

**NMR evidence of the kinetic and thermodynamic products in
the NIS promoted cyclization of 1-phenyl-4-pentenylamines.
Synthesis and reactivity of *trans*-2-phenyl-5-iodopiperidines.**

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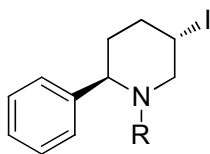
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Table 1. ^{13}C NMR Chemical shifts of piperidines **2**^a



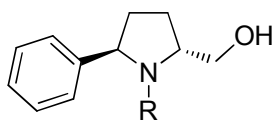
2a R = Me
2b Et
2c Pr
2d allyl
2e Bn
2f *i*Pr
2g *c*Hex

| | 2a | 2b | 2c | 2d | 2e | 2f | 2g |
|--------------|-------------------|-------------------|-------------------|--------------------|-------------------|-------------------|-------------------|
| C-2 | 69.4 | 67.1 | 67.5 | 67.0 | 67.6 | 64.7 | 64.4 |
| C-3 | 38.0 | 38.2 | 38.7 | 38.6 | 38.9 | 39.4 | 39.3 |
| C-4 | 38.7 | 38.7 | 39.0 | 38.9 | 39.0 | 39.4 | 39.3 |
| C-5 | 24.6 | 25.1 | 25.8 | 25.3 | 25.4 | 27.7 | 28.1 |
| C-6 | 66.9 | 62.2 | 63.1 | 63.0 | 62.9 | 55.4 | 57.2 |
| C-Ar | 127.3 | 127.5 | 127.1 | 127.3 | 126.9 | 127.1 | 127.1 |
| | 128.5 | 128.6 | 127.5 | 127.4 | 127.3 | 127.4 | 127.4 |
| | 143.6 | 142.8 | 128.4 | 128.6 | 127.4 | 128.5 | 128.5 |
| | | | 144.0 | 143.6 | 128.2 | 144.0 | 144.0 |
| | | | | | 128.5 | | |
| | | | | | 128.7 | | |
| | | | | | 138.7 | | |
| | | | | | 144.1 | | |
| Other | 43.5 ^b | 10.5 ^b | 11.6 ^b | 57.3 ^c | 58.6 ^c | 12.5 ^b | 24.1 ^c |
| | | 48.2 ^c | 19.0 ^c | 117.9 ^d | | 21.3 ^b | 25.6 ^c |
| | | | 56.1 ^c | 134.4 ^e | | 48.4 ^f | 26.2 ^c |
| | | | | | | | 26.3 ^c |
| | | | | | | | 31.4 ^c |
| | | | | | | | 58.0 ^f |

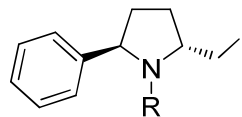
^aValues were assigned on the basis of gCOSY and gHSQC spectra.

^bCH₃, ^cCH₂, ^dCH₂=, ^eCH=, ^fCH.

Table 2. ^{13}C NMR Chemical shifts of pyrrolidines **3** and **4**^a



4a R = Me
4b Et
4c Pr
4d allyl
4e Bn
4h tBu



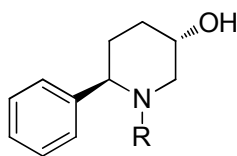
3a R = Me
3h tBu

| | 4a | 4b | 4c | 4d | 4e | 4h | 3a^h | 3h |
|------------------------------------|-------------------|-------------------|-------------------|--------------------|-------------------|-------------------|-----------------------|-------------------|
| C-2 | 63.7 | 62.6 | 62.8 | 62.4 | 62.3 | 59.9 | 65.3 | 62.2 |
| C-3 | 27.3 | 27.2 | 27.1 | 27.1 | 27.0 | 29.3 | 31.2 | 29.9 |
| C-4 | 31.9 | 32.5 | 32.6 | 32.4 | 32.6 | 37.0 | 33.7 | 36.8 |
| C-5 | 68.8 | 65.6 | 65.8 | 66.4 | 66.2 | 64.6 | 67.8 | 65.6 |
| CH₂X^b | 61.6 | 62.1 | 62.1 | 62.4 | 62.4 | 65.1 | 11.4 | 14.6 |
| C-Ar | 127.0 | 126.8 | 126.7 | 126.9 | 126.8 | 126.1 | 127.8 | 125.9 |
| | 127.9 | 127.7 | 127.7 | 127.9 | 126.9 | 128.3 | 128.2 | 126.1 |
| | 128.3 | 128.3 | 128.3 | 128.2 | 127.9 | 149.0 | 129.3 | 128.1 |
| | 142.6 | 143.3 | 143.5 | 143.2 | 128.2 | | 145.1 | 148.8 |
| | | | | 128.3 | | | | |
| | | | | 139.9 | | | | |
| | | | | 143.3 | | | | |
| Other | 35.1 ^c | 14.3 ^c | 11.8 ^c | 50.5 ^d | 51.6 ^d | 28.0 ^c | 34.7 ^c | 27.7 ^c |
| | | 41.2 ^d | 21.9 ^d | 116.1 ^e | | 55.4 ^g | | 54.9 ^g |
| | | | 49.0 ^d | 136.7 ^f | | | | |

^aValues were assigned on the basis of gCOSY and gHSQC spectra.

^bX = OH or I, ^cCH₃, ^dCH₂, ^eCH₂=, ^fCH=, ^gC, ^hSpectrum registered in C₆D₆.

Table 3. ^{13}C NMR Chemical shifts of piperidines **5**^a



5a R = Me
5b Et
5c Pr
5d allyl
5e Bn
5f *i*Pr
5g *c*Hex

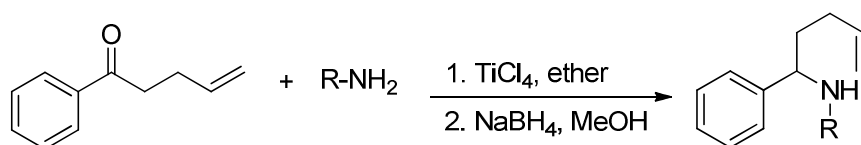
| | 5a | 5b | 5c | 5d | 5e | 5f | 5g |
|--------------|-------------------|-------------------|-------------------|--------------------|-------------------|-------------------|-------------------|
| C-2 | 64.3 | 59.5 | 60.0 | 60.1 | 60.1 | 51.8 | 53.8 |
| C-3 | 67.7 | 68.1 | 67.8 | 67.9 | 67.9 | 68.5 | 68.7 |
| C-4 | 34.3 | 34.5 | 34.4 | 34.4 | 34.3 | 34.7 | 34.7 |
| C-5 | 33.9 | 34.4 | 34.4 | 34.4 | 34.7 | 34.7 | 34.8 |
| C-6 | 69.8 | 67.3 | 67.8 | 67.4 | 67.9 | 64.9 | 64.4 |
| C-Ar | 127.2 | 127.0 | 127.0 | 127.2 | 126.8 | 127.0 | 126.9 |
| | 127.5 | 127.5 | 127.6 | 127.5 | 127.1 | 127.5 | 127.5 |
| | 128.5 | 128.4 | 128.4 | 128.5 | 127.5 | 128.5 | 128.4 |
| | 143.6 | 144.1 | 144.0 | 143.8 | 128.1 | 143.9 | 144.2 |
| | | | | | 128.6 | | |
| | | | | | 128.7 | | |
| | | | | | 139.1 | | |
| | | | | | 144.4 | | |
| Other | 44.2 ^b | 10.7 ^b | 11.6 ^b | 58.0 ^c | 59.3 ^c | 12.2 ^b | 23.7 ^c |
| | | 48.6 ^c | 18.8 ^c | 117.7 ^d | | 21.1 ^b | 25.7 ^c |
| | | | 56.6 ^c | 134.7 ^e | | 48.4 ^f | 26.4 ^c |
| | | | | | | | 26.5 ^c |
| | | | | | | | 31.5 ^c |
| | | | | | | | 57.8 ^f |

^aValues were assigned on the basis of gCOSY and gHSQC spectra.

^bCH₃, ^cCH₂, ^dCH₂=, ^eCH=, ^fCH.

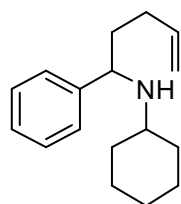
General procedures. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 or C_6D_6 solution. Chemical shifts are reported as δ values (ppm) relative to internal Me_4Si . Infrared spectra were recorded on a Nicolet 320 FT-IR spectrophotometer. TLC was performed on SiO_2 (silica gel 60 F₂₅₄, Merck) or on Al_2O_3 (aluminium oxide 60 F254, Merck). The spots were located by UV light, a 1% KMnO_4 aqueous solution or a 1.5% K_2PtCl_6 aqueous solution. Chromatography refers to flash chromatography and was achieved on SiO_2 (silica gel 60, SDS, 230–400 mesh) or on Al_2O_3 (aluminium oxide activity II-III, 70-230 mesh). All reactions were carried out under an argon atmosphere with dry, freshly distilled solvents and under anhydrous conditions. Drying of the organic extracts during the work-up of reactions was performed over anhydrous Na_2SO_4 .

I. Synthesis of 1-phenylpent-4-enamines (1c-h)¹



Representative example, preparation of 1g: A solution of 1-phenylpent-4-en-1-one² (0.3 g, 1.87 mmol) and cyclohexylamine (0.85 ml, 7.48 mmol) in 10 mL of dry diethylether was cooled to 0°, and TiCl_4 (0.12 mL, 1.12 mmol) was added dropwise. The mixture was stirred overnight allowing the temperature to rise to rt then it was quenched with aqueous 0.5 M NaOH solution and extracted with ether. The combined organic layers were dried and concentrated. The residue was dissolved in MeOH (10 mL), cooled to 0° and then NaBH_4 (0.088 g, 2.24 mmol) was added portionwise. The mixture was stirred at rt for 1h then the solvent was removed under vacuum, water was added and the aqueous extracted with CH_2Cl_2 . The organic layers were dried, concentrated and purified by chromatography (SiO_2 , CH_2Cl_2 - CH_2Cl_2 /MeOH 95:5) to yield **1g** (0.35 g, 78%) as a colourless oil.

N-Cyclohexyl-1-phenylpent-4-en-1-amine (1g)

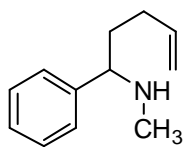


IR (NaCl, neat): 3321, 3062, 3024, 2926, 2851, 1640, 1492, 1450, 1366, 1124, 993, 909, 759, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.90-1.20 (m, 5H), 1.53 (brs, 1H), 1.60-1.84 (m, 5H), 1.96 (m, 3H), 2.23 (tt, 1H, $J = 10, 3.6$ Hz), 3.76 (t, 1H, $J = 6.8$ Hz), 4.95 (m, 2H, = CH_2), 5.78 (ddt, 1H, $J = 16.8, 10.4, 6.4$ Hz, =CH), 7.20-7.35 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 24.8 (CH_2), 25.2 (CH_2), 26.2 (CH_2), 30.6 (CH_2), 33.0 (CH_2), 34.7 (CH_2), 37.7 (CH_2), 53.5 (CH), 59.0 (CH), 114.6 (=CH₂), 126.7, 127.1, 128.3 (Ar-CH), 138.4 (=CH), 144.8 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{17}\text{H}_{26}\text{N}$ 244.2059 [$\text{M}+\text{H}$]⁺, found 244.2064.

¹ G. Verniest, E. Van Hende, R. Surmont and N. De Kimpe, *Org. Lett.*, 2006, **8**, 4767. For the preparation of the imine in the synthesis of **1a** and **1b** see: J. S. M. Samec, A. H. Ell, J. B. Aaberg, T. Privalov, L. Eriksson, J.-E. Bäckvall, *J. Am. Chem. Soc.*, 2006, **128**, 14293.

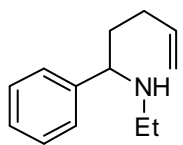
² D. V. Gribkov, K. C. Hultzsich, F. Hampel, *J. Am. Chem. Soc.*, 2006, **128**, 3748.

N-Methyl-1-phenylpent-4-en-1-amine (1a)



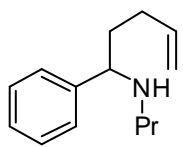
Yield: 47%; IR (NaCl, neat): 3330, 3077, 3063, 3025, 2973, 2933, 2848, 2788, 1640, 1492, 1475, 1450, 1354, 1133, 994, 911, 760, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.65-2.05 (m, 5H), 2.27 (s, 3H, CH_3), 3.47 (dd, 1H, $J = 8, 6$ Hz), 4.95 (m, 2H, $=\text{CH}_2$), 5.78 (ddt, 1H, $J = 16.8, 10, 6.8$ Hz, $=\text{CH}$), 7.22-7.36 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 30.5 (CH_2), 34.4 (CH_3), 36.8 (CH_2), 64.9 (CH), 114.7 ($=\text{CH}_2$), 127.0, 127.3, 128.4 (Ar-CH), 138.3 ($=\text{CH}$), 143.5 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{12}\text{H}_{18}\text{N}$ 176.1434 $[\text{M}+\text{H}]^+$, found 176.1432.

N-Ethyl-1-phenylpent-4-en-1-amine (1b)



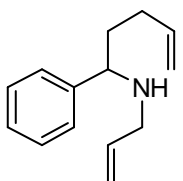
Yield: 30%; IR (NaCl, neat): 3321, 3077, 3063, 3025, 2967, 2930, 2870, 2847, 1640, 1492, 1452, 1379, 1127, 993, 910, 760, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.05 (t, 3H, $J = 7.2$ Hz, CH_3), 1.50 (brs, 1H, NH), 1.65-2.05 (m, 4H), 2.47 (m, 2H, CH_2), 3.60 (dd, 1H, $J = 8, 5.6$ Hz), 4.96 (m, 2H, $=\text{CH}_2$), 5.78 (ddt, 1H, $J = 16.8, 10, 6.8$ Hz, $=\text{CH}$), 7.21-7.35 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 15.4 (CH_3), 30.5 (CH_2), 37.1 (CH_2), 41.9 (CH_2), 62.8 (CH), 114.6 ($=\text{CH}_2$), 126.9, 127.3, 128.3 (Ar-CH), 138.4 ($=\text{CH}$), 144.1 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{13}\text{H}_{20}\text{N}$ 190.1590 $[\text{M}+\text{H}]^+$, found 190.1591.

1-Phenyl-N-propylpent-4-en-1-amine (1c)



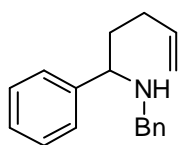
Yield: 75%; IR (NaCl, neat): 3327, 3079, 3063, 3025, 2958, 2930, 2872, 2802, 1640, 1492, 1453, 1379, 1357, 1125, 993, 910, 759, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.86 (t, 3H, $J = 7.6$ Hz, CH_3), 1.45 (m, 2H), 1.73 (m, 1H), 1.82 (m, 1H), 1.96 (m, 2H), 2.39 (m, 2H), 3.58 (dd, 1H, $J = 8.0, 6.4$ Hz), 4.96 (m, 2H, $=\text{CH}_2$), 5.78 (ddt, 1H, $J = 16.8, 10.4, 6.4$ Hz, $=\text{CH}$), 7.20-7.36 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 11.8 (CH_3), 23.3 (CH_2), 30.5 (CH_2), 37.2 (CH_2), 49.6 (CH_2), 62.9 (CH), 114.6 ($=\text{CH}_2$), 126.9, 127.3, 128.3 (Ar-CH), 138.4 ($=\text{CH}$), 144.2 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{22}\text{N}$ 204.1746 $[\text{M}+\text{H}]^+$, found 204.1746.

N-Allyl-1-phenylpent-4-en-1-amine (1d)



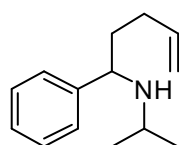
Yield: 72%; IR (NaCl, neat): 3328, 3077, 3025, 2977, 2924, 2844, 1640, 1492, 1452, 1357, 1114, 993, 913, 760, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.50 (brs, 1H, NH), 1.67-1.88 (m, 2H), 1.97 (m, 2H), 3.05 (m, 2H), 3.63 (dd, 1H, $J = 8, 6.4$ Hz), 4.90-5.15 (m, 4H, $=\text{CH}_2$), 5.72-5.92 (m, 2H, $=\text{CH}$), 7.21-7.35 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 30.5 (CH_2), 37.1 (CH_2), 50.1 (CH_2), 62.0 (CH), 114.7 ($=\text{CH}_2$), 115.7 ($=\text{CH}_2$), 127.0, 127.3, 128.4 (Ar-CH), 137.0 ($=\text{CH}$), 138.3 ($=\text{CH}$), 143.8 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{20}\text{N}$ 202.1590 $[\text{M}+\text{H}]^+$, found 202.1589.

***N*-Benzyl-1-phenylpent-4-en-1-amine (1e)**



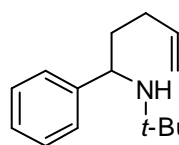
Yield: 71%; IR (NaCl, neat): 3326, 3062, 3026, 2975, 2924, 2844, 1639, 1601, 1492, 1452, 1115, 1026, 994, 910, 744, 699 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.60 (brs, 1H, NH), 1.68-1.88 (m, 2H), 1.98 (m, 2H), 3.53 (d, 1H, $J = 13.2$ Hz), 3.63 (t, 1H, $J = 6.4$ Hz), 3.64 (d, 1H, $J = 13.2$ Hz), 4.94 (m, 2H, =CH₂), 5.76 (ddt, 1H, $J = 16.8, 10.4, 6.4$ Hz, =CH), 7.20-7.38 (m, 10H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 30.5 (CH₂), 37.3 (CH₂), 51.5 (CH₂), 62.0 (CH), 114.6 (=CH₂), 126.8, 127.0, 127.4, 128.2, 128.3, 128.4 (Ar-CH), 138.4 (=CH), 140.6 (*ipso*-C), 144.0 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₈H₂₂N 252.1746 [M+H]⁺, found 252.1749.

***N*-Isopropyl-1-phenylpent-4-en-1-amine (1f)**



Yield: 71%; IR (NaCl, neat): 3324, 3077, 3064, 3025, 2962, 2930, 2862, 1640, 1491, 1469, 1451, 1378, 1366, 1171, 1124, 994, 910, 760, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.96 (d, 3H, $J = 6.4$ Hz, CH₃), 1.00 (d, 3H, $J = 6.4$ Hz, CH₃), 1.28 (brs, 1H, NH), 1.69 (m, 1H), 1.79 (m, 1H), 1.95 (m, 2H), 2.58 (sept, 1H, $J = 6.4$ Hz), 3.69 (t, 1H, $J = 7.2$ Hz), 4.95 (m, 2H, =CH₂), 5.78 (ddt, 1H, $J = 16.8, 10.0, 6.8$ Hz, =CH), 7.20-7.35 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.0 (CH₃), 24.2 (CH₃), 30.6 (CH₂), 37.6 (CH₂), 45.4 (CH), 59.6 (CH), 114.6 (=CH₂), 126.8, 127.1, 128.3 (Ar-CH), 138.4 (=CH), 144.6 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₄H₂₂N 204.1746 [M+H]⁺, found 204.1747.

***N*-(*tert*-Butyl)-1-phenylpent-4-en-1-amine (1h)**

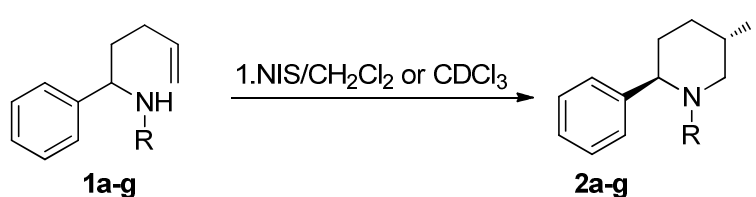


Yield: 64%; IR (NaCl, neat): 3341, 3077, 3024, 2962, 2929, 2864, 1640, 1480, 1452, 1388, 1363, 1228, 993, 910, 758, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.99 (s, 9H, CH₃), 1.17 (brs, 1H, NH), 1.68 (m, 2H), 1.84-2.04 (m, 2H), 3.72 (t, 1H, $J = 6.8$ Hz), 4.96 (m, 2H, =CH₂), 5.78 (ddt, 1H, $J = 16.8, 10.0, 6.8$ Hz, =CH), 7.16-7.34 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 30.2 (CH₃), 30.9 (CH₂), 39.6 (CH₂), 51.3 (C), 57.0 (CH), 114.6 (=CH₂), 126.4, 127.0, 128.1 (Ar-CH), 138.5 (=CH), 147.7 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₅H₂₄N 218.1903 [M+H]⁺, found 218.1903.

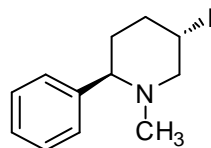
II. Iodoaminocyclization of alkenylamines 1a-h³

General procedure: To a solution of alkenylamine **1a-h** (0.28 mmol) in CH₂Cl₂ (3 mL) or CDCl₃ (1 mL) was added N-iodosuccinimide (0.28 mmol) and the mixture was stirred at room temperature for 10-15 min. The mixture was then purified on a short silicagel pad (CH₂Cl₂) to yield iododerivatives **2a-g** from **1a-g** and **3h** from **1h**.

³ Iodocyclizations using CDCl₃ or C₆D₆ as solvent were carried out in an NMR tube.

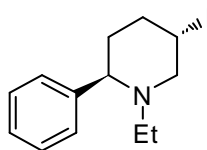


(2*RS*,5*SR*)-5-Iodo-1-methyl-1-phenylpiperidine (2a)



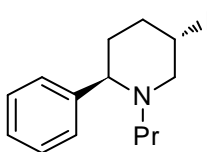
Yield: 75%; IR (NaCl, neat): 3027, 2989, 2938, 2840, 2781, 1490, 1452, 1195, 1153, 1118, 1079, 1044, 1003, 980, 952, 890, 779, 759, 700, 537 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.62-1.76 (m, 2H, CH_2 -3), 1.99 (s, 3H, CH_3), 2.01 (m, 1H, H-4ax), 2.48 (m, 1H, H-4eq), 2.59 (t, 1H, $J = 11.2$ Hz, H-6ax), 2.88 (dd, 1H, $J = 10, 4.4$, H-2), 3.43 (ddd, 1H, $J = 11.2, 4, 2$ Hz, H-6eq), 4.33 (tt, 1H, $J = 12, 4$ Hz, H-5), 7.22-7.34 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 24.6 (C-5), 38.0 (C-3), 38.7 (C-4), 43.5 (CH_3), 66.9 (C-6), 69.4 (C-2), 127.3, 128.5 (Ar-CH), 143.6 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{12}\text{H}_{17}\text{IN}$ 302.0400 $[\text{M}+\text{H}]^+$, found 302.0400.

(2*RS*,5*SR*)-1-Ethyl-5-iodo-1-phenylpiperidine (2b)



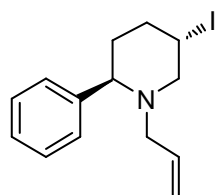
Yield: 70%; IR (NaCl, neat): 3027, 2967, 2937, 2796, 2717, 2660, 1491, 1452, 1383, 1187, 1151, 1120, 1080, 1014, 979, 758, 736, 701, 540 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.94 (t, 3H, $J = 7.2$ Hz, CH_3), 1.64-1.83 (m, 2H, CH_2 -3), 2.03 (qd, 1H, $J = 12.8, 4.8$ Hz, H-4ax), 2.14 (m, 1H, CH_2), 2.50 (m, 2H), 2.64 (t, 1H, $J = 10$ Hz, H-6ax), 3.22 (brs, 1H, H-2), 3.57 (d, 1H, $J = 10$ Hz, H-6eq), 4.40 (brs, 1H, H-5), 7.22-7.36 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 10.5 (CH_3), 25.1 (C-5), 38.2 (C-3), 38.7 (C-4), 48.2 (CH_2), 62.2 (C-6), 67.1 (C-2), 127.5, 128.6 (Ar-CH), 142.8 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{13}\text{H}_{19}\text{IN}$ 316.0557 $[\text{M}+\text{H}]^+$, found 316.0555.

(2*RS*,5*SR*)-5-Iodo-2-phenyl-1-propylpiperidine (2c)



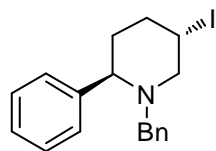
Yield: 69%; IR (NaCl, neat): 3081, 3060, 3027, 2958, 2973, 2869, 2798, 1491, 1453, 1385, 1339, 1307, 1190, 1148, 1120, 1078, 1027, 978, 893, 759, 700, 537 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.70 (t, 3H, $J = 7.6$ Hz, CH_3), 1.37 (m, 2H), 1.66 (m, 2H, CH_2 -3), 1.95 (m, 1H), 2.00 (m, 1H, H-4ax), 2.31 (dt, 1H, $J = 12.8, 8.4$ Hz), 2.46 (m, 1H, H-4eq), 2.55 (t, 1H, $J = 11.6$ Hz, H-6ax), 3.12 (t, 1H, $J = 6.8$ Hz, H-2), 3.54 (dm, 1H, $J = 11.2$ Hz, H-6eq), 4.32 (tm, 1H, $J = 11.6$ Hz, H-5), 7.20-7.33 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 11.6 (CH_3), 19.0 (CH_2), 25.8 (C-5), 38.7 (C-3), 39.0 (C-4), 56.1 (CH_2), 63.1 (C-6), 67.5 (C-2), 127.1, 127.5, 128.4 (Ar-CH), 144.0 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{21}\text{IN}$ 330.0713 $[\text{M}+\text{H}]^+$, found 330.0713.

