
Pulse Sequence: s2pul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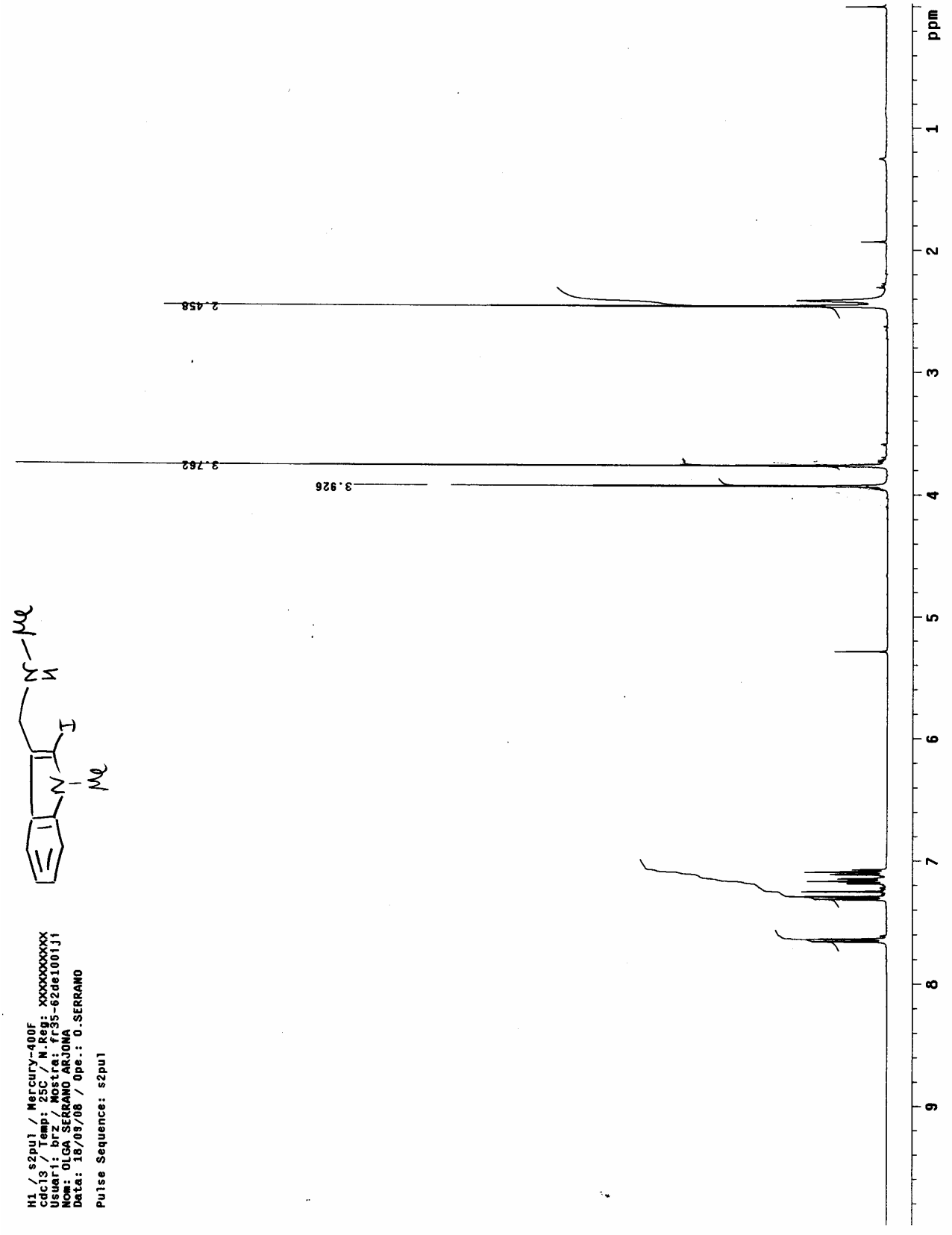
I
N
CH3

3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3.762
3.926
ppm
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2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
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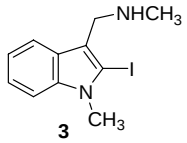
Data: 18/09/08 / Ope.: O.SERRANO
Pulse Sequence: s2pul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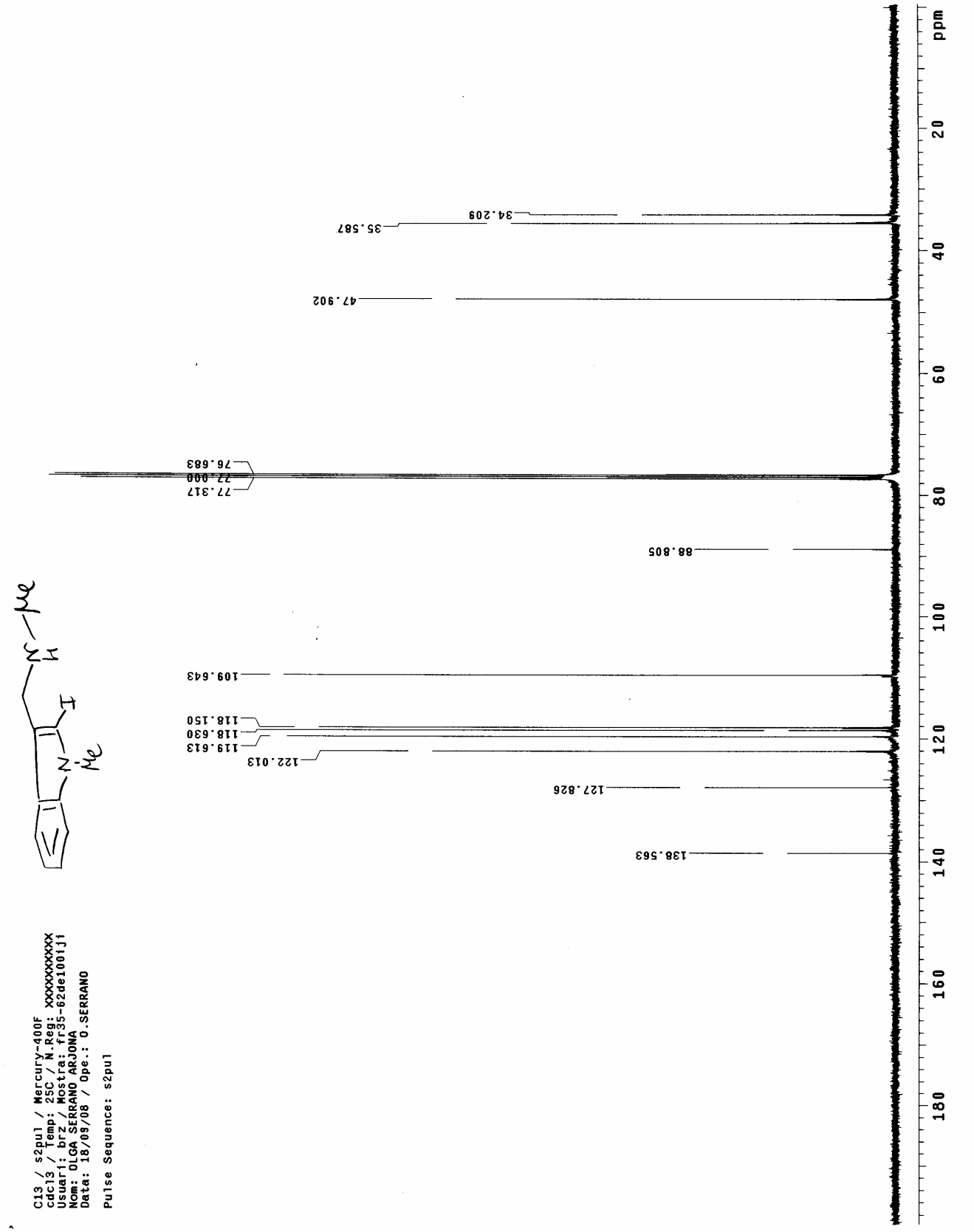
I
N
CH3

3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35.587
47.902
76.683
77.000
77.317
88.805
109.643
118.150
118.630
119.613
122.013
127.826
138.563
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
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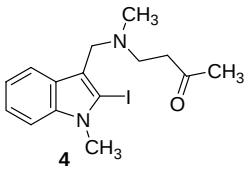
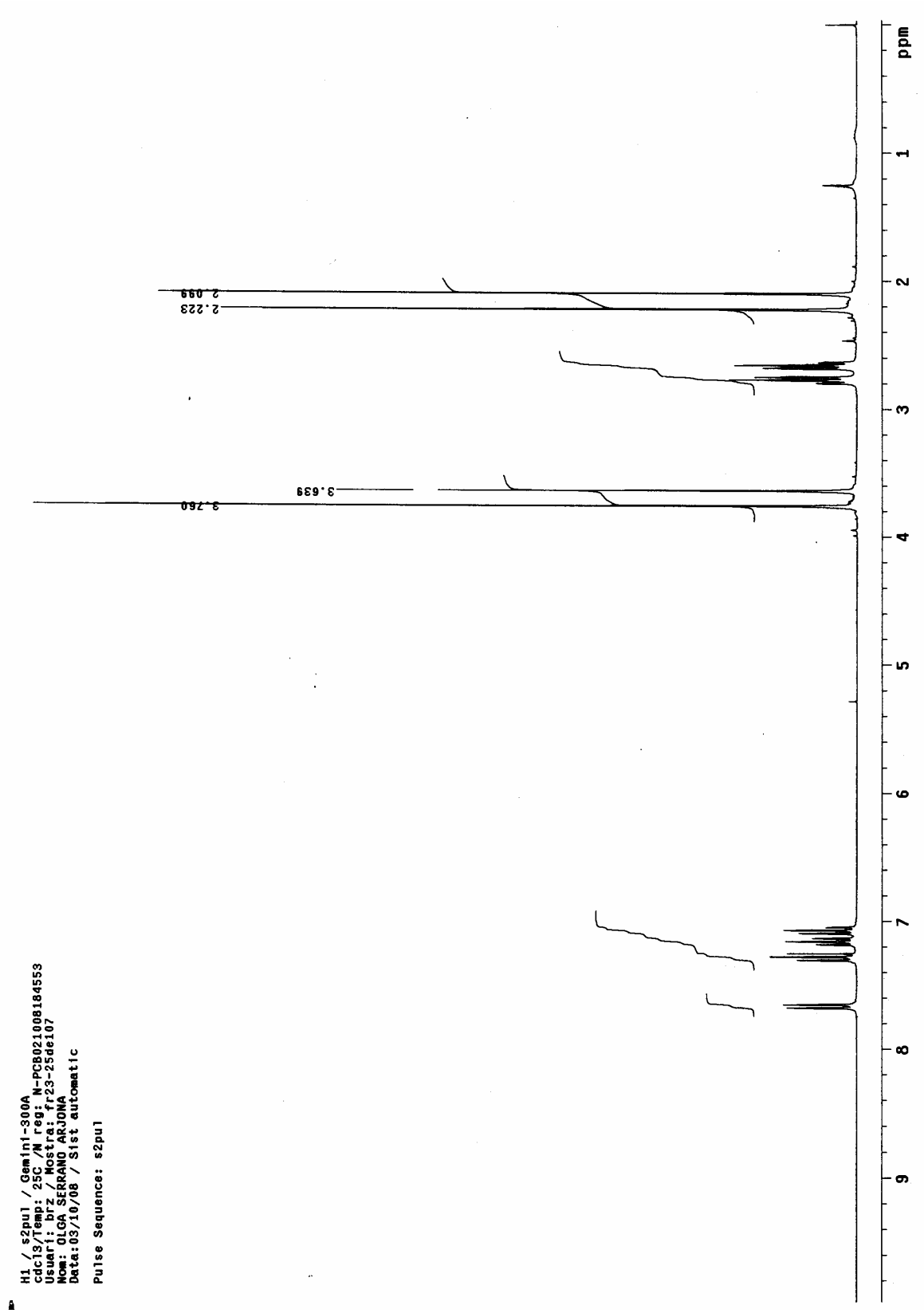
Data: 18/09/08 / Ope.: O.SERRANO
Pulse Sequence: s2pul


CH3
N
CH3
I
O
N
CH3

4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3.639
3.760
ppm
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2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB021008184553
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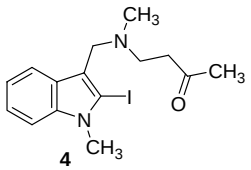
Nom: OLGA SERRANO ARJONA
Data:03/10/08 / Sist automatic
Pulse Sequence: s2pul



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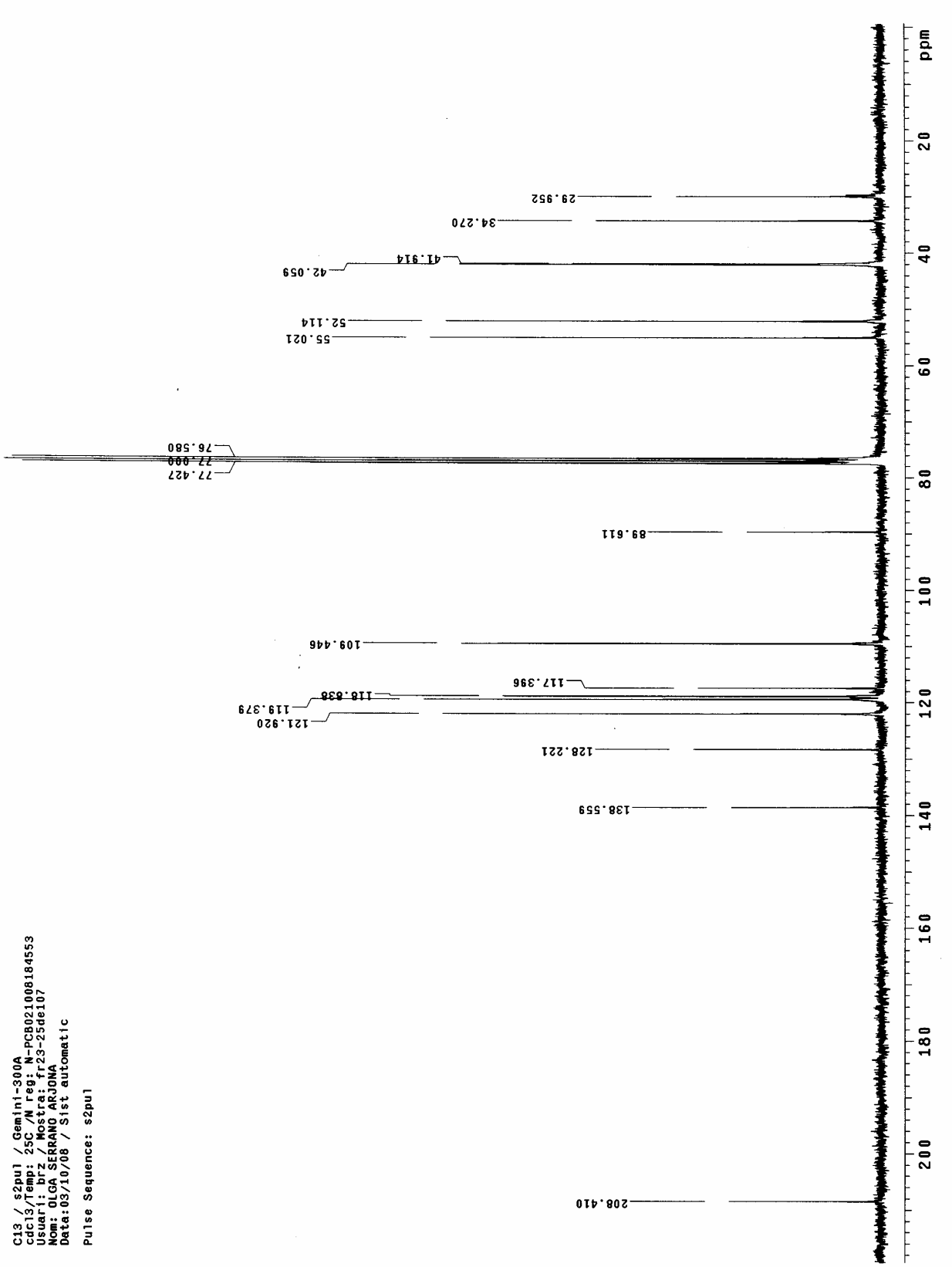
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Usuari: brz / Mostra: fr23-25de107
Nom: OLGA SERRANO ARJONA
Data:03/10/08 / Sist automatic
Pulse Sequence: s2pul
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138.559
128.221
121.920
119.379
118.838
117.396
109.446
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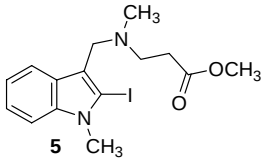
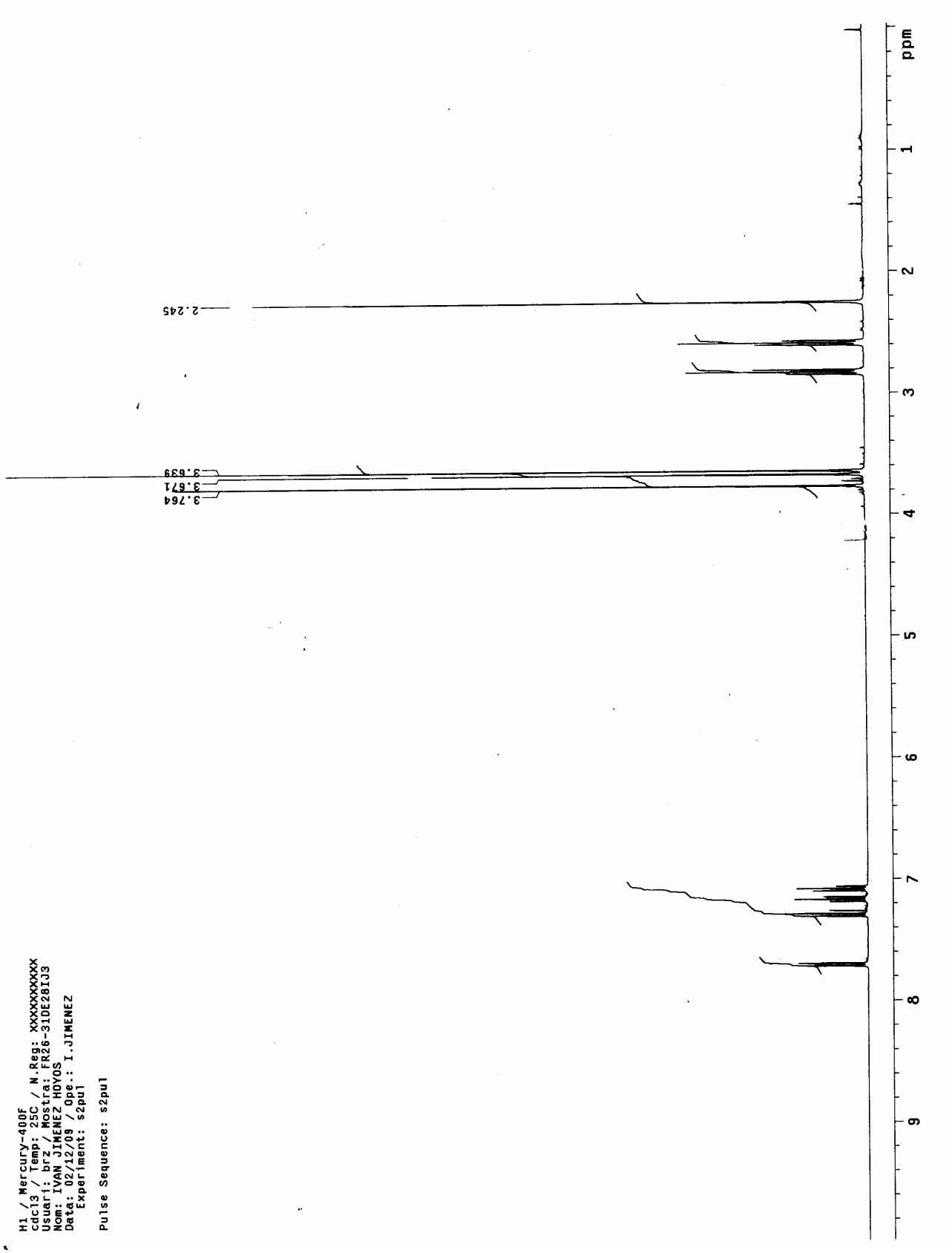
77.427
77.000
76.580
55.021
52.114
42.059
41.914
34.270
29.952
ppm


CH3
N
OCH3
I
O
N
CH3

5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3.639
3.671
3.764
ppm
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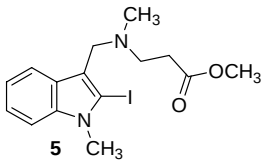
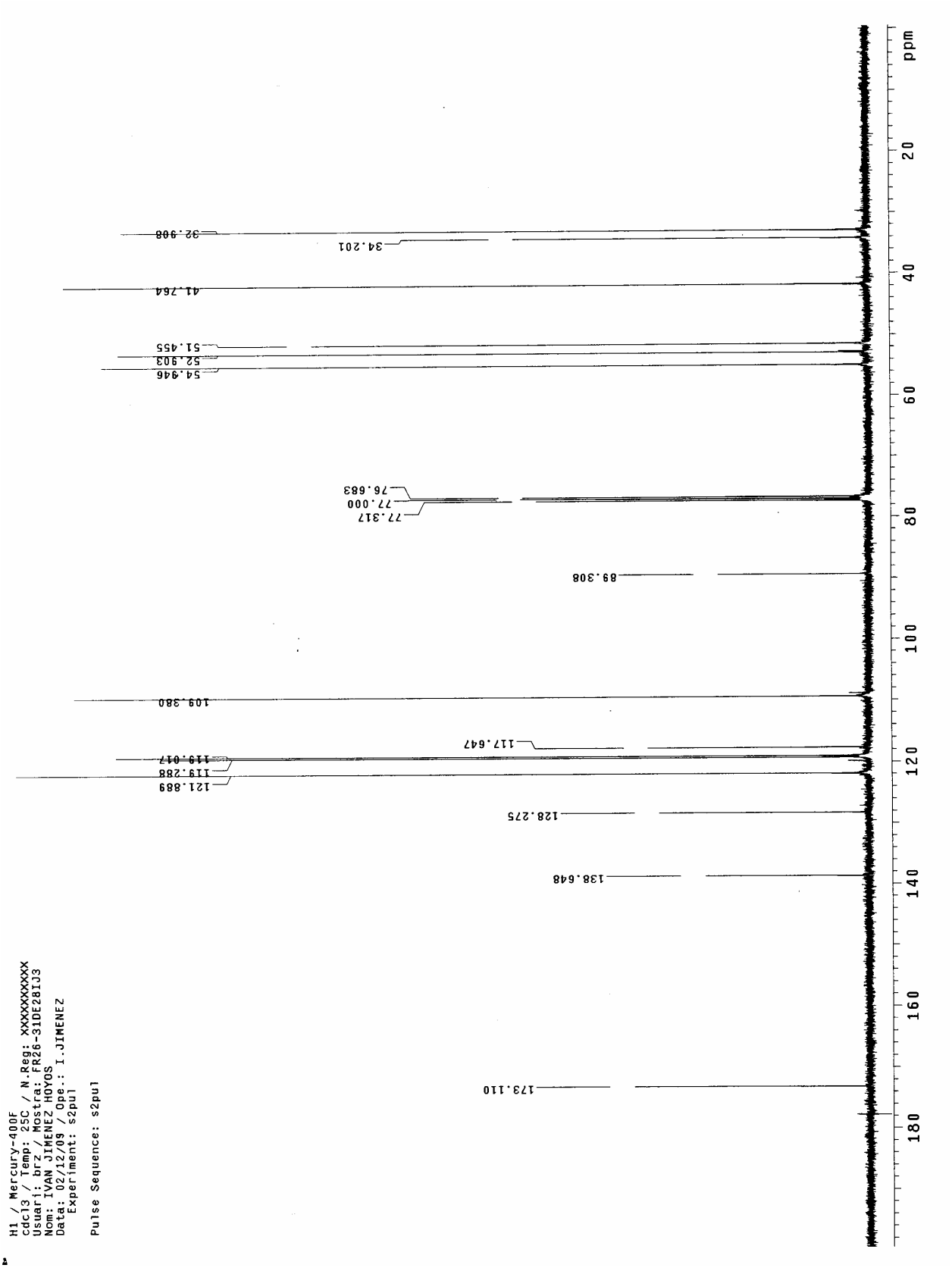
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: FR26-31DE28IJ3
Nom: IVAN JIMENEZ HOYOS
Data: 02/12/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul


CH3
N
OCH3
I
O
N
CH3

5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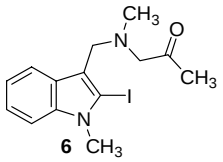
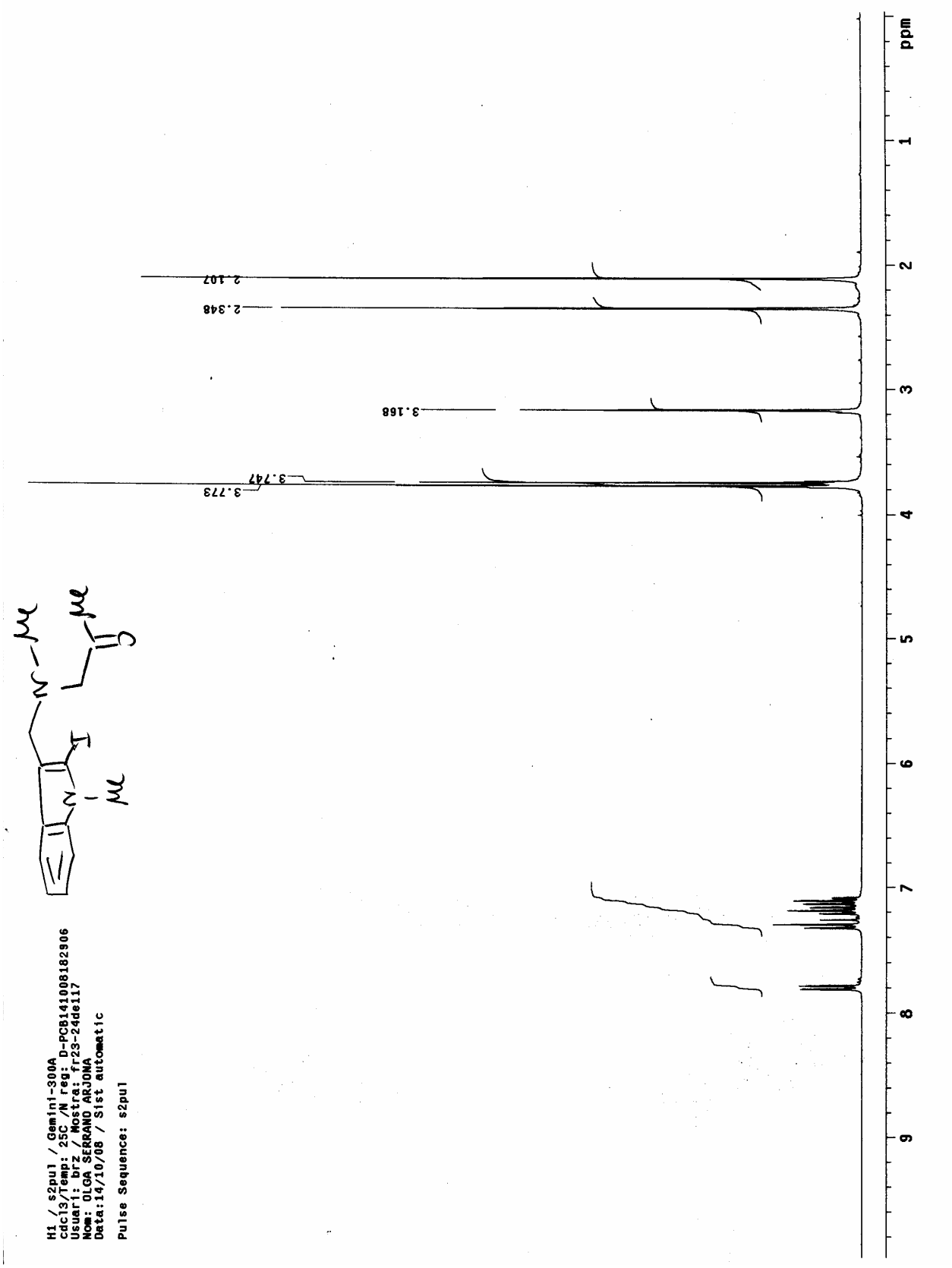
51.455
52.903
54.946
76.683
77.000
77.317
89.308
109.380
117.647
119.017
119.288
121.889
128.275
138.648
173.110
ppm
20
40
60
80
100
120
140
160
180
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: FR26-31DE28IJ3
Nom: IVAN JIMENEZ HOYOS
Data: 02/12/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul


CH3
N
O
CH3
I
N
CH3

6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3.747
3.773
ppm
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2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: D-PCB141008182906
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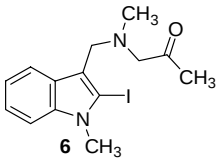
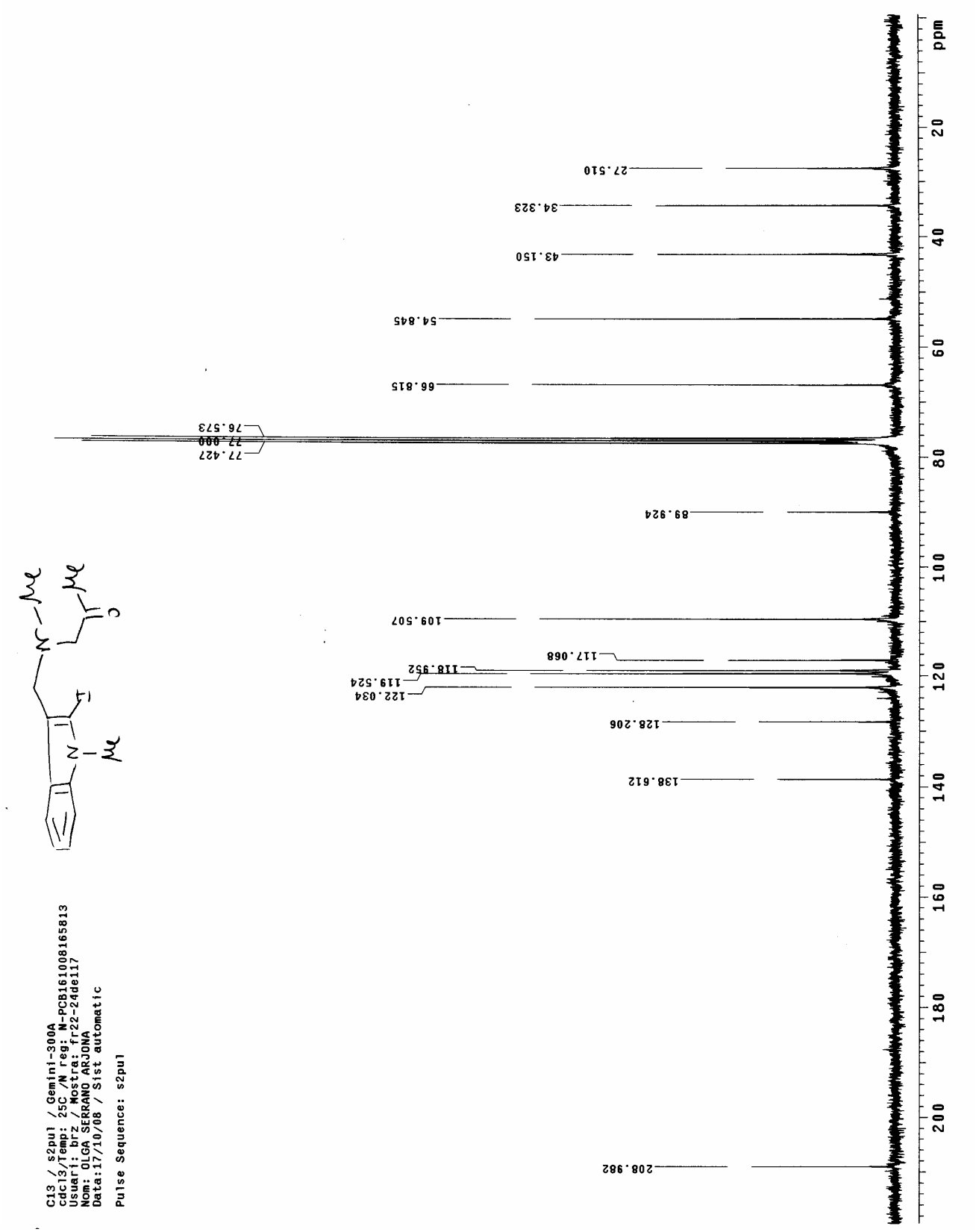
Nom: OLGA SERRAMO ARJONA
Data:14/10/08 / Sist automatic
Pulse Sequence: s2pul


