

## SUPPORTING INFORMATION

### **Synthesis of the 10-oxabicyclo[5.2.1]decane framework, present in bioactive natural products**

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and Gabriele Kociok-Köhn*

**Document S1:** Protocols of synthesis for compounds **4** to **9**.

**Document S2:** Anticancer *in vitro* testing protocol.

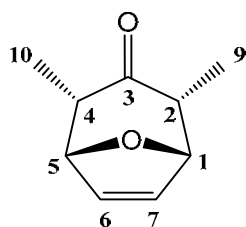
**Document S3:** Spectra of compounds **4** to **17** described in the present work (IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, DEPT, COSY, HSQC and MS).

## DOCUMENT S1

### PROTOCOLS OF SYNTHESIS FOR COMPOUNDS 4 TO 9

(The numbering of the following sections continues in the main text of the article)

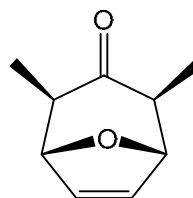
#### *S1.1. Synthesis of cycloadduct 2,4-dimethyl-8-oxabicyclo[3.2.1]oct-6-en-3-one, (4)*



**4a**

*diequatorial*

**(1*R*\*, 2*S*\*, 4*R*\*, 5*S*\*)**



**4b**

*diaxial*

**(1*R*\*, 2*R*\*, 4*S*\*, 5*S*\*)**

#### *Copper activation:*

Copper (24 g, 378 mmol) was added to a 2% solution of I<sub>2</sub> in acetone (240 mL). The solution was stirred for 20 minutes and filtered through a porous plate. The solid was first washed with a 37% HCl solution and acetone in a 1:1 ratio (100 mL) and then with acetone (60 mL). Finally, the obtained solid (23.5 g) was dried at high vacuum for several hours.

#### *Cycloaddition reaction:*

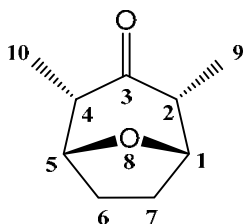
In a 500 mL three necked flask, fitted with a condenser and a pressure equalizing addition funnel, dry acetonitrile (220 mL), NaI (101.62 g, 678 mmol), activated copper (23.69 g, 373 mmol) and furan (32 mL, 30.08 g, 442 mmol) were placed. The funnel was charged with 2,4-dibromopentanone, **2**, (27.56 g, 113 mmol) and acetonitrile (60 mL) as a solvent. The solution was added rapidly and the crude was kept between 50 and 60 °C for 3.5 hours till the reaction controlled by GC reached a 100 % conversion. The flask was cooled to 0 °C, and CH<sub>2</sub>Cl<sub>2</sub> (100 mL) were added while stirring. The reaction mixture was then poured into a 2 L beaker containing water (500 mL) and ice (500 mL). The mixture was stirred rapidly until the ice melted. The mixture was filtered by a Büchner and washed with CH<sub>2</sub>Cl<sub>2</sub> (50 mL). The filtrate was transferred to a 2 L separatory funnel while still cold. Extraction with CH<sub>2</sub>Cl<sub>2</sub> was done several times till the organic phase showed no more colour. The organic phases were combined together and extracted with an aqueous solution of NH<sub>3</sub> (35%) until the aqueous phase became transparent (no blue colour was observed), meaning that no more amino copper complex (blue) was present. The organic phase was then extracted with water, dried over anhydrous MgSO<sub>4</sub> and concentrated to dryness to afford 16.85 g (98% yield) of a mixture containing, by GC, 92.5 % of the diequatorial product, **4a** and 7.23 % of the diaxial product, **4b**. Both diastereoisomers are

useful for the synthetic purpose, and the mixture may be used for the next step. However, these stereoisomers were easily separated by conventional flash column chromatography on silica gel, eluting with hexane and ethyl acetate or alternatively by crystallization in hexane at  $-20\text{ }^{\circ}\text{C}$ , due to the preferential crystallization of the major isomer.

**4a:** Colourless oil at rt. **IR** (film,  $\text{cm}^{-1}$ ):  $\nu_{\text{max}}$  3405 (C=O, Fermi's resonance), 3085 (H—Csp<sup>2</sup>, st), 2971, 2876 (H—Csp<sup>3</sup>, st), 1713 (C=O, st), 1449 (C—C, deform.), 1155, 1053 (C—O, st). **<sup>1</sup>H NMR** (200 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  0.96 (6H, d,  $J_1 = 7\text{ Hz}$ , H9 and H10), 2.78 (2H, q,  $J_1 = 7\text{ Hz}$ ,  $J_2 = 4.8\text{ Hz}$ , H2 and H4), 4.84 (2H, d,  $J_2 = 4.8\text{ Hz}$ , H6 and H7), 6.34 (2H, s, H1 and H5). **<sup>13</sup>C NMR** (50 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  10.1 (C9, C10), 50.3 (C2, C4), 82.7 (C6, C7), 133.5 (C1, C5), 208.9 (C3). **MS** (DIP-CI-NH<sub>3</sub>, 70 eV, 150°C):  $m/z$  (%) 152 (4, M), 170 (100, M+NH<sub>4</sub>), 183 (38, M+N<sub>2</sub>H<sub>7</sub>). **Anal.** Calcd for C<sub>9</sub>H<sub>12</sub>O<sub>2</sub> (%): C, 71.06; H, 7.89. Found: C, 70.88; H, 7.64. **GC** (Conditions, type D):  $t_R = 9.15\text{ min}$ . **TLC** (SiO<sub>2</sub>; hexane : ethyl acetate, 7:3):  $R_F = 0.73$  (3 elutions and development with anisaldehyde reagent).

**4b:** Colourless oil at rt. **IR** (film,  $\text{cm}^{-1}$ ):  $\nu_{\text{max}}$  3092 (H—Csp<sup>2</sup>, st), 2975, 2938, 2878 (H—Csp<sup>3</sup>, st), 1711 (C=O, st), 1458 (C—C, deform.), 1180, 1092 (C—O, st). **<sup>1</sup>H NMR** (200 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  1.36 (6H, d,  $J = 7.5$ , H9 and H10), 2.28 (2H, q,  $J = 7.5$ , H2 and H4), 4.65 (2H, s, H6 and H7), 6.27 (2H, s, H1 and H5). **<sup>13</sup>C NMR** (50 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  17.7 (C9 and C10), 49.8 (C2 and C4), 82.0 (C6 and C7), 133.6 (C1 and C5), 213.7 (C3). **MS** (DIP-CI-NH<sub>3</sub>, 70 eV, 150°C):  $m/z$  (%) 152 (6, M), 170 (100, M+NH<sub>4</sub>), 183 (38, M+N<sub>2</sub>H<sub>7</sub>). **Anal.** Calcd for C<sub>9</sub>H<sub>12</sub>O<sub>2</sub> (%): C, 71.06; H, 7.89. Found: C, 70.95; H, 7.83. **GC** (Conditions, type D):  $t_R = 9.54\text{ min}$ . **TLC** (SiO<sub>2</sub>; hexane / ethyl acetate, 7:3):  $R_F = 0.63$  (3 elutions and development with anisaldehyde reagent).

### *S1.2. Synthesis of cycloadduct (1R\*,2S\*,4R\*,5S\*)-2,4-dimethyl-8-oxabicyclo[3.2.1]oct-6-en-3-one, (5a).*

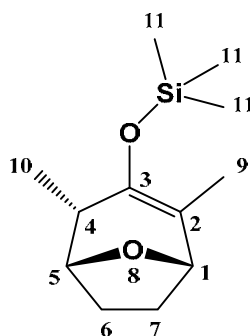


In a 500 mL round-bottomed flask, 10% Pd/C (3.29 g) was placed under a continuous flow of argon in order to preserve the catalyst. Subsequently, absolute ethanol (100 mL) was added and the system was purged again with argon. In another flask, compound **4a** (12.19 g, 80 mmol), was dissolved in absolute ethanol (200 mL) and the organic solution was transferred via cannula to the flask containing the catalyst, by using a positive pressure of argon. The reaction mixture was then submitted to 10 cycles of vacuum and hydrogen in order to remove the possible oxygen present in the system. The crude was

stirred at room temperature with a positive pressure of hydrogen for 16 hours. The reaction was controlled by GC and once complete the crude was first filtered with a funnel containing a double paper filter and then through a Celite<sup>®</sup> column in order to separate all the Pd from the organic solution. The column was washed with ethanol, and the obtained organic fraction was concentrated to dryness to afford 12.10 g of hydrogenated product, **5a** (98% yield, 100% conversion).

Colourless oil. **IR** (film, cm<sup>-1</sup>):  $\nu_{max}$  3407 (C=O, Fermi's resonance), 2973, 2880 (H—Csp<sup>3</sup>, st), 1713 (C=O, st), 1472 (C—C, deform.), 1379, 1155, 1045 (C—O, st), 1026, 951, 930. **<sup>1</sup>H NMR** (200 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  0.87 (6H, d,  $J$  = 7 Hz, H9 y H10), 1.54-1.79 (4H, m, H6 and H7), 2.72 (2H, qd,  $J_1$  = 7 Hz,  $J_2$  = 7 Hz, H2 and H4), 4.39-4.42 (2H, m, H1 and H5). **<sup>13</sup>C NMR** (50 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  9.6 (C9 and C10), 24.8 (C6 and C7), 50.3 (C2 and C4), 80.9 (C1 and C5), 210.2 (C3). **MS** (DIP-CI-NH<sub>3</sub>, 70 eV, 150°C):  $m/z$  (%): 154 (7, M), 172 (100, M+NH<sub>4</sub>), 185 (52, M+N<sub>2</sub>H<sub>7</sub>). **Anal.** Calcd for C<sub>9</sub>H<sub>14</sub>O<sub>2</sub> (%): C, 70.13; H, 9.09. Found: C, 69.98; H, 8.81. **GC** (Conditions, type D):  $t_R$  = 9.95 min. **TLC** (SiO<sub>2</sub>; hexane : ethyl acetate, 7:3):  $R_F$  = 0.62.

### S1.3. Synthesis of {(1*S*\*,4*S*\*,5*R*\*)-2,4-dimethyl-8-oxa-bicyclo[3.2.1]oct-2-en-3-yloxy}trimethylsilane, (**6a**)

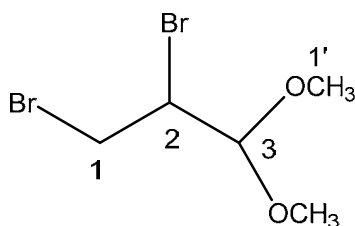


In a 500 mL three neck round bottom flask previously dried by heating under vacuum and purged with argon, fitted with a magnetic stirrer and a pressure equalizing addition funnel, anhydrous THF (150 mL) and pure diisopropylamine (99+ %) (24.42 mL) were placed. The solution was cooled to -78 °C and butyllithium (1.6 M in THF, 71.1 mL) was introduced into the funnel. The base was added dropwise and the mixture was left to react at -78 °C for 30 minutes. Subsequently, compound **5a** (15.8 g, 102.5 mmol), previously dissolved in anhydrous THF (60 mL) was slowly added via cannula to the reaction flask. The temperature of the system was raised to 0 °C and it was left to react for two hours. At this point, trimethylsilylchloride (17.50 mL) was added to the reaction flask by a syringe and the system was first maintained at 0 °C for 15 minutes and then at room temperature for 2 hours. The crude obtained was filtered via cannula to separate the salt (LiCl) generated in the reaction. The organic solution was then concentrated to dryness and the solid redissolved in hexane (20 mL). The heterogeneous solution was cooled

down to  $-20\text{ }^{\circ}\text{C}$ , in order to induce the precipitation of the remaining salt, and then filtered via cannula. This operation was repeated till no salt was present in the organic fraction. The final organic fraction was rotary evaporated to afford 23.20 g of the desired silyl enol ether **6a** (99% yield, 100% conversion).

Colourless oil. **IR** (film,  $\text{cm}^{-1}$ ):  $\nu_{\text{max}}$  2959, 2869 ( $\text{H}-\text{Csp}^3$ , st), 1680 ( $\text{C}=\text{C}$ , st), 1460 ( $\text{C}-\text{C}$ , deform.), 1252, 1202, 1122, 1049 ( $\text{C}-\text{O}$ , st), 893, 843 ( $\text{Si}-\text{C}$ , st).  **$^1\text{H}$  NMR** (200 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  0.18 (9H, s, H11), 0.91 (3H, d,  $J = 7.2\text{ Hz}$ , H9), 1.52 (3H, d,  $J = 2.2\text{ Hz}$ , H10), 1.70-1.90 (4H, m, H6 and H7), 2.78-2.86 (1H, m, H4), 4.25 (1H, s, H1), 4.30-4.40 (1H, dd, H5).  **$^{13}\text{C}$  NMR** (50 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  2.5 (C11), 11.6 (C10), 12.3 (C9), 22.6 (C7), 32.5 (C6), 39.0 (C4), 76.9 (C5), 78.9 (C1), 126.3 (C2), 127.9 (C3). **MS** (DIP-Cl-NH<sub>3</sub>, 70 eV,  $150^{\circ}\text{C}$ ):  $m/z$  (%) 226 (100, M), 227 (24, M+H), 244 (25, M+NH<sub>4</sub>). **HRMS** Calcd for  $\text{C}_{12}\text{H}_{22}\text{O}_2\text{Si}$ : 226.13891. Found: 226.13895. **TLC** ( $\text{SiO}_2$ ; hexane : ether, 9:1):  $R_F = 0.28$ .

#### S1.4. Synthesis of 1,2-dibromo-3,3-dimethoxypropane, (**8**)

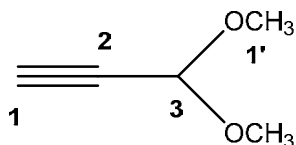


A 100 mL three-necked flask was equipped with a magnetic stirrer, a pressure-equalizing addition funnel, a condenser fitted with a  $\text{CaCl}_2$  tube, and a thermometer. The flask and the addition funnel were charged with acrolein (24.7 mL, 370 mmol) and (19.5 mL, 60.50 g, 378 mmol) of bromine, respectively. Acrolein, **7**, was stirred rapidly and cooled to  $0\text{ }^{\circ}\text{C}$  in an ice bath, then bromine was added at a rate such that the temperature is kept between  $0$  and  $5\text{ }^{\circ}\text{C}$  (3 hours), until all bromine was added and the reaction mixture acquired a permanent red color. At this point, the funnel was charged with methyl orthoformate (60.72 mL, 555 mmol) and methanol (190 mL). The solution was added along 10-15 minutes. Then, the solution was heated to  $45\text{ }^{\circ}\text{C}$  for 3-4 hours after the removal of the addition funnel. Methyl orthoformate and methanol in excess were eliminated by evaporation using the rotary evaporator, obtaining 83.35 g of **8** (86% yield, 100% conversion).

Colourless oil. **IR** (film,  $\text{cm}^{-1}$ ):  $\nu_{\text{max}}$  2936, 2836 ( $\text{H}-\text{Csp}^3$ , st), 1734 ( $\text{C}=\text{O}$ , st), 1447 ( $\text{C}-\text{C}$ , deform.), 1358, 1240, 1221, 1198, 1115, 1067 ( $\text{C}-\text{O}$ , st), 966.  **$^1\text{H}$  NMR** (200 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  3.52 (6H, d,  $J = 4\text{ Hz}$ , H1'), 3.77-3.82 (2H, m, H1), 4.18-4.22 (1H, m, H2), 4.55 (1H, d,  $J = 4.4\text{ Hz}$ , H3).  **$^{13}\text{C}$  NMR** (50 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  33.1 (C1), 51.5 (C2), 56.4 (C1'), 104.2 (C3). **MS** (DIP-EI, 70 eV,  $150^{\circ}\text{C}$ ):  $m/z$  (%) 261 (2, M-1), 231

(100, M-OMe), 199 (3, M-(OMe)<sub>2</sub>), 149 (24), 135 (31), 123 (49), 107 (57). **TLC** (SiO<sub>2</sub>; hexane : ether, 9:1) : R<sub>f</sub> = 0.26. **GC** (Parameters and conditions, type C) : t<sub>R</sub> = 3.14 min.

### *S1.5. Synthesis of 1,1-dimethoxy-propyne, (9)*



In a 1 L three necked, round-bottomed flask equipped with a mechanical stirrer, a condenser, and a pressure equalizing addition funnel, tetrabutylammonium hydrogen sulfate (222 g, 654 mmol) and water (44 mL) were placed. The mixture was stirred to form a thick paste to which a solution of 1,2-dibromo-3,3-dimethoxypropane, **8**, (57.09 g, 218 mmol) in hexane (150 mL) was added. The resulting mixture was vigorously stirred and cooled to 10-15 °C and a cold (10-15 °C) solution of NaOH (130.8 g, 3270 mmol) in water (130 mL) was added along 10 minutes. The mixture was stirred at room temperature for 2 hours, cooled to 5 °C, and neutralized by adding H<sub>2</sub>SO<sub>4</sub> (25% w/w) (260 mL) at 0 °C. Stirring was stopped, three layers formed and were allowed to separate for 30 min. The two upper layers were carefully decanted and extracted with ether (8 x 30 mL). The lowest layer was filtered to remove sodium sulfate and extracted with diethyl ether (5 x 30 mL). The organic fractions were combined, dried over anhydrous magnesium sulfate and filtered to give an organic solution of the product in ether. The % of product in this solution was determined by GC giving a total of 10.91 g of **9** (50% yield, 100% conversion). The product was isolated by slow and careful distillation at atmospheric pressure using a 50 cm packed column to avoid loss of product due to its low boiling point (86.0 °C at 760 Torr).

Colourless oil. **IR** (film, cm<sup>-1</sup>): ν<sub>max</sub> 3276 (H—C<sub>sp</sub>, st), 2940, 2834 (H—C<sub>sp</sub><sup>3</sup>, st), 2128 (C≡C, st), 1358, 1113, 1061 (C-O, st), 966. **<sup>1</sup>H NMR** (200 MHz, CDCl<sub>3</sub>, ppm): δ 2.51 (1H, d, J = 1.8 Hz, H1), 3.32 (9H, s, H1'), 5.09 (1H, d, J = 1.8 Hz, H3). **<sup>13</sup>C NMR** (50 MHz, CDCl<sub>3</sub>, ppm): δ 52.4 (C1'), 74.1 (C1), 78.1 (C2), 92.6 (C3). **MS** (DIP-CI-NH<sub>3</sub>, 70 eV, 150°C): m/z (%) 69 (100, M-OMe), 118 (3, M+NH<sub>4</sub>), 135 (4, M+N<sub>2</sub>H<sub>7</sub>). **CG** (parameters and conditions, type A): t<sub>R</sub> = 1.6 min. **TLC** (SiO<sub>2</sub>; hexane : ether, 9:1): R<sub>F</sub> = 0.36.

## DOCUMENT S2

### Methodology for the *in Vitro* Cancer Testing<sup>1</sup>

The human tumor cell lines of the cancer screening panel are grown in RPMI 1640 medium containing 5% fetal bovine serum and 2 mM *L*-glutamine. For a typical screening experiment, cells are inoculated into 96 well microtiter plates in 100  $\mu$ L at plating densities ranging from 5.000 to 40.000 cells/well depending on the doubling time of individual cell lines. After cell inoculation, the microtiter plates are incubated at 37° C, 5 % CO<sub>2</sub>, 95 % air and 100 % relative humidity for 24 h prior to addition of experimental drugs.

After 24 hours, two plates of each cell line are fixed *in situ* with TCA, to represent a measurement of the cell population for each cell line at the time of drug addition (T<sub>z</sub>). Experimental drugs are solubilized in dimethyl sulfoxide at 400-fold the desired final maximum test concentration and stored frozen prior to use. At the time of drug addition, an aliquot of frozen concentrate is thawed and diluted to twice the desired final maximum test concentration with complete medium containing 50  $\mu$ g/mL of gentamicin. Additional four, 10-fold or ½ log serial dilutions are made to provide a total of five drug concentrations plus control. Aliquots of 100  $\mu$ L of these different drug dilutions are added to the appropriate microtiter wells already containing 100  $\mu$ L of medium, resulting in the required final drug concentrations.

Following drug addition, the plates are incubated for an additional 48 h at 37°C, 5 % CO<sub>2</sub>, 95 % air, and 100 % relative humidity. For adherent cells, the assay is terminated by the addition of cold TCA. Cells are fixed *in situ* by the gentle addition of 50  $\mu$ L of cold 50 % (w/v) TCA (final concentration, 10 % TCA) and incubated for 60 minutes at 4 °C. The supernatant is discarded, and the plates are washed five times with water and air dried. Sulforhodamine B (SRB) solution (100  $\mu$ L) at 0.4 % (w/v) in 1 % acetic acid is added to each well, and plates are incubated for 10 minutes at room temperature. After staining, unbound dye is removed by washing five times with 1 % acetic acid and the plates are air dried. Bound stain is subsequently solubilized with 10 mM Trizma base [2-Amino-2-(hydroxymethyl)propane-1,3-diol], and the absorbance is read on an automated plate reader at a wavelength of 515 nm. For suspension cells, the methodology is the same except that the assay is terminated by fixing settled cells at the

bottom of the wells by gently adding 50 µL of 80 % TCA (final concentration, 16 % TCA). Using the seven absorbance measurements [time zero, (Tz), control growth, (C), and test growth in the presence of drug at the five concentration levels (Ti)], the percentage growth is calculated at each of the drug concentrations levels. Percentage growth inhibition (PGI) is calculated as:

$[(Ti - Tz)/(C - Tz)] \times 100$  for concentrations for which  $Ti > \text{or} = Tz$

$[(Ti - Tz)/Tz] \times 100$  for concentrations for which  $Ti < Tz$ .

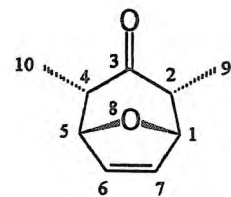
Three dose response parameters are calculated for each experimental agent. Growth inhibition of 50 % (**GI<sub>50</sub>**) is calculated from  $[(Ti - Tz)/(C - Tz)] \times 100 = 50$ , which is the drug concentration resulting in a 50% reduction in the net protein increase (as measured by SRB staining) in control cells during the drug incubation. The drug concentration resulting in total growth inhibition (**TGI**) is calculated from  $Ti = Tz$ . The **LC<sub>50</sub>** (concentration of drug resulting in a 50% reduction in the measured protein at the end of the drug treatment as compared to that at the beginning) indicating a net loss of cells following treatment is calculated from  $[(Ti - Tz)/Tz] \times 100 = -50$ . Values are calculated for each of these three parameters if the level of activity is reached; however, if the effect is not reached or is exceeded, the value for that parameter is expressed as greater as or lower than the maximum or minimum concentration tested, respectively.

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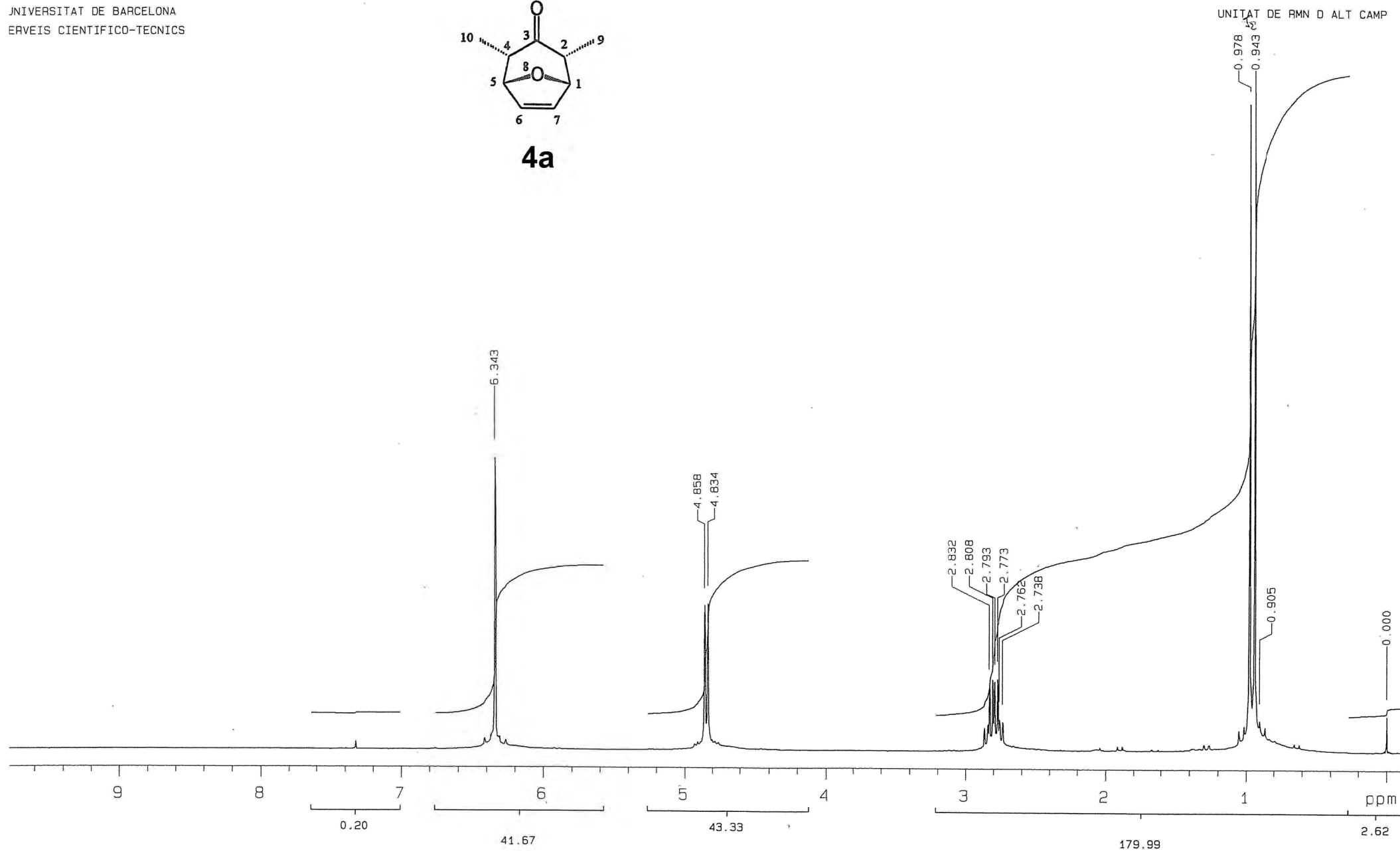
<sup>1</sup>(a) M. C. Alley, D. A. Scudiero, P. A. Monks, M. L Hursey, M. J. Czerwinski, D. L. Fine, B. J. Abbott, J. G. Mayo, R. H. Shoemaker, M. R. Boyd, *Cancer Res.*, **1988**, *48*, 589-601 ; (b) M. R. Grever, S. A. Schepartz, B. A. Chabner, The National Cancer Institute: Cancer Drug Discovery and Development Program. *Seminars in Oncology*, **1992**, *19*, 622-638; (c) M. R. Boyd, K. D. Paul, *Drug Develop. Res.*, **1995**, *34*, 91-109.

## DOCUMENT S3

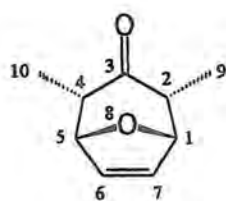
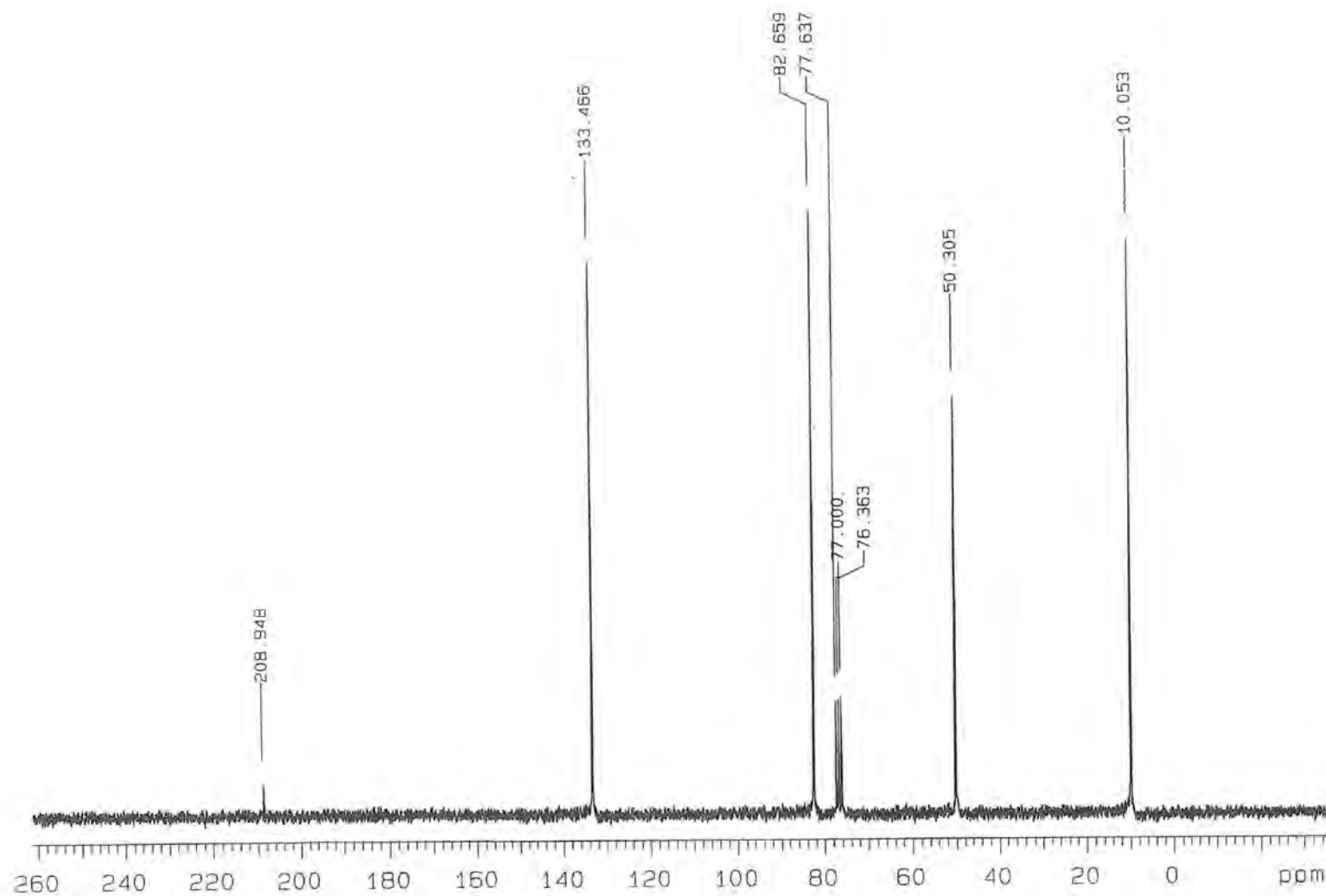
Spectra of compounds **4** to **17** described in the present work  
(IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, DEPT, COSY, HSQC and MS)



4a



|        |          |     |          |                   |          |                            |        |
|--------|----------|-----|----------|-------------------|----------|----------------------------|--------|
| H1     | 199.9750 | H1  | -300.0   | not used          | s2pu1    |                            |        |
| 3000.3 | 300.0    | nnn | 20       | not used not used |          | DACA EXP-5 A IV-V DAVID 04 | daca02 |
| 1.994  | 0        | c   | 200      | 2000.0 -25.3      | not used | usuario: jmv               |        |
| 7.5    | 32       | --- | not used |                   | cdc13    |                            | 200    |

**4a**

C13 50.2894  
 14992.5 0  
 1.001 0 200  
 15.0 2500

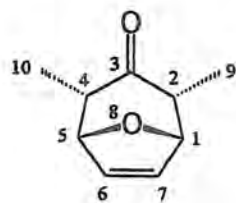
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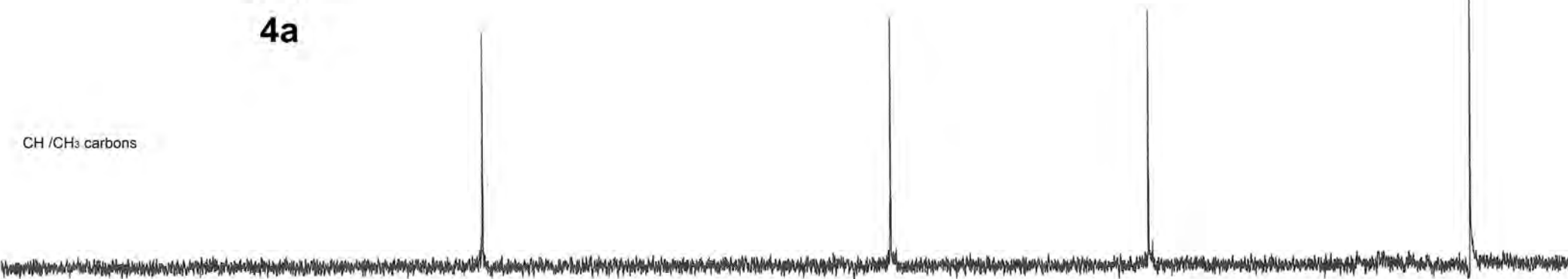
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\* 500

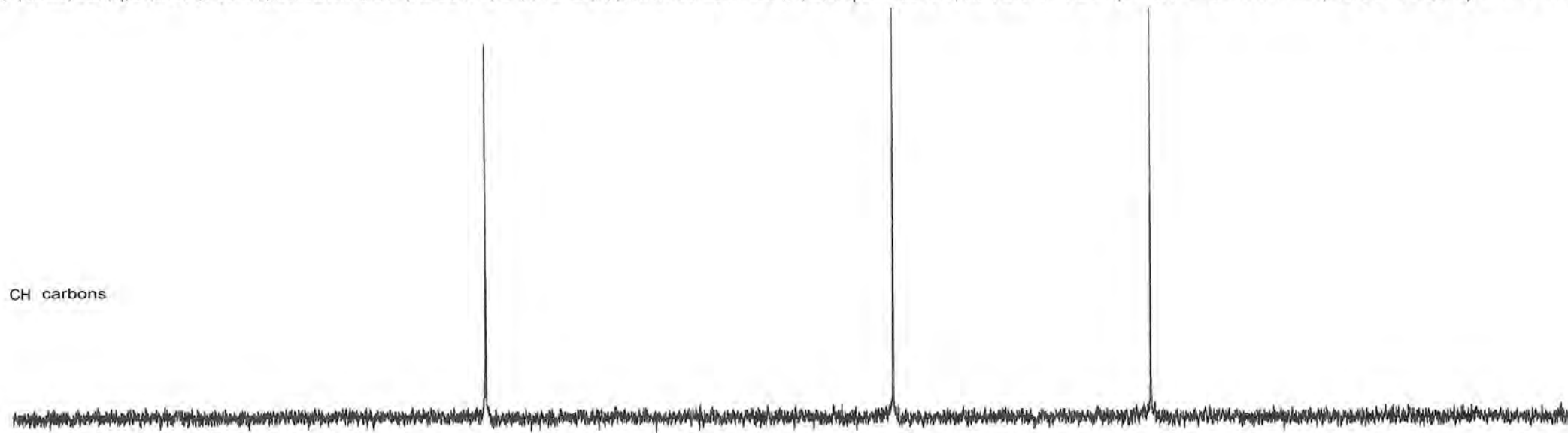


**4a**

CH /CH<sub>3</sub> carbons

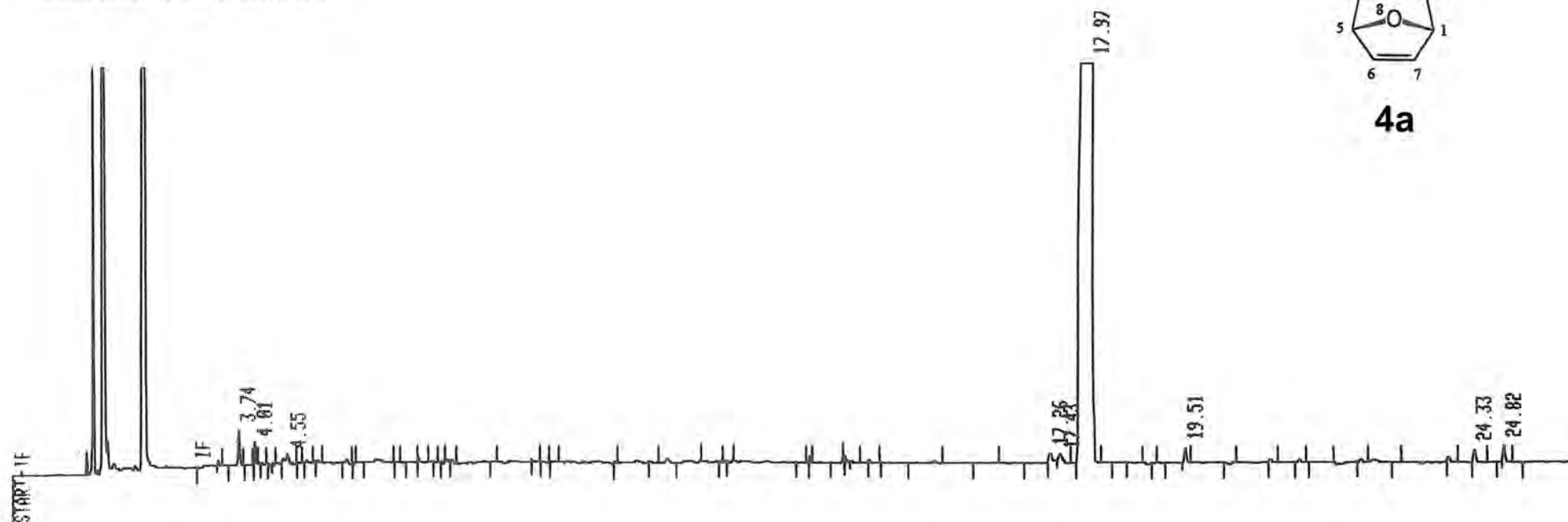
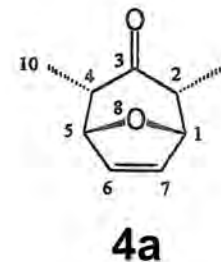


CH carbons



|         |         |      |        |                |      |                        |              |
|---------|---------|------|--------|----------------|------|------------------------|--------------|
| C13     | 50.2894 | H1   | -800.0 | 65536          | dept |                        |              |
| 14992.5 | 0       | nny  | 20     | 1.000 not used |      | DACE EXP-5 PF DAVID 04 | dace03       |
| 1.001   | 2.000   | ccs  | 10500  | 10057.8        | -0.2 | not used               |              |
| 19.0    | 240     | 30.0 | 30     |                |      | cdc13                  | usuar10: jmy |
|         |         |      |        |                |      |                        | -200         |

Conditions: 50°C, 1 min; 5°C/min; 250°C, 20 min.  
 Capillary column: 5% MePhe Silicone 20 m



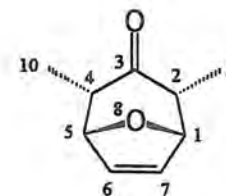
LIST: LIST  
 PEAK CAPACITY: 1159

ZERO = 5,-0.8  
 ATT 2↑ = 1  
 CHT SP = 1.0  
 PK WD = 0.04  
 THRSH = 1  
 AR RE.I = 100

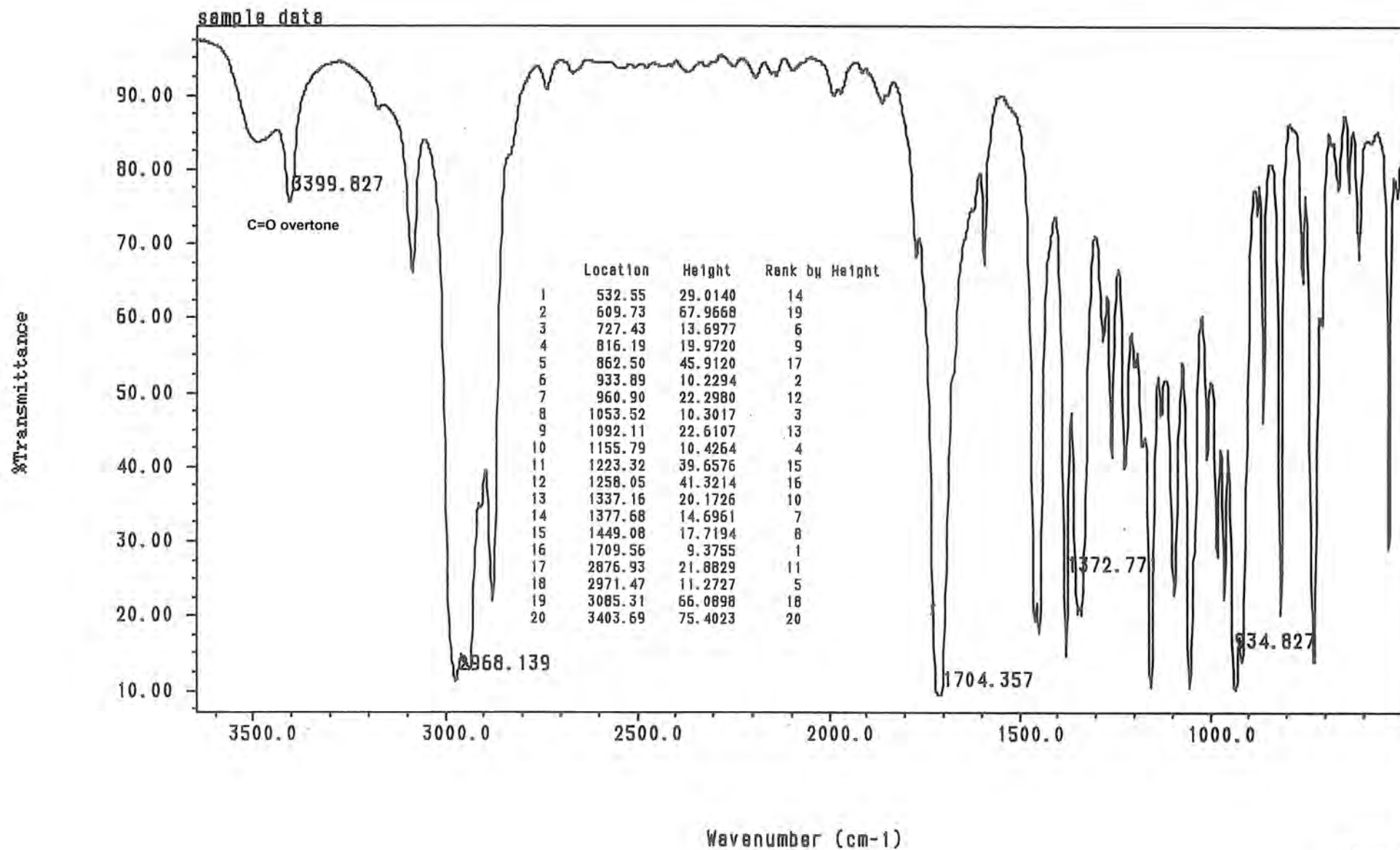
| AREA% | RT    | AREA   | TYPE | AR/HT | AREA%  |
|-------|-------|--------|------|-------|--------|
|       | 3.74  | 2441   | PB   | 0.032 | 0.278  |
|       | 4.01  | 1064   | D PB | 0.021 | 0.121  |
|       | 4.55  | 1585   | BB   | 0.073 | 0.181  |
|       | 17.26 | 1576   | BV   | 0.067 | 0.180  |
|       | 17.43 | 1861   | VB   | 0.090 | 0.212  |
|       | 17.97 | 858800 | PB   | 0.135 | 97.909 |
|       | 19.51 | 1423   | PB   | 0.045 | 0.162  |
|       | 24.33 | 1469   | BB   | 0.051 | 0.168  |
|       | 24.82 | 1792   | VB   | 0.050 | 0.204  |
|       | 26.89 | 5127   | PB   | 0.051 | 0.585  |