(2*RS*,5*SR*)-1-Allyl-5-iodo-2-phenylpiperidine (2d)



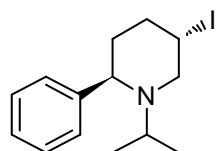
Yield: 66%; IR (NaCl, neat): 3062, 3026, 2939, 2793, 1641, 1601, 1490, 1451, 1360, 1332, 1189, 1147, 1100, 1080, 983, 920, 795, 758, 700, 537 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.68 (m, 2H, CH_2 -3), 2.01 (m, 1H, H-4ax), 2.47 (dm, 1H, $J = 12.8$, H-4eq), 2.52 (m, 1H), 2.53 (t, 1H, $J = 11.6$ Hz, H-6ax), 3.08 (ddt, 1H, $J = 14$, 4.8, 1.6 Hz), 3.16 (t, 1H, $J = 7$ Hz, H-2), 3.52 (ddd, 1H, $J = 11.6$, 4, 2 Hz, H-6eq), 4.30 (tt, 1H, $J = 12$, 4 Hz, H-5), 5.07 (m, 2H, $=\text{CH}_2$), 5.72 (m, 1H, $=\text{CH}$), 7.20-7.33 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 25.3 (C-5), 38.6 (C-3), 38.9 (C-4), 57.3 (CH_2), 63.0 (C-6), 67.0 (C-2), 117.9 ($=\text{CH}_2$), 127.3, 127.4, 128.6 (Ar-CH), 134.4 ($=\text{CH}$), 143.6 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{19}\text{IN}$ 328.0556 [$\text{M}+\text{H}$] $^+$, found 328.0560.

(2*RS*,5*SR*)-1-Benzyl-5-iodo-2-phenylpiperidine (2e)



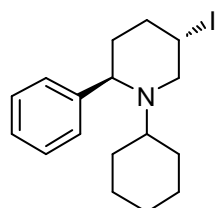
Yield: 79%; IR (NaCl, neat): 3060, 3027, 2939, 2793, 1492, 1451, 1376, 1336, 1306, 1190, 1148, 1104, 1077, 1027, 990, 910, 795, 758, 737, 698, 536 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.73 (m, 2H, CH_2 -3), 2.03 (m, 1H, H-4ax), 2.45 (t, 1H, $J = 11.6$ Hz, H-6ax), 2.47 (m, 1H, H-4eq), 2.88 (d, 1H, $J = 13.6$ Hz), 3.22 (m, 1H, H-2), 3.35 (ddd, 1H, $J = 11.2$, 4, 2 Hz, H-6eq), 3.70 (d, 1H, $J = 13.6$ Hz), 4.22 (tt, 1H, $J = 12$, 4 Hz, H-5), 7.18-7.43 (m, 10H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 25.4 (C-5), 38.9 (C-3), 39.0 (C-4), 58.6 (CH_2), 62.9 (C-6), 67.6 (C-2), 126.9, 127.3, 127.4, 128.2, 128.5, 128.7 (Ar-CH), 138.7 (*ipso*-C), 144.1 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{18}\text{H}_{21}\text{IN}$ 378.0713 [$\text{M}+\text{H}$] $^+$, found 378.0714.

(2*RS*,5*SR*)-5-Iodo-1-isopropyl-2-phenylpiperidine (2f)



Yield: 63%; IR (NaCl, neat): 3081, 3061, 3027, 2963, 2934, 2869, 2796, 1491, 1453, 1385, 1368, 1193, 1162, 1116, 1076, 1042, 1027, 971, 884, 759, 701, 540 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.77 (d, 3H, $J = 6.4$ Hz, CH_3), 0.97 (d, 3H, $J = 6.8$ Hz, CH_3), 1.66 (m, 2H, CH_2 -3), 2.00 (m, 1H, H-4ax), 2.46 (brd, 1H, $J = 11.6$ Hz, H-4eq), 2.62 (t, 1H, $J = 11.6$ Hz, H-6ax), 2.74 (m, 1H), 3.38 (m, 2H, H-2 and H-6eq), 4.27 (brt, 1H, $J = 11.6$ Hz, H-5), 7.18-7.37 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 12.5 (CH_3), 21.3 (CH_3), 27.7 (C-5), 39.4 (C-3 and C-4), 48.4 (CH), 55.4 (C-6), 64.7 (C-2), 127.1, 127.4, 128.5 (Ar-CH), 144.0 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{21}\text{IN}$ 330.0713 [$\text{M}+\text{H}$] $^+$, found 330.0716.

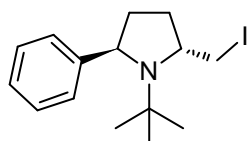
(2*RS*,5*SR*)-1-Cyclohexyl-5-iodo-2-phenylpiperidine (2g)



Yield: 55%; IR (NaCl, neat): 3061, 3027, 2930, 2853, 2795, 1491, 1450, 1188, 1141, 1116, 1077, 1010, 909, 758, 732, 700, 540 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.80-1.85 (m, 13H), 1.99 (brs, 1H, H-4ax), 2.25 (brs, 1H), 2.46 (brs, 1H, H-4eq), 2.73 (brs, 1H, H-6ax), 3.48 (brs, 2H, H-2 and H-6eq), 4.28 (brs, 1H, H-5), 7.15-7.45 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 24.1 (CH_2), 25.6 (CH_2), 26.2 (CH_2), 26.3 (CH_2), 28.1 (C-5), 31.4 (CH_2), 39.3 (C-3 and C-4), 57.2 (C-6), 58.0 (CH), 64.4 (C-2), 127.1, 127.4, 128.5 (Ar-CH), 144.0

(*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₇H₂₅IN 370.1026 [M+H]⁺, found 370.1024.

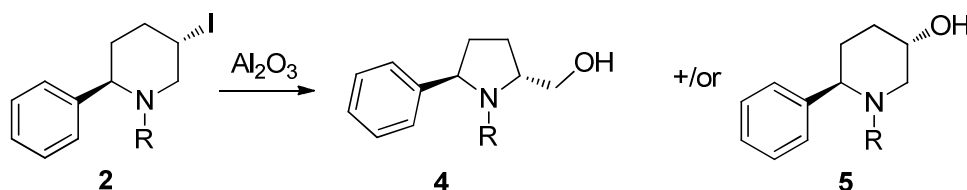
(2*RS*,5*RS*)-1-*tert*-Butyl-2-iodomethyl-5-phenyl-pyrrolidine (3h)



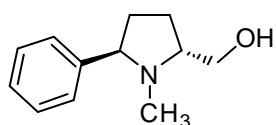
Yield: 50%; IR (NaCl, neat): 3059, 3023, 2966, 2870, 1489, 1450, 1422, 1392, 1366, 1222, 1200, 1160, 1100, 1031, 976, 944, 909, 757, 701, 580 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.97 (s, 9H, CH₃), 1.65 (m, 1H, H-4), 1.78 (m, 1H, H-3), 2.00 (m, 1H, H-3), 2.14 (m, 1H, H-4), 3.13 (dd, 1H, *J* = 11.6, 9.6 Hz, CH₂I), 3.25 (ddd, 1H, *J* = 9.6, 3.2, 1.6 Hz, CH₂I), 3.54 (m, 1H, H-2), 4.13 (dd, 1H, *J* = 9, 7 Hz, H-5), 7.10-7.50 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 14.6 (CH₂I), 27.7 (CH₃), 29.9 (C-3), 36.8 (C-4), 54.9 (C), 62.2 (C-2), 65.6 (C-5), 125.9, 126.1, 128.1 (Ar-CH), 148.8 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₅H₂₃IN 344.0870 [M+H]⁺, found 344.0873.

III. Evolution of 2a-g and 3h on alumina

General procedure: Iodo derivatives **2a-g** or **3h** (20-50 mg) were adsorbed on alumina (2 g) overnight then the formed alcohols were separated by chromatography on Al₂O₃ (CH₂Cl₂/NH₃-CH₂Cl₂/MeOH 99:1).⁴

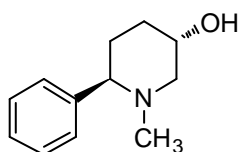


(2*RS*,5*RS*)-1-Methyl-5-phenyl-2-pyrrolidinemethanol (4a)



IR (NaCl, neat): 3069, 3085, 3061, 3027, 2945, 2873, 2794, 1601, 1491, 1452, 1363, 1285, 1200, 1163, 1057, 1032, 974, 933, 855, 757, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.86 (m, 1H, H-4), 1.96 (m, 1H, H-3), 2.15 (s, 3H, CH₃), 2.19-2.34 (m, 2H, H-3 and H-4), 2.40 (brs, 1H, OH), 3.13 (m, 1H, H-2), 3.57 (dd, 1H, *J* = 10.8, 2.4 Hz, CH₂OH), 3.69 (dd, 1H, *J* = 10.8, 3.6 Hz, CH₂OH), 4.15 (dd, 1H, *J* = 7.6, 4 Hz, H-5), 7.18-7.36 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 27.3 (C-3), 31.9 (C-4), 35.1 (CH₃), 61.6 (CH₂OH), 63.7 (C-2), 68.8 (C-5), 127.0, 127.9, 128.3 (Ar-CH), 142.6 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₂H₁₈NO 192.1383 [M+H]⁺, found 192.1383.

(3*RS*,6*SR*)-1-Methyl-6-phenylpiperidin-3-ol (5a)

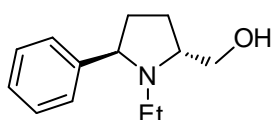


IR (NaCl, neat): 3327, 3061, 3028, 2938, 2854, 2781, 1492, 1451, 1251, 1199, 1126, 1103, 1076, 1011, 974, 962, 881, 759, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.36 (dddd, 1H, *J* = 13.3, 12.1, 11, 4 Hz, H-4ax), 1.60 (brs, 1H, OH), 1.66 (tdd, 1H, *J* = 13.6, 11.2, 3.6 Hz, H-5ax), 1.80 (ddt, 1H, *J* = 13.6, 4,

⁴ For the yields of **4** and **5** obtained in each case see the article.

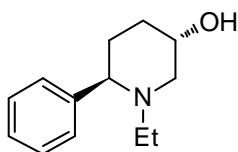
3.2 Hz, H-5eq), 2.02 (t, 1H, $J = 10.4$ Hz, H-2ax), 2.02 (s, 3H, CH₃), 2.11 (dm, 1H, $J = 12.0$ Hz, H-4eq), 2.76 (dd, 1H, $J = 11.2, 3.2$ Hz, H-6), 3.21 (ddd, 1H, $J = 10.4, 4.4, 2$ Hz, H-2eq), 3.93 (tt, 1H, $J = 10.4, 4.4$ Hz, H-3), 7.22-7.34 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 33.9 (C-5), 34.3 (C-4), 44.2 (CH₃), 64.3 (C-2), 67.7 (C-3), 69.8 (C-6), 127.2, 127.5, 128.5 (Ar-CH), 143.6 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₂H₁₈NO 192.1383 [M+H]⁺, found 192.1382.

(2*RS*,5*RS*)-1-Ethyl-5-phenyl-2-pyrrolidinemethanol (4b)



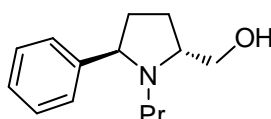
IR (NaCl, neat): 3367, 3084, 3061, 3026, 2966, 2874, 2834, 1600, 1491, 1452, 1385, 1306, 1196, 1067, 1033, 758, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.99 (t, 3H, $J = 7.2$ Hz, CH₃), 1.76 (m, 1H, H-4), 1.92 (m, 1H, H-3), 2.21-2.33 (m, 2H, H-3 and H-4), 2.41 (dq, 1H, $J = 12.4, 7.2$ Hz), 2.56 (dq, 1H, $J = 12.4, 7.2$ Hz), 2.65 (brs, 1H, OH), 3.34 (m, 1H, H-2), 3.52 (dd, 1H, $J = 10.8, 2$ Hz, CH₂OH), 3.67 (dd, 1H, $J = 10.4, 4$ Hz, CH₂OH), 4.31 (dd, 1H, $J = 7.2, 2.8$ Hz, H-5), 7.17-7.35 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 14.3 (CH₃), 27.2 (C-3), 32.5 (C-4), 41.2 (CH₂), 62.1 (CH₂OH), 62.6 (C-2), 65.6 (C-5), 126.8, 127.7, 128.3 (Ar-CH), 143.3 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₃H₂₀NO 206.1539 [M+H]⁺, found 206.1536.

(3*RS*,6*SR*)-1-Ethyl-6-phenylpiperidin-3-ol (5b)



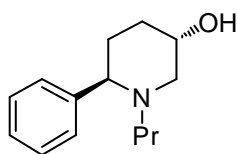
IR (NaCl, neat): 3343, 3061, 3027, 2968, 2935, 2872, 2800, 1601, 1492, 1452, 1382, 1233, 1184, 1130, 1103, 1075, 1060, 1022, 973, 860, 758, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.92 (t, 3H, $J = 7.2$ Hz, CH₃), 0.92 (t, 3H, $J = 7.2$ Hz, CH₃), 1.35 (dddd, 1H, $J = 13.3, 12.2, 11, 4.4$ Hz, H-4ax), 1.62 (tdd, 1H, $J = 13.4, 11.2, 4$ Hz, H-5ax), 1.63 (brs, 1H, OH), 1.79 (ddt, 1H, $J = 13.6, 4.4, 3.2$ Hz, H-5eq), 1.97 (t, 1H, $J = 10.4$ Hz, H-2ax), 2.06 (dq, 1H, $J = 12.8, 7.2$ Hz), 2.09 (m, 1H, H-4eq), 2.51 (dq, 1H, $J = 13.2, 7.2$ Hz), 3.01 (dd, 1H, $J = 11.2, 3.2$ Hz, H-6), 3.31 (ddd, 1H, $J = 10.4, 4.4, 2$ Hz, H-2eq), 3.89 (tt, 1H, $J = 10.4, 4.4$ Hz, H-3), 7.20-7.34 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 10.7 (CH₃), 34.4 (C-5), 34.5 (C-4), 48.6 (CH₂), 59.5 (C-2), 67.3 (C-6), 68.1 (C-3), 127.0, 127.5, 128.4 (Ar-CH), 144.1 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₃H₂₀NO 206.1539 [M+H]⁺, found 206.1537.

(2*RS*,5*RS*)-5-Phenyl-1-propyl-2-pyrrolidinemethanol (4c)



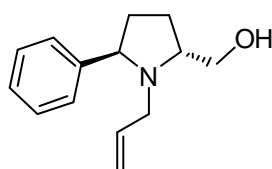
IR (NaCl, neat): 3380, 3083, 3061, 3027, 2958, 2934, 2872, 2829, 1491, 1454, 1384, 1284, 1222, 1189, 1071, 1032, 758, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.81 (t, 3H, $J = 7.2$ Hz, CH₃), 1.40 (m, 2H), 1.75 (m, 1H, H-4), 1.92 (m, 1H, H-3), 2.20-2.32 (m, 2H, H-3 and H-4), 2.39 (m, 2H), 3.32 (m, 1H, H-2), 3.51 (dd, 1H, $J = 10.8, 1.6$ Hz, CH₂OH), 3.67 (dd, 1H, $J = 10.8, 3.6$ Hz, CH₂OH), 4.28 (dd, 1H, $J = 7.2, 2.4$ Hz, H-5), 7.15-7.34 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 11.8 (CH₃), 21.9 (CH₂), 27.1 (C-3), 32.6 (C-4), 49.0 (CH₂), 62.1 (CH₂OH), 62.8 (C-2), 65.8 (C-5), 126.7, 127.7, 128.3 (Ar-CH), 143.5 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₄H₂₂NO 220.1696 [M+H]⁺, found 220.1695.

(3*RS*,6*SR*)-6-Phenyl-1-propylpiperidin-3-ol (5c)



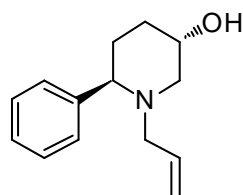
IR (NaCl, neat): 3295, 3062, 3028, 2959, 2935, 2871, 2798, 1492, 1452, 1378, 1237, 1179, 1102, 1059, 1015, 975, 759, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 0.72 (t, 3H, $J = 7.2$ Hz, CH_3), 1.35 (m, 1H, H-4ax), 1.41 (sext, 2H, $J = 7.2$ Hz, CH_2), 1.64 (qd, 1H, $J = 13.6, 3.2$ Hz, H-5ax), 1.79 (ddt, 1H, $J = 13.6, 4, 3.2$ Hz, H-5eq), 1.94 (m, 1H), 1.96 (t, 1H, $J = 10.4$ Hz, H-2ax), 2.09 (brd, 1H, $J = 12.4$ Hz, H-4eq), 2.37 (dt, 1H, $J = 12.8, 8.4$ Hz), 3.01 (dd, 1H, $J = 11.6, 3.2$ Hz, H-6), 3.35 (dm, 1H, $J = 10.4$ Hz, H-2eq), 3.90 (tt, 1H, $J = 10.4, 4.8$ Hz, H-3), 7.18-7.34 (m, 5H, Ar-H), ^{13}C NMR (CDCl_3 , 100 MHz) δ 11.6 (CH_3), 18.8 (CH_2), 34.4 (C-5 and C-4), 56.6 (CH_2), 60.0 (C-2), 67.8 (C-3 and C-6), 127.0, 127.6, 128.4 (Ar-CH), 144.0 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{22}\text{NO}$ 220.1696 $[\text{M}+\text{H}]^+$, found 220.1696.

(2*RS*,5*RS*)-1-Allyl-5-phenyl-2-pyrrolidinemethanol (4d)



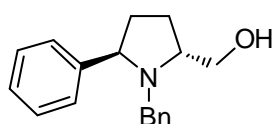
IR (NaCl, neat): 3395, 3078, 3027, 2953, 2876, 1491, 1452, 1418, 1356, 1285, 1149, 1070, 1032, 994, 916, 757, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.70 (brs, 1H, OH), 1.79 (m, 1H, H-4), 1.93 (m, 1H, H-3), 2.21-2.33 (m, 2H, H-3 and H-4), 3.02 (dd, 1H, $J = 14.4, 6.8$ Hz), 3.14 (ddt, 1H, $J = 14.4, 5.6, 2$ Hz), 3.35 (m, 1H, H-2), 3.54 (dd, 1H, $J = 10.8, 2.4$ Hz, CH_2OH), 3.68 (dd, 1H, $J = 10.8, 4$ Hz, CH_2OH), 4.27 (dd, 1H, $J = 7.2, 3.2$ Hz, H-5), 5.01 (m, 2H, $=\text{CH}_2$), 5.76 (m, 1H, $=\text{CH}$), 7.17-7.34 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 27.1 (C-3), 32.4 (C-4), 50.5 (CH_2), 62.4 (C-2 and CH_2OH), 66.4 (C-5), 116.1 ($=\text{CH}_2$), 126.9, 127.9, 128.2 (Ar-CH), 136.7 ($=\text{CH}$), 143.2 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{20}\text{NO}$ 218.1539 $[\text{M}+\text{H}]^+$, found 218.1537.

(3*RS*,6*SR*)-1-Allyl-6-phenylpiperidin-3-ol (5d)



IR (NaCl, neat): 3340, 3077, 3027, 2936, 2857, 2794, 1492, 1452, 1418, 1373, 1333, 1239, 1123, 1098, 1058, 1024, 996, 919, 759, 701 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.35 (dddd, 1H, $J = 13.4, 12.1, 11, 4.4$ Hz, H-4ax), 1.60 (brs, 1H, OH), 1.63 (tdd, 1H, $J = 14, 11, 3.6$ Hz, H-5ax), 1.81 (ddt, 1H, $J = 13.6, 4, 3.6$ Hz, H-5eq), 1.94 (t, 1H, $J = 10.8$ Hz, H-2ax), 2.10 (dm, 1H, $J = 12$ Hz, H-4eq), 2.53 (dd, 1H, $J = 13.6, 8$ Hz), 3.02 (dd, 1H, $J = 10.8, 3.2$ Hz, H-6), 3.12 (ddt, 1H, $J = 13.6, 4.8, 2$ Hz), 3.31 (ddd, 1H, $J = 10.8, 4.4, 2$ Hz, H-2eq), 3.87 (tt, 1H, $J = 10.4, 4.4$ Hz, H-3), 5.06 (m, 2H, $=\text{CH}_2$), 5.76 (m, 1H, $=\text{CH}$), 7.21-7.34 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 34.4 (C-4 and C-5), 58.0 (CH_2), 60.1 (C-2), 67.4 (C-6), 67.9 (C-3), 117.7 ($=\text{CH}_2$), 127.2, 127.5, 128.5 (Ar-CH), 134.7 ($=\text{CH}$), 143.8 (*ipso*-C). HRMS (ESI-TOF) calcd. for $\text{C}_{14}\text{H}_{20}\text{NO}$ 218.1539 $[\text{M}+\text{H}]^+$, found 218.1538.

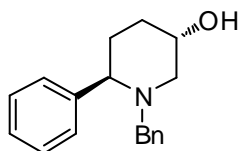
(2*RS*,5*RS*)-1-Benzyl-5-phenyl-2-pyrrolidinemethanol (4e)



IR (NaCl, neat): 3367, 3060, 3027, 2942, 2874, 1492, 1452, 1361, 1131, 1072, 1028, 757, 736, 699 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.76 (brs, 1H, OH), 1.80 (m, 1H,

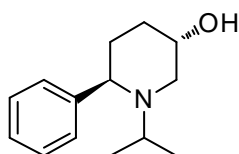
H-4), 1.97 (m, 1H, H-3), 2.29 (m, 2H, H-3 and H-4), 3.41 (m, 1H, H-2), 3.51 (d, 1H, $J = 14$ Hz, CH₂Ar), 3.55 (dd, 1H, $J = 10.8, 2$ Hz, CH₂OH), 3.67 (dd, 1H, $J = 10.8, 4$ Hz, CH₂OH), 3.72 (d, 1H, $J = 14$ Hz, CH₂Ar), 4.18 (dd, 1H, $J = 7.2, 3.2$ Hz, H-5), 7.12-7.38 (m, 10H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 27.0 (C-3), 32.6 (C-4), 51.6 (CH₂Ar), 62.3 (C-2), 62.4 (CH₂OH), 66.2 (C-5), 126.8, 126.9, 127.9, 128.2, 128.3 (Ar-CH), 139.9 (*ipso*-C), 143.3 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₈H₂₂NO 268.1696 [M+H]⁺, found 268.1696.

(3*RS*,6*SR*)-1-Benzyl-6-phenylpiperidin-3-ol (5e)



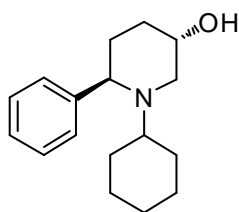
IR (NaCl, neat): 3324, 3060, 3027, 2933, 2857, 2791, 1492, 1451, 1376, 1273, 1179, 1123, 1101, 1074, 1022, 759, 740, 699 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.38 (m, 2H, H-4ax and OH), 1.67 (tdd, 1H, $J = 13.8, 11, 4$ Hz, H-5ax), 1.84 (t, 1H, $J = 10.4$ Hz, H-2ax), 1.86 (m, 1H, H-5eq), 2.09 (dm, 1H, $J = 11.6$ Hz, H-4eq), 2.86 (d, 1H, $J = 13.2$ Hz, CH₂Ar), 3.11 (m, 2H, H-2eq and H-6), 3.75 (d, 1H, $J = 13.6$ Hz, CH₂Ar), 3.79 (brs, 1H, H-3), 7.18-7.46 (m, 10H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 34.3 (C-4), 34.7 (C-5), 59.3 (CH₂Ar), 60.1 (C-2), 67.9 (C-3 and C-6), 126.8, 127.1, 127.5, 128.1, 128.6, 128.7 (Ar-CH), 139.1 (*ipso*-C), 144.4 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₈H₂₂NO 268.1696 [M+H]⁺, found 268.1696.

(3*RS*,6*SR*)-1-Isopropyl-6-phenylpiperidin-3-ol (5f)



IR (NaCl, neat): 3324, 3061, 3027, 2965, 2934, 2869, 2794, 1492, 1452, 1362, 1242, 1187, 1162, 1122, 1062, 1007, 972, 879, 760, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.77 (d, 3H, $J = 6.4$ Hz, CH₃), 0.98 (d, 3H, $J = 6.8$ Hz, CH₃), 1.32 (tdd, 1H, $J = 13.6, 12.4, 4.4$ Hz, H-4ax), 1.47 (brs, OH), 1.60 (m, 1H, H-5ax), 1.77 (ddt, 1H, $J = 13.6, 4, 3.2$ Hz, H-5eq), 2.03 (t, 1H, $J = 10.4$ Hz, H-2ax), 2.08 (m, 1H, H-4eq), 2.77 (sept, 1H, $J = 6.8$ Hz), 3.15 (ddd, 1H, $J = 10.4, 4.4, 2$ Hz, H-2eq), 3.25 (dd, 1H, $J = 10.8, 2.8$ Hz, H-6), 3.81 (brt, 1H, $J = 10$ Hz, H-3), 7.20-7.33 (m, 5H, Ar-H), ¹³C NMR (CDCl₃, 100 MHz) δ 12.2 (CH₃), 21.1 (CH₃), 34.7 (C-5 and C-4), 48.4 (CH), 51.8 (C-2), 64.9 (C-6), 68.5 (C-3), 127.0, 127.5, 128.5 (Ar-CH), 143.9 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₄H₂₂NO 220.1696 [M+H]⁺, found 220.1695.