CH3
N
O
CH3
I
N
CH3

6


ppm
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40
60
80
100
120
140
160
180
200
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43.150
54.845
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77.427
89.924
109.507
117.068
118.952
119.524
122.034
128.206
138.612
208.962
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB161008165813
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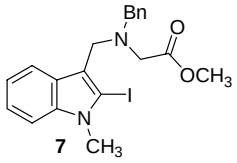
Nom: OLGA SERRANO ARJONA
Data:17/10/08 / Sist automatic
Pulse Sequence: s2pul



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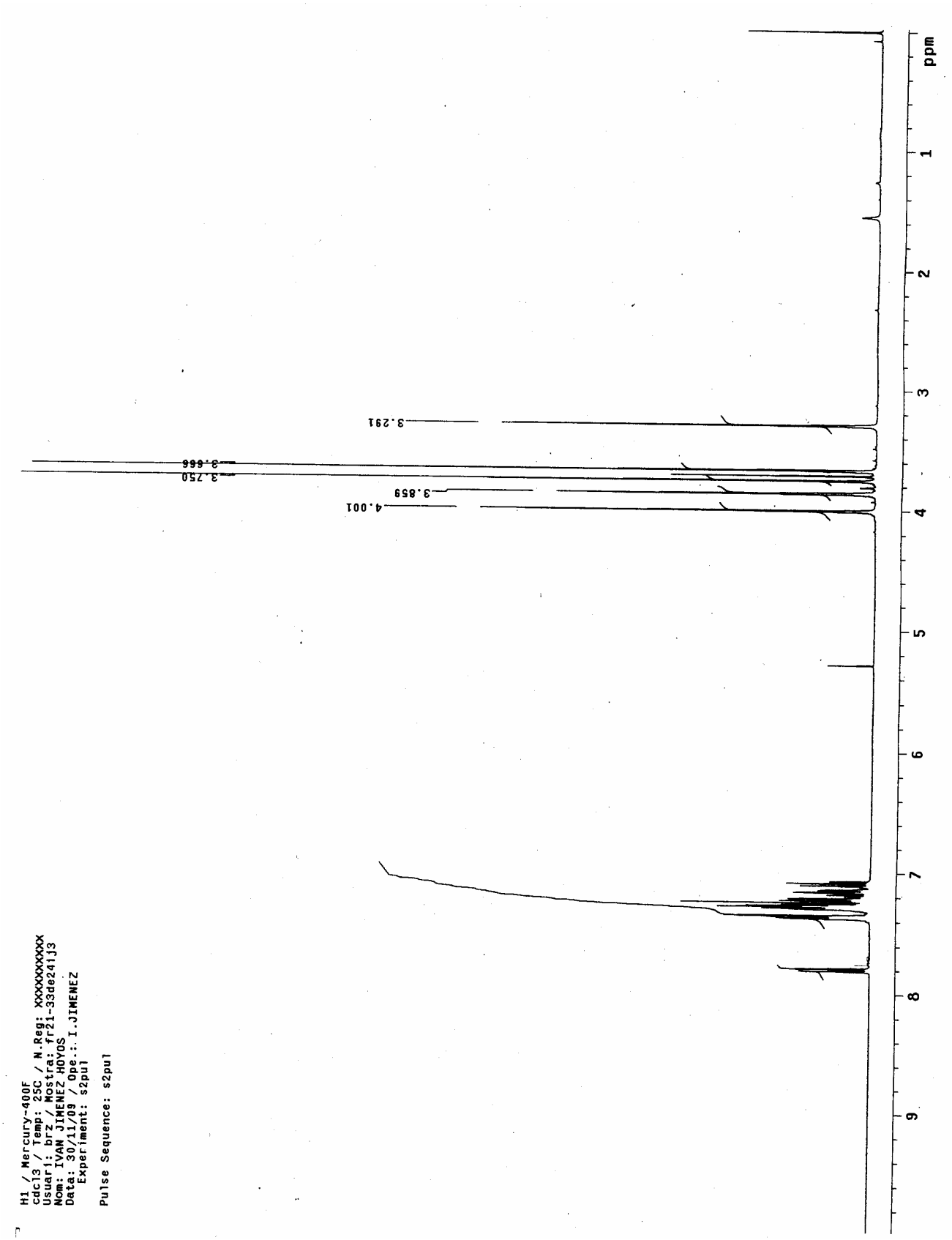
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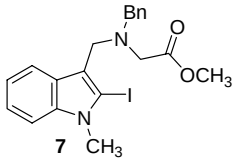
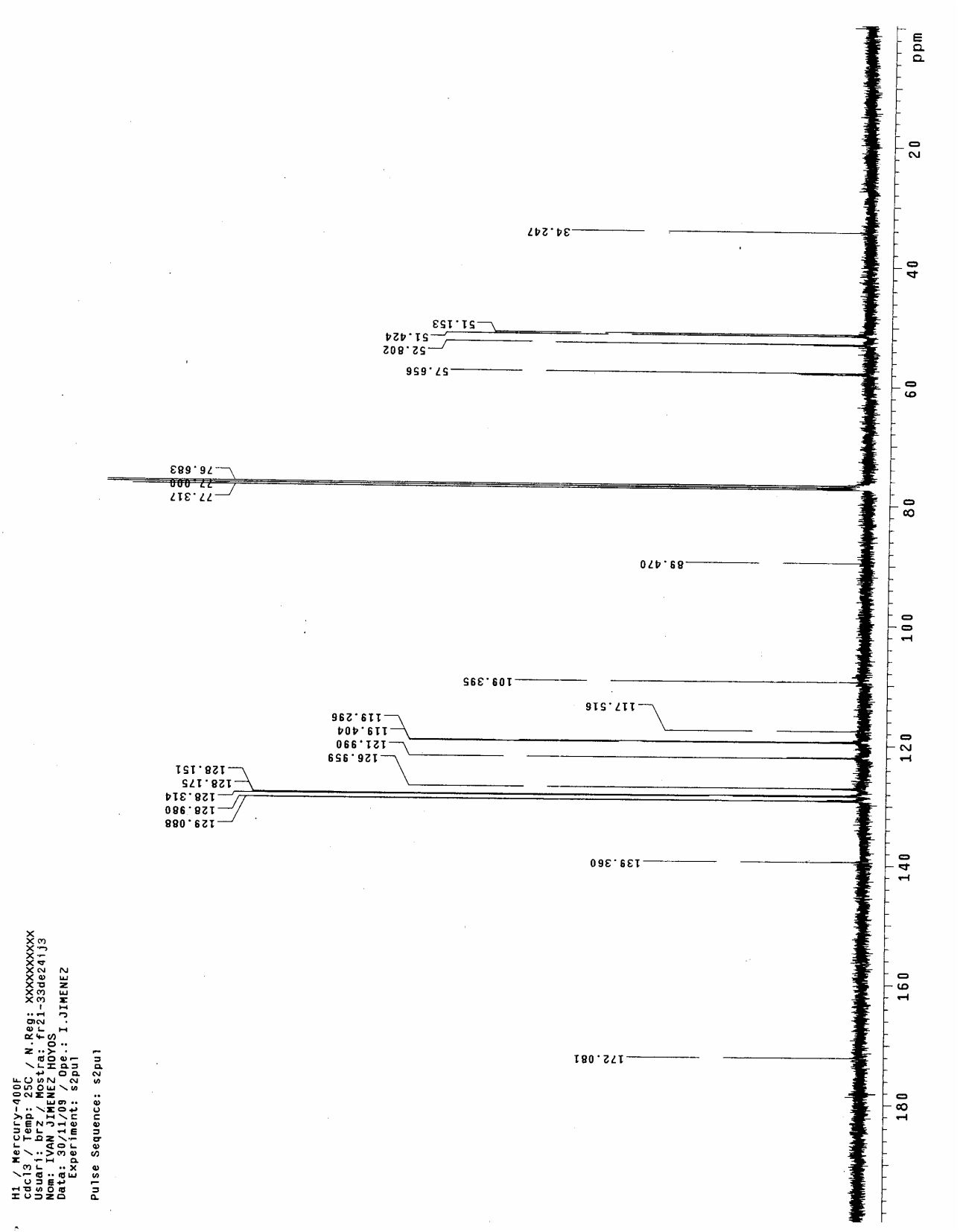
3.859
4.001
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr21-33de24ij3
Nom: IVAN JIMENEZ HOYOS
Data: 30/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul


Bn
N
O
OCH3
I
N
CH3

7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109.395
117.516
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119.404
121.990
126.959
128.151
128.175
128.314
128.980
129.088
139.360
172.081
ppm
20
40
60
80
100
120
140
160
180
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
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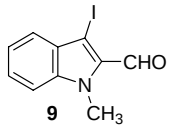
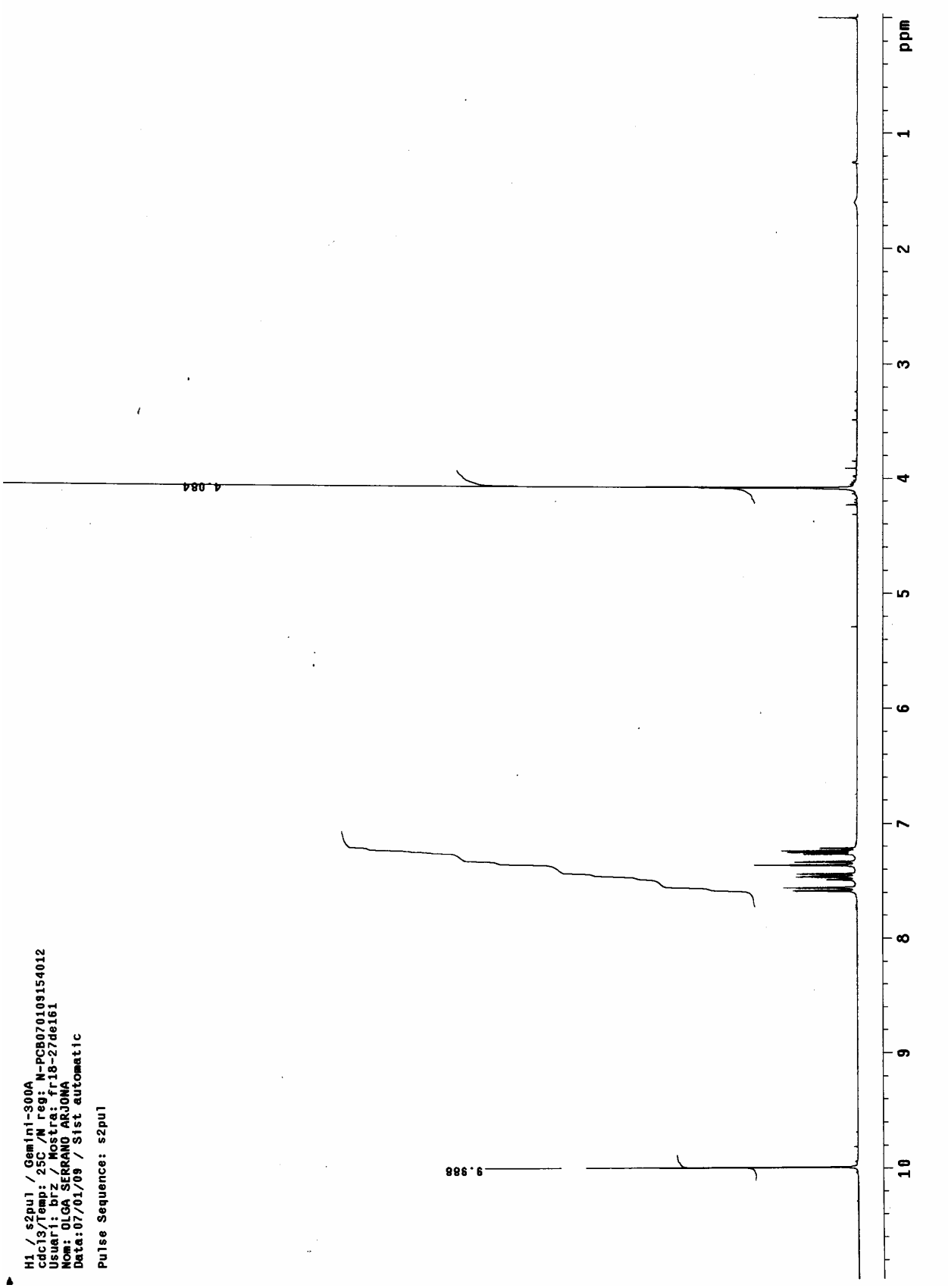
Data: 30/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul


I
CHO
N
CH3

9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9.986
ppm
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4
5
6
7
8
9
10
H1 / s2pul / Gemini-300A
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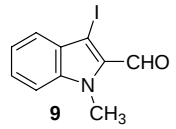
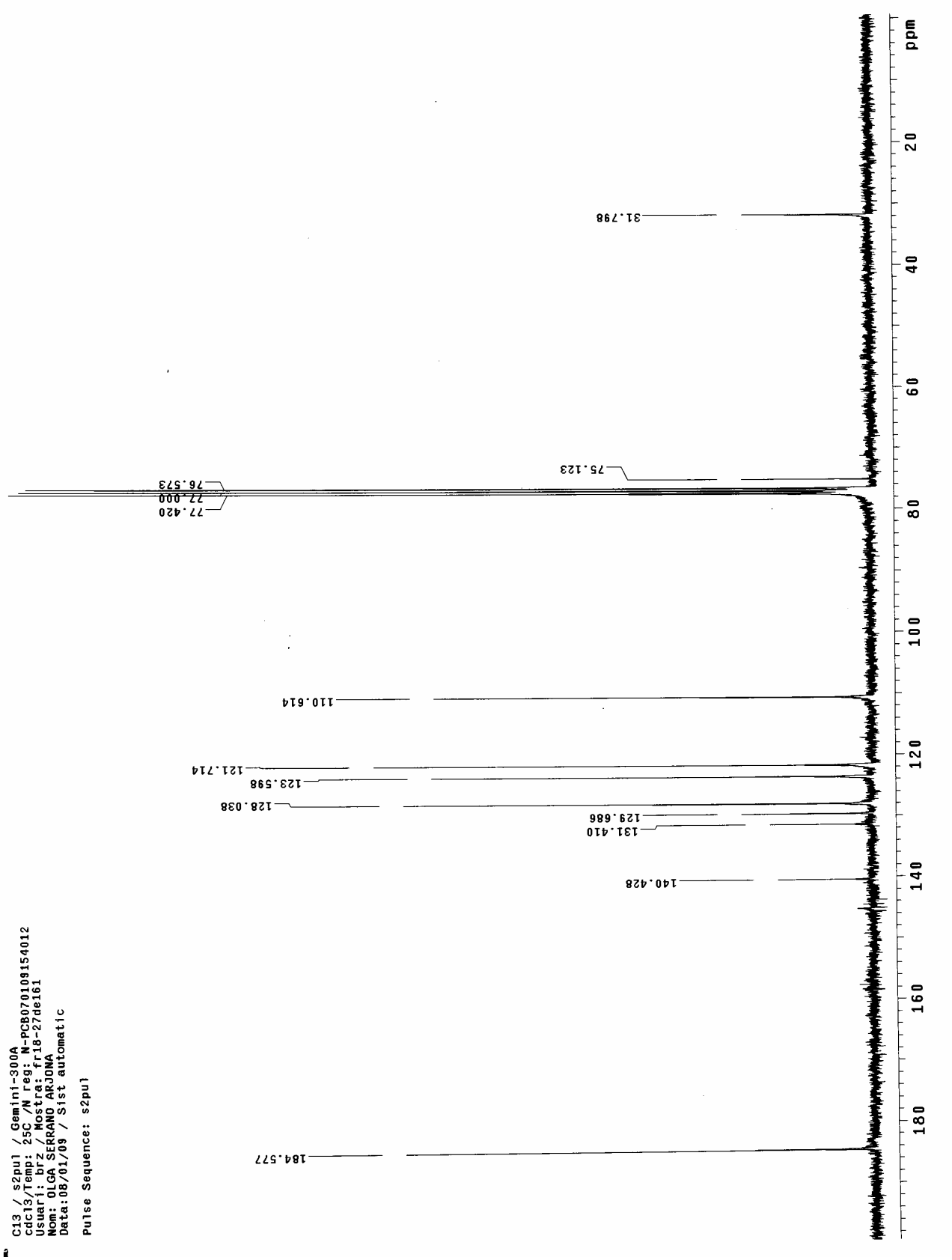
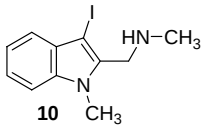
NOM: OLGA SERRANO ARJONA
Data:07/01/09 / Sist automatic
Pulse Sequence: s2pul

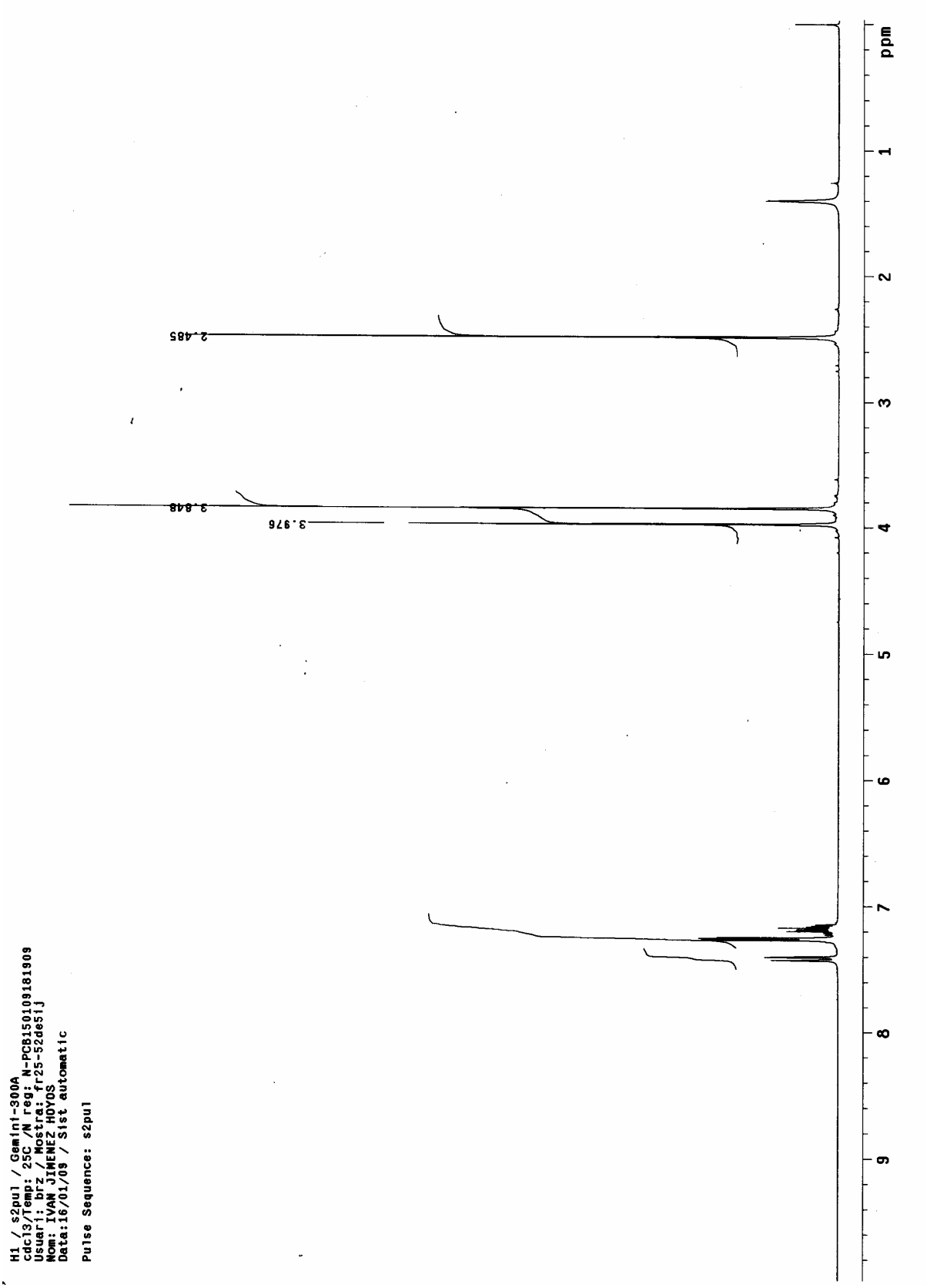

I
CHO
N
CH3

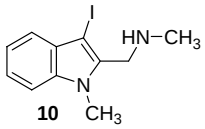
9


ppm
20
40
60
80
100
120
140
160
180
31.798
75.123
77.420
77.000
76.573
110.614
121.714
123.598
128.038
129.686
131.410
140.428
184.577
C13 / s2pul / Gemini-300A
Cdcl3/Temp: 25C /N reg: N-PCB070109154012
Usuari: brz / Mostra: fr18-27de161
Nom: OLGA SERRANO ARJONA
Data:08/01/09 / Sist automatic
Pulse Sequence: s2pul

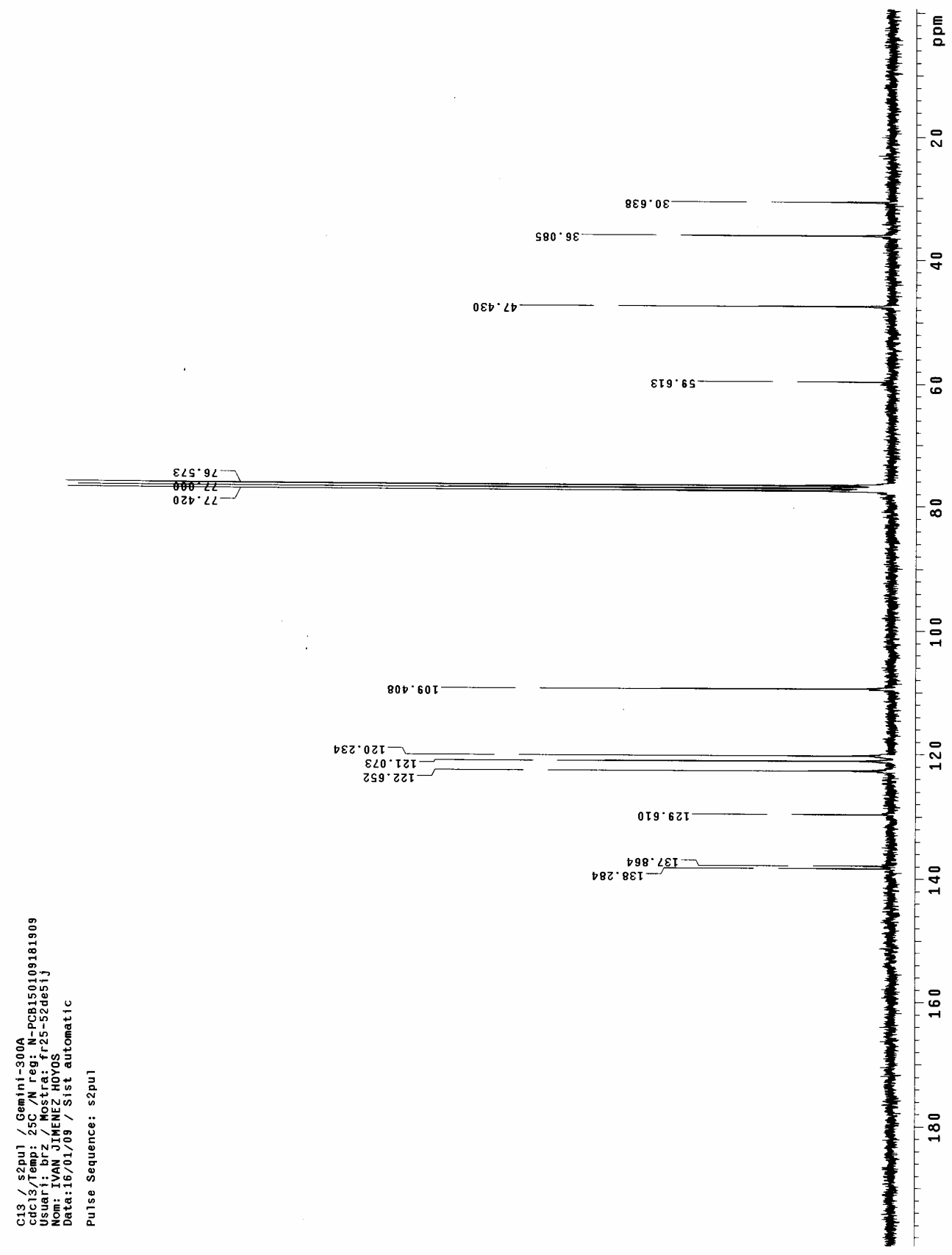

I
HN−CH3
N
CH3

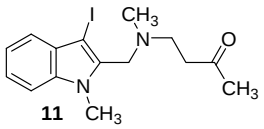
10


2.485
3.848
3.976
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB15010918190 9
Usuari: brz / Mostra: fr25-52de5ij
Nom: IVAN JIMENEZ HOYOS
Data:16/01/09 / Sist automatic
Pulse Sequence: s2pul

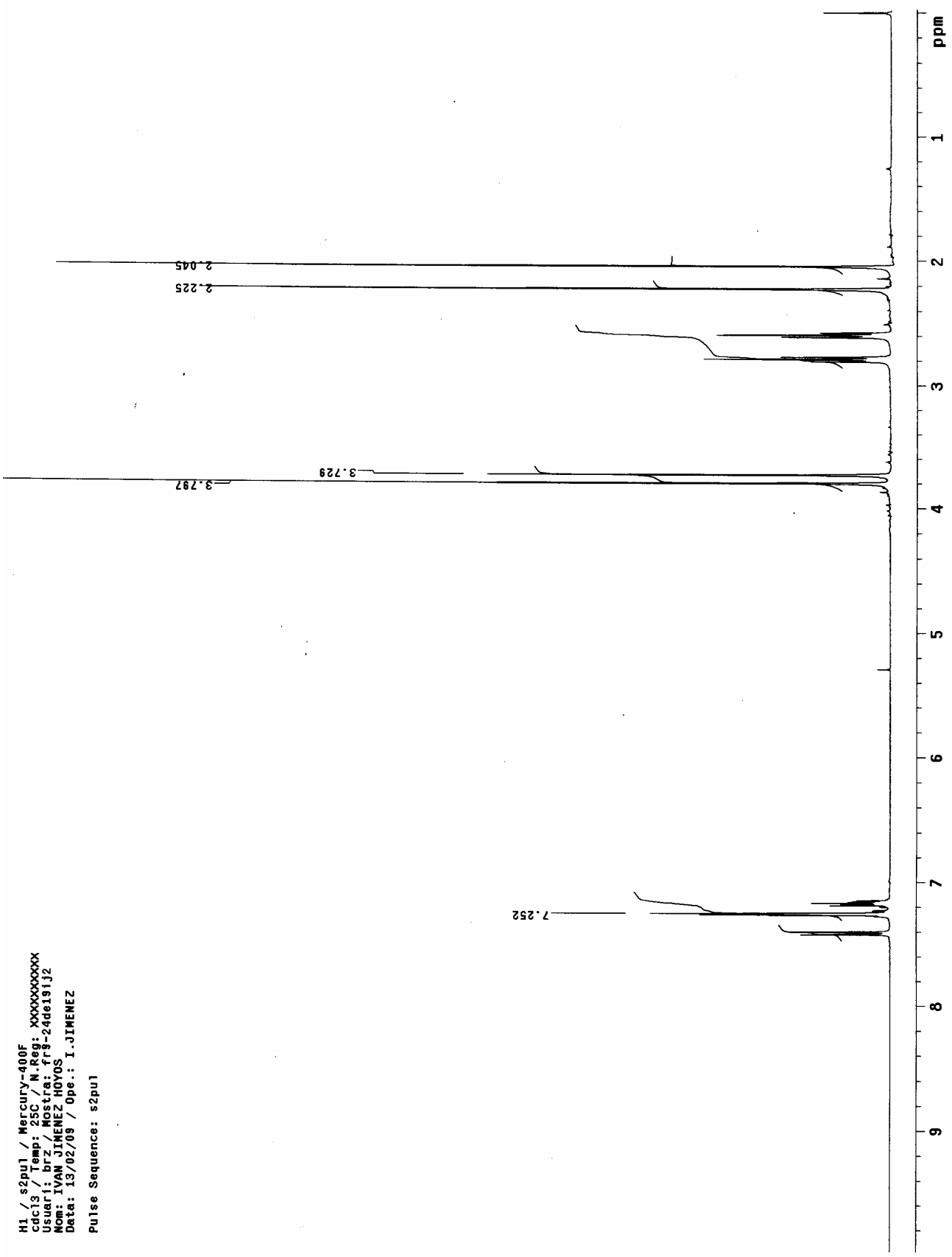

I
HN−CH3
N
CH3

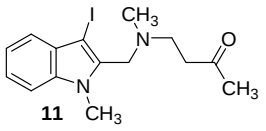
10


30.638
36.085
47.430
59.613
76.573
77.000
77.420
109.408
120.234
121.073
122.652
129.610
137.864
138.284
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB150109181909
Usuari: brz / Mostra: fr25-52de5ij
Nom: IVAN JIMENEZ HOYOS
Data:16/01/09 / Sist automatic
Pulse Sequence: s2pul

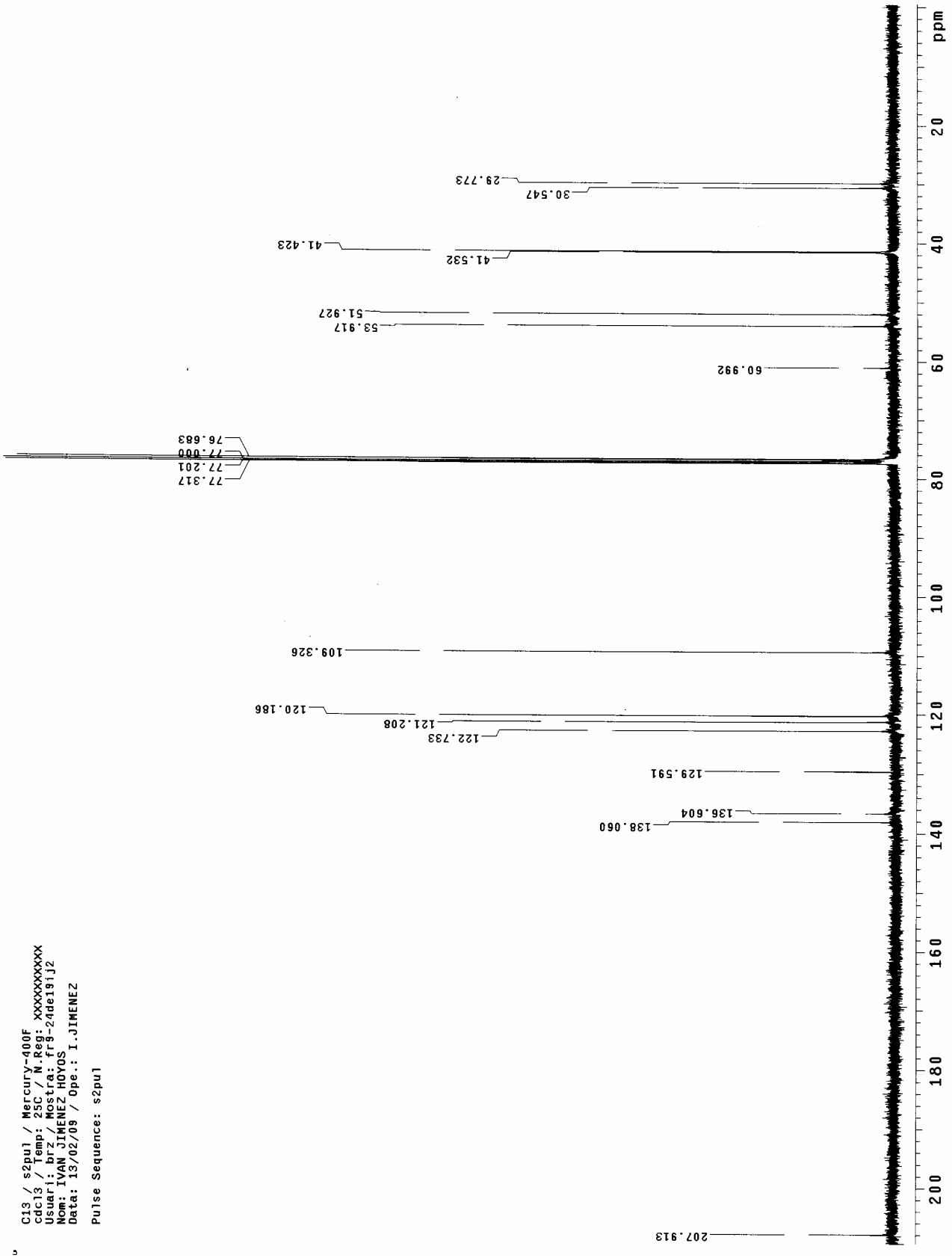


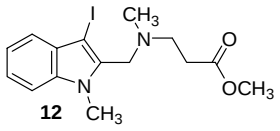
11


2.045
2.225
3.729
3.797
7.252
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr9-24de191j2
Nom: IVAN JIMENEZ HOYOS
Data: 13/02/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