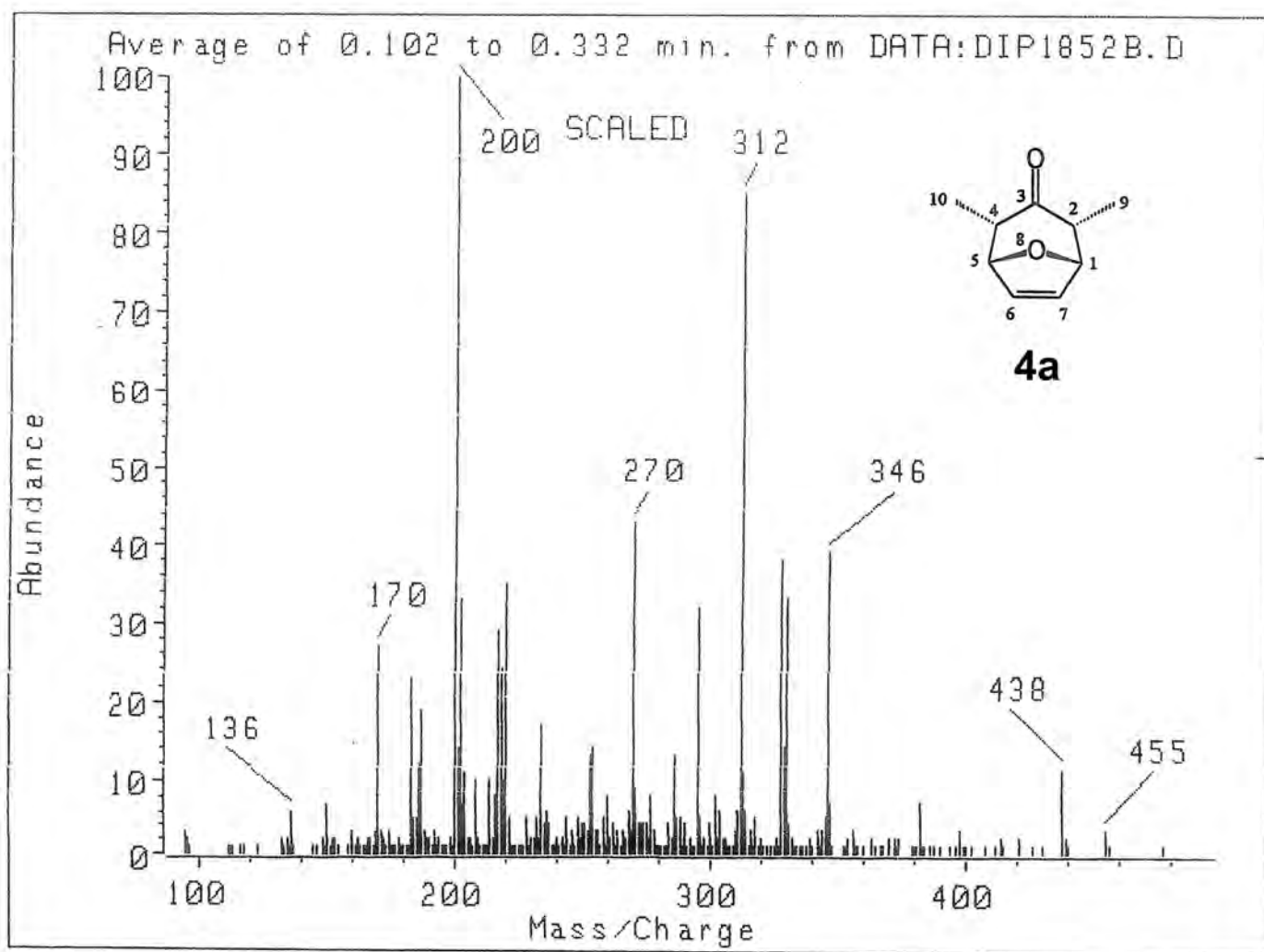
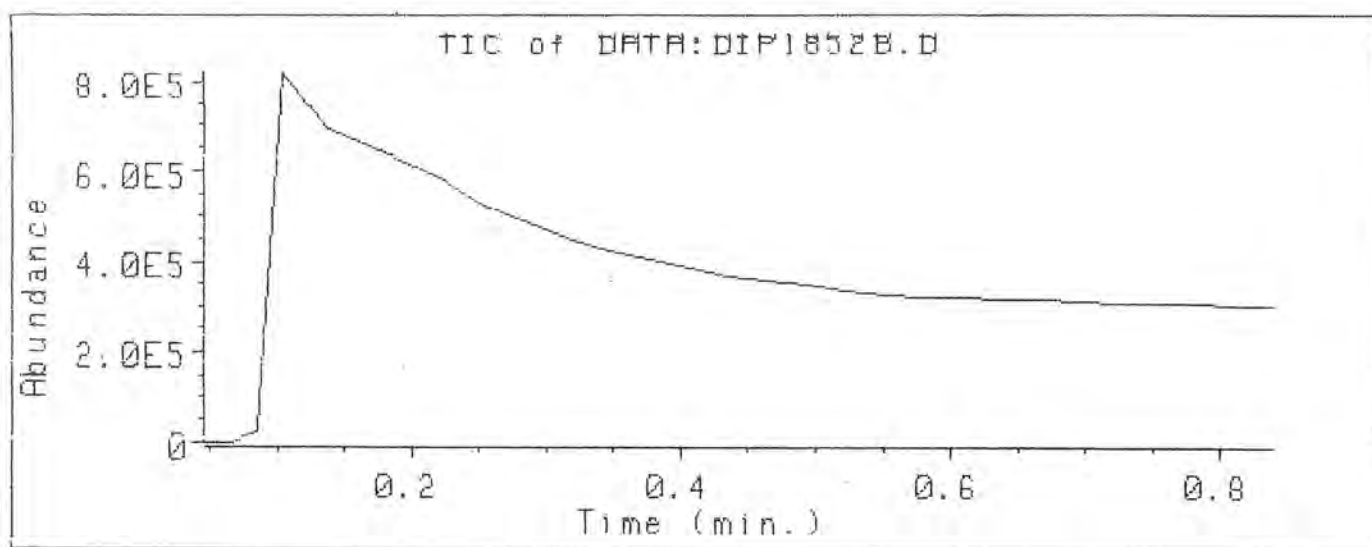
TOTAL AREA= 877140  
 MUL FACTOR= 1.0000E+00

exp-5 PF  
sample data



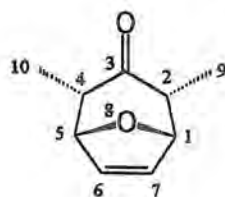
4a





Average of 0.102 to 0.332 min. from DATA:DIP1852B.D  
 REF:EXP-21, H-III DAVID DIP-CI  $MH_3$

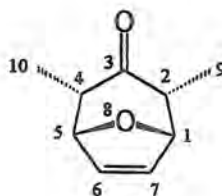
| m/z    | abund. | m/z    | abund. | m/z    | abund. | m/z    | abund. |
|--------|--------|--------|--------|--------|--------|--------|--------|
| 94.00  | 3      | 175.00 | 1      | 215.00 | 2      | 255.00 | 3      |
| 95.00  | 2      | 176.00 | 1      | 216.00 | 8      | 256.00 | 3      |
| 96.00  | 1      | 177.00 | 1      | 217.00 | 29     | 257.00 | 1      |
| 111.00 | 1      | 178.00 | 2      | 218.00 | 24     | 258.00 | 5      |
| 112.00 | 1      | 179.00 | 1      | 219.00 | 10     | 259.00 | 1      |
| 113.00 | 1      | 180.00 | 1      | 220.00 | 35     | 260.00 | 8      |
| 116.00 | 1      | 181.00 | 1      | 221.00 | 5      | 261.00 | 2      |
| 117.00 | 1      | 182.00 | 1      | 222.00 | 1      | 262.00 | 4      |
| 123.00 | 1      | 183.00 | 23     | 223.00 | 1      | 263.00 | 1      |
| 132.00 | 2      | 184.00 | 5      | 224.00 | 1      | 264.00 | 3      |
| 133.00 | 1      | 185.00 | 5      | 225.00 | 1      | 265.00 | 1      |
| 134.00 | 2      | 186.00 | 12     | 226.00 | 1      | 266.00 | 3      |
| 135.00 | 1      | 187.00 | 19     | 227.00 | 1      | 267.00 | 2      |
| 136.00 | 6      | 188.00 | 3      | 228.00 | 5      | 268.00 | 6      |
| 137.00 | 1      | 189.00 | 2      | 229.00 | 1      | 269.00 | 3      |
| 144.00 | 1      | 190.00 | 2      | 230.00 | 2      | 270.00 | 43     |
| 146.00 | 1      | 191.00 | 1      | 231.00 | 2      | 271.00 | 9      |
| 148.00 | 2      | 192.00 | 3      | 232.00 | 5      | 272.00 | 4      |
| 149.00 | 1      | 193.00 | 1      | 233.00 | 2      | 273.00 | 4      |
| 150.00 | 7      | 194.00 | 2      | 234.00 | 17     | 274.00 | 4      |
| 151.00 | 1      | 195.00 | 1      | 235.00 | 4      | 275.00 | 4      |
| 152.00 | 2      | 196.00 | 1      | 236.00 | 6      | 276.00 | 2      |
| 153.00 | 2      | 197.00 | 1      | 237.00 | 4      | 277.00 | 8      |
| 154.00 | 1      | 198.00 | 2      | 238.00 | 1      | 278.00 | 3      |
| 158.00 | 1      | 199.00 | 1      | 239.00 | 1      | 279.00 | 1      |
| 160.00 | 3      | 200.00 | 100    | 240.00 | 2      | 280.00 | 1      |
| 161.00 | 1      | 201.00 | 14     | 241.00 | 1      | 281.00 | 1      |
| 162.00 | 2      | 202.00 | 33     | 242.00 | 2      | 282.00 | 1      |
| 163.00 | 1      | 203.00 | 7      | 243.00 | 1      | 283.00 | 1      |
| 164.00 | 1      | 204.00 | 11     | 244.00 | 5      | 284.00 | 4      |
| 165.00 | 1      | 205.00 | 2      | 245.00 | 1      | 285.00 | 2      |
| 166.00 | 2      | 206.00 | 2      | 246.00 | 3      | 286.00 | 13     |
| 167.00 | 2      | 207.00 | 1      | 247.00 | 1      | 287.00 | 5      |
| 168.00 | 1      | 208.00 | 10     | 248.00 | 5      | 288.00 | 5      |
| 169.00 | 3      | 209.00 | 2      | 249.00 | 2      | 289.00 | 1      |
| 170.00 | 27     | 210.00 | 1      | 250.00 | 4      | 290.00 | 4      |
| 171.00 | 3      | 211.00 | 1      | 251.00 | 4      | 291.00 | 1      |
| 172.00 | 2      | 212.00 | 1      | 252.00 | 3      | 292.00 | 2      |
| 173.00 | 1      | 213.00 | 1      | 253.00 | 13     | 293.00 | 1      |
| 174.00 | 3      | 214.00 | 10     | 254.00 | 14     | 294.00 | 1      |



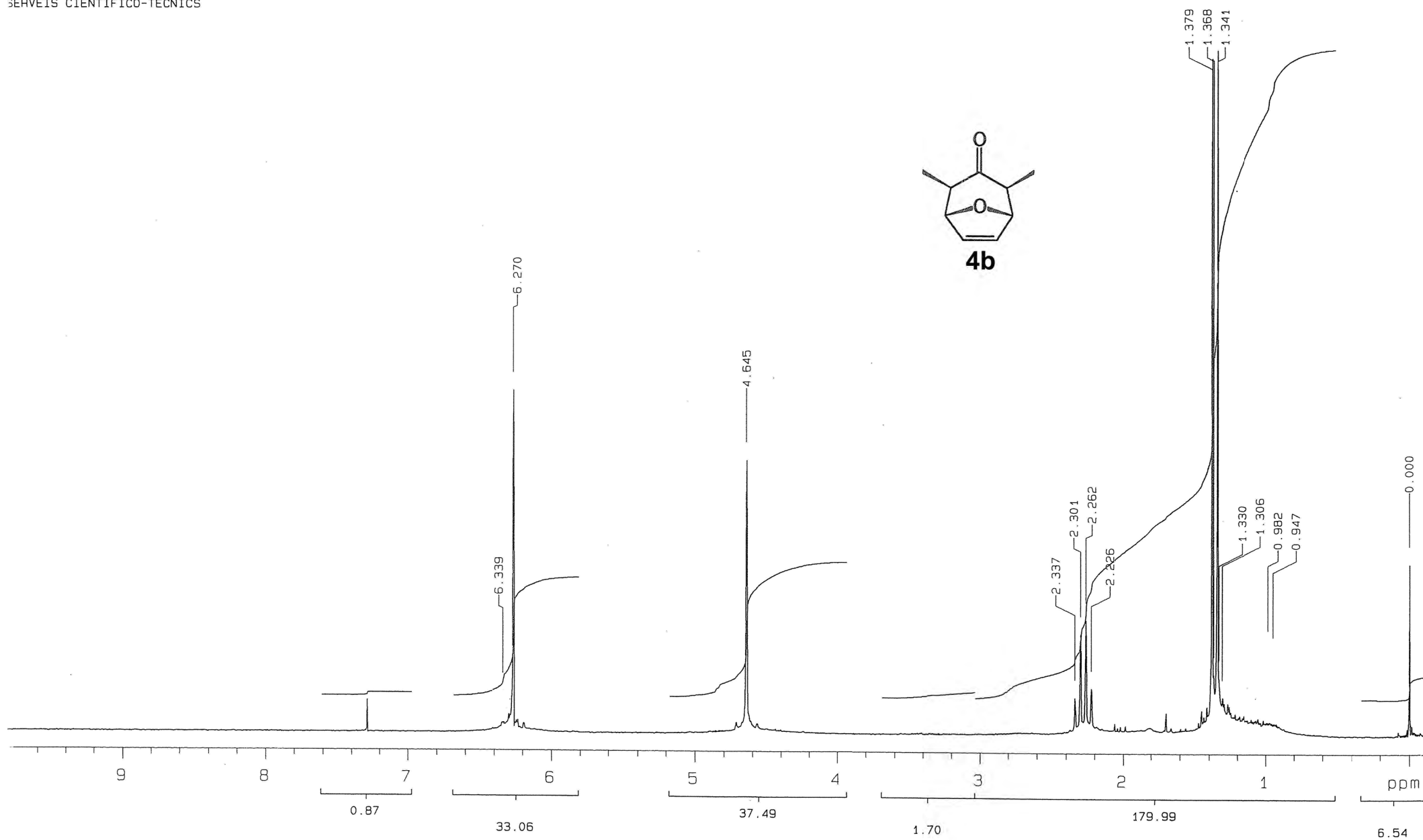
4a

Average of 0.102 to 0.332 min. from DATA:DIP1852B.D  
 REF:EXP-21, H-III DAVID DIP-CI

| m/z    | abund. | m/z    | abund. | m/z    | abund. | m/z    | abund. |
|--------|--------|--------|--------|--------|--------|--------|--------|
| 295.00 | 32     | 319.00 | 1      | 346.00 | 39     | 384.00 | 1      |
| 296.00 | 5      | 320.00 | 2      | 347.00 | 7      | 386.00 | 1      |
| 297.00 | 1      | 321.00 | 1      | 348.00 | 1      | 388.00 | 1      |
| 298.00 | 2      | 322.00 | 1      | 352.00 | 1      | 390.00 | 1      |
| 299.00 | 1      | 324.00 | 1      | 353.00 | 1      | 394.00 | 1      |
| 300.00 | 4      | 325.00 | 1      | 354.00 | 2      | 396.00 | 1      |
| 301.00 | 1      | 326.00 | 2      | 356.00 | 3      | 398.00 | 3      |
| 302.00 | 8      | 327.00 | 1      | 357.00 | 1      | 399.00 | 1      |
| 303.00 | 2      | 328.00 | 38     | 358.00 | 1      | 400.00 | 1      |
| 304.00 | 6      | 329.00 | 14     | 360.00 | 1      | 402.00 | 1      |
| 305.00 | 2      | 330.00 | 33     | 363.00 | 2      | 408.00 | 1      |
| 306.00 | 2      | 331.00 | 4      | 365.00 | 1      | 412.00 | 1      |
| 307.00 | 1      | 332.00 | 2      | 367.00 | 1      | 414.00 | 2      |
| 308.00 | 1      | 333.00 | 1      | 368.00 | 1      | 415.00 | 1      |
| 309.00 | 1      | 334.00 | 1      | 370.00 | 2      | 421.00 | 2      |
| 310.00 | 3      | 335.00 | 1      | 372.00 | 2      | 426.00 | 1      |
| 311.00 | 6      | 336.00 | 1      | 373.00 | 1      | 430.00 | 1      |
| 312.00 | 85     | 338.00 | 1      | 374.00 | 2      | 438.00 | 11     |
| 313.00 | 11     | 339.00 | 2      | 379.00 | 1      | 439.00 | 2      |
| 314.00 | 6      | 340.00 | 1      | 380.00 | 1      | 440.00 | 1      |
| 315.00 | 1      | 342.00 | 3      | 381.00 | 1      | 455.00 | 3      |
| 316.00 | 3      | 343.00 | 1      | 382.00 | 7      | 456.00 | 1      |
| 317.00 | 1      | 344.00 | 3      | 383.00 | 1      | 477.00 | 1      |
| 318.00 | 5      | 345.00 | 5      |        |        |        |        |



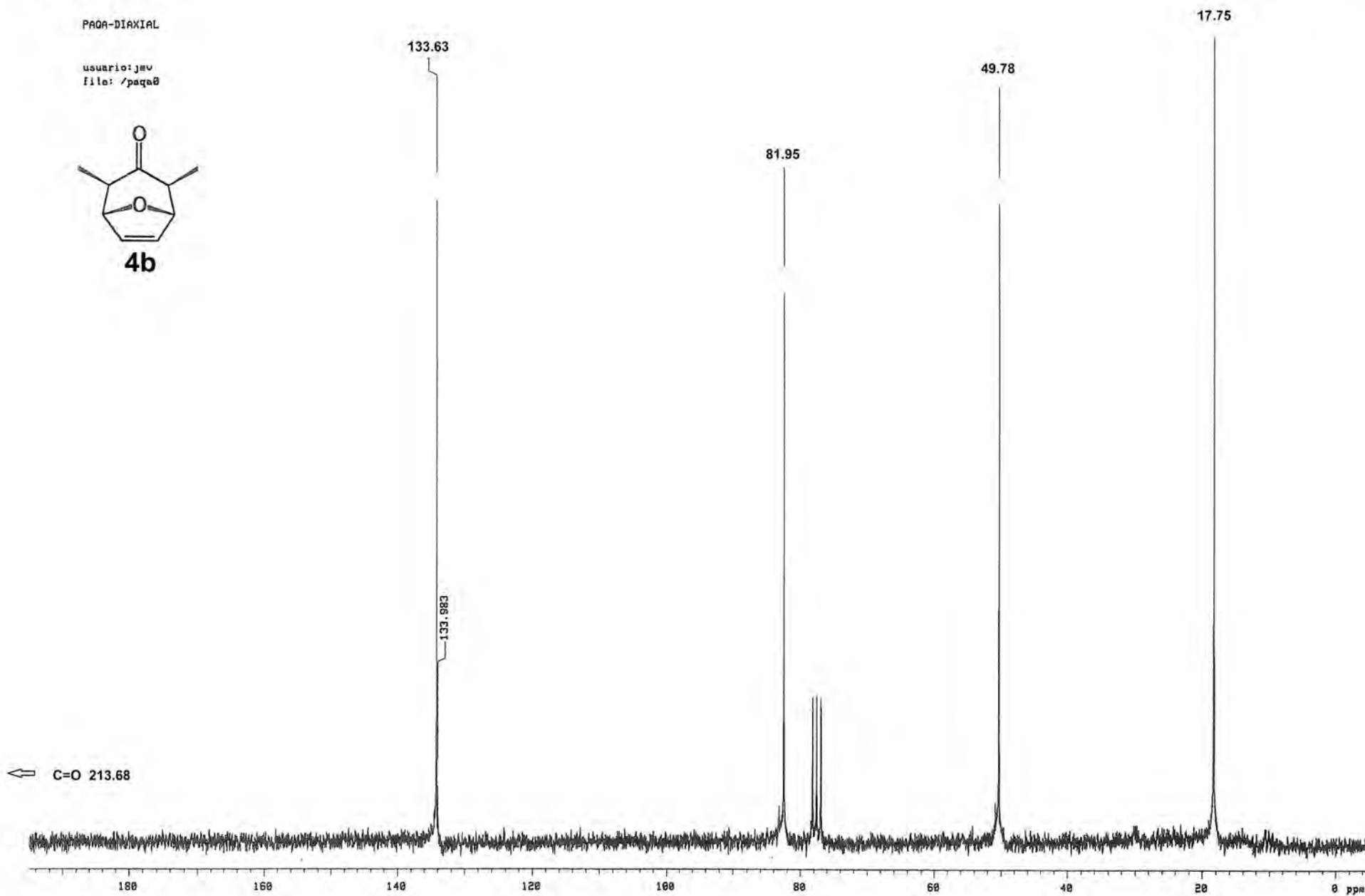
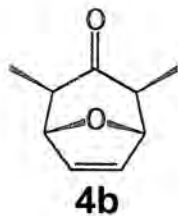
**4a**



|        |          |     |          |                   |       |                 |              |
|--------|----------|-----|----------|-------------------|-------|-----------------|--------------|
| H1     | 199.9750 | H1  | -300.0   | not used          | s2pu1 |                 |              |
| 3000.3 | 300.0    | nnn | 20       | not used not used |       | DADAC E-21/F-VI | dada02       |
| 1.994  | 0        | c   | 200      | 2000.0            | -25.3 | not used        | usuario: jmv |
| 7.5    | 32       | --- | not used |                   |       | cdc13           | 500          |

PAQA-DIAXIAL

usuari: jmv  
file: /paqa0



PAQA-DIAXIAL

usuario:jmv

dept

Frequency 50.289 MHz

Spectral width 14992.5 Hz

Acquisition time 1.001 sec

Relaxation delay 2.000 sec

Pulse width 95.0 degrees

Ambient temperature

No. repetitions 240

DECOUPLE H1

High power 30

Decoupler gated on during acquisition

Decoupler gated off during delay

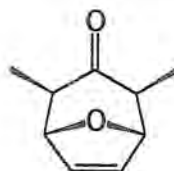
Double precision acquisition

DATA PROCESSING

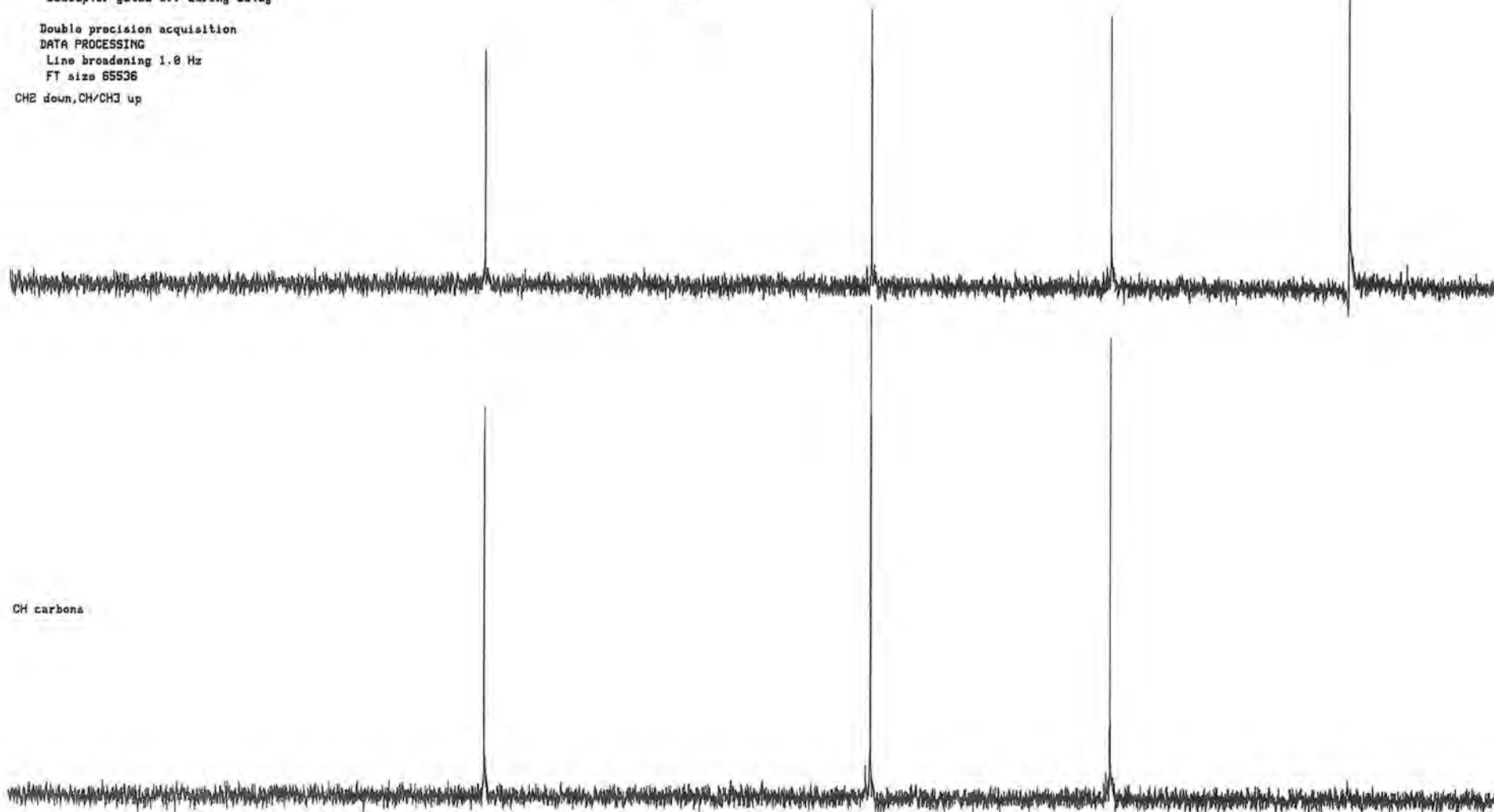
Line broadening 1.0 Hz

FT size 65536

CH2 down,CH/CH3 up

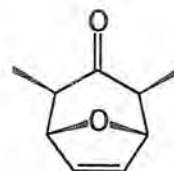


4b

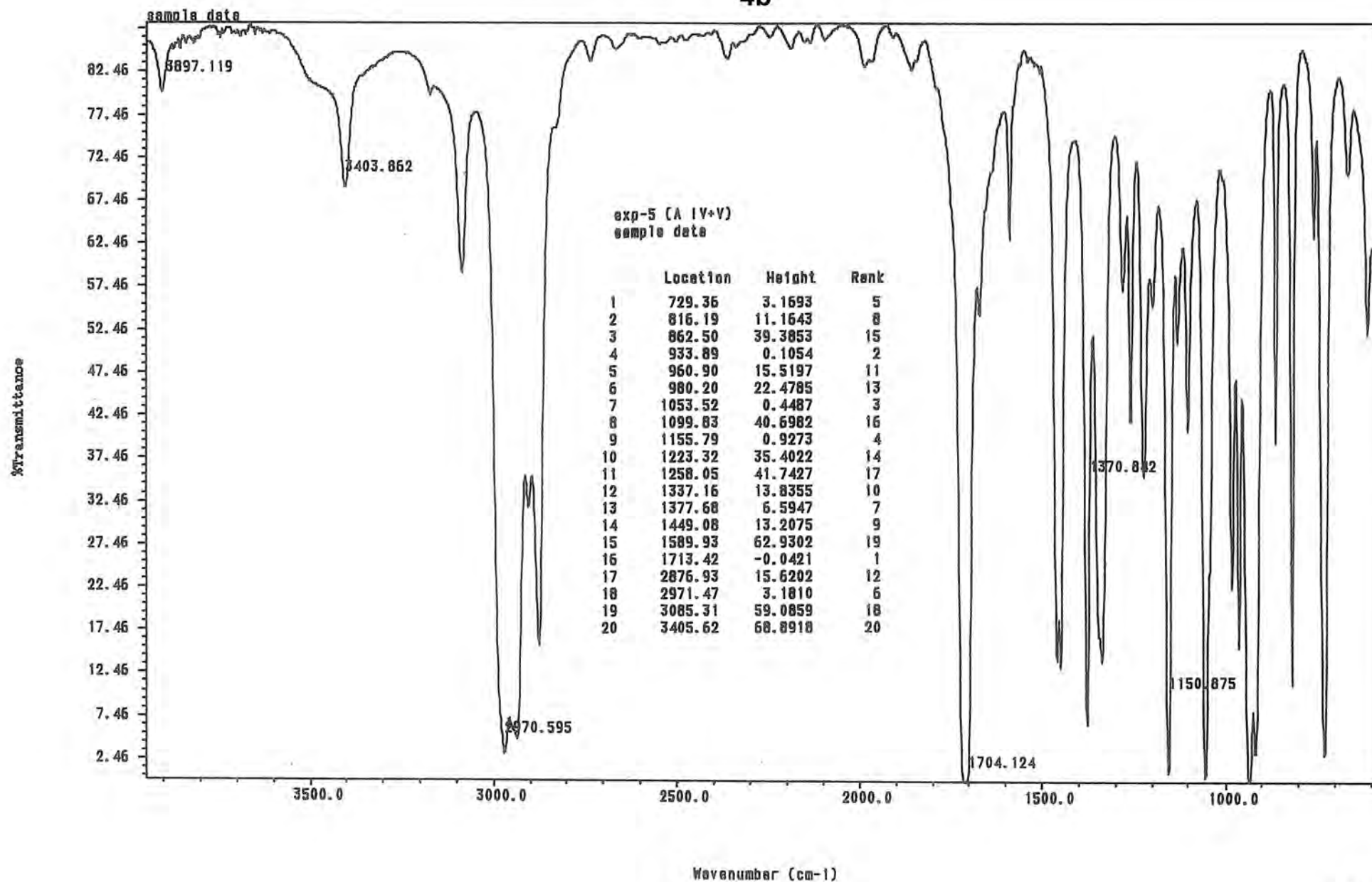


CH carbons

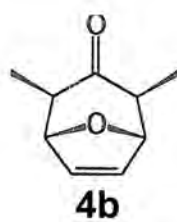
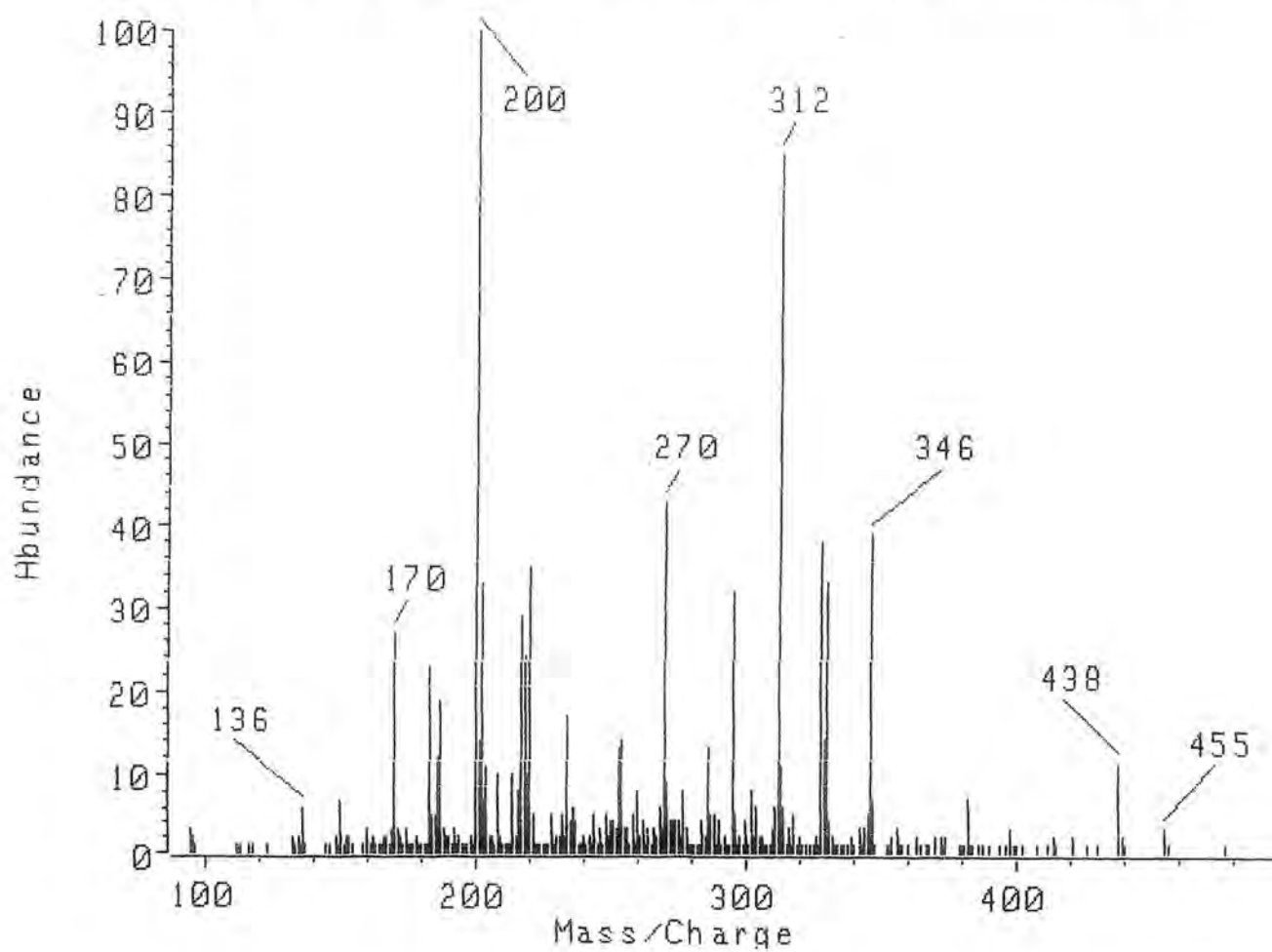
exp-5 (A IV+V)  
sample data

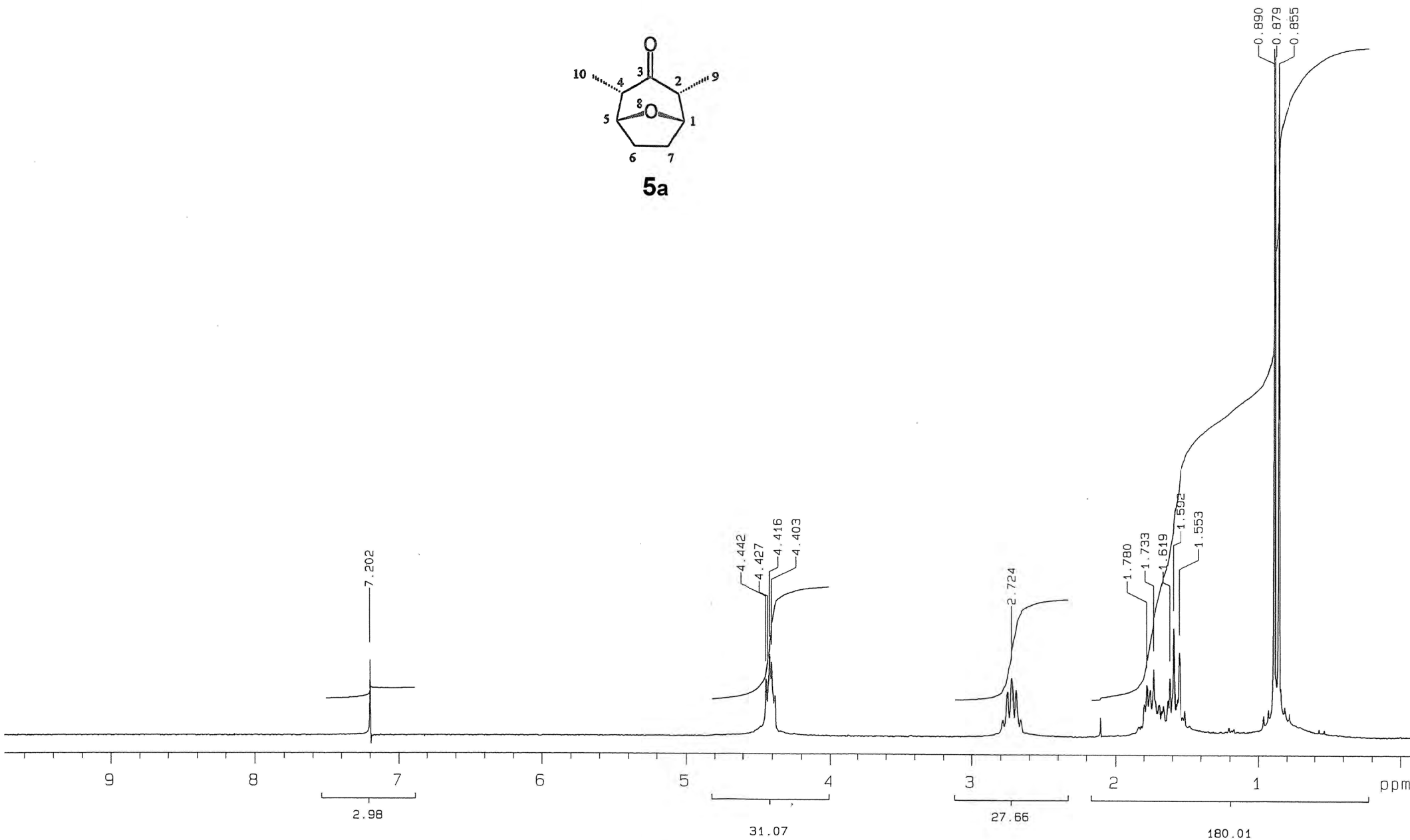
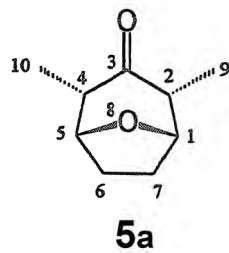


4b

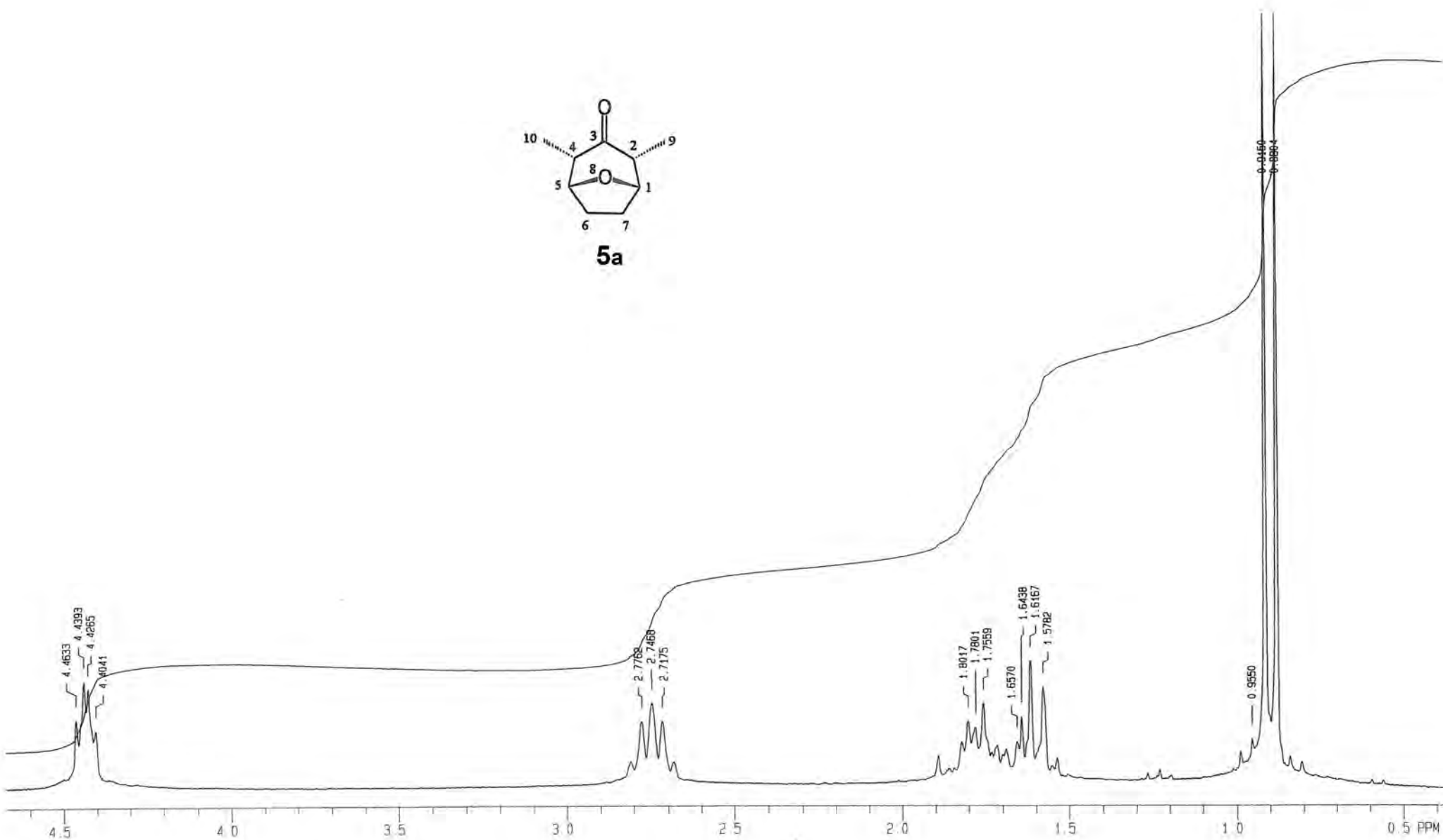
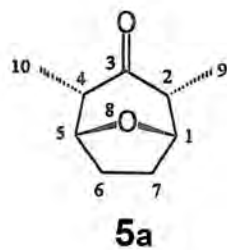


Average of 0.10 to 0.33 min. from DATA:DIP1852B.