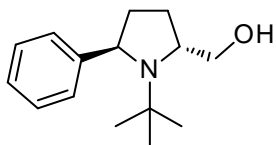
(3*RS*,6*SR*)-1-Cyclohexyl-6-phenylpiperidin-3-ol (5g)



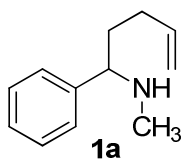
IR (NaCl, neat): 3308, 3027, 2930, 2854, 2793, 1492, 1451, 1265, 1126, 1073, 1057, 1019, 973, 758, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 0.79 (qt, 1H, $J = 12.8, 3.2$ Hz), 0.98 (m, 2H), 1.09 (qd, 1H, $J = 12, 3.2$ Hz), 1.31 (m, 1H, H-4ax), 1.37-1.76 (m, 8H), 1.77 (ddt, 1H, $J = 13.6, 4.4, 3.2$ Hz, H-5eq), 2.08 (m, 1H, H-4eq), 2.14 (t, 1H, $J = 10.4$ Hz, H-2ax), 2.29 (tt, 1H, $J = 11.6, 3.2$ Hz), 3.24 (ddd, 1H, $J = 10.4, 4.4, 2$ Hz, H-2eq), 3.39 (dd, 1H, $J = 10.8, 2.8$ Hz, H-6), 3.84 (tt, 1H, $J = 10.8, 4.4$ Hz, H-3), 7.21-7.33 (m, 5H, Ar-H), ¹³C NMR (CDCl₃, 100 MHz) δ 23.7 (CH₂), 25.7 (CH₂), 26.4 (CH₂), 26.5 (CH₂), 31.5 (CH₂), 34.7 (C-4), 34.8 (C-5), 53.8 (C-2), 57.8 (CH), 64.4 (C-6), 68.7 (C-3),

126.9, 127.5, 128.4 (Ar-CH), 144.2 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₇H₂₆NO 260.2008 [M+H]⁺, found 260.2016.

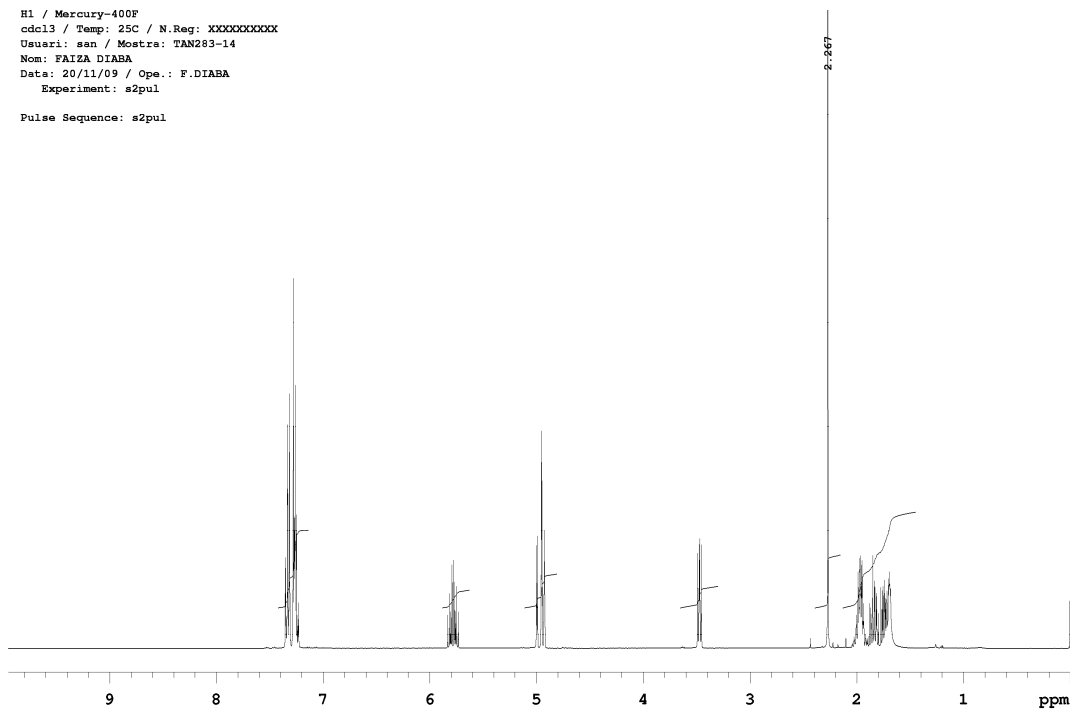
(2*RS*,5*RS*)-1-*tert*-Butyl-5-phenyl-2-pyrrolidinemethanol (4h)



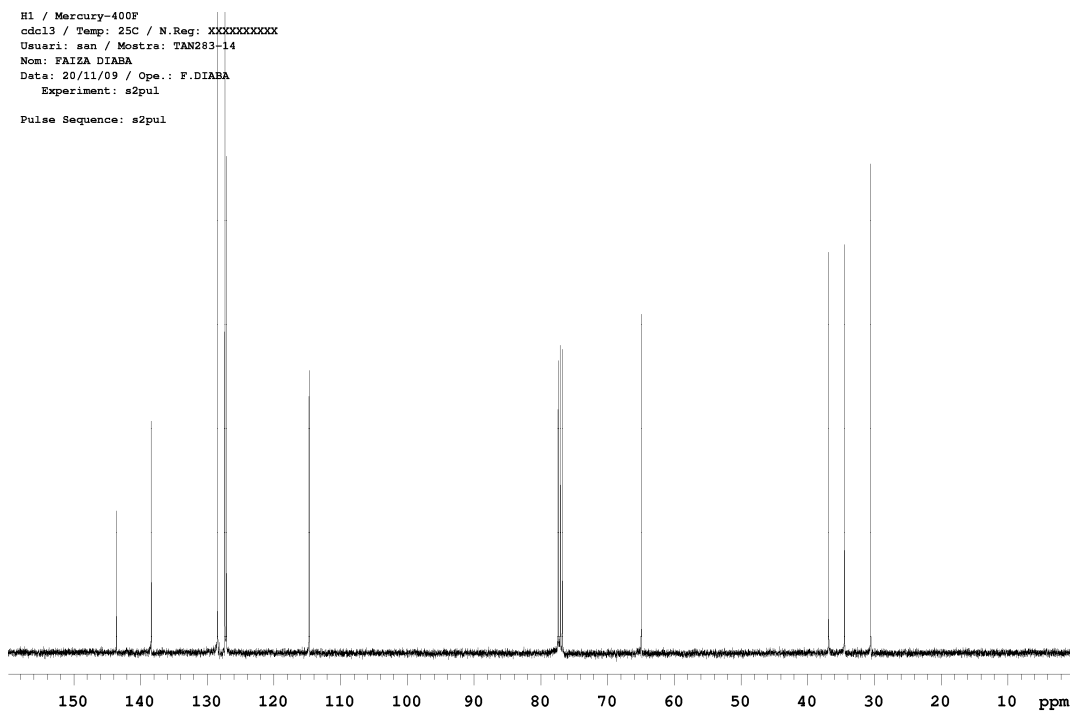
IR (NaCl, neat): 3355, 3059, 3023, 2964, 2870, 1491, 1470, 1451, 1391, 1366, 1223, 1122, 1073, 1027, 757, 701 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.02 (s, 9H, CH₃), 1.66 (m, 2H, H-3 and H-4), 1.79 (m, 1H, H-3), 2.19 (m, 1H, H-4), 3.38 (dd, 1H, *J* = 8.8, 7.6 Hz, CH₂OH), 3.44 (dt, 1H, *J* = 6.8, 6.4 Hz, H-2), 3.55 (dd, 1H, *J* = 8.8, 6.4 Hz, CH₂OH), 4.19 (t, 1H, *J* = 8.4 Hz, H-5), 7.14-7.38 (m, 5H, ArH); ¹³C NMR (CDCl₃, 100 MHz) δ 28.0 (CH₃), 29.3 (C-3), 37.0 (C-4), 55.4 (C), 59.9 (C-2), 64.6 (C-5), 65.1 (CH₂OH), 126.1, 128.3 (Ar-CH), 149.0 (*ipso*-C). HRMS (ESI-TOF) calcd. for C₁₅H₂₄NO 234.1852 [M+H]⁺, found 234.1854.

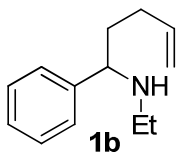


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: TAN283-14
Nom: FAIZA DIABA
Data: 20/11/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

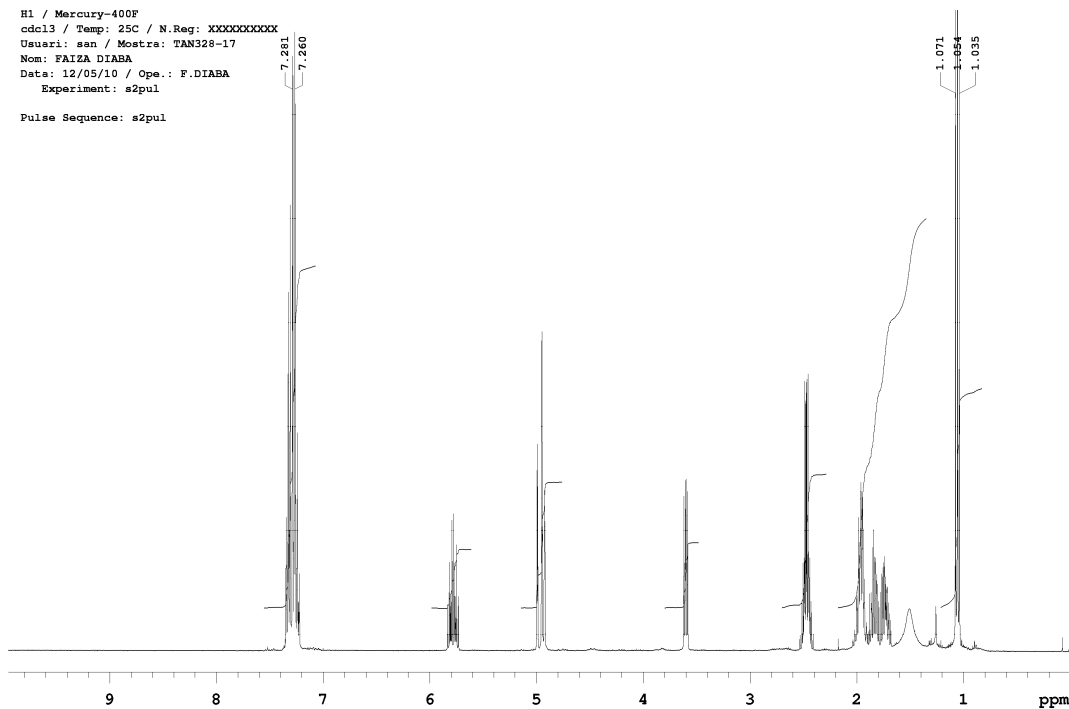


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: TAN283-14
Nom: FAIZA DIABA
Data: 20/11/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

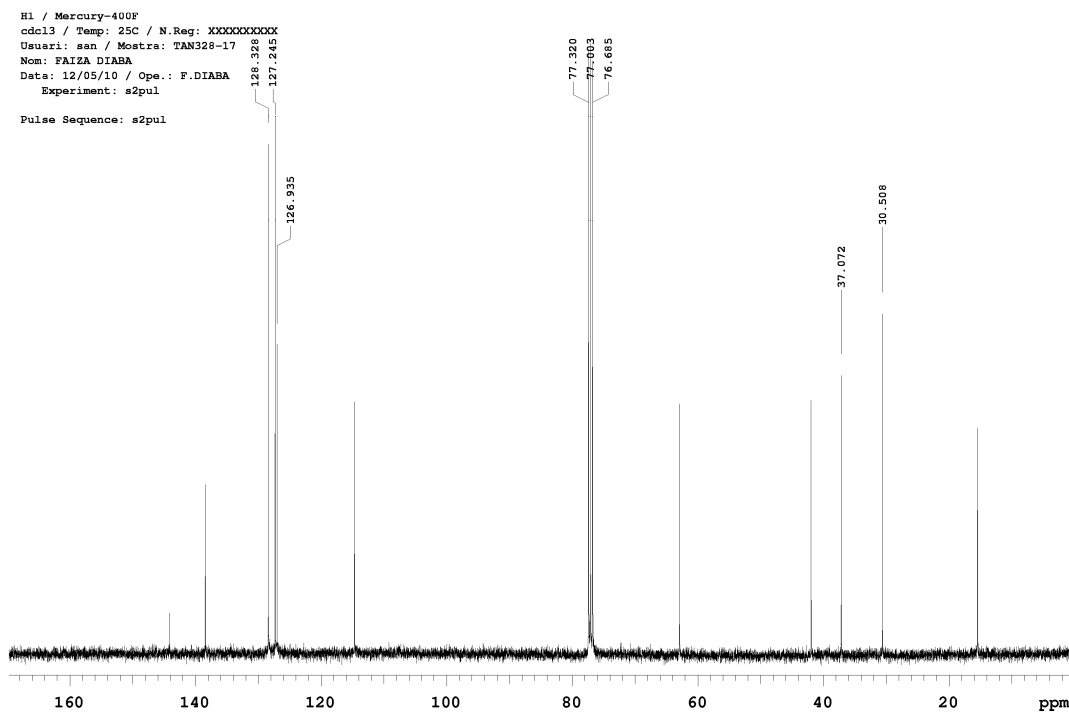


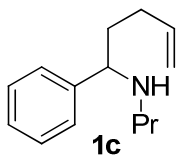


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: TAN326-17
Nom: FAIZA DIABA
Data: 12/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

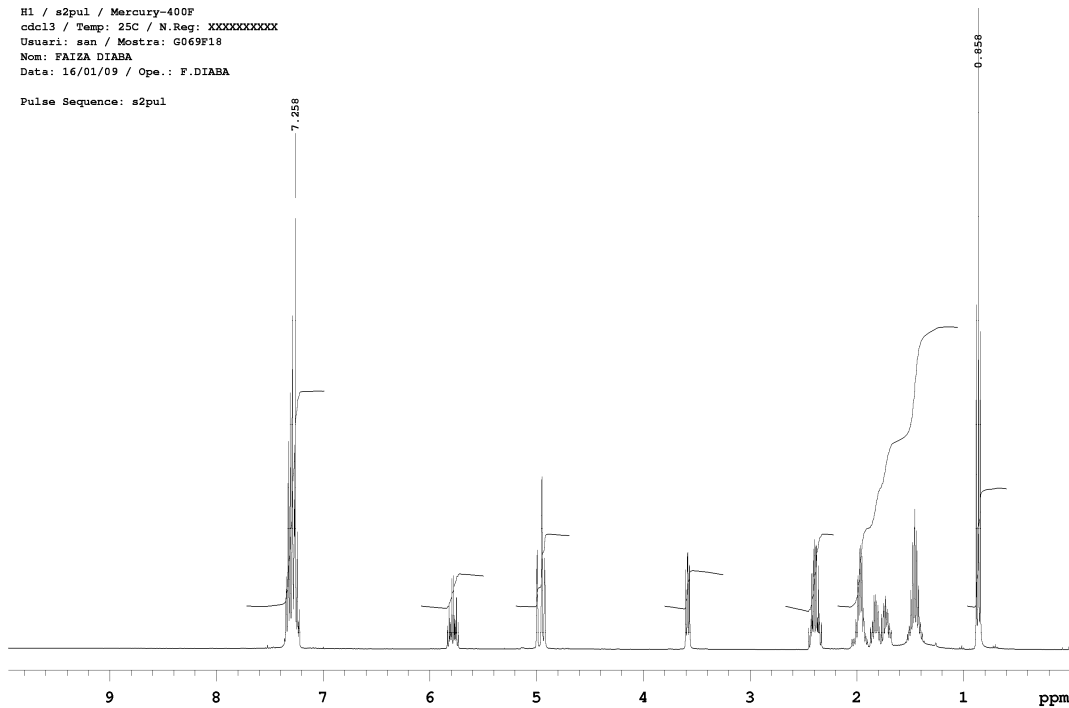


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: TAN326-17
Nom: FAIZA DIABA
Data: 12/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

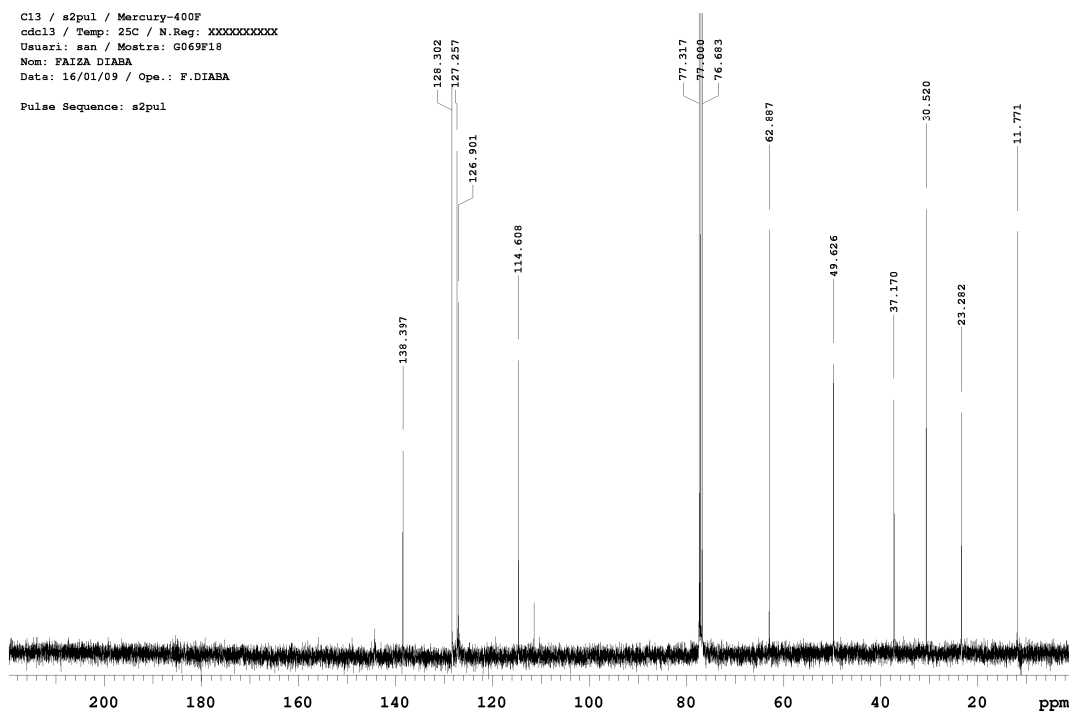


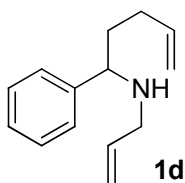


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: G069F18
Nom: FAIZA DIABA
Data: 16/01/09 / Ope.: F.DIABA
Pulse Sequence: s2pul

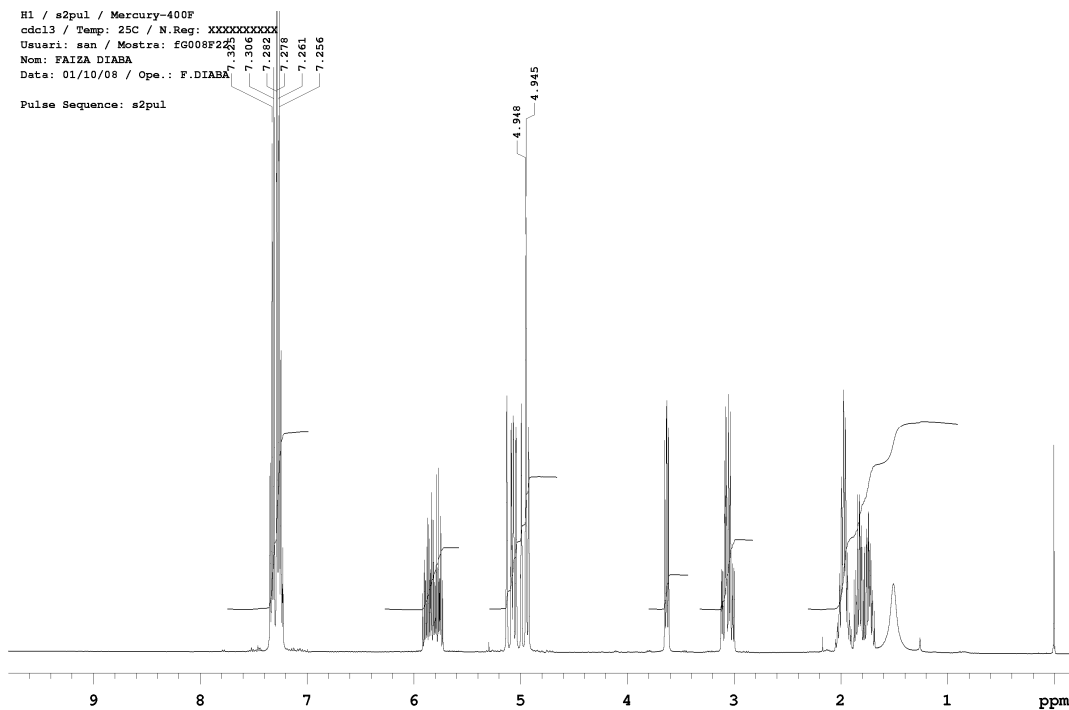


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: G069F18
Nom: FAIZA DIABA
Data: 16/01/09 / Ope.: F.DIABA
Pulse Sequence: s2pul

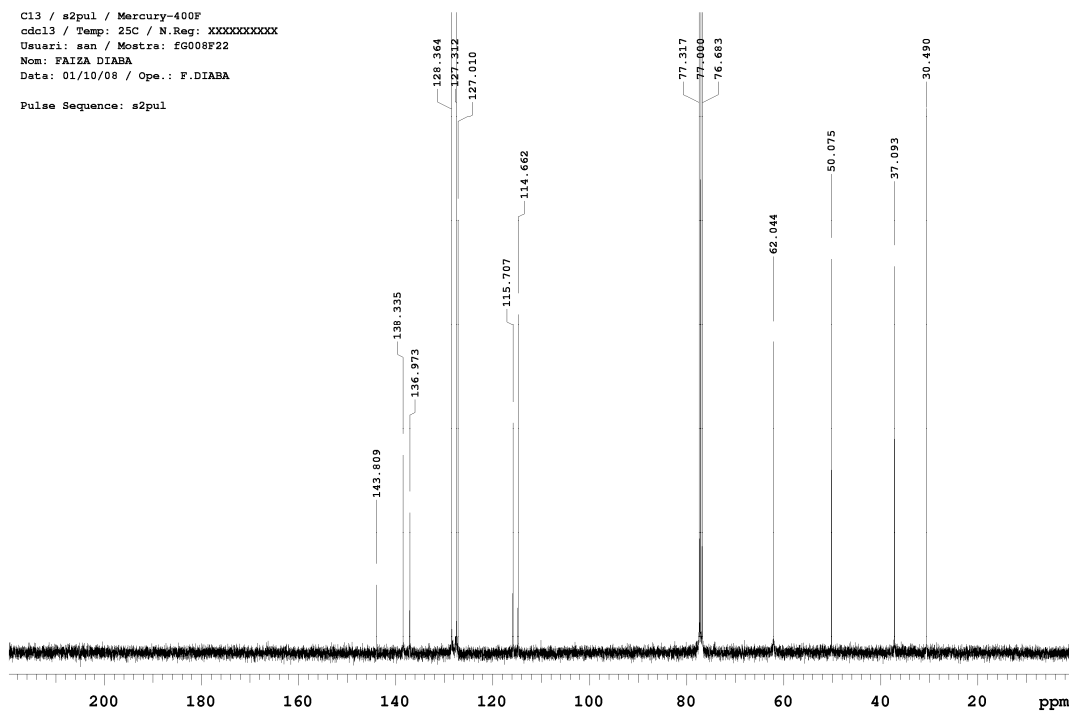


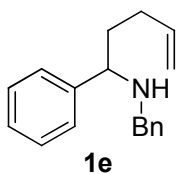


H1 / s2pul / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: sen / Mostra: FG008F22
Nom: FAIZA DIABA
Data: 01/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