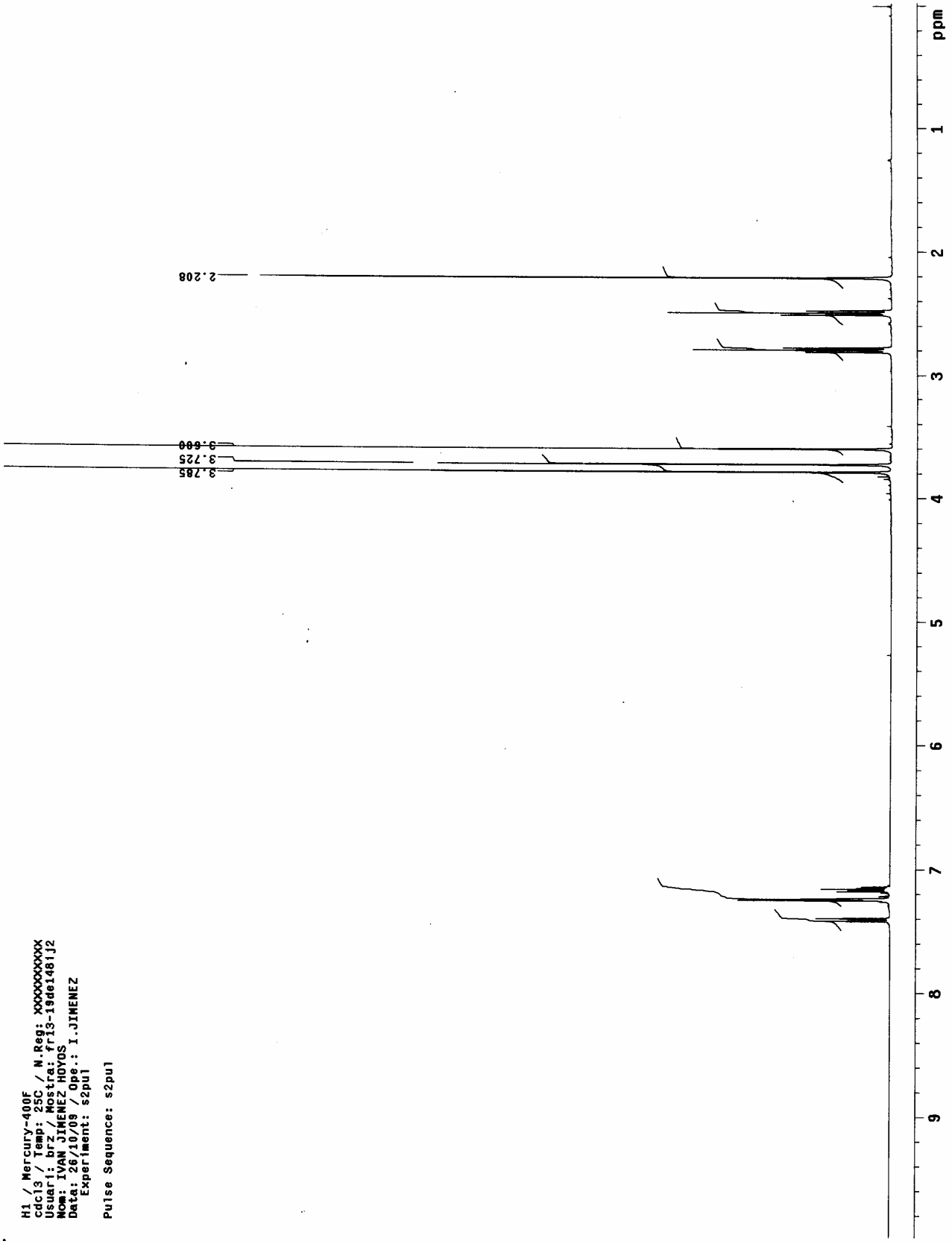

I
CH3
N
O
CH3
N
CH3

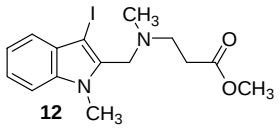
11


ppm
20
40
60
80
100
120
140
160
180
200
29.773
30.547
41.423
41.532
51.927
53.917
60.992
76.683
77.000
77.201
77.317
109.326
120.186
121.208
122.733
129.591
136.604
138.060
207.913
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr9-24de19ij2
Nom: IVAN JIMENEZ HOYOS
Data: 13/02/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

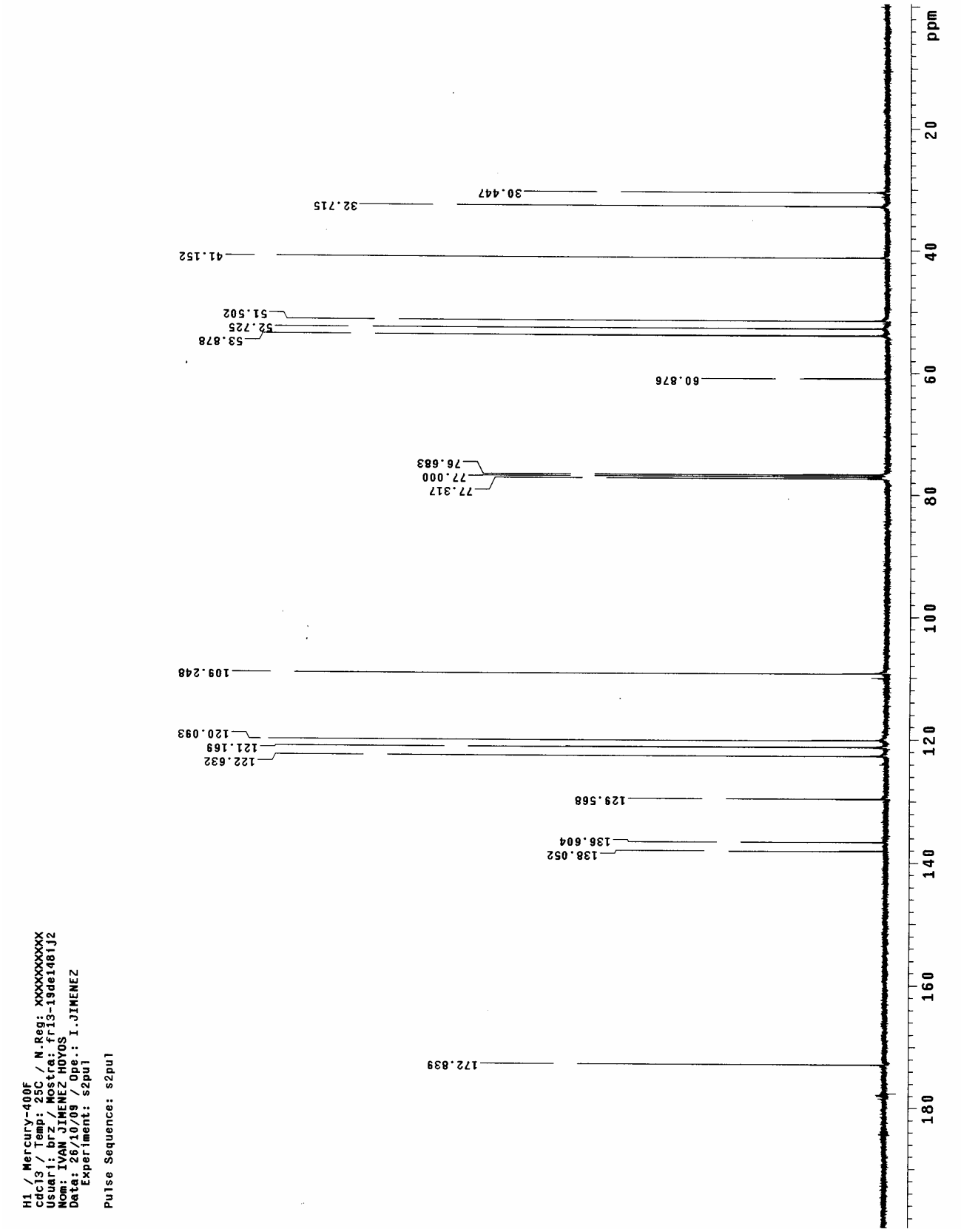

I
CH3
N
O
N
OCH3
CH3

12


2.208
3.600
3.725
3.785
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fri3-19de148ij2
Nom: IVAN JIMENEZ HOYOS
Data: 26/10/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

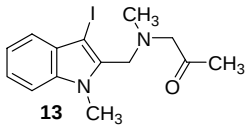


12

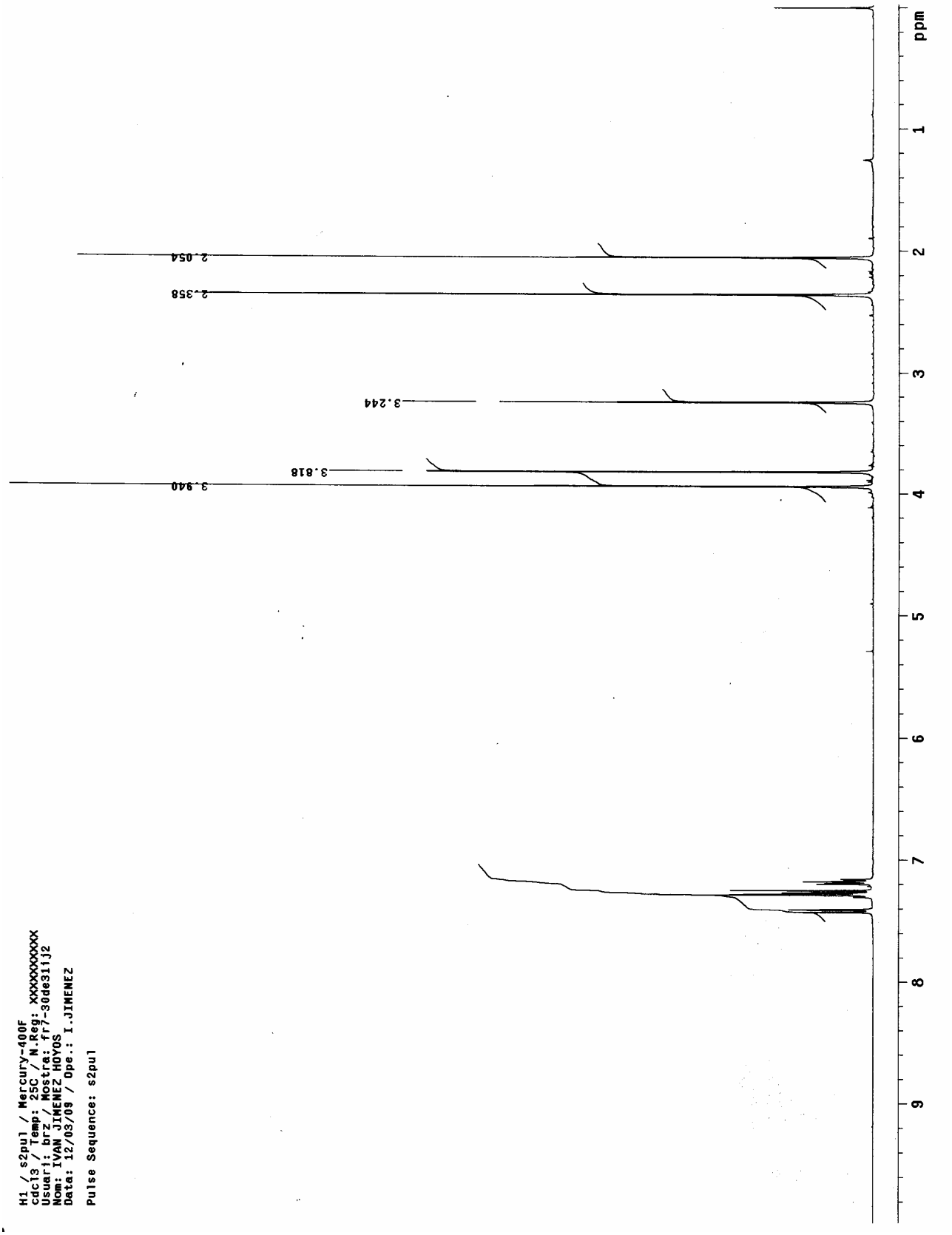


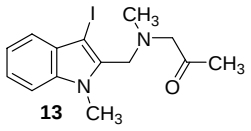
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fri3-19de148ij2
Nom: IVAN JIMENEZ HOYOS
Data: 26/10/09 / Ope.: I.JIMENEZ
Experiment: s2pul

Pulse Sequence: s2pul

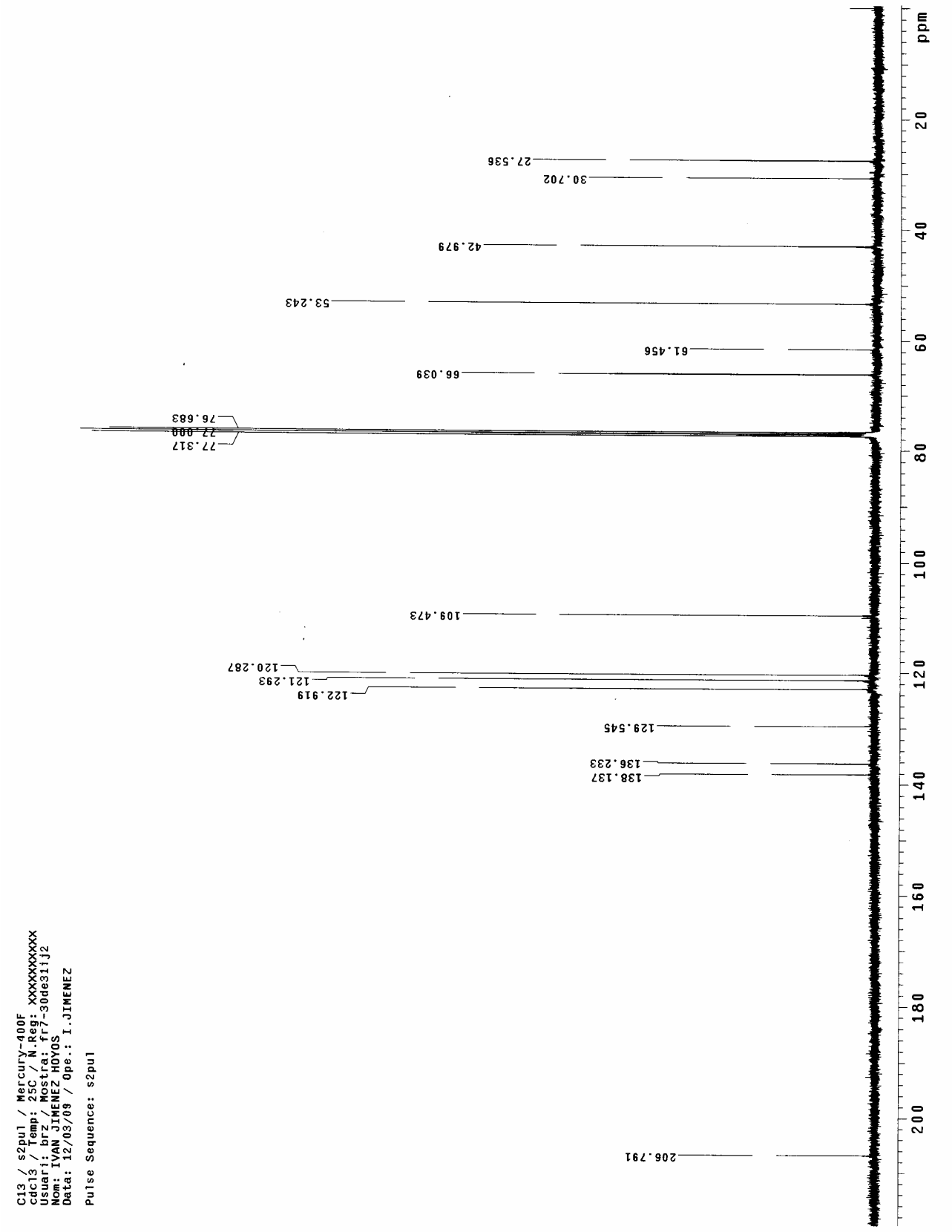

I
CH3
N
CH3
O
N
CH3

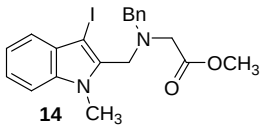
13


2.054
2.358
3.244
3.818
3.940
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr7-30de311j2
Nom: IVAN JIMENEZ HOYOS
Data: 12/03/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

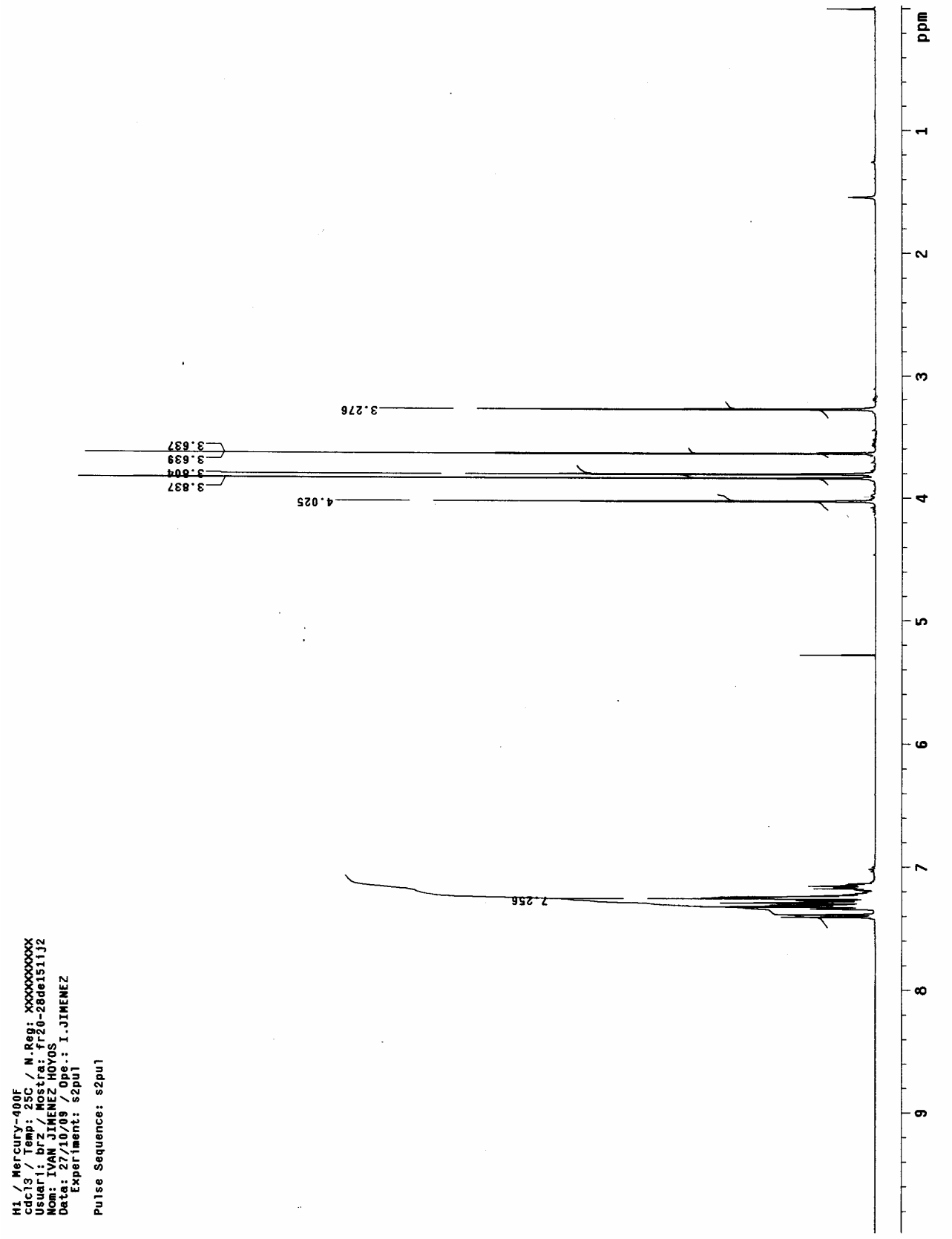

I
CH3
N
CH3
O
N
CH3

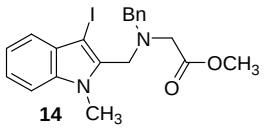
13


ppm
20
40
60
80
100
120
140
160
180
200
27.536
30.702
42.979
53.243
61.456
66.039
76.683
77.000
77.317
109.473
120.287
121.293
122.919
129.545
136.233
138.137
206.791
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr7-30de31ij2
Nom: IVAN JIMENEZ HOYOS
Data: 12/03/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

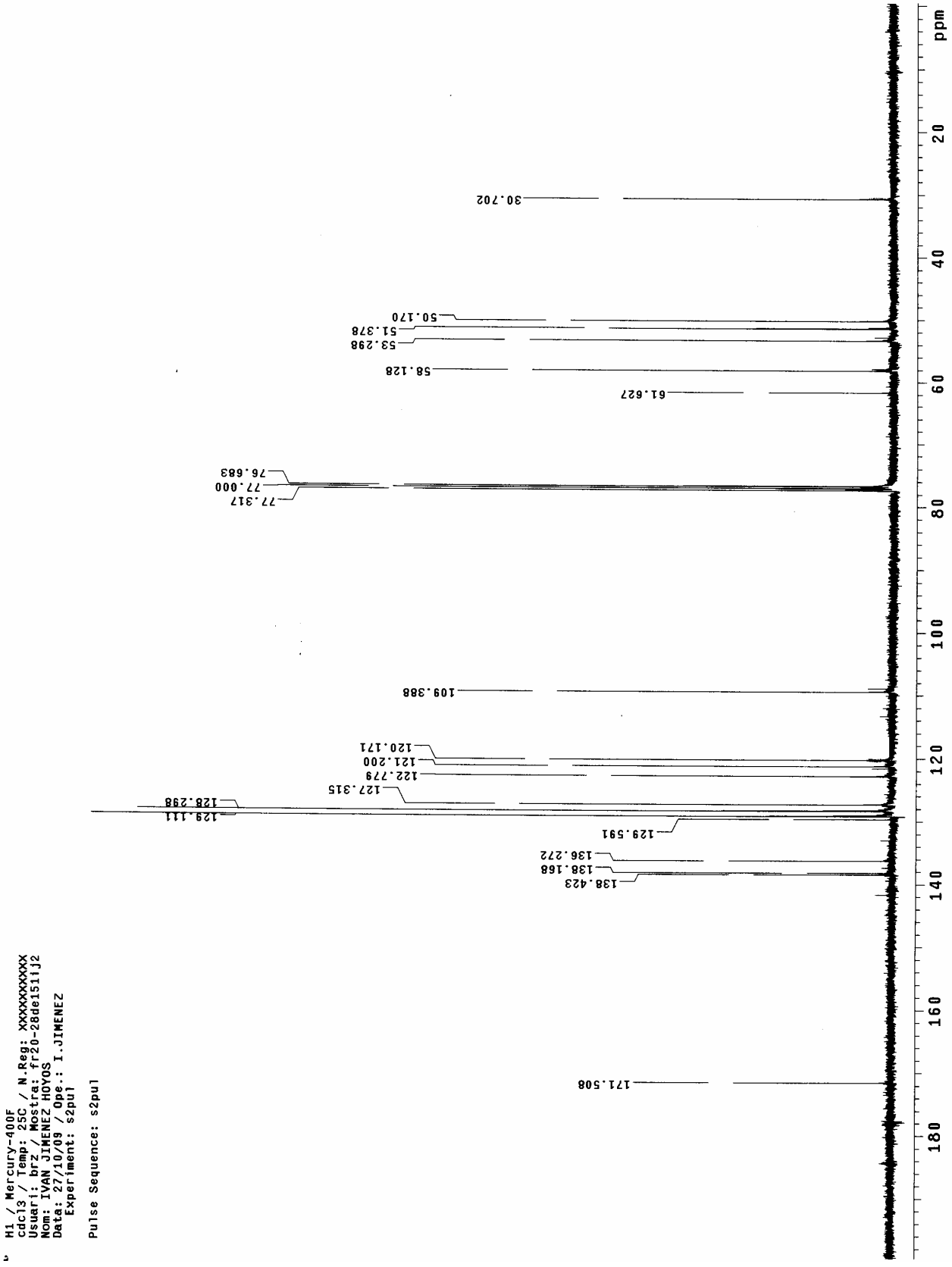

I
Bn
N
OCH3
O
N
CH3

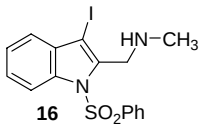
14


3.276
3.637
3.639
3.804
3.837
4.025
7.256
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr20-28d6151ij2
Nom: IVAN JIMENEZ HOYOS
Data: 27/10/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

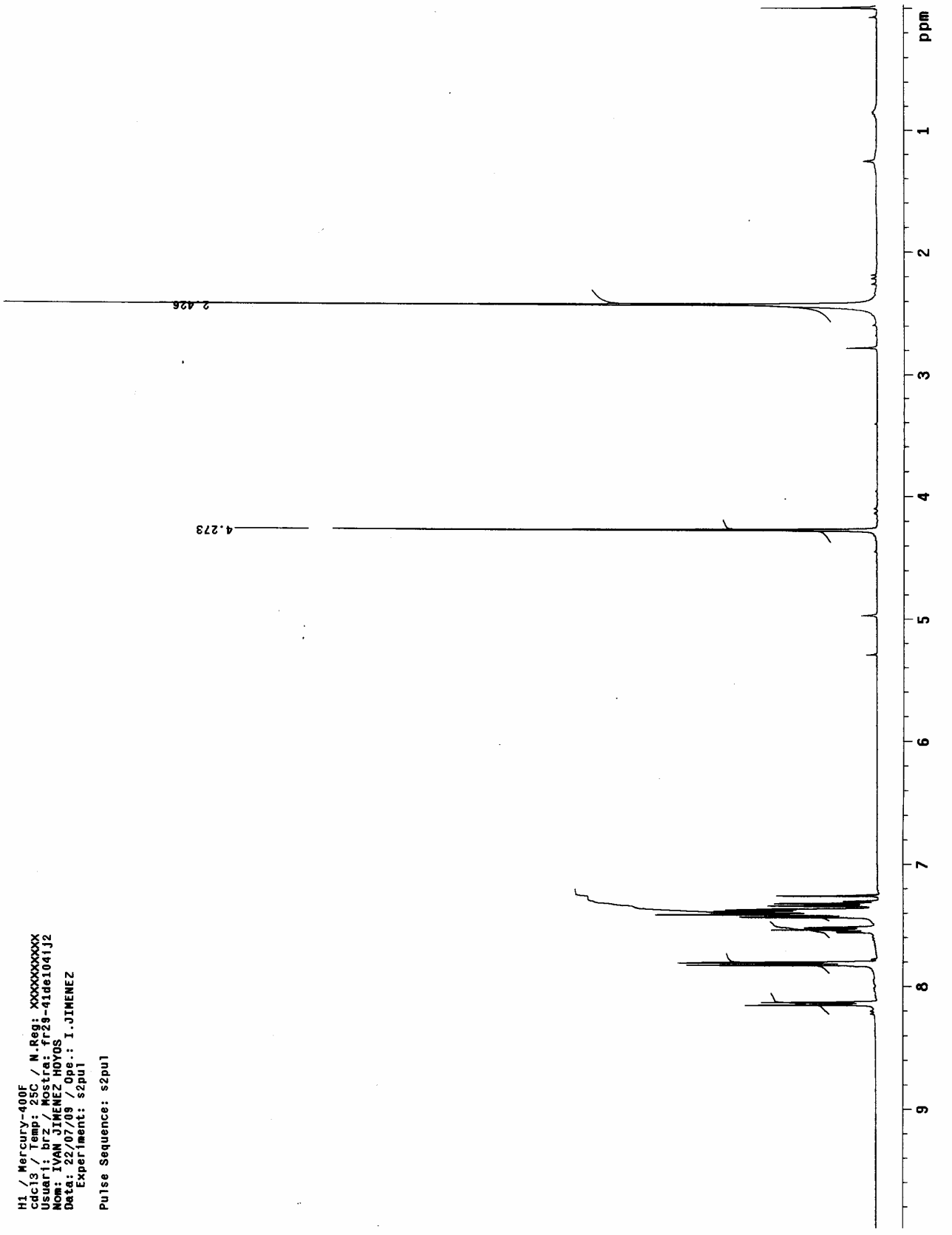

I
Bn
N
OCH3
O
N
CH3

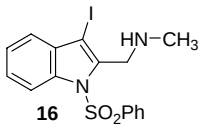
14


ppm
20
40
60
80
100
120
140
160
180
30.702
50.170
51.378
53.298
58.128
61.627
76.683
77.000
77.317
109.388
120.171
121.200
122.779
127.315
128.298
129.111
129.591
136.272
138.168
138.423
171.508
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: br2 / Mostra: fr20-28de151ij2
Nom: IVAN JIMENEZ HOYOS
Data: 27/10/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

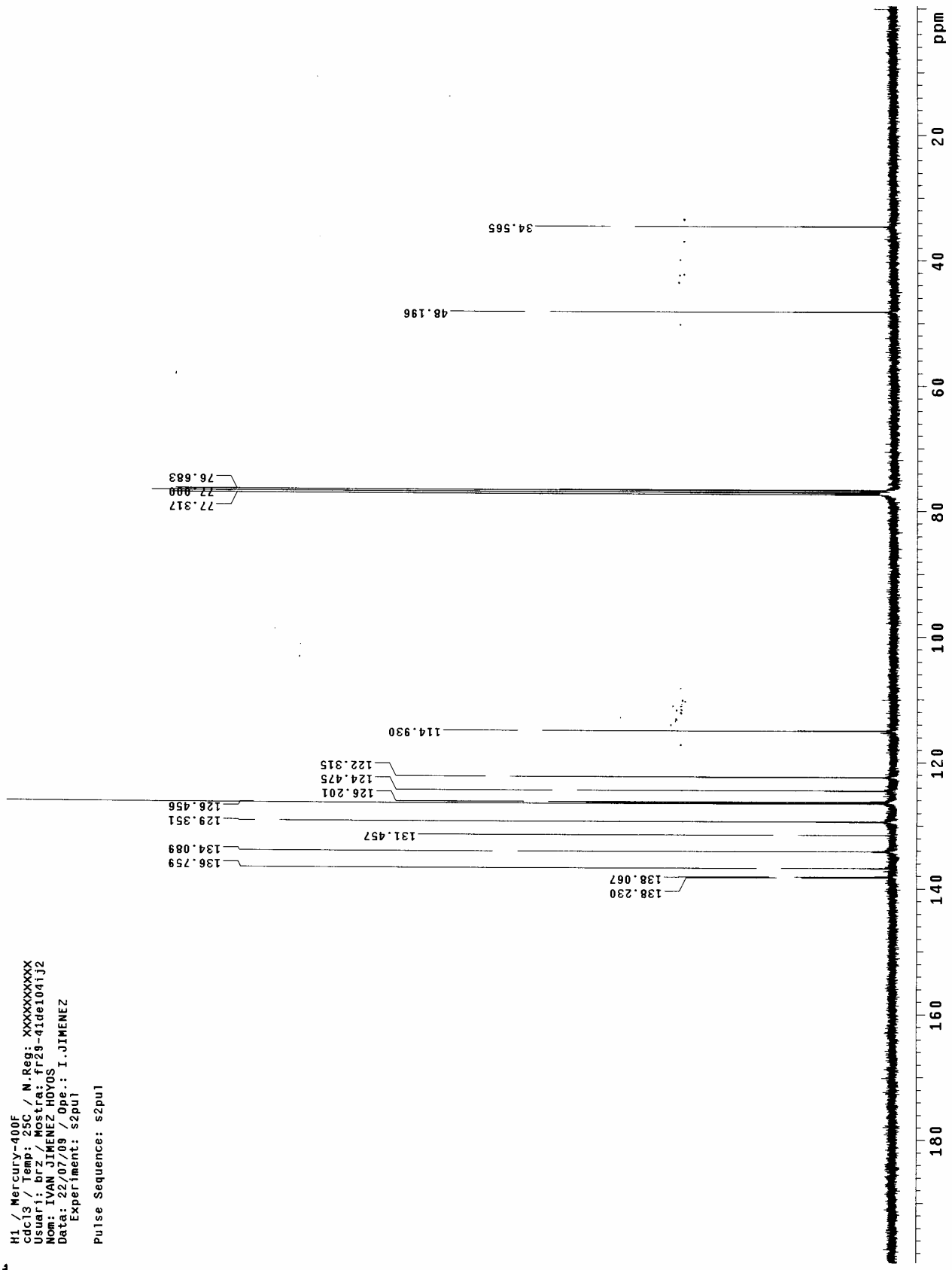

I
HN−CH3
N
SO2Ph

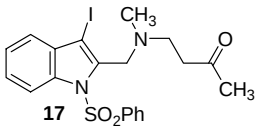
16


2.426
4.273
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr29-41de1041j2
Nom: IVAN JIMENEZ HOYOS
Data: 22/07/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