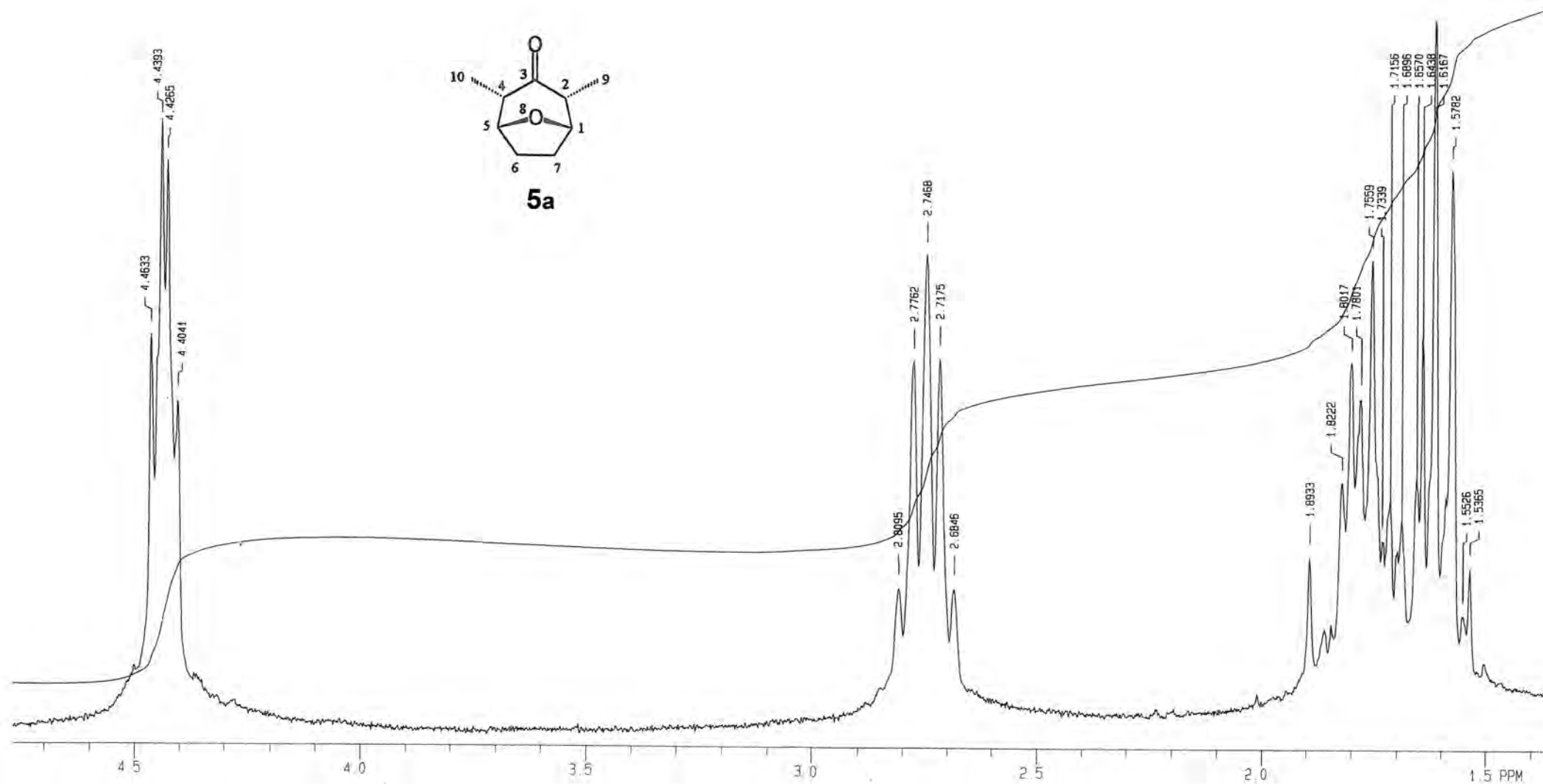
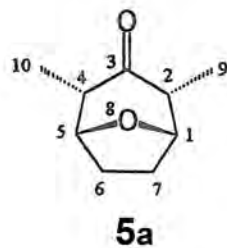




|        |          |     |          |                   |          |               |        |
|--------|----------|-----|----------|-------------------|----------|---------------|--------|
| H1     | 199.9750 | H1  | -300.0   | not used          | s2pul    |               |        |
| 3000.3 | 300.0    | nnn | 20       | not used not used |          | DACB EXP-8 PF | dacb01 |
| 1.994  | 0        | c   | 200      | 2000.0            | not used | usuario: jmv  |        |
| 15.0   | 64       | --- | not used |                   | cdc13    |               | 500    |



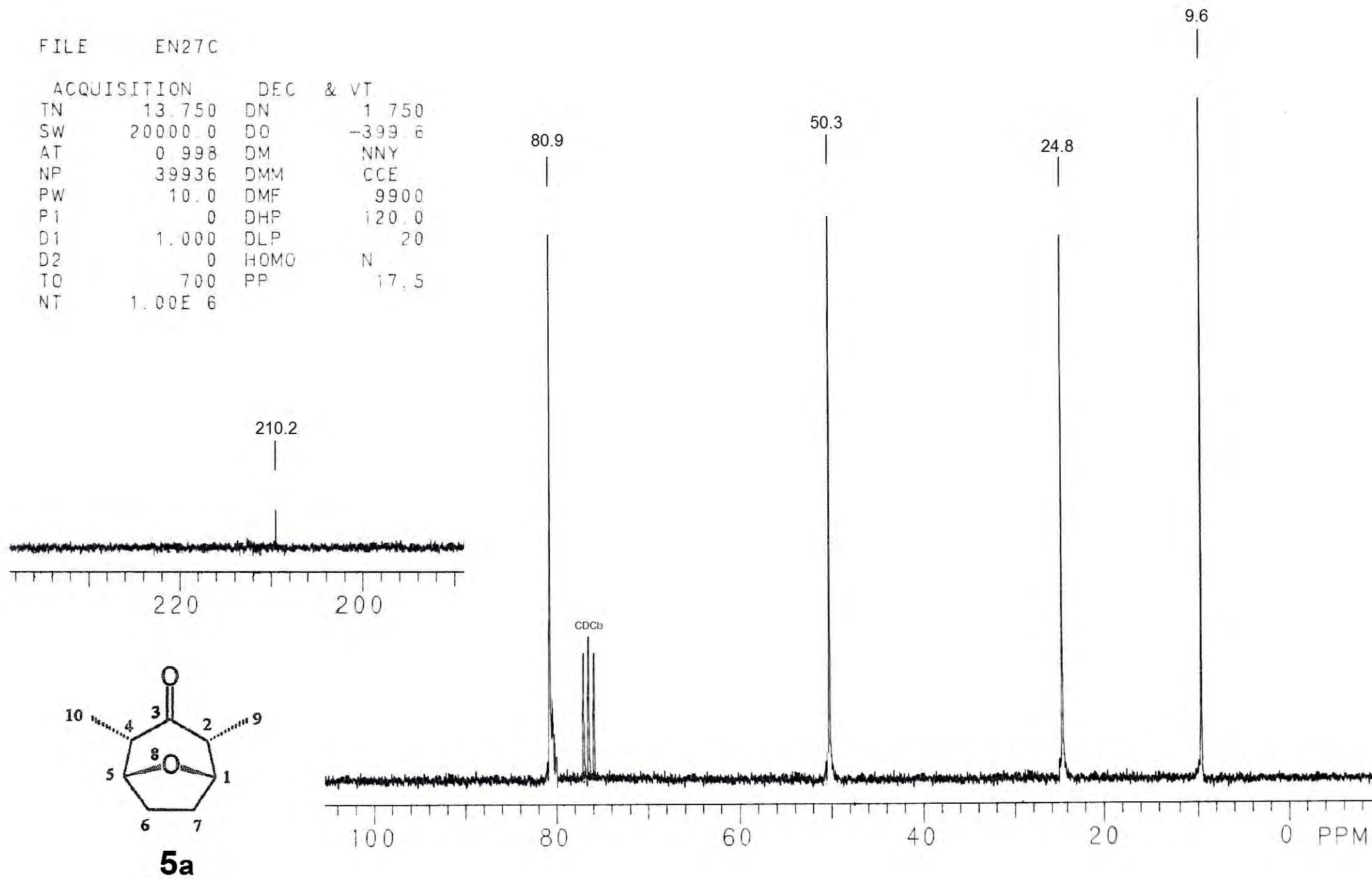
|        |     |       |        |       |      |     |       |          |
|--------|-----|-------|--------|-------|------|-----|-------|----------|
| 1.000  | 200 | 1.000 | -300.0 | 16    | ---  | --- | S2PUL |          |
| 3000.3 | 0   | NNN   | 20.0   | ---   | ---  | --- |       | H        |
| 2.666  | 0   | C     | 200    | 889.0 | 75.8 | --- |       |          |
| 4.9    | 16  |       | 1.0    |       |      |     | CDCL3 | GEMA 200 |



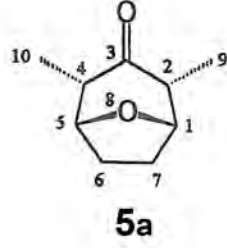
|        |     |       |        |       |       |     |          |
|--------|-----|-------|--------|-------|-------|-----|----------|
| 1.000  | 200 | 1.000 | -300.0 | 16    | ---   | --- | S2PUL    |
| 3000.3 | 0   | NNN   | 20.0   | ---   | ---   | --- |          |
| 2.666  | 0   | C     | 200    | 691.2 | 273.6 | --- |          |
| 4.9    | 16  |       | 1.0    |       |       |     | CDCL3    |
|        |     |       |        |       |       |     | GEMA 200 |

FILE EN27C

| ACQUISITION |         | DEC  | & VT   |
|-------------|---------|------|--------|
| TN          | 13.750  | DN   | 1.750  |
| SW          | 20000.0 | DO   | -399.6 |
| AT          | 0.998   | DM   | NNY    |
| NP          | 39936   | DMM  | CCE    |
| PW          | 10.0    | DMF  | 9900   |
| P1          | 0       | DHP  | 120.0  |
| D1          | 1.000   | DLP  | 20     |
| D2          | 0       | HOMO | N      |
| TO          | 700     | PP   | 17.5   |
| NT          | 1.00E 6 |      |        |



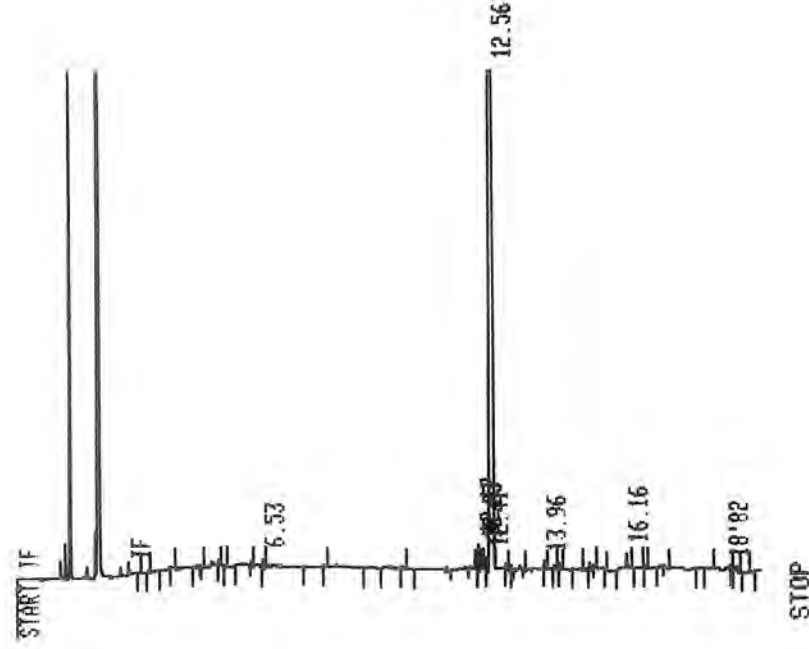
Conditions: 50°C, 1 min; 5°C/min; 250°C, 20 min.  
 Capillary column: 5% MePhe Silicone 20 m



LIST: LIST  
 PEAK CAPACITY: 1158

ZERO = 5,-0.7  
 ATT 24 = 1  
 CHT SP = 0.5  
 PK MD = 0.04  
 THRESH = 1  
 OR REJ = 100

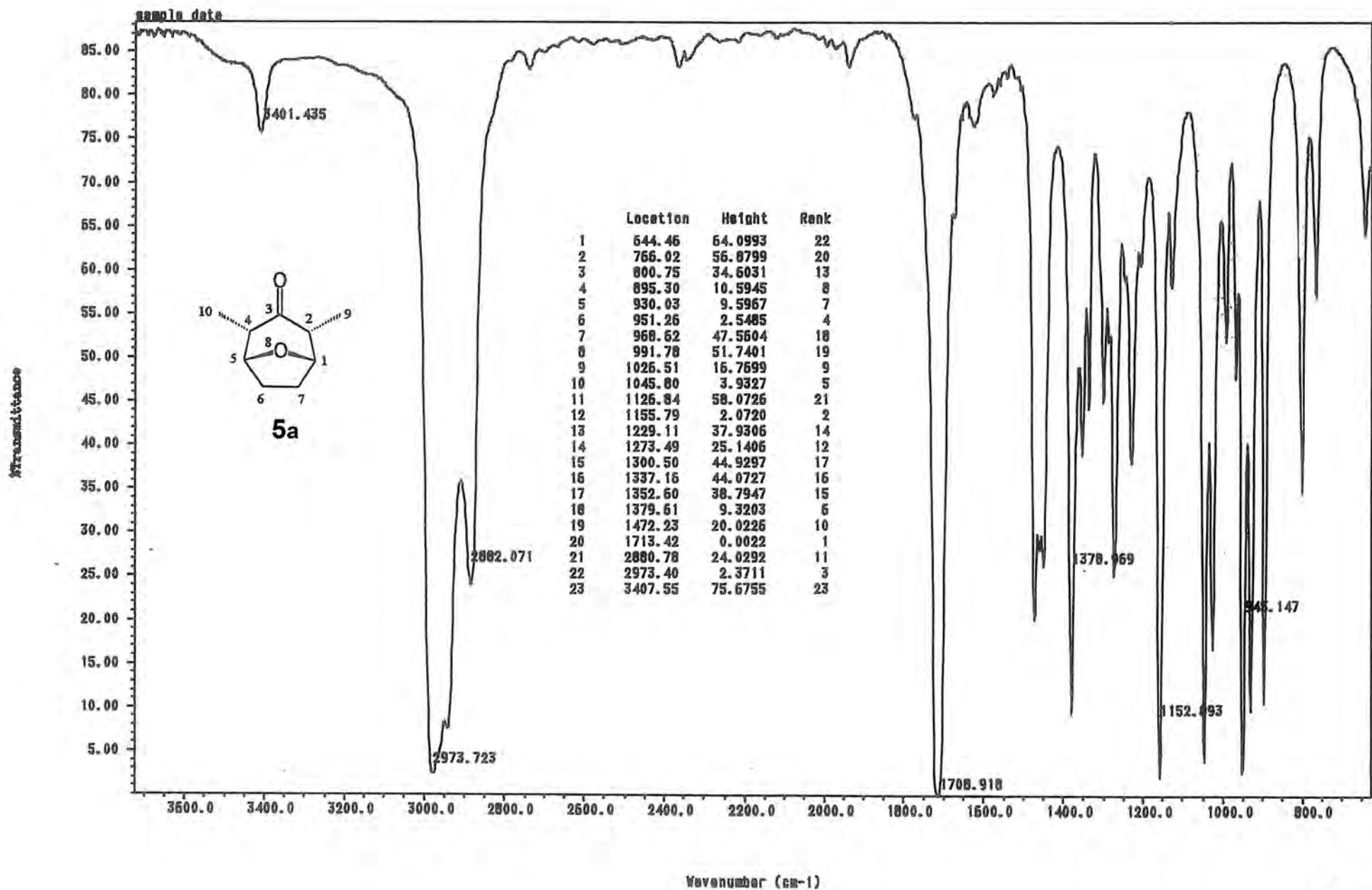
LIST: TIME 0  
 0.00 INTG # = 9  
 3.00 INTG # = -9  
 40.00 STOP

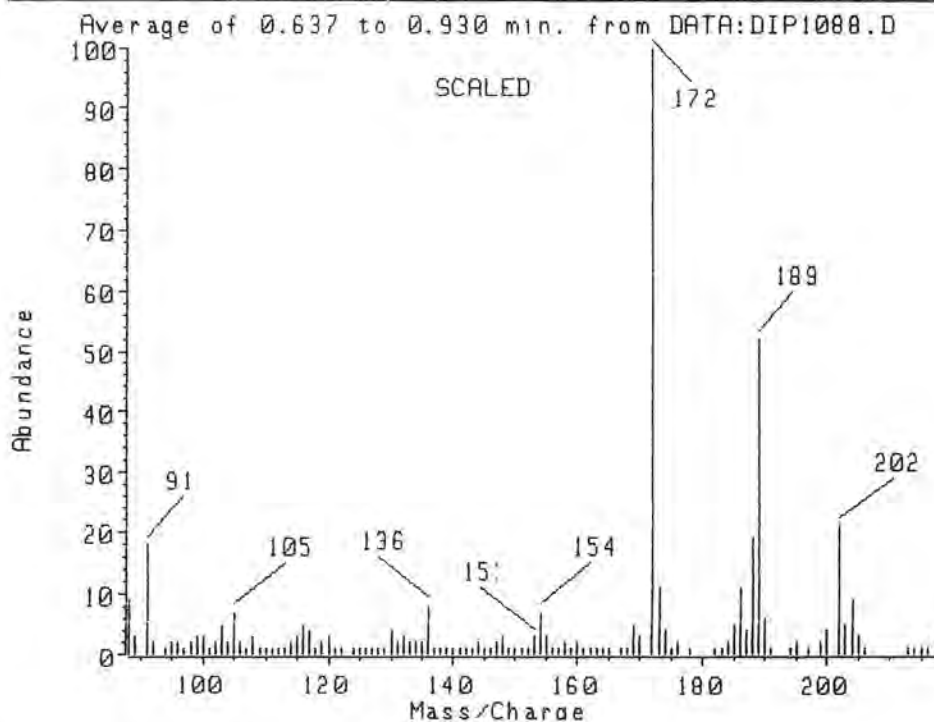
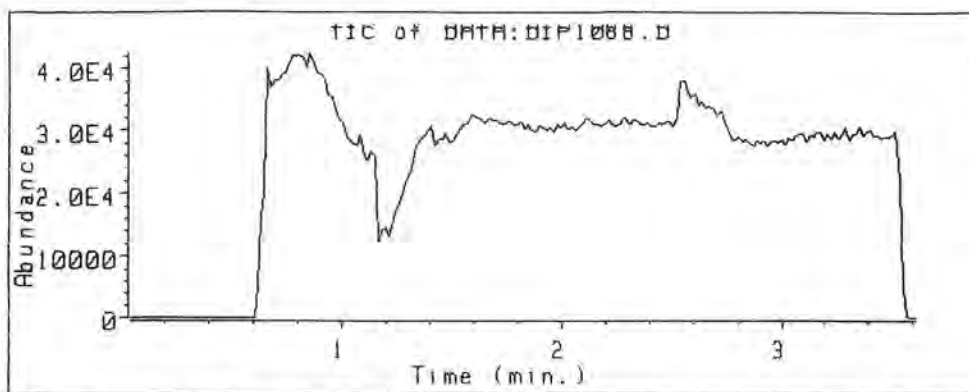
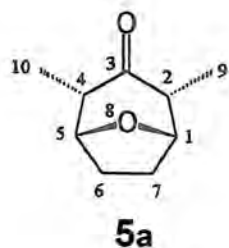


RUN # 14

| AREA% | RT | AREA   | TYPE | AR/HT | AREA2  |
|-------|----|--------|------|-------|--------|
| 6.53  |    | 344    | BB   | 0.022 | 0.049  |
| 12.17 |    | 1453   | BY   | 0.033 | 0.207  |
| 12.23 |    | 1289   | VB   | 0.038 | 0.183  |
| 12.41 |    | 694    | BY   | 0.036 | 0.099  |
| 12.56 |    | 697370 | VB   | 0.066 | 99.124 |
| 13.96 |    | 597    | BB   | 0.035 | 0.085  |
| 16.16 |    | 1788   | BB   | 0.068 | 0.254  |

TOTAL AREA= 703530  
 MUL FACTOR= 1.0000E+00





Average of 0.637 to 0.930 min. from DATA:DIP1088.D  
CICLODUCTE HIDOGENAT M.CANO DIP-CI-NH3

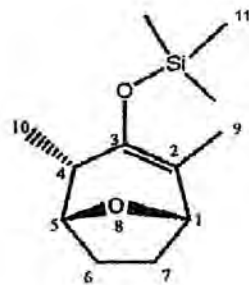
| m/z    | abund. | m/z    | abund. | m/z    | abund. | m/z    | abund. |
|--------|--------|--------|--------|--------|--------|--------|--------|
| 88.00  | 9      | 118.00 | 1      | 147.00 | 2      | 176.00 | 2      |
| 89.00  | 3      | 119.00 | 2      | 148.00 | 3      | 178.00 | 1      |
| 91.00  | 18     | 120.00 | 3      | 149.00 | 1      | 182.00 | 1      |
| 92.00  | 2      | 121.00 | 1      | 150.00 | 1      | 183.00 | 1      |
| 94.00  | 1      | 122.00 | 1      | 151.00 | 1      | 184.00 | 2      |
| 95.00  | 2      | 124.00 | 1      | 152.00 | 1      | 185.00 | 5      |
| 96.00  | 2      | 125.00 | 1      | 153.00 | 3      | 186.00 | 11     |
| 97.00  | 1      | 126.00 | 1      | 154.00 | 7      | 187.00 | 4      |
| 98.00  | 2      | 127.00 | 1      | 155.00 | 3      | 188.00 | 19     |
| 99.00  | 3      | 128.00 | 1      | 156.00 | 1      | 189.00 | 52     |
| 100.00 | 3      | 129.00 | 1      | 157.00 | 1      | 190.00 | 6      |
| 101.00 | 1      | 130.00 | 4      | 158.00 | 2      | 191.00 | 1      |
| 102.00 | 2      | 131.00 | 2      | 159.00 | 1      | 194.00 | 1      |
| 103.00 | 5      | 132.00 | 3      | 160.00 | 2      | 195.00 | 2      |
| 104.00 | 2      | 133.00 | 2      | 161.00 | 1      | 197.00 | 1      |
| 105.00 | 7      | 134.00 | 2      | 162.00 | 1      | 199.00 | 2      |
| 106.00 | 2      | 135.00 | 2      | 163.00 | 1      | 200.00 | 4      |
| 107.00 | 1      | 136.00 | 8      | 164.00 | 1      | 202.00 | 21     |
| 108.00 | 3      | 137.00 | 1      | 165.00 | 1      | 203.00 | 5      |
| 109.00 | 1      | 138.00 | 1      | 167.00 | 1      | 204.00 | 9      |
| 110.00 | 1      | 139.00 | 1      | 168.00 | 1      | 205.00 | 3      |
| 111.00 | 1      | 140.00 | 1      | 169.00 | 5      | 206.00 | 1      |
| 112.00 | 1      | 141.00 | 1      | 170.00 | 3      | 213.00 | 1      |
| 113.00 | 1      | 142.00 | 1      | 172.00 | 100    | 214.00 | 1      |
| 114.00 | 2      | 143.00 | 1      | 173.00 | 11     | 215.00 | 1      |
| 115.00 | 3      | 144.00 | 2      | 174.00 | 4      | 216.00 | 1      |
| 116.00 | 5      | 145.00 | 1      | 175.00 | 1      | 218.00 | 3      |
| 117.00 | 4      | 146.00 | 1      |        |        |        |        |

UNIVERSITAT DE BARCELONA

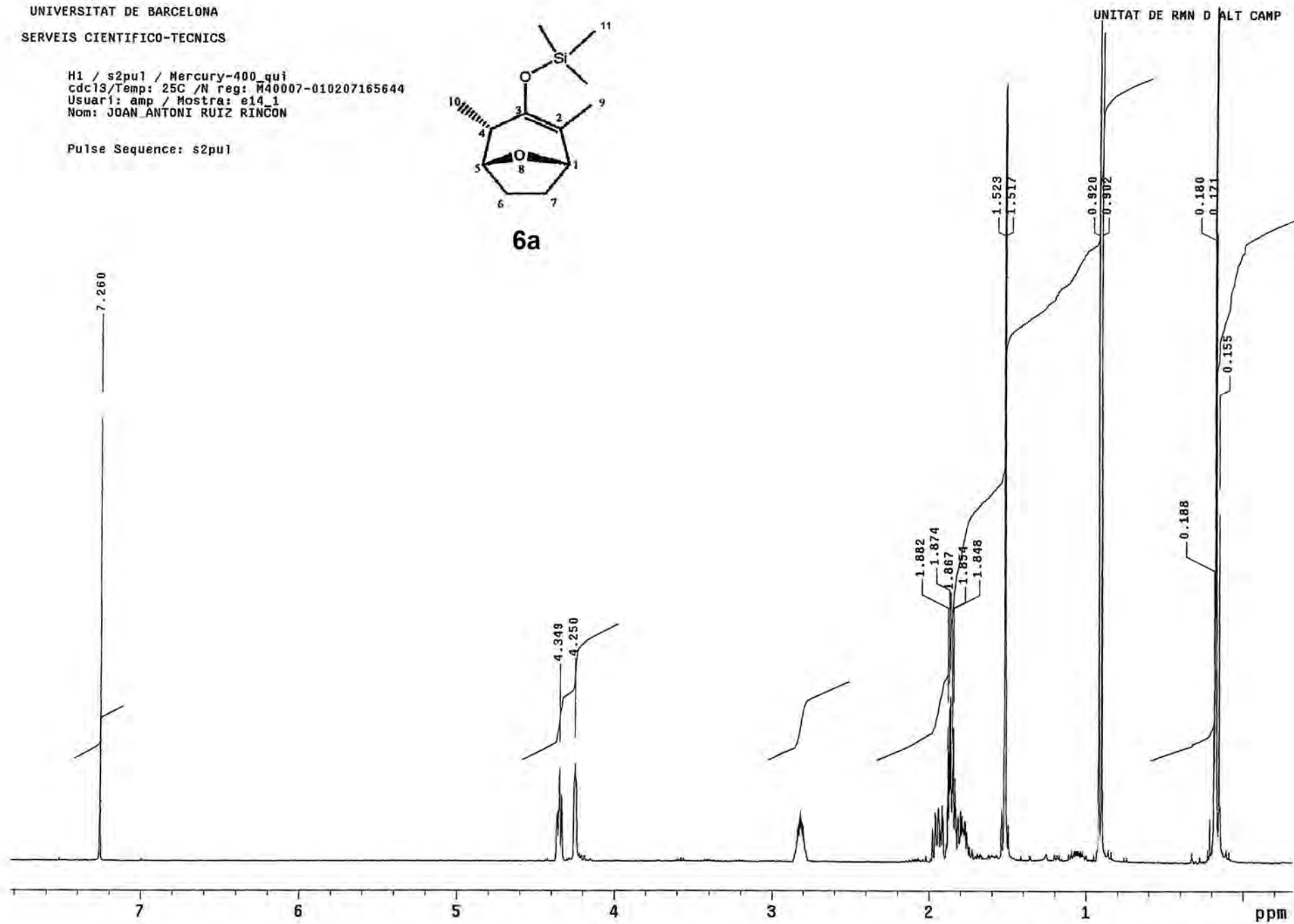
SERVEIS CIENTIFICOS-TECNICS

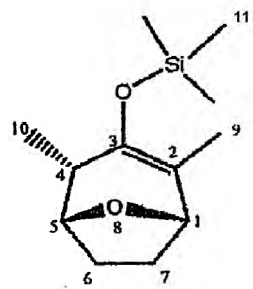
H1 / s2pu1 / Mercury-400. qui  
cdc13/Temp: 25C /N reg: M40007-010207165644  
Usuari: amp / Mostra: e14\_1  
Nom: JOAN ANTONI RUIZ RINCON

Pulse Sequence: s2pu1

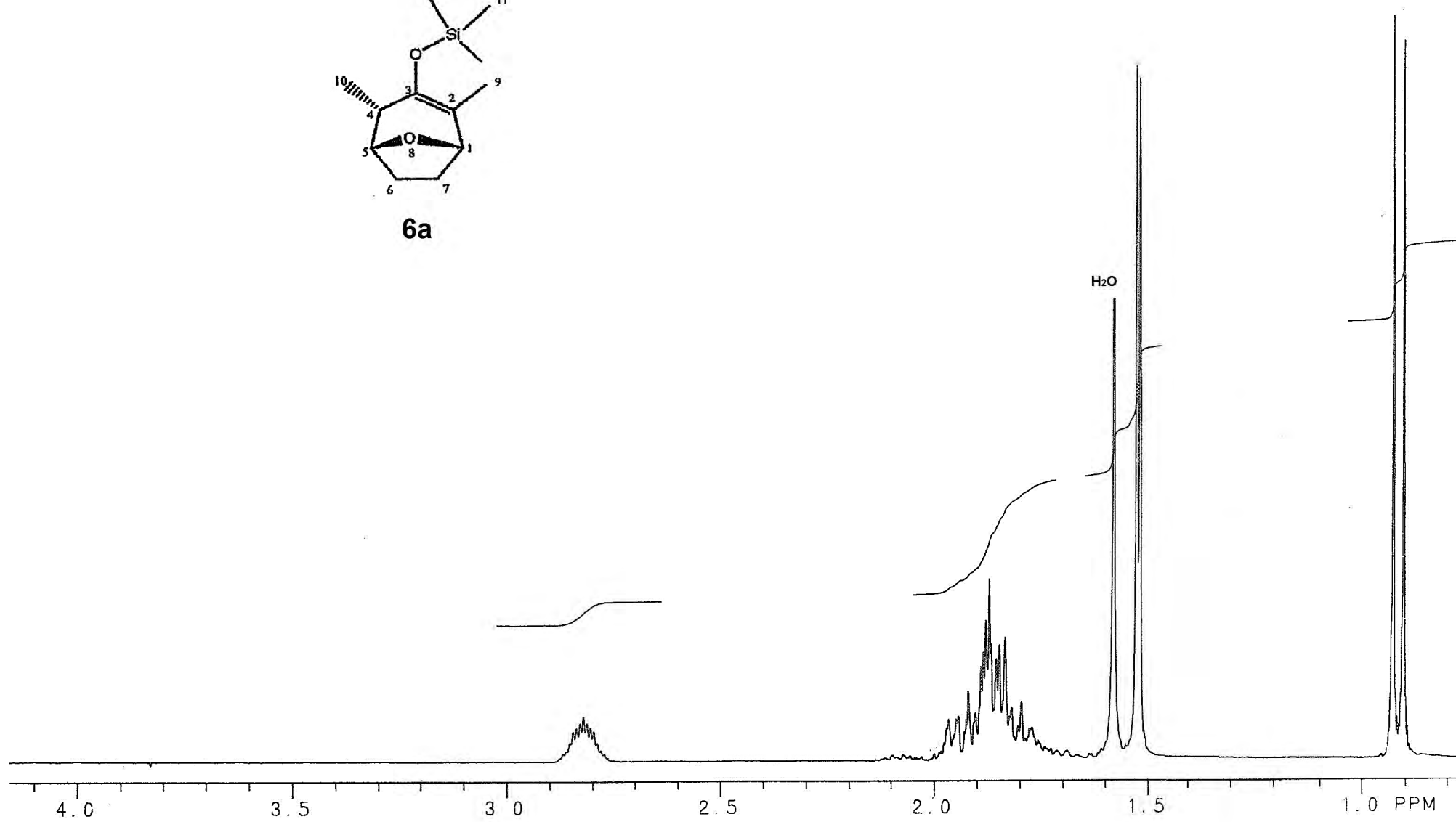


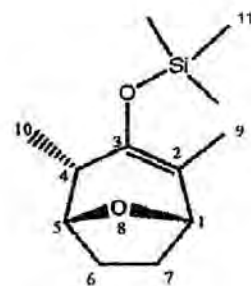
6a



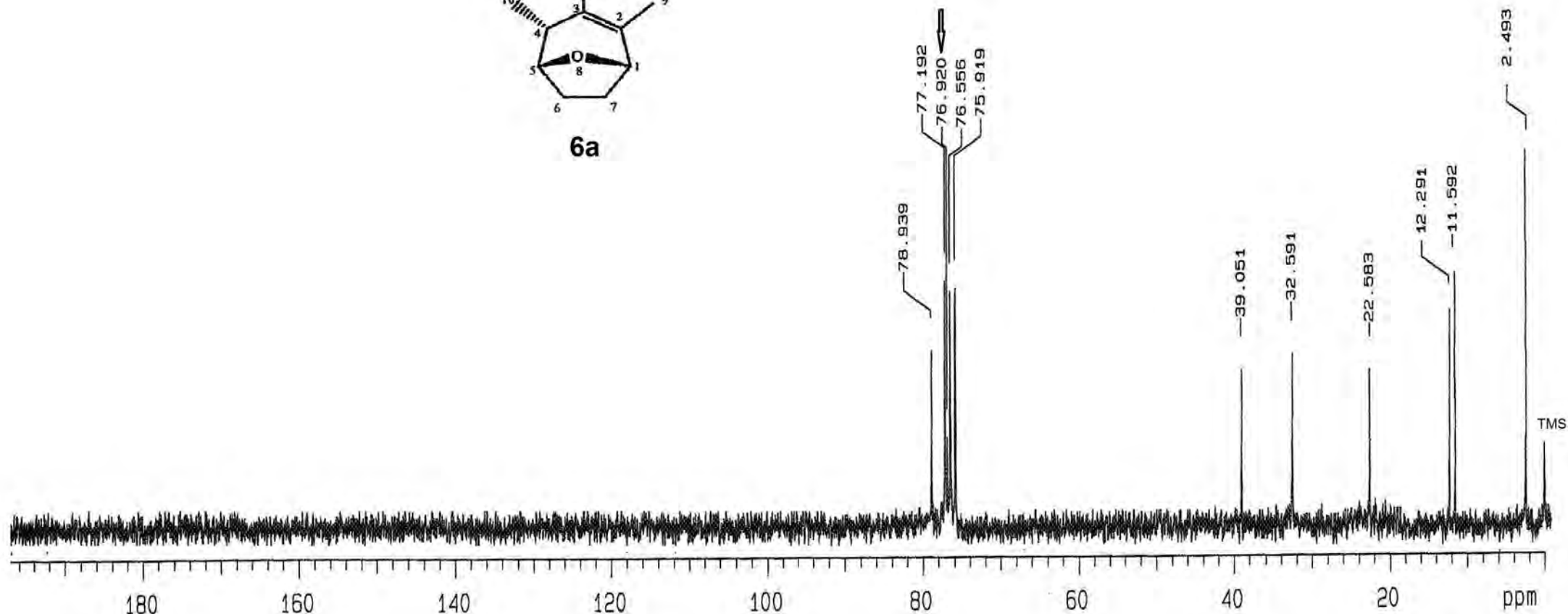


**6a**



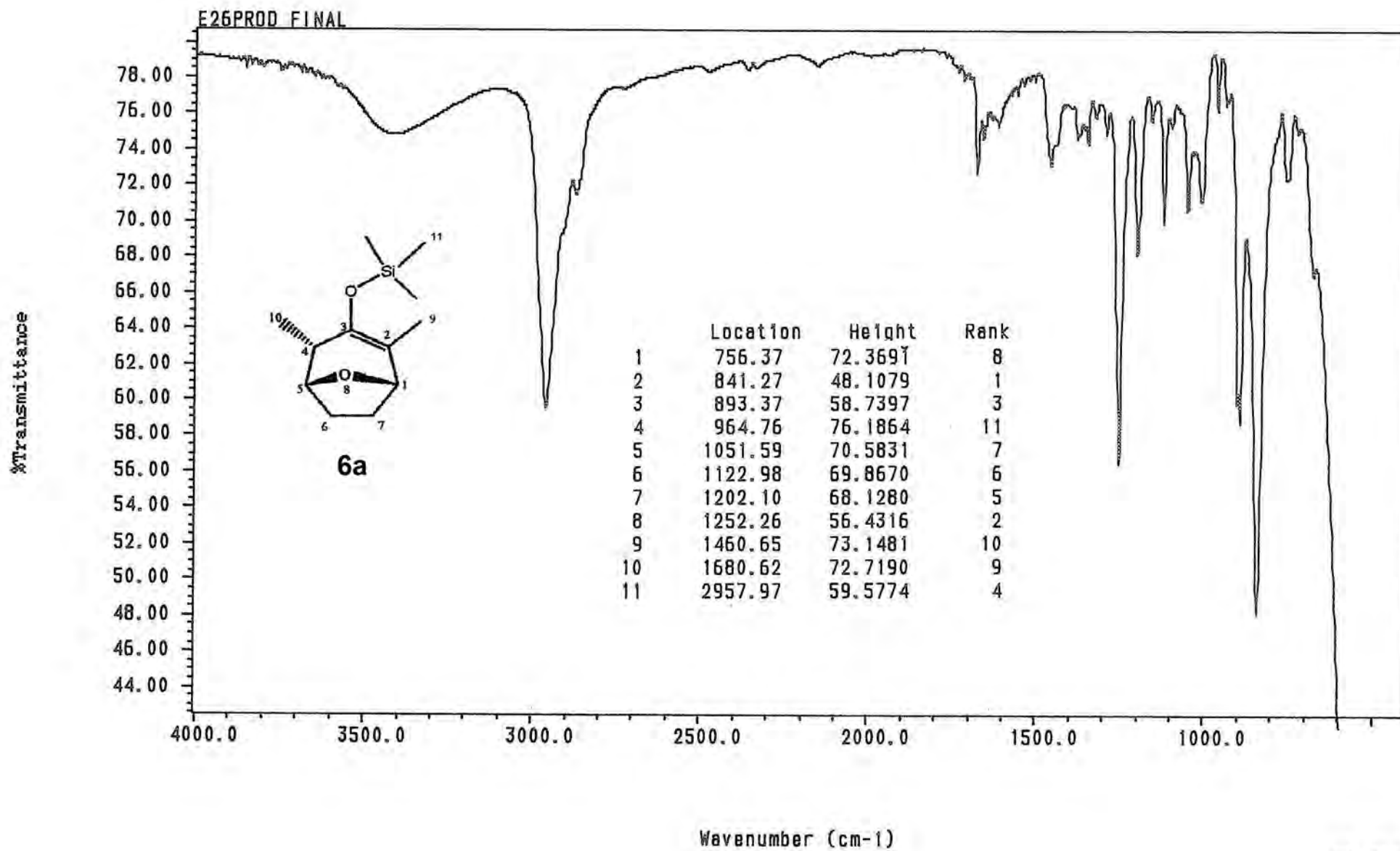


6a



|         |         |      |          |                |              |
|---------|---------|------|----------|----------------|--------------|
| C13     | 50.2894 | H1   | -300.0   | not used       | s2p1         |
| .4992.5 | 0       | yyy  | 20       | 1.000 not used | EXPBE26FINAL |
| 1.001   | 0.200   | s    | 10500    | 10058.1 -0.9   | not used     |
| 15.0    | 2500    | 29.0 | not used | cdc13          | usuario: jmv |

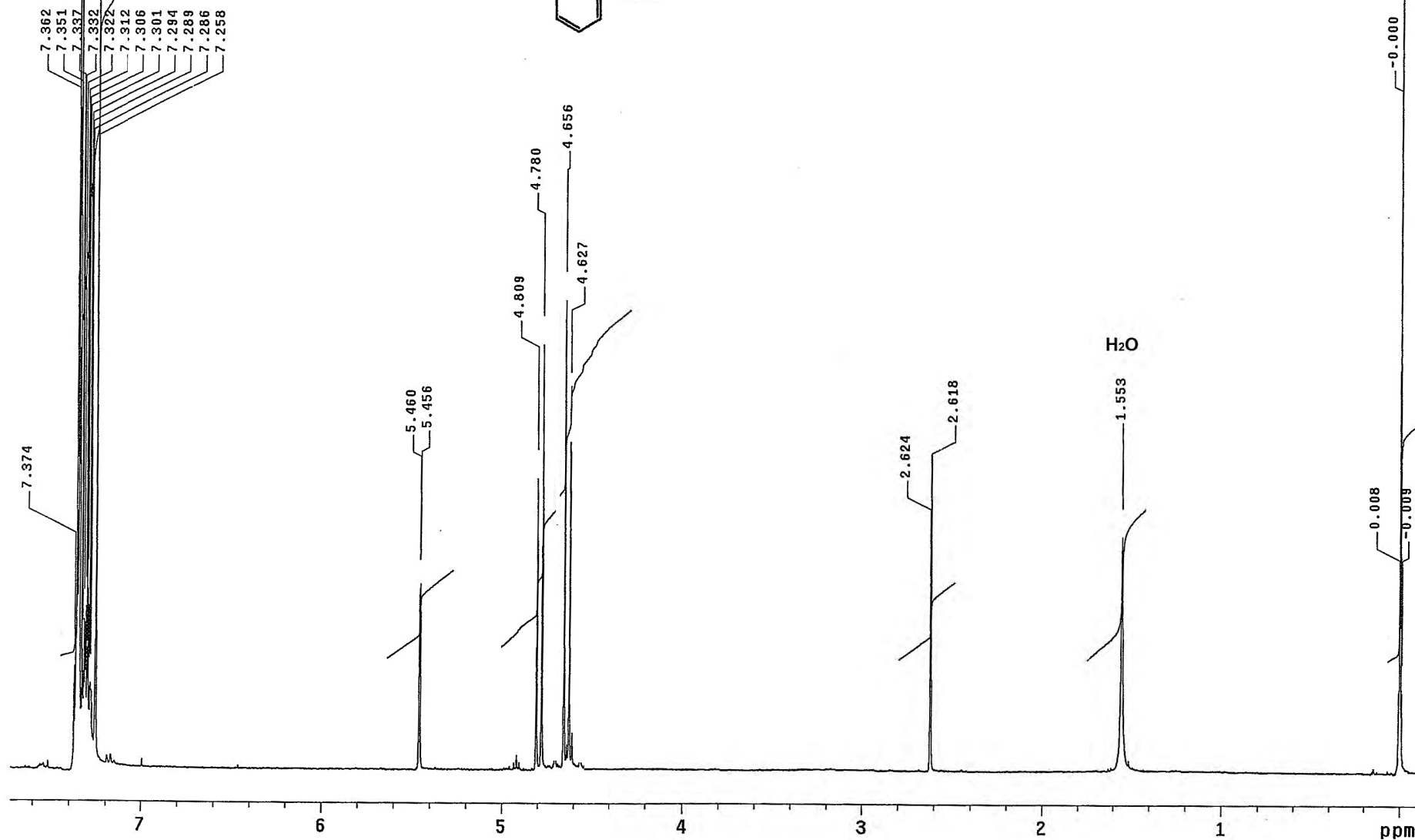
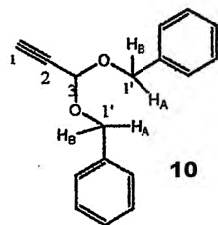
sample  
E26PROD FINAL



Nic/IR

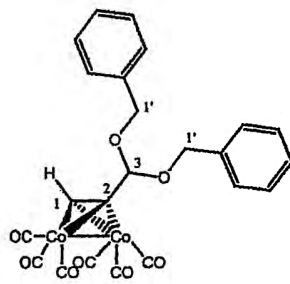
H1 / s2pu1 / Mercury-400\_qui  
c4c13/Temp: 25C /N reg: M40007-220107124234  
Usuari: amp / Mostra: e12\_5  
Npm: JOAN\_ANTI RUIZ RINCON  
Data

Pulse Sequence: s2pu1

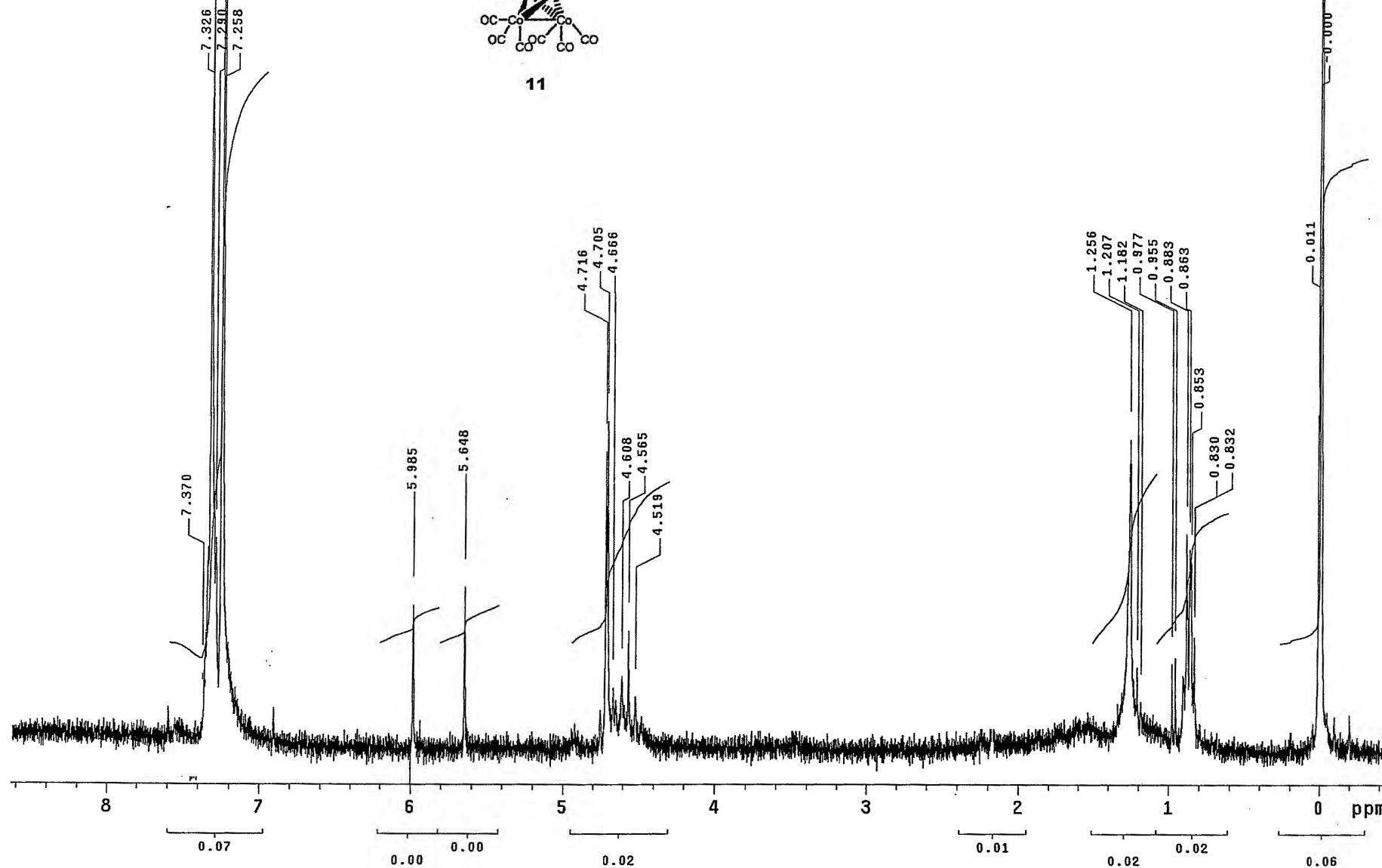


H1 / s2pu1 / Unitat 300  
cdc13/Temp: 25C / N reg: U30007-240107163135  
Usuari: amp / Mostra: e13-2  
Nom: JUAN-ANTONIO BARCIA UBEDA  
Sist automatic

Pulse Sequence: s2pu1

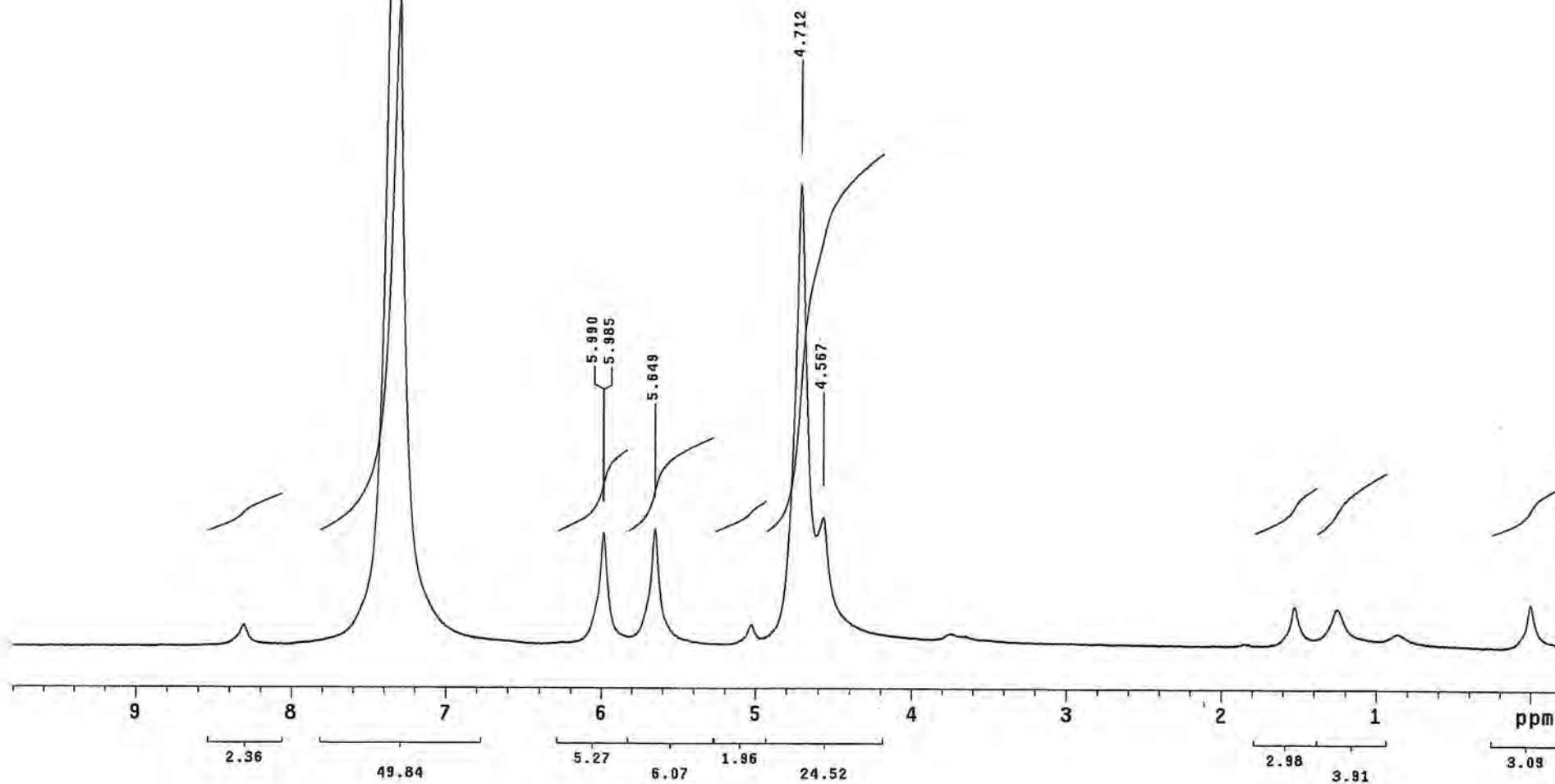
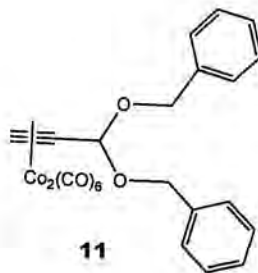