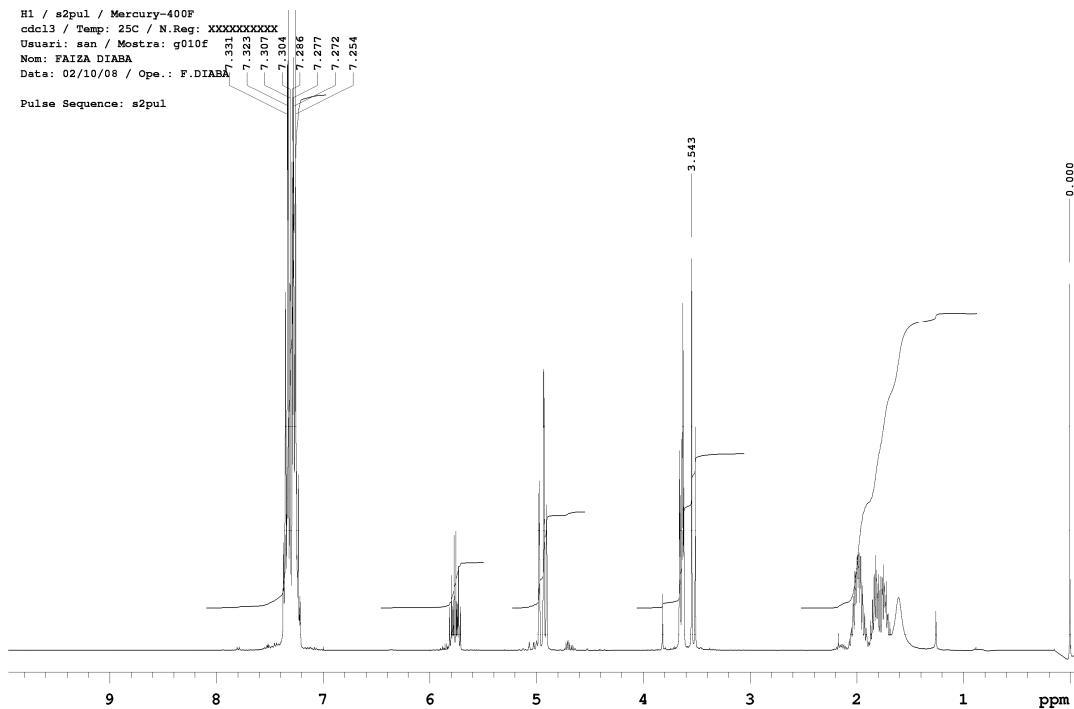


C13 / s2pul / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: sen / Mostra: FG008F22
Nom: FAIZA DIABA
Data: 01/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

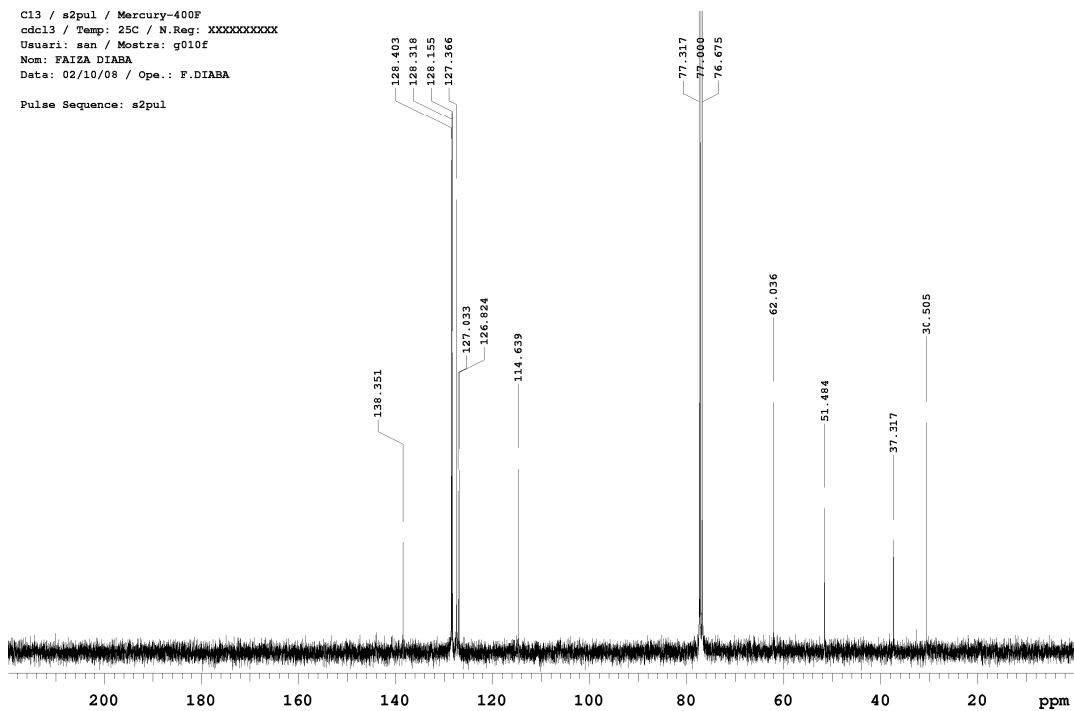


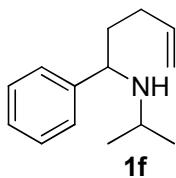


H1 / s2pul / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: g010f
Nom: FAIZA DIABA
Data: 02/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

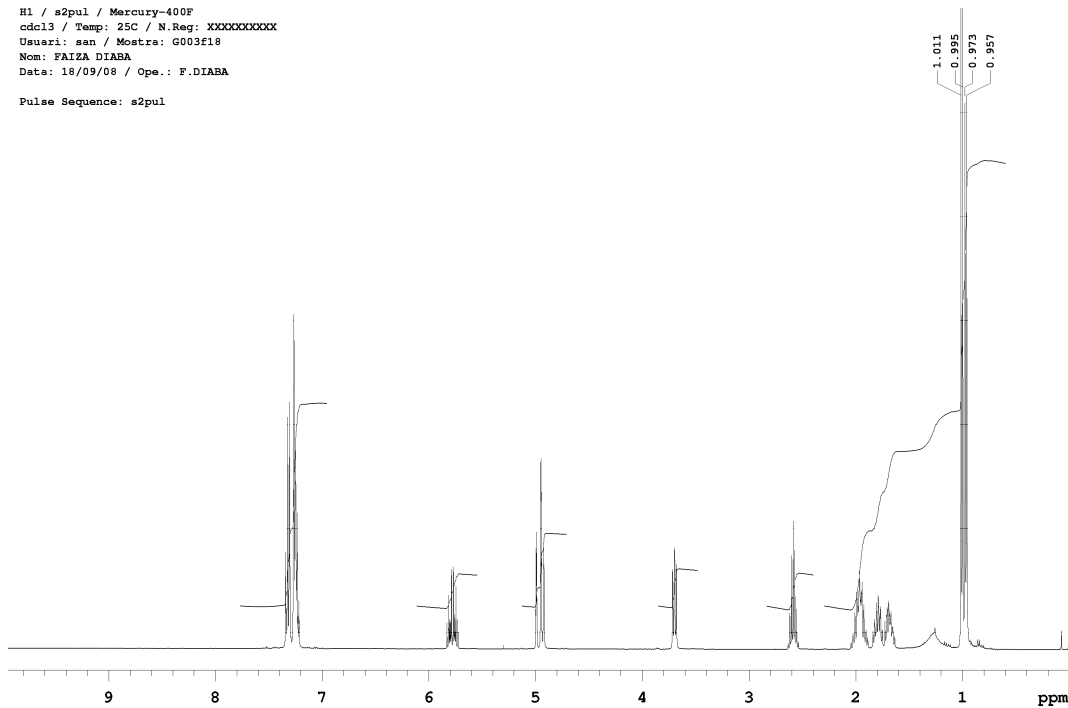


C13 / s2pul / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: g010f
Nom: FAIZA DIABA
Data: 02/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

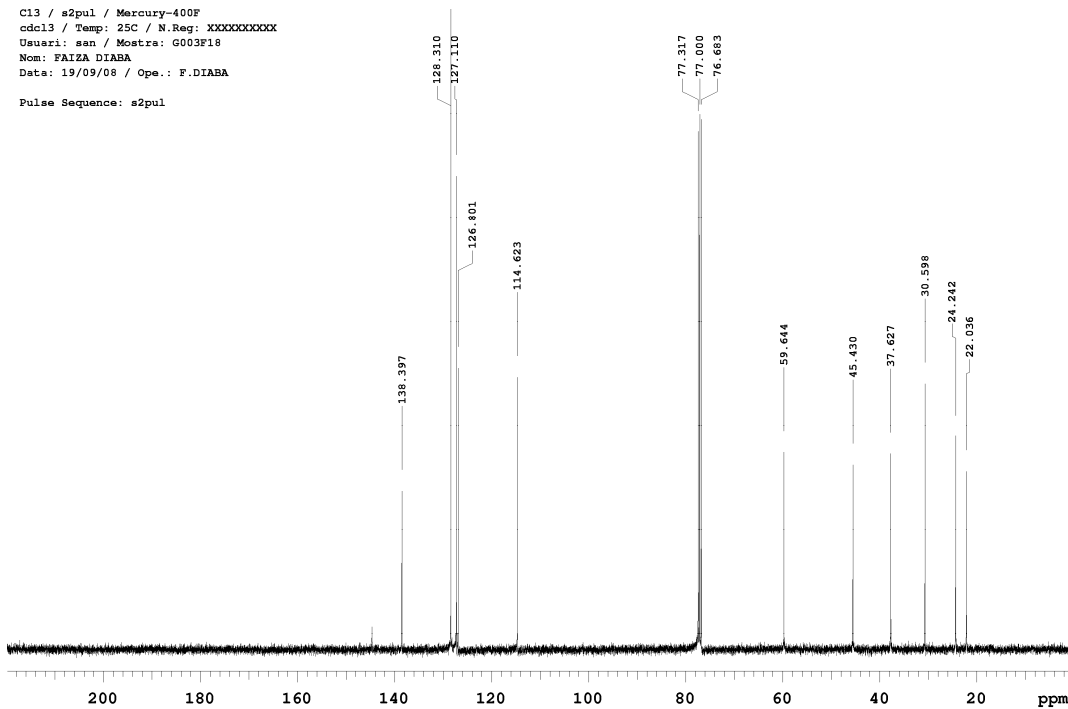


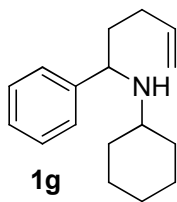


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: G003F18
Nom: FAIZA DIABA
Data: 18/09/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

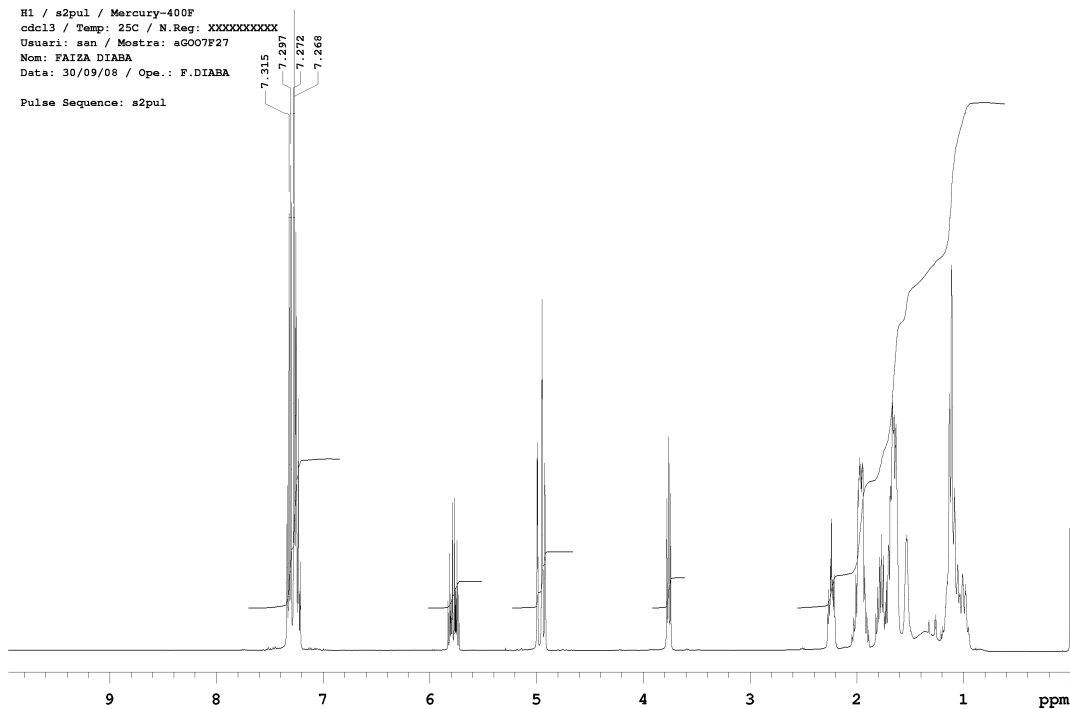


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: G003F18
Nom: FAIZA DIABA
Data: 19/09/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

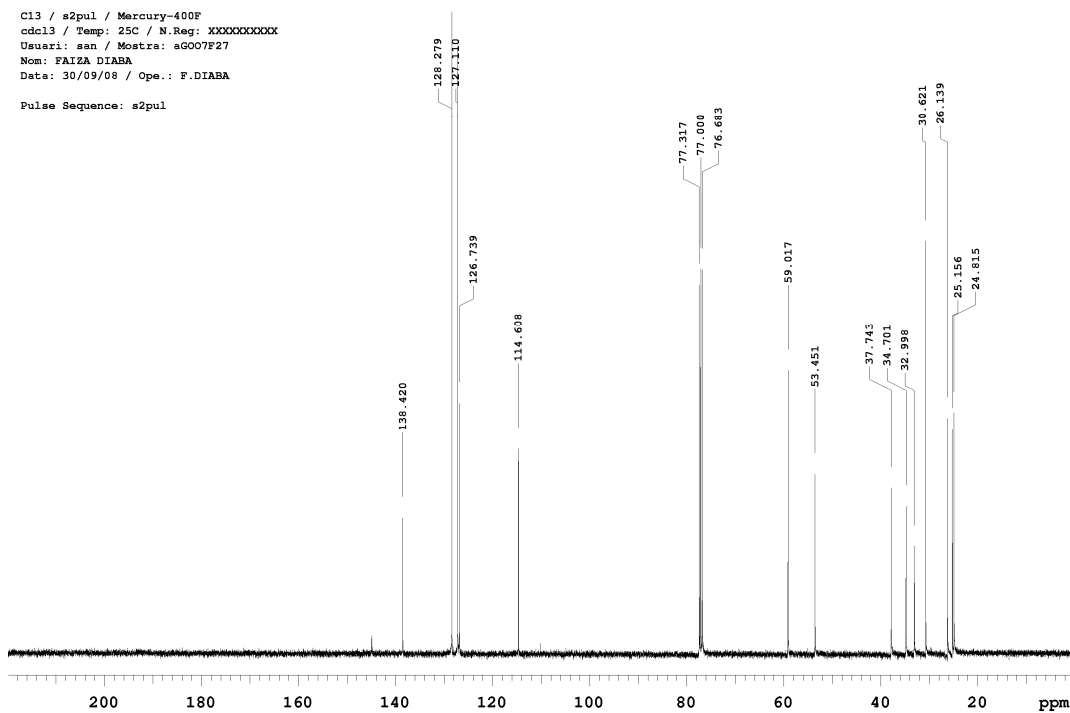


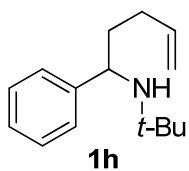


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: aG007E27
Nom: FAIZA DIABA
Data: 30/09/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

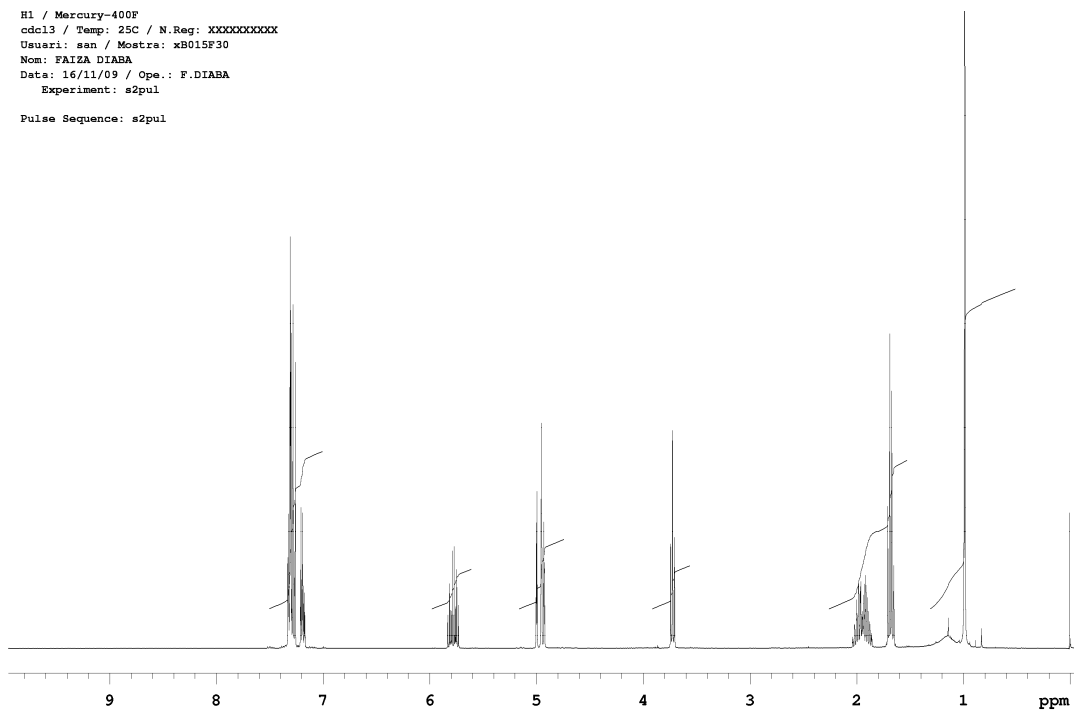


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: aG007E27
Nom: FAIZA DIABA
Data: 30/09/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

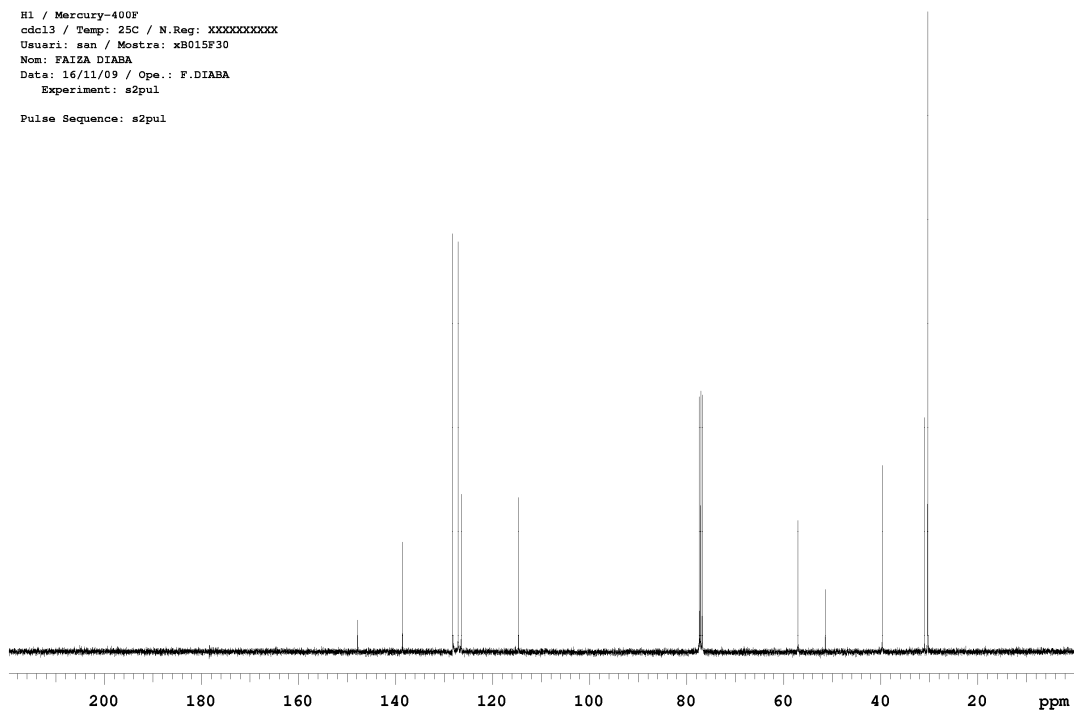


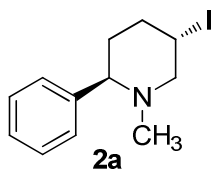


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: xB015F30
Nom: FAIZA DIABA
Data: 16/11/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

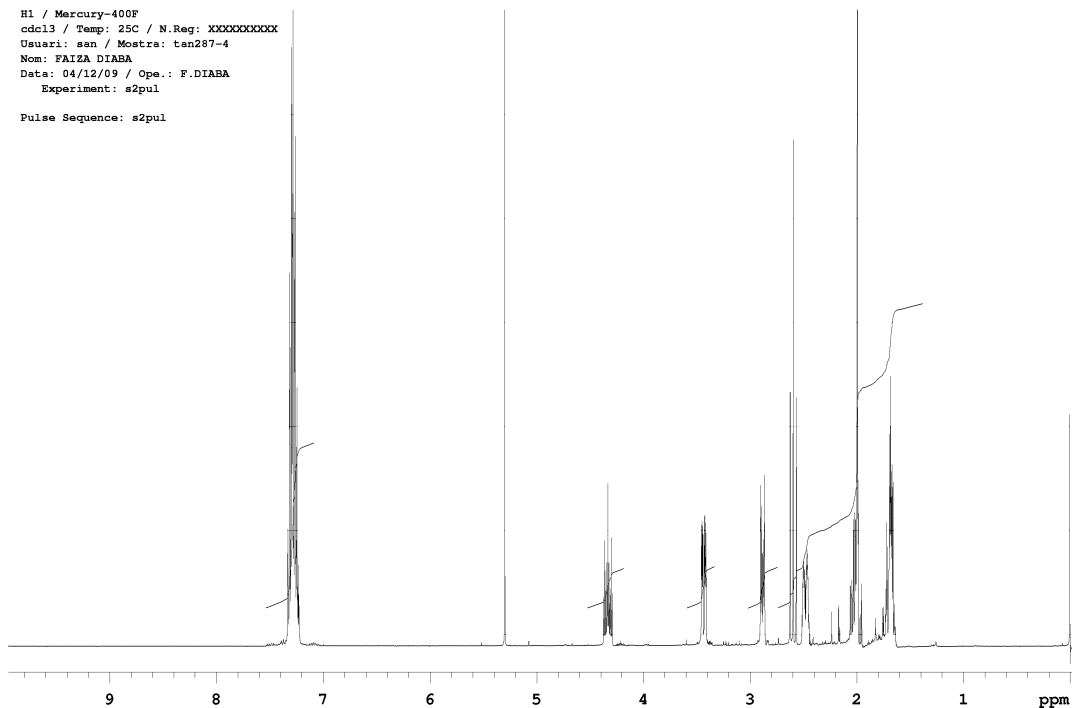


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: xB015F30
Nom: FAIZA DIABA
Data: 16/11/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

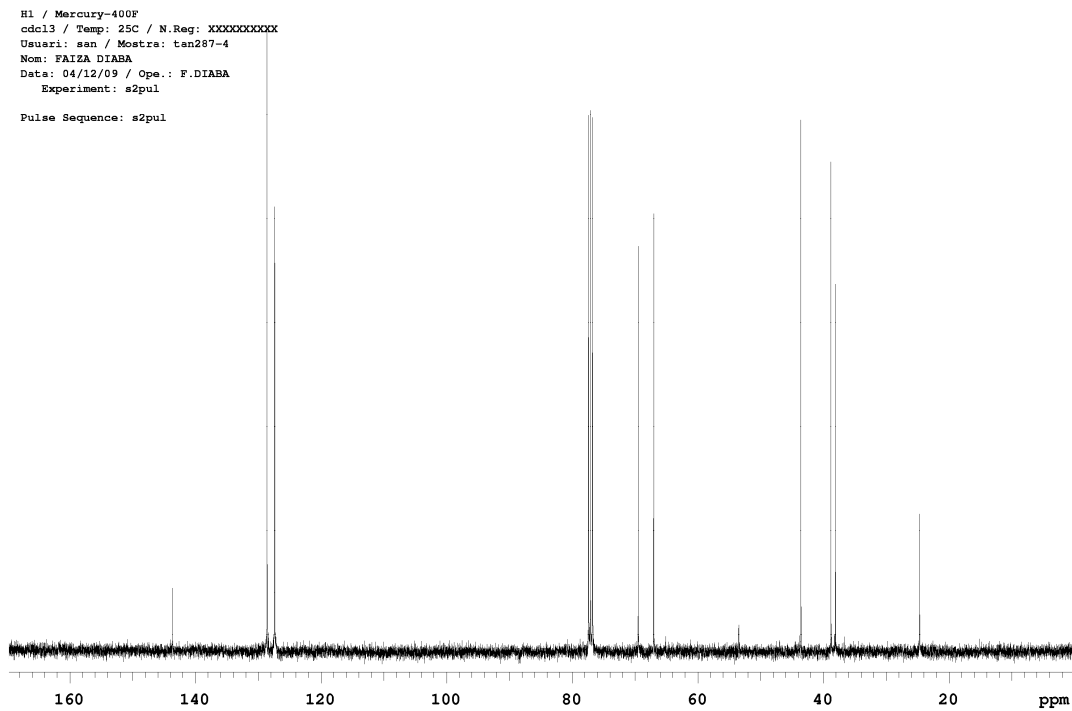


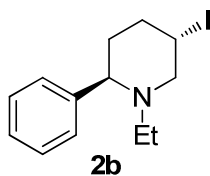


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: tan287-4
Nom: FAIZA DIABA
Data: 04/12/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