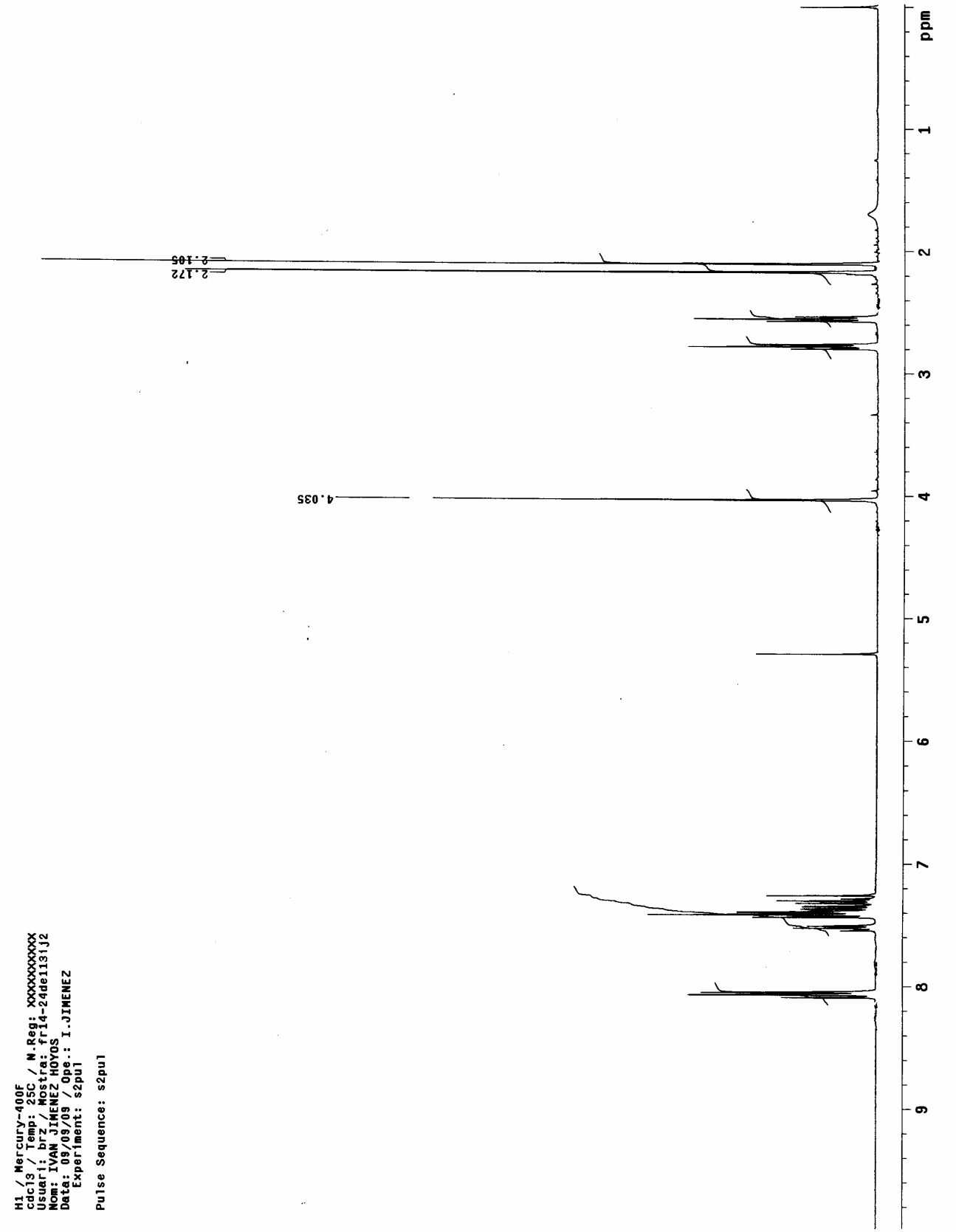

I
HN−CH3
N
SO2Ph

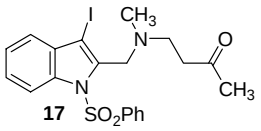
16


34.565
48.196
76.683
77.000
77.317
114.930
122.315
124.475
126.201
126.456
129.351
131.457
134.089
136.759
138.067
138.230
ppm
20
40
60
80
100
120
140
160
180
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr29-41de1041j2
Nom: IVAN JIMENEZ HOYOS
Data: 22/07/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

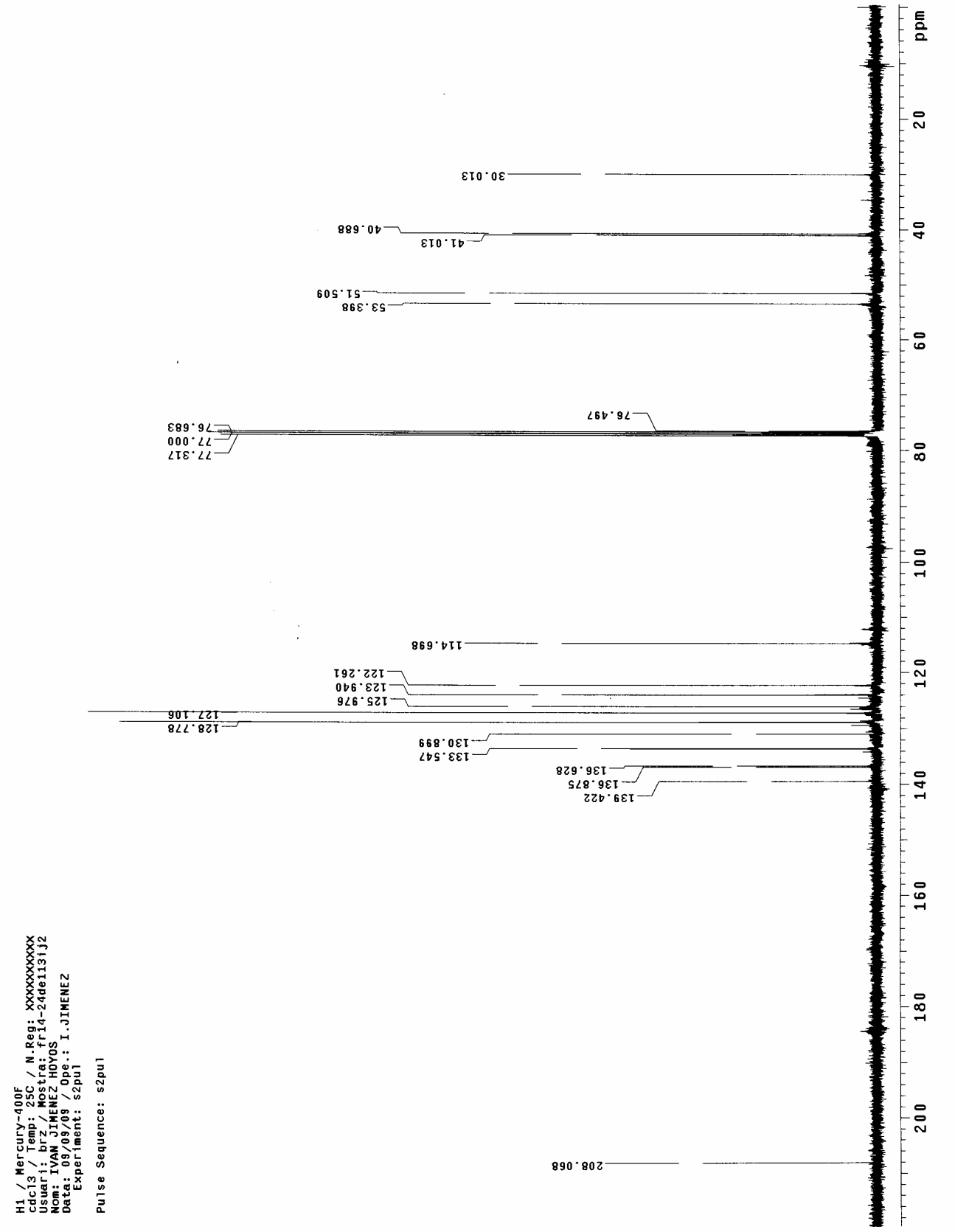

I
CH3
N
O
CH3
N
SO2Ph

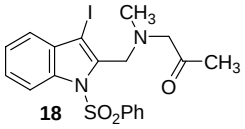
17


2.185
2.172
4.035
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr14-24de113jj2
Nom: IVAN JIMENEZ HOYOS
Data: 09/09/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

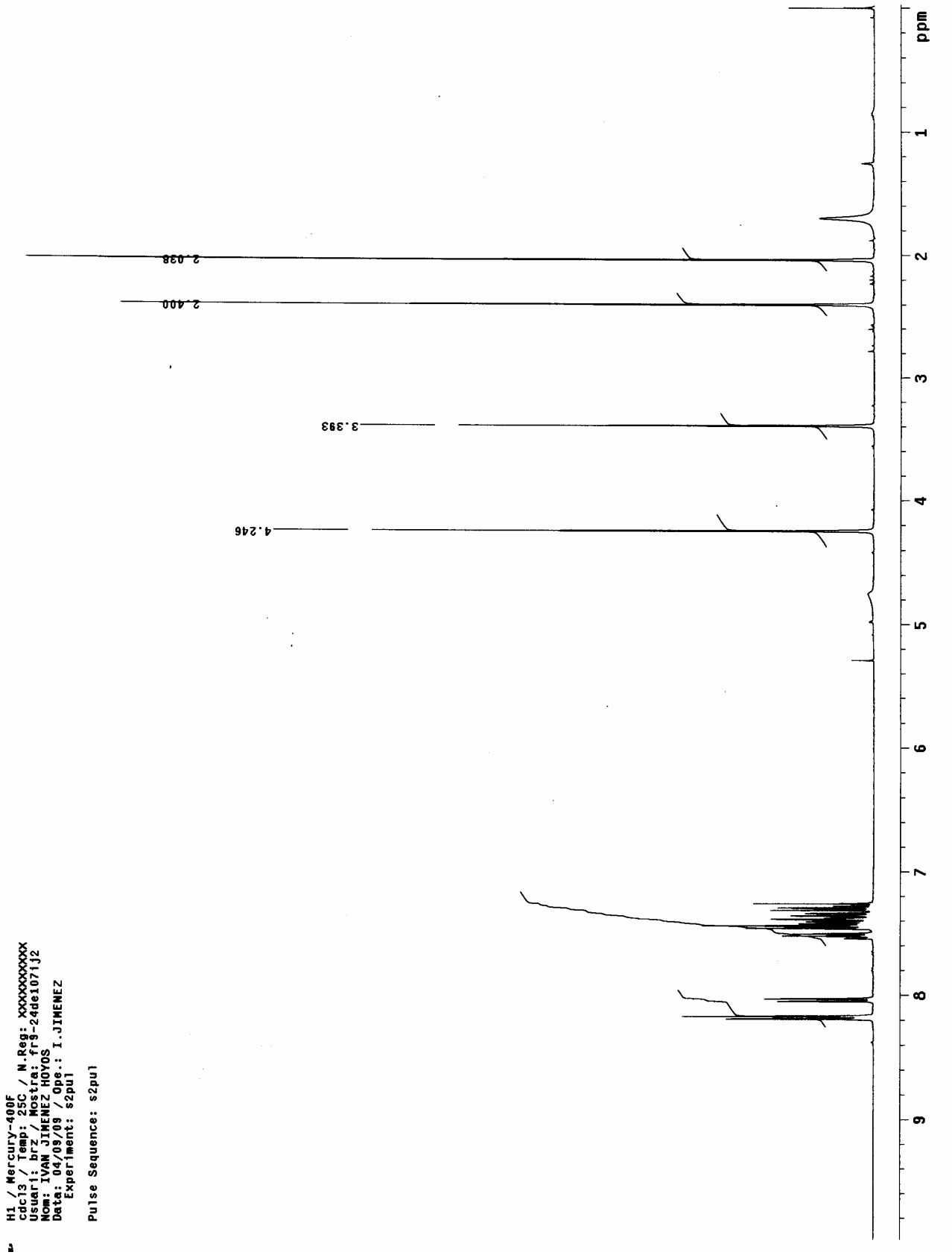

I
CH3
N
O
CH3
N
SO2Ph

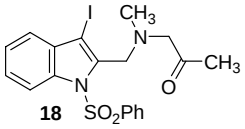
17


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brZ / Mostra: fr14-24de113ij2
Nom: IVAN JIMENEZ HOYOS
Data: 09/09/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul
208.068
139.422
136.875
136.628
133.547
130.899
128.778
127.106
125.976
123.940
122.261
114.698
77.317
77.000
76.683
76.497
53.398
51.509
41.013
40.688
30.013
ppm

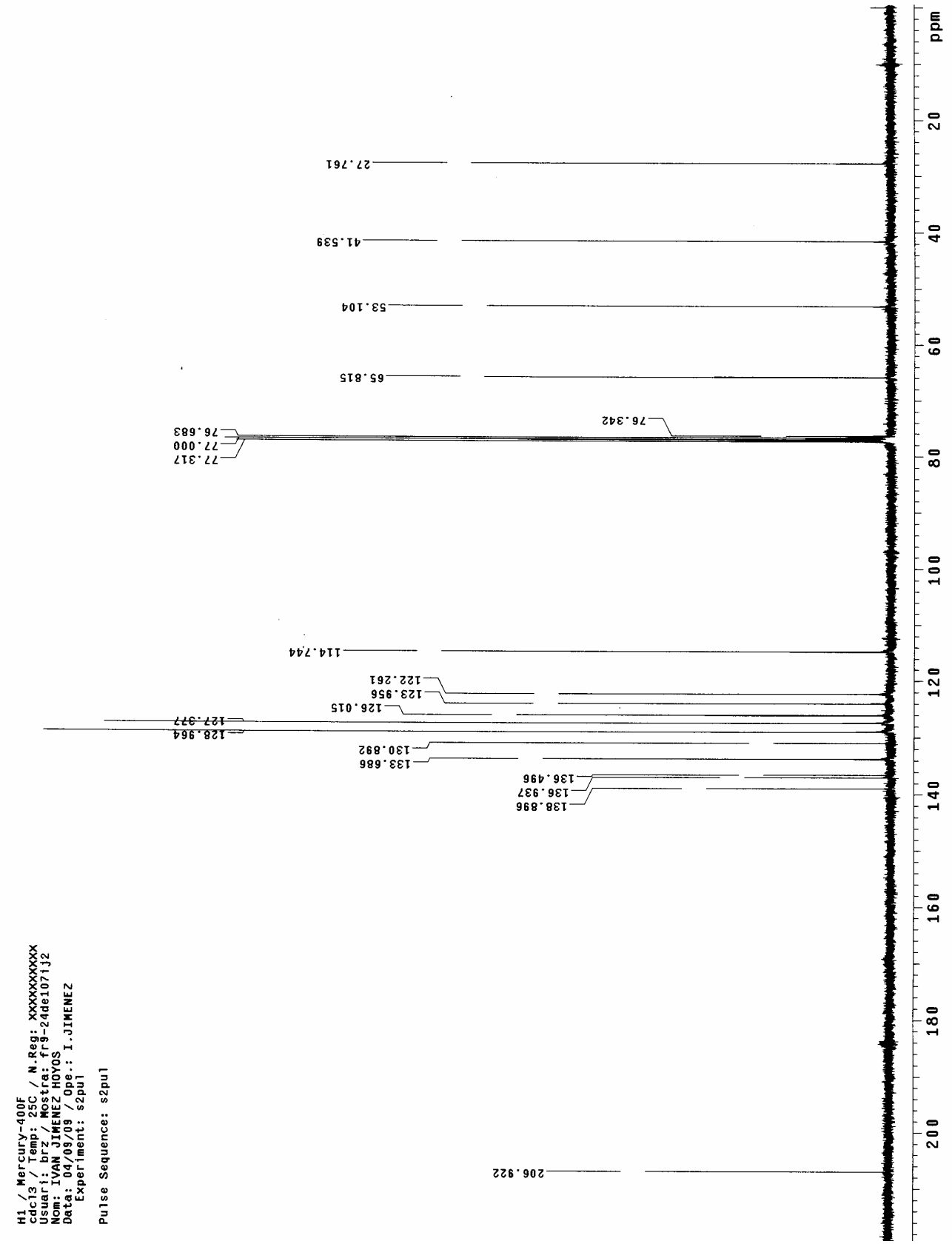

I
CH3
N
CH3
O
N
SO2Ph

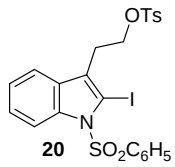
18


2.038
2.400
3.393
4.246
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr9-24de107ij2
Nom: IVAN JIMENEZ HOYOS
Data: 04/09/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

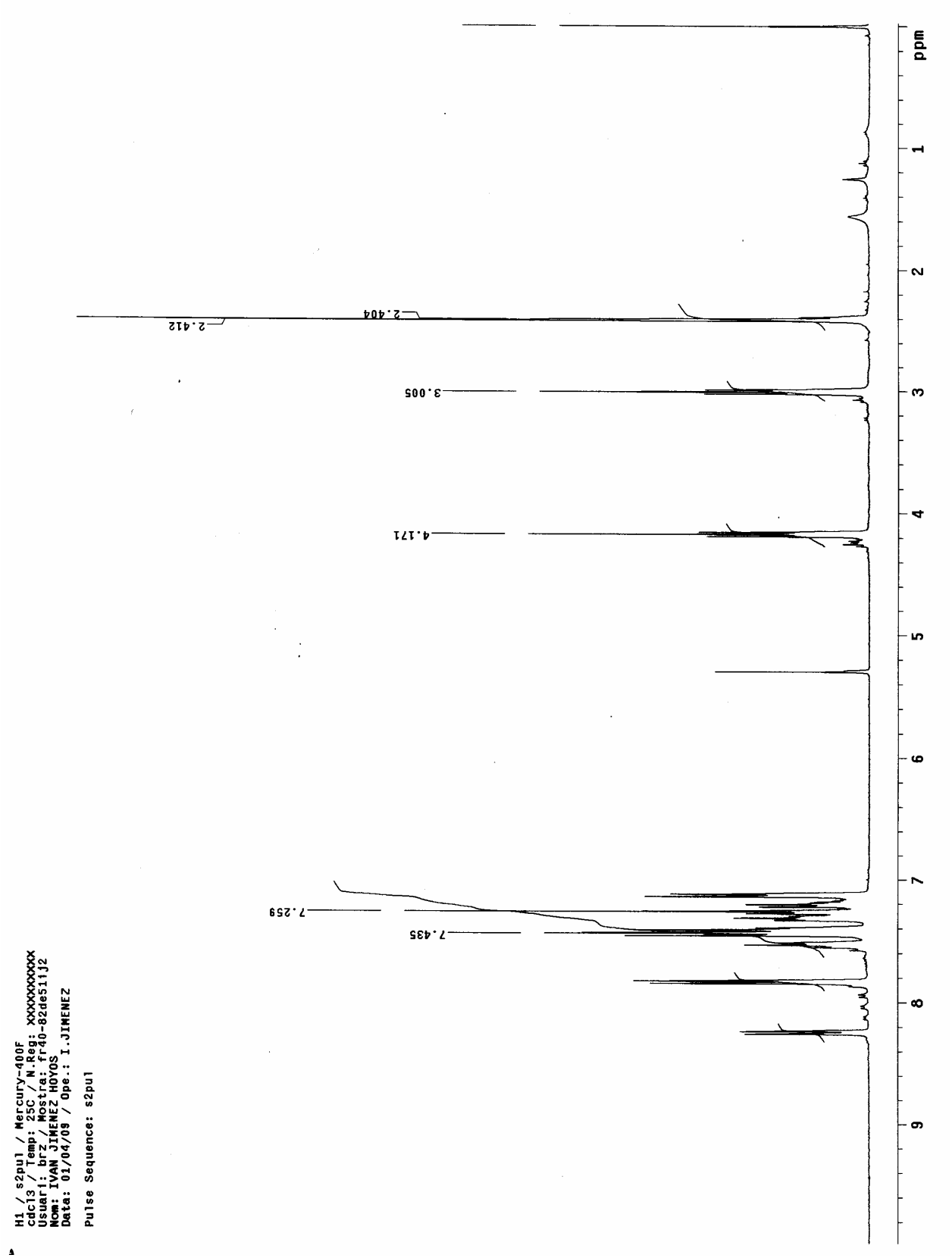

I
CH3
N
CH3
O
N
SO2Ph

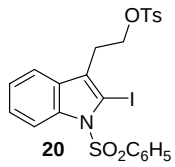
18


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr9-24de107ij2
Nom: IVAN JIMENEZ HOYOS
Data: 04/09/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul
206.922
138.896
136.937
136.496
133.686
130.892
128.964
127.377
126.015
123.956
122.261
114.744
77.317
77.000
76.683
76.342
65.815
53.104
41.539
27.761
ppm
200
180
160
140
120
100
80
60
40
20

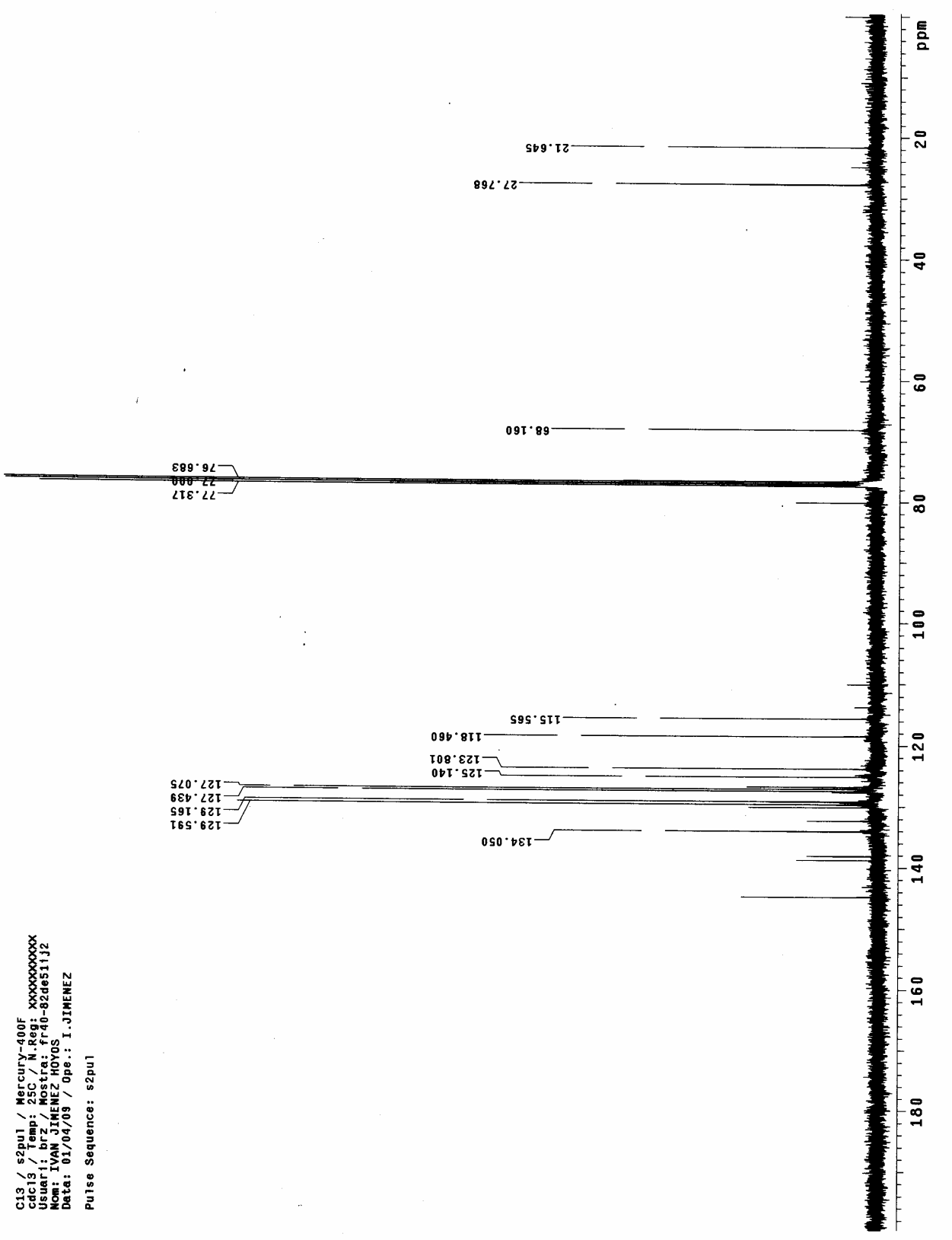

OTs
I
N
SO2C6H5

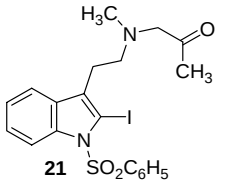
20


2.412
2.404
3.005
4.171
7.259
7.435
ppm
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr40-82de51j2
Nom: IVAM JIMENEZ HOYOS
Data: 01/04/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

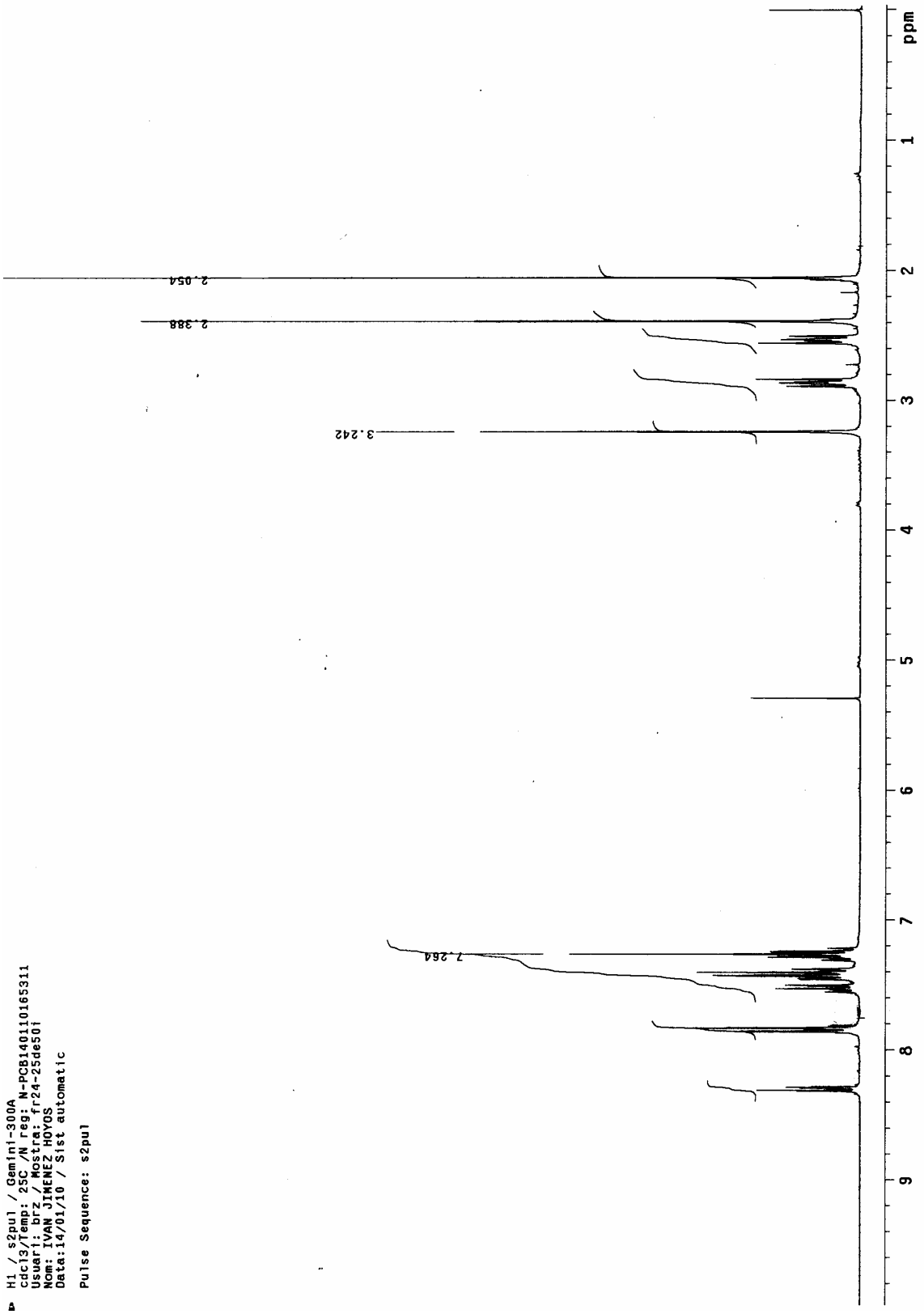

OTs
I
N
SO2C6H5

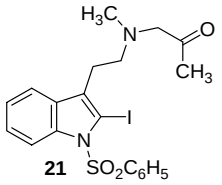
20


21.645
27.768
68.160
76.683
77.000
77.317
115.565
118.460
123.801
125.140
127.075
127.439
129.165
129.591
134.050
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr40-82de51ij2
Nom: IVAN JIMENEZ HOYOS
Data: 01/04/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

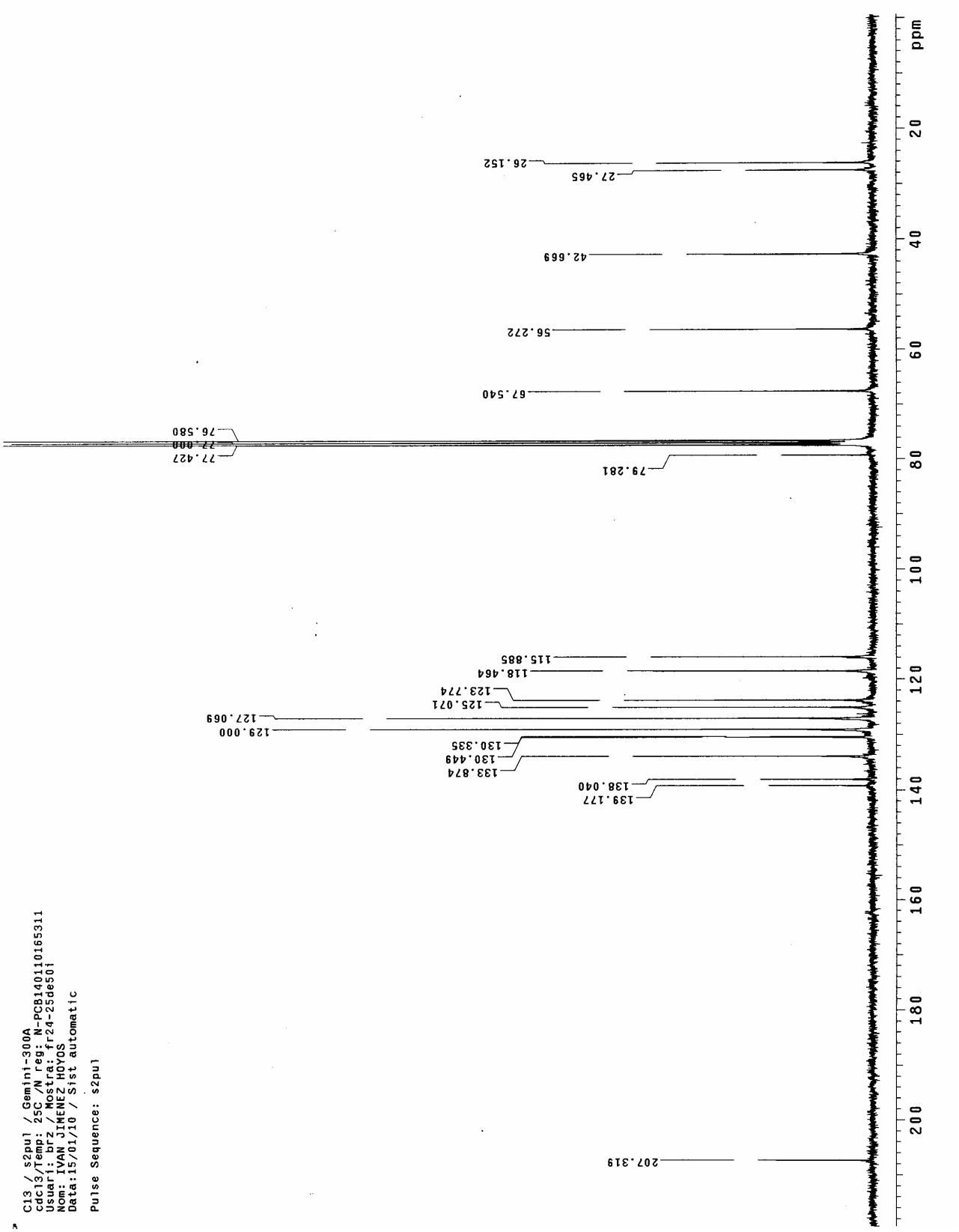

H3C
N
O
CH3
I
N
SO2C6H5

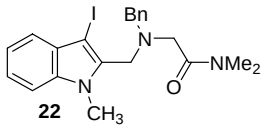
21


2.054
2.388
3.242
7.264
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB140110165311
Usuari: brz /Mostra: fr24-25de50i
Nom: IVAN JIMENEZ HOYOS
Data:14/01/10 / Sist automatic
Pulse Sequence: s2pul