11



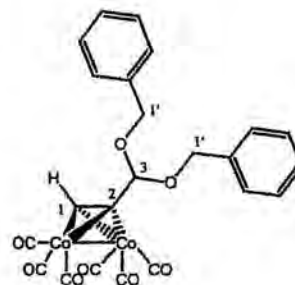
H1 / s2pu1 / Mercury-400.qui  
cdc13/Temp: 25C /N reg: M40006-080506180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pu1

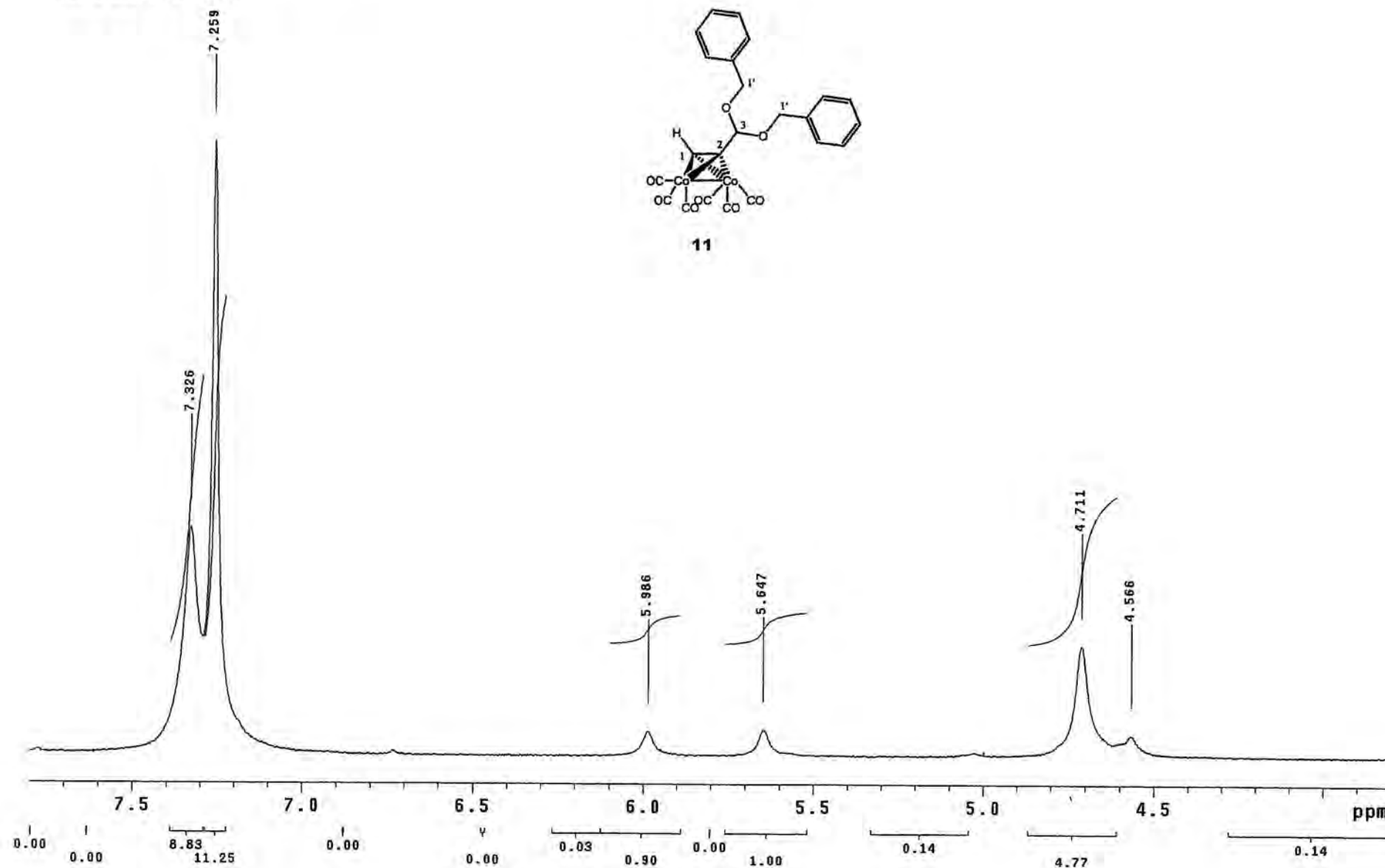


H1 / s2pu1 / Gem-200.DivIII  
LOC: #28 / Disolvent: cdc13/ temp ambient  
Usuari: amp/ Mostra: sp89\_1  
Nom: STEFANO PONZANO  
Sistema automatic

Pulse Sequence: s2pu1



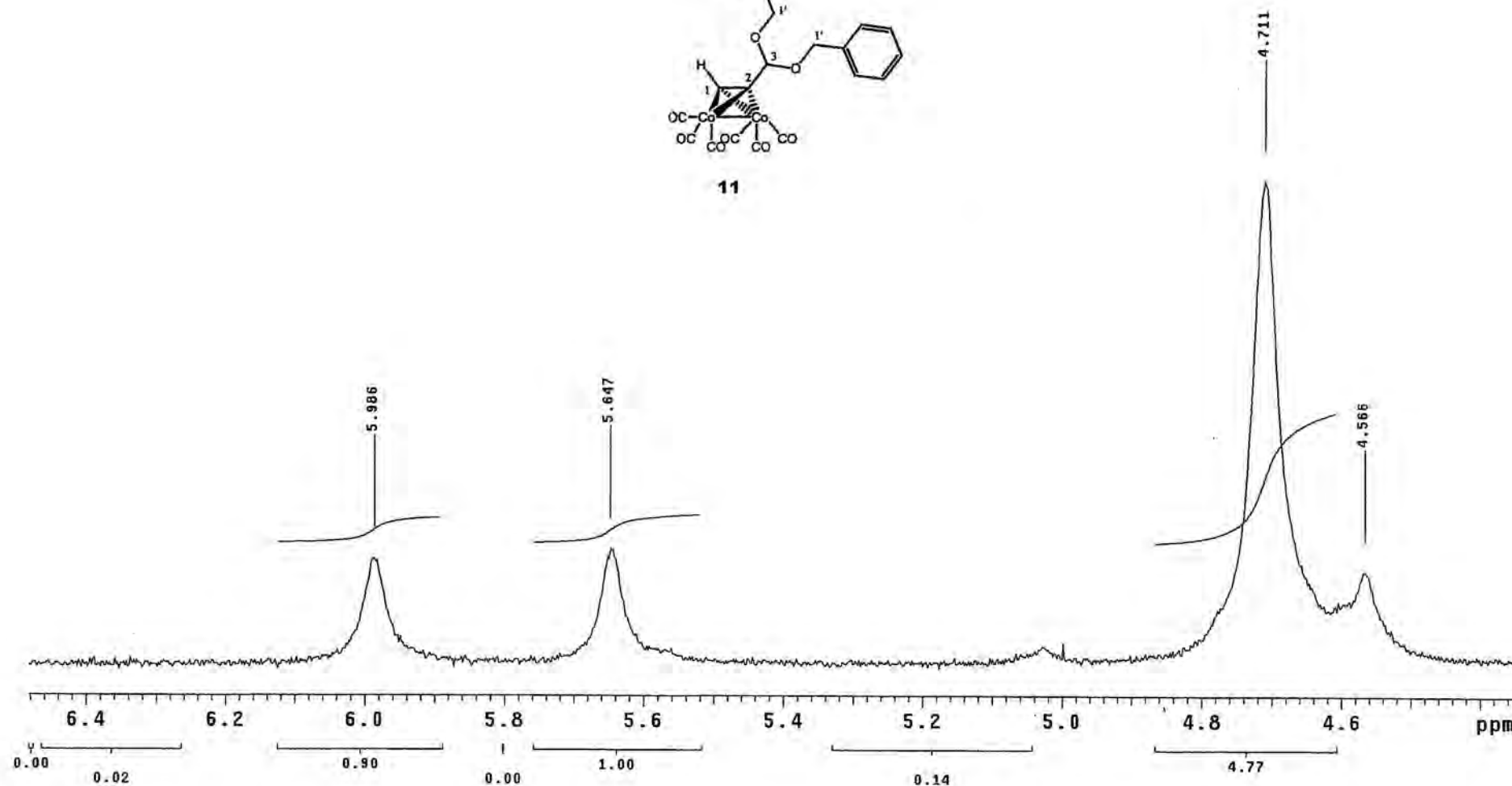
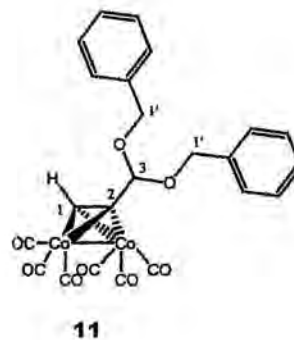
11



## SERVEIS CIENTIFICO-TECNICS

H1 / s2pu1 / Gem-200.DivIII  
LOC: #28 / Disolvent: cdc13/ temp ambient  
Usuari: amp/ Mostra: sp89\_1  
Nom: STEFANO PONZANO  
Sistema automatic

Pulse Sequence: s2pu1



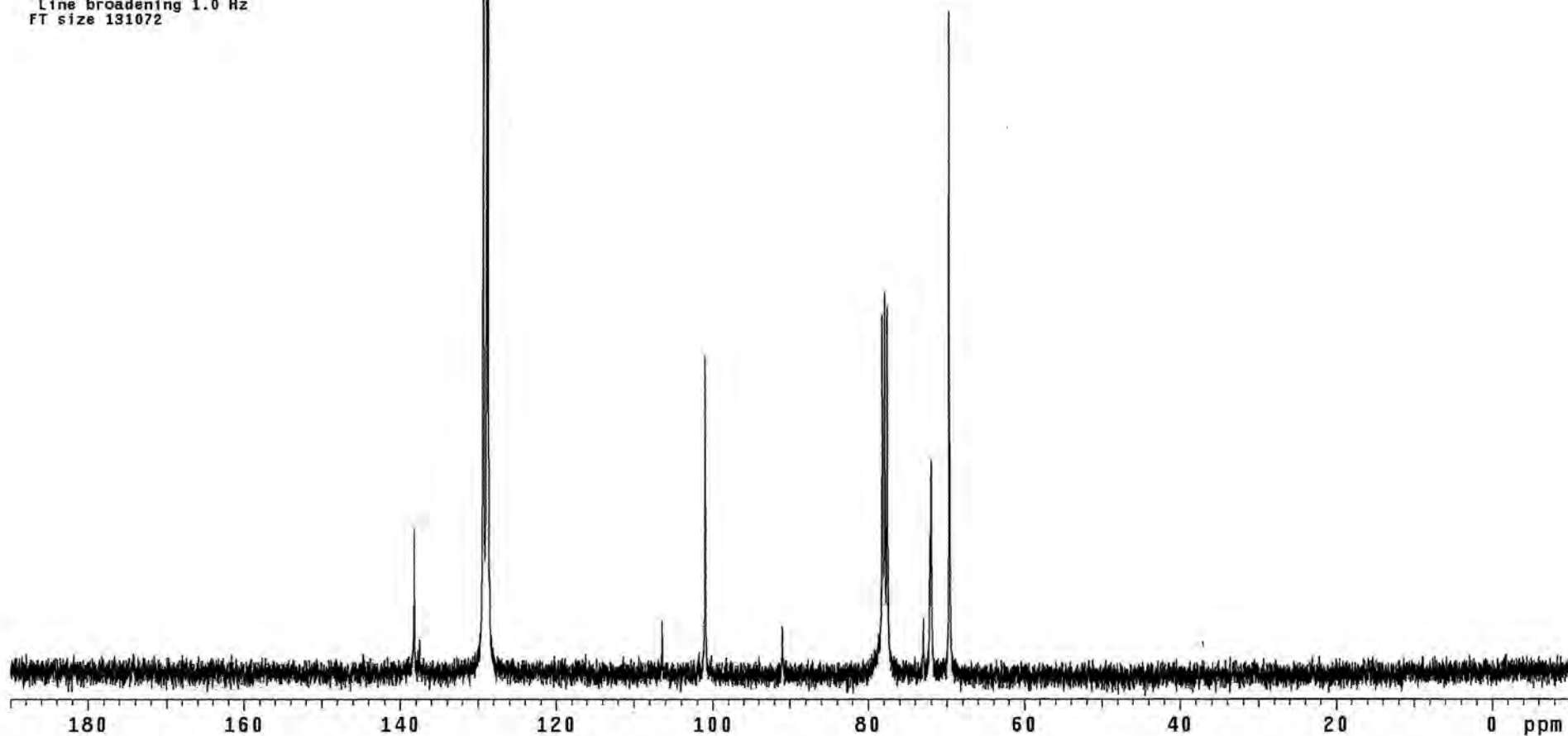
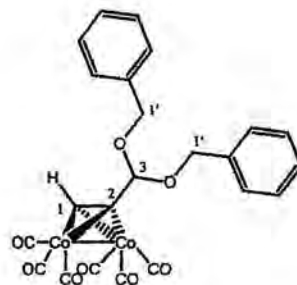
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

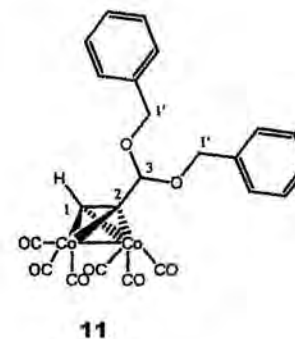
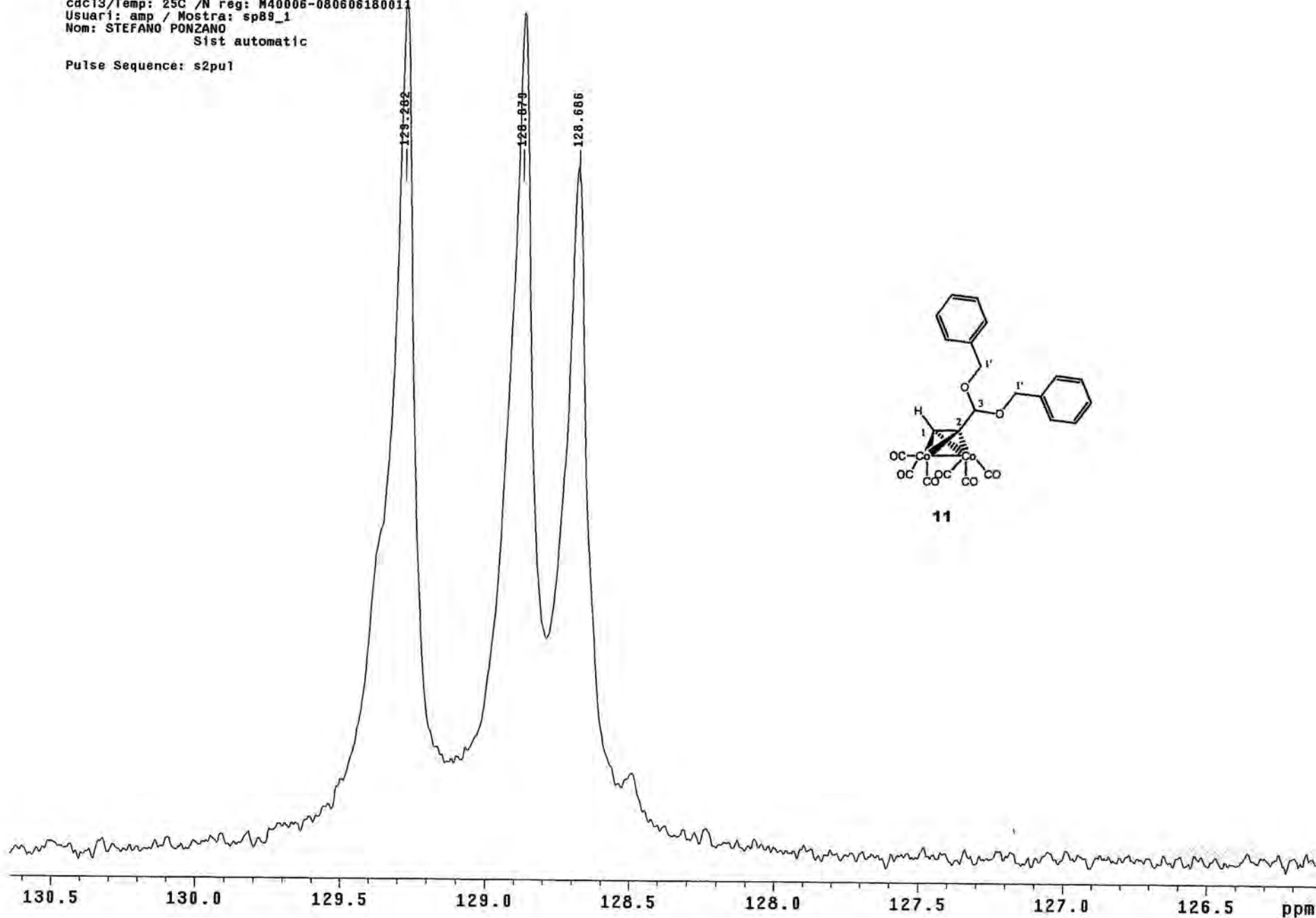
Pulse Sequence: s2pu1  
\*\*\*\*\*@\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072

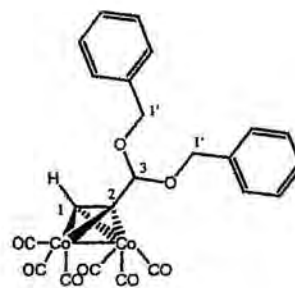


C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO  
Sist automatic  
Pulse Sequence: s2pu1

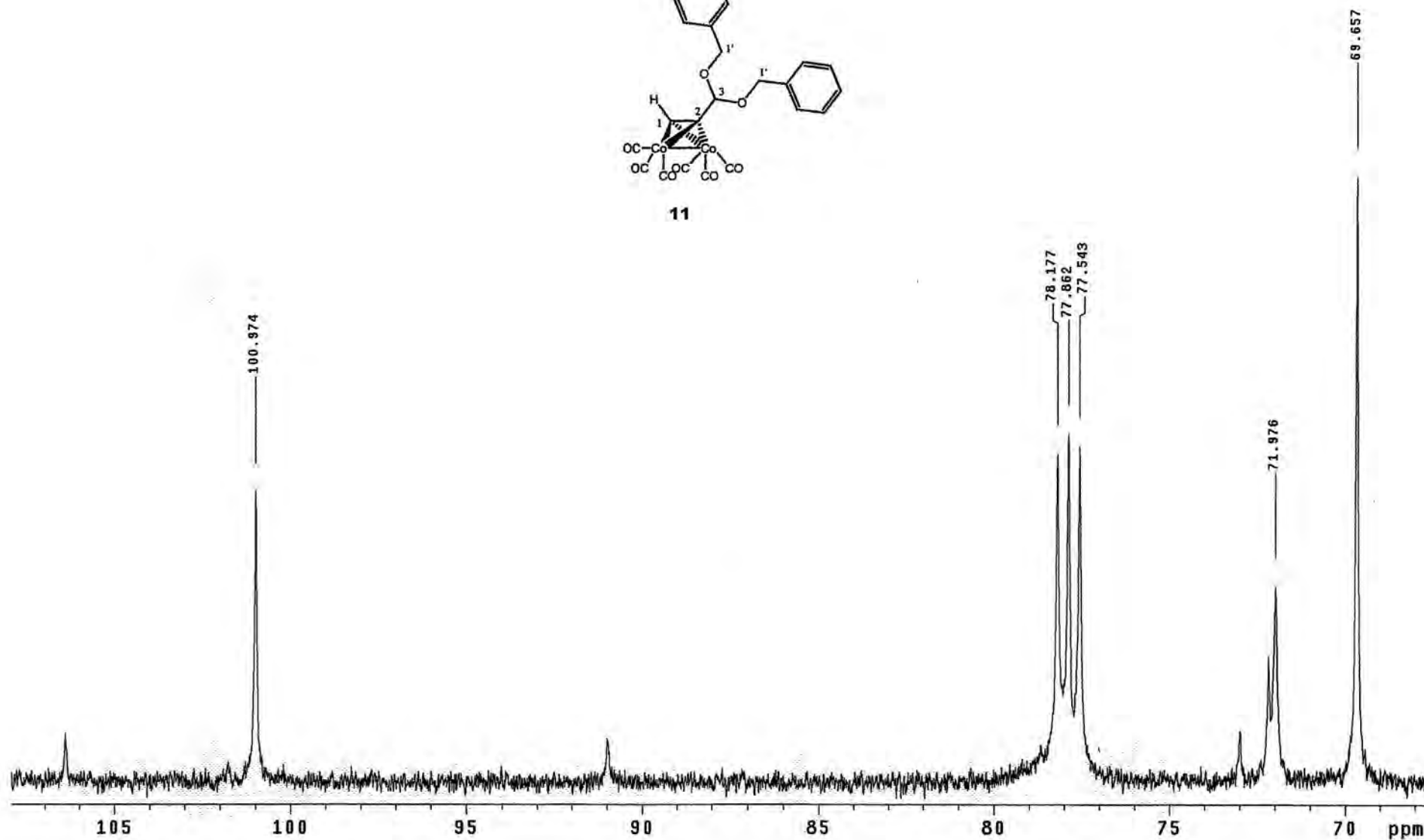


C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



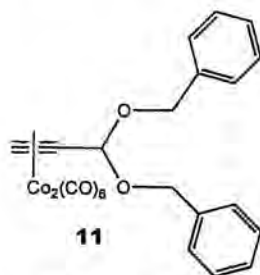
11



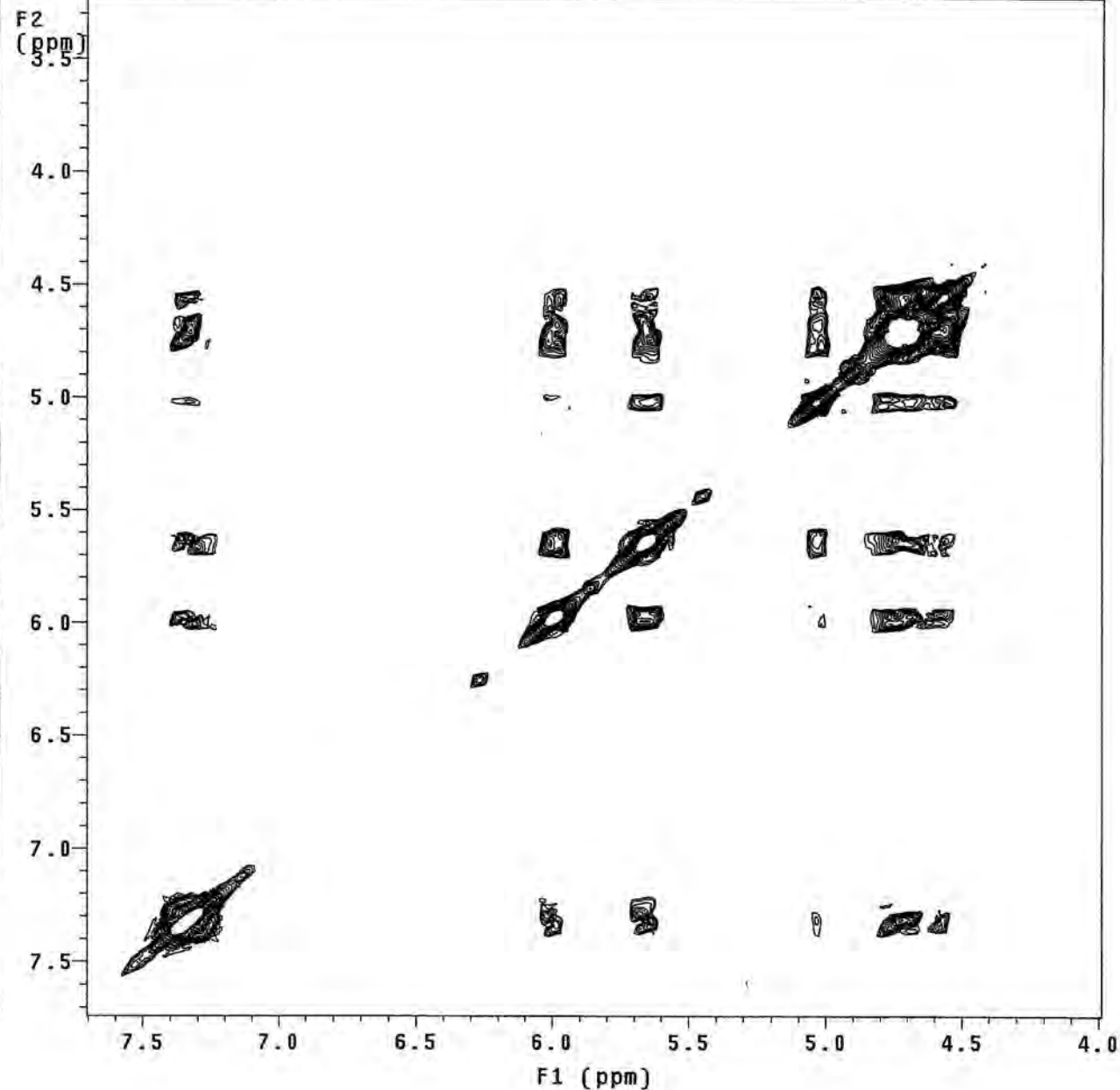
H1 / gCOSY / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-080606180  
011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

exp3 gCOSY

| SAMPLE             | FLAGS         | nn       |
|--------------------|---------------|----------|
| solvent cdc13      | hs            | n        |
| sample auto_Custom | sspul         | 1015     |
| Q_08Jun2006        | hsglv1        |          |
| SPECIAL            |               |          |
| ACQUISITION        | temp          | 25.0     |
| sw 3968.3          | gain          | 28       |
| at 0.150           | spin          | 0        |
| np 1190            | F2 PROCESSING |          |
| fb not used        | sb            | -0.075   |
| ss 16              | sbs           | not used |
| d1 1.000           | fn            | 2048     |
| nt 2               | F1 PROCESSING |          |
| 2D ACQUISITION     | sb1           | -0.065   |
| sw1 3968.3         | sbs1          | not used |
| ni 256             | proc1         | lp       |
| PRESATURATION      | fn1           | 2048     |
| satmode n          | DISPLAY       |          |
| satfrq 0           | sp            | 1291.5   |
| satdly 0           | wp            | 1803.8   |
| satpwr 0           | sp1           | 1596.1   |
| TRANSMITTER        | wp1           | 1485.7   |
| tn H1              | rfl           | 733.4    |
| sfrq 400.113       | rfp           | 0        |
| tof -519.2         | rfl1          | 727.5    |
| tpwr 59            | rfp1          | 0        |
| pw 11.400          | PLOT          |          |
| GRADIENTS          | wc            | 153.0    |
| gzlv11 1015        | sc            | 6.2      |
| gt1 0.001000       | wc2           | 153.0    |
| gstap 0.000500     | sc2           | 0        |
| DECOUPLING         |               | 3        |
| dn                 |               | 7        |
| dm                 |               |          |



REF: EXP 89  
Libreta n° 2  
(Experimento 89)



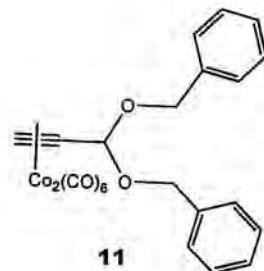
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gCOSY / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

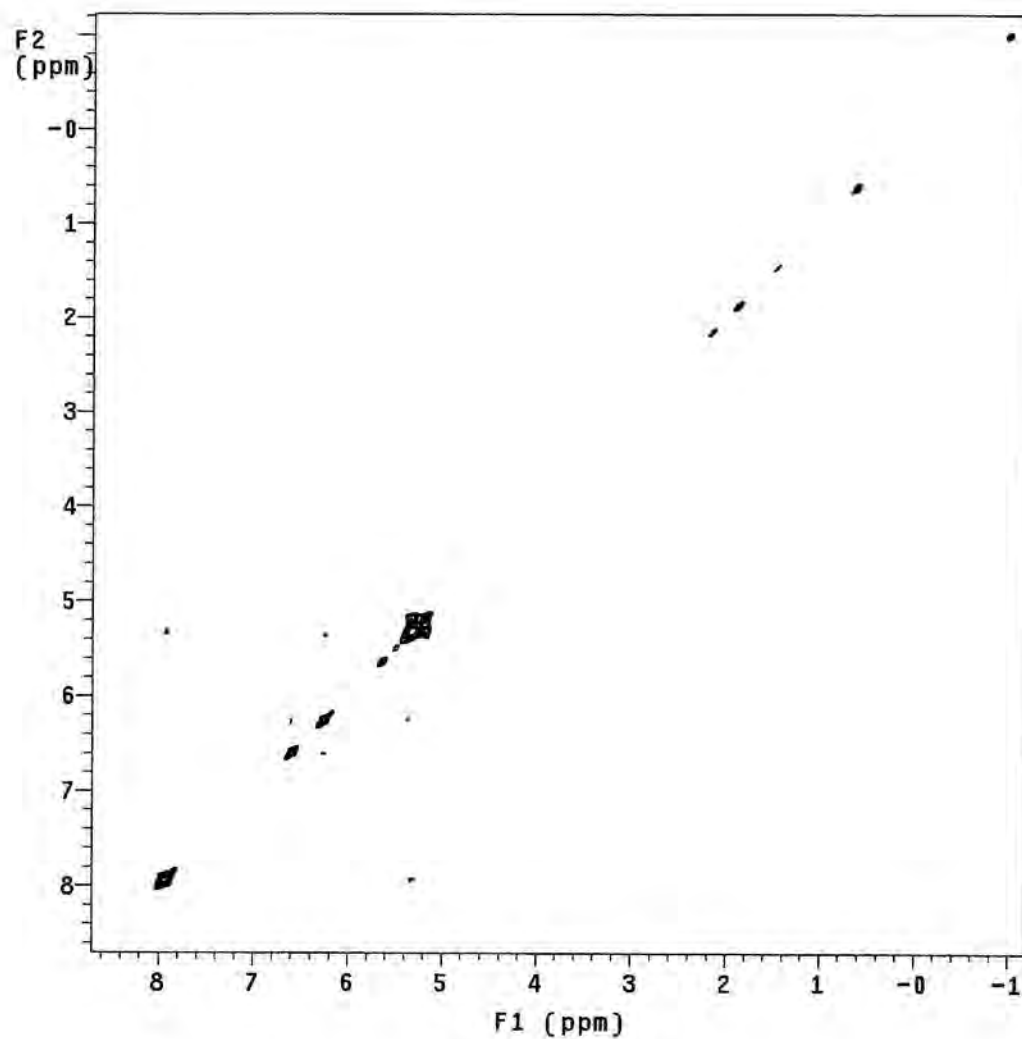
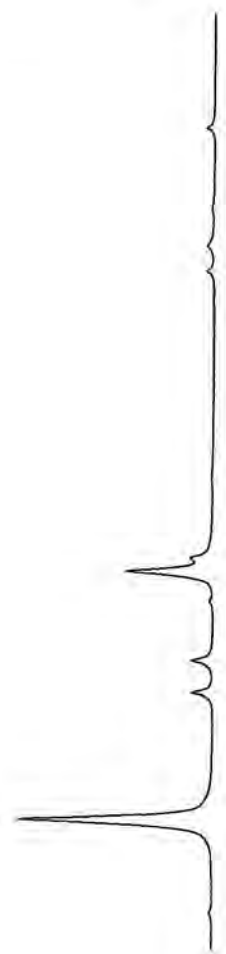
Pulse Sequence: gCOSY  
\*\*\*\*\*@\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3968.3 Hz  
2D Width 3968.3 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.065 sec  
FT size 2048 x 2048



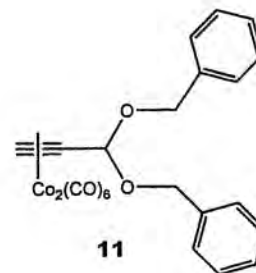
REF: EXP 89  
Libreta n° 2  
(Experimento 89)



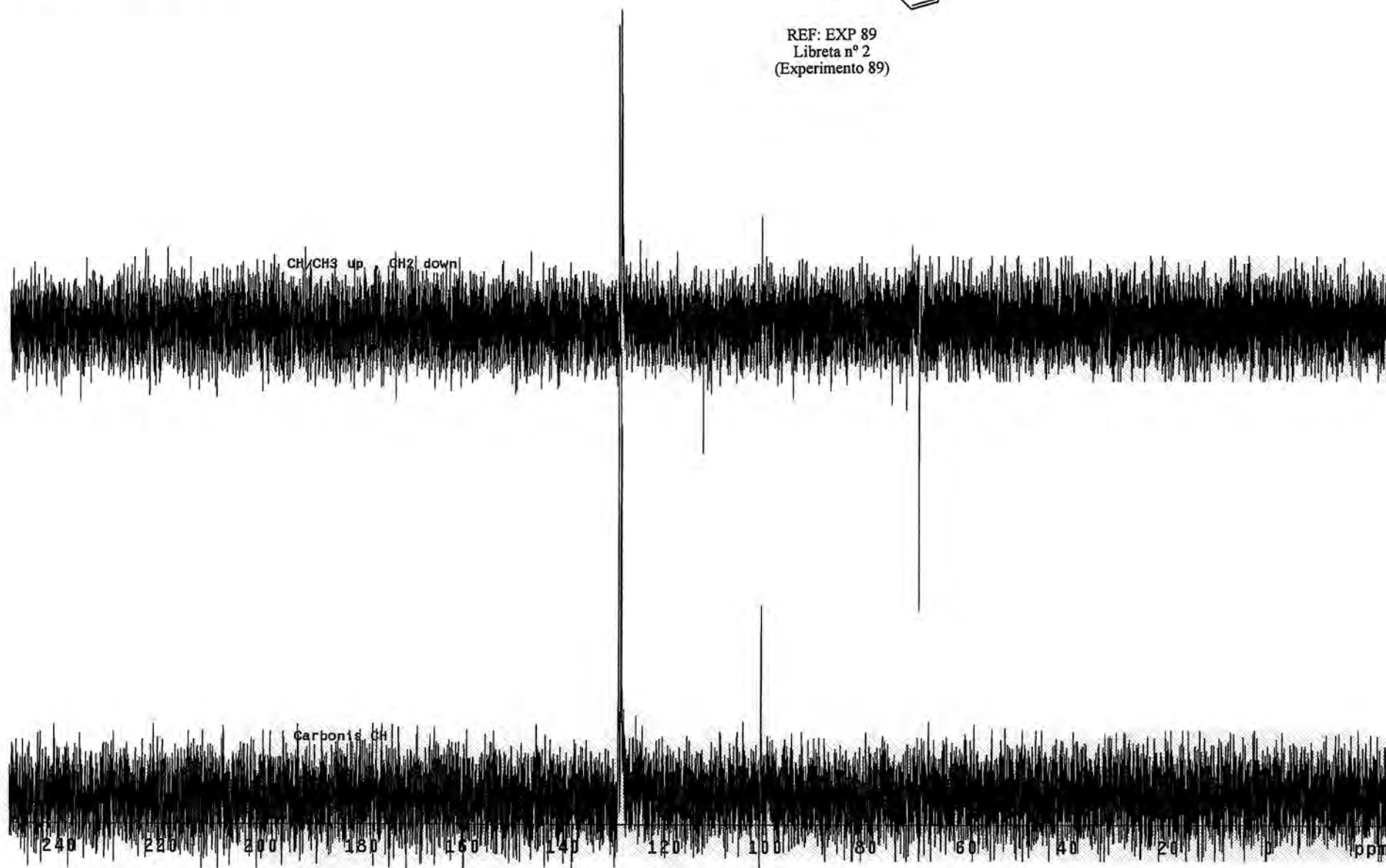
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / DEPT / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

Pulse Sequence: DEPT  
\*\*\*\*\*



REF: EXP 89  
Libreta nº 2  
(Experimento 89)



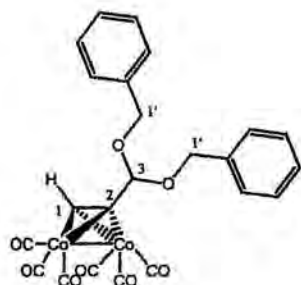
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*

H1 / gHSQC / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

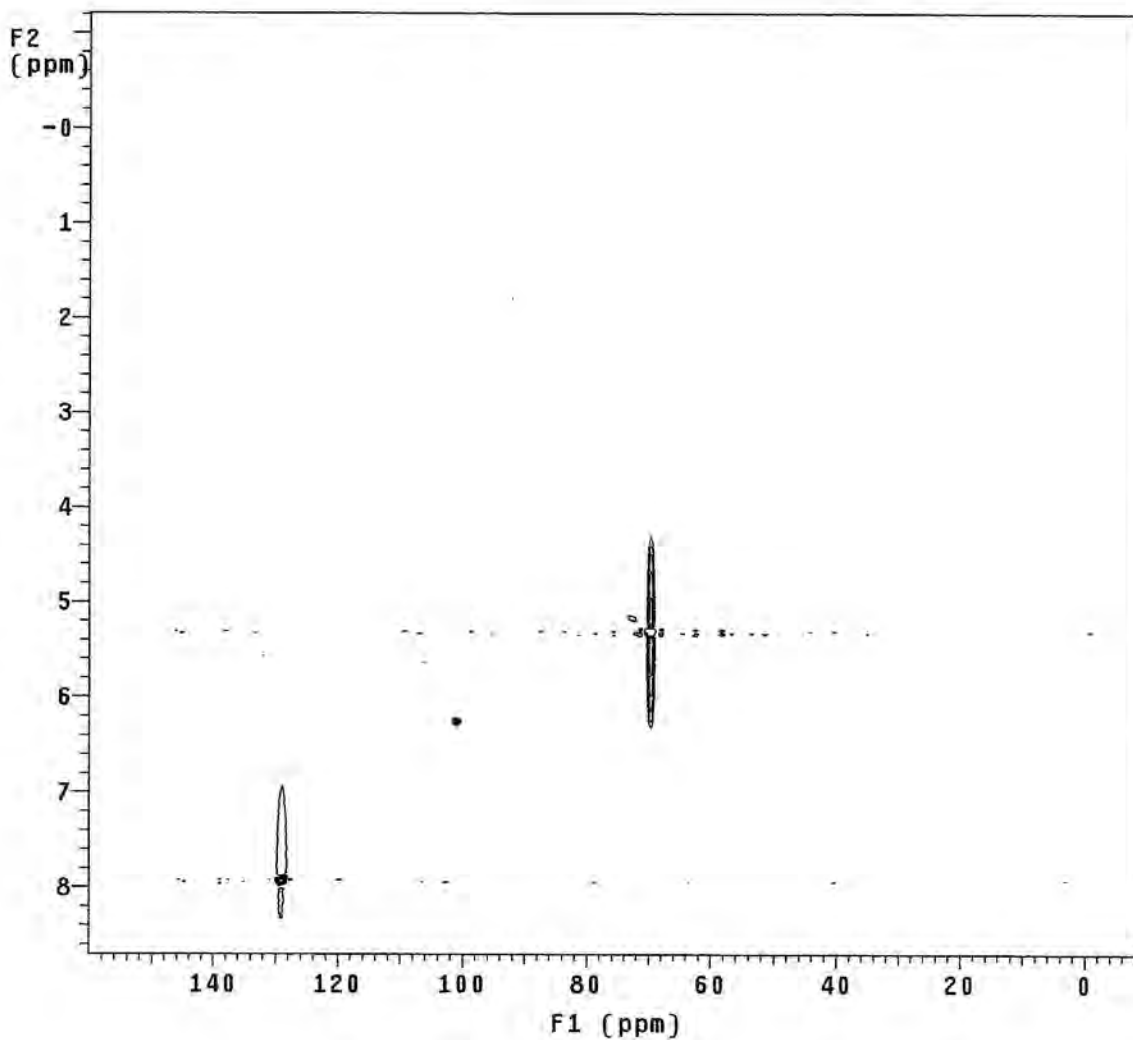
Pulse Sequence: gHSQC  
\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.129 sec  
Width 3968.3 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048

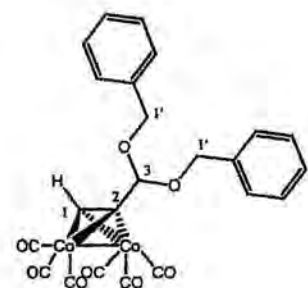


11

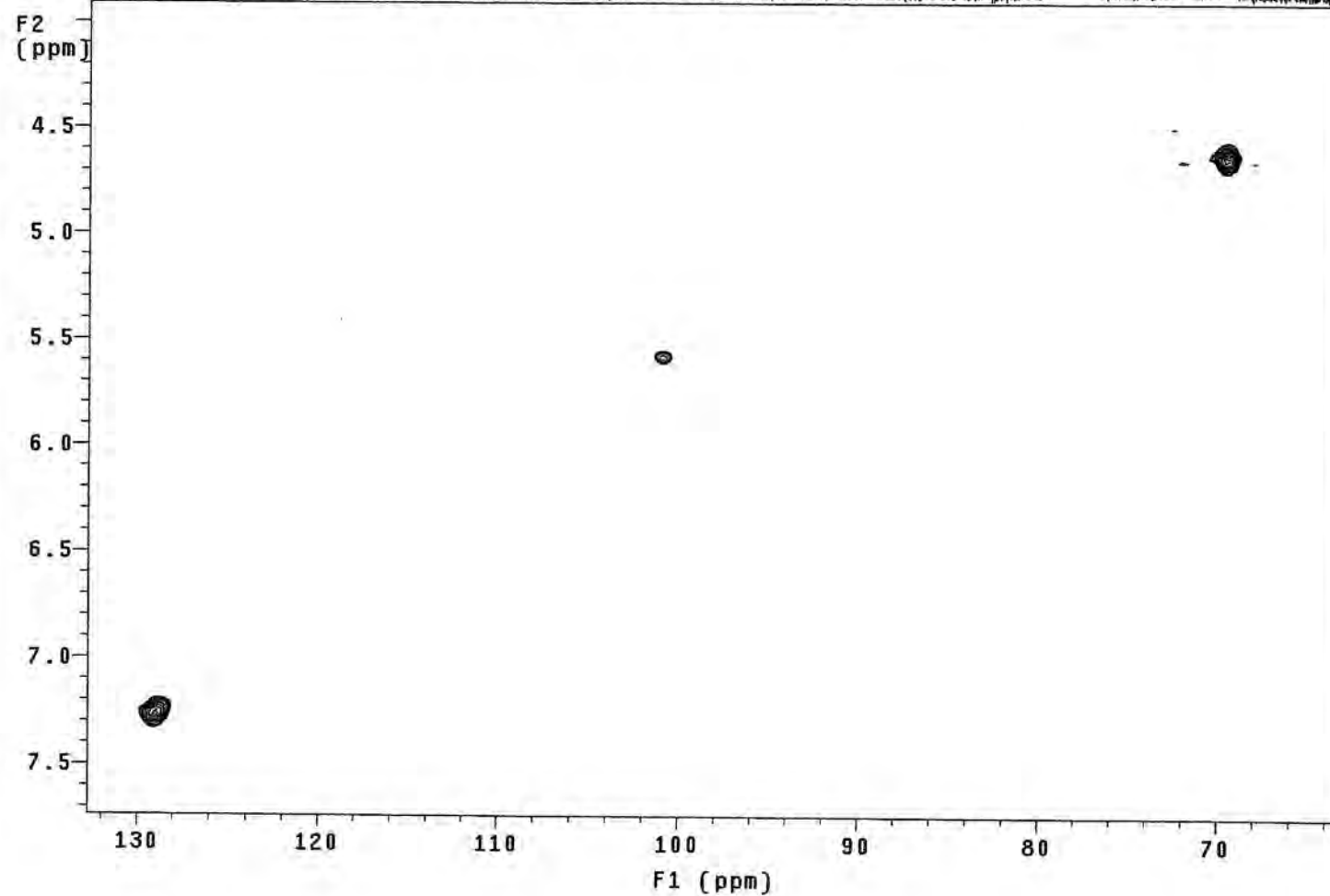


H1 / gHSQC / Mercury-400\_qul  
cdc13/Temp: 25C /N reg: M40006-080606180011  
Usuari: amp / Mostra: sp89\_1  
Nom: STEFANO PONZANO