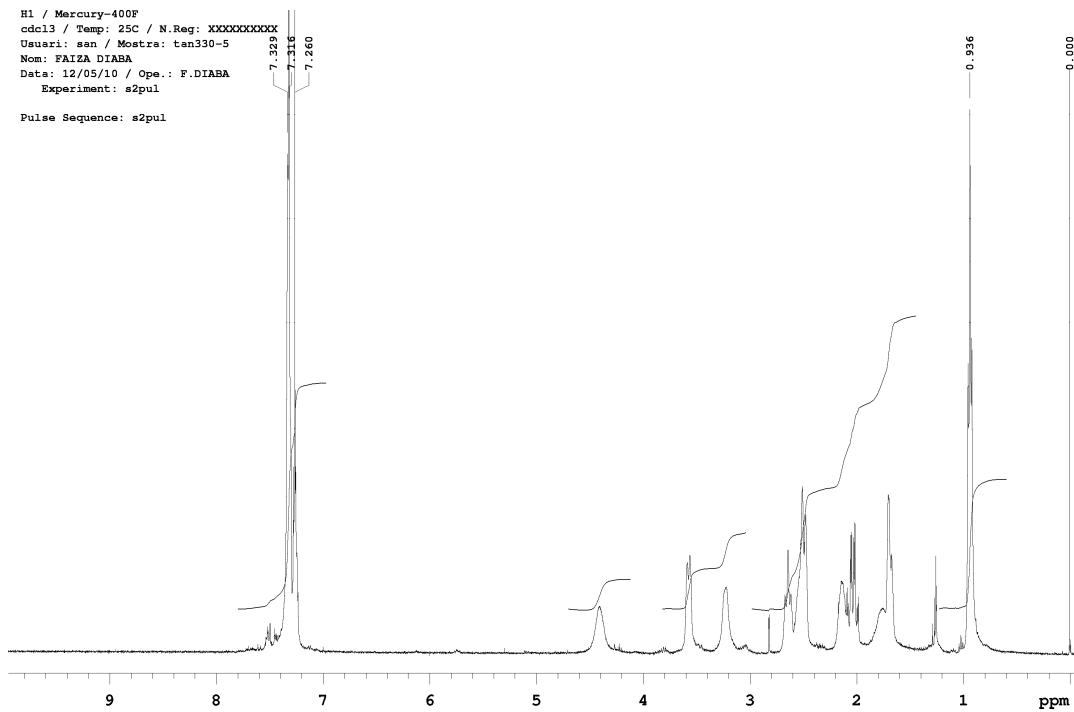


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: tan287-4
Nom: FAIZA DIABA
Data: 04/12/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

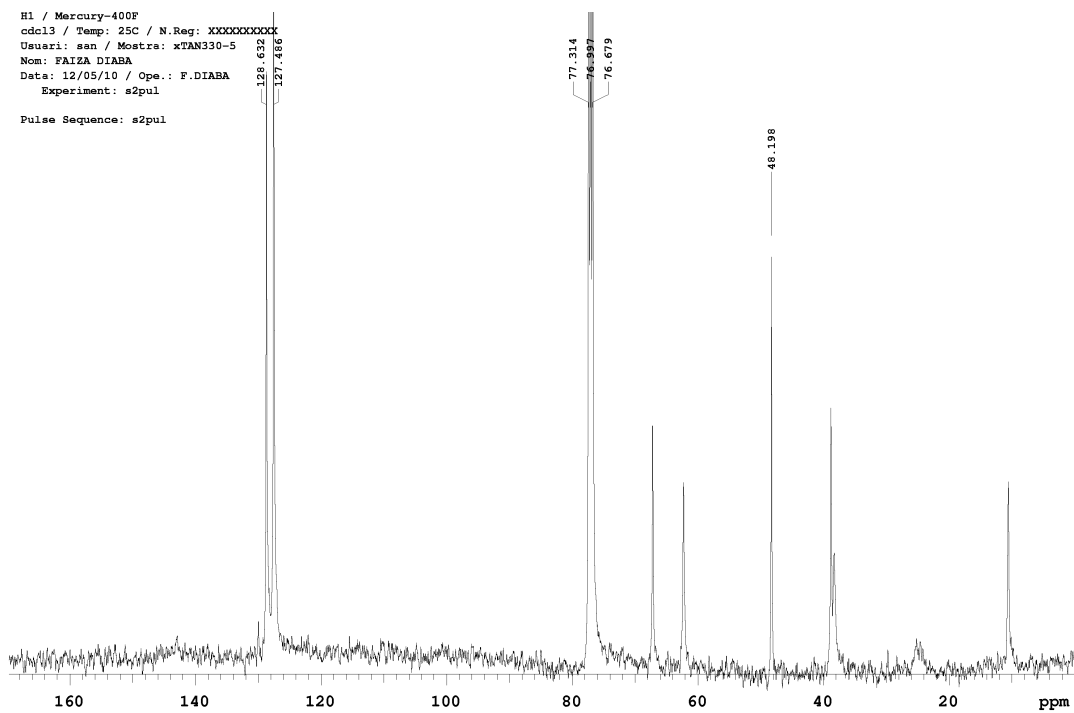


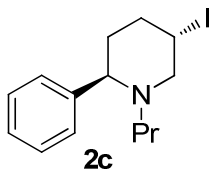


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: tan330-5
Nom: FAIZA DIABA
Data: 12/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

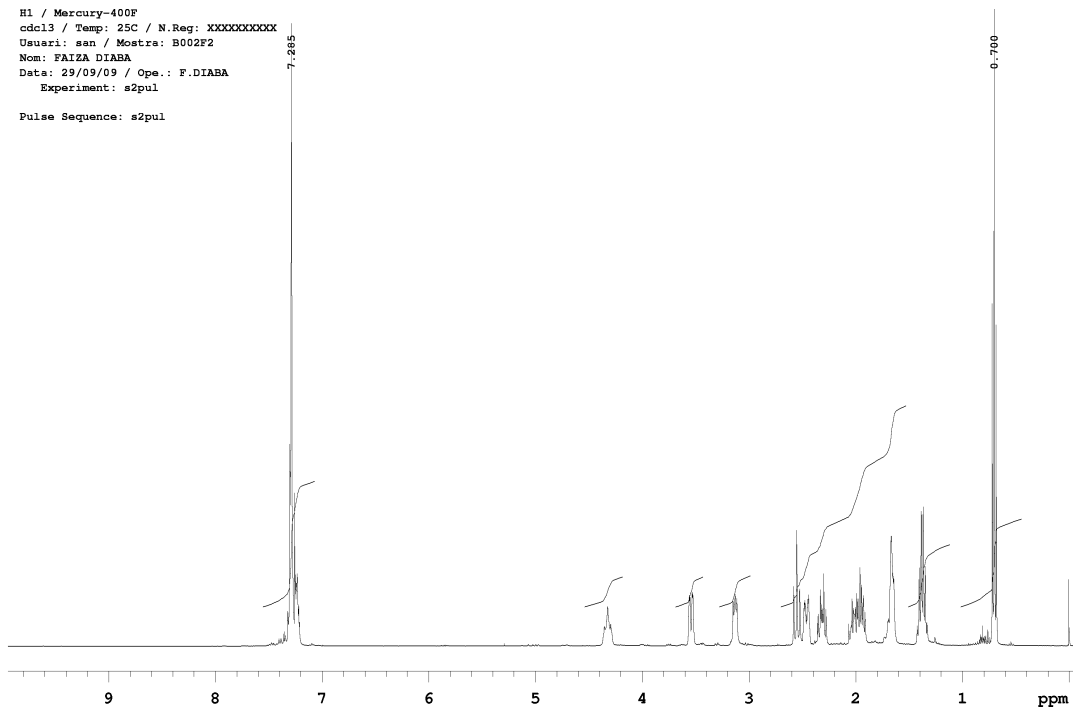


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: XTAN330-5
Nom: FAIZA DIABA
Data: 12/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

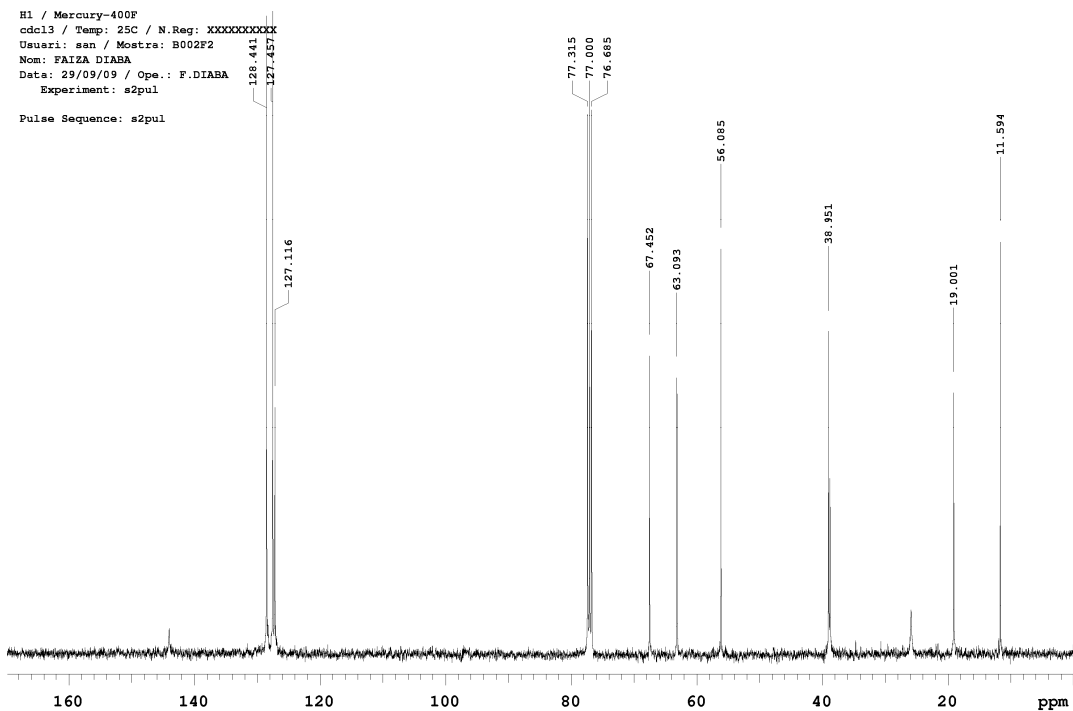


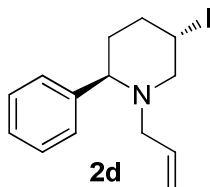


H1 / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: B002F2
Nom: FAIZA DIABA
Data: 29/09/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

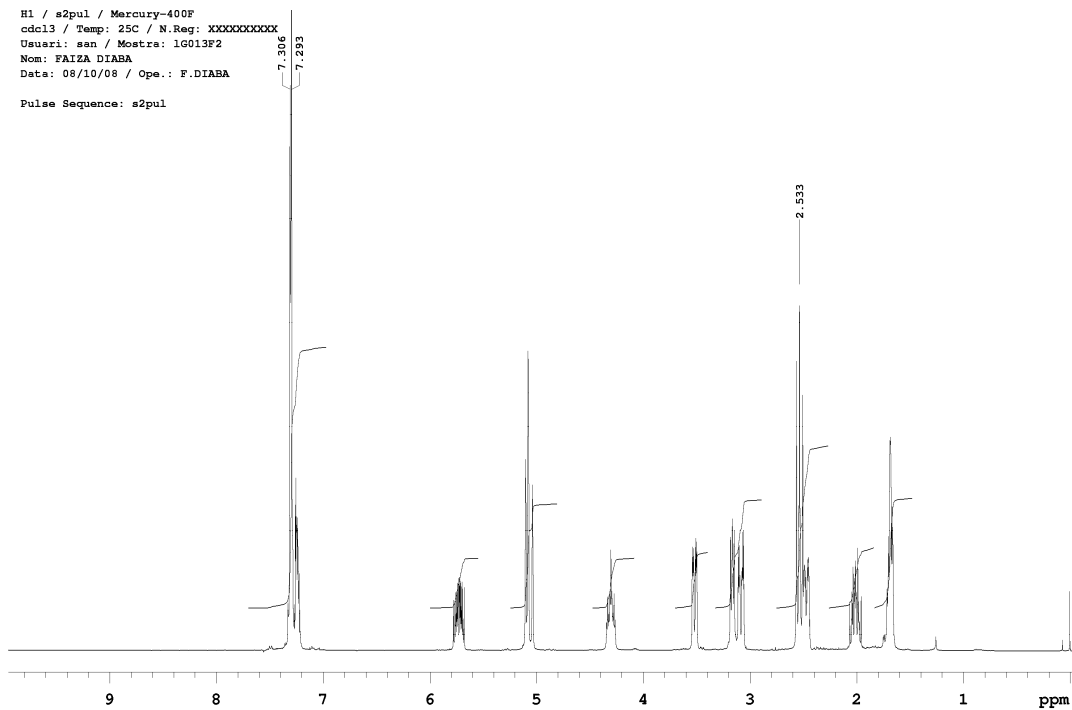


H1 / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: B002F2
Nom: FAIZA DIABA
Data: 29/09/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

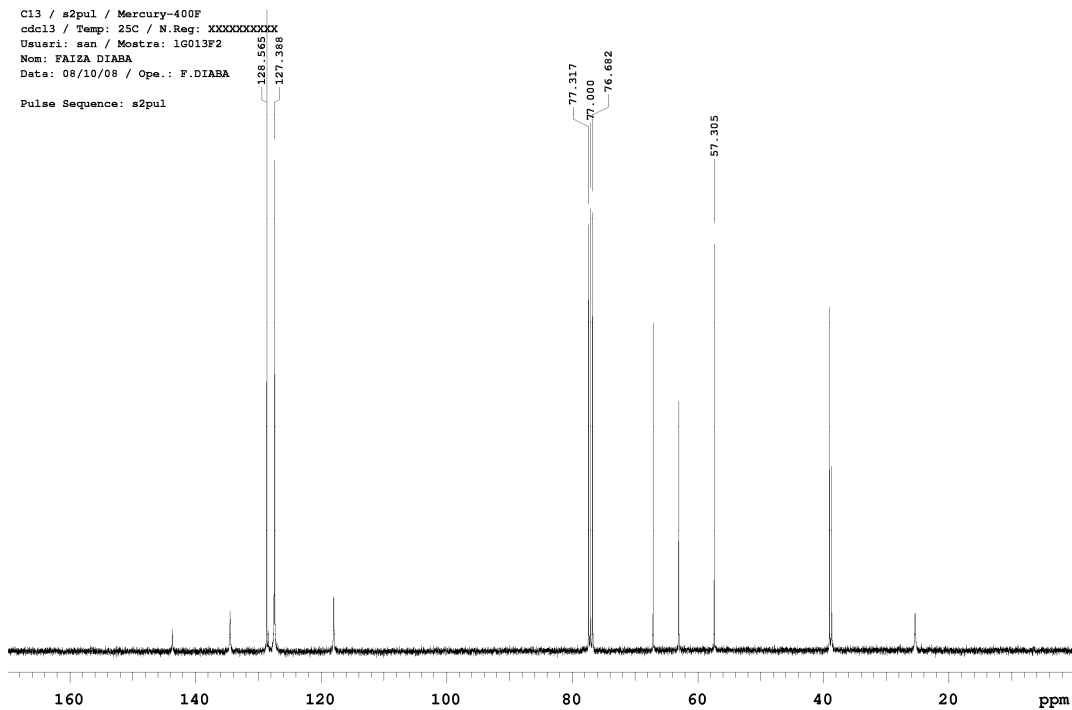


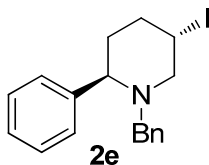


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: ssn / Mostre: 1G013F2
Nom: FAIZA DIABA
Data: 08/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

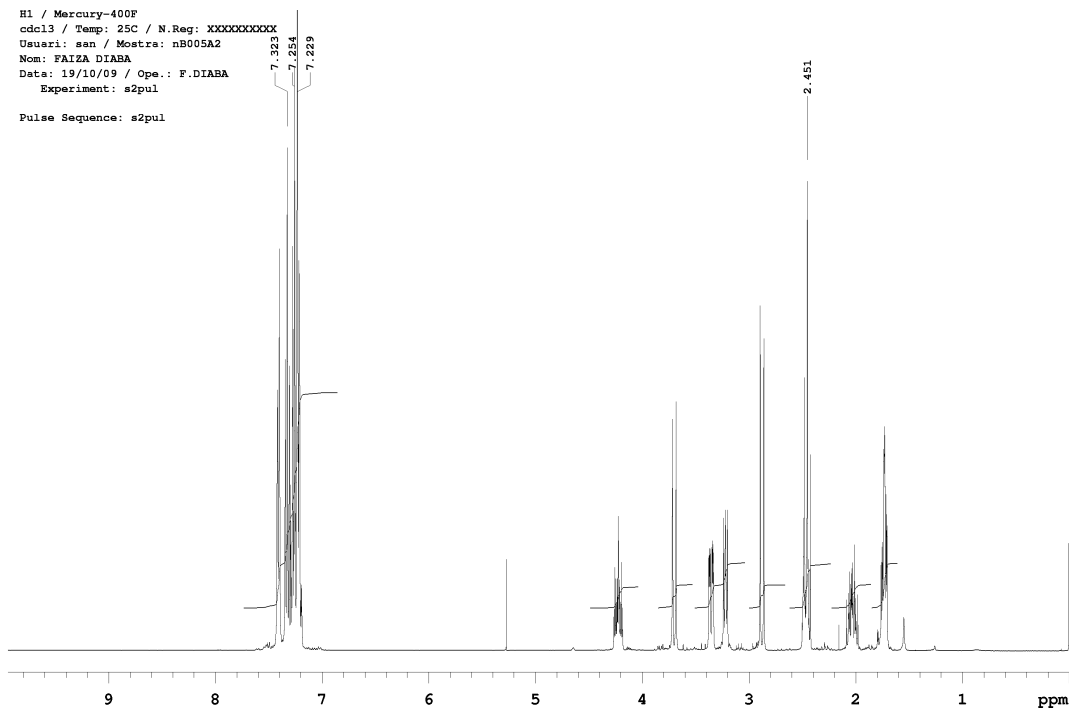


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: ssn / Mostre: 1G013F2
Nom: FAIZA DIABA
Data: 08/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

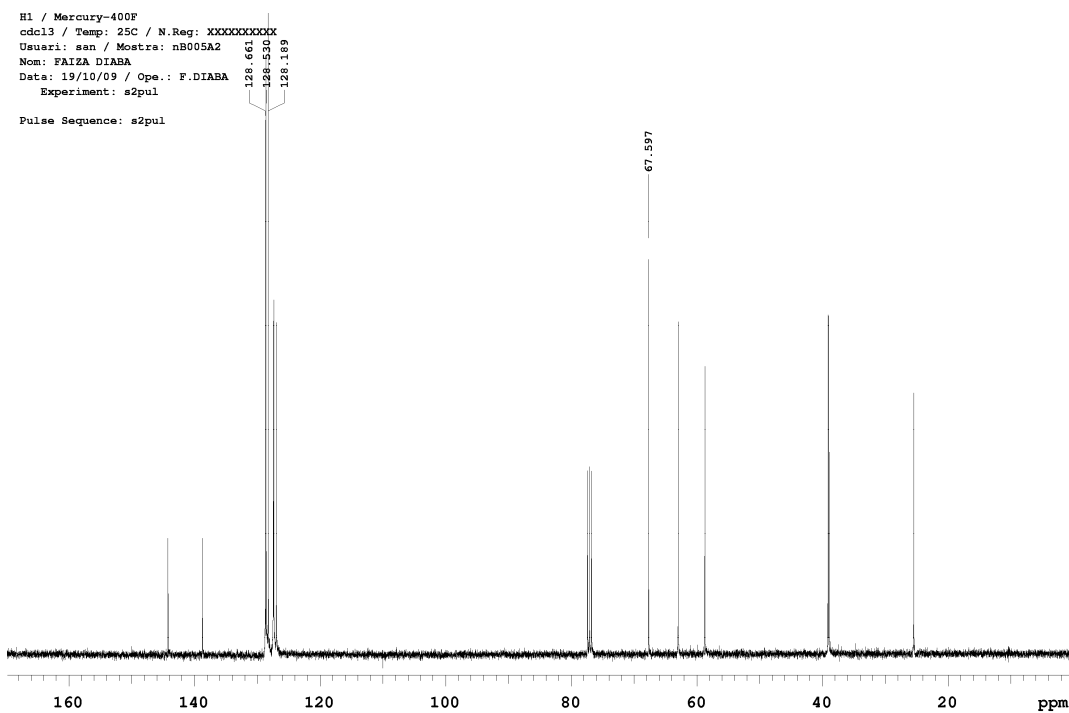


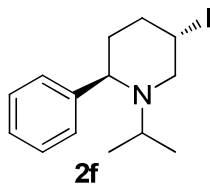


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: nB005A2
Nom: FAIZA DIABA
Data: 19/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

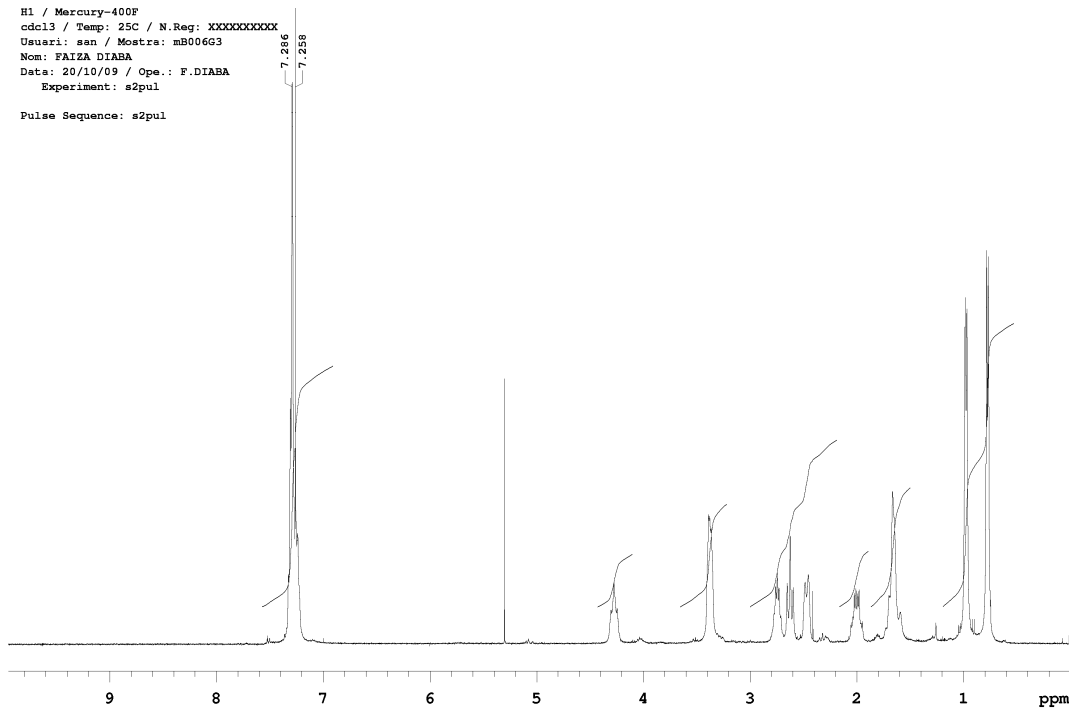


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: nB005A2
Nom: FAIZA DIABA
Data: 19/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