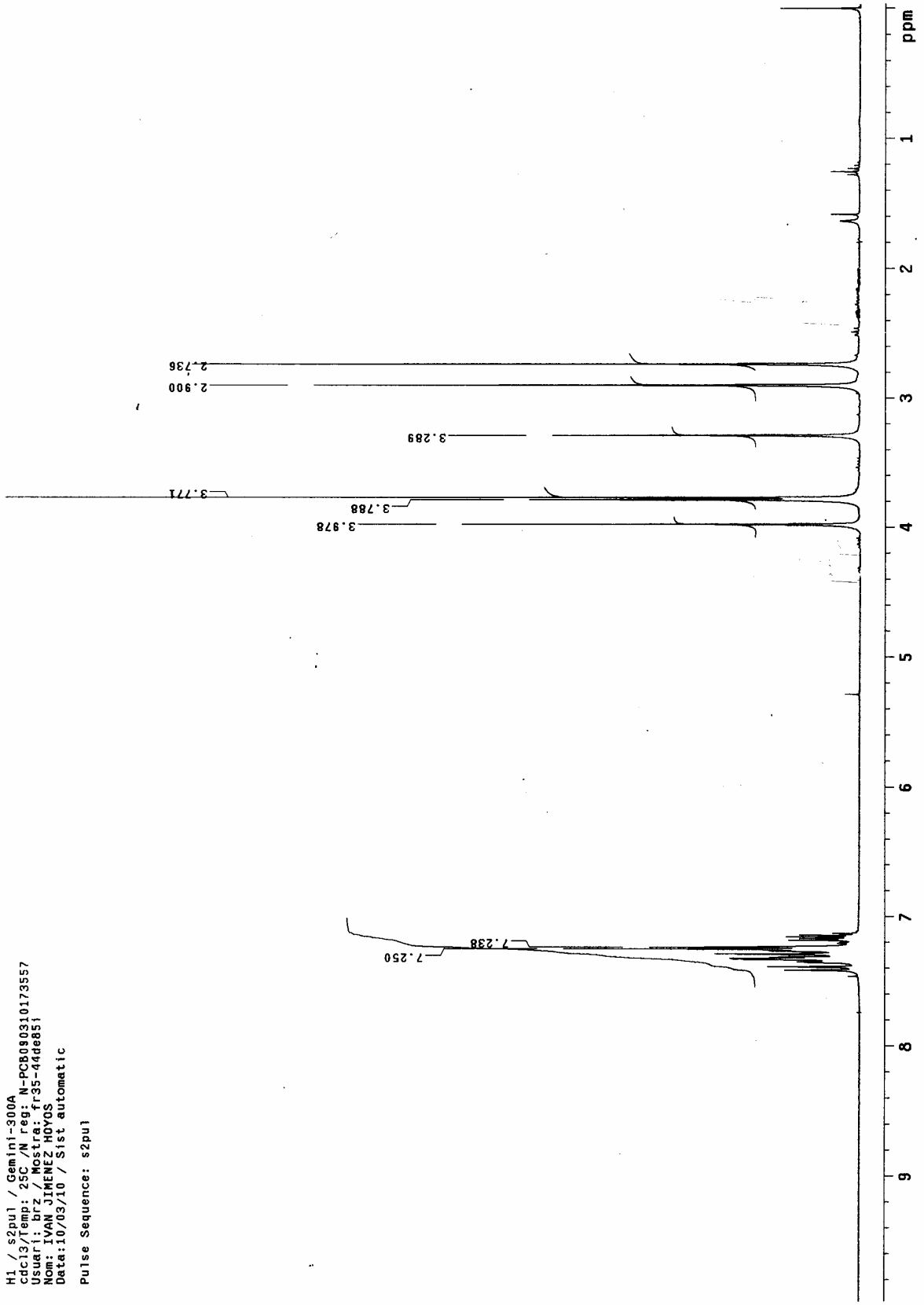

H3C
N
O
CH3
I
N
SO2C6H5

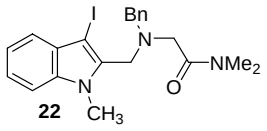
21


26.152
27.465
42.669
56.272
67.540
76.580
77.000
77.427
79.281
115.885
118.464
123.774
125.071
127.069
129.000
130.335
130.449
133.874
138.040
139.177
207.319
ppm
20
40
60
80
100
120
140
160
180
200
C13 / s2pul / Gemini-300A
CdCl3/Temp: 25C /N reg: N-PCB140110165311
Usuari: brz / Mostra: fr24-25de50i
Nom: IVAN JIMENEZ HOYOS
Data:15/01/10 / Sist automatic
Pulse Sequence: s2pul

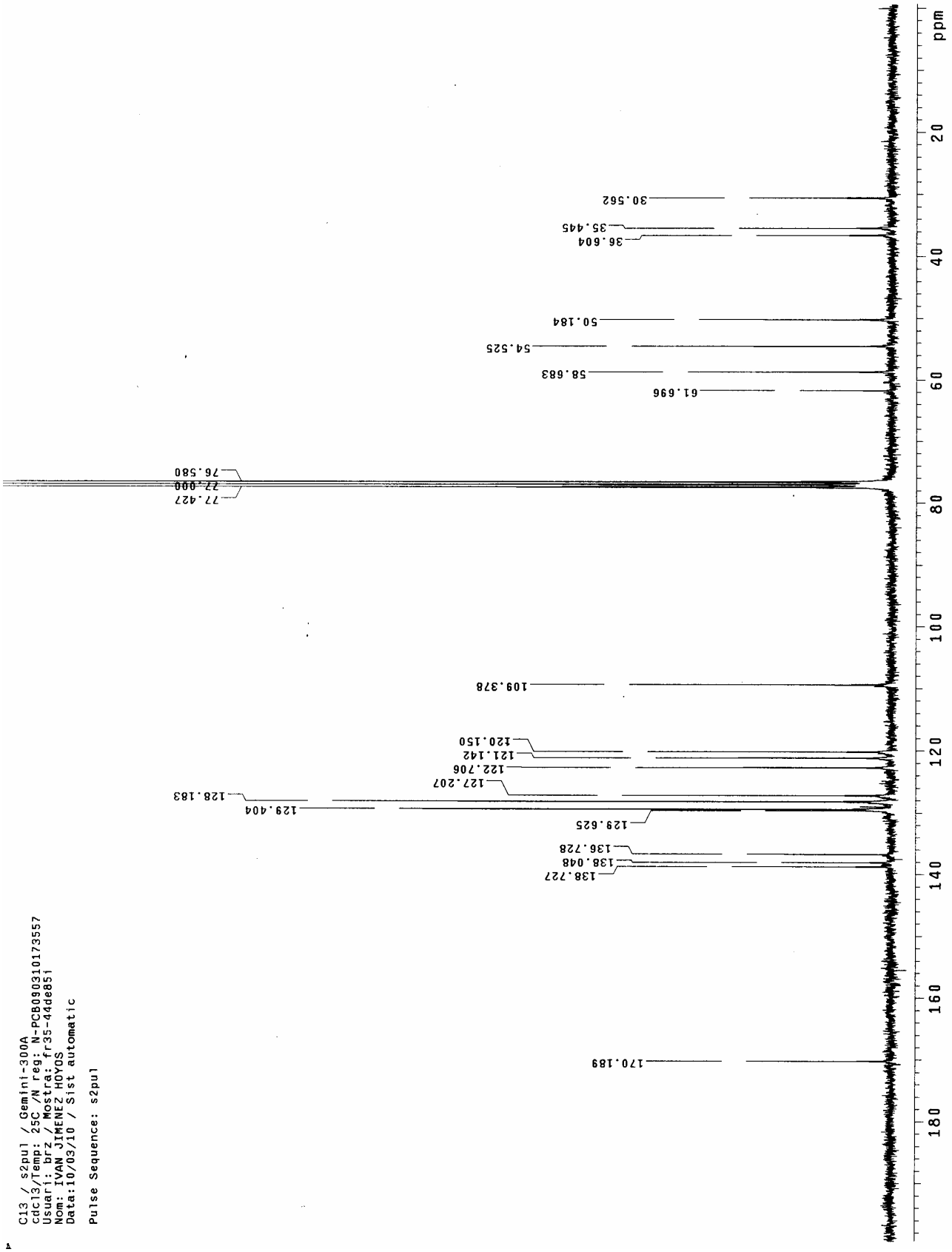

I
Bn
N
NMe2
O
N
CH3

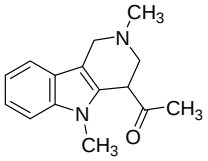
22


2.736
2.900
3.289
3.771
3.788
3.978
7.238
7.250
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
Cdcl3/Temp: 25C /N reg: N-PCB090310173557
Usuari: brz / Mostra: fr35-44de851
Nom: IVAN JIMENEZ HOYOS
Data:10/03/10 / Sist automatic
Pulse Sequence: s2pul

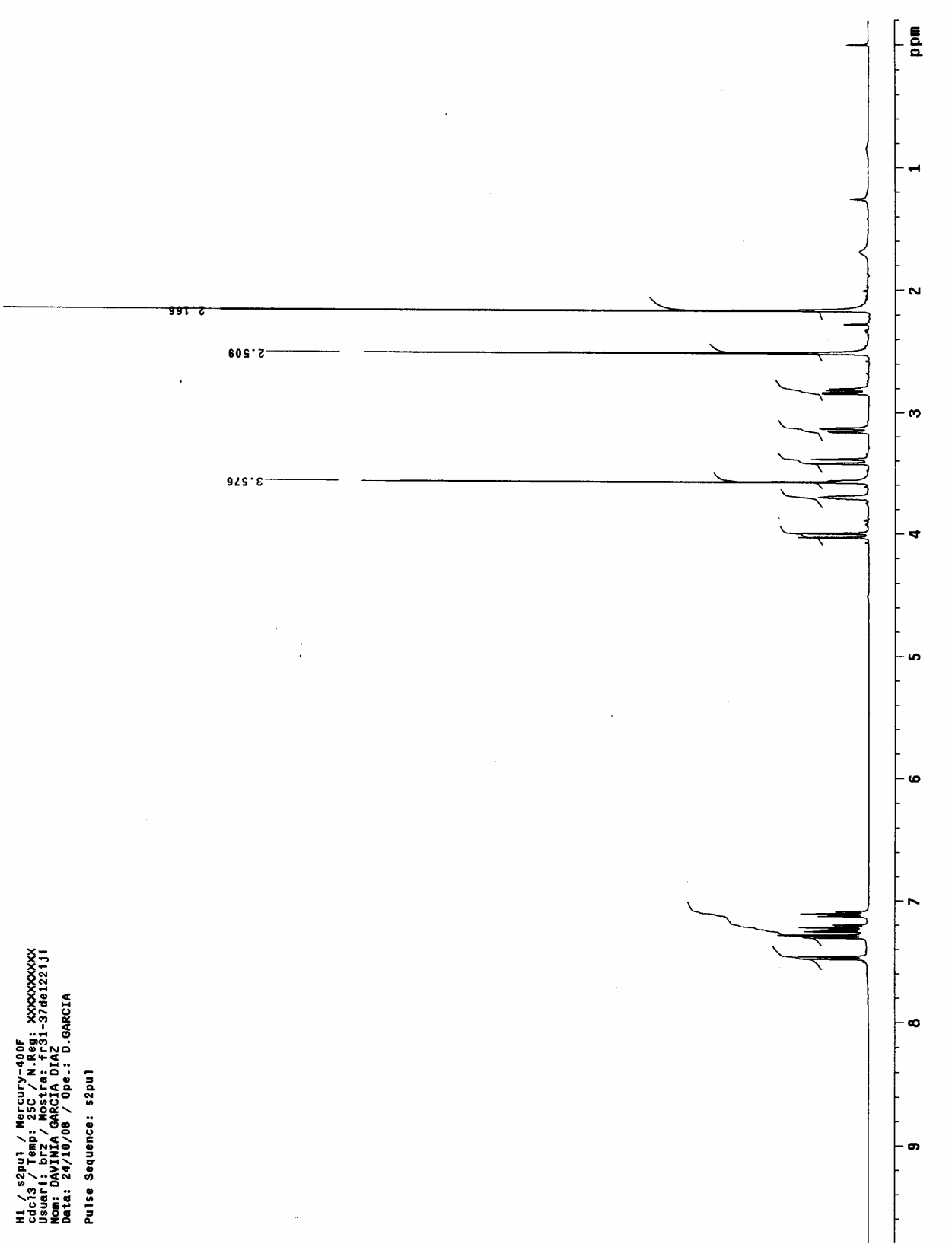

I
Bn
N
NMe2
O
N
CH3

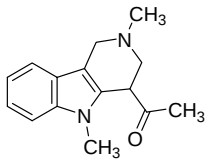
22


ppm
20
40
60
80
100
120
140
160
180
30.562
35.445
36.604
50.184
54.525
58.683
61.696
76.580
77.000
77.427
109.378
120.150
121.142
122.706
127.207
128.183
129.404
129.625
136.728
138.048
138.727
170.189
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB090310173557
Usuari: brz / Mostra: fr35-44de85i
Nom: IVAN JIMENEZ HOYOS
Data:10/03/10 / Sist automatic
Pulse Sequence: s2pul

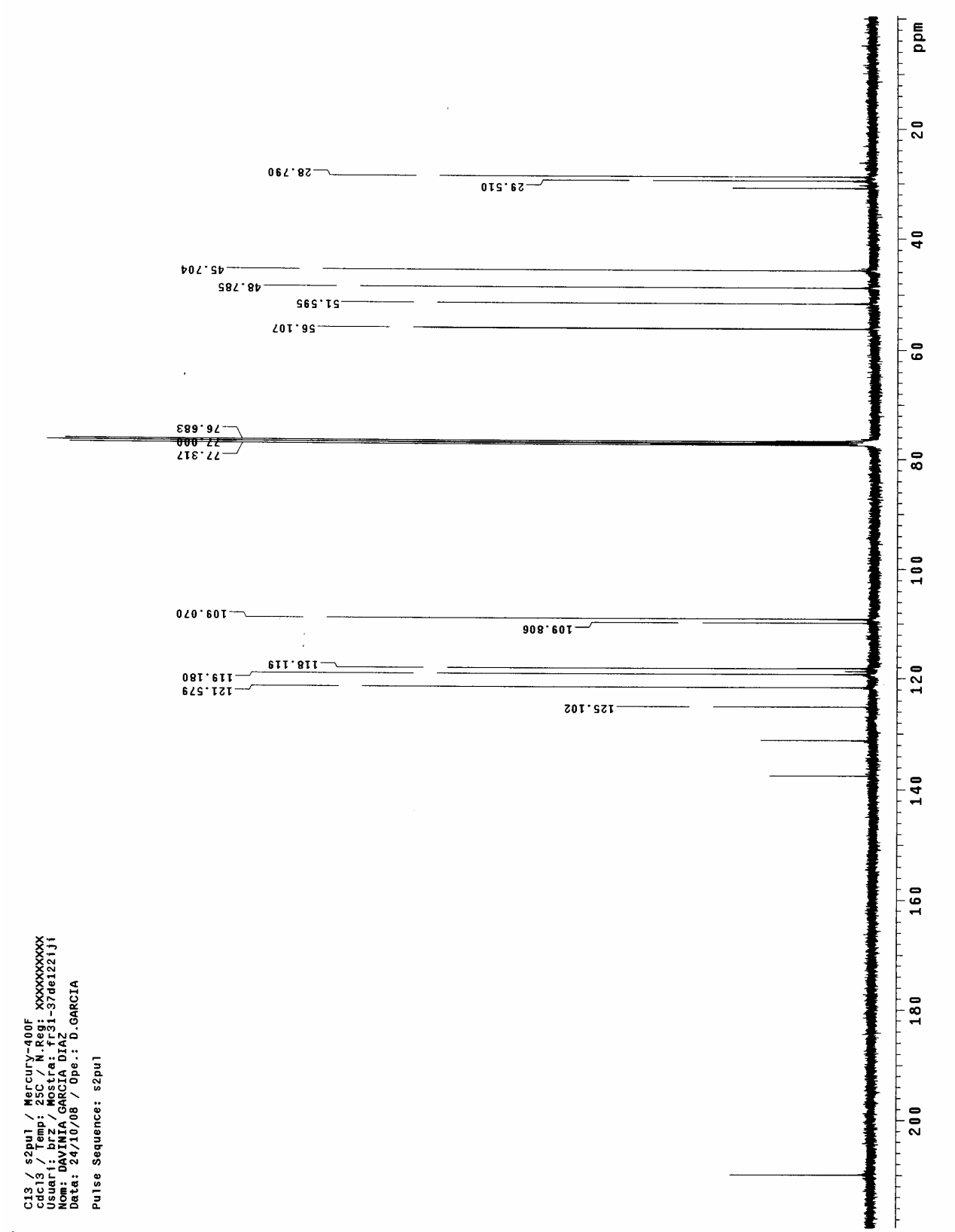


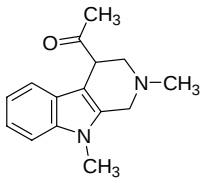
23


2.166
2.509
3.576
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
Cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr31-37de122lj1
Nom: DAVINIA GARCIA DIAZ
Data: 24/10/08 / Ope.: D.GARCIA
Pulse Sequence: s2pul

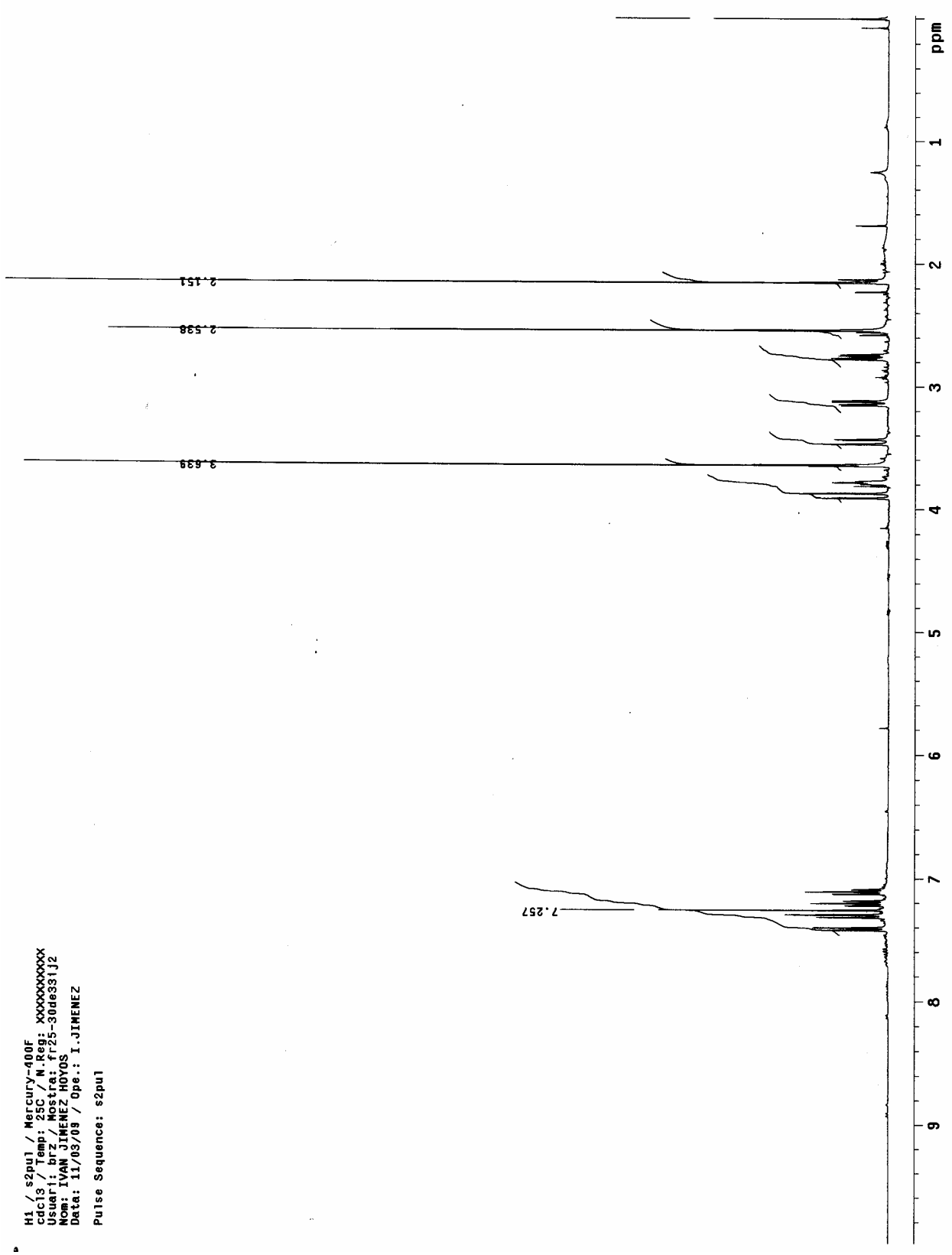


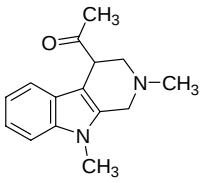
23


28.790
29.510
45.704
48.785
51.595
56.107
76.683
77.000
77.317
109.070
109.806
118.119
119.180
121.579
125.102
ppm
20
40
60
80
100
120
140
160
180
200
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brZ / Mostra: fr31-37de122iji
Nom: DAVINIA GARCIA DIAZ
Data: 24/10/08 / Ope.: D.GARCIA
Pulse Sequence: s2pul

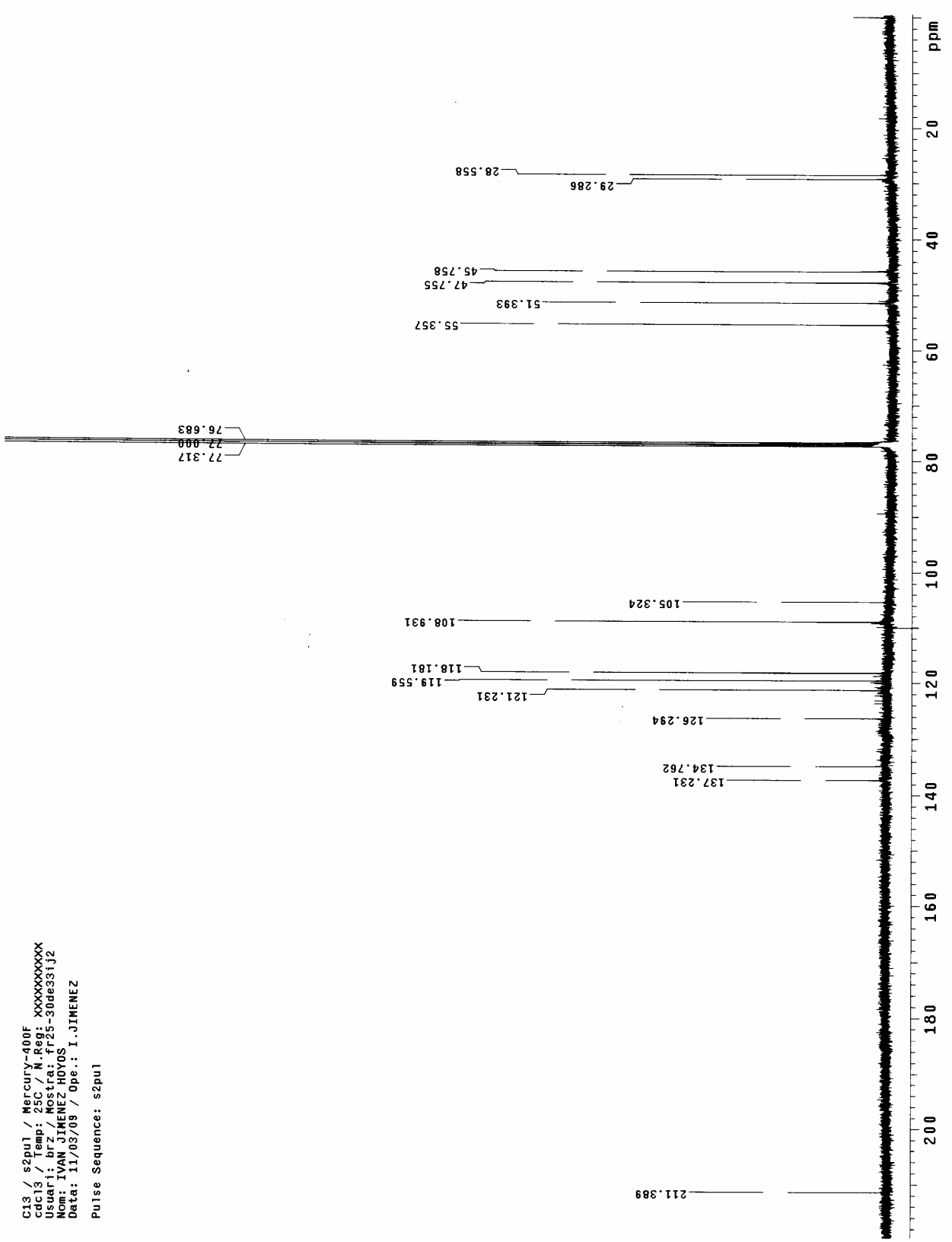

CH3
O
N−CH3
N
CH3

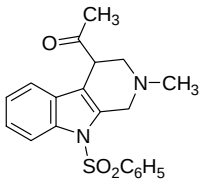
24


2.151
2.538
3.639
7.257
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brZ / Mostra: fr25-30de33ij2
Nom: IVAN JIMENEZ HOYOS
Data: 11/03/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

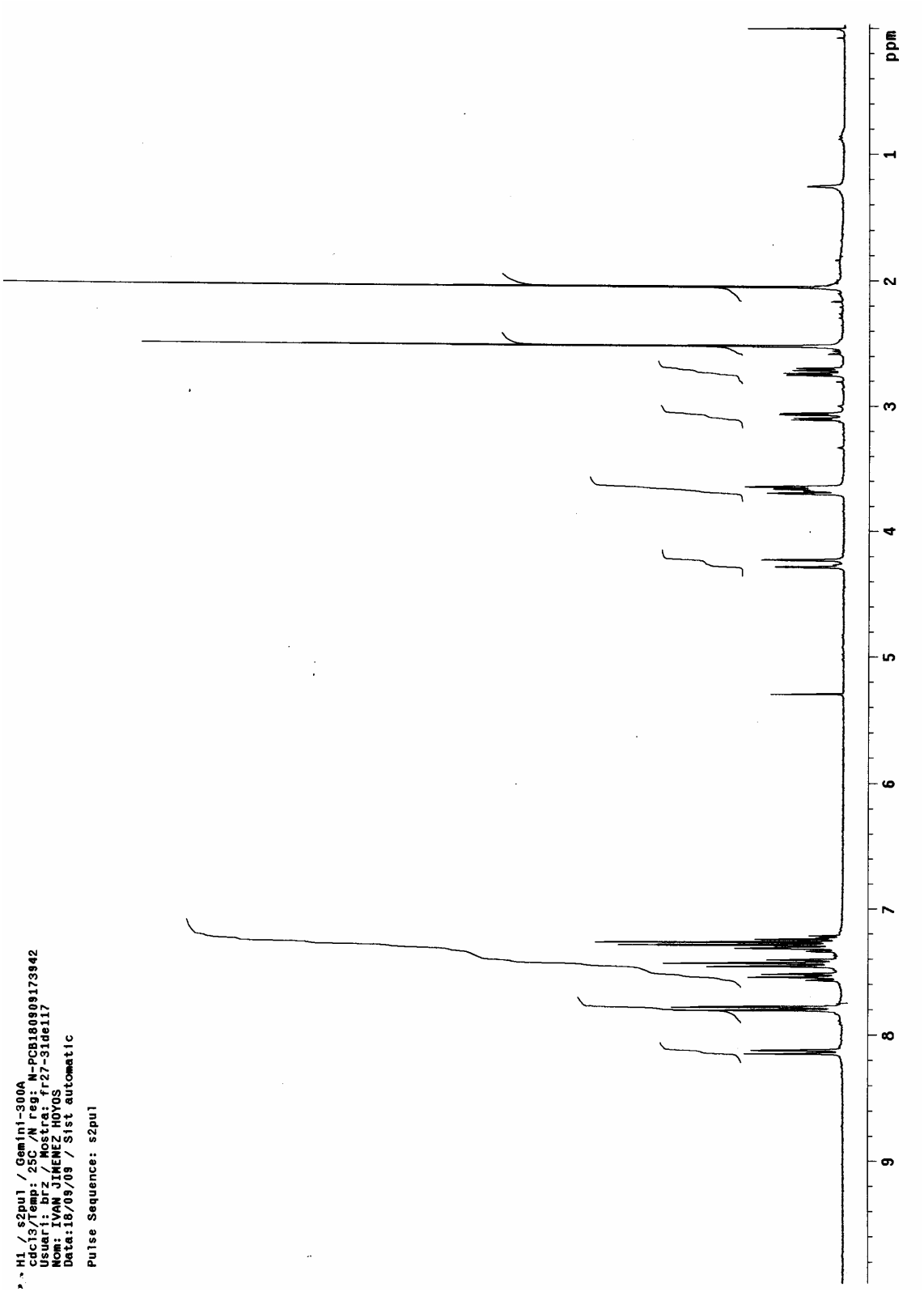

CH3
O
N−CH3
N
CH3

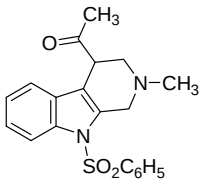
24


28.558
29.286
45.758
47.755
51.383
55.357
76.683
77.000
77.317
105.324
108.931
118.181
119.559
121.231
126.294
134.762
137.231
211.389
ppm
20
40
60
80
100
120
140
160
180
200
C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr25-30de33ij2
Nom: IVAN JIMENEZ HOYOS
Data: 11/03/09 / Ope.: I.JIMENEZ
Pulse Sequence: s2pul

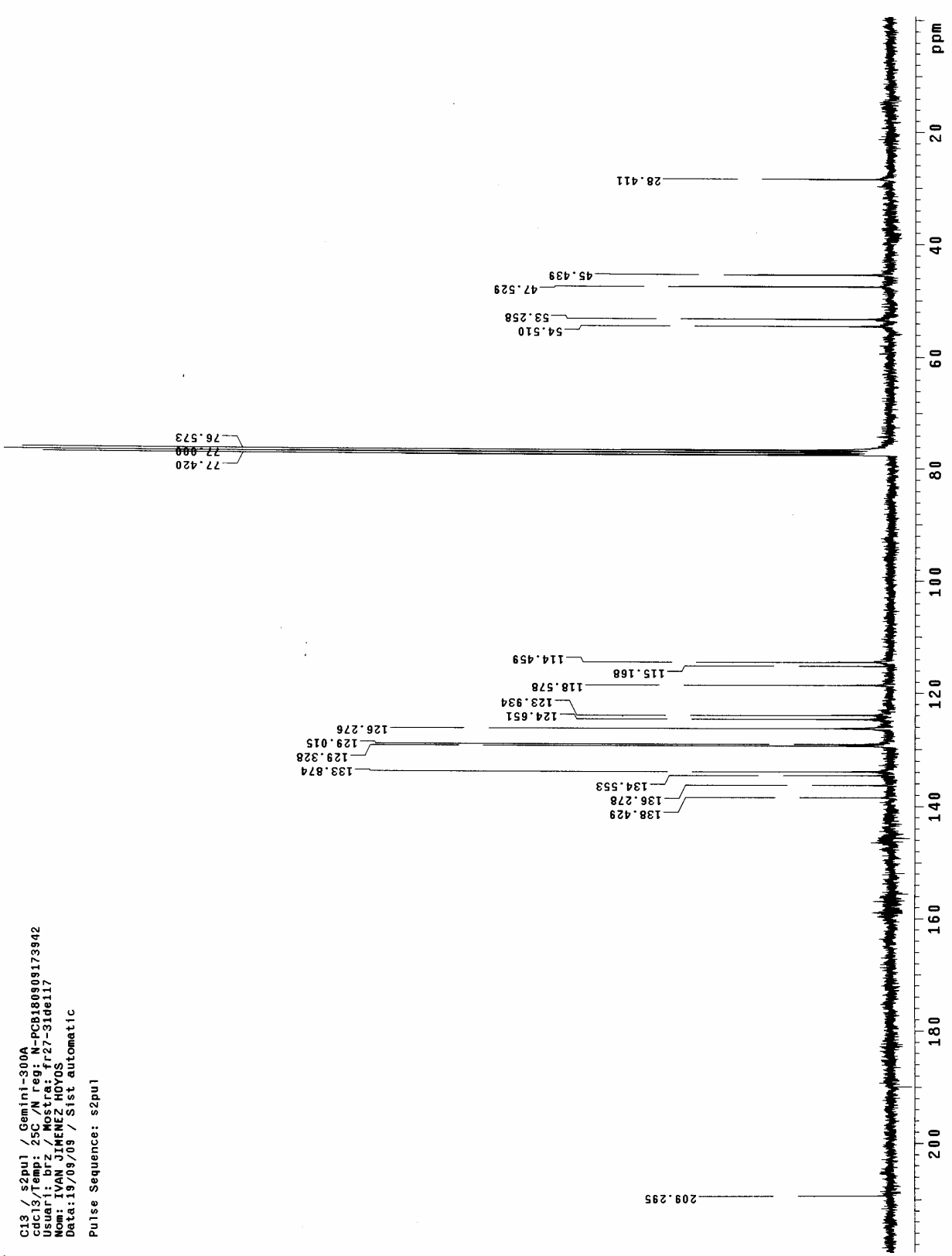

CH3
O
N−CH3
N
SO2C6H5

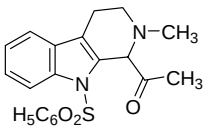
25


ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB180909173942
Usuari: brz / Mostra: fr27-31de117
Nom: IVAN JIMENEZ HOYOS
Data:18/09/09 / Sist automatic
Pulse Sequence: s2pul