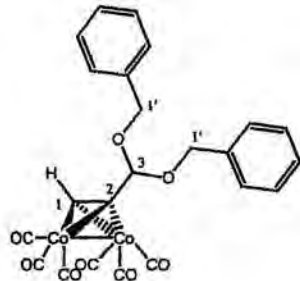
Pulse Sequence: gHSQC



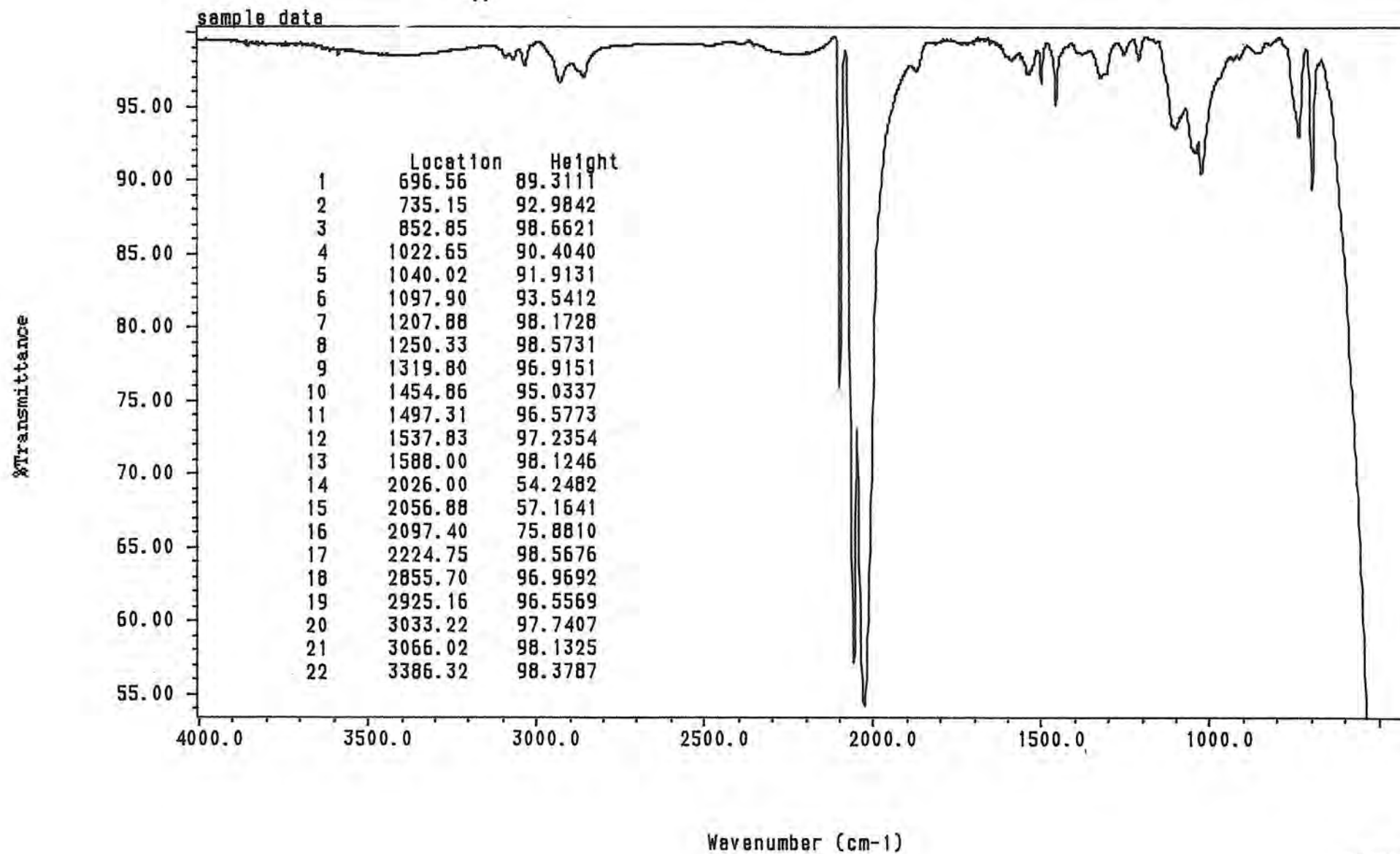
11



sp92-1  
sample data



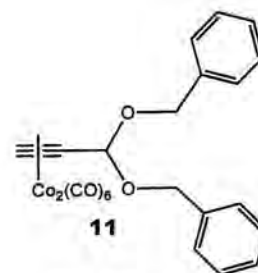
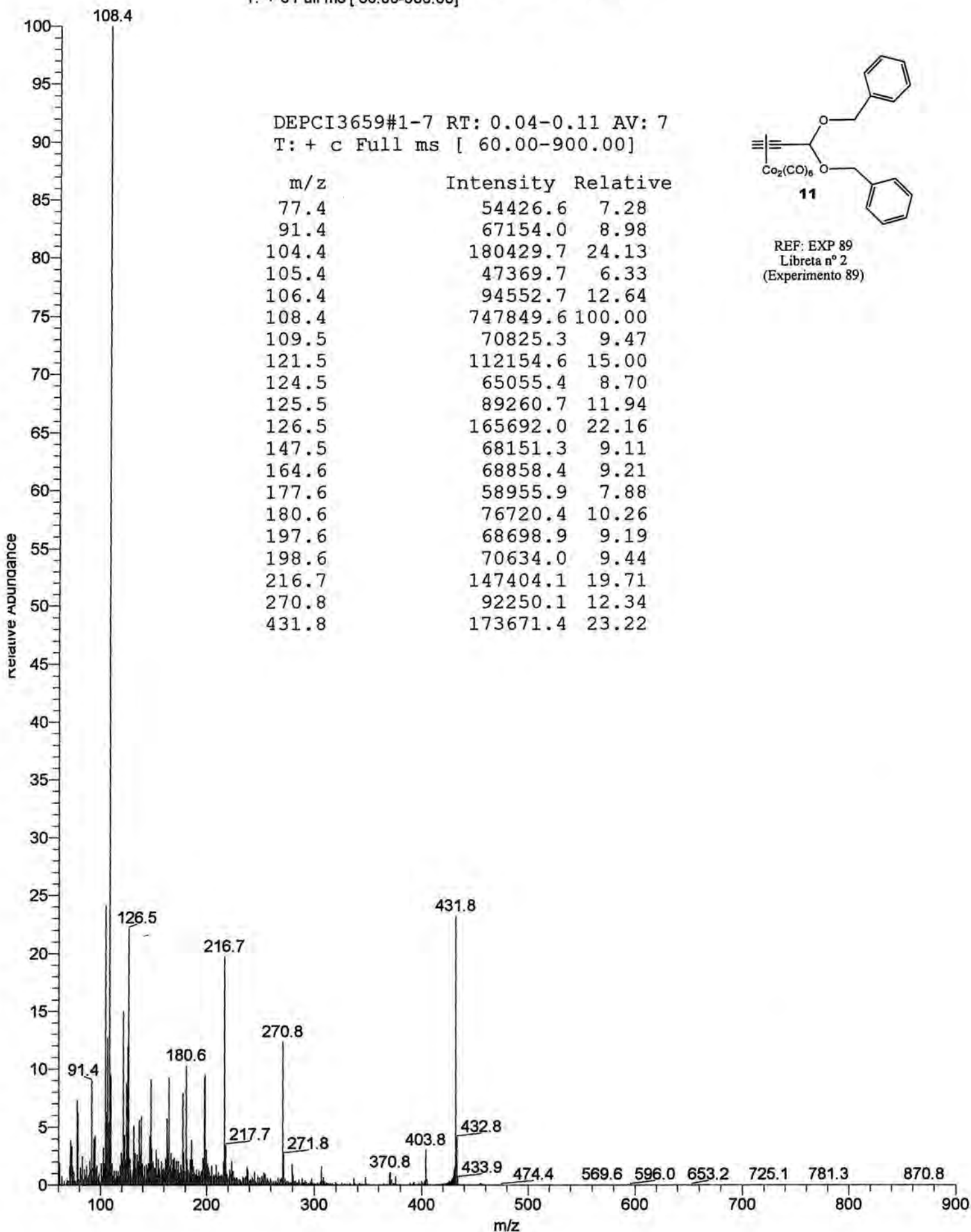
11

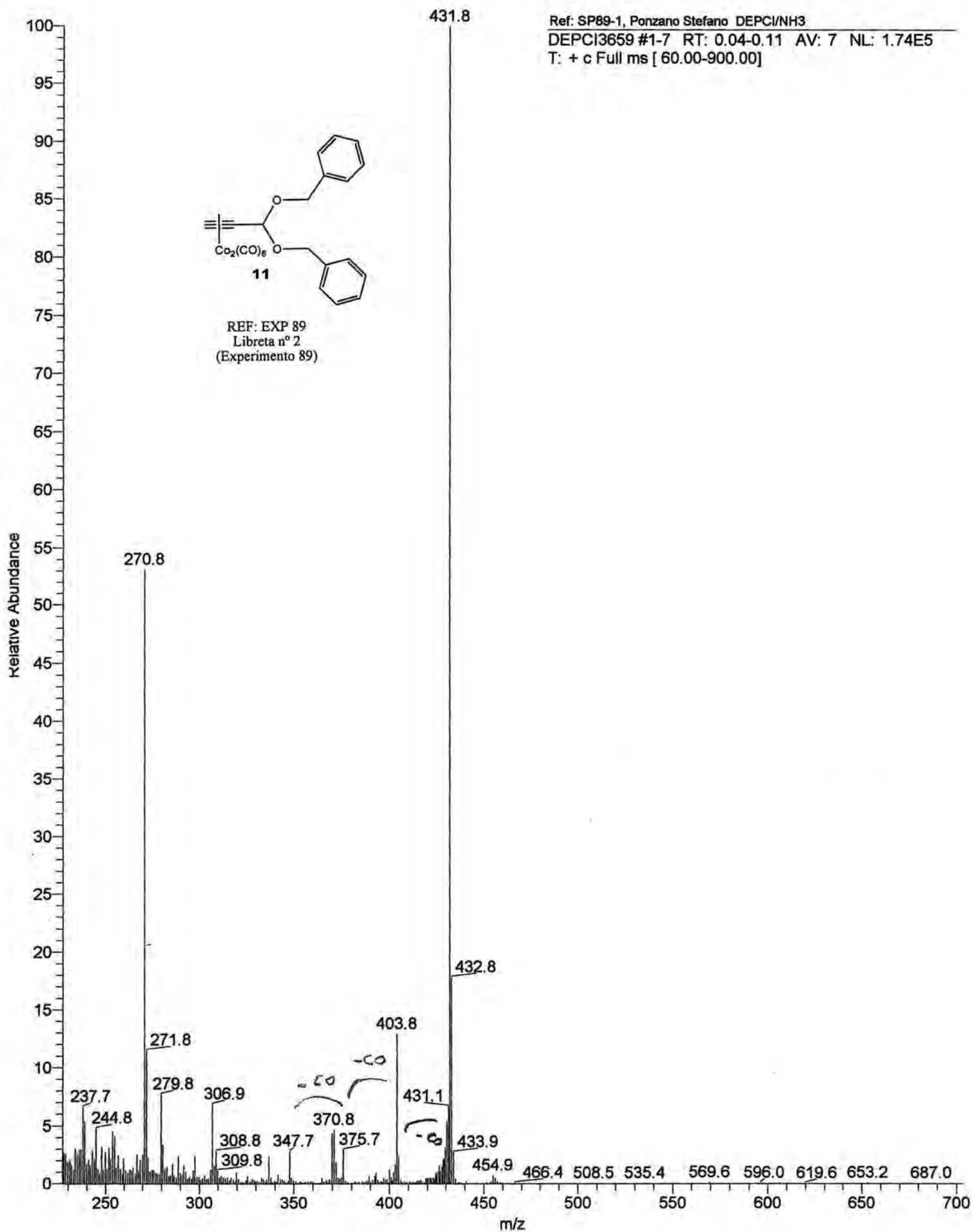


N1c/IR

DEPCI3659#1-7 RT: 0.04-0.11 AV: 7  
T: + c Full ms [ 60.00-900.00]

| m/z   | Intensity | Relative |
|-------|-----------|----------|
| 77.4  | 54426.6   | 7.28     |
| 91.4  | 67154.0   | 8.98     |
| 104.4 | 180429.7  | 24.13    |
| 105.4 | 47369.7   | 6.33     |
| 106.4 | 94552.7   | 12.64    |
| 108.4 | 747849.6  | 100.00   |
| 109.5 | 70825.3   | 9.47     |
| 121.5 | 112154.6  | 15.00    |
| 124.5 | 65055.4   | 8.70     |
| 125.5 | 89260.7   | 11.94    |
| 126.5 | 165692.0  | 22.16    |
| 147.5 | 68151.3   | 9.11     |
| 164.6 | 68858.4   | 9.21     |
| 177.6 | 58955.9   | 7.88     |
| 180.6 | 76720.4   | 10.26    |
| 197.6 | 68698.9   | 9.19     |
| 198.6 | 70634.0   | 9.44     |
| 216.7 | 147404.1  | 19.71    |
| 270.8 | 92250.1   | 12.34    |
| 431.8 | 173671.4  | 23.22    |

REF: EXP 89  
Libreta n° 2  
(Experimento 89)



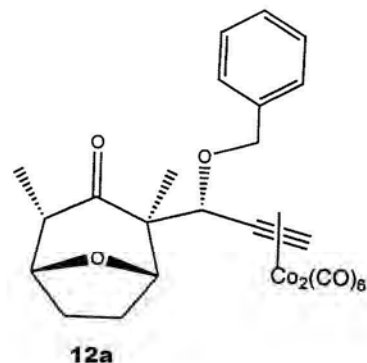
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFIC-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*

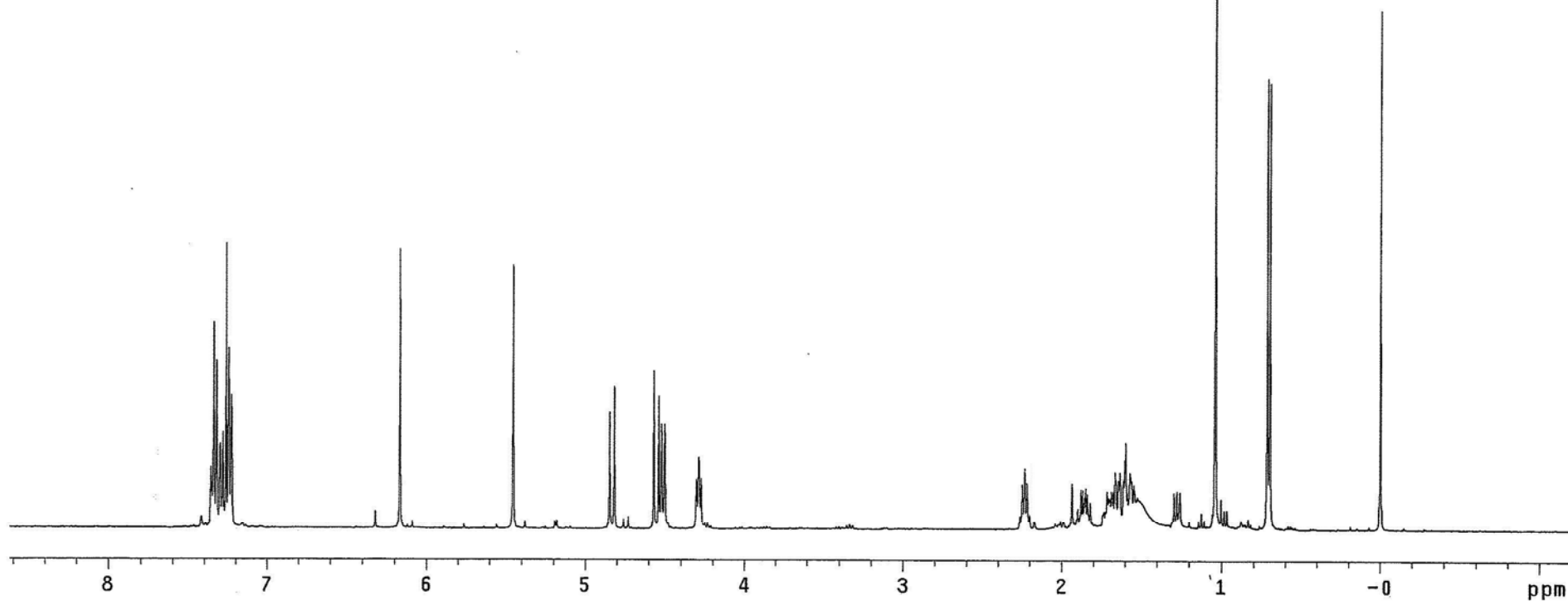
Mercury-400BB "fenix"

Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.503 sec  
Width 3937.0 Hz  
16 repetitions  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 32768



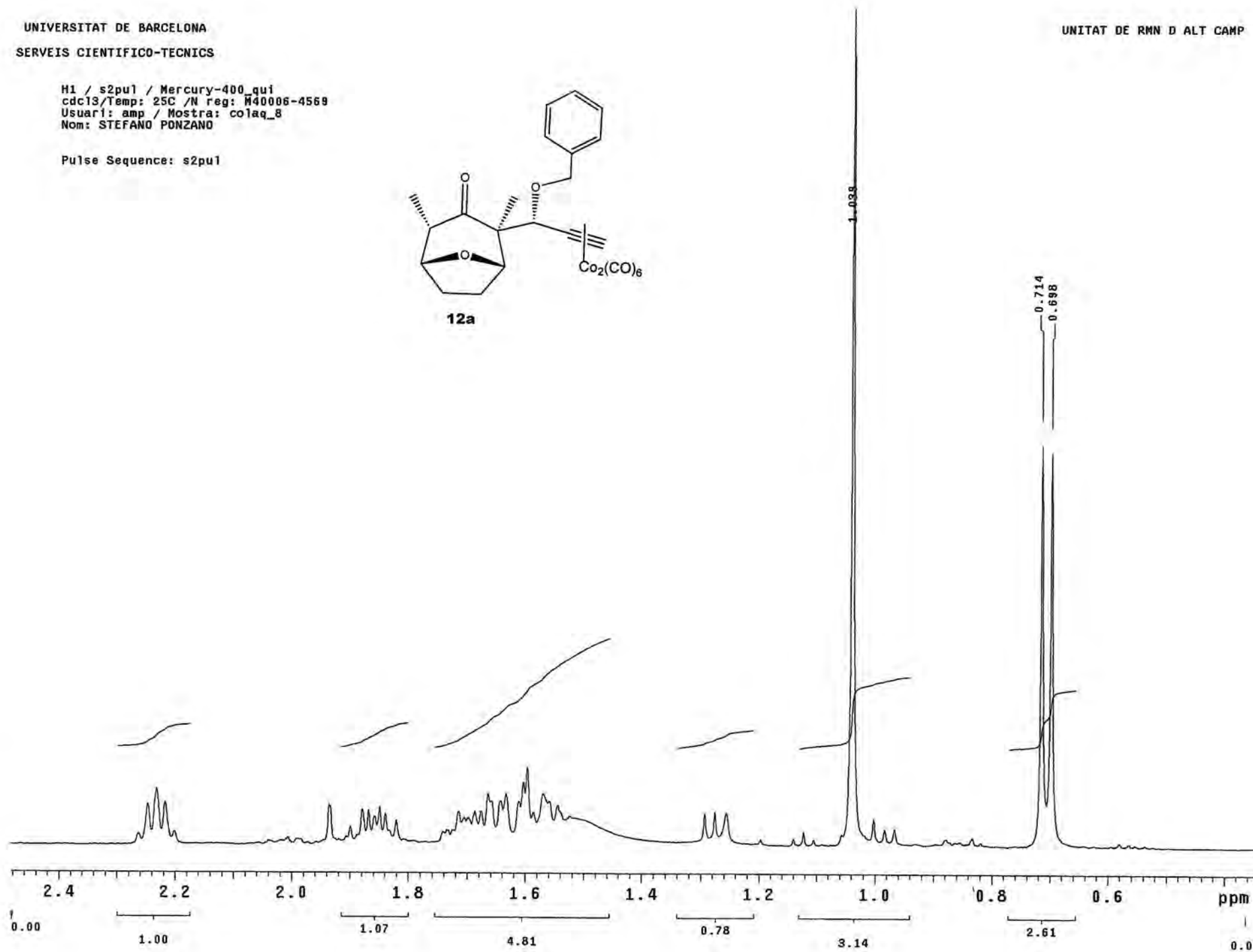
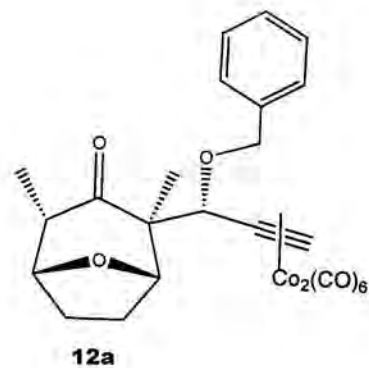
REF: COL AQ-8  
Libreta n° 3  
(COLUMN AQ)

| INDEX | FREQUENCY | PPM    | HEIGHT |
|-------|-----------|--------|--------|
| 1     | 2936.416  | 7.339  | 32.6   |
| 2     | 2928.967  | 7.320  | 26.5   |
| 3     | 2904.937  | 7.260  | 45.3   |
| 4     | 2899.410  | 7.247  | 25.5   |
| 5     | 2897.968  | 7.243  | 28.4   |
| 6     | 2467.839  | 6.168  | 44.9   |
| 7     | 2182.127  | 5.454  | 41.7   |
| 8     | 1926.933  | 4.816  | 22.6   |
| 9     | 1827.450  | 4.567  | 24.9   |
| 10    | 415.712   | 1.039  | 151.2  |
| 11    | 285.952   | 0.715  | 71.8   |
| 12    | 279.224   | 0.698  | 71.1   |
| 13    | -0.000    | -0.000 | 82.6   |



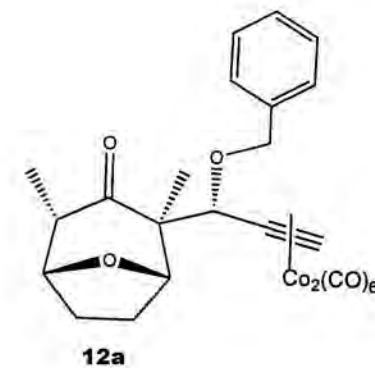
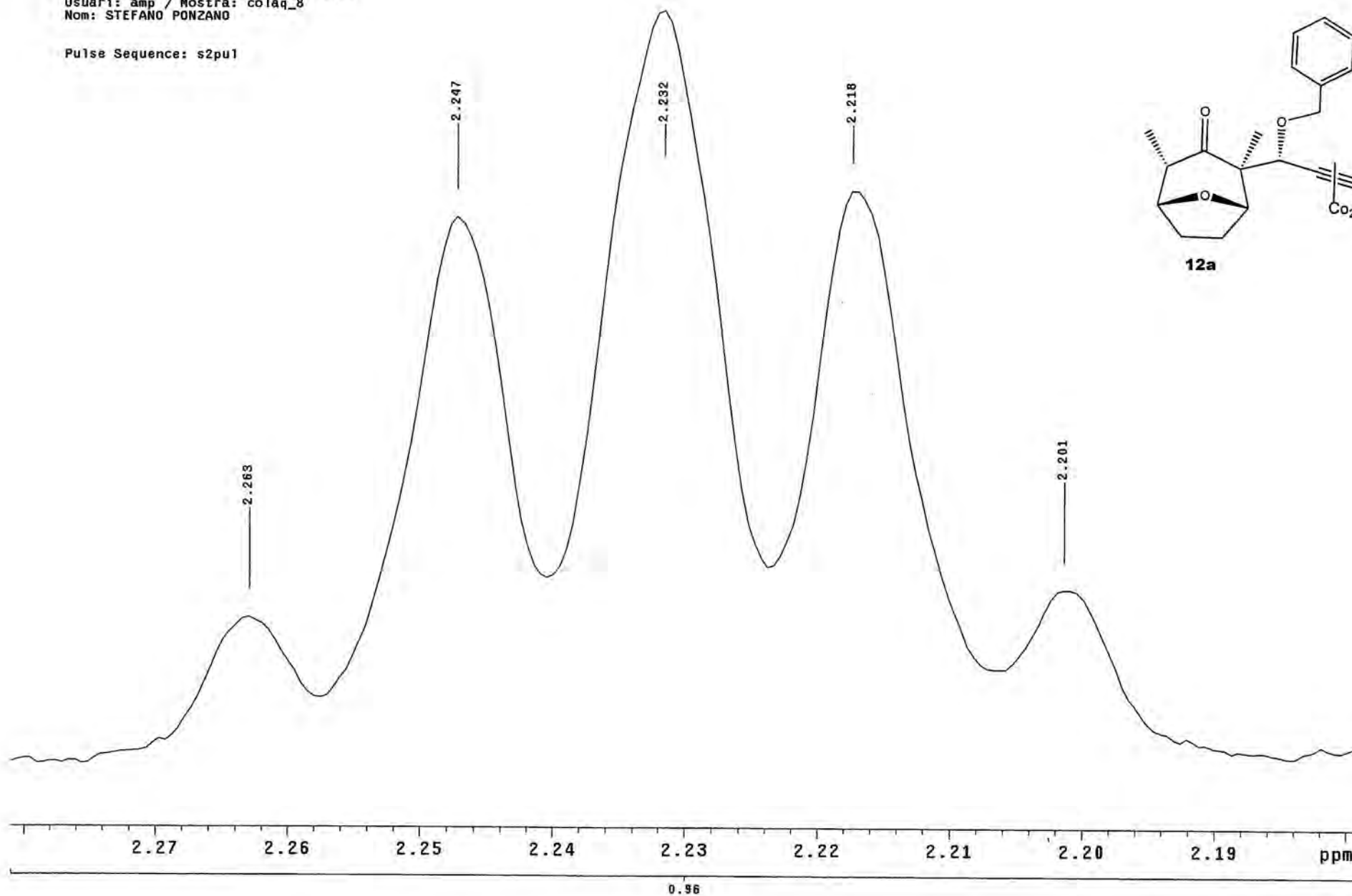
H1 / s2pu1 / Mercury-400. qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



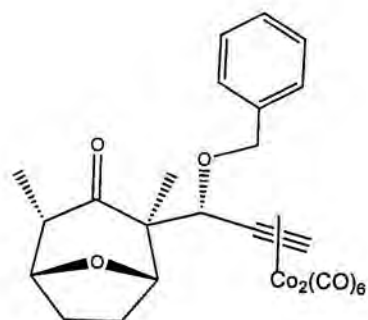
H1 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

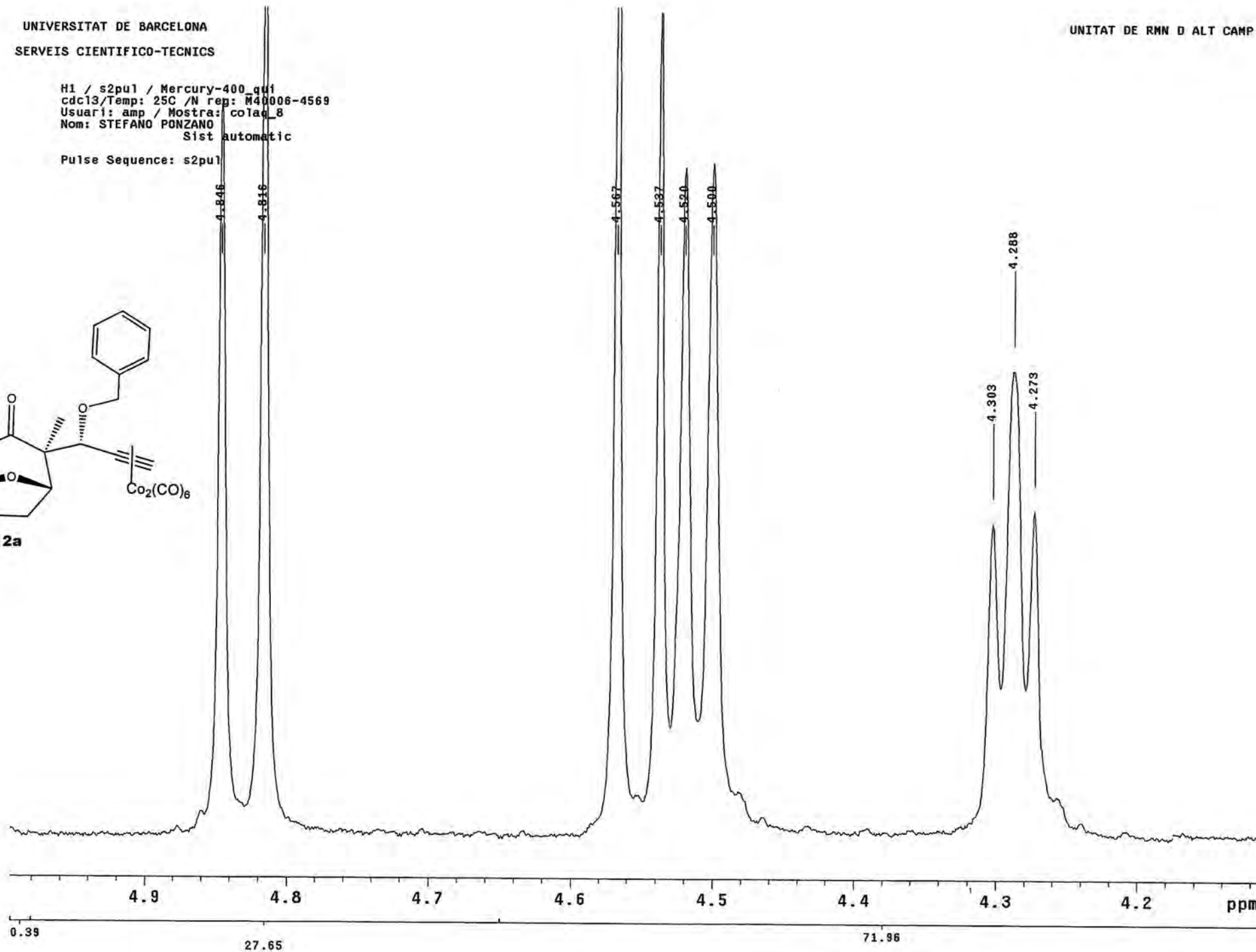


H1 / s2pu1 / Mercury-400.qul  
cdc13/Temp: 25C /N rep: M40006-4569  
Usuari: amp / Mostra: cola\_8  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pu1

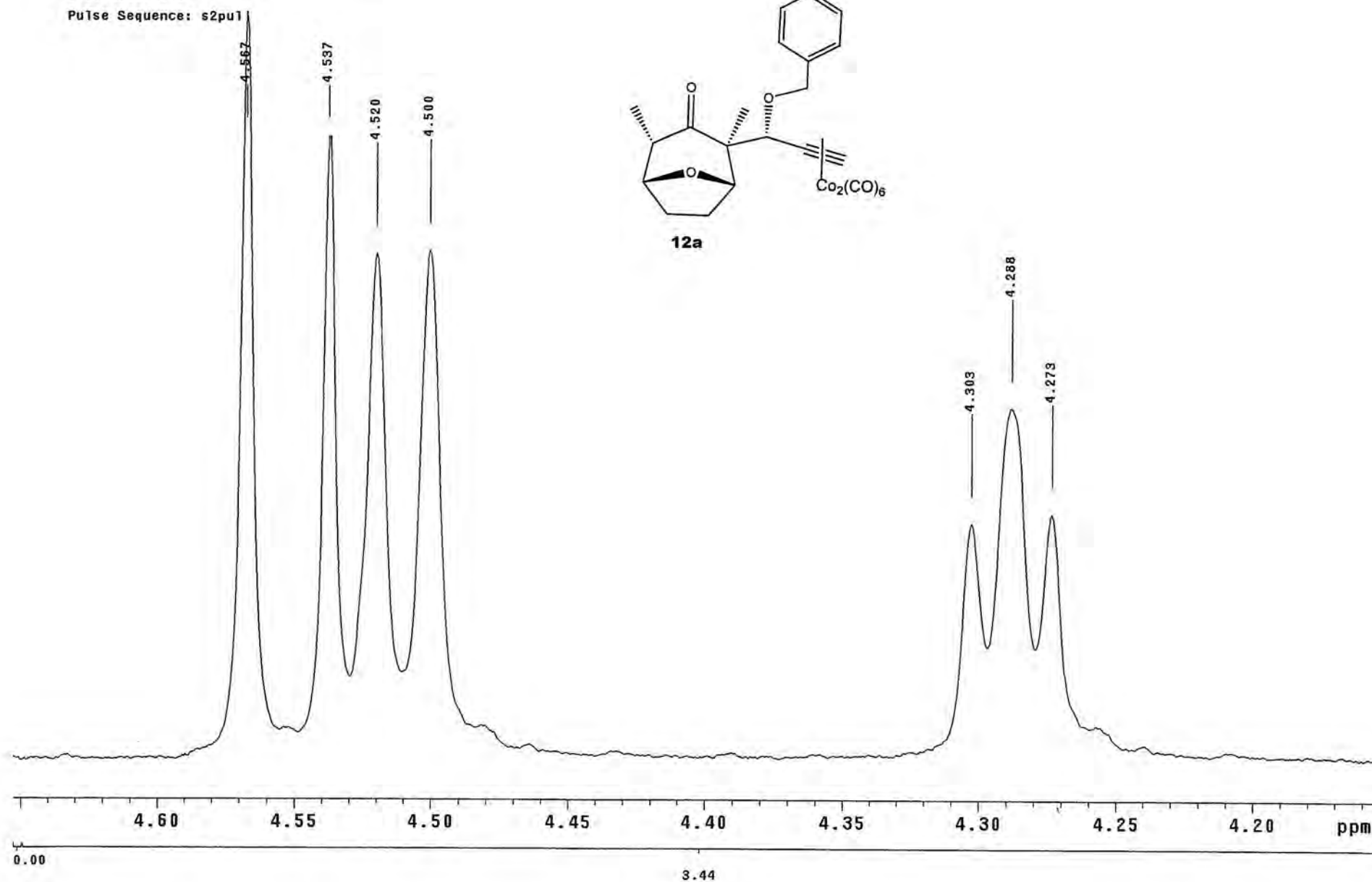
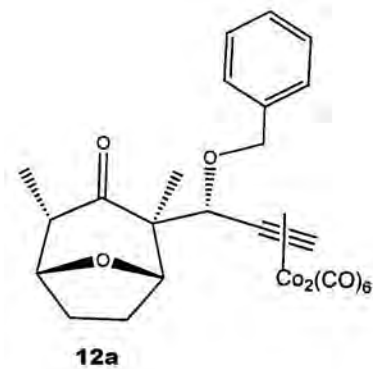


12a



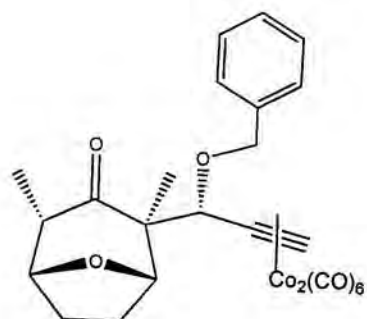
H1 / s2pu1 / Mercury-400\_gui  
cdcl3/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pu1

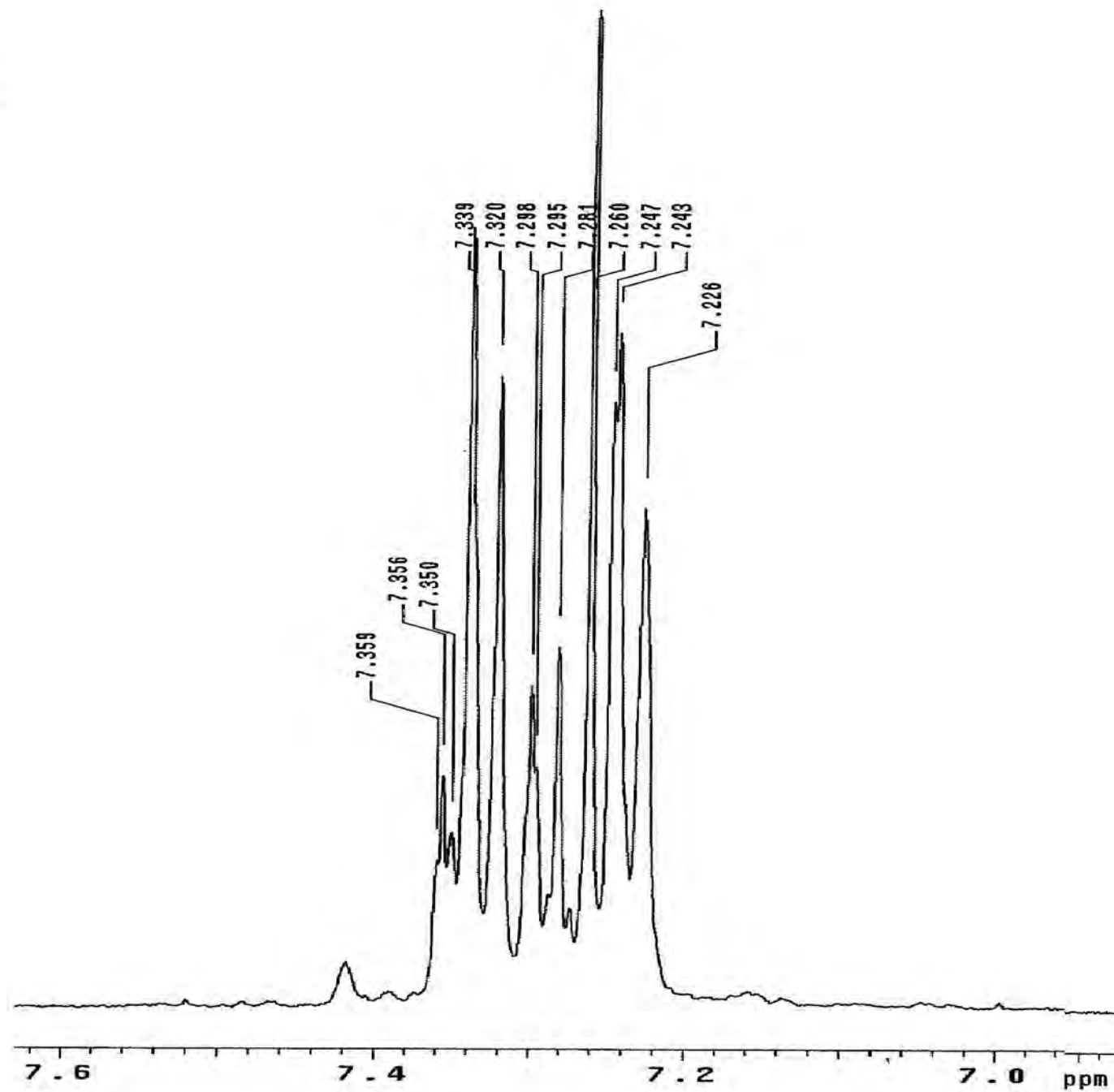


H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

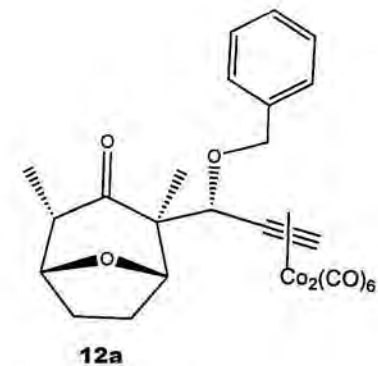
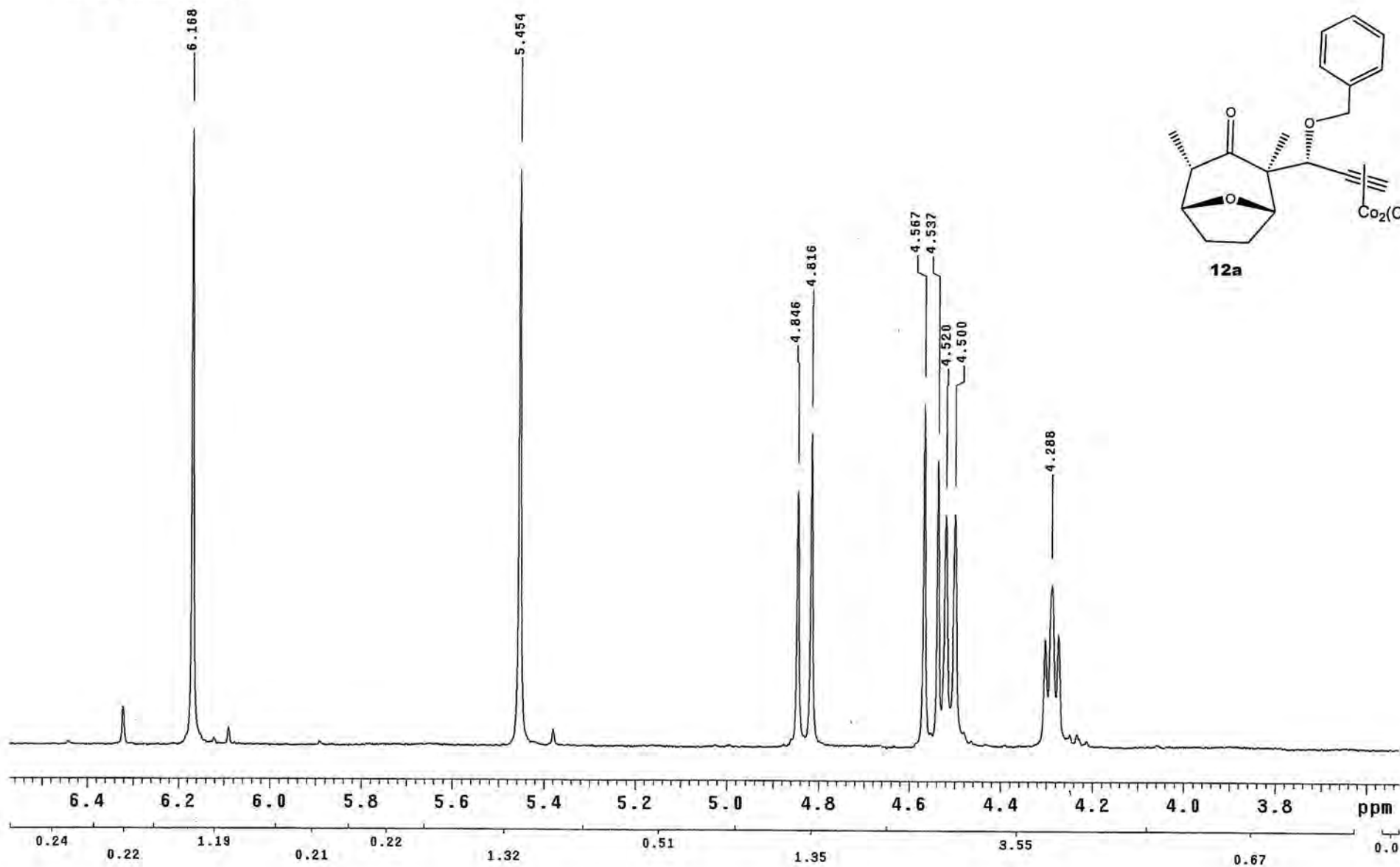
Pulse Sequence: s2pu1



12a



Pulse Sequence: s2pu1



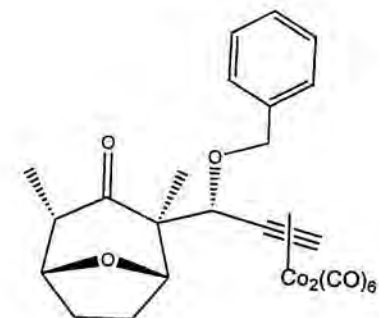
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