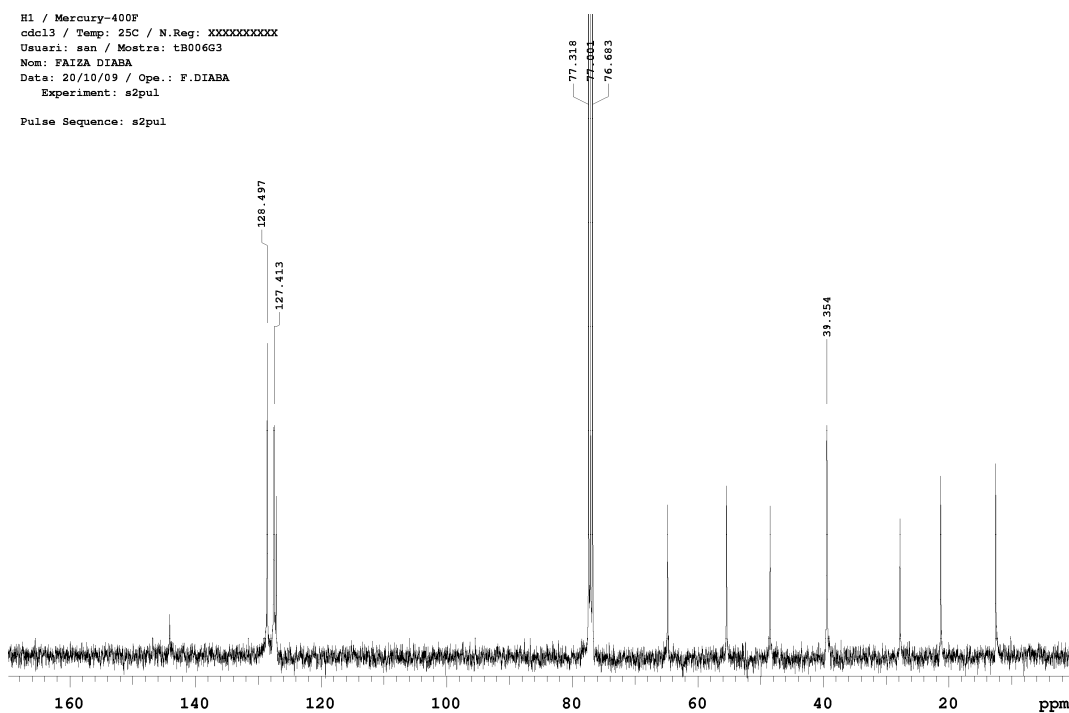


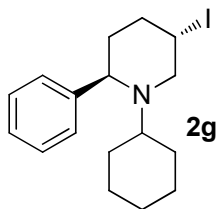


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: mB006G3
Nom: FAIZA DIABA
Data: 20/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

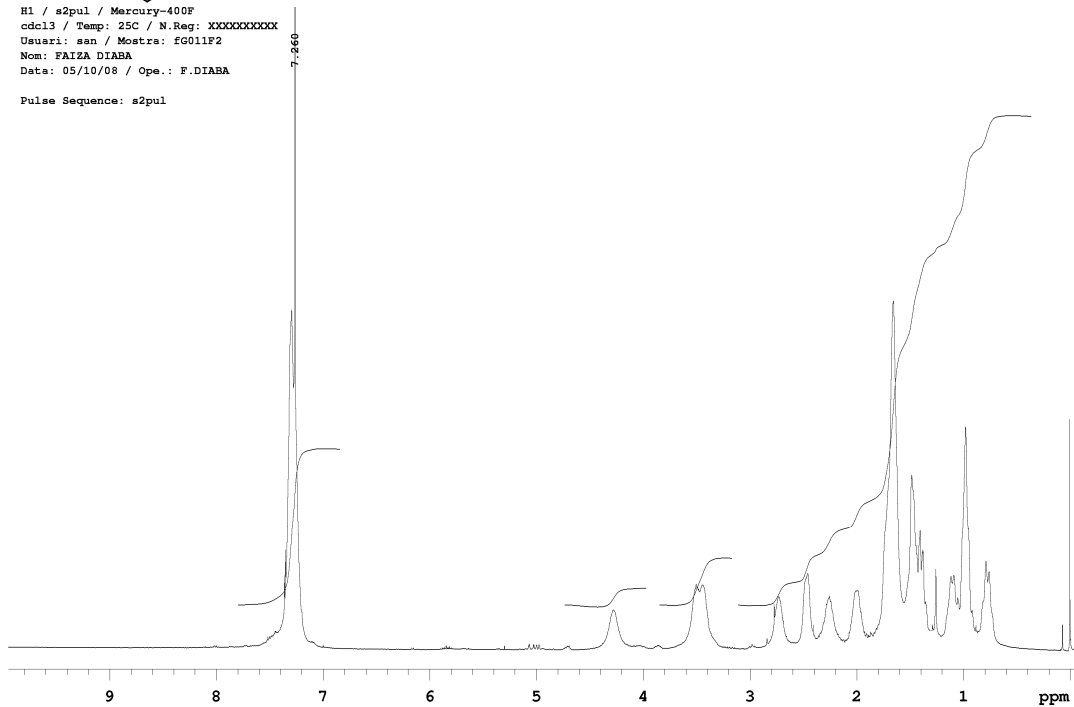


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: tB006G3
Nom: FAIZA DIABA
Data: 20/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

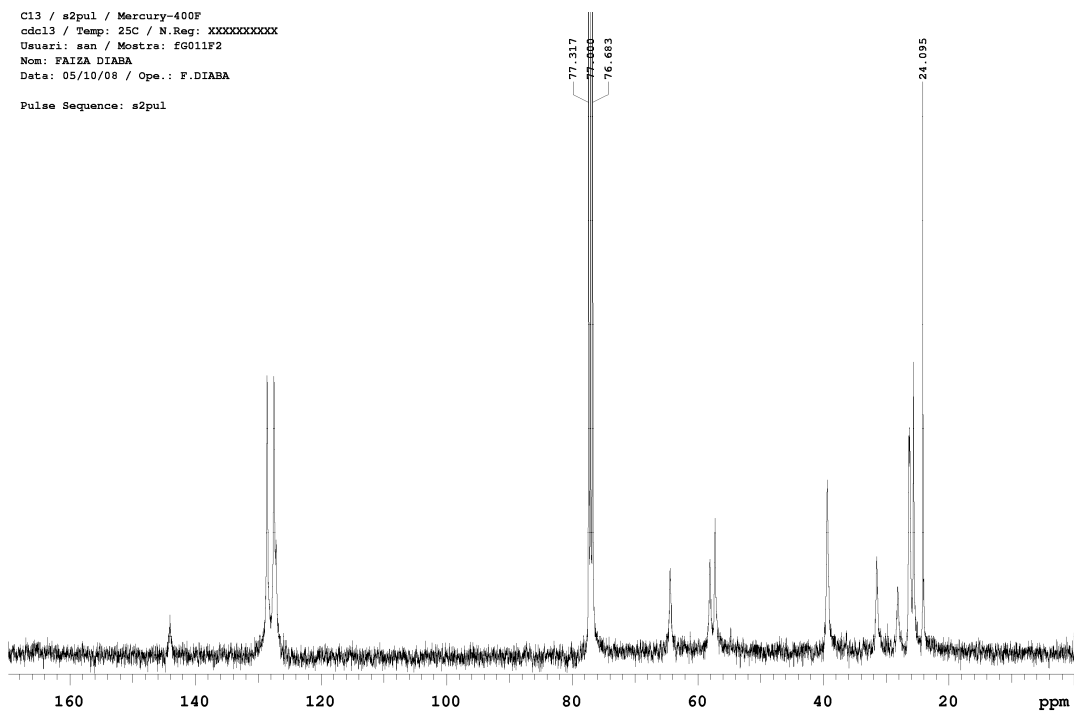


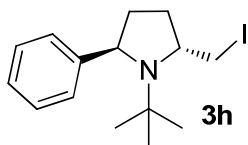


H1 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: fg011F2
Nom: FAIZA DIABA
Data: 05/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

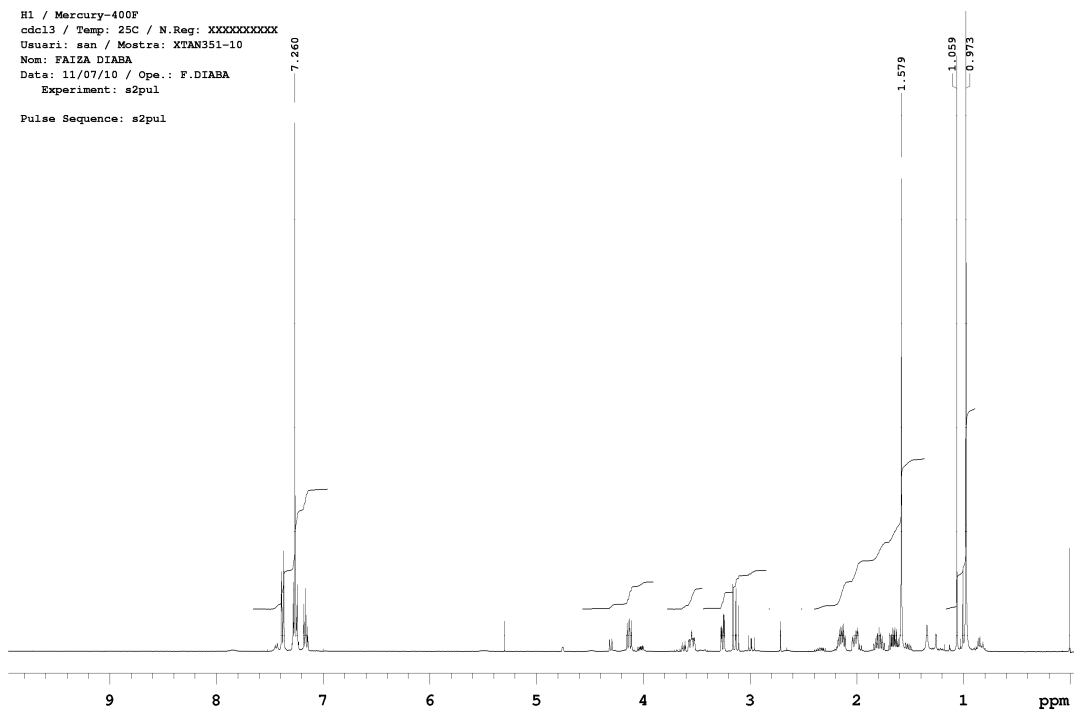


C13 / s2pul / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: fg011F2
Nom: FAIZA DIABA
Data: 05/10/08 / Ope.: F.DIABA
Pulse Sequence: s2pul

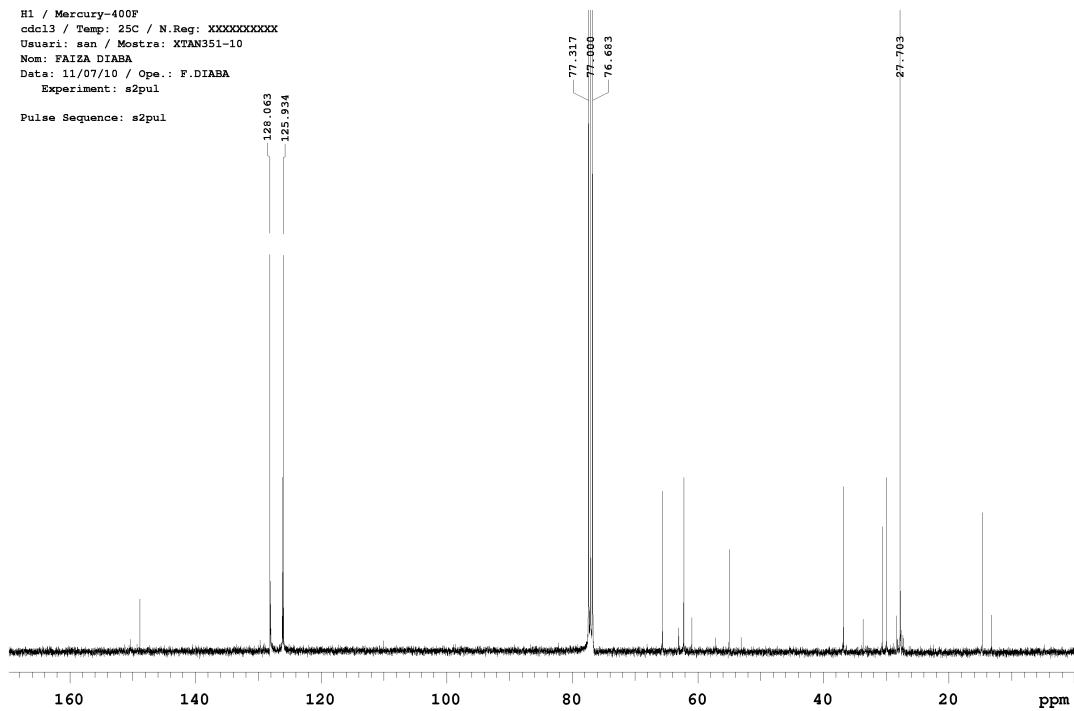


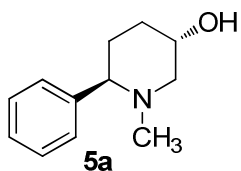


H1 / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: XTAN351-10
Nom: FAIZA DIABA
Data: 11/07/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

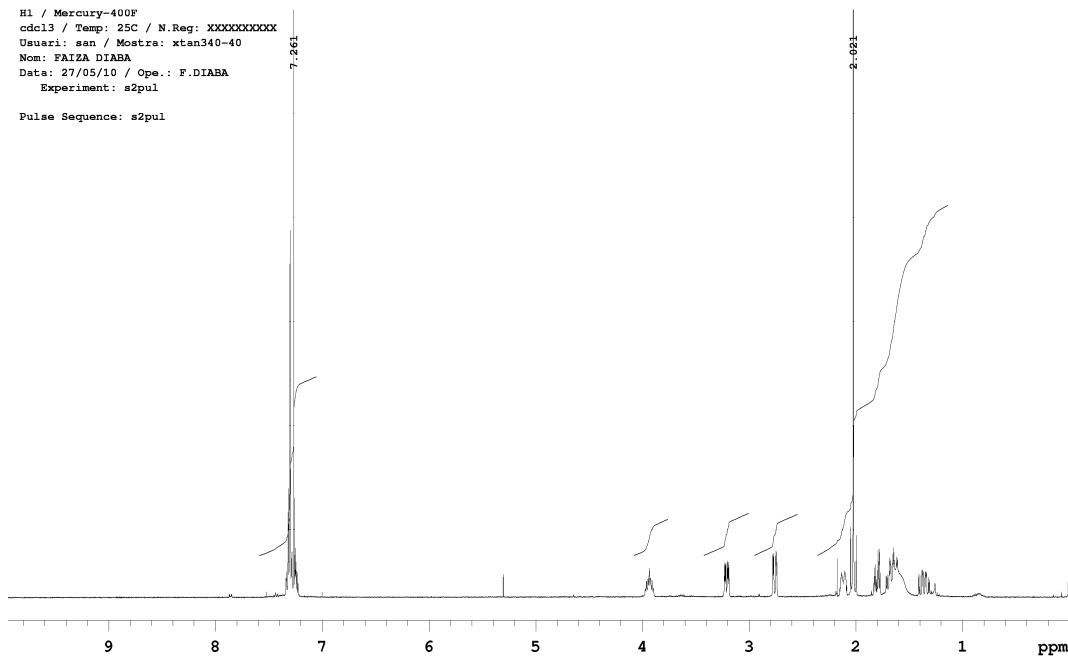


H1 / Mercury-400F
cdc13 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: XTAN351-10
Nom: FAIZA DIABA
Data: 11/07/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

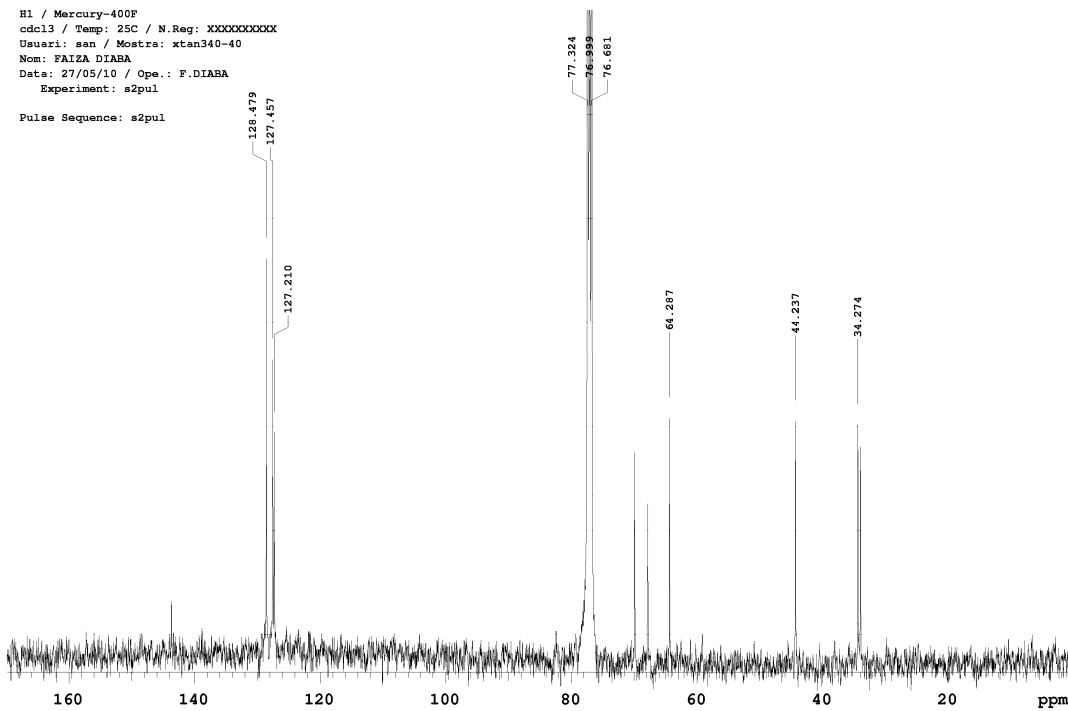


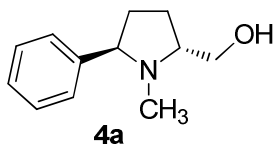


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: xtan340-40
Nom: FAIZA DIABA
Data: 27/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

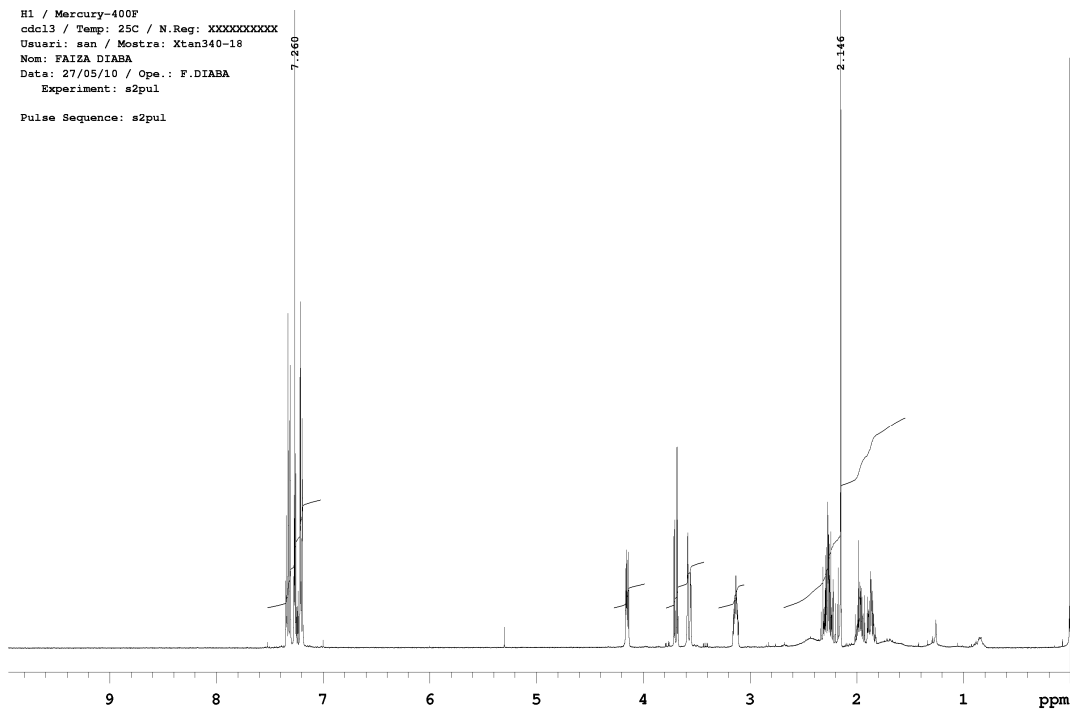


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: xtan340-40
Nom: FAIZA DIABA
Data: 27/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

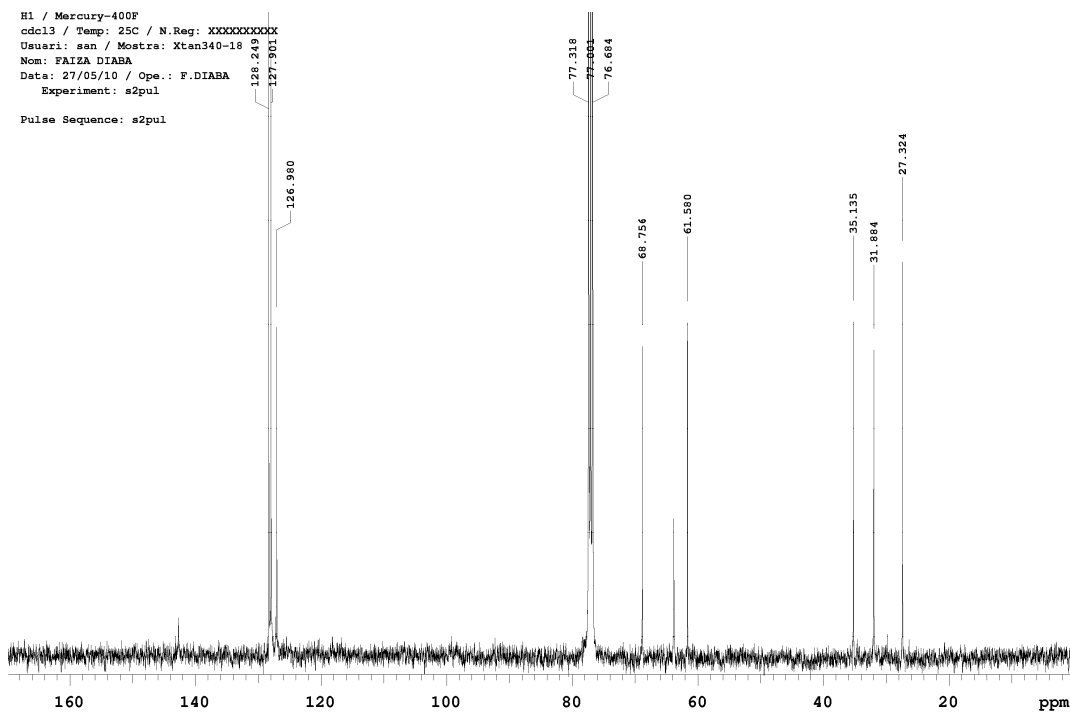


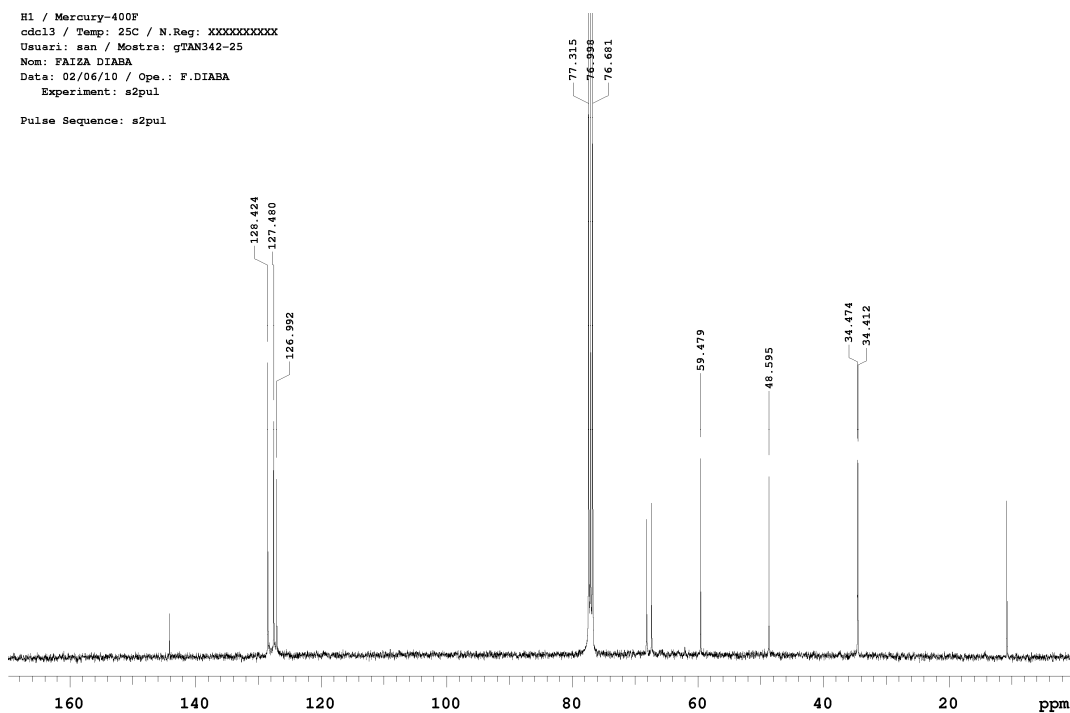
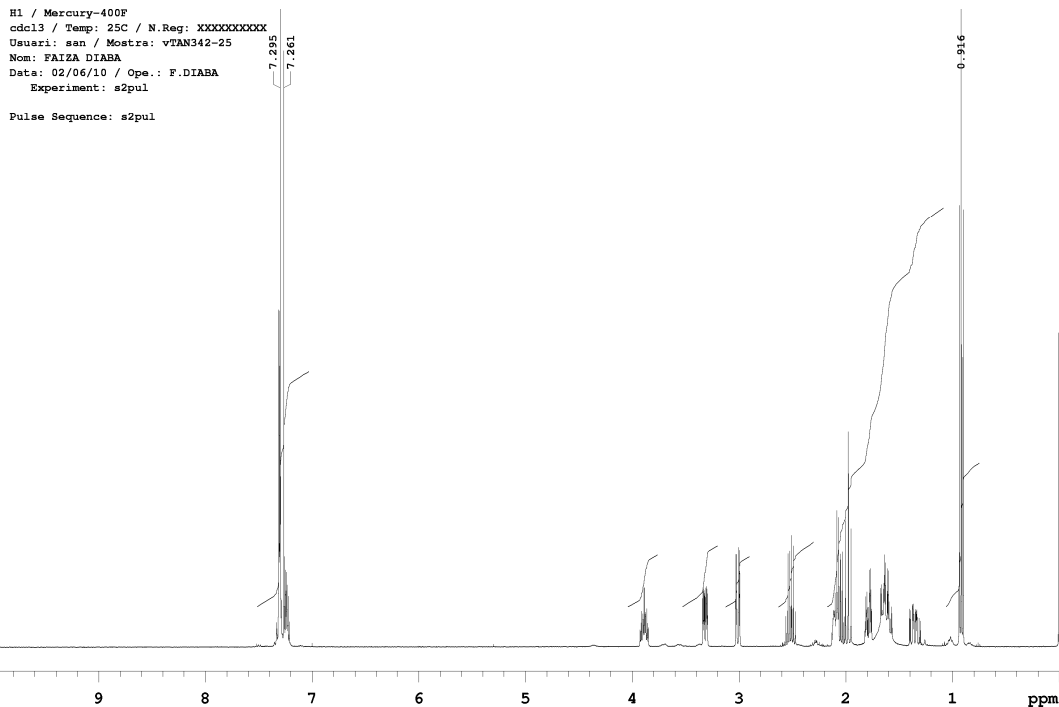
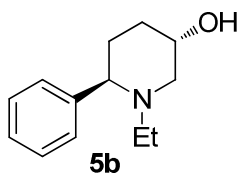