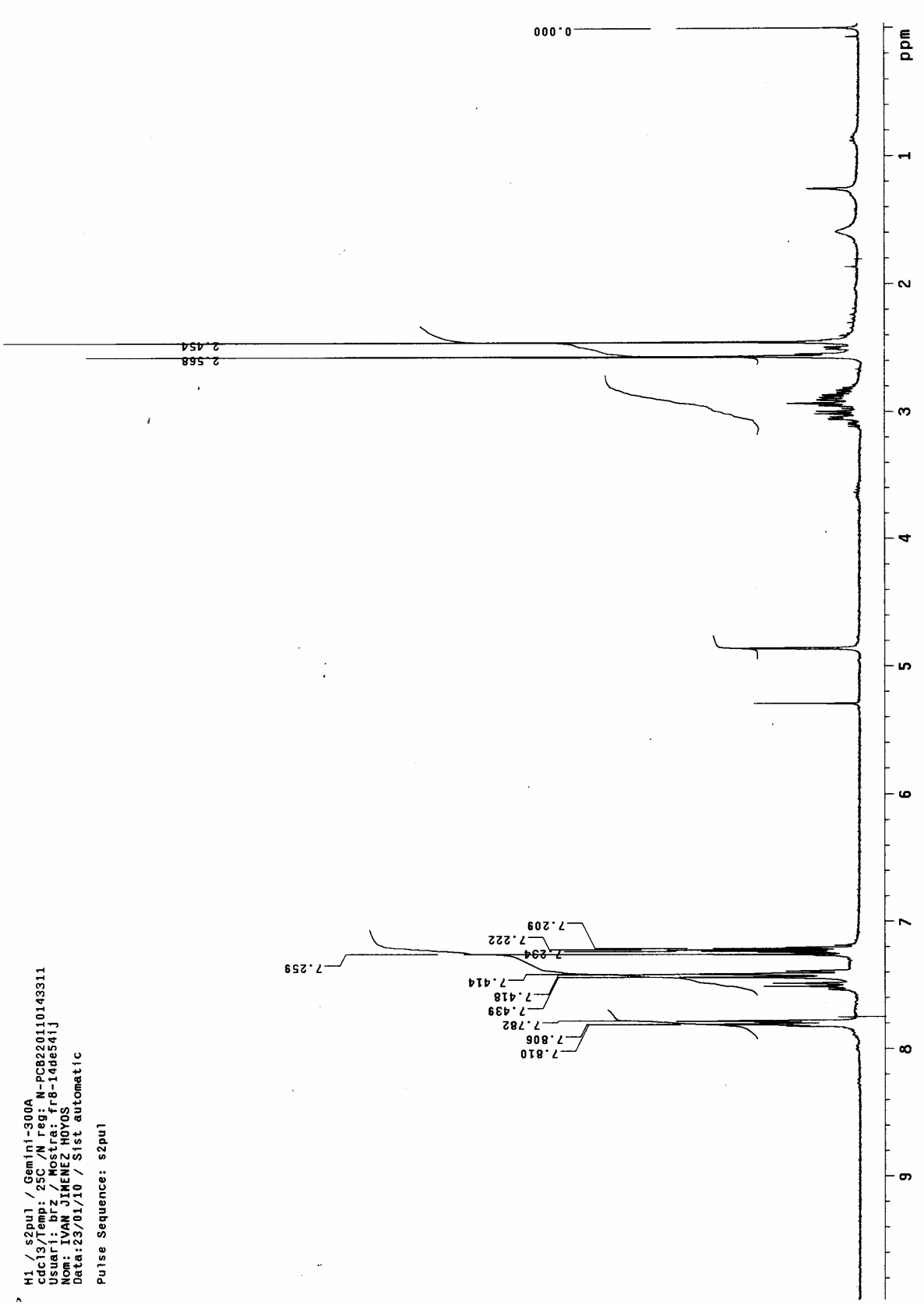

CH3
O
N−CH3
N
SO2C6H5

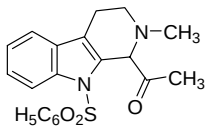
25


28.411
45.439
47.529
53.258
54.510
76.573
77.420
114.459
115.168
118.578
123.934
124.651
126.276
129.015
129.328
133.874
134.553
136.278
138.429
209.295
ppm
20
40
60
80
100
120
140
160
180
200
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB180909173942
Usuari: brz / Mostra: fr27-31de117
Nom: IVAN JIMENEZ HOYOS
Data:19/09/09 / Sist automatic
Pulse Sequence: s2pul

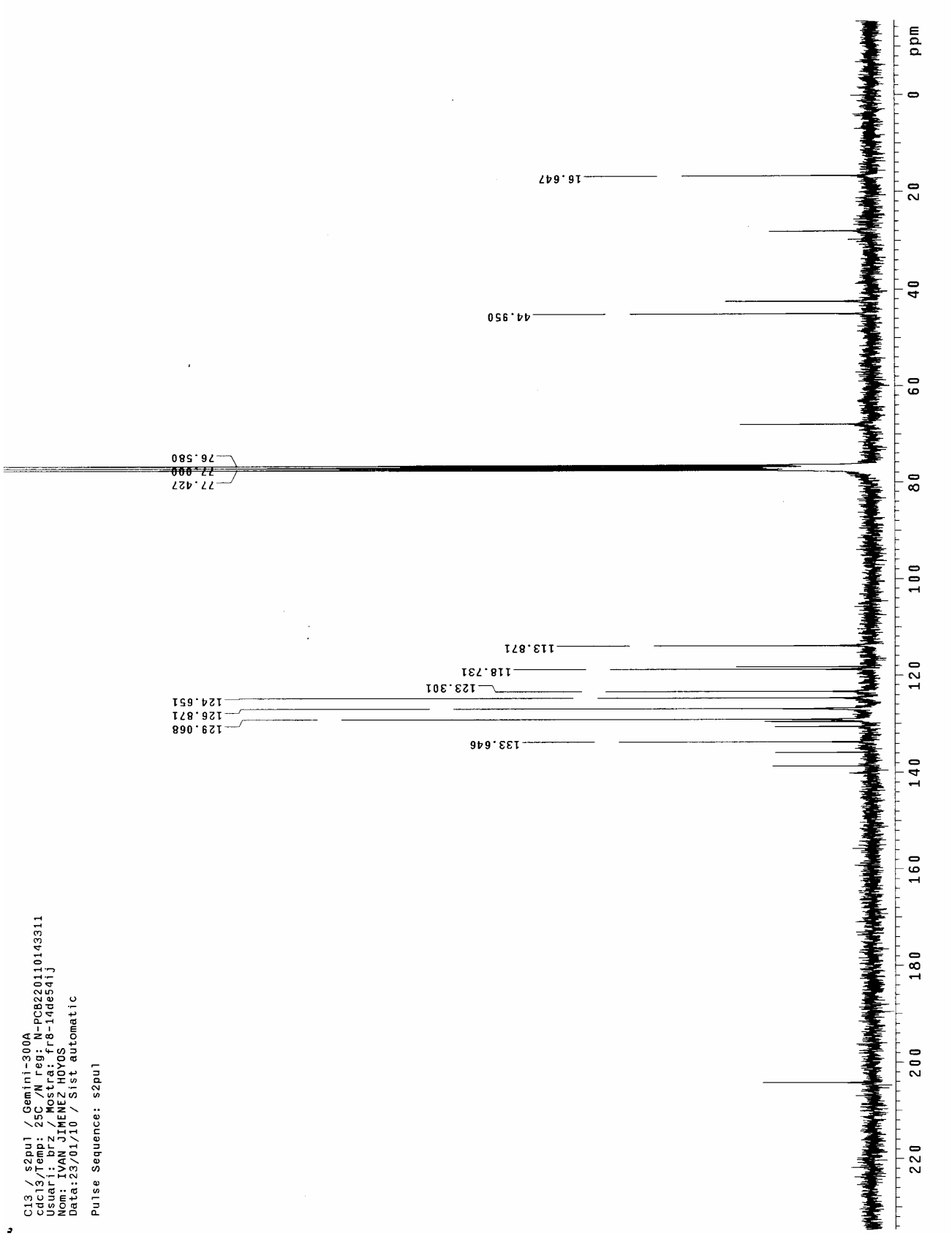

N−CH3
CH3
N
O
H5C6O2S

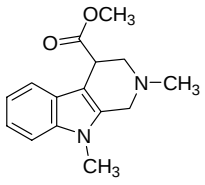
26


0.000
2.454
2.568
7.209
7.222
7.234
7.259
7.414
7.418
7.439
7.782
7.806
7.810
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB220110143311
Usuari: brz / Mostra: fr8-14de54ij
Nom: IVAN JIMENEZ HOYOS
Data:23/01/10 / Sist automatic
Pulse Sequence: s2pul

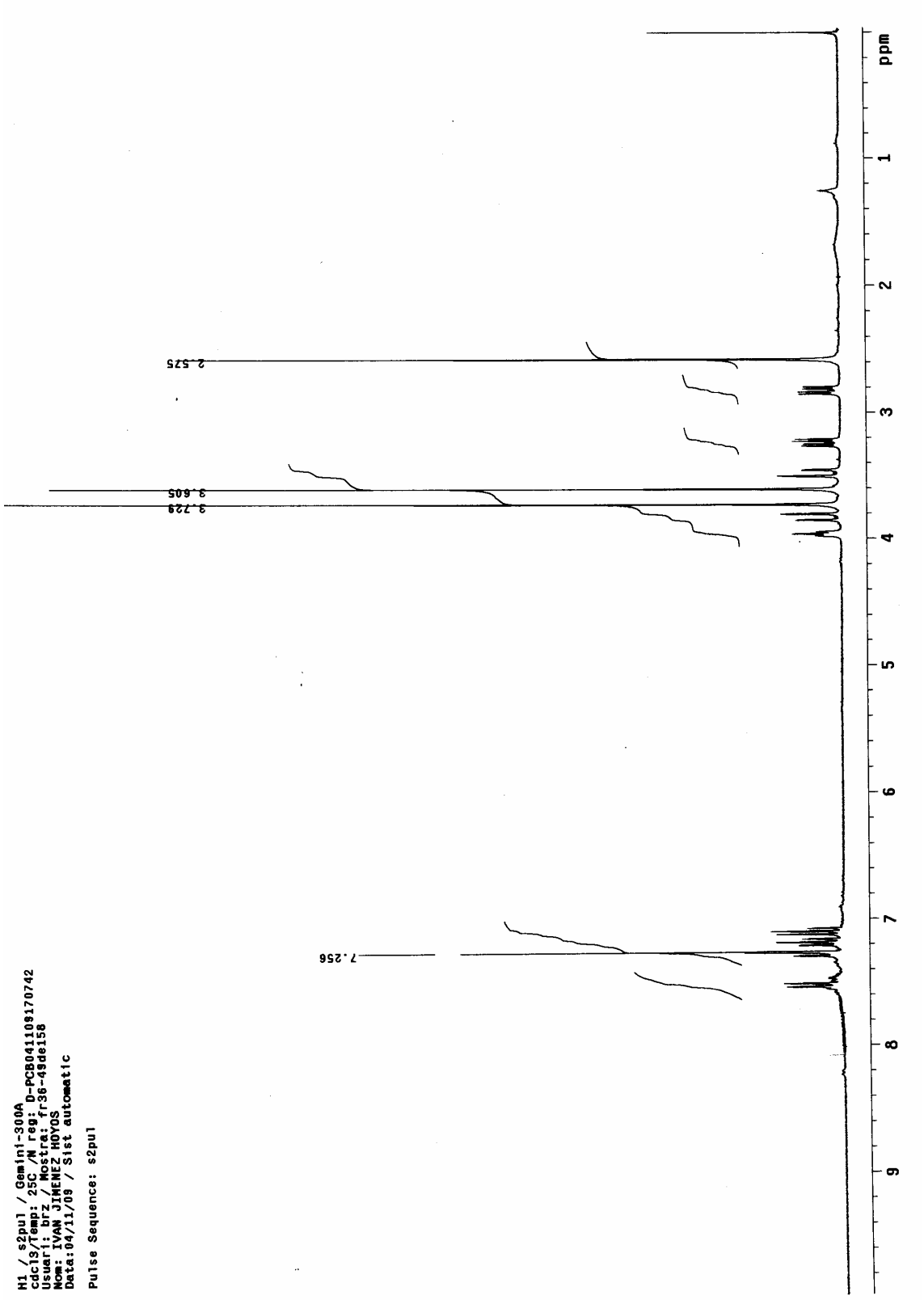

N−CH3
CH3
N
O
H5C6O2S

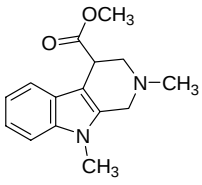
26


16.647
44.950
76.580
77.000
77.427
113.871
118.731
123.301
124.651
126.871
129.068
133.646
ppm
0
20
40
60
80
100
120
140
160
180
200
220
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB220110143311
Usuari: brz / Mostra: fr8-14de54ij
Nom: IVAN JIMENEZ HOYOS
Data:23/01/10 / Sist automatic
Pulse Sequence: s2pul

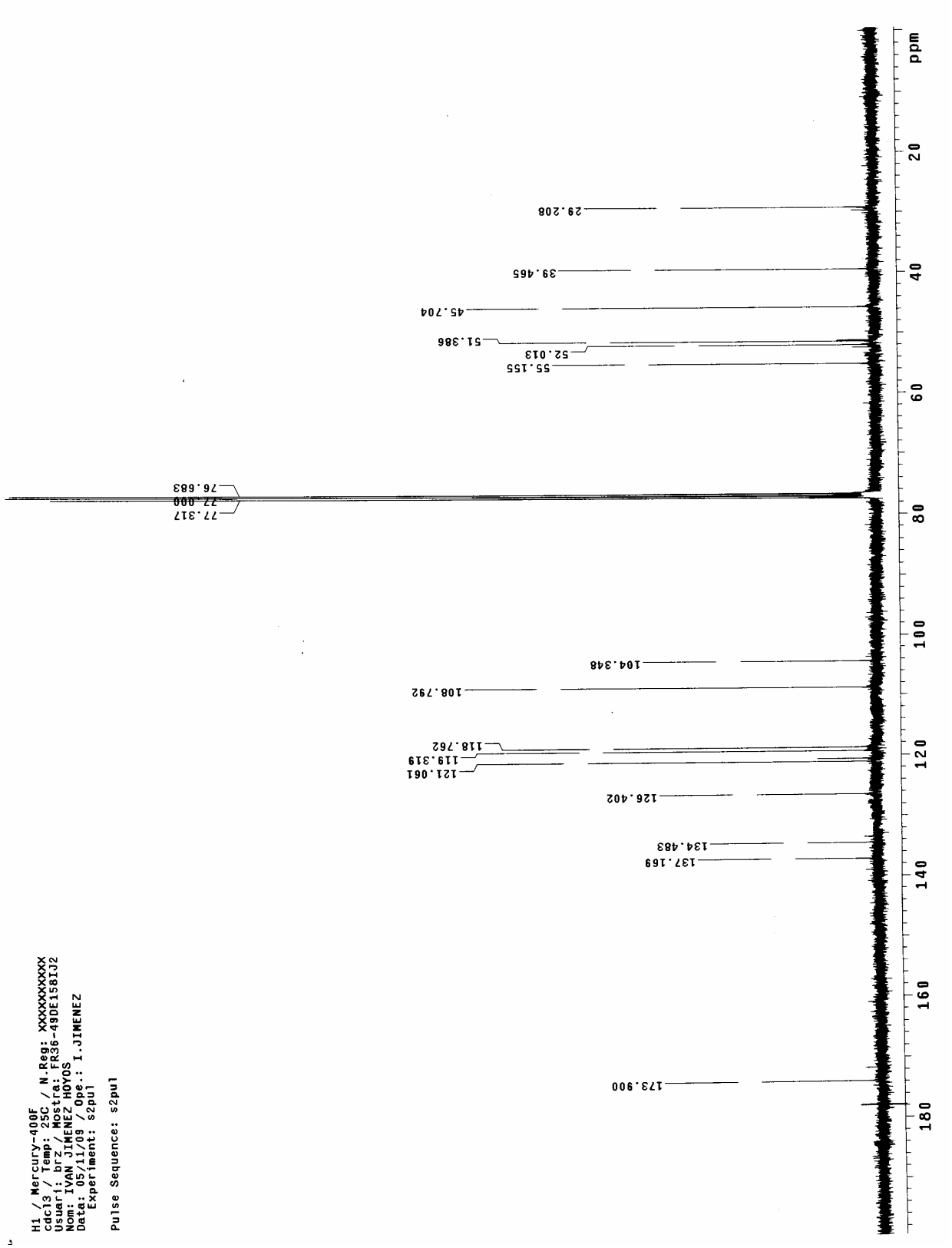

OCH3
O
N−CH3
N
CH3

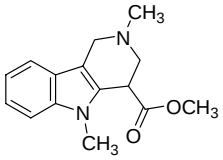
27


2.525
3.605
3.729
7.256
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: D-PCB04110917074 2
Usuari: brz / Mostra: fr36-49de158
NOM: IVAN JIMENEZ HOYOS
Data:04/11/09 / Sist automatic
Pulse Sequence: s2pul

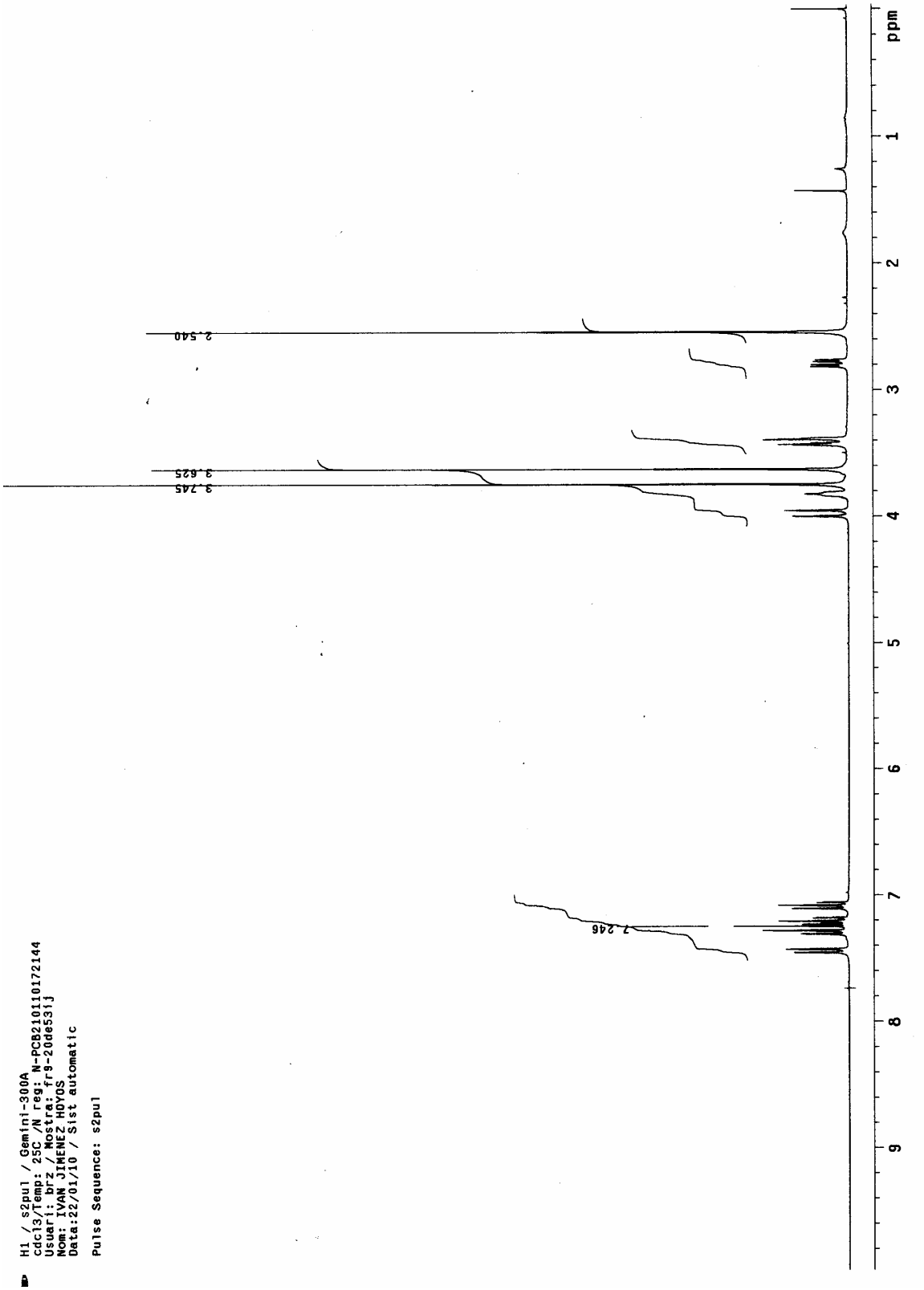

OCH3
O
N−CH3
N
CH3

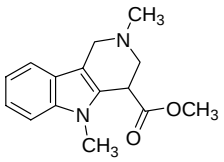
27


ppm
20
40
60
80
100
120
140
160
180
29.208
39.465
45.704
51.386
52.013
55.155
76.683
77.000
77.317
104.348
108.792
118.762
119.319
121.061
126.402
134.483
137.169
173.900
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: FR36-49DE158IJ2
Nom: IVAN JIMENEZ HOYOS
Date: 05/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

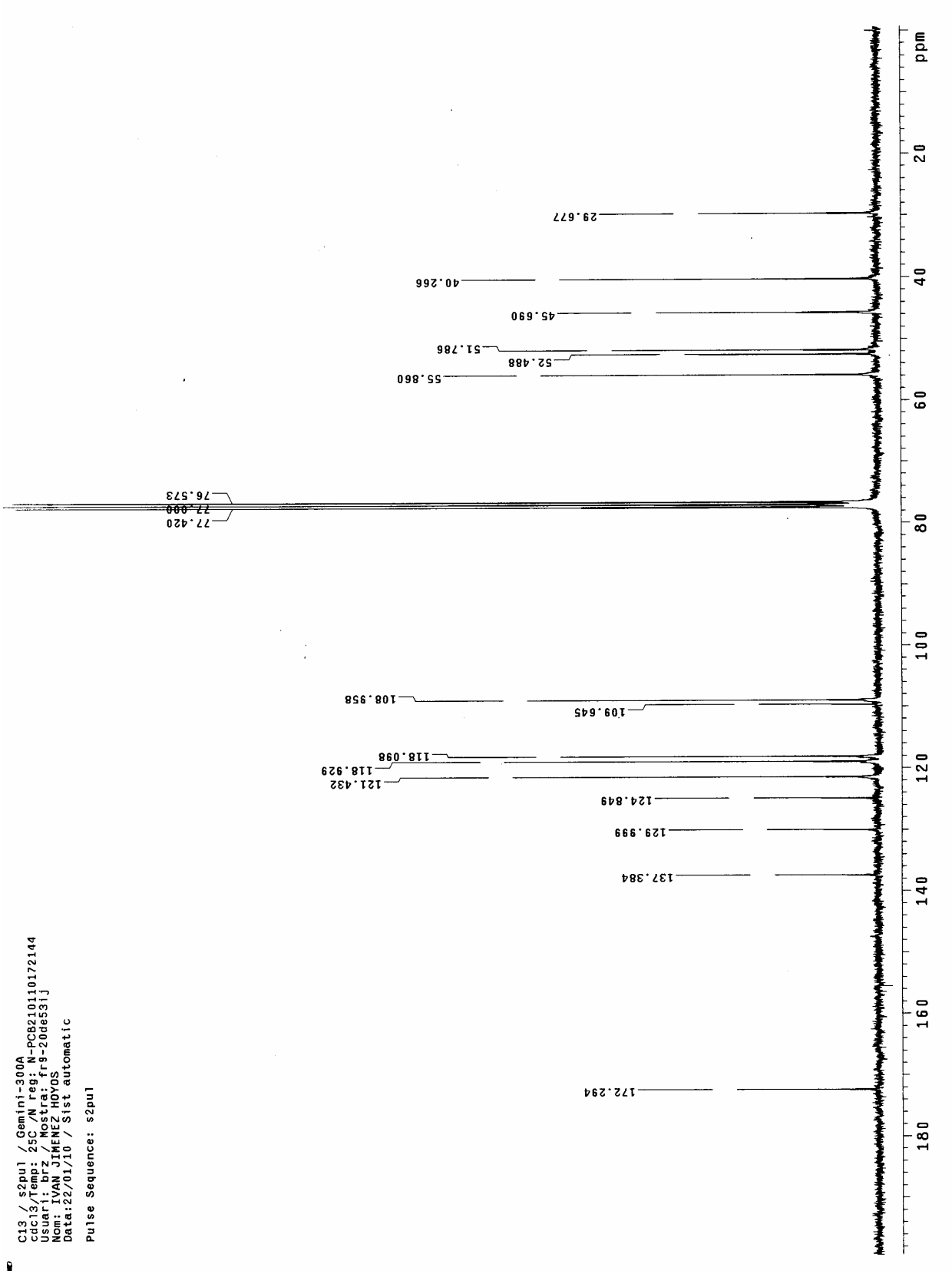

CH3
N
N
CH3
OCH3
O

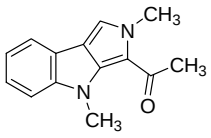
28


2.540
3.745
3.625
7.246
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB210110172144
Usuari: brz / Mostra: fr9-20de531j
Nom: IVAN JIMENEZ HOYOS
Data:22/01/10 / Sist automatic
Pulse Sequence: s2pul

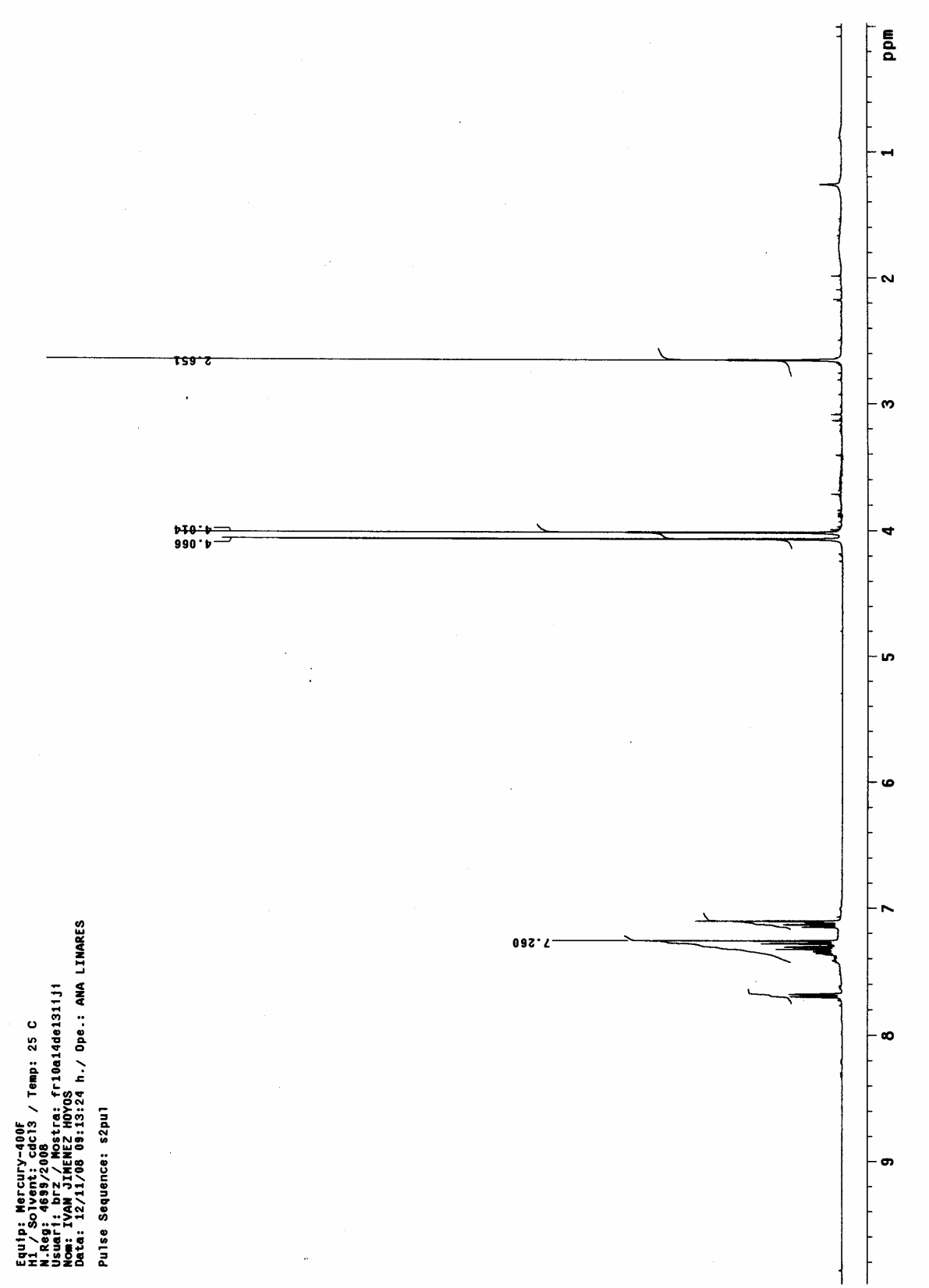

CH3
N
N
CH3
OCH3
O

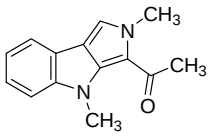
28


29.677
40.266
45.690
51.786
52.488
55.860
76.573
77.089
77.420
108.958
109.645
118.098
118.929
121.432
124.849
129.999
137.384
172.294
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB21011017214
Usuari: brz /Mostra: fr9-20de53ij
Nom: IVAN JIMENEZ,HOYOS
Data:22/01/10 / Sist automatic
Pulse Sequence: s2pul