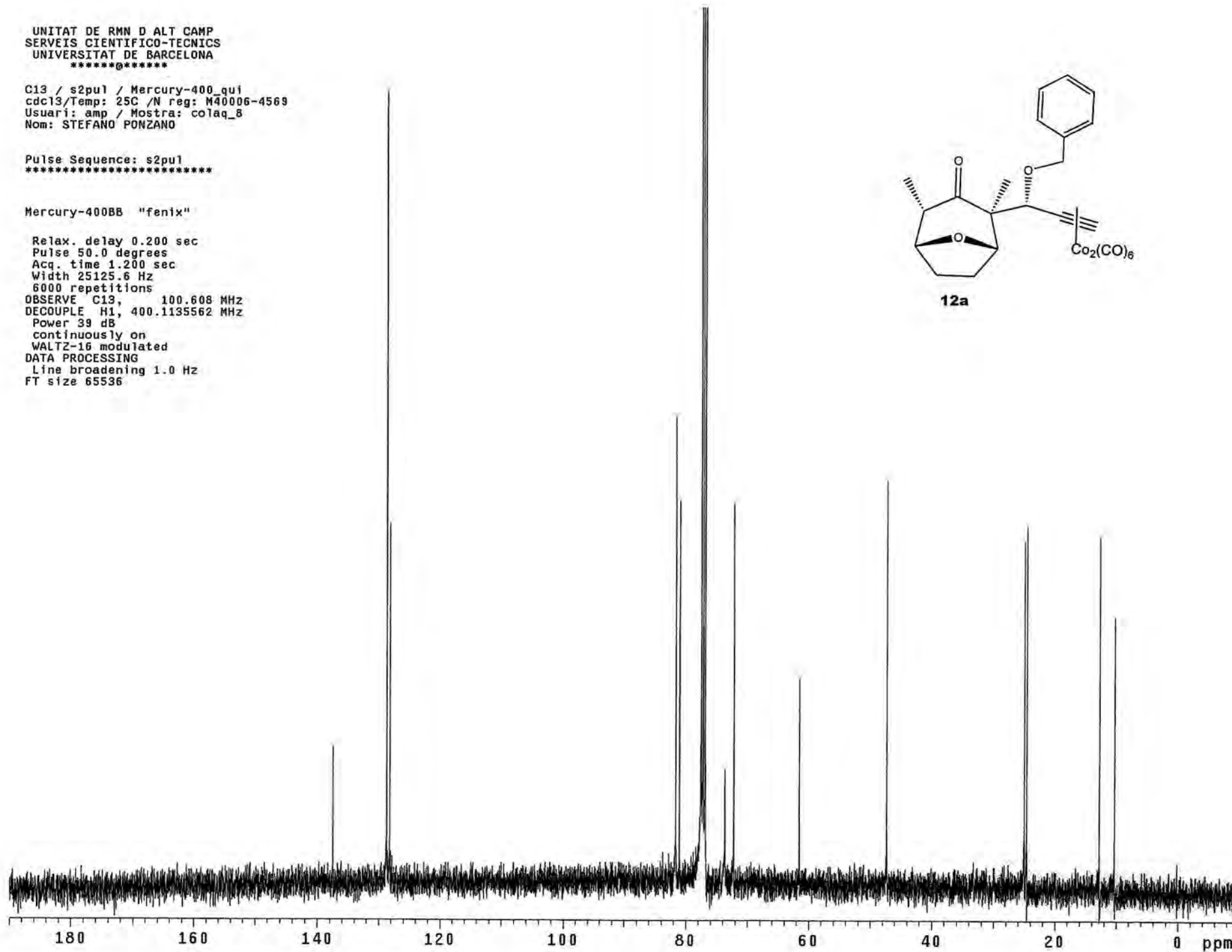
Pulse Sequence: s2pu1  
\*\*\*\*\*

Mercury-400BB "fenix"

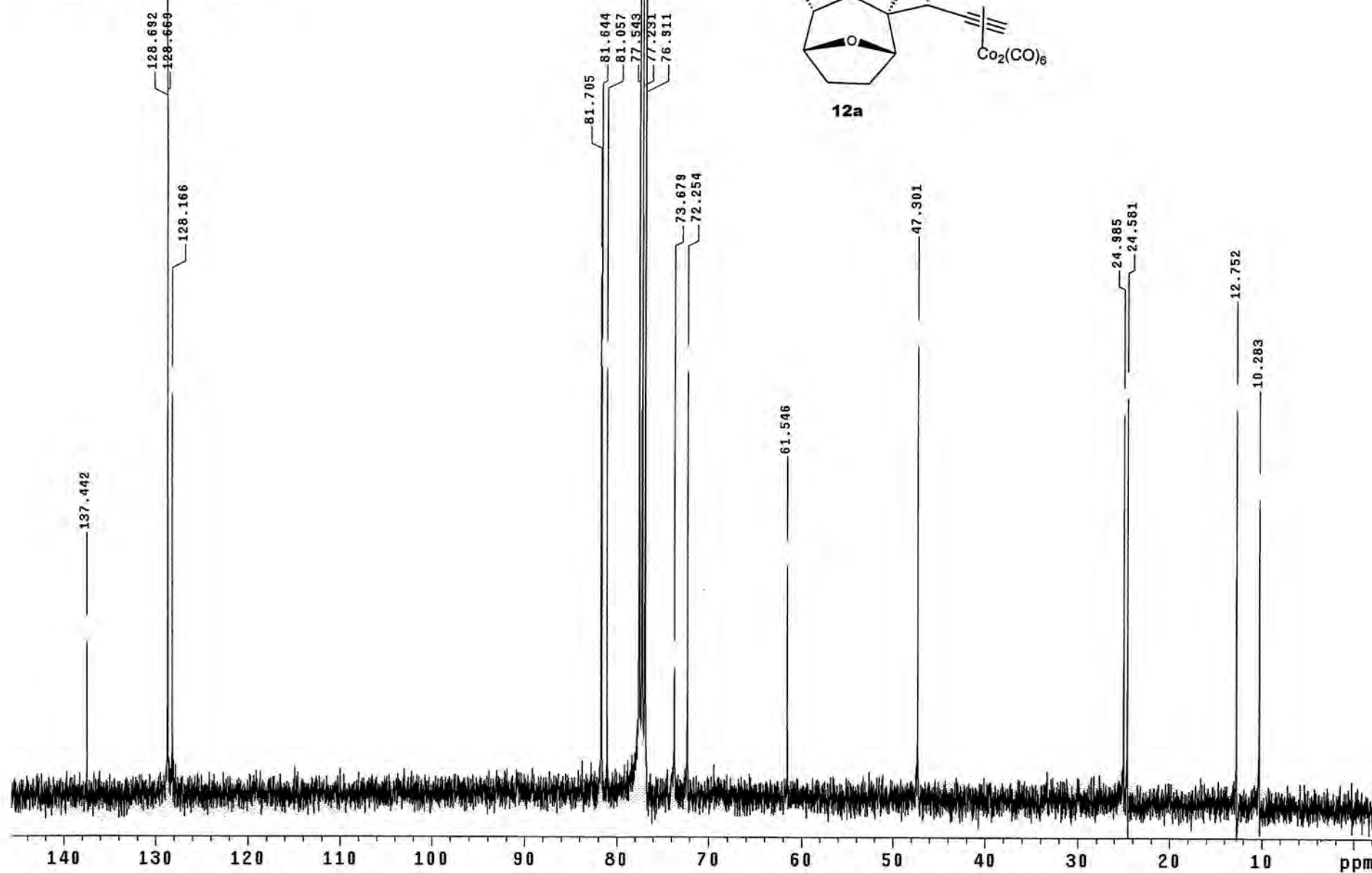
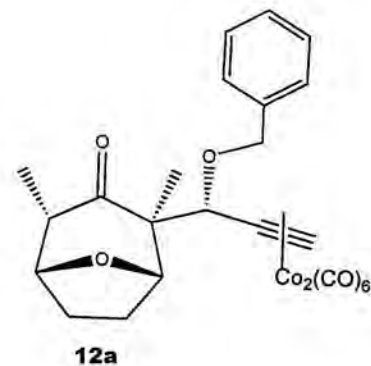
Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 25125.6 Hz  
6000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536



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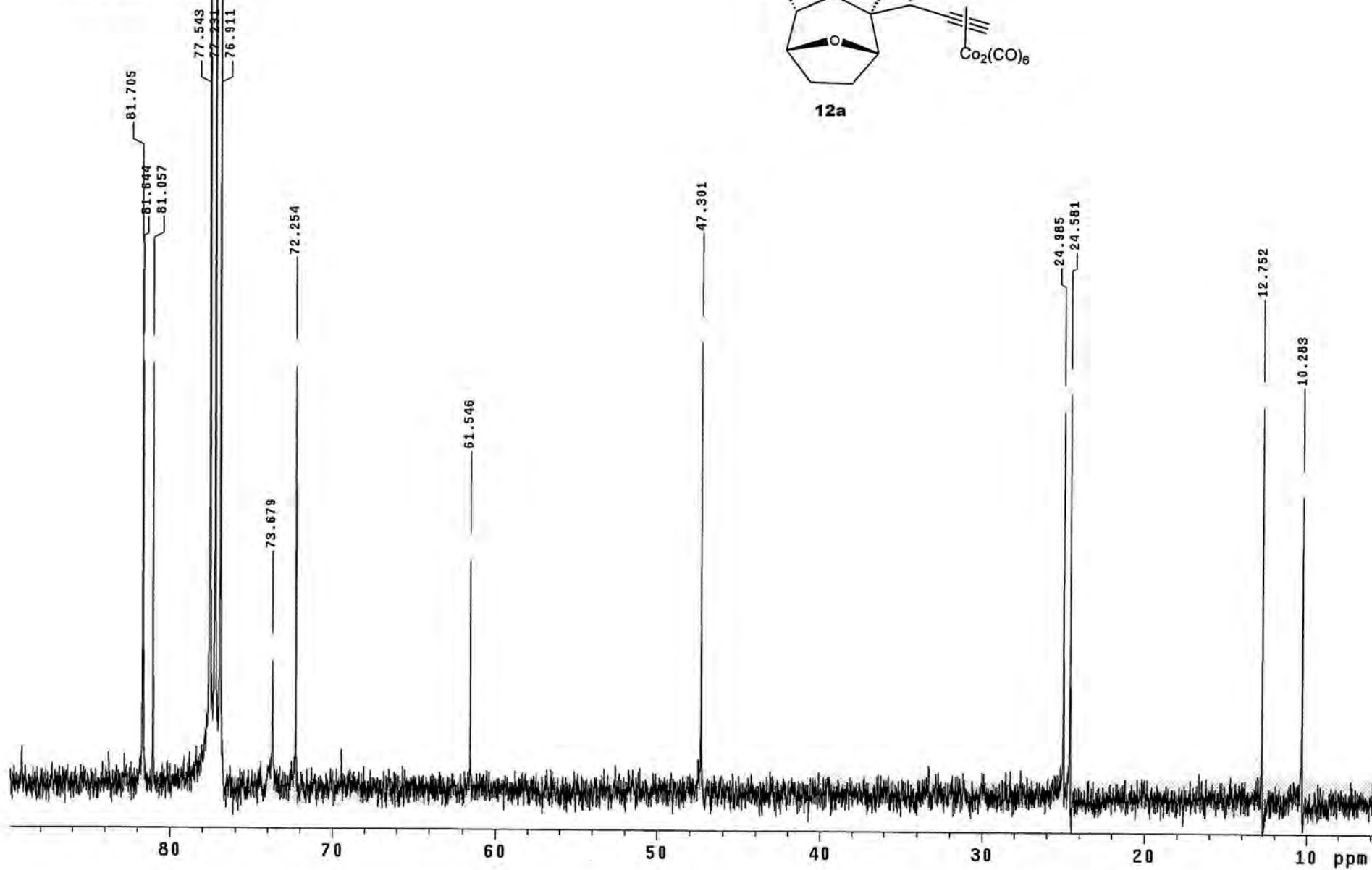
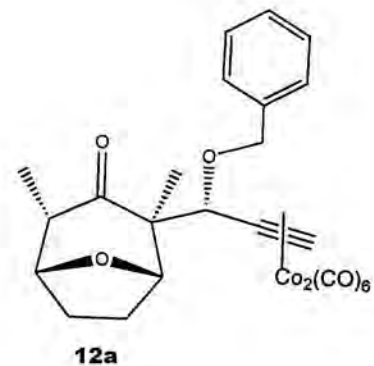


Pulse Sequence: s2pu1



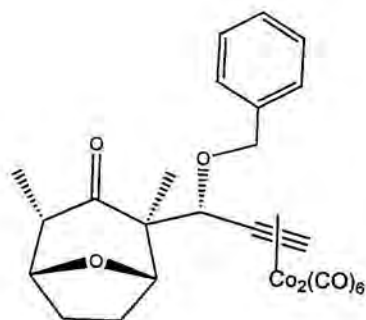
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Tmp: 25C / W reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pu1

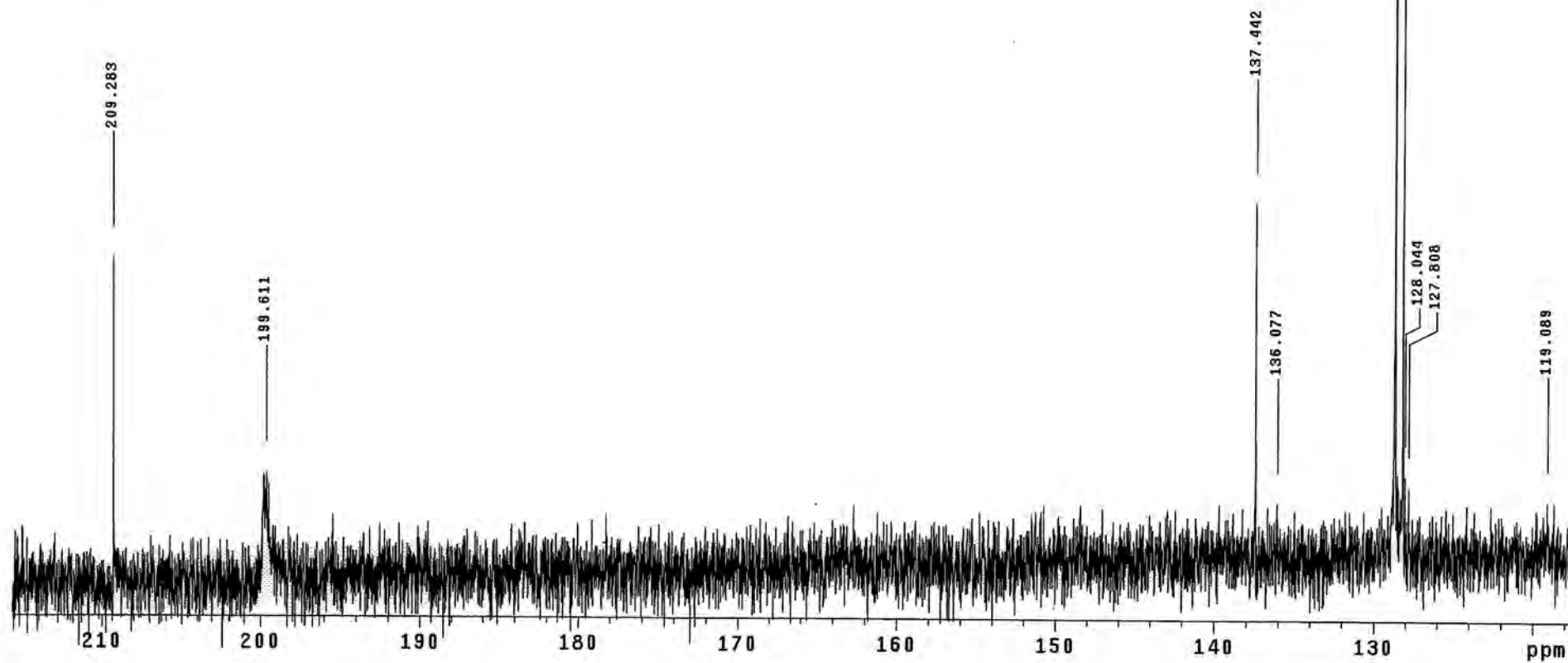


C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

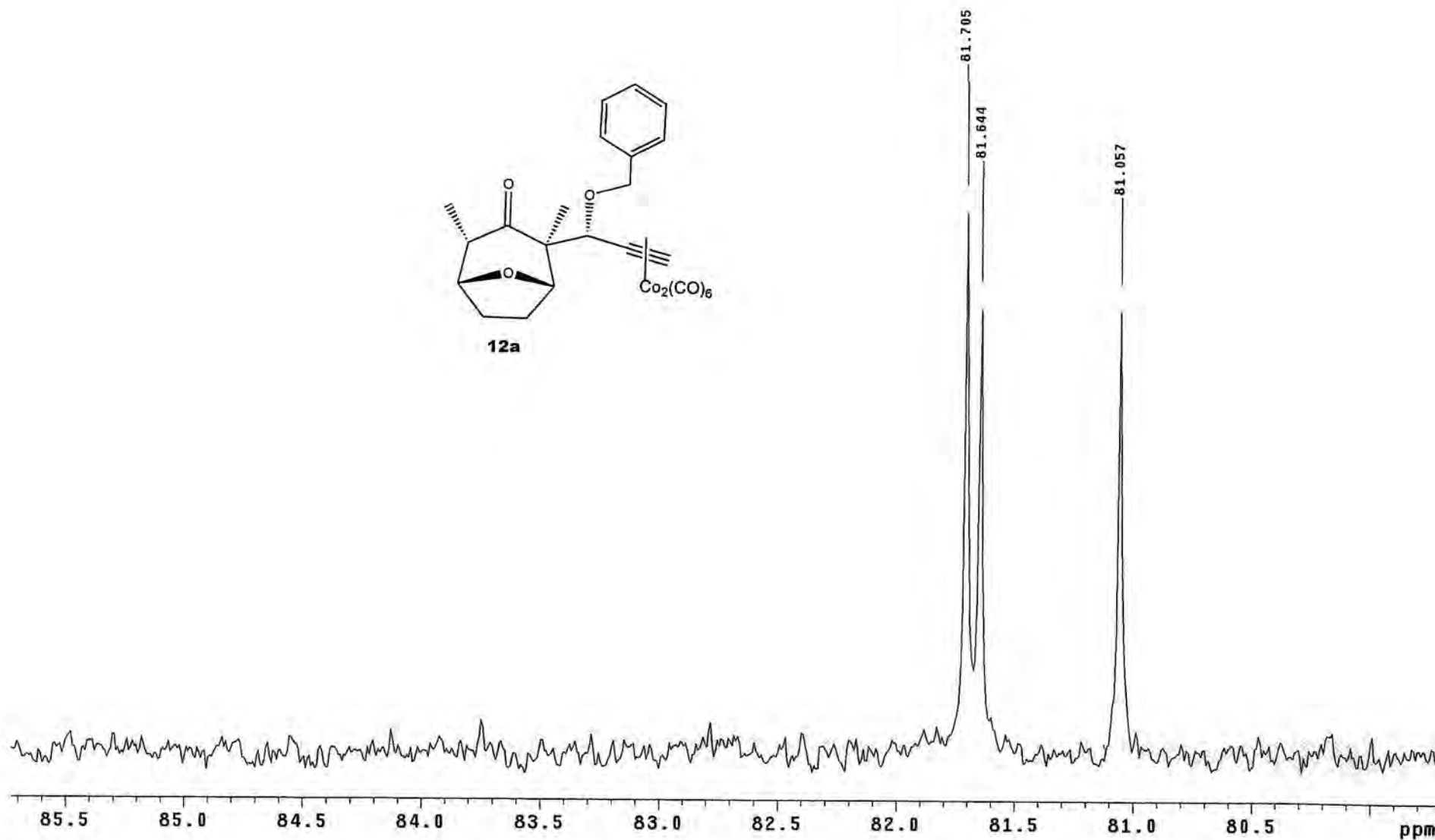
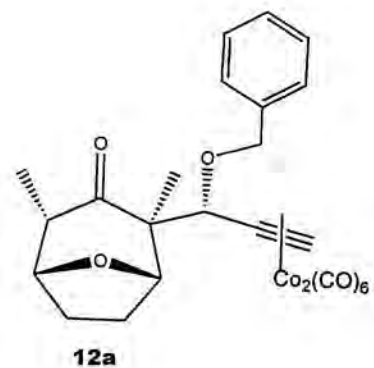


12a



C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

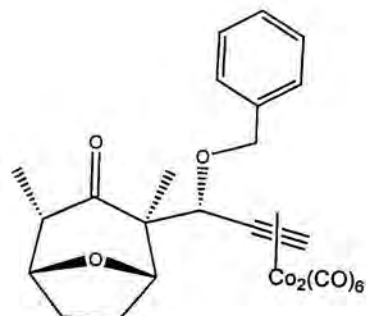
Pulse Sequence: s2pu1



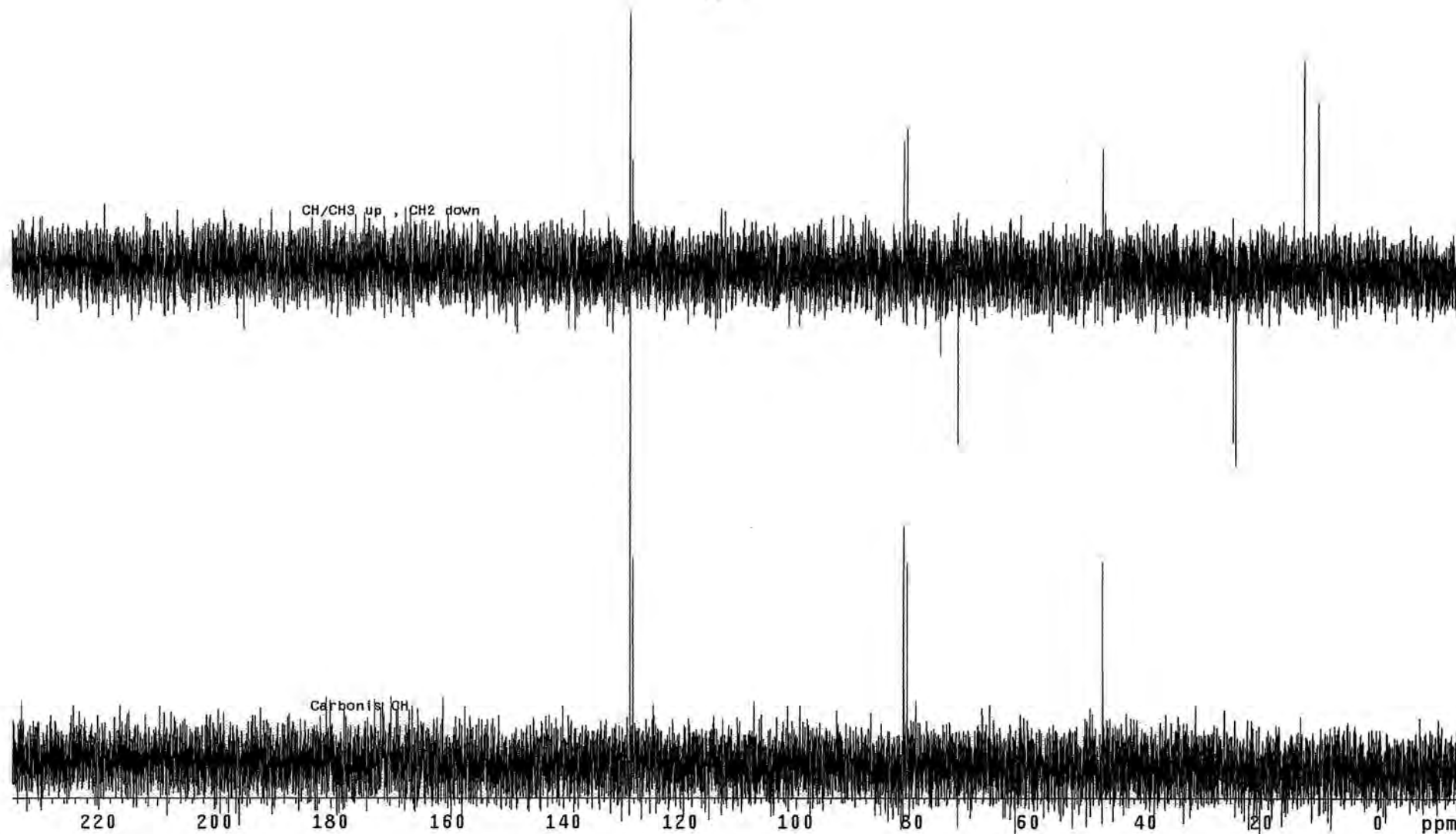
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / DEPT / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-45  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: DEPT  
\*\*\*\*\*



12a

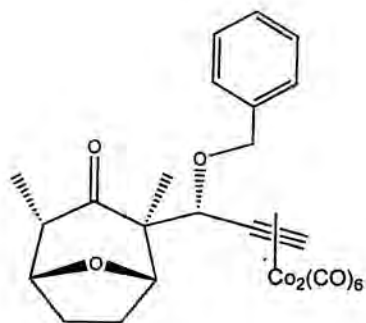


H1 / gHSQC / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*

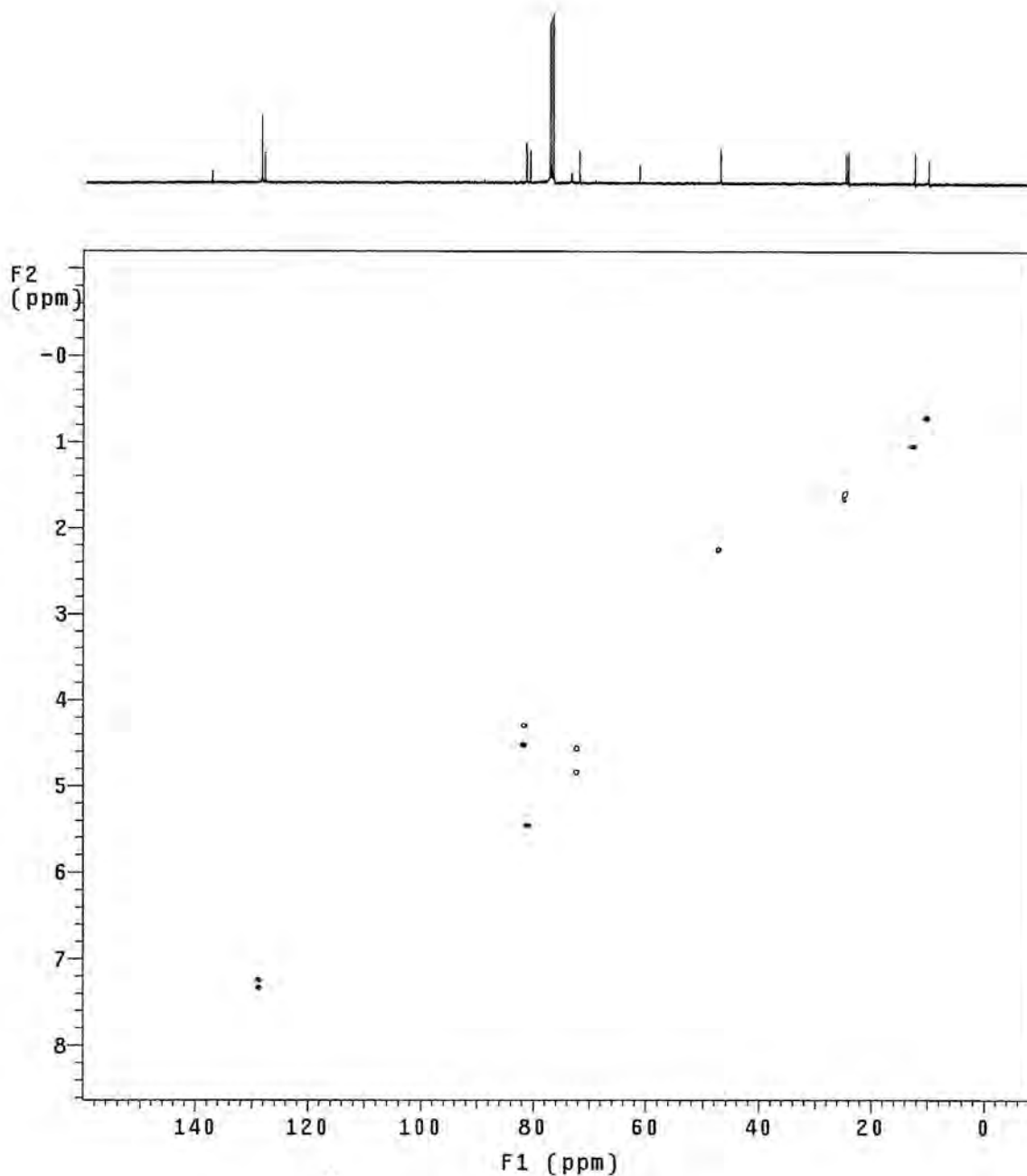
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.130 sec  
Width 3837.0 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048



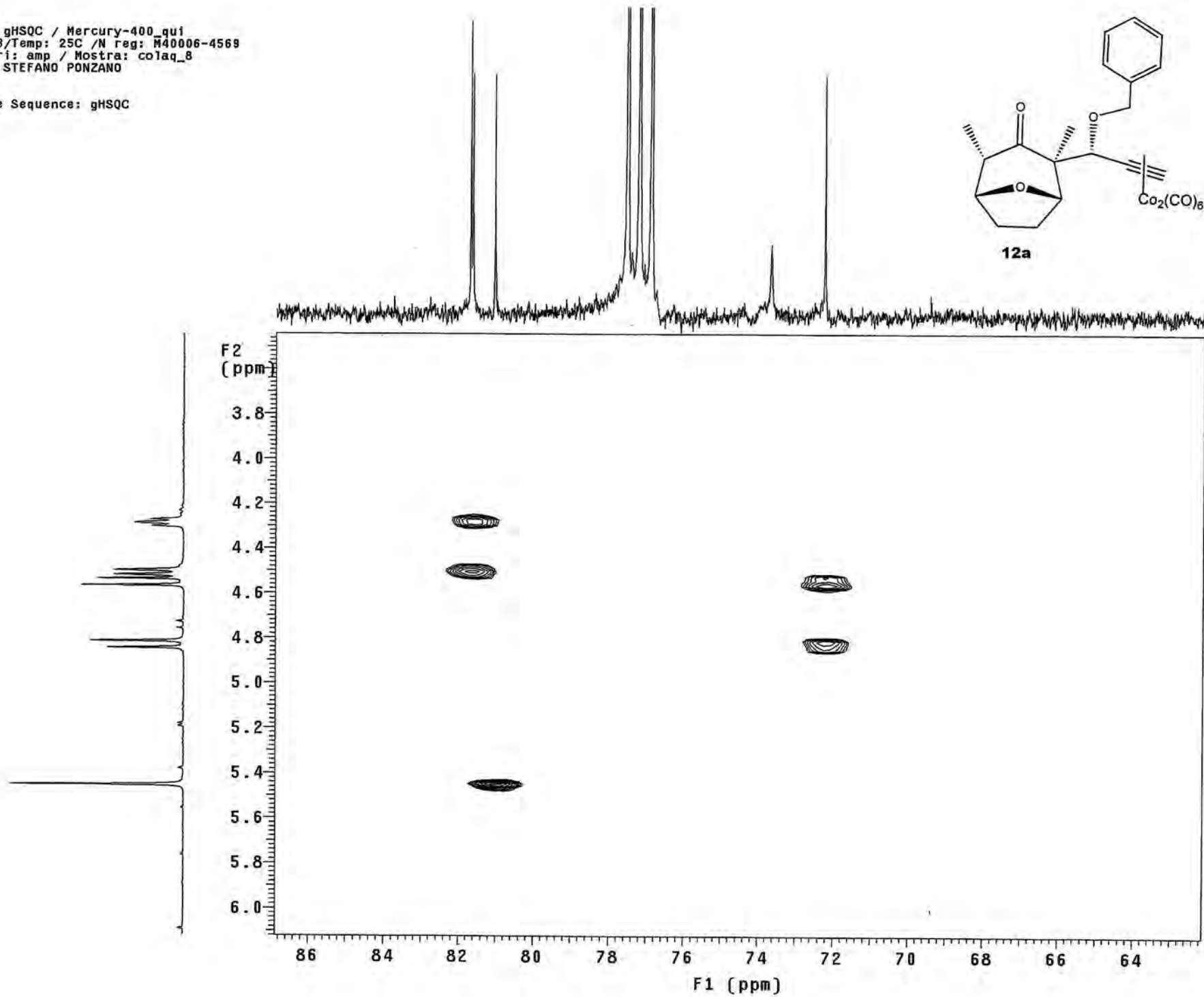
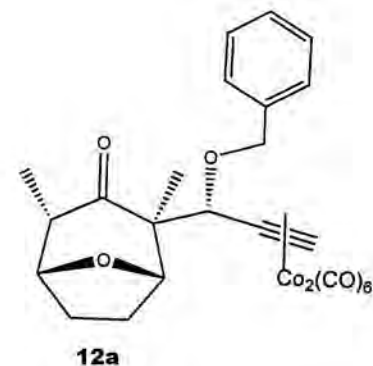
**12a**

REF: COL AQ-8  
Libreta nº 3  
(COLUMN AQ)



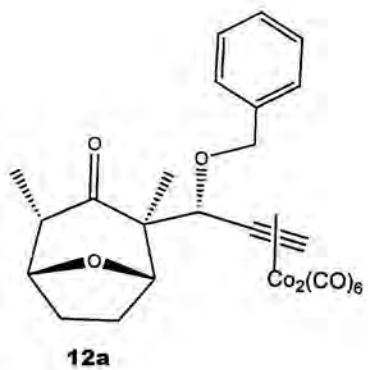
H1 / gHSQC / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC

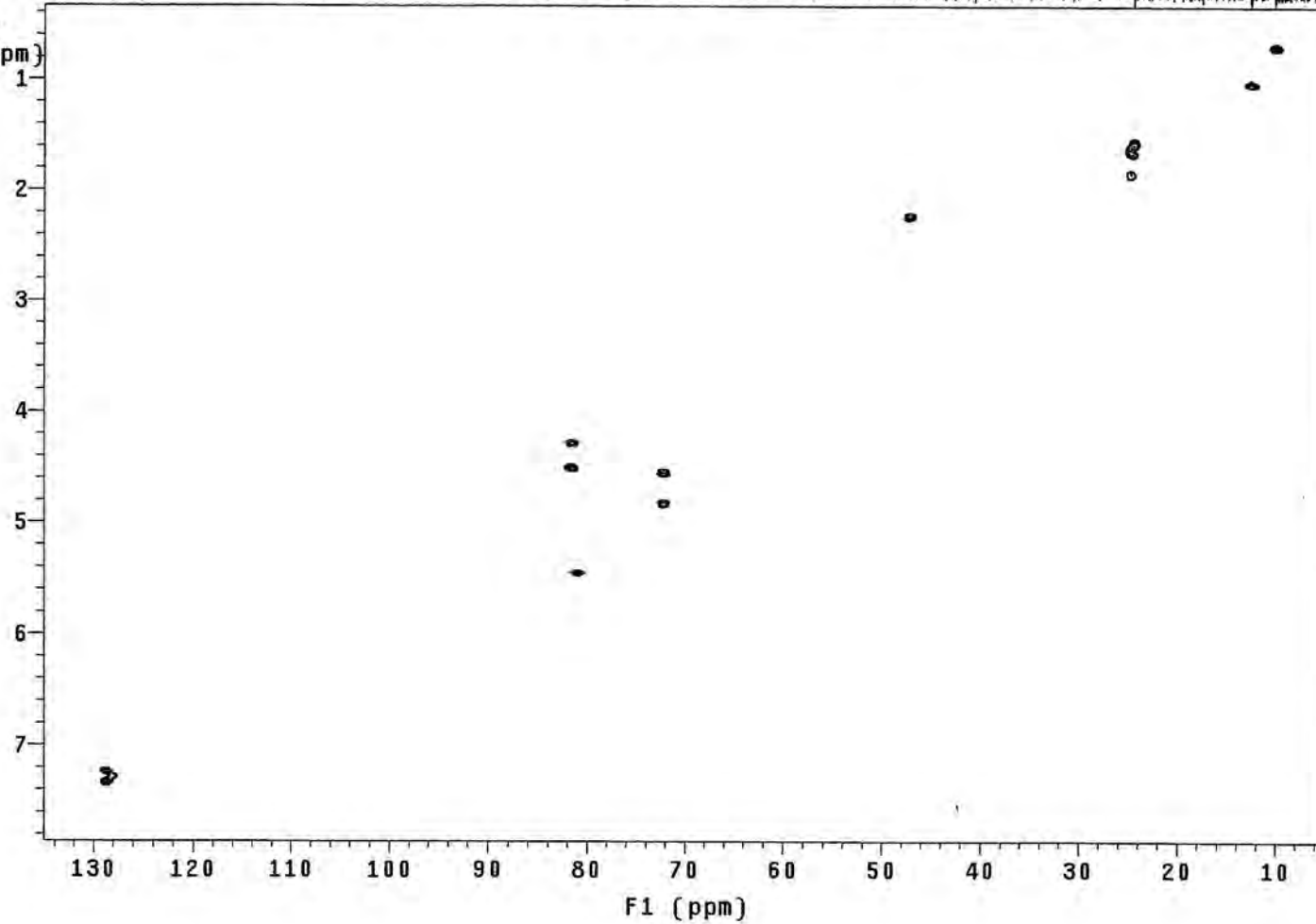


H1 / gHSQC / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-4569  
Usuari: amp / Mostra: colaq\_8  
Nom: STEFANO PONZANO

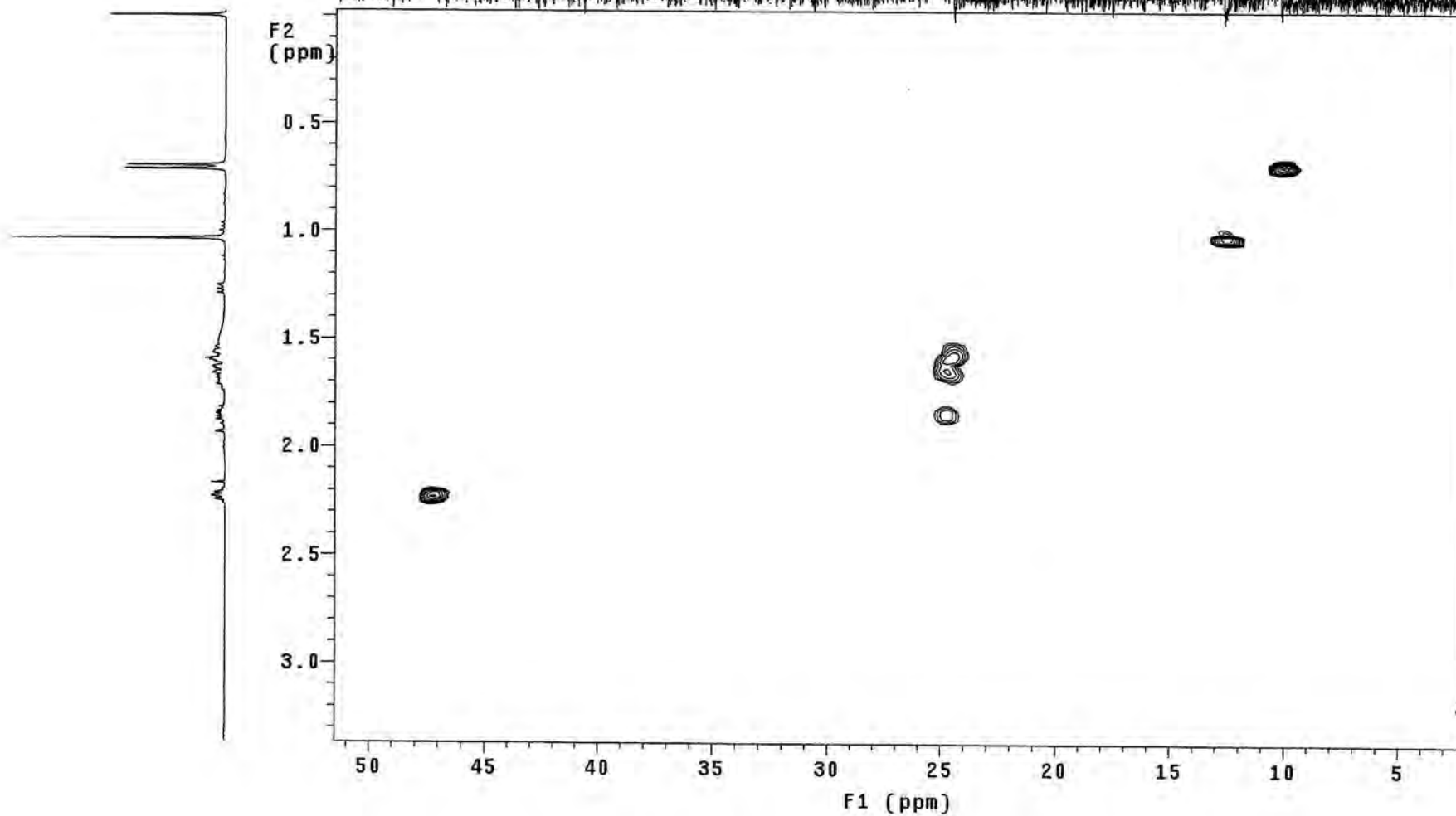
Pulse Sequence: gHSQC



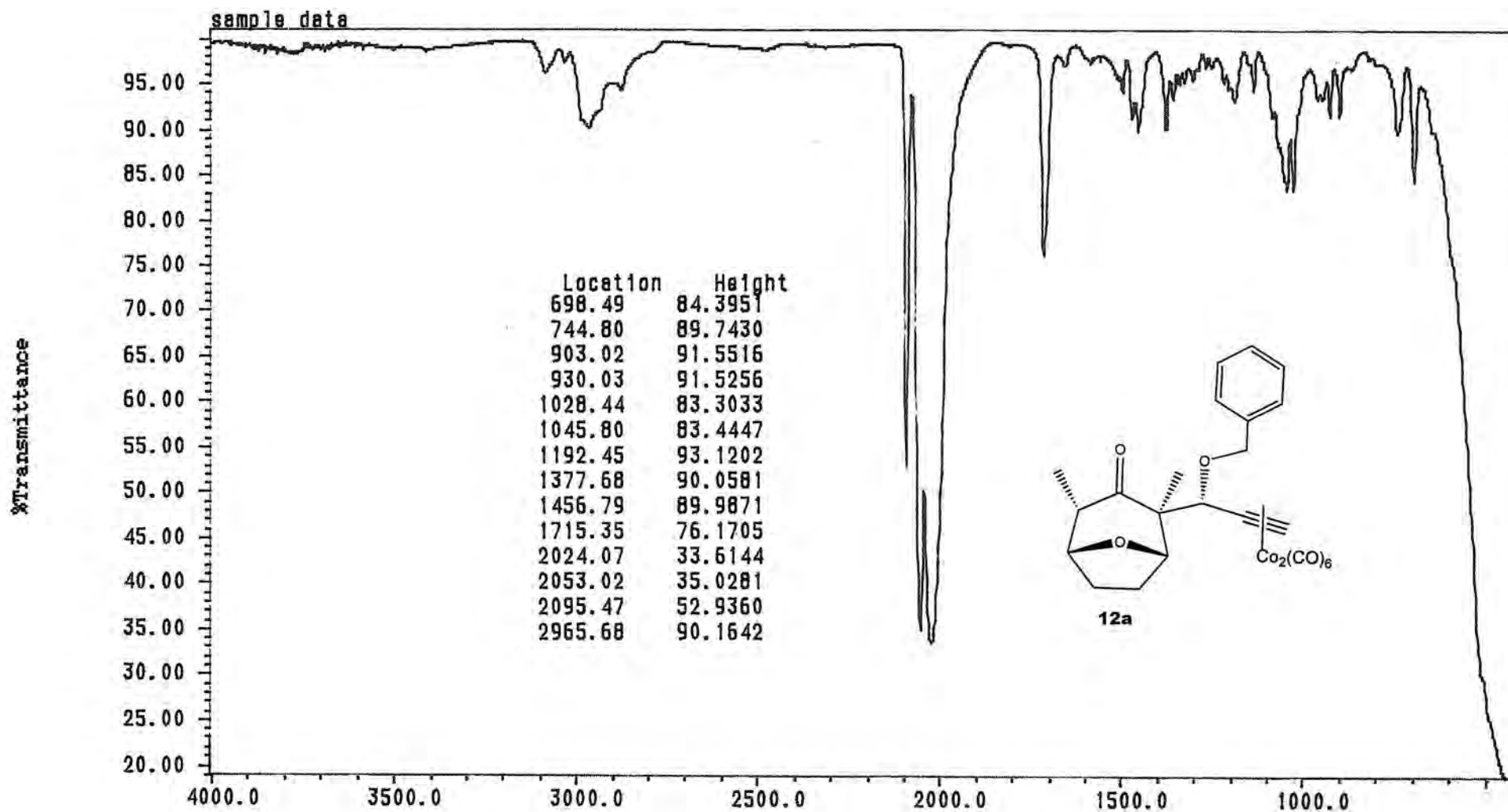
F2  
(ppm)