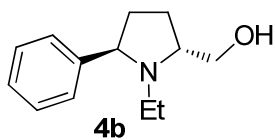
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: Xtan340-18
Nom: FAIZA DIABA
Data: 27/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul



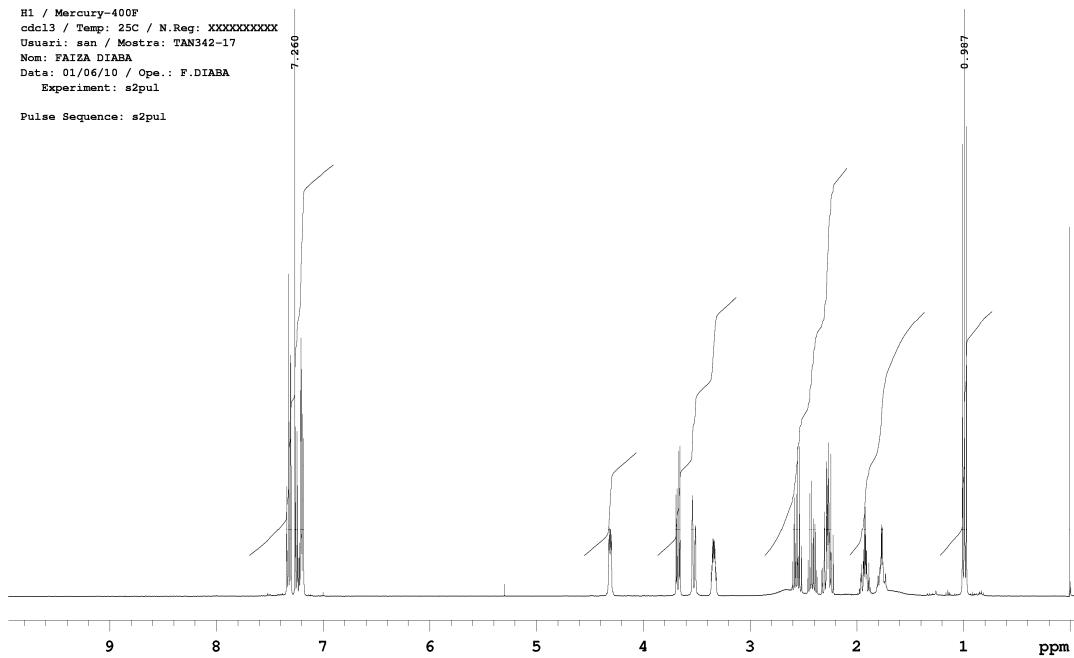
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: Xtan340-18
Nom: FAIZA DIABA
Data: 27/05/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul



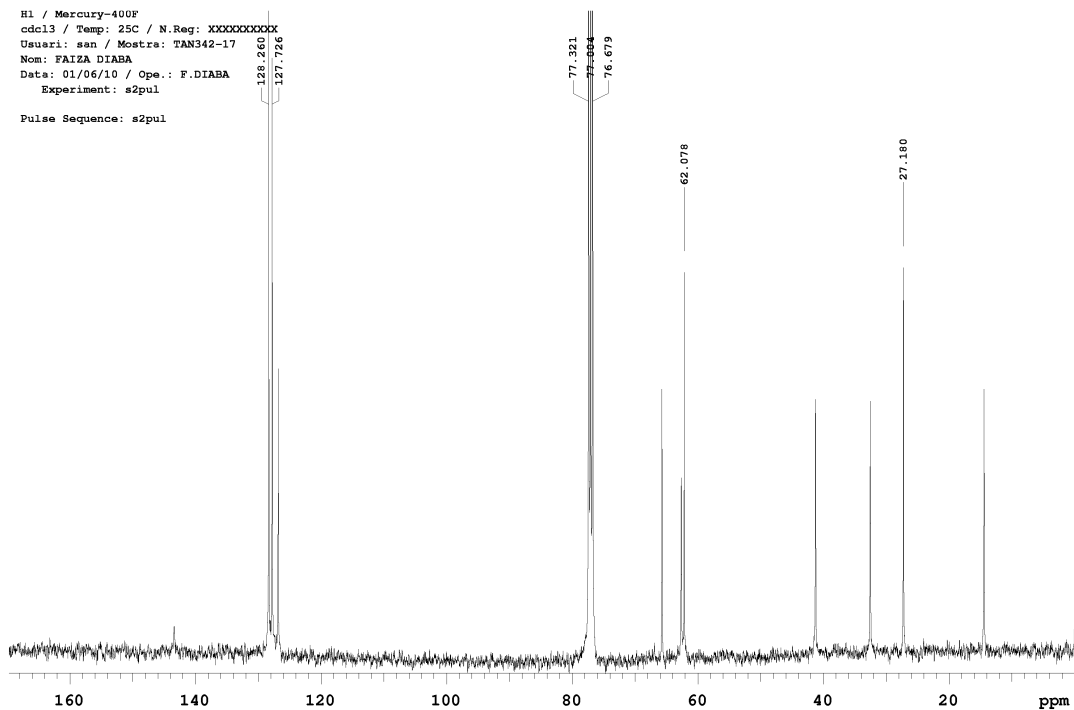


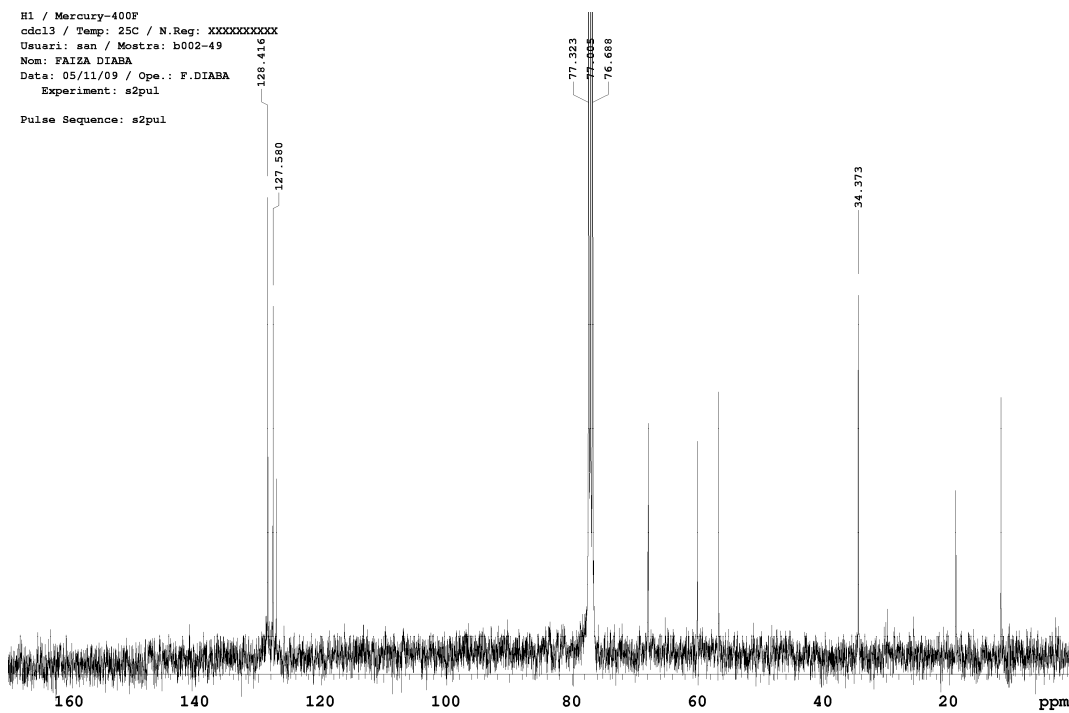
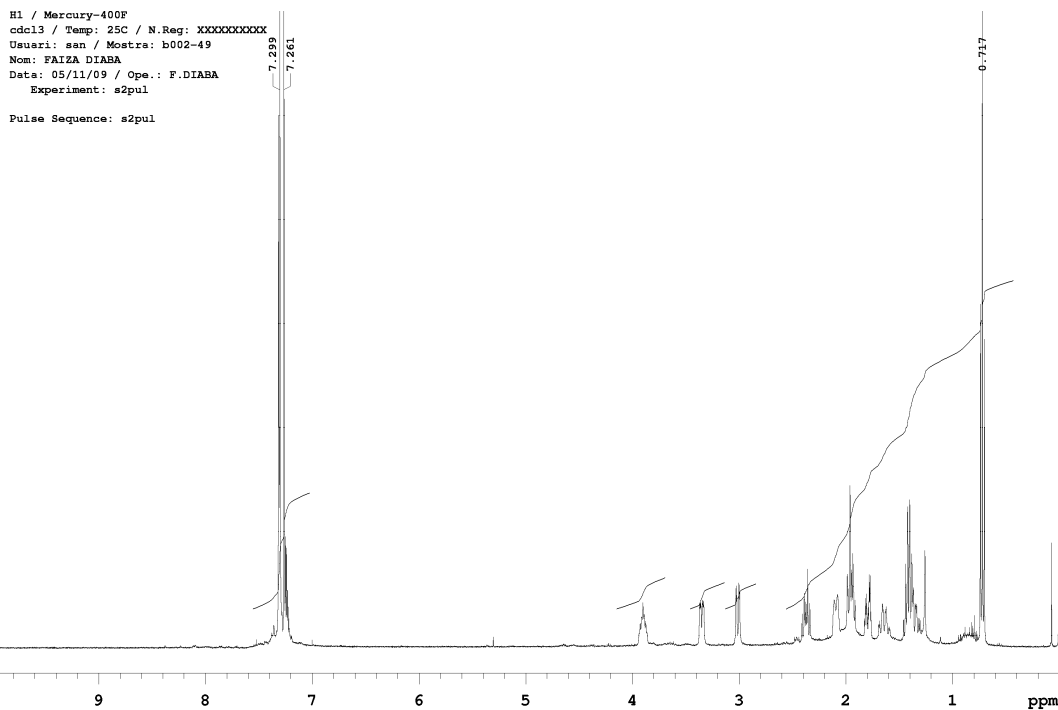
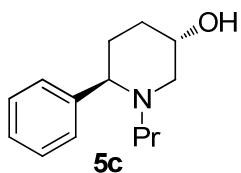


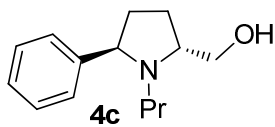
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: TAN342-17
Nom: FAIZA DIABA
Data: 01/06/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul



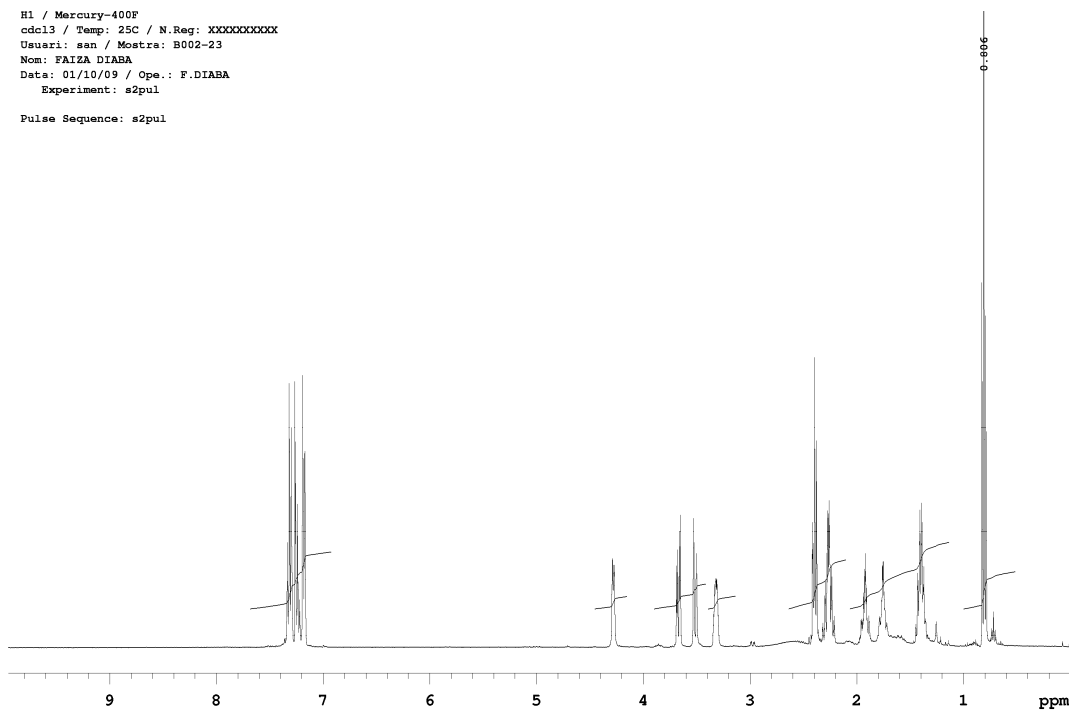
H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: TAN342-17
Nom: FAIZA DIABA
Data: 01/06/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul



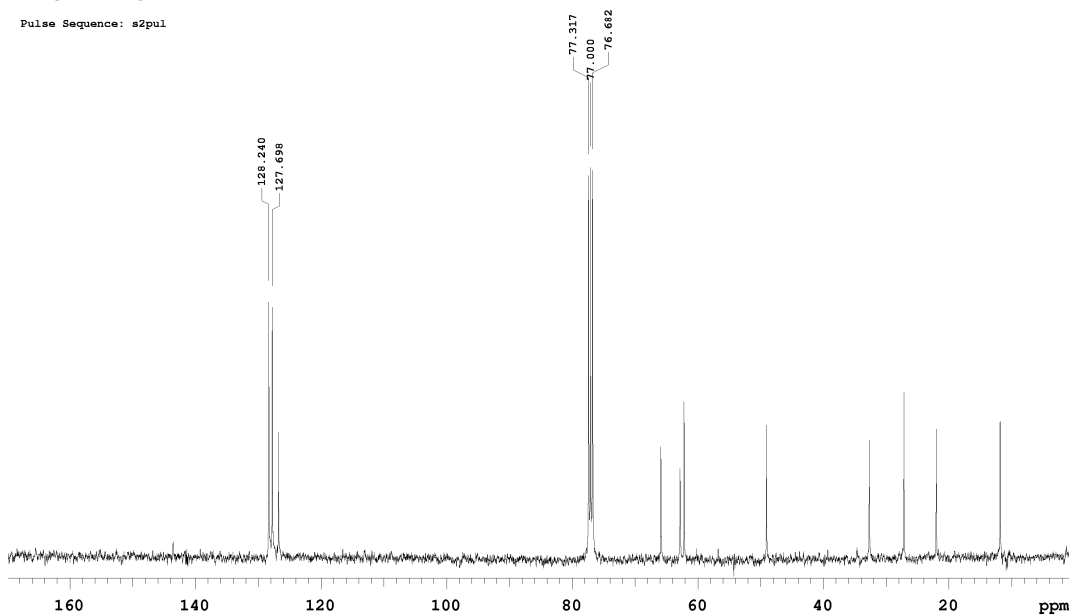


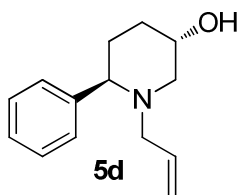


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: B002-23
Nom: FAIZA DIABA
Data: 01/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

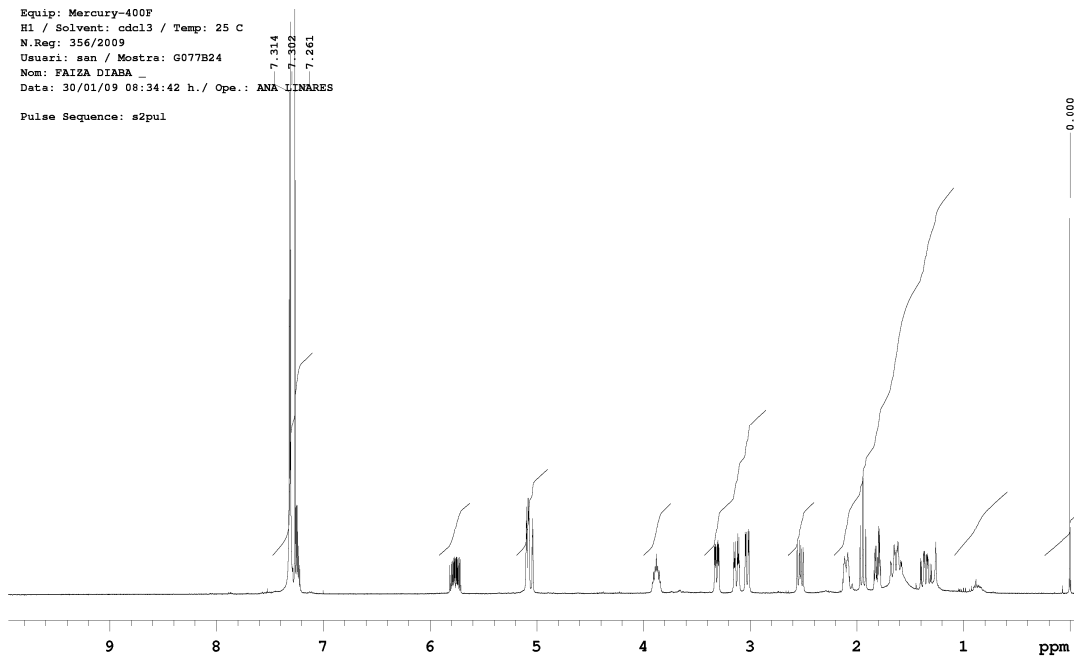


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: B002-23
Nom: FAIZA DIABA
Data: 01/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

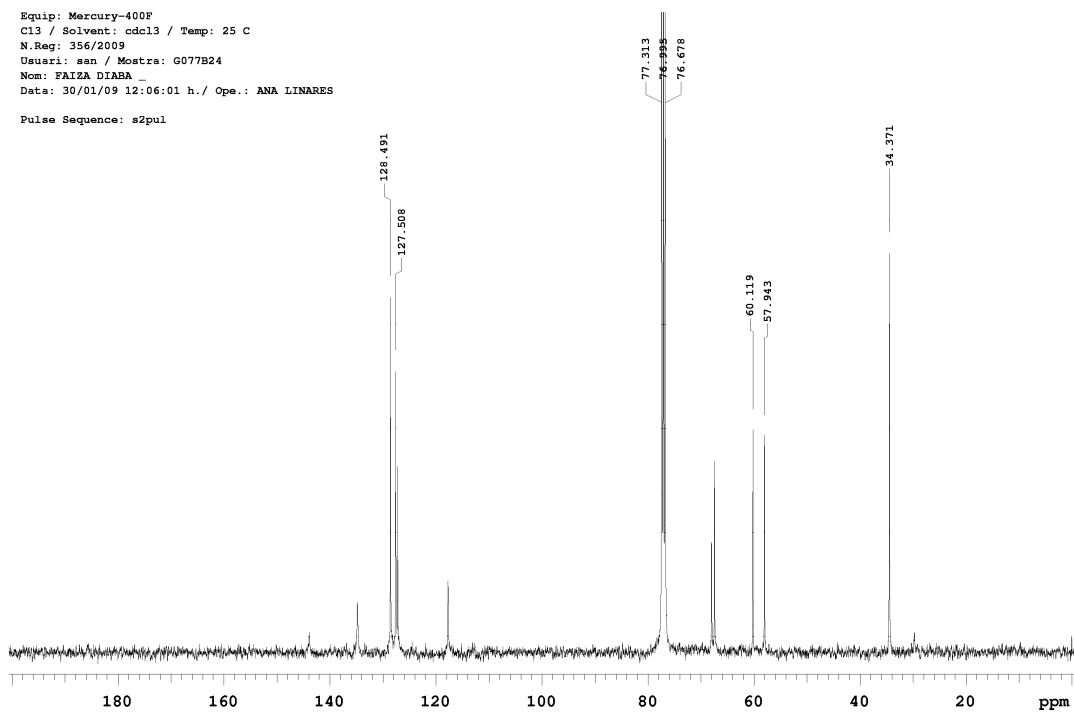


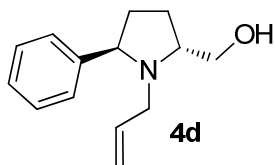


Equip: Mercury-400F
H1 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 356/2009
Usuari: san / Mostra: G077B24
Nom: FAIZA DIABA _
Data: 30/01/09 08:34:42 h. / Ope.: ANA LINARES
Pulse Sequence: s2pul

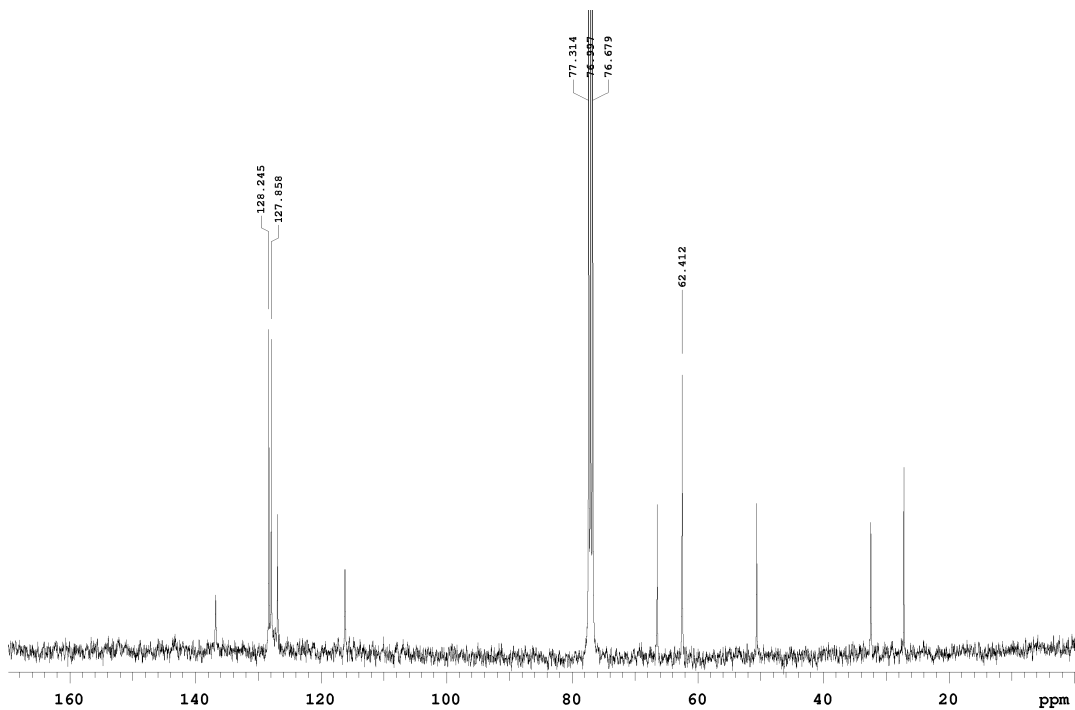
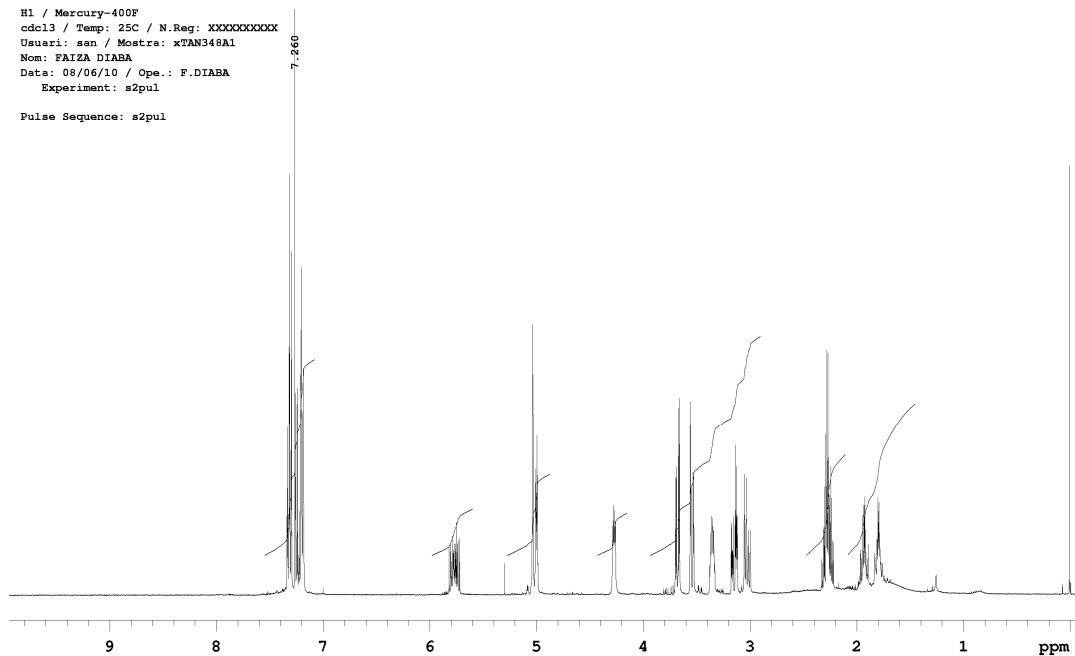