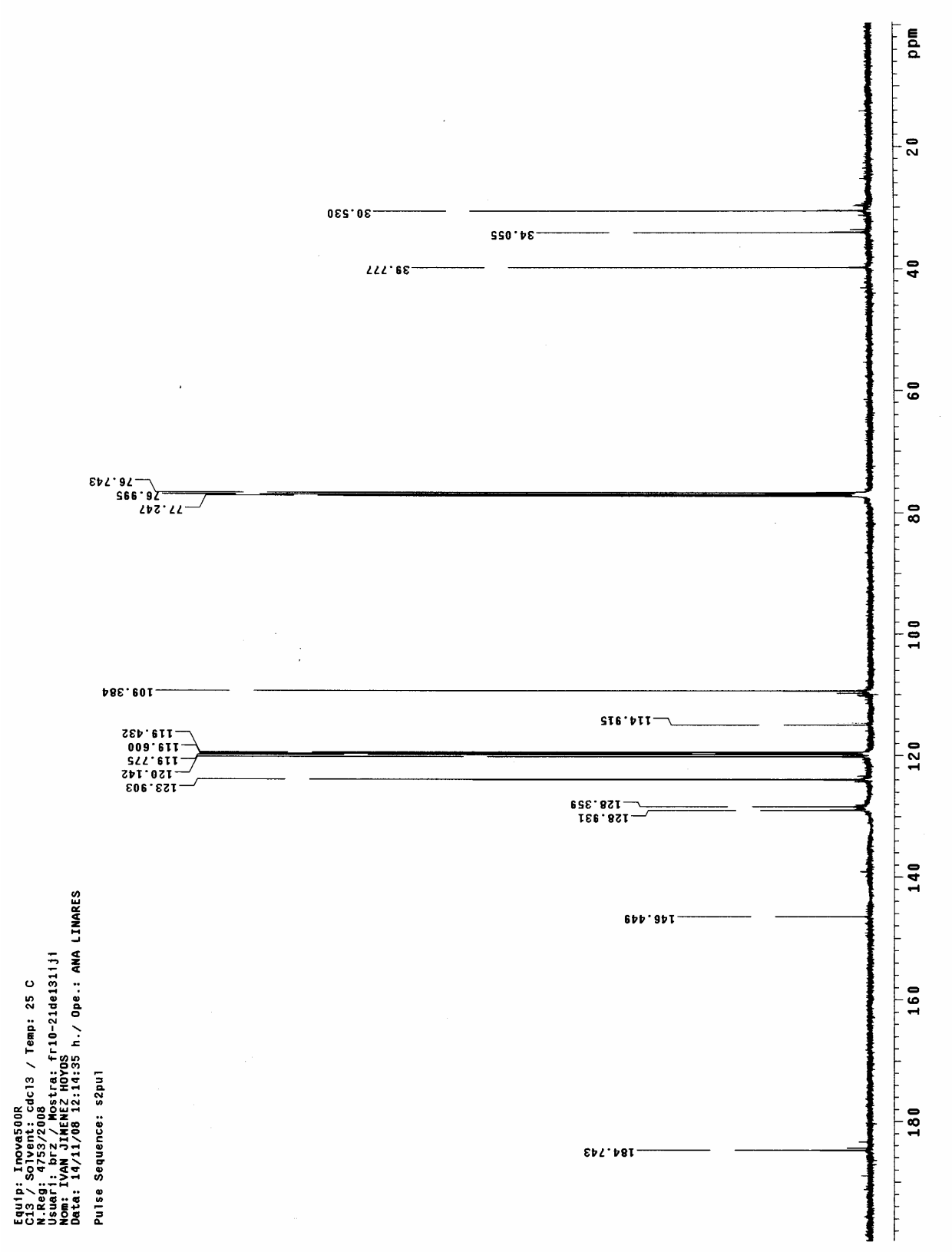

N
CH3
CH3
O
N
CH3

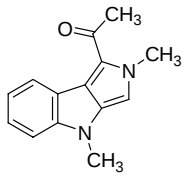
29


2.651
4.014
4.066
7.260
ppm
1
2
3
4
5
6
7
8
9
Equip: Mercury-400F
H1 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 4699/2008
Usuari: brz / Mostra: fr10a14de1311j1
Nom: IVAN JIMENEZ HOYOS
Data: 12/11/08 09:13:24 h./ Ope.: ANA LINARES
Pulse Sequence: s2pul

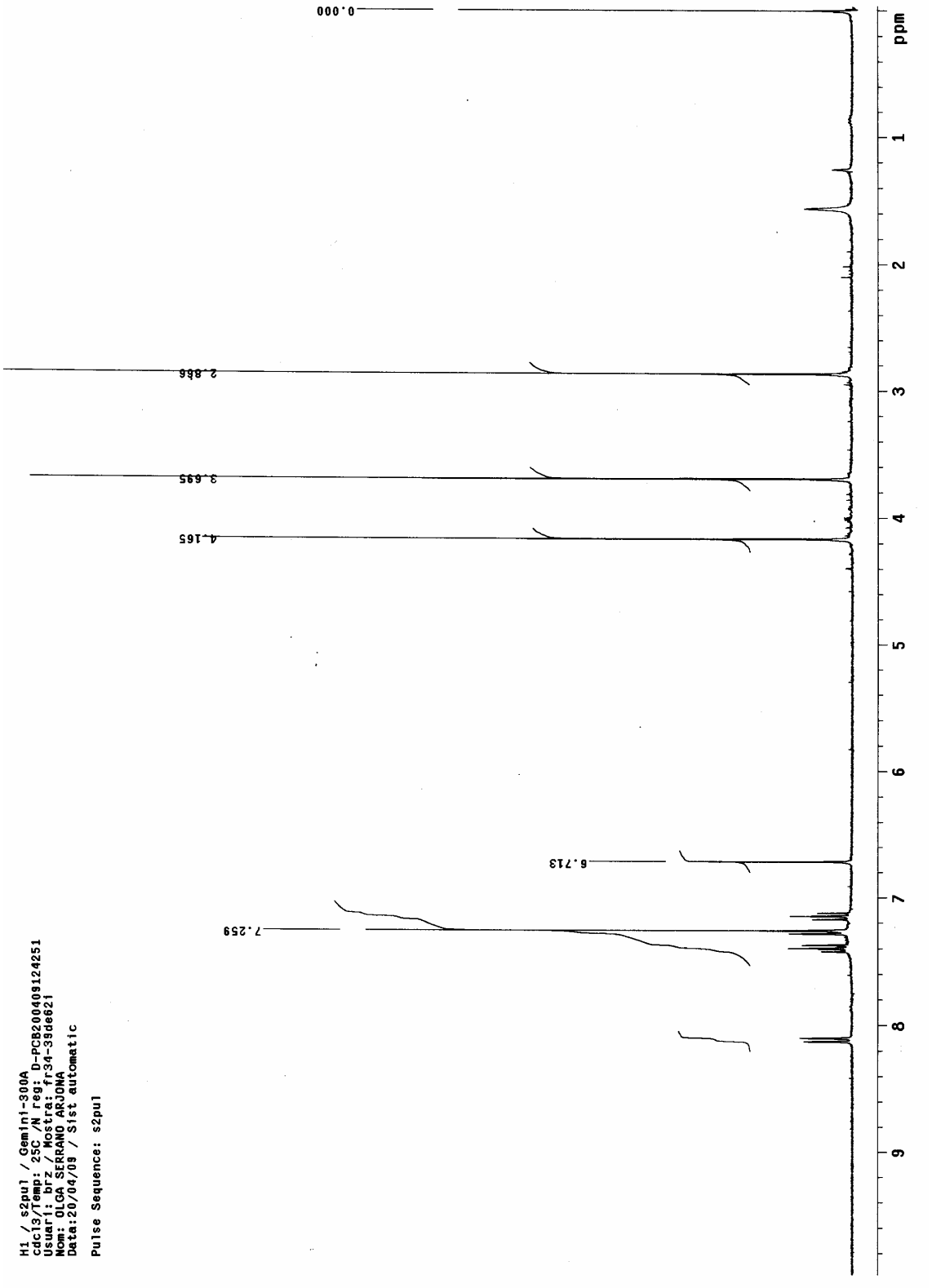

N
CH3
CH3
O
N
CH3

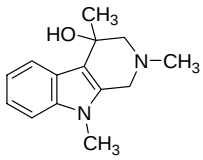
29


ppm
20
40
60
80
100
120
140
160
180
30.530
34.055
39.727
76.743
76.995
77.247
109.384
114.915
119.432
119.600
119.775
120.142
123.903
128.359
128.931
146.449
184.743
Equip: Inova500R
C13 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 4753/2008
Usuari: br2 / Mostra: fr10-21de131ij1
Nom: IVAN JIMENEZ HOYOS
Data: 14/11/08 12:14:35 h./ Ope.: ANA LINARES
Pulse Sequence: s2pul

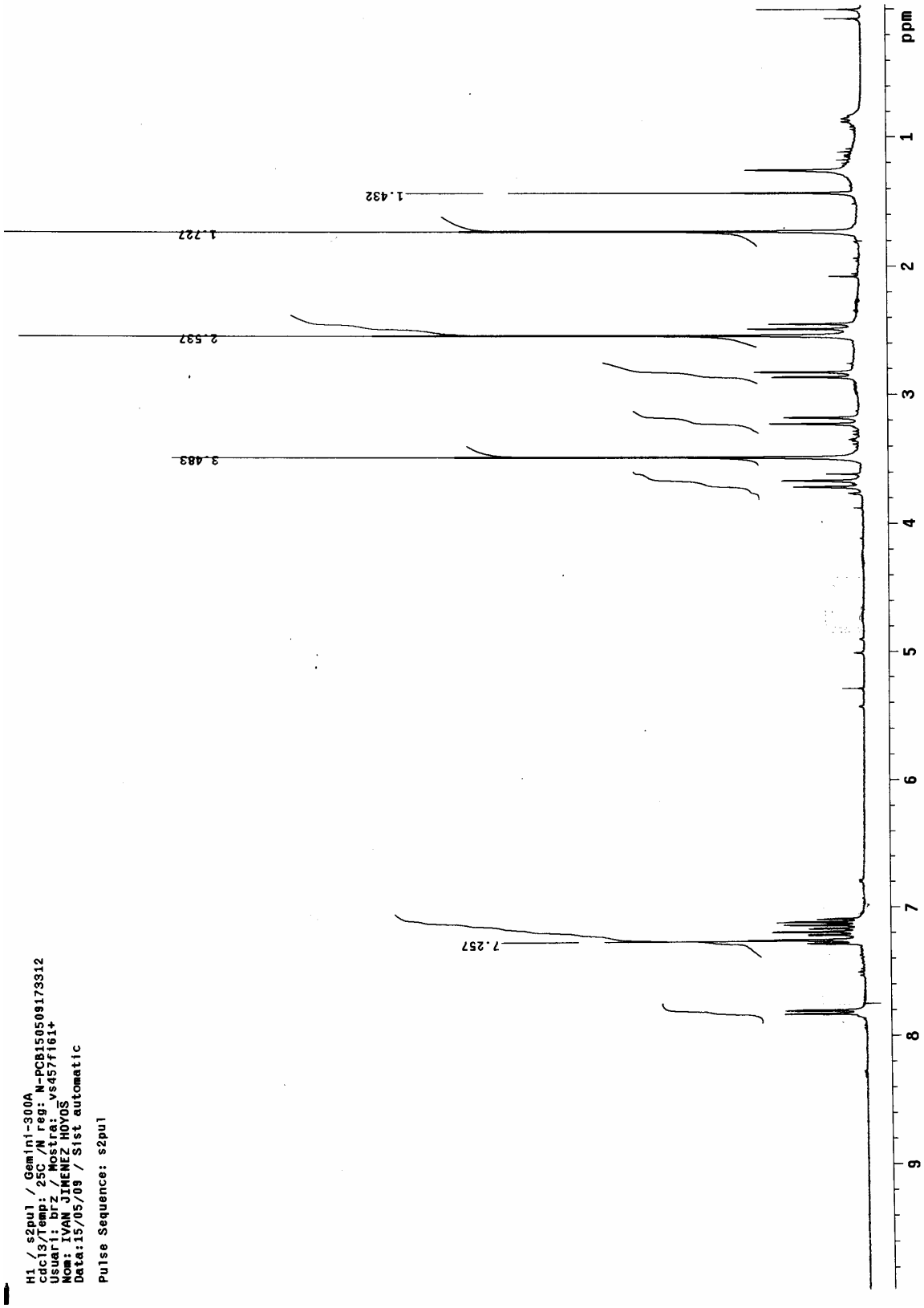

O
CH3
N
CH3
N
CH3

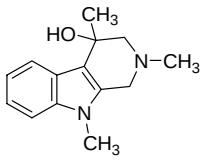
30


0.000
2.886
3.695
4.165
6.713
7.259
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: D-PCB200409124251
Usuari: brz / Mostra: fr34-39de021
Nom: OLGA SERRANO ARJONA
Data:20/04/09 / Sist automatic
Pulse Sequence: s2pul

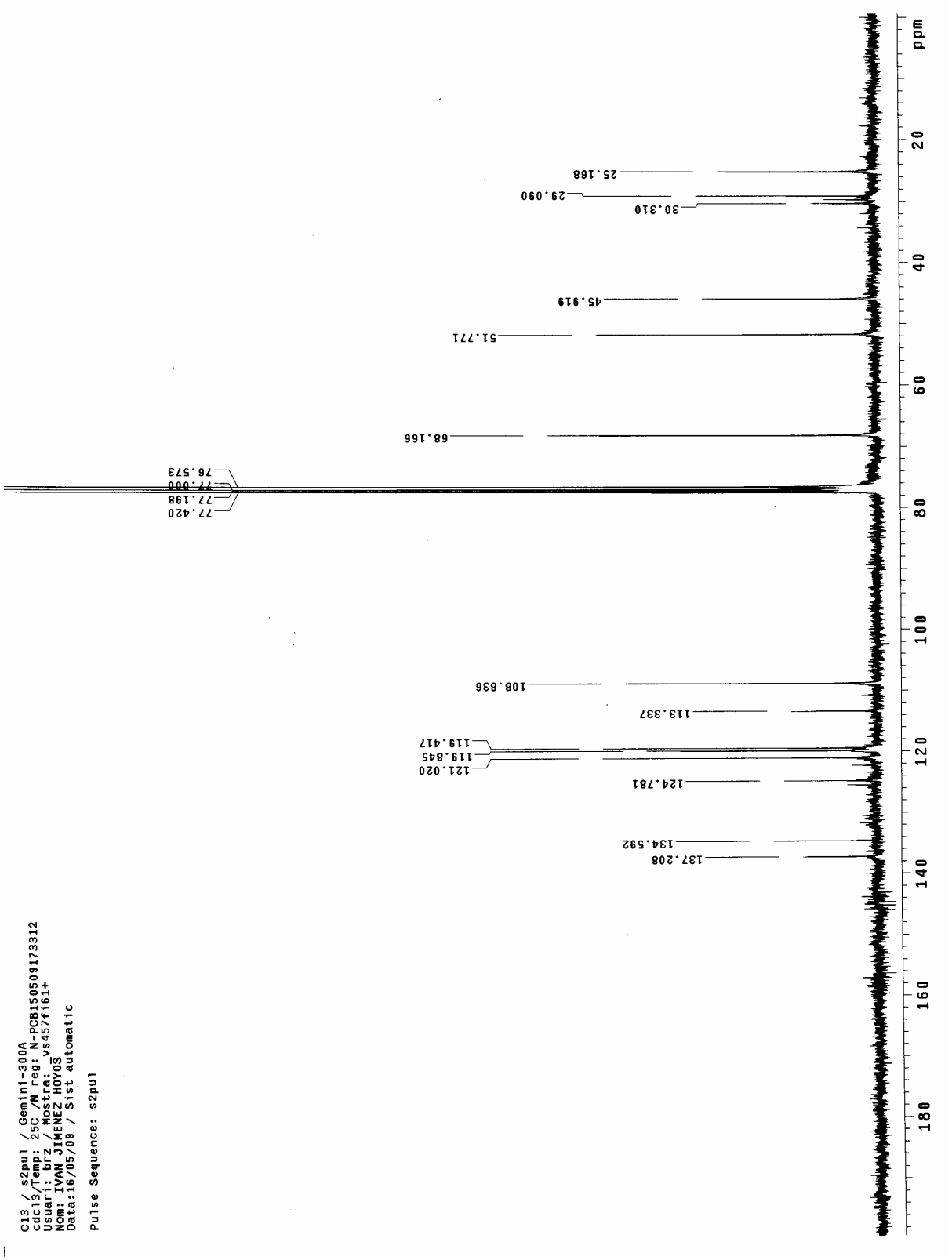

CH3
HO
N−CH3
N
CH3

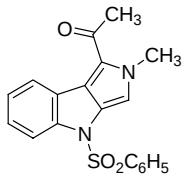
31


1.432
1.727
2.587
3.483
7.257
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB150509173312
Usuari: brz / Mostra: _vs457f161+
Nom: IVAN JIMENEZ HOYOS
Data:15/05/09 / Sist automatic
Pulse Sequence: s2pul

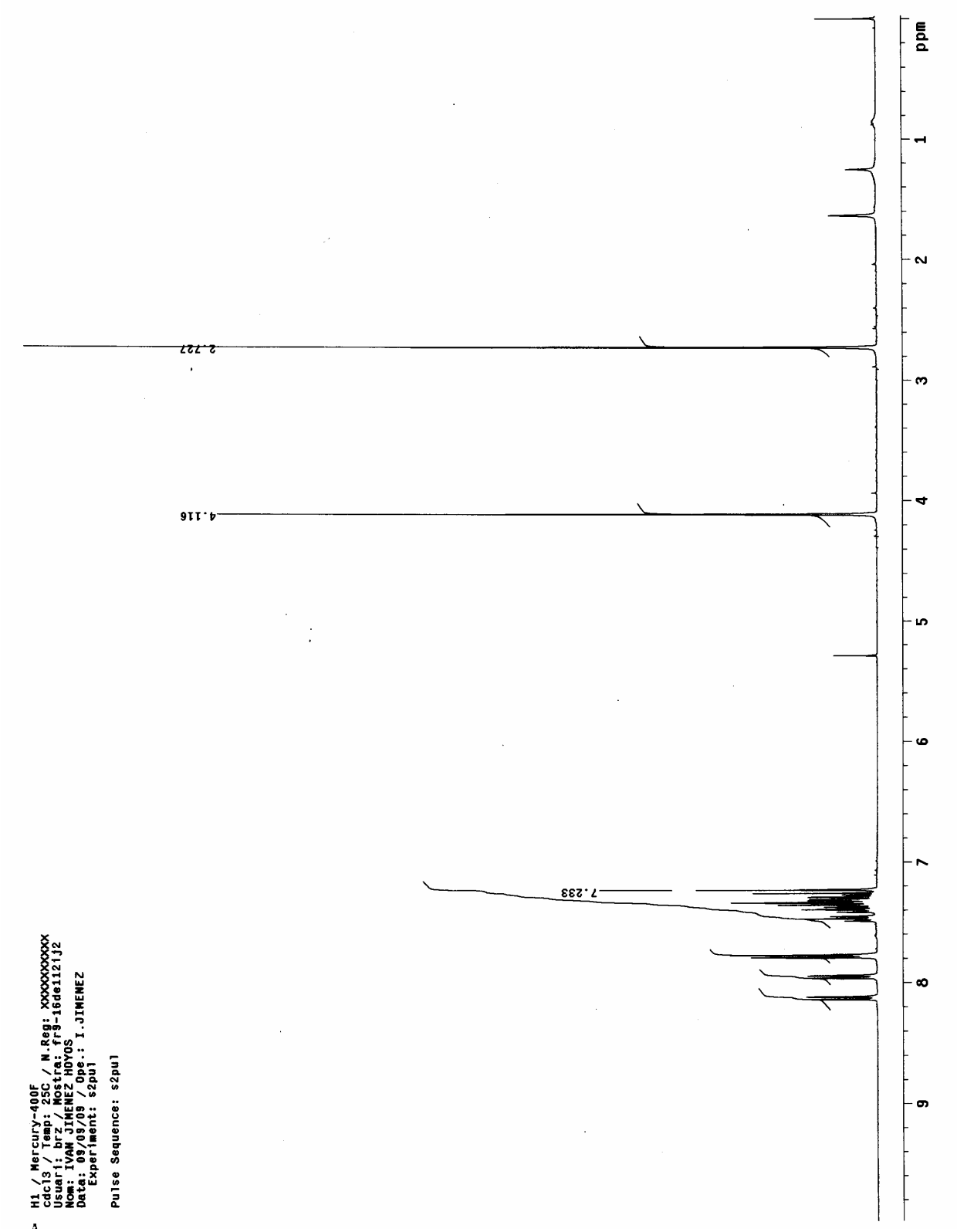

CH3
HO
N−CH3
N
CH3

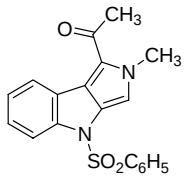
31


ppm
20
40
60
80
100
120
140
160
180
25.168
29.090
30.310
45.919
51.771
68.166
76.573
77.000
77.198
77.420
108.836
113.337
119.417
119.845
121.020
124.781
134.592
137.208
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB150509173312
Usuari: brz /Mostra: vs457fi61+
Nom: IVAN JIMENEZ HOYOS
Data:16/05/09 / Sist automatic
Pulse Sequence: s2pul

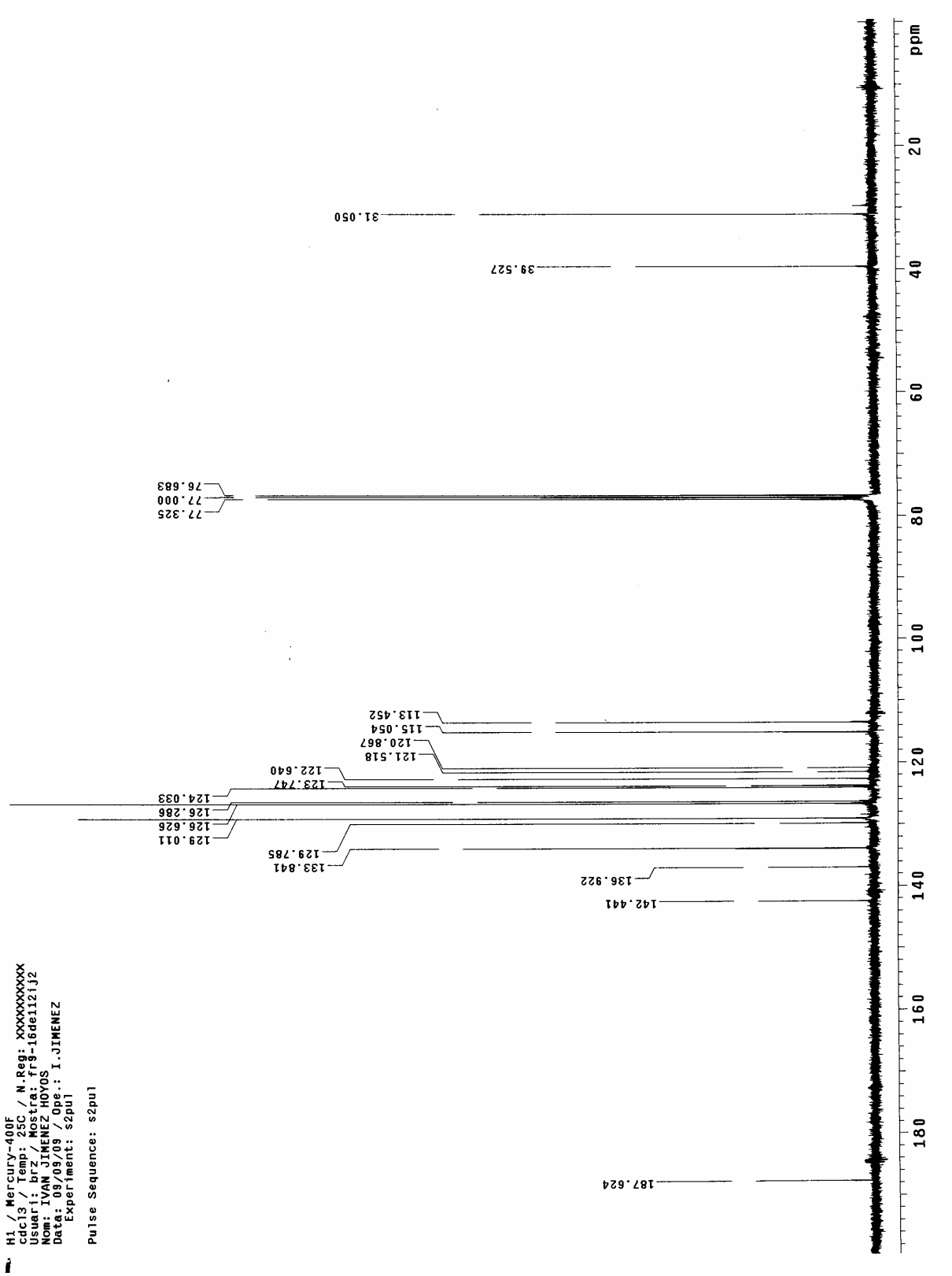

CH3
O
CH3
N
N
SO2C6H5

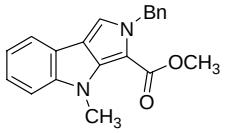
32


2.727
4.116
7.233
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: frg-16de1121j2
Nom: IVAN JIMENEZ HOYOS
Data: 09/09/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

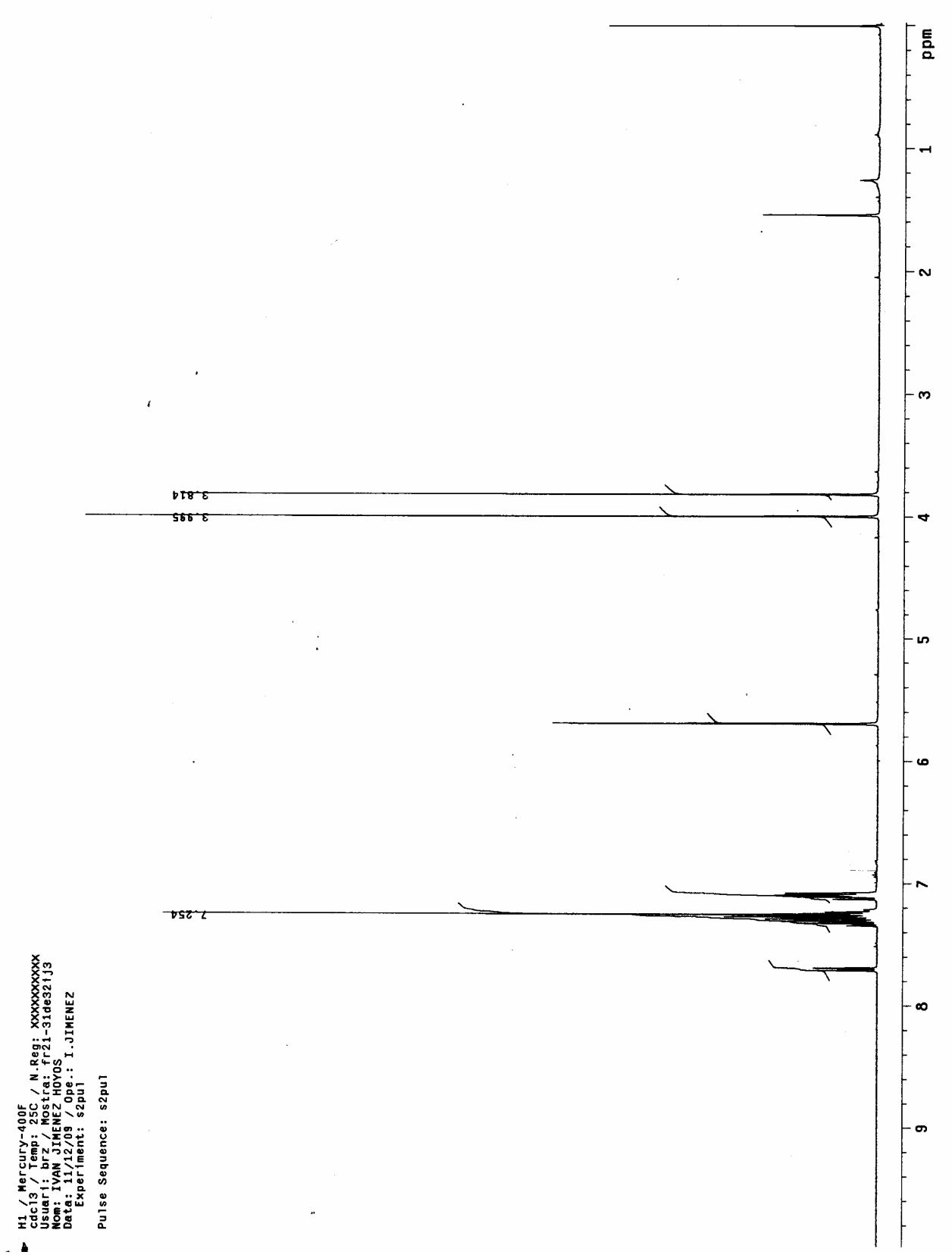

CH3
O
N
CH3
N
SO2C6H5

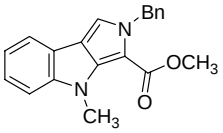
32


31.050
39.527
76.683
77.000
77.325
113.452
115.054
120.867
121.518
122.640
123.747
124.033
126.286
126.626
129.011
129.785
133.841
136.922
142.441
187.624
ppm
20
40
60
80
100
120
140
160
180
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra:frg-16de1121j2
Nom: IVAN JIMENEZ HOYOS
Data: 09/09/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

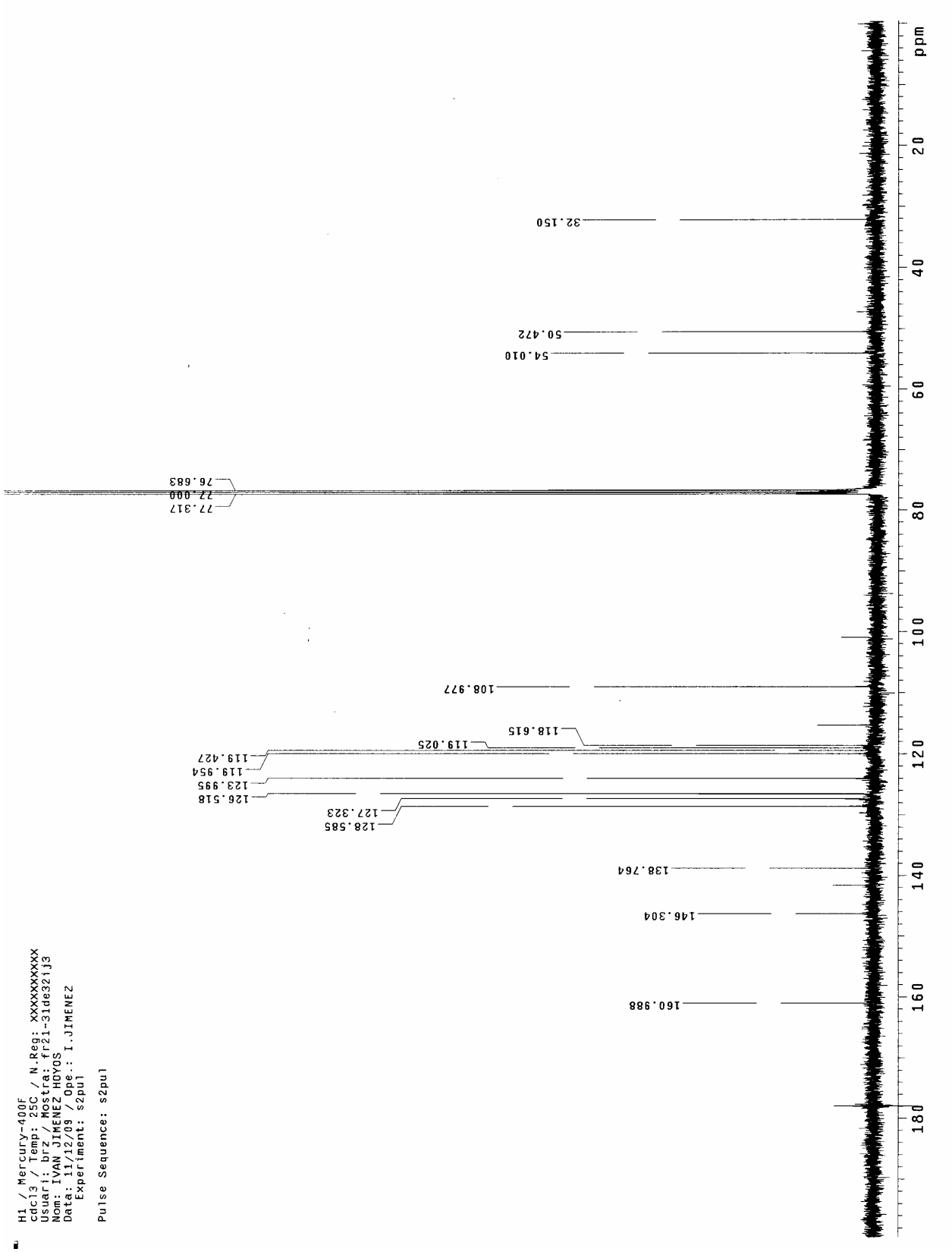

Bn
N
OCH3
O
N
CH3

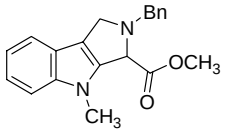
33


3.814
3.995
7.254
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr21-31de32ij3
Nom: IVAN JIMENEZ HOYOS
Data: 11/12/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