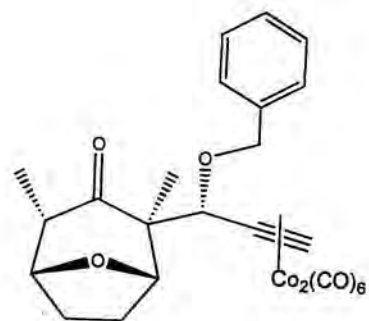
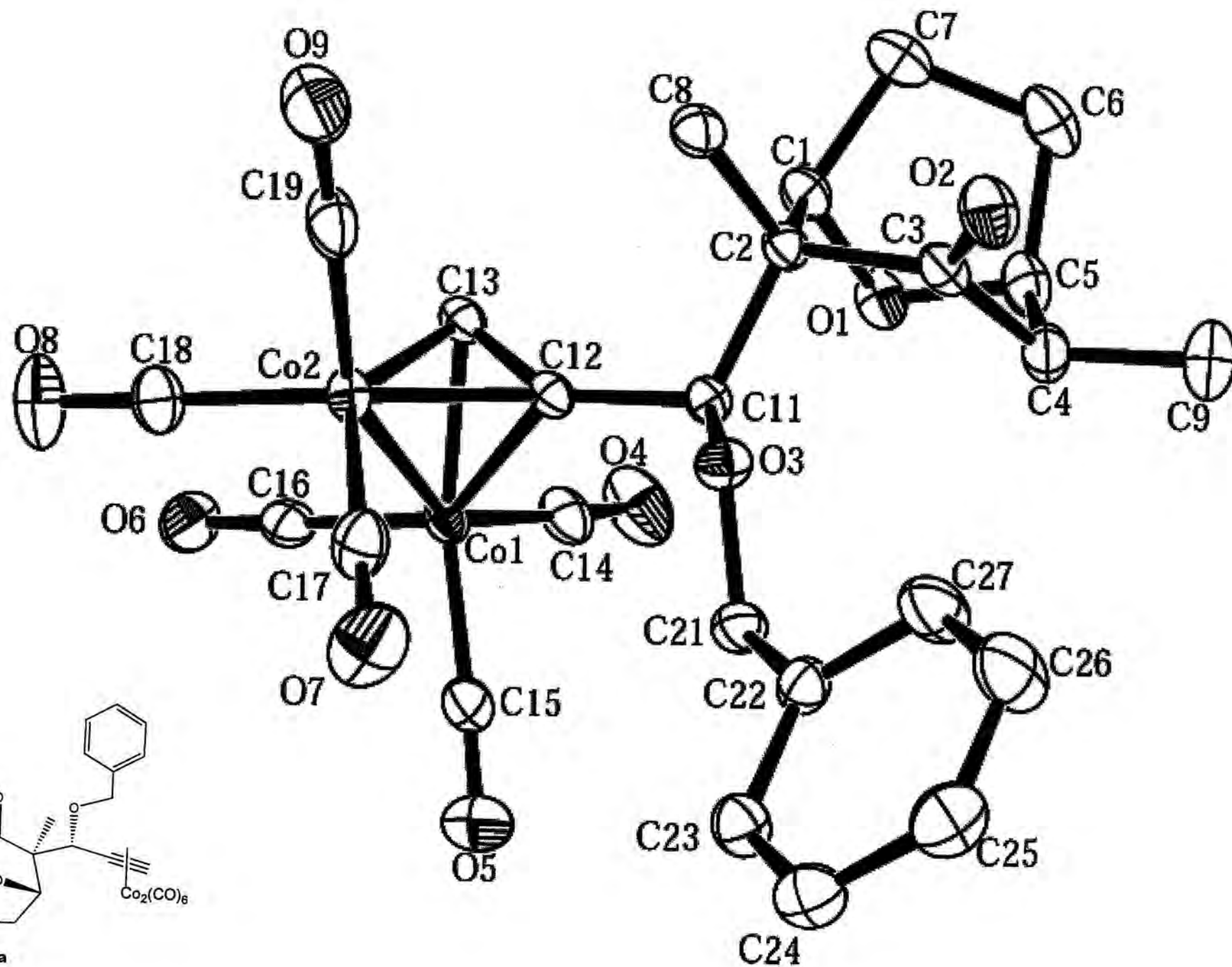


Pulse Sequence: gHSQC



colaq\_8  
sample data

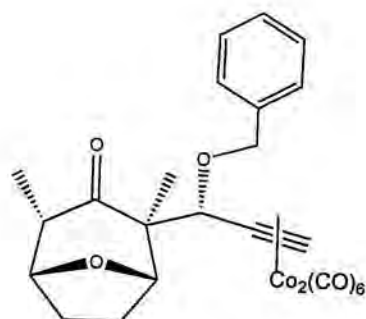




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Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for h06rdk15. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

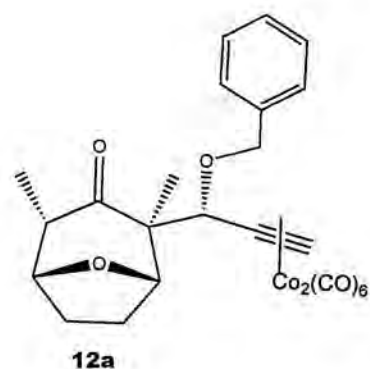
|       | x        | y       | z       | U(eq) |
|-------|----------|---------|---------|-------|
| Co(1) | 4772(1)  | 6593(1) | 3291(1) | 20(1) |
| Co(2) | 2858(1)  | 4881(1) | 3631(1) | 20(1) |
| O(1)  | 9184(1)  | 2893(1) | 2842(1) | 26(1) |
| O(2)  | 6901(1)  | -30(1)  | 1990(1) | 31(1) |
| O(3)  | 4709(1)  | 3272(1) | 1933(1) | 22(1) |
| O(4)  | 8134(1)  | 6822(2) | 3201(1) | 49(1) |
| O(5)  | 3447(2)  | 8378(2) | 1691(1) | 45(1) |
| O(6)  | 3666(2)  | 8640(1) | 4510(1) | 39(1) |
| O(7)  | 891(2)   | 5583(2) | 2095(1) | 47(1) |
| O(8)  | 893(2)   | 6653(2) | 4772(1) | 48(1) |
| O(9)  | 2217(2)  | 1956(1) | 4460(1) | 51(1) |
| C(1)  | 8242(2)  | 2012(2) | 3374(1) | 23(1) |
| C(2)  | 6746(2)  | 1878(1) | 2888(1) | 19(1) |
| C(3)  | 7365(2)  | 1080(2) | 2144(1) | 23(1) |
| C(4)  | 8681(2)  | 1707(2) | 1664(1) | 27(1) |
| C(5)  | 9933(2)  | 1854(2) | 2302(1) | 30(1) |
| C(6)  | 10525(2) | 364(2)  | 2897(1) | 38(1) |
| C(7)  | 9377(2)  | 467(2)  | 3628(1) | 32(1) |
| C(8)  | 5690(2)  | 967(2)  | 3420(1) | 24(1) |
| C(9)  | 9382(2)  | 786(2)  | 989(1)  | 42(1) |
| C(11) | 5808(2)  | 3499(1) | 2526(1) | 18(1) |
| C(12) | 5007(2)  | 4402(1) | 3172(1) | 18(1) |
| C(13) | 5094(2)  | 4587(2) | 3967(1) | 20(1) |
| C(14) | 6824(2)  | 6770(2) | 3213(1) | 30(1) |
| C(15) | 3981(2)  | 7691(2) | 2299(1) | 28(1) |
| C(16) | 4105(2)  | 7878(2) | 4029(1) | 26(1) |
| C(17) | 1627(2)  | 5288(2) | 2685(1) | 30(1) |
| C(18) | 1652(2)  | 5979(2) | 4330(1) | 29(1) |
| C(19) | 2453(2)  | 3073(2) | 4110(1) | 33(1) |
| C(21) | 4383(2)  | 4460(2) | 1235(1) | 23(1) |
| C(22) | 3627(2)  | 3881(2) | 555(1)  | 23(1) |
| C(23) | 2333(2)  | 4794(2) | 103(1)  | 26(1) |
| C(24) | 1689(2)  | 4301(2) | -560(1) | 30(1) |
| C(25) | 2324(2)  | 2894(2) | -774(1) | 33(1) |
| C(26) | 3603(2)  | 1979(2) | -323(1) | 40(1) |
| C(27) | 4247(2)  | 2464(2) | 340(1)  | 36(1) |



**12a**

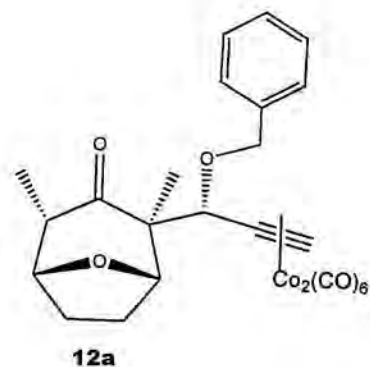
Bond lengths [Å] for h06rdk15.

|             |            |
|-------------|------------|
| Co(1)-C(14) | 1.7992(16) |
| Co(1)-C(16) | 1.8124(15) |
| Co(1)-C(15) | 1.8236(16) |
| Co(1)-C(13) | 1.9444(13) |
| Co(1)-C(12) | 1.9952(13) |
| Co(1)-Co(2) | 2.4731(3)  |
| Co(2)-C(19) | 1.7979(17) |
| Co(2)-C(18) | 1.8103(15) |
| Co(2)-C(17) | 1.8271(16) |
| Co(2)-C(13) | 1.9565(14) |
| Co(2)-C(12) | 1.9734(13) |
| O(1)-C(1)   | 1.4362(18) |
| O(1)-C(5)   | 1.4415(18) |
| O(2)-C(3)   | 1.2126(17) |
| O(3)-C(11)  | 1.4228(16) |
| O(3)-C(21)  | 1.4289(17) |
| O(4)-C(14)  | 1.1333(19) |
| O(5)-C(15)  | 1.133(2)   |
| O(6)-C(16)  | 1.1376(19) |
| O(7)-C(17)  | 1.124(2)   |
| O(8)-C(18)  | 1.1328(19) |
| O(9)-C(19)  | 1.135(2)   |
| C(1)-C(7)   | 1.5402(19) |
| C(1)-C(2)   | 1.5511(19) |
| C(2)-C(8)   | 1.5180(19) |
| C(2)-C(3)   | 1.5438(19) |
| C(2)-C(11)  | 1.5590(18) |
| C(3)-C(4)   | 1.516(2)   |
| C(4)-C(9)   | 1.530(2)   |
| C(4)-C(5)   | 1.542(2)   |
| C(5)-C(6)   | 1.535(2)   |
| C(6)-C(7)   | 1.538(3)   |
| C(11)-C(12) | 1.5093(18) |
| C(12)-C(13) | 1.338(2)   |
| C(21)-C(22) | 1.502(2)   |
| C(22)-C(27) | 1.386(2)   |
| C(22)-C(23) | 1.388(2)   |
| C(23)-C(24) | 1.391(2)   |
| C(24)-C(25) | 1.380(2)   |
| C(25)-C(26) | 1.380(2)   |
| C(26)-C(27) | 1.387(2)   |



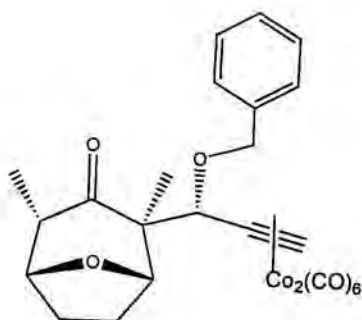
## Bond angles [deg] for h06rdk15.

|                   |            |                   |            |
|-------------------|------------|-------------------|------------|
| C(14)-Co(1)-C(16) | 99.74(7)   | C(11)-C(12)-Co(2) | 137.29(9)  |
| C(14)-Co(1)-C(15) | 100.57(7)  | C(13)-C(12)-Co(1) | 68.12(8)   |
| C(16)-Co(1)-C(15) | 103.52(7)  | C(11)-C(12)-Co(1) | 131.92(9)  |
| C(14)-Co(1)-C(13) | 97.84(6)   | Co(2)-C(12)-Co(1) | 77.09(5)   |
| C(16)-Co(1)-C(13) | 103.51(6)  | C(12)-C(13)-Co(1) | 72.21(8)   |
| C(15)-Co(1)-C(13) | 143.97(6)  | C(12)-C(13)-Co(2) | 70.78(8)   |
| C(14)-Co(1)-C(12) | 99.46(6)   | Co(1)-C(13)-Co(2) | 78.69(5)   |
| C(16)-Co(1)-C(12) | 140.54(6)  | O(4)-C(14)-Co(1)  | 176.25(16) |
| C(15)-Co(1)-C(12) | 106.37(6)  | O(5)-C(15)-Co(1)  | 178.04(15) |
| C(13)-Co(1)-C(12) | 39.68(6)   | O(6)-C(16)-Co(1)  | 177.36(13) |
| C(14)-Co(1)-Co(2) | 147.08(5)  | O(7)-C(17)-Co(2)  | 177.60(16) |
| C(16)-Co(1)-Co(2) | 97.97(5)   | O(8)-C(18)-Co(2)  | 179.28(16) |
| C(15)-Co(1)-Co(2) | 101.95(5)  | O(9)-C(19)-Co(2)  | 175.56(17) |
| C(13)-Co(1)-Co(2) | 50.87(4)   | O(3)-C(21)-C(22)  | 108.82(11) |
| C(12)-Co(1)-Co(2) | 51.06(4)   | C(27)-C(22)-C(23) | 118.71(14) |
| C(19)-Co(2)-C(18) | 95.30(7)   | C(27)-C(22)-C(21) | 120.96(13) |
| C(19)-Co(2)-C(17) | 102.16(8)  | C(23)-C(22)-C(21) | 120.26(13) |
| C(18)-Co(2)-C(17) | 103.04(7)  | C(22)-C(23)-C(24) | 120.53(14) |
| C(19)-Co(2)-C(13) | 99.11(7)   | C(25)-C(24)-C(23) | 120.32(14) |
| C(18)-Co(2)-C(13) | 108.70(6)  | C(24)-C(25)-C(26) | 119.32(15) |
| C(17)-Co(2)-C(13) | 139.64(6)  | C(25)-C(26)-C(27) | 120.54(15) |
| C(19)-Co(2)-C(12) | 104.99(6)  | C(22)-C(27)-C(26) | 120.56(15) |
| C(18)-Co(2)-C(12) | 144.21(6)  |                   |            |
| C(17)-Co(2)-C(12) | 101.11(6)  |                   |            |
| C(13)-Co(2)-C(12) | 39.80(6)   |                   |            |
| C(19)-Co(2)-Co(1) | 149.46(5)  |                   |            |
| C(18)-Co(2)-Co(1) | 97.18(5)   |                   |            |
| C(17)-Co(2)-Co(1) | 101.98(5)  |                   |            |
| C(13)-Co(2)-Co(1) | 50.44(4)   |                   |            |
| C(12)-Co(2)-Co(1) | 51.85(4)   |                   |            |
| C(1)-O(1)-C(5)    | 103.57(11) |                   |            |
| C(11)-O(3)-C(21)  | 114.61(10) |                   |            |
| O(1)-C(1)-C(7)    | 103.70(12) |                   |            |
| O(1)-C(1)-C(2)    | 109.46(11) |                   |            |
| C(7)-C(1)-C(2)    | 112.85(11) |                   |            |
| C(8)-C(2)-C(3)    | 110.38(11) |                   |            |
| C(8)-C(2)-C(1)    | 112.09(12) |                   |            |
| C(3)-C(2)-C(1)    | 106.15(11) |                   |            |
| C(8)-C(2)-C(11)   | 111.17(11) |                   |            |
| C(3)-C(2)-C(11)   | 107.05(11) |                   |            |
| C(1)-C(2)-C(11)   | 109.76(10) |                   |            |
| O(2)-C(3)-C(4)    | 122.66(13) |                   |            |
| O(2)-C(3)-C(2)    | 122.06(13) |                   |            |
| C(4)-C(3)-C(2)    | 115.11(11) |                   |            |
| C(3)-C(4)-C(9)    | 113.60(13) |                   |            |
| C(3)-C(4)-C(5)    | 107.56(13) |                   |            |
| C(9)-C(4)-C(5)    | 113.34(13) |                   |            |
| O(1)-C(5)-C(6)    | 103.68(13) |                   |            |
| O(1)-C(5)-C(4)    | 108.53(11) |                   |            |
| C(6)-C(5)-C(4)    | 112.96(13) |                   |            |
| C(5)-C(6)-C(7)    | 103.71(12) |                   |            |
| C(6)-C(7)-C(1)    | 103.47(13) |                   |            |
| O(3)-C(11)-C(12)  | 112.48(10) |                   |            |
| O(3)-C(11)-C(2)   | 105.78(10) |                   |            |
| C(12)-C(11)-C(2)  | 114.19(11) |                   |            |
| C(13)-C(12)-C(11) | 143.46(12) |                   |            |
| C(13)-C(12)-Co(2) | 69.42(8)   |                   |            |



Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for h06rdk15.  
The anisotropic displacement factor exponent takes the form:  
 $-2 \pi^2 [ h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12} ]$

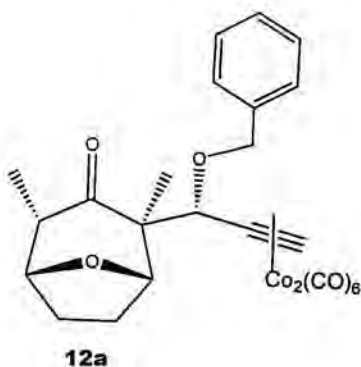
|       | U11   | U22   | U33   | U23    | U13    | U12    |
|-------|-------|-------|-------|--------|--------|--------|
| Co(1) | 20(1) | 15(1) | 24(1) | -5(1)  | 1(1)   | -3(1)  |
| Co(2) | 18(1) | 21(1) | 23(1) | -7(1)  | 3(1)   | -4(1)  |
| O(1)  | 21(1) | 24(1) | 37(1) | -9(1)  | 3(1)   | -7(1)  |
| O(2)  | 35(1) | 26(1) | 37(1) | -15(1) | 2(1)   | -7(1)  |
| O(3)  | 25(1) | 22(1) | 19(1) | -4(1)  | -3(1)  | -6(1)  |
| O(4)  | 25(1) | 41(1) | 83(1) | -15(1) | 3(1)   | -9(1)  |
| O(5)  | 51(1) | 41(1) | 35(1) | 4(1)   | -4(1)  | 3(1)   |
| O(6)  | 60(1) | 24(1) | 34(1) | -10(1) | 11(1)  | -7(1)  |
| O(7)  | 34(1) | 76(1) | 34(1) | -7(1)  | -7(1)  | -15(1) |
| O(8)  | 35(1) | 60(1) | 56(1) | -34(1) | 16(1)  | -7(1)  |
| O(9)  | 41(1) | 29(1) | 82(1) | -2(1)  | 26(1)  | -10(1) |
| C(1)  | 22(1) | 20(1) | 27(1) | -5(1)  | -2(1)  | -4(1)  |
| C(2)  | 20(1) | 16(1) | 21(1) | -4(1)  | 1(1)   | -3(1)  |
| C(3)  | 22(1) | 19(1) | 26(1) | -6(1)  | -2(1)  | 1(1)   |
| C(4)  | 27(1) | 26(1) | 29(1) | -7(1)  | 7(1)   | -3(1)  |
| C(5)  | 22(1) | 30(1) | 40(1) | -12(1) | 7(1)   | -4(1)  |
| C(6)  | 26(1) | 34(1) | 50(1) | -14(1) | -5(1)  | 7(1)   |
| C(7)  | 28(1) | 24(1) | 40(1) | -3(1)  | -8(1)  | 1(1)   |
| C(8)  | 26(1) | 17(1) | 30(1) | -3(1)  | 3(1)   | -6(1)  |
| C(9)  | 41(1) | 48(1) | 39(1) | -18(1) | 14(1)  | -3(1)  |
| C(11) | 18(1) | 17(1) | 20(1) | -4(1)  | 0(1)   | -4(1)  |
| C(12) | 16(1) | 15(1) | 23(1) | -3(1)  | 0(1)   | -3(1)  |
| C(13) | 19(1) | 16(1) | 24(1) | -3(1)  | 0(1)   | -2(1)  |
| C(14) | 30(1) | 20(1) | 41(1) | -8(1)  | 2(1)   | -4(1)  |
| C(15) | 29(1) | 22(1) | 31(1) | -4(1)  | 4(1)   | -1(1)  |
| C(16) | 33(1) | 17(1) | 28(1) | -1(1)  | 2(1)   | -6(1)  |
| C(17) | 22(1) | 40(1) | 31(1) | -9(1)  | 2(1)   | -9(1)  |
| C(18) | 22(1) | 33(1) | 34(1) | -11(1) | 2(1)   | -6(1)  |
| C(19) | 24(1) | 29(1) | 47(1) | -11(1) | 12(1)  | -6(1)  |
| C(21) | 27(1) | 22(1) | 20(1) | -3(1)  | -2(1)  | -3(1)  |
| C(22) | 23(1) | 27(1) | 20(1) | -5(1)  | 1(1)   | -4(1)  |
| C(23) | 23(1) | 27(1) | 28(1) | -5(1)  | 0(1)   | -3(1)  |
| C(24) | 25(1) | 35(1) | 29(1) | -2(1)  | -5(1)  | -8(1)  |
| C(25) | 37(1) | 38(1) | 27(1) | -9(1)  | -5(1)  | -12(1) |
| C(26) | 48(1) | 33(1) | 38(1) | -17(1) | -10(1) | 3(1)   |
| C(27) | 40(1) | 32(1) | 32(1) | -10(1) | -11(1) | 5(1)   |



12a

Hydrogen coordinates (  $\times 10^4$  ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for h06rdk15.

|        | x     | y    | z     | U(eq) |
|--------|-------|------|-------|-------|
| H(1)   | 7921  | 2503 | 3876  | 28    |
| H(4)   | 8210  | 2760 | 1383  | 33    |
| H(5)   | 10847 | 2232 | 2012  | 36    |
| H(6A)  | 11634 | 295  | 3083  | 45    |
| H(6B)  | 10469 | -532 | 2628  | 45    |
| H(7A)  | 9956  | 447  | 4152  | 38    |
| H(7B)  | 8790  | -381 | 3697  | 38    |
| H(8A)  | 5309  | 1484 | 3891  | 36    |
| H(8B)  | 6300  | -54  | 3627  | 36    |
| H(8C)  | 4779  | 882  | 3086  | 36    |
| H(9A)  | 10225 | 1248 | 705   | 63    |
| H(9B)  | 8544  | 784  | 586   | 63    |
| H(9C)  | 9828  | -264 | 1243  | 63    |
| H(11)  | 6574  | 4081 | 2223  | 22    |
| H(13)  | 5759  | 4056 | 4465  | 24    |
| H(21A) | 5383  | 4774 | 1038  | 28    |
| H(21B) | 3656  | 5357 | 1395  | 28    |
| H(23)  | 1884  | 5762 | 247   | 31    |
| H(24)  | 808   | 4937 | -868  | 36    |
| H(25)  | 1885  | 2557 | -1227 | 39    |
| H(26)  | 4046  | 1009 | -467  | 48    |
| H(27)  | 5121  | 1820 | 649   | 43    |



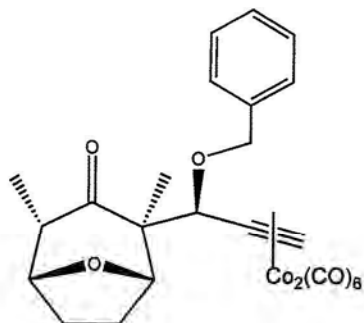
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*

Mercury-400BB "fenix"

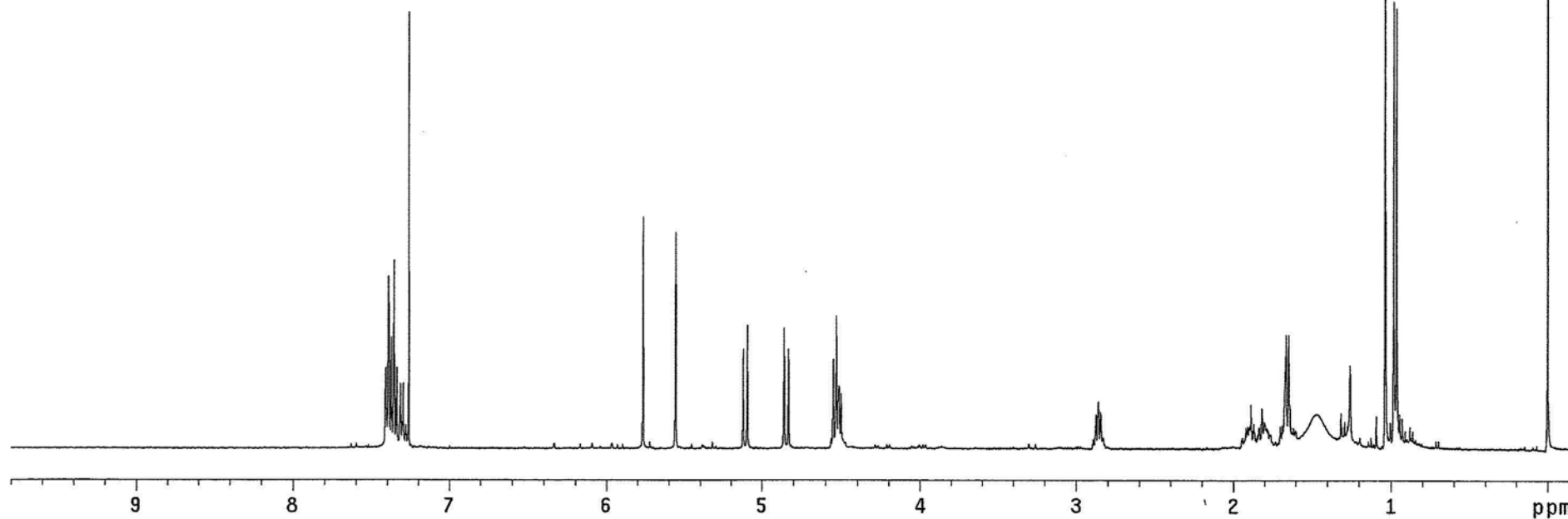
Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 5307.9 Hz  
8 repetitions  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536



**12b**

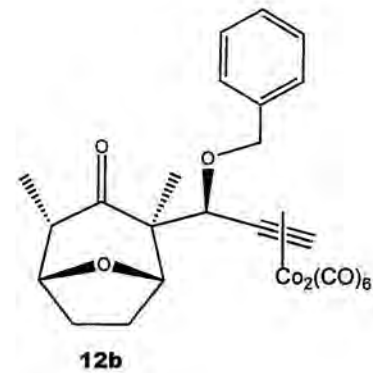
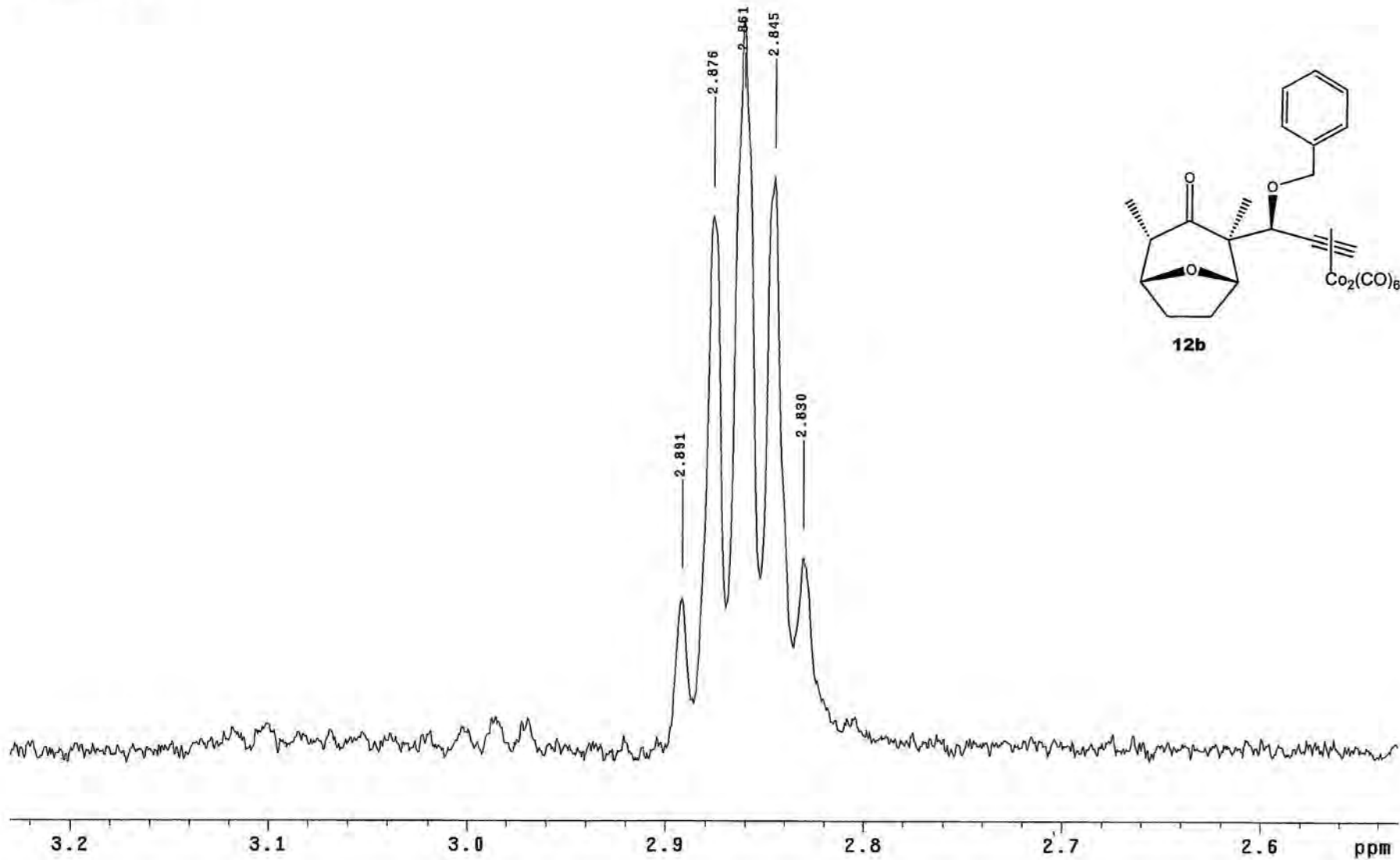
REF: COL AP-3  
Libreta nº 3  
(COLUMNNA AP)

| INDEX | FREQUENCY | PPM   | HEIGHT |
|-------|-----------|-------|--------|
| 1     | 2957.484  | 7.392 | 27.2   |
| 2     | 2943.229  | 7.356 | 29.7   |
| 3     | 2905.163  | 7.261 | 69.2   |
| 4     | 2306.313  | 5.764 | 36.6   |
| 5     | 2305.665  | 5.763 | 34.6   |
| 6     | 2223.053  | 5.556 | 34.2   |
| 7     | 414.514   | 1.036 | 151.2  |
| 8     | 392.971   | 0.982 | 70.9   |
| 9     | 386.329   | 0.966 | 69.8   |
| 10    | 0.000     | 0.000 | 119.4  |



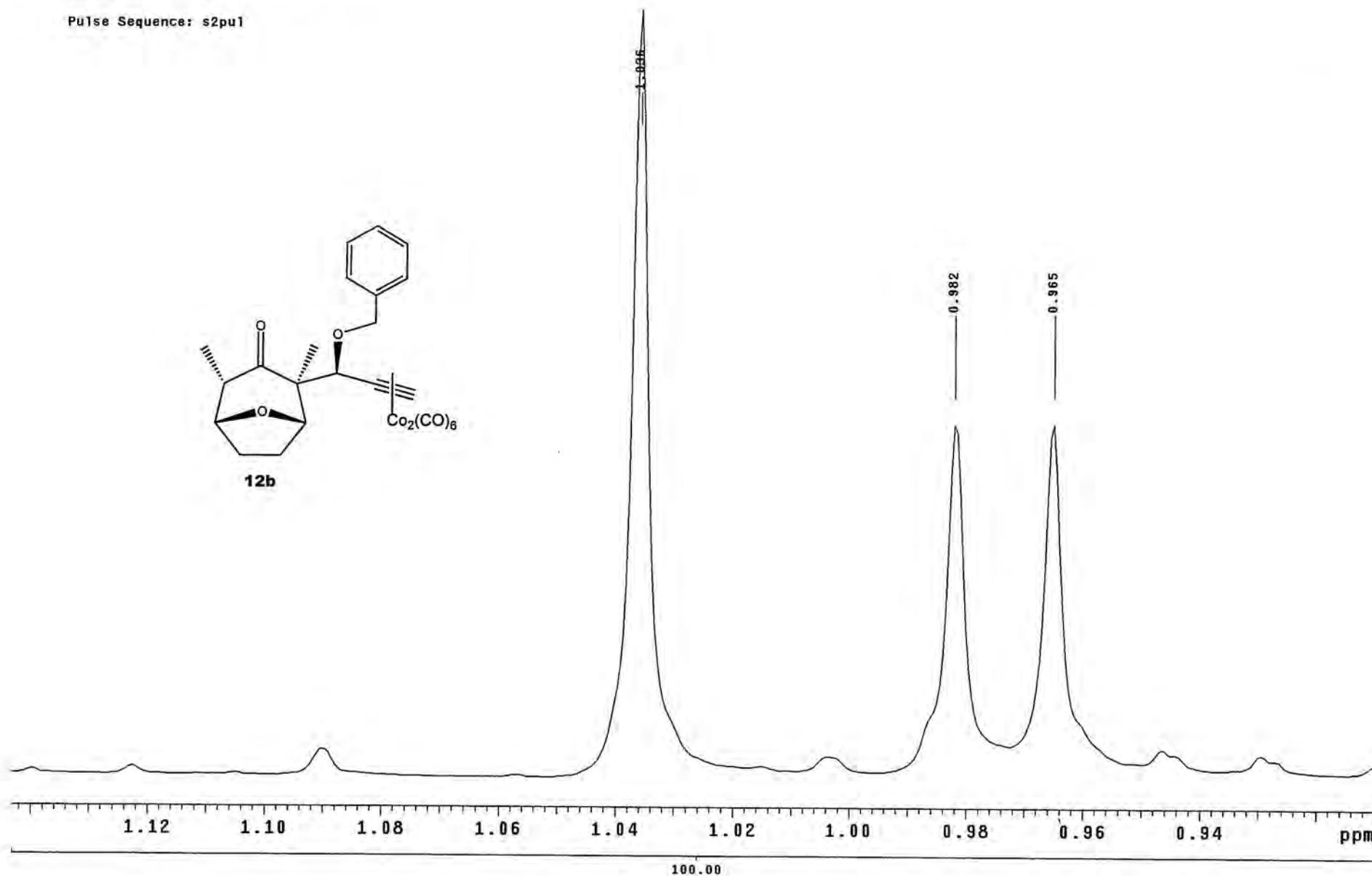
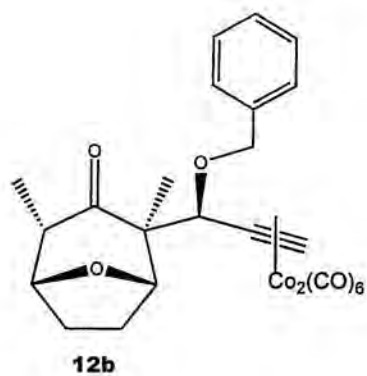
```
H1 / s2pu1 / Mercury-400_qui
cdc13/Temp: 25C /N reg: M40006-200906180511
Usuari: amp / Mostra: colap3
Nom: STEFANO PONZANO
```

Pulse Sequence: s2pu1



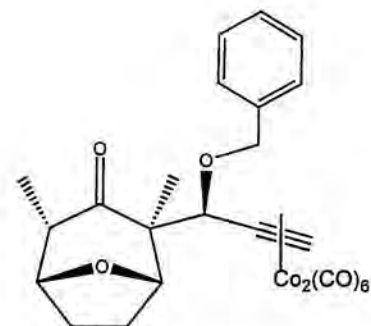
H1 / s2pu1 / Mercury-400.qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

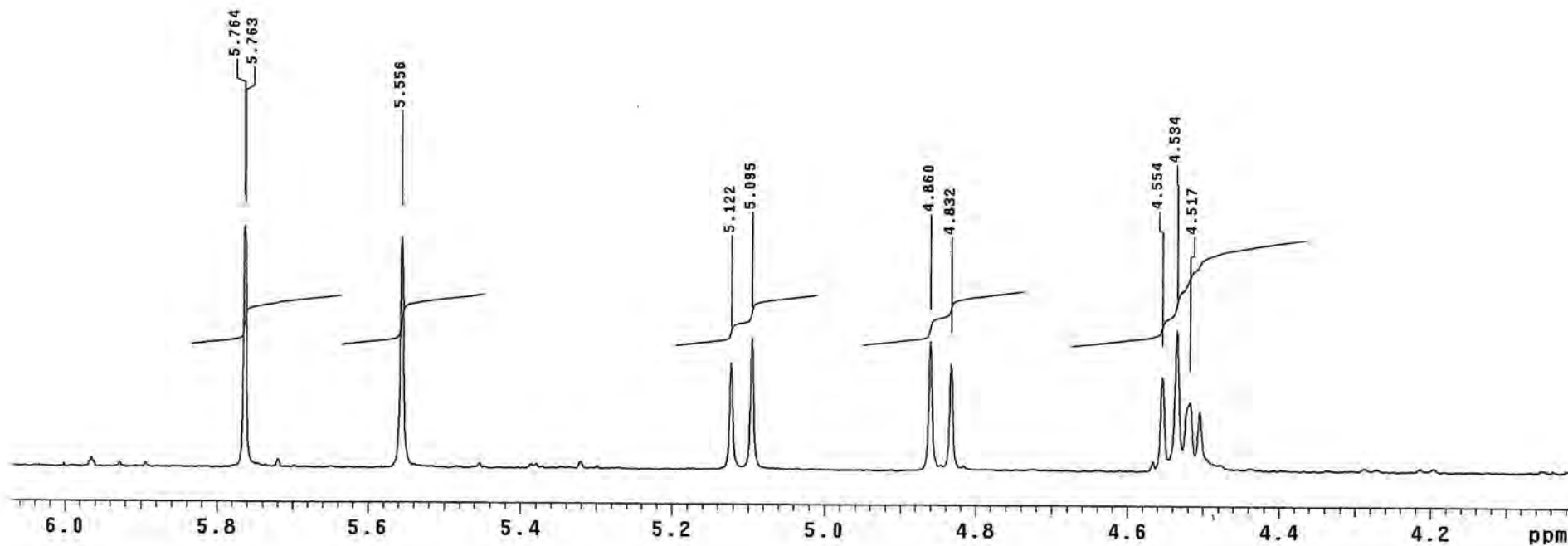


H1 / s2pu1 / Mercury-400\_qi  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

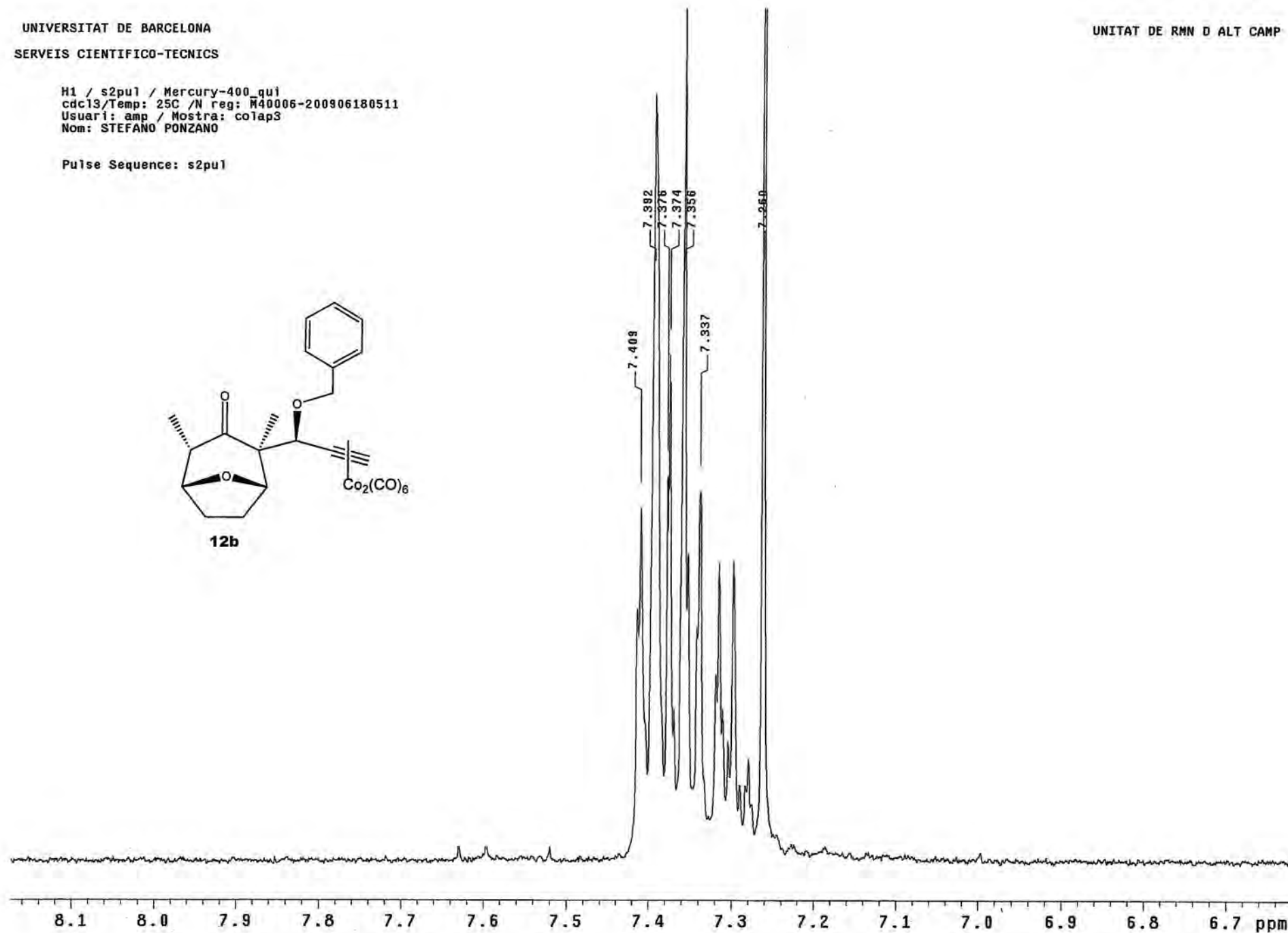
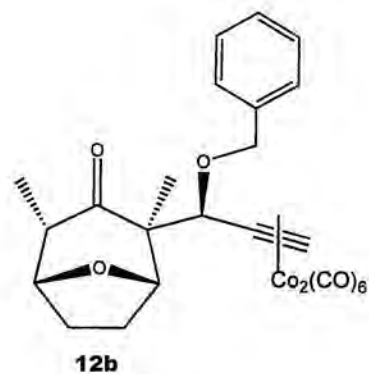


12b



H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



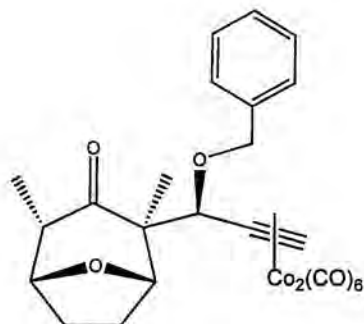
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*

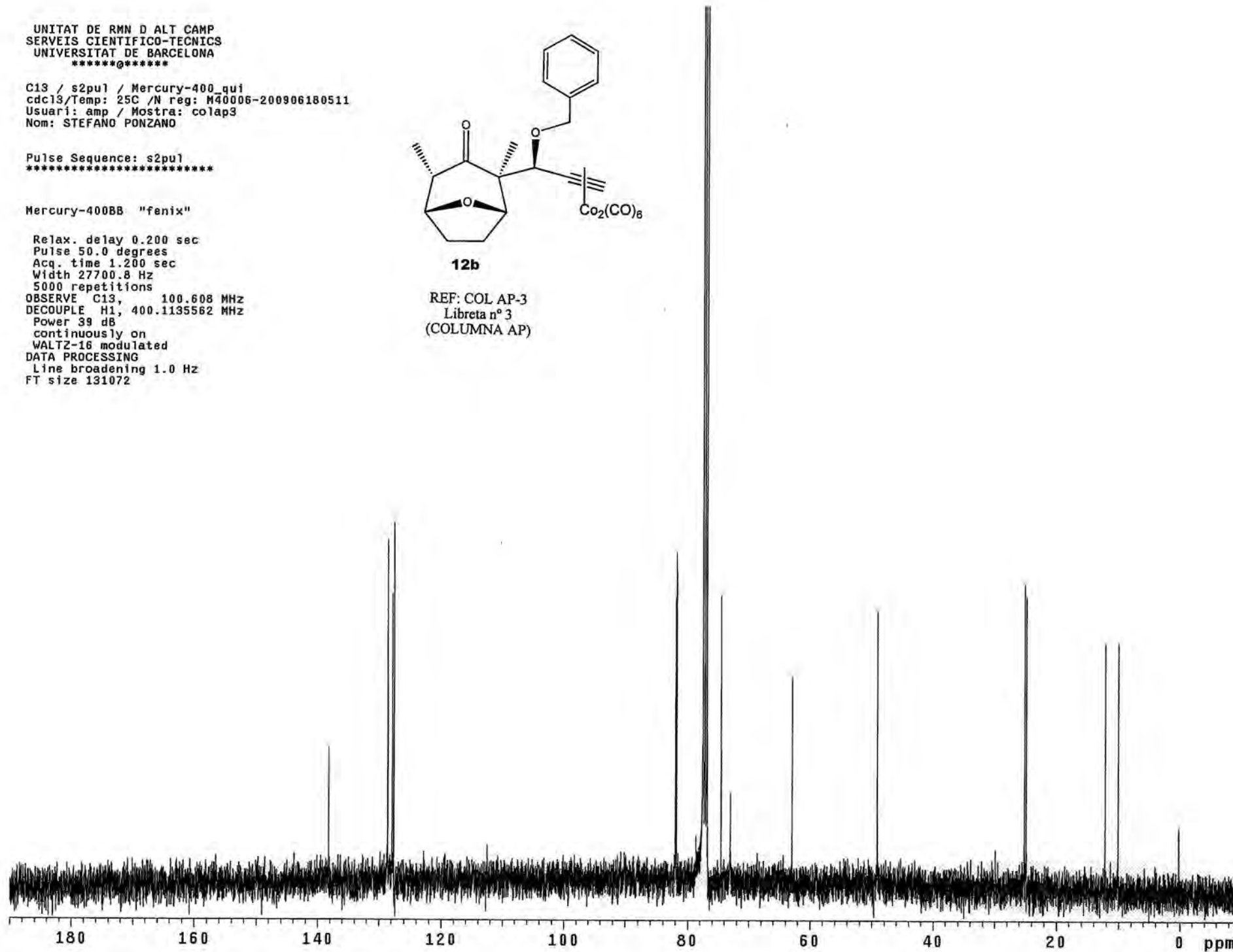
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