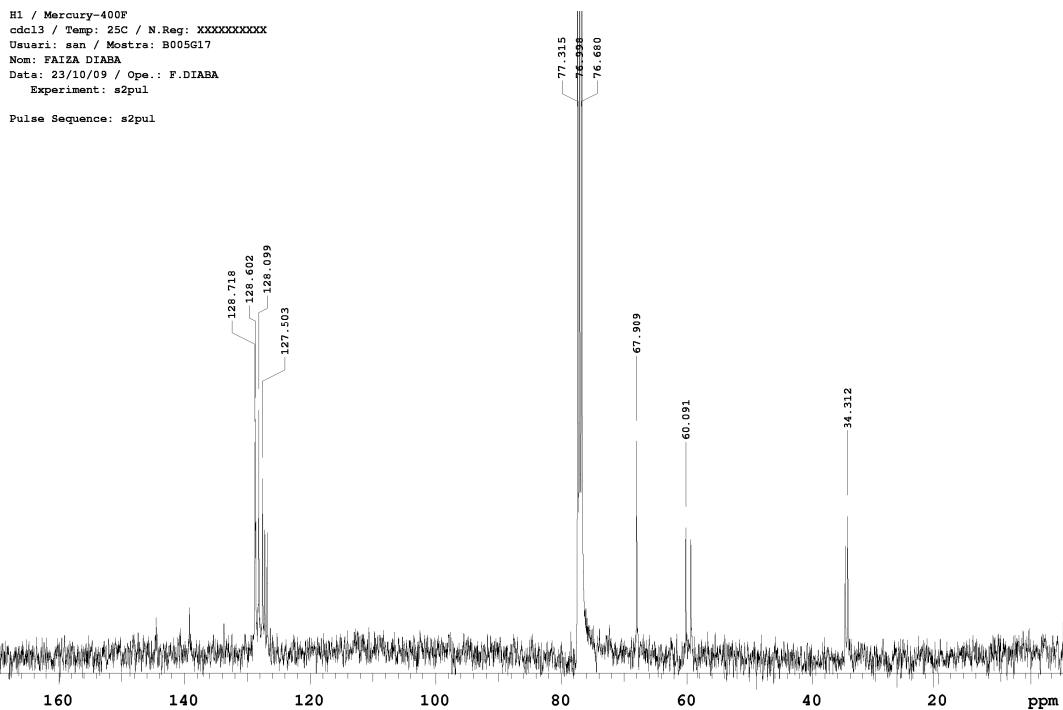
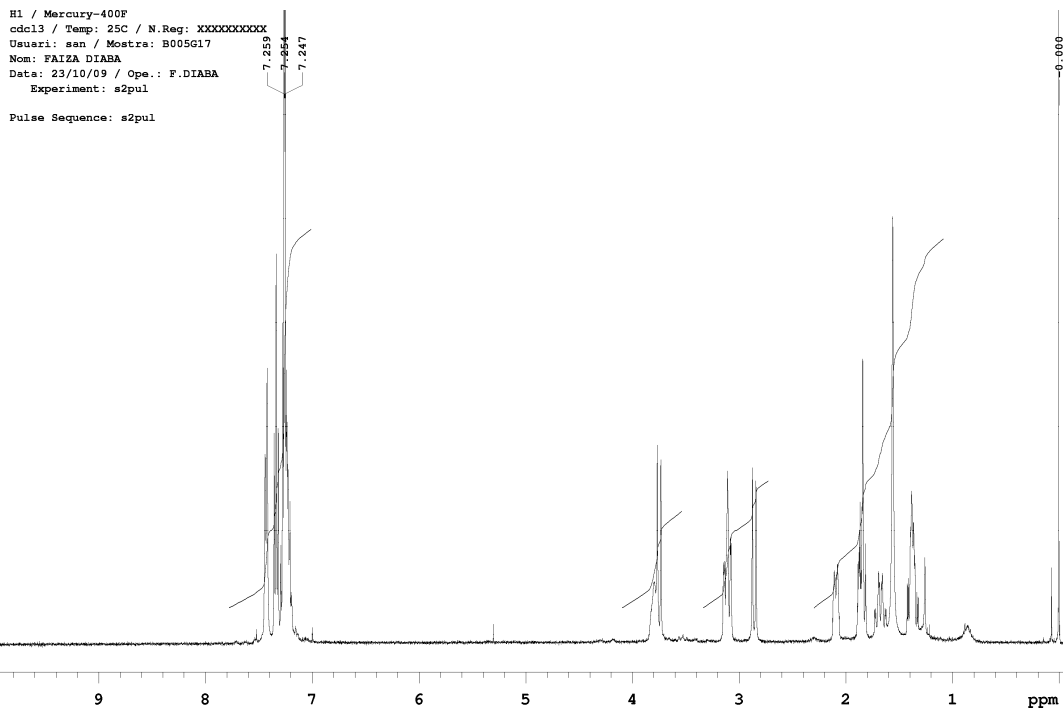
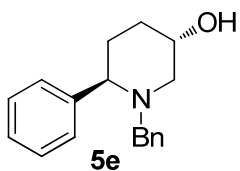
Equip: Mercury-400F
C13 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 356/2009
Usuari: san / Mostra: G077B24
Nom: FAIZA DIABA _
Data: 30/01/09 12:06:01 h. / Ope.: ANA LINARES
Pulse Sequence: s2pul

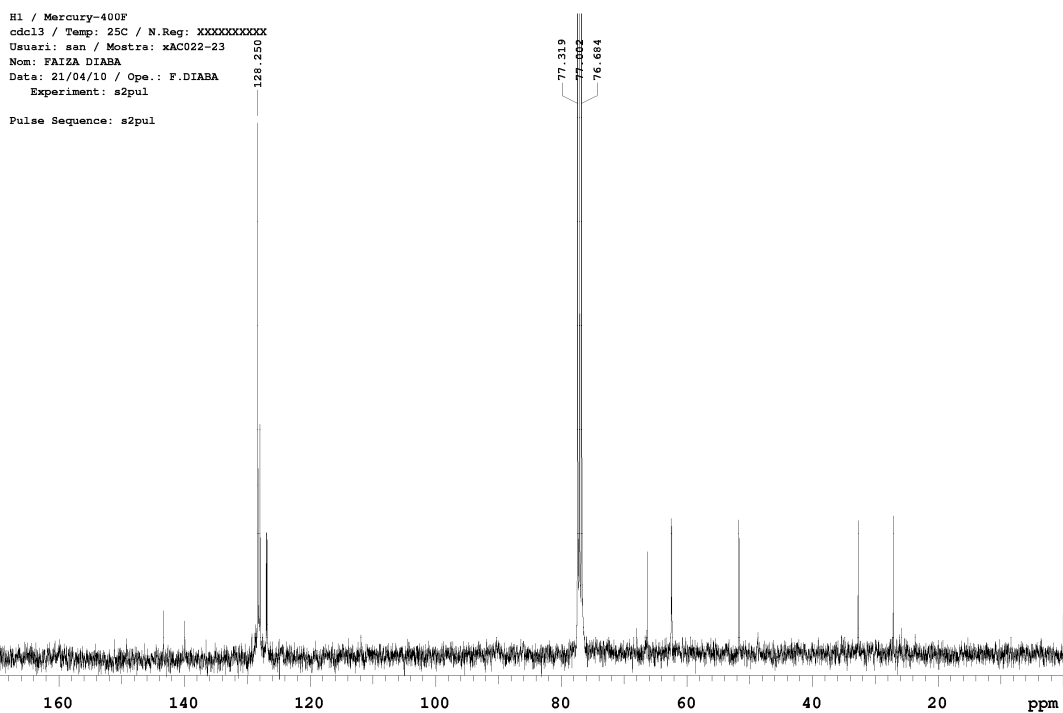
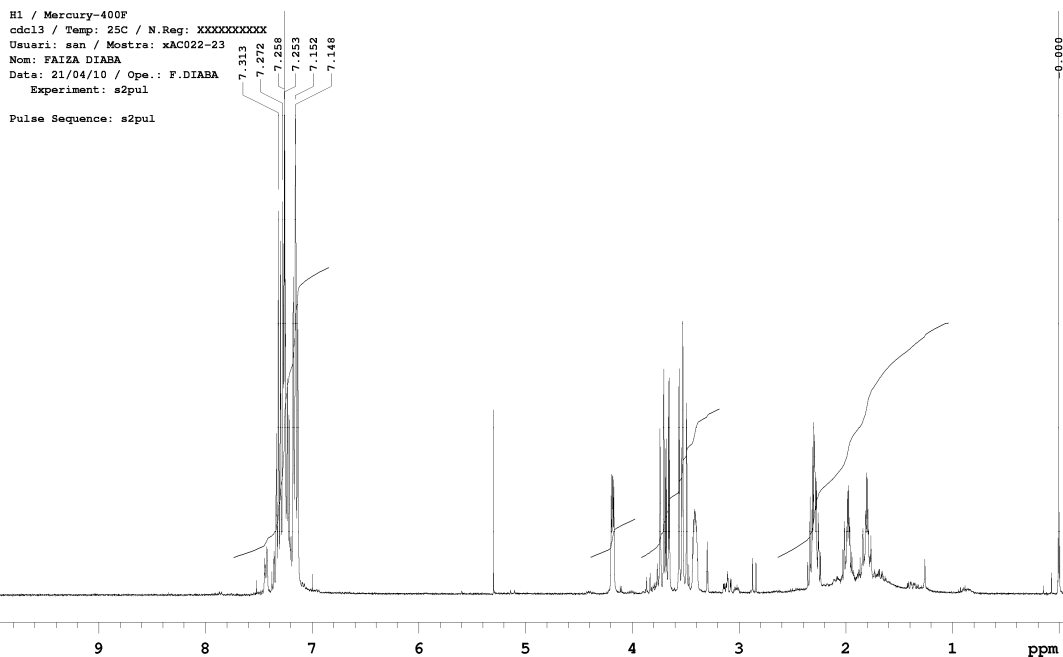
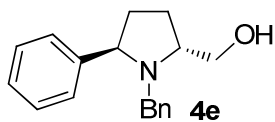


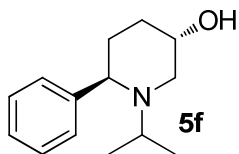


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XTAN348A1
Nom: FAIZA DIABA
Data: 08/06/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

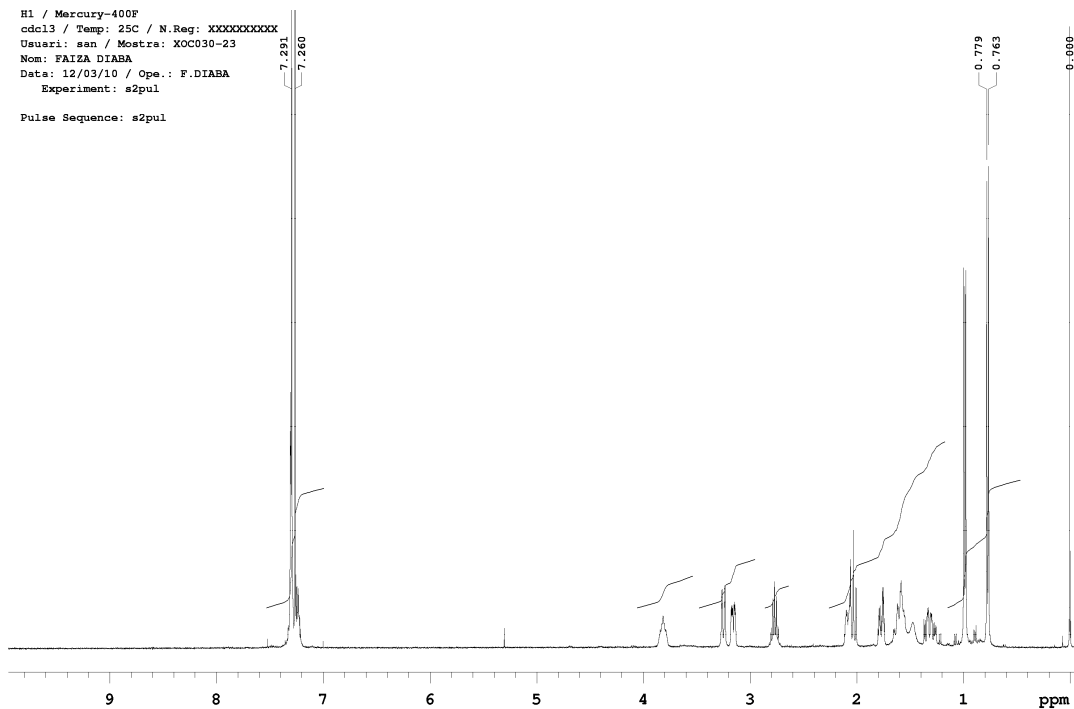




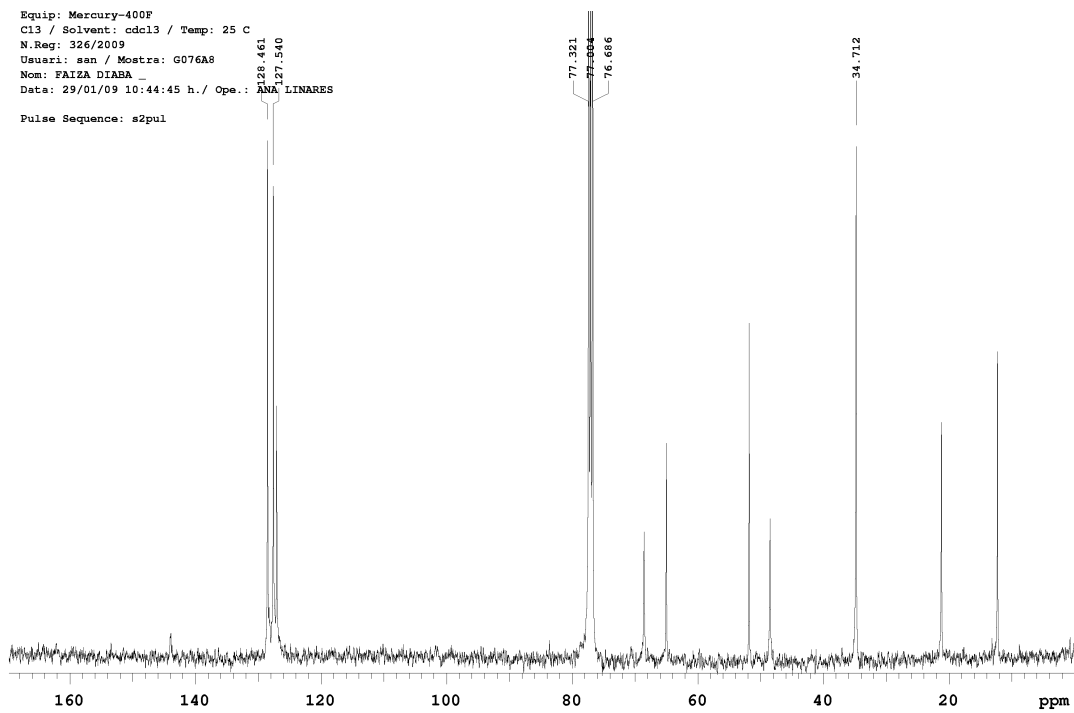


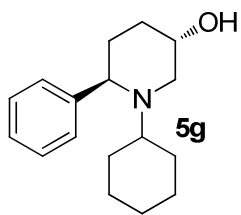


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: sen / Mostra: XOC030-23
Nom: FAIZA DIABA
Data: 12/03/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

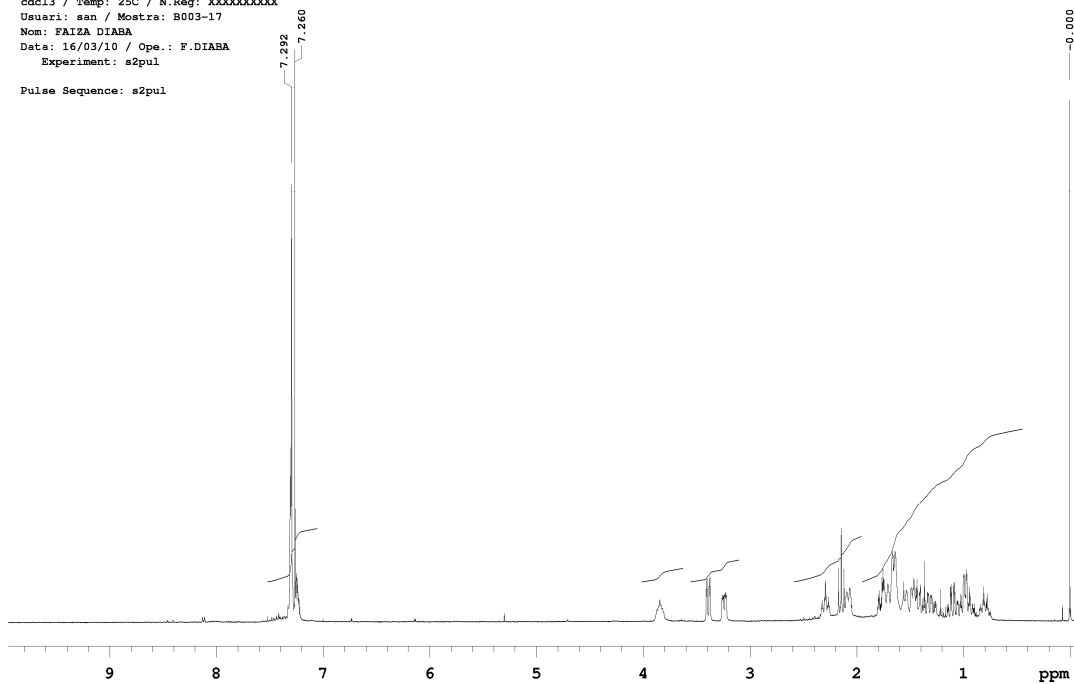


Equip: Mercury-400F
C13 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 326/2009
Usuari: sen / Mostra: G076A8
Nom: FAIZA DIABA _
Data: 29/01/09 10:44:45 h. / Ope.: ANA LINARES
Pulse Sequence: s2pul

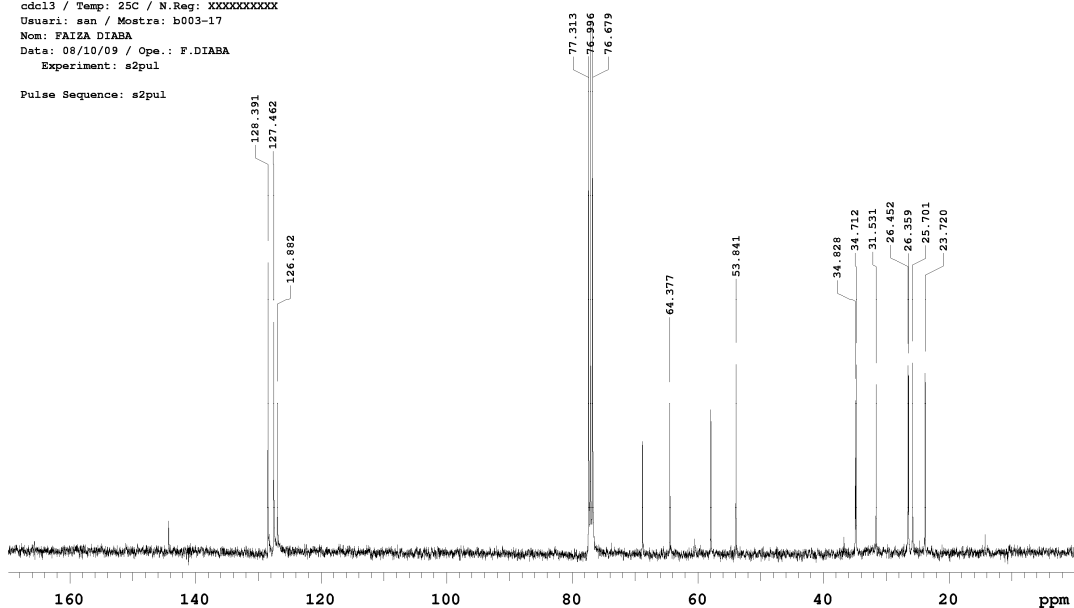


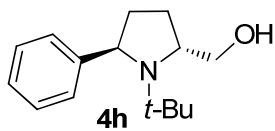


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: B003-17
Nom: FAIZA DIABA
Data: 16/03/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

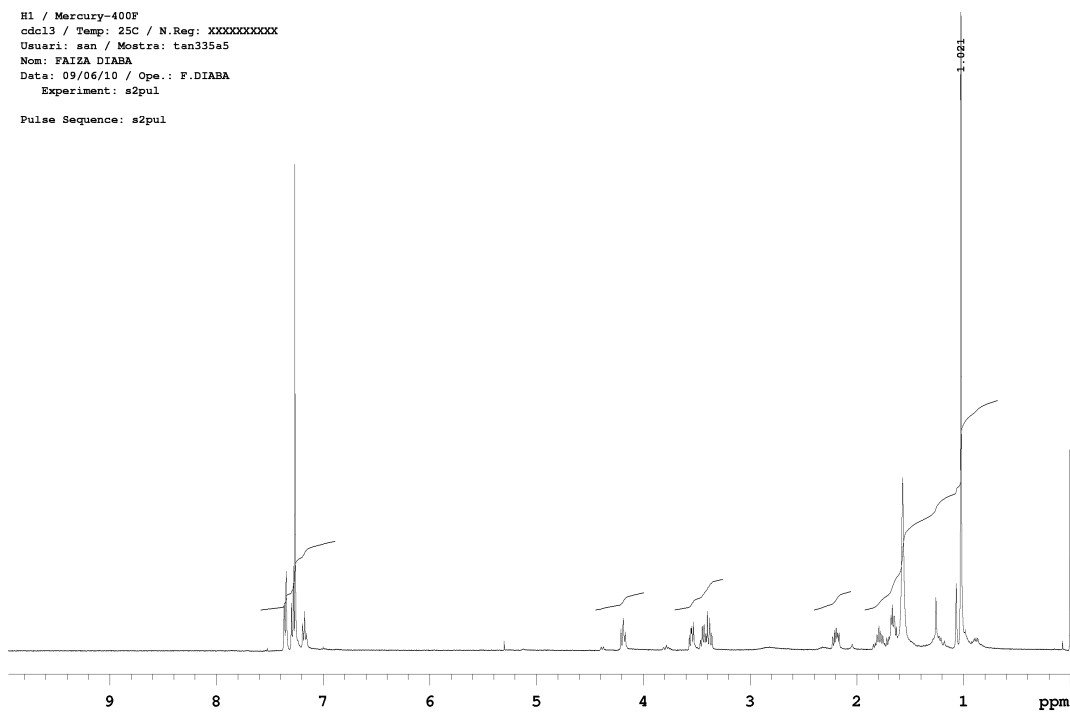


H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: b003-17
Nom: FAIZA DIABA
Data: 08/10/09 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul





H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: tan335a5
Nom: FAIZA DIABA
Data: 09/06/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul



H1 / Mercury-400F
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXXXX
Usuari: san / Mostra: tan335a5
Nom: FAIZA DIABA
Data: 09/06/10 / Ope.: F.DIABA
Experiment: s2pul
Pulse Sequence: s2pul