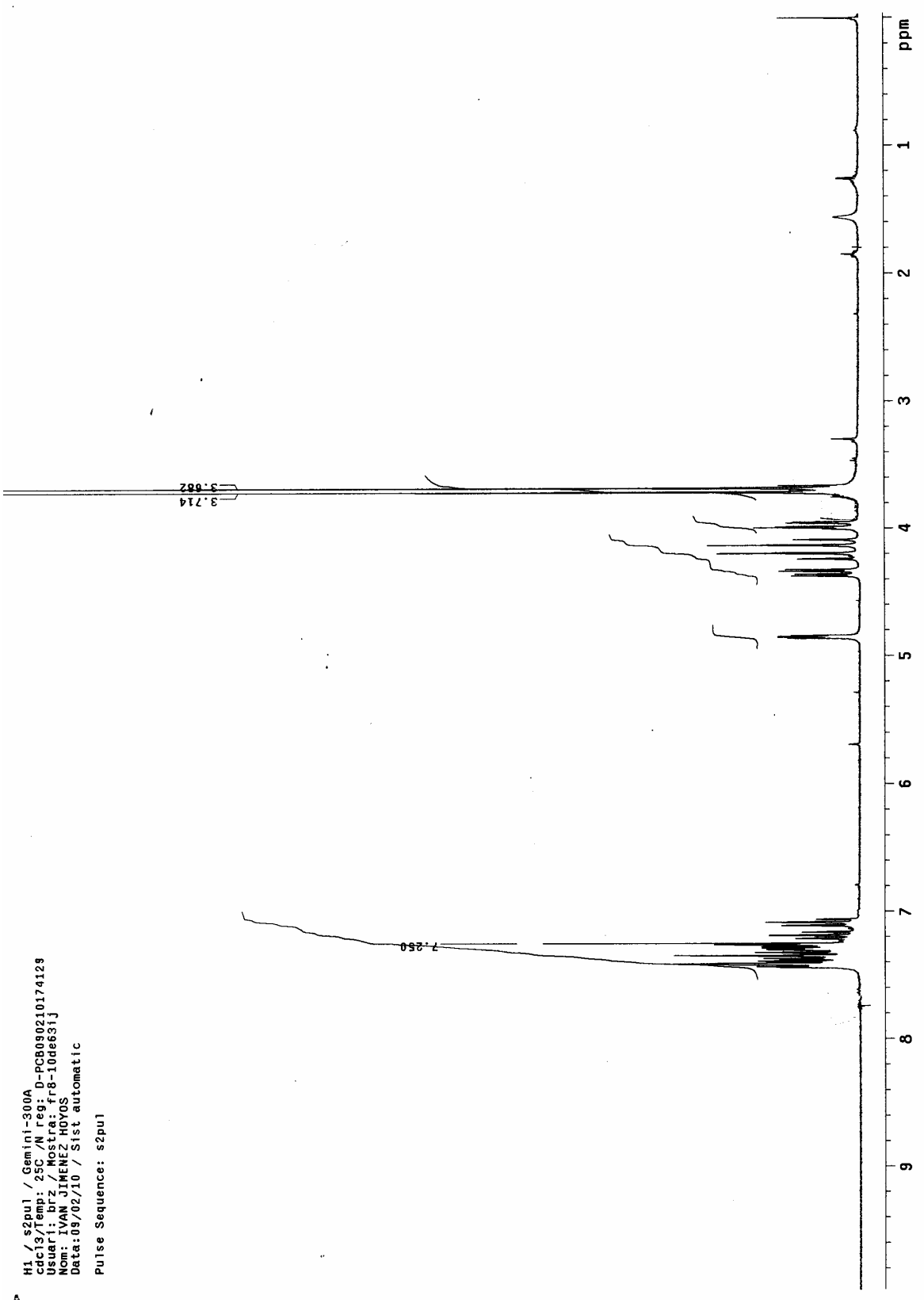

Bn
N
OCH3
O
N
CH3

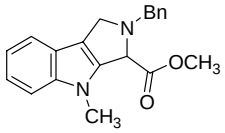
33


32.150
50.472
54.010
76.683
77.000
77.317
108.977
118.615
119.025
119.427
119.954
123.995
126.518
127.323
128.585
138.764
146.304
160.988
ppm
20
40
60
80
100
120
140
160
180
H1 / Mercury-400F
Cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr21-31de321j3
Nom: IVAN JIMENEZ HOYOS
Data: 11/12/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

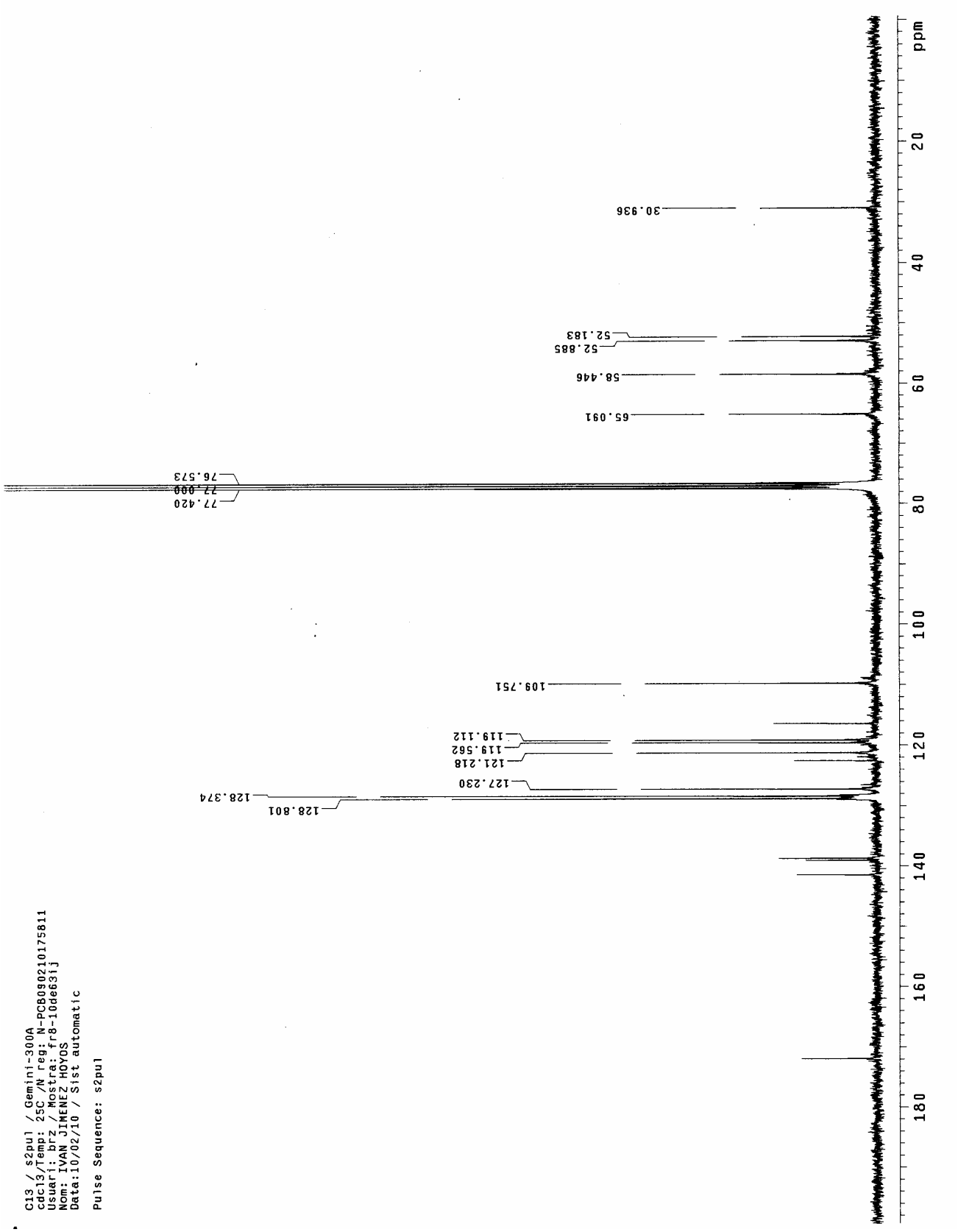

N
Bn
OCH3
O
N
CH3

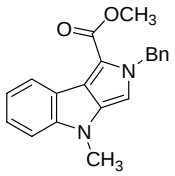
34


3.682
3.714
7.250
ppm
1
2
3
4
5
6
7
8
9
H1 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: D-PCB030210174129
Usuari: brz / Mostra: fr8-10de63ij
Nom: IVAN JIMENEZ HOYOS
Data:09/02/10 / Sist automatic
Pulse Sequence: s2pul

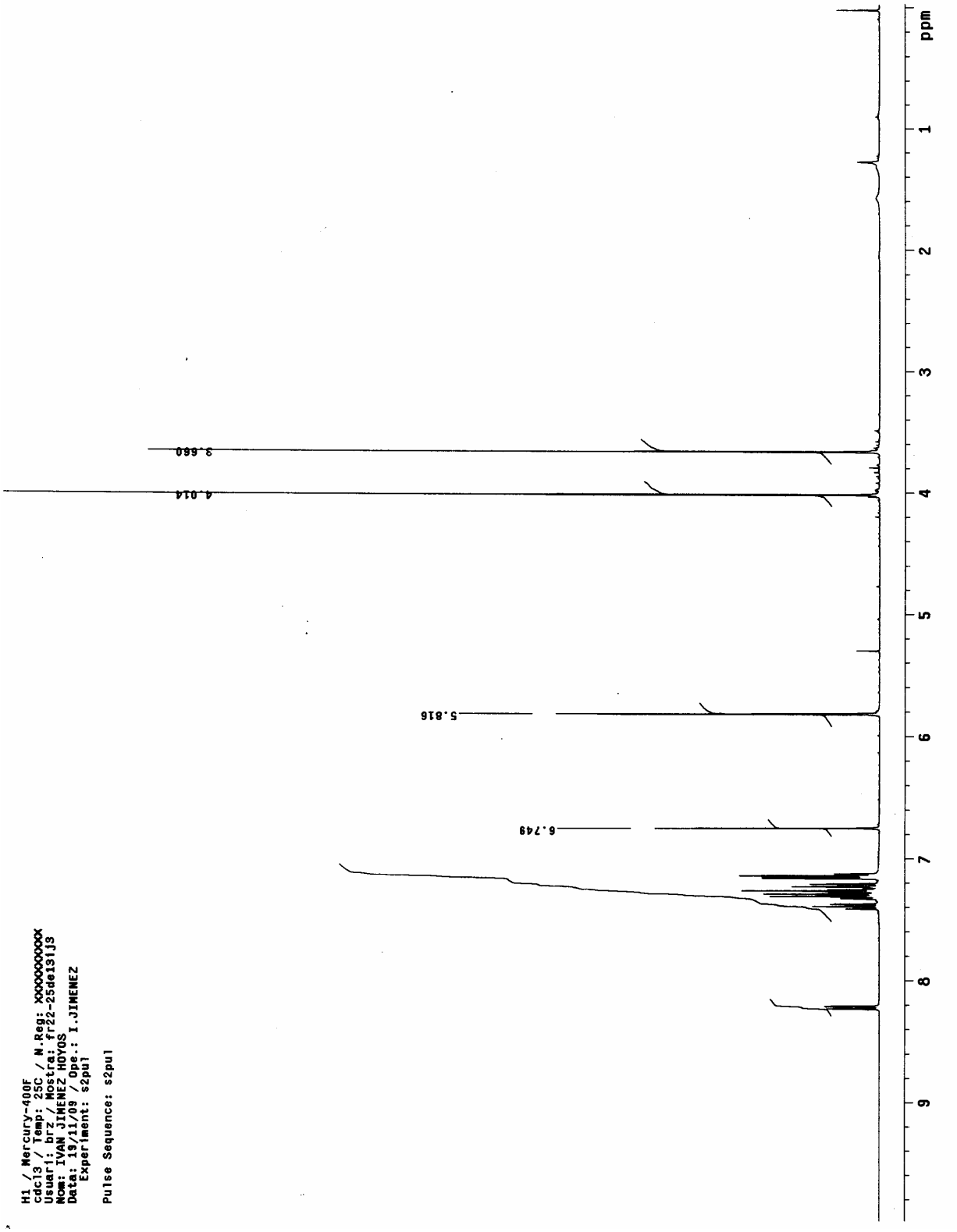

Bn
N
OCH3
O
N
CH3

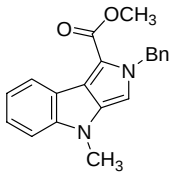
34


30.936
52.183
52.885
58.446
65.091
76.573
77.000
77.420
109.751
119.112
119.562
121.218
127.230
128.374
128.801
ppm
20
40
60
80
100
120
140
160
180
C13 / s2pul / Gemini-300A
cdcl3/Temp: 25C /N reg: N-PCB090210175811
Usuari: brz / Mostra: fr8-10de631j
Nom: IVAN JIMENEZ HOYOS
Data:10/02/10 / Sist automatic
Pulse Sequence: s2pul

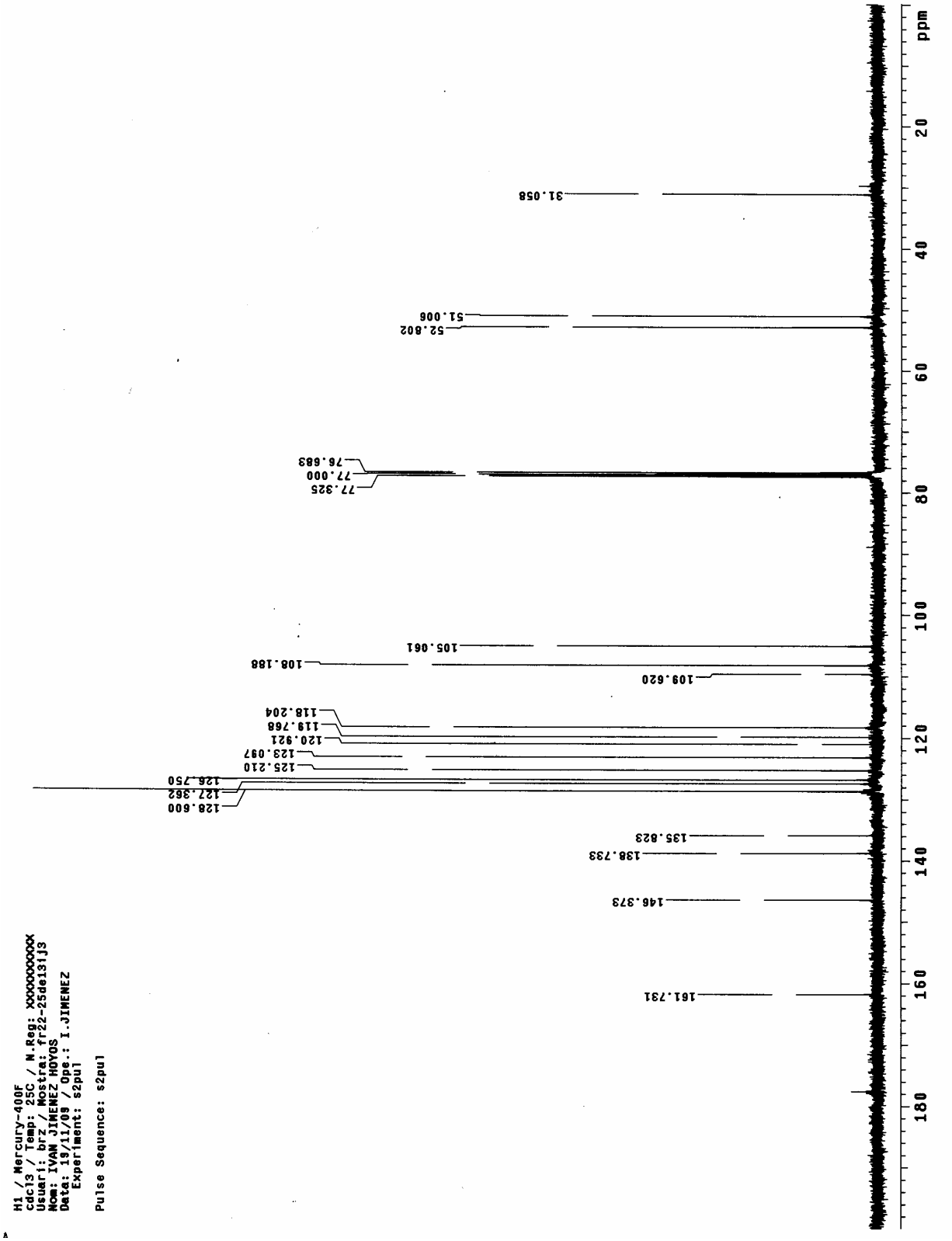

OCH3
O
Bn
N
N
CH3

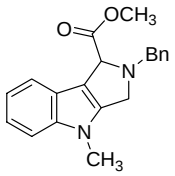
35


3.660
4.014
5.816
6.749
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr22-25de13ij3
Nom: IVAN JIMENEZ HOYOS
Data: 19/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

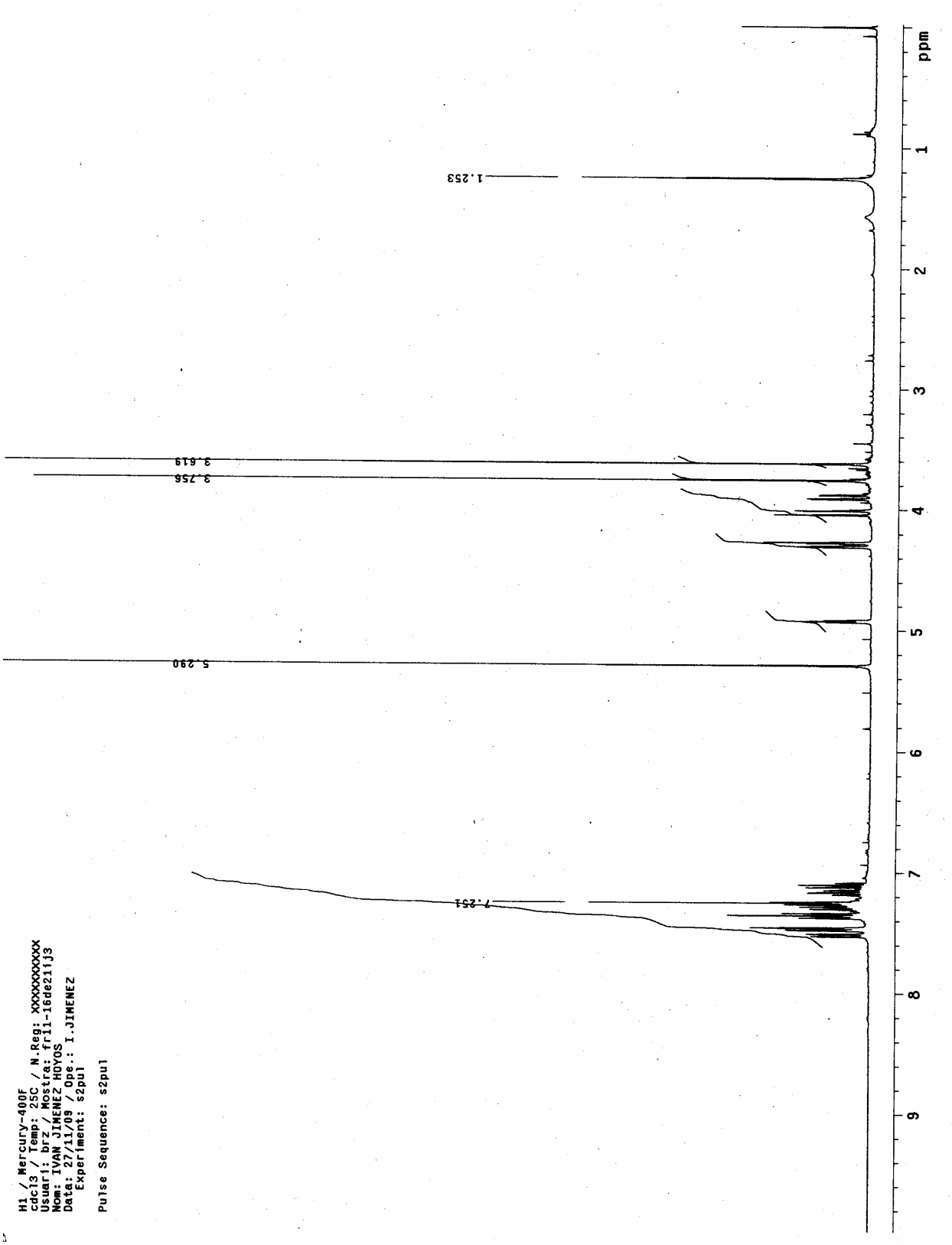

OCH3
O
Bn
N
N
CH3

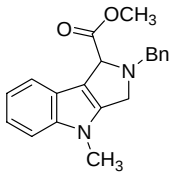
35


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: brz / Mostra: fr22-25de131j3
Nom: IVAN JIMENEZ HOYOS
Date: 19/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul
31.058
51.006
52.802
76.683
77.000
77.325
105.061
108.186
109.620
118.204
118.768
120.921
123.097
125.210
126.750
127.362
128.600
135.823
138.733
146.373
161.731
ppm
20
40
60
80
100
120
140
160
180

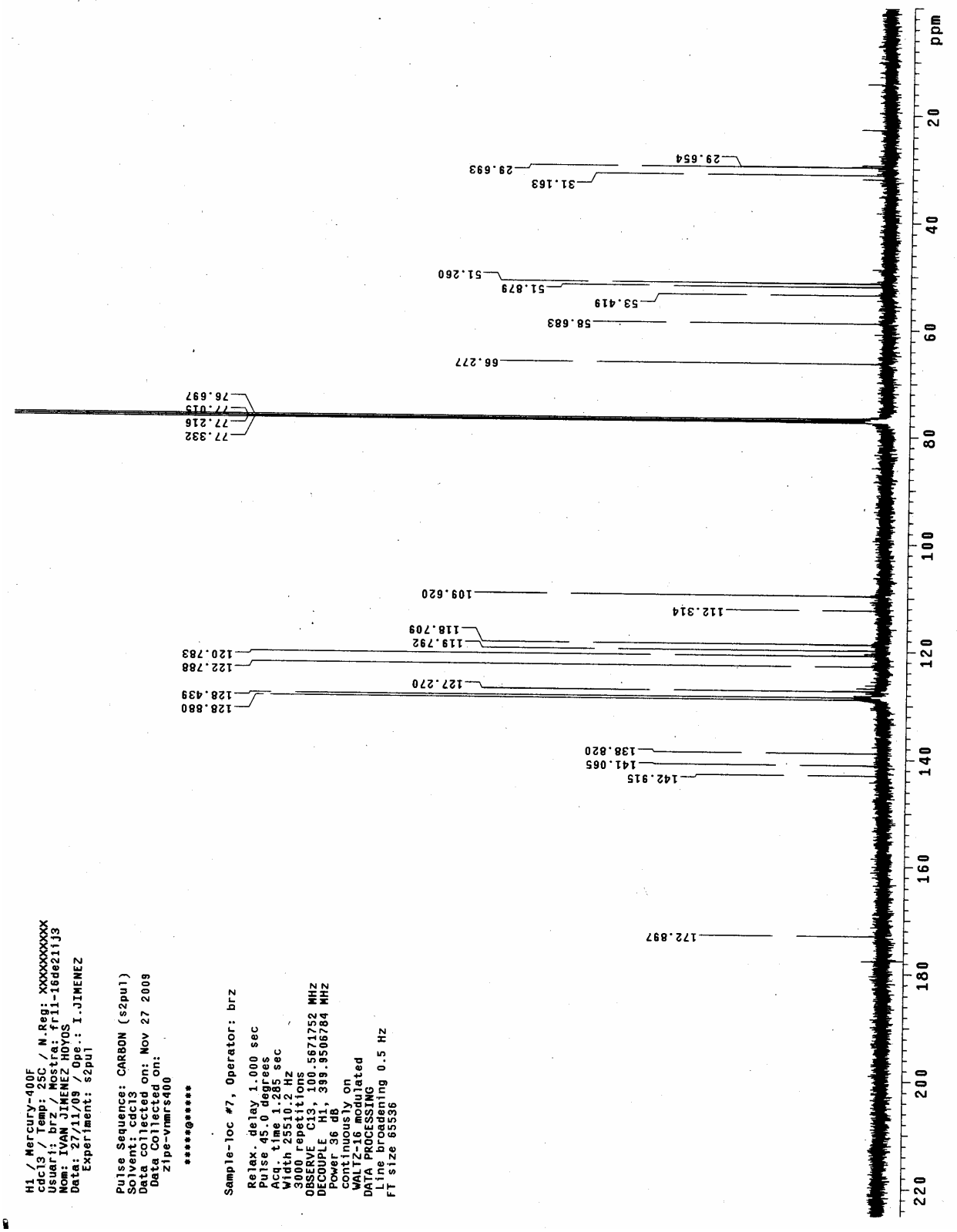

OCH3
O
Bn
N
N
CH3

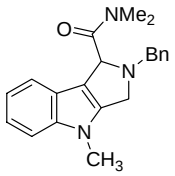
36


1.253
3.619
3.756
5.290
7.251
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: br2 / Mostra: fr11-16de211j3
Nom: IVAN JIMENEZ HOYOS
Data: 27/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul

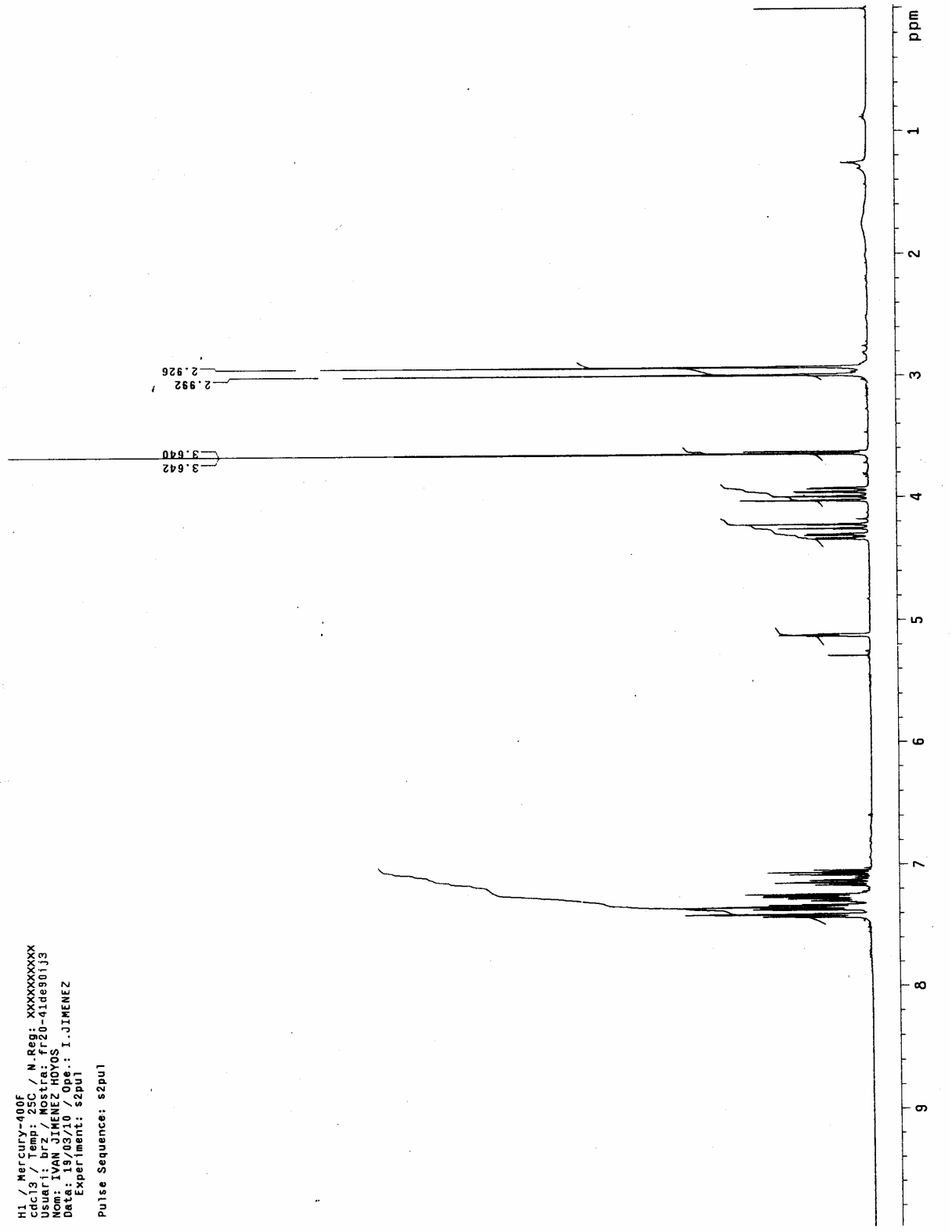

OCH3
O
Bn
N
N
CH3

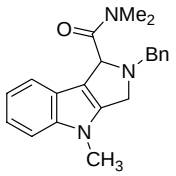
36


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fri1-166e21fj3
Nom: IVAN JIMENEZ HOYOS
Data: 27/11/09 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 27 2009
Data Collected on:
zipe-vmmrs400
*****@******
Sample-loc #7, Operator: brz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
3000 repetitions
OBSERVE C13, 100.5671752 MHz
DECOUPLE H1, 399.9506784 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
29.654
29.693
31.163
51.260
51.879
53.419
58.683
66.277
76.697
77.015
77.216
77.332
109.620
112.314
118.709
119.792
120.783
122.788
127.270
128.439
128.880
138.820
141.065
142.915
172.897
ppm
20
40
60
80
100
120
140
160
180
200
220

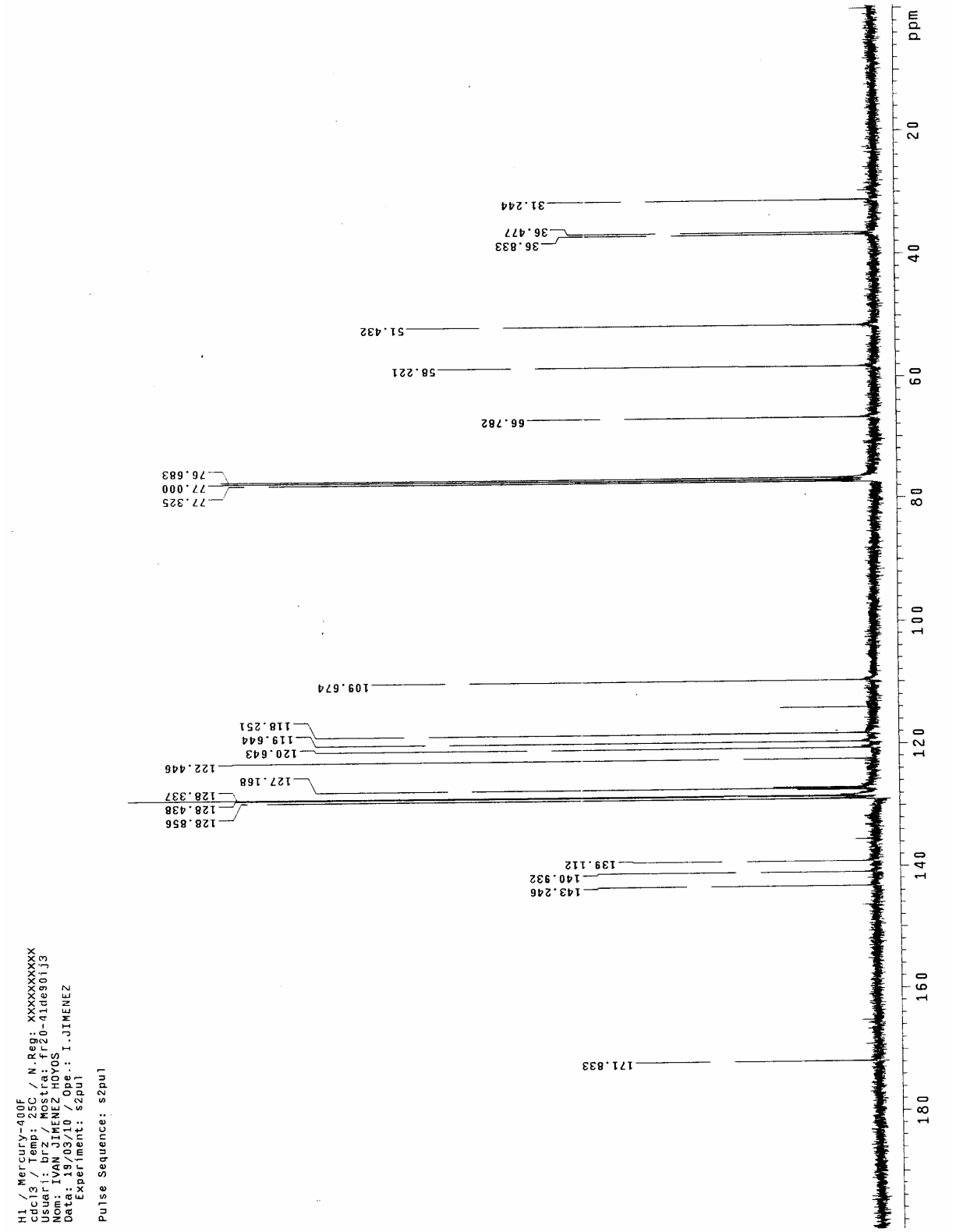

NMe2
O
Bn
N
N
CH3

37


2.926
2.992
3.640
3.642
ppm
1
2
3
4
5
6
7
8
9
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr20-41de90ij3
Nom: IVAN JIMENEZ HOYOS
Date: 19/03/10 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul


NMe2
O
Bn
N
N
CH3

37


31.244
36.477
36.833
51.432
58.221
66.782
76.683
77.000
77.325
109.674
118.251
119.644
120.643
122.446
127.168
128.337
128.438
128.856
139.112
140.932
143.246
171.833
ppm
20
40
60
80
100
120
140
160
180
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: brz / Mostra: fr20-41de90ij3
Nom: IVAN JIMENEZ HOYOS
Data: 19/03/10 / Ope.: I.JIMENEZ
Experiment: s2pul
Pulse Sequence: s2pul