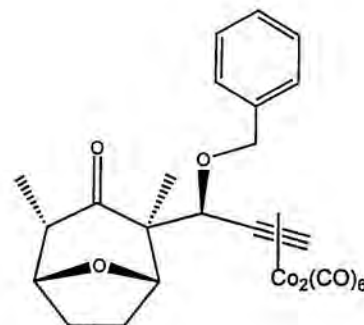
**12b**

REF: COL AP-3  
Libreta n° 3  
(COLUMNNA AP)

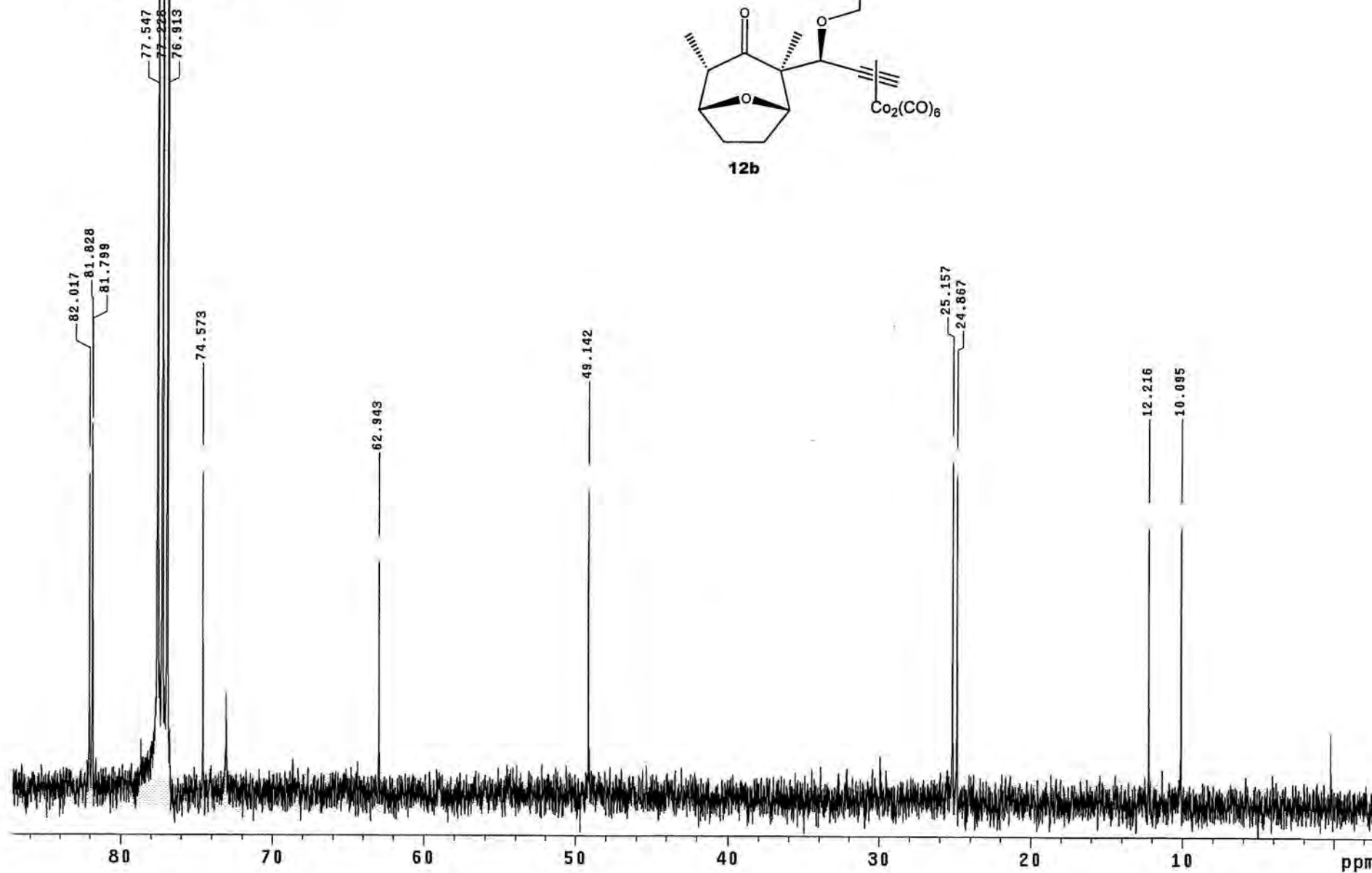


C13 / s2pul / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pul

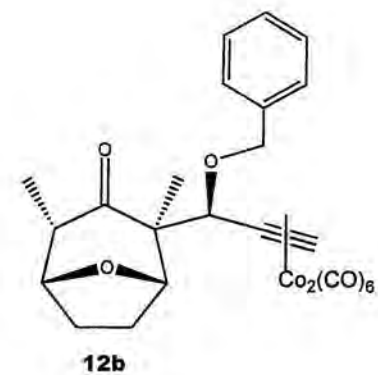
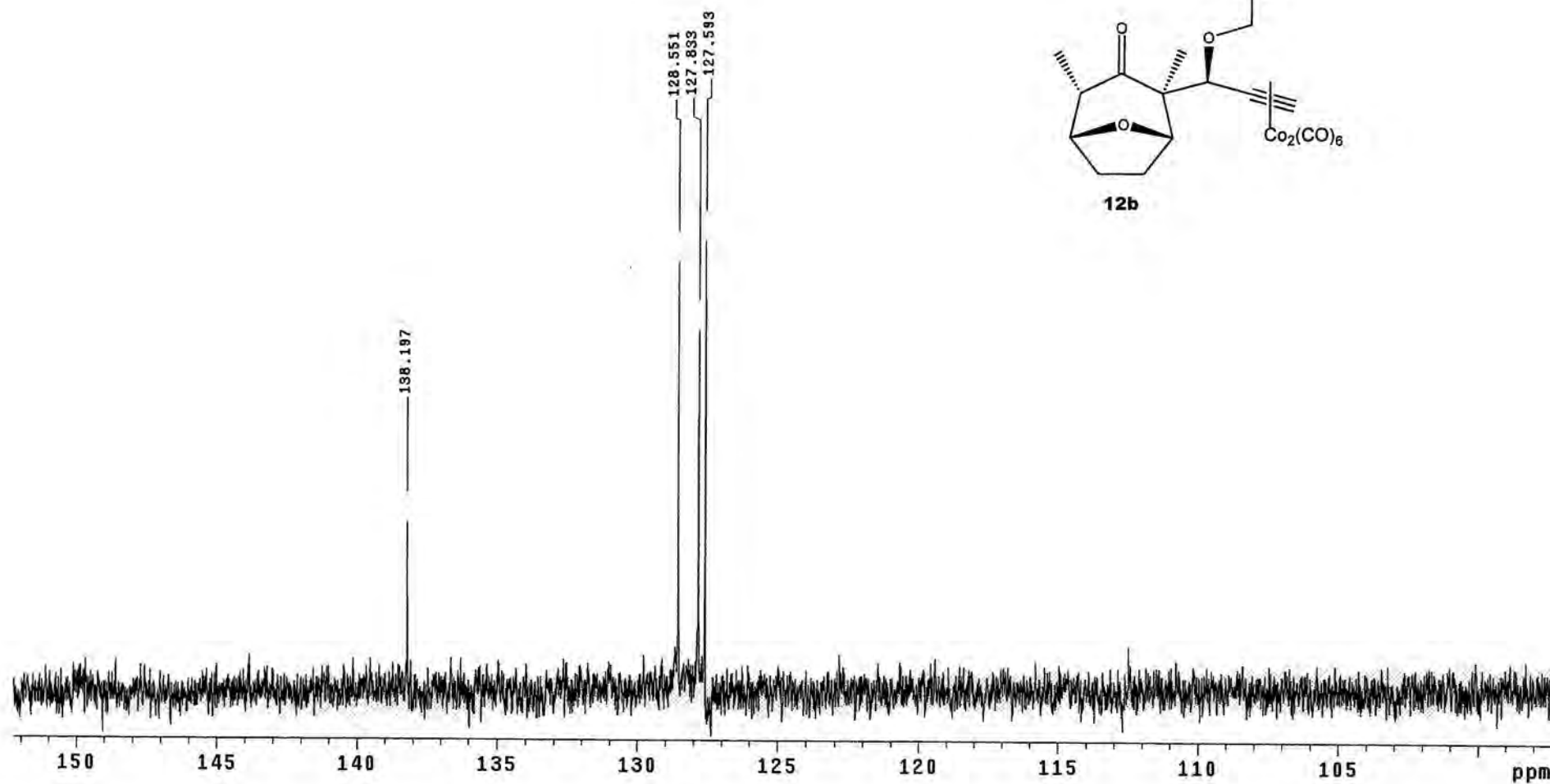


12b



C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

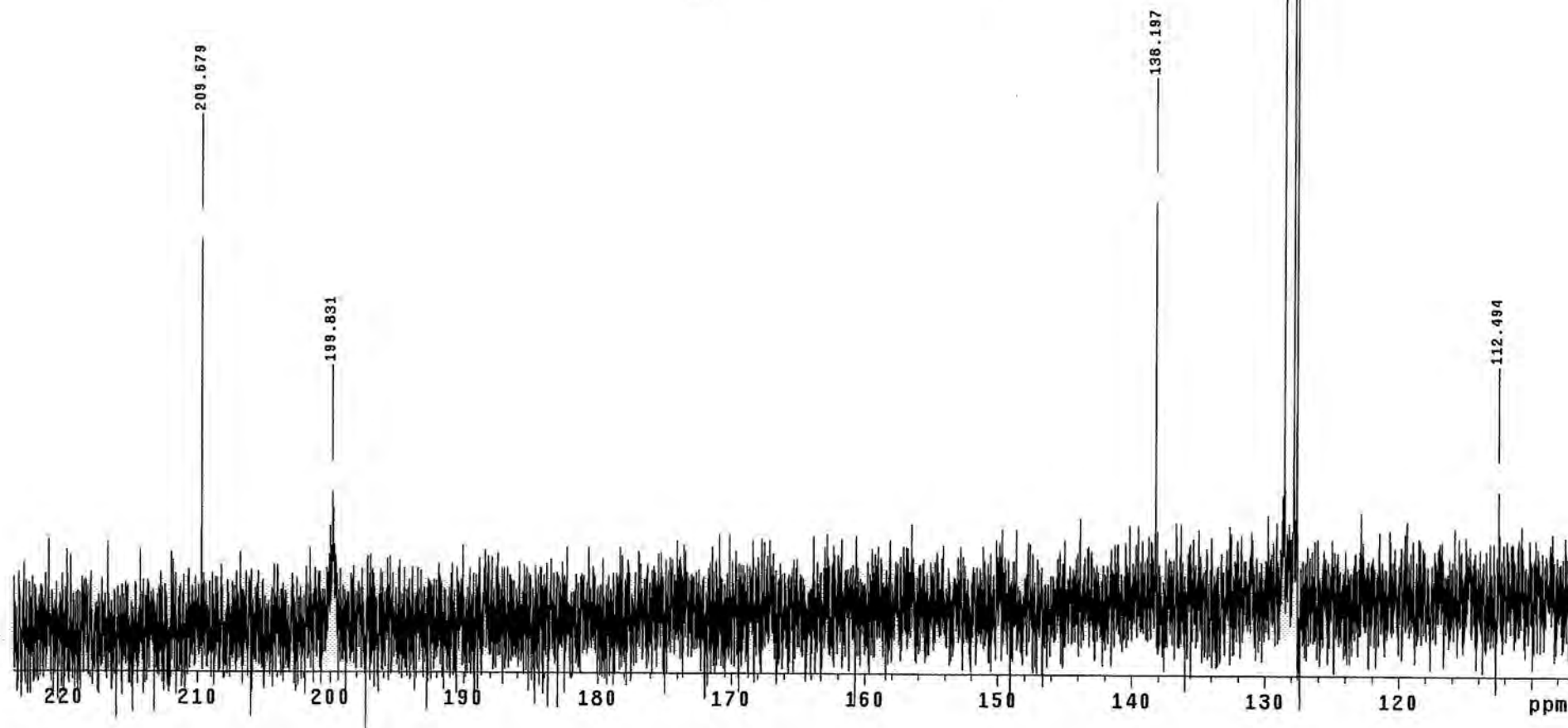
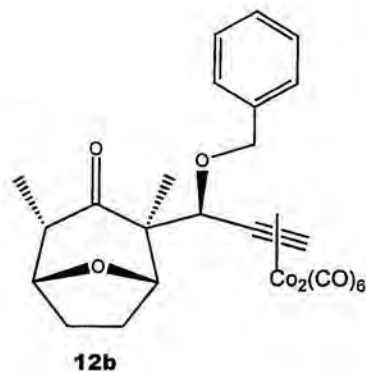
Pulse Sequence: s2pu1



C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

UNITAT DE RMN D ALT CAMP



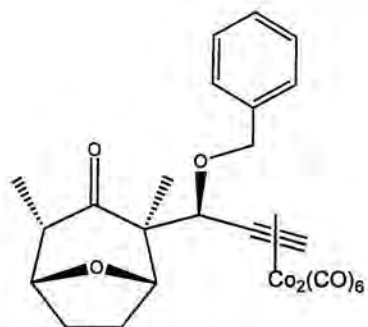
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gHSQC / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-200906180511  
Usuari: amp / Mostra: colap3  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*

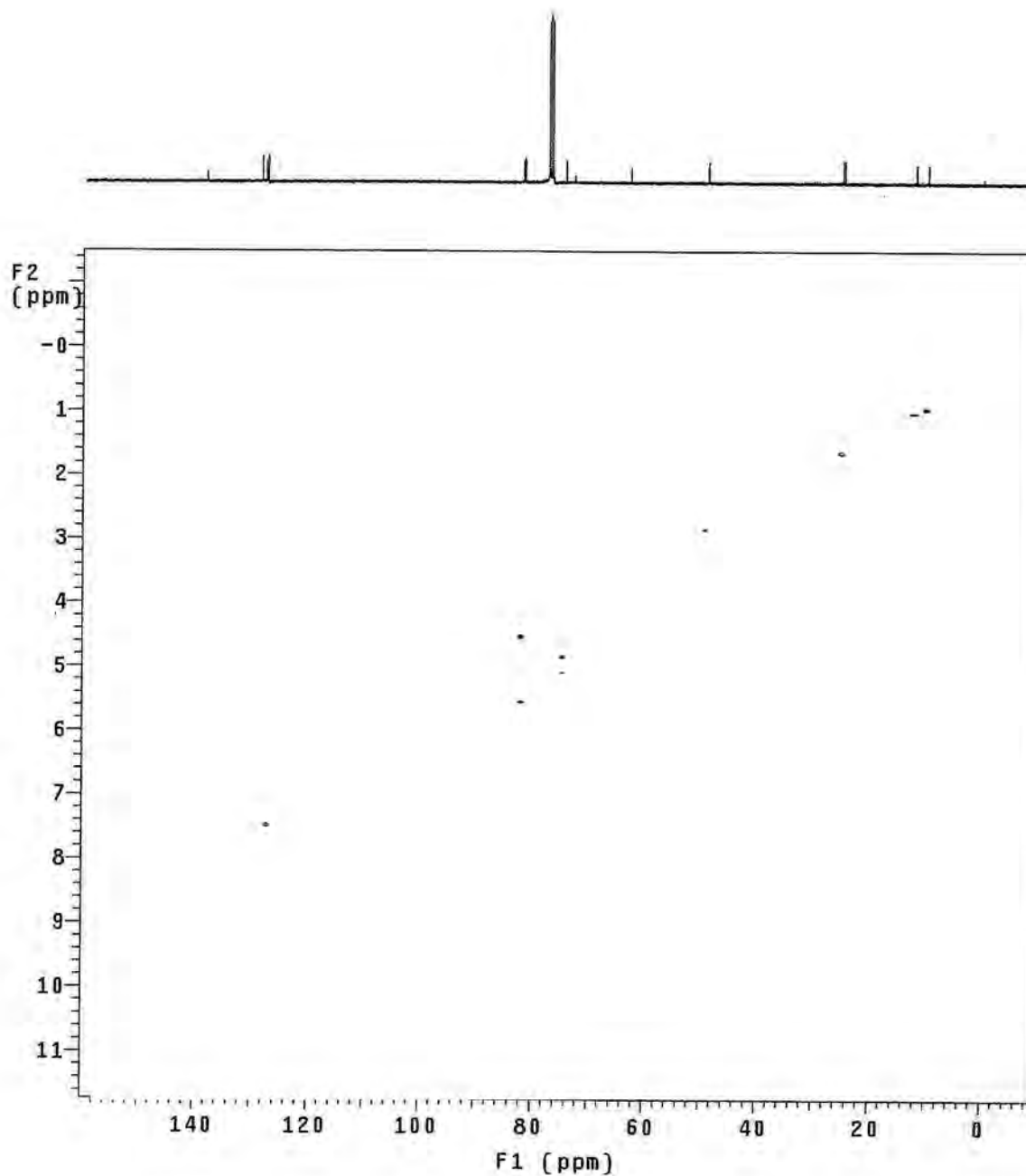
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.194 sec  
Width 5291.0 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.089 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 2048 x 2048

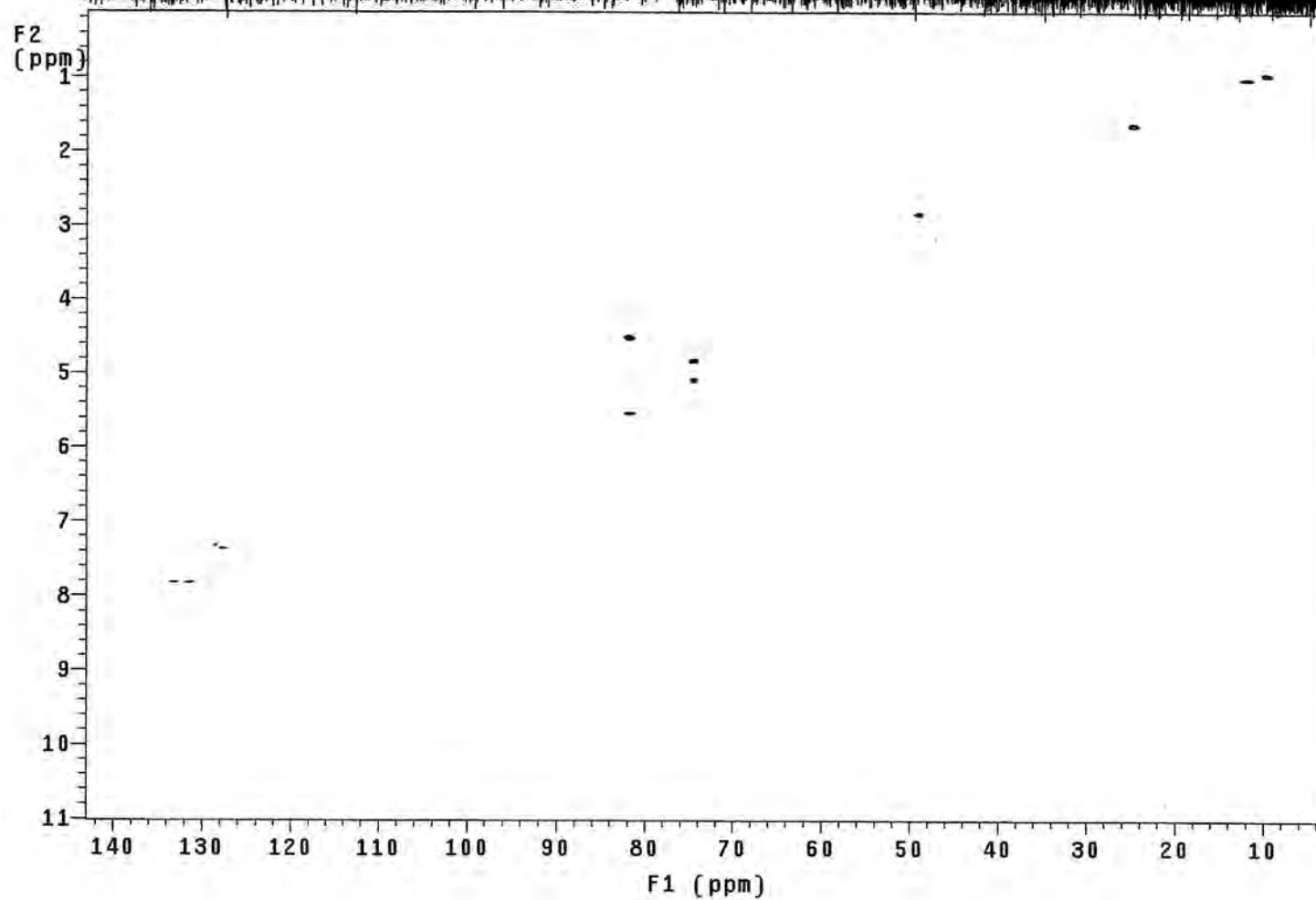


**12b**

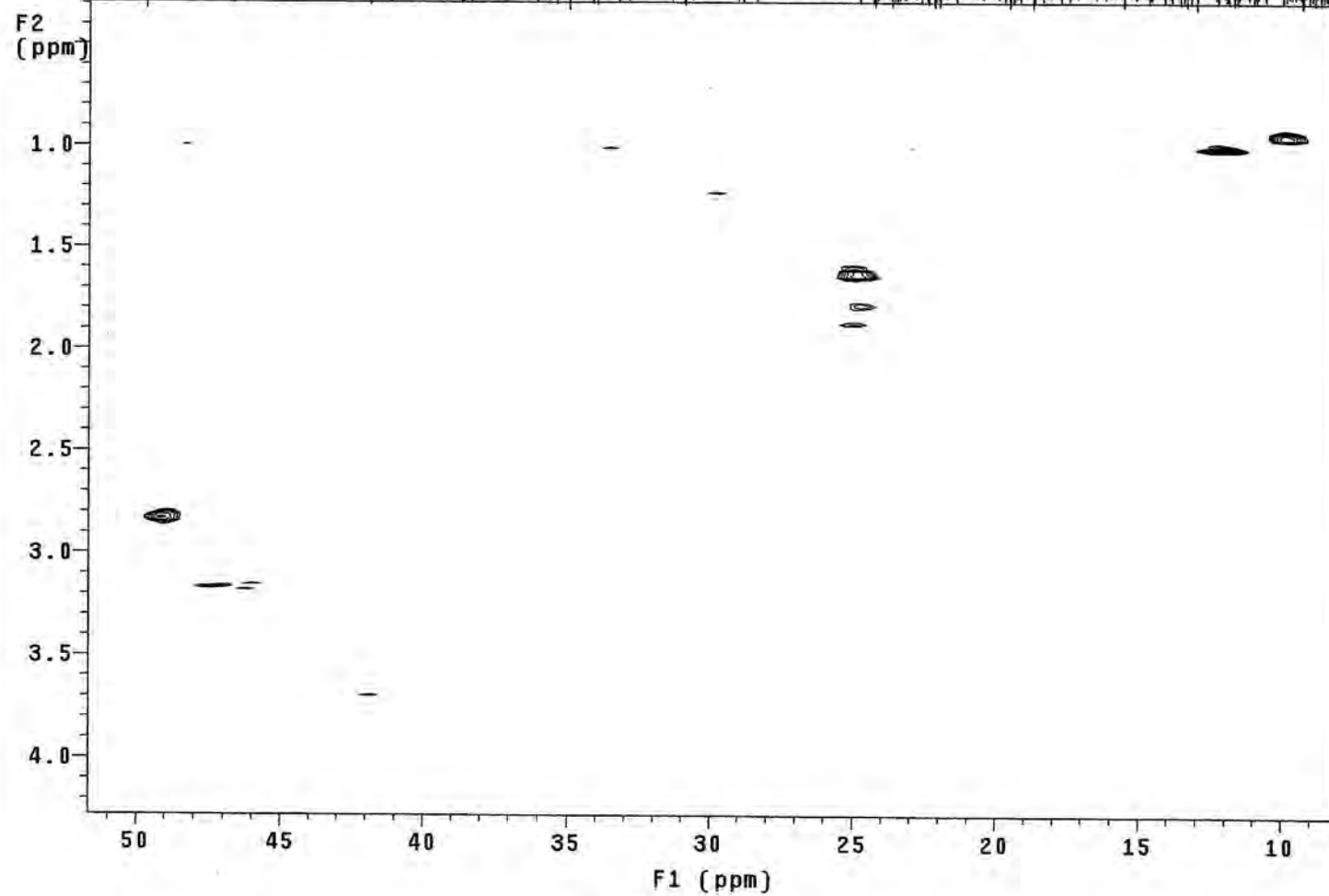
REF: COL AP-3  
Libreta n° 3  
(COLUMNNA AP)



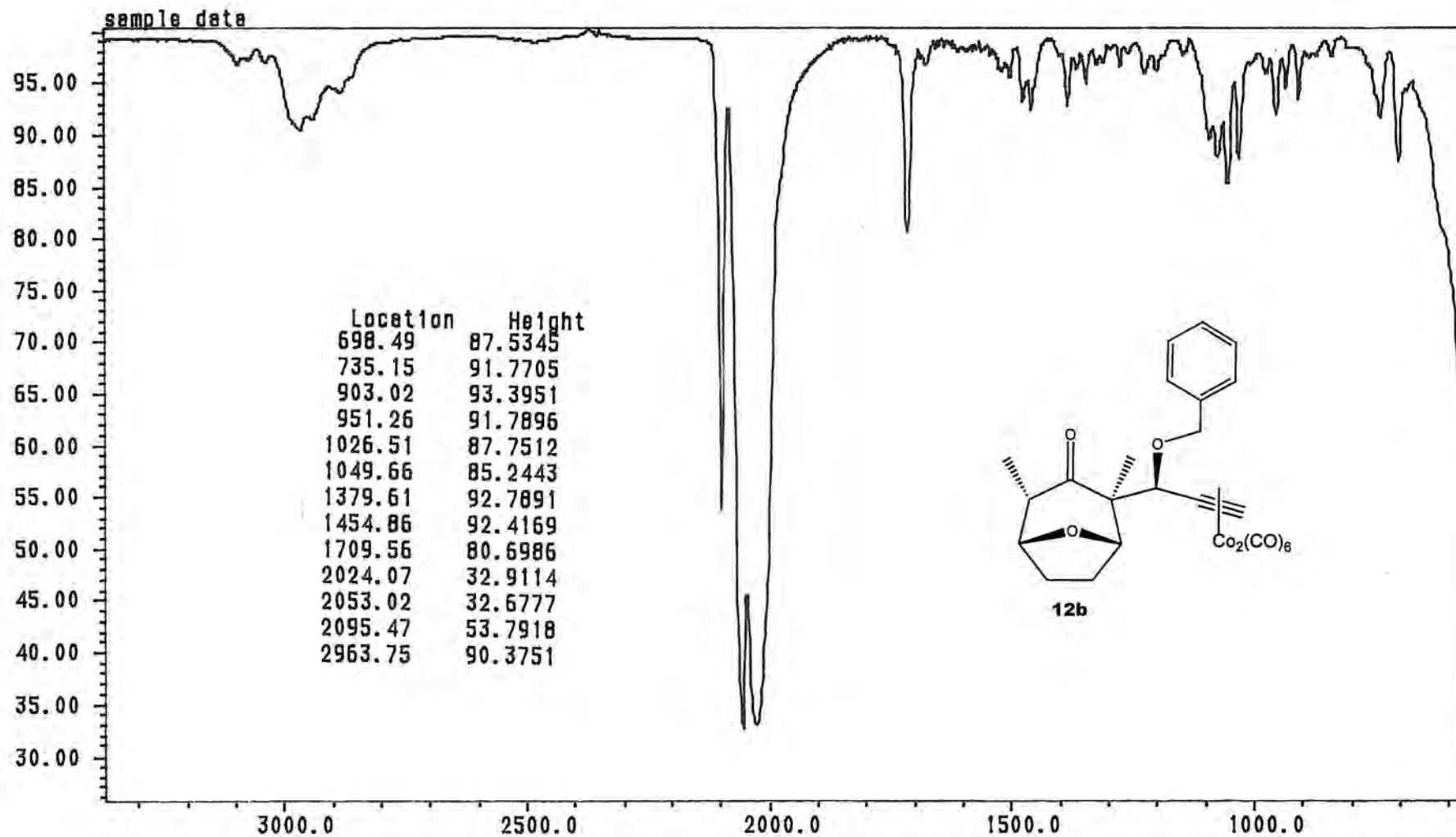
Pulse Sequence: gHSQC



Pulse Sequence: gHSQC

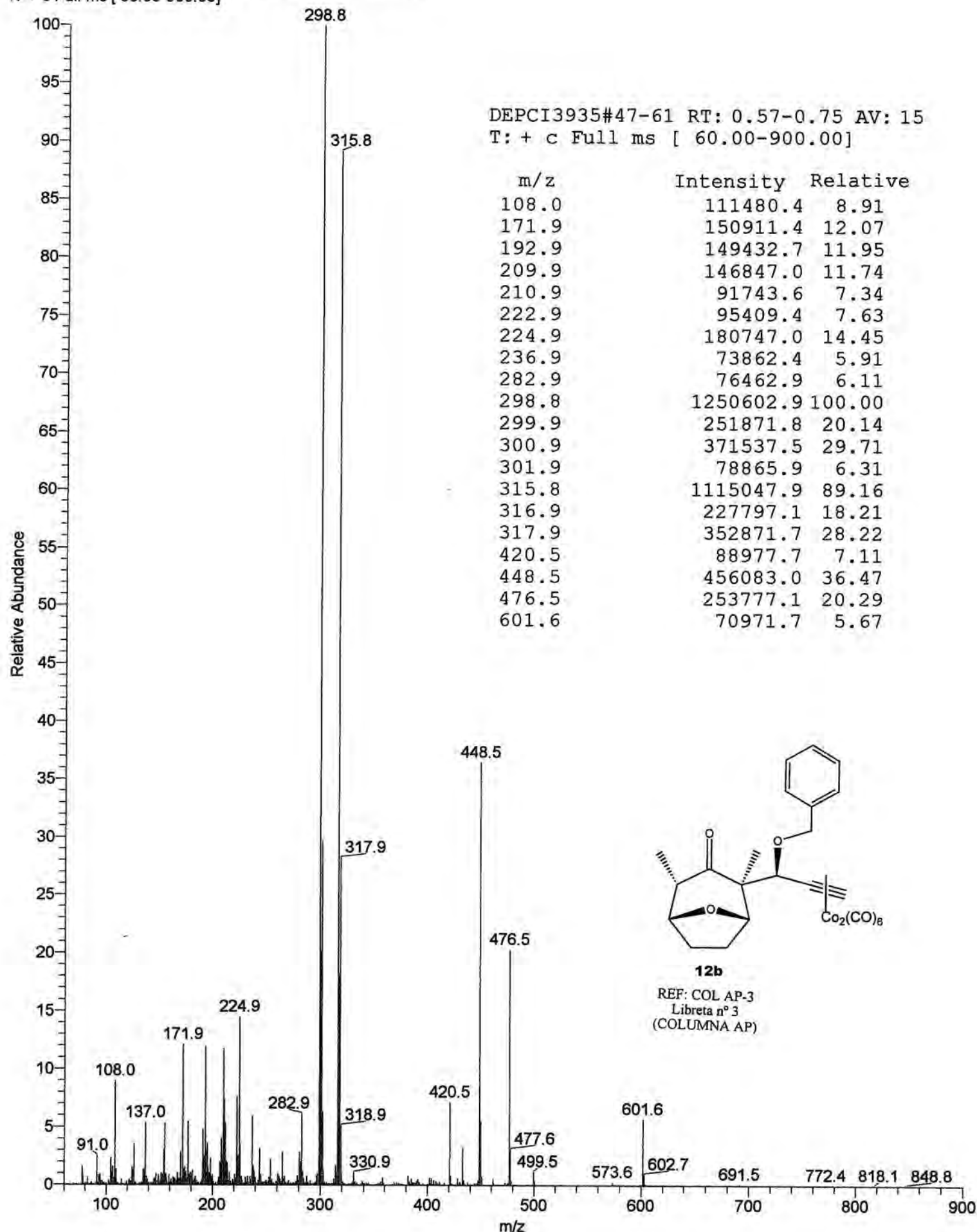


### Transmittance



Wavenumber (cm-1)

N1c/IR



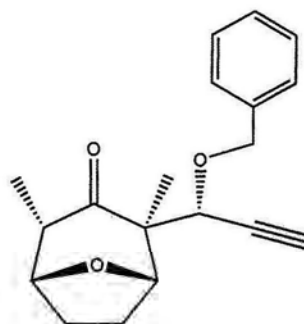
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*

Mercury-400BB "fenix"

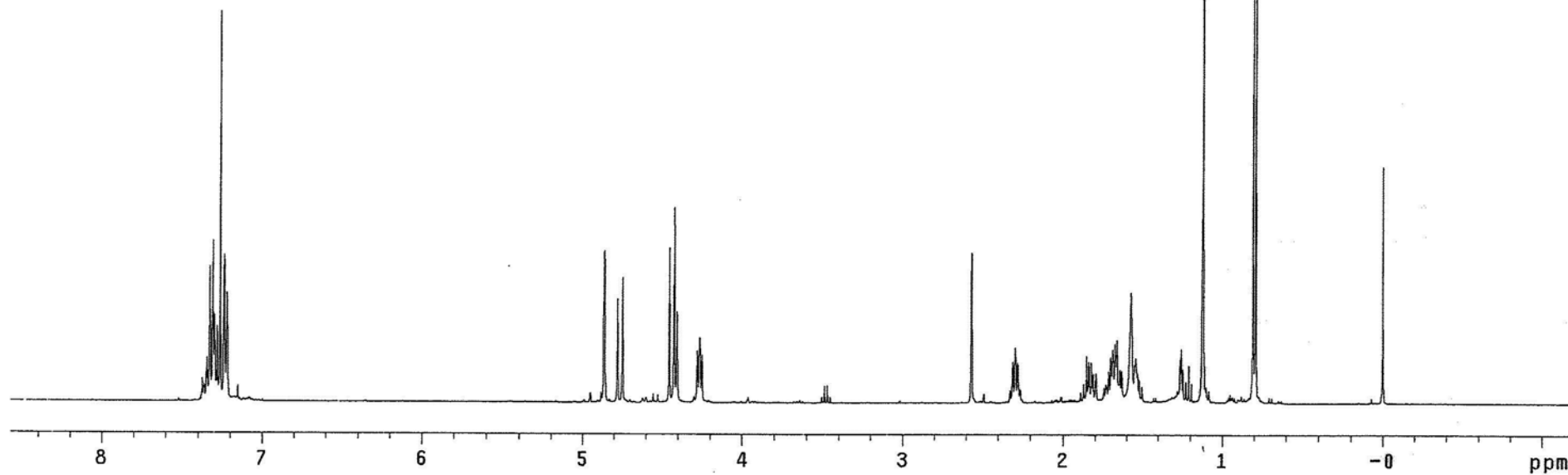
Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 3918.5 Hz  
8 repetitions  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 32768



13a

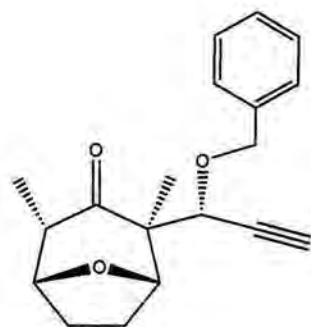
REF: COL-AT  
Libreta n° 3  
(COLUMNNA AT)

| INDEX | FREQUENCY | PPM    | HEIGHT |
|-------|-----------|--------|--------|
| 1     | 2924.043  | 7.308  | 25.5   |
| 2     | 2905.388  | 7.261  | 62.1   |
| 3     | 2895.104  | 7.236  | 23.4   |
| 4     | 1947.050  | 4.866  | 23.8   |
| 5     | 1945.137  | 4.861  | 24.1   |
| 6     | 1782.265  | 4.454  | 24.6   |
| 7     | 1770.067  | 4.424  | 31.0   |
| 8     | 1028.175  | 2.570  | 23.9   |
| 9     | 1026.022  | 2.564  | 23.8   |
| 10    | 448.915   | 1.122  | 151.2  |
| 11    | 323.592   | 0.809  | 78.3   |
| 12    | 316.895   | 0.792  | 76.5   |
| 13    | -0.000    | -0.000 | 37.5   |



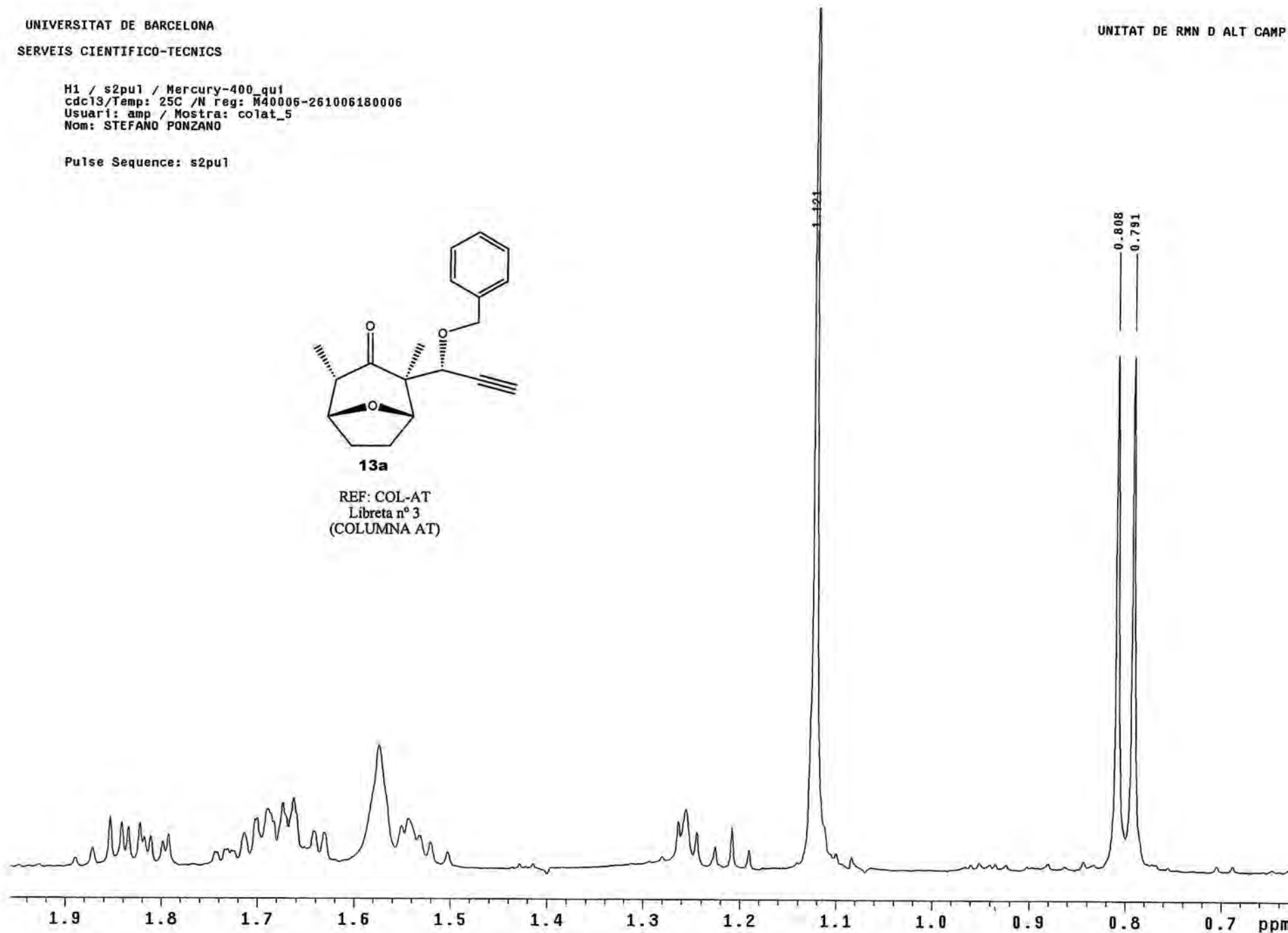
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



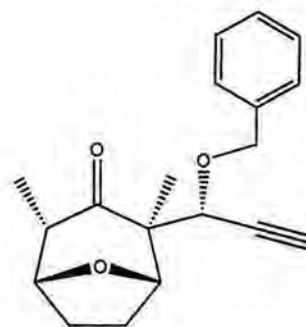
**13a**

REF: COL-AT  
Libreta nº 3  
(COLUMN A T)



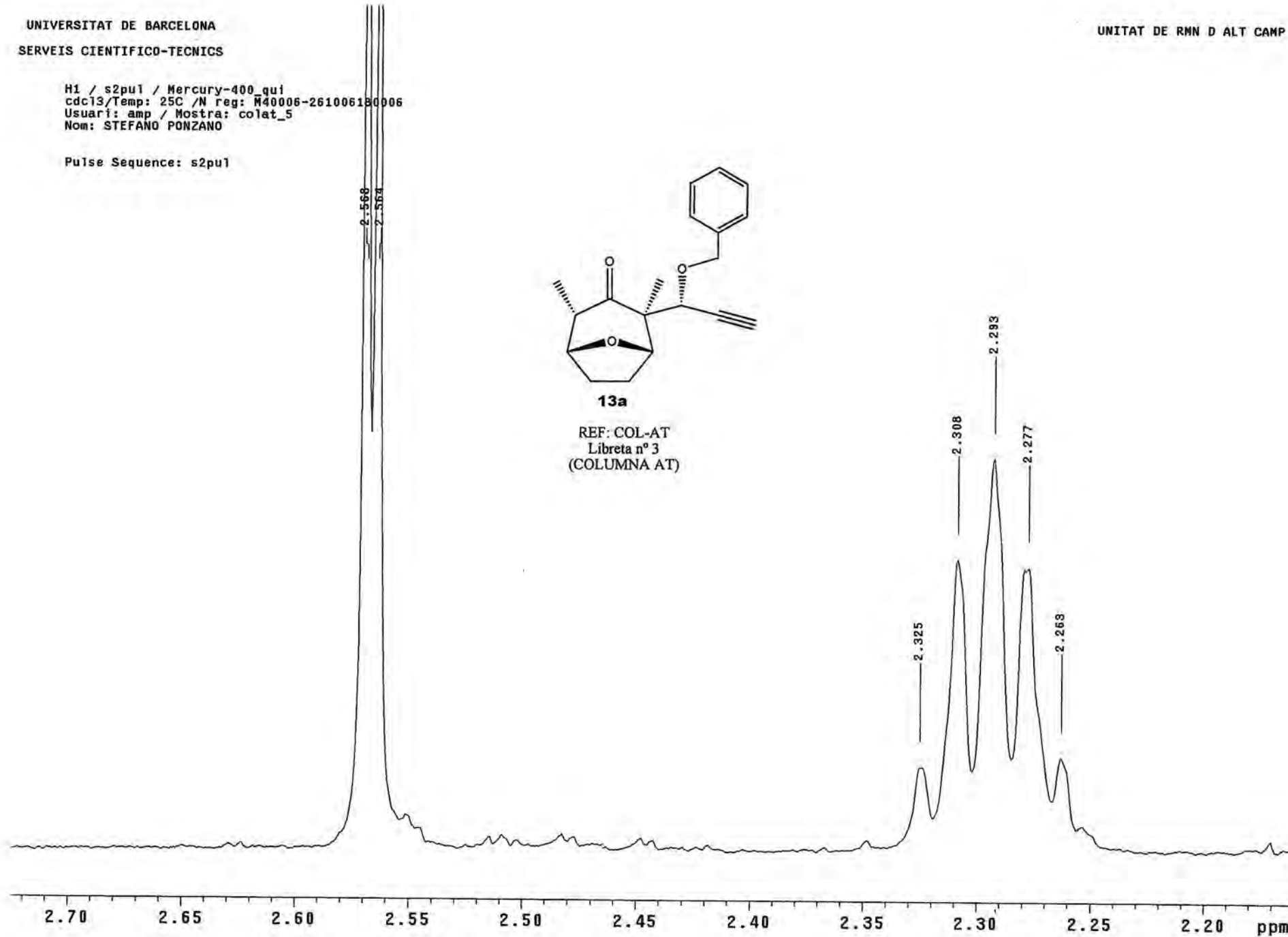
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006130006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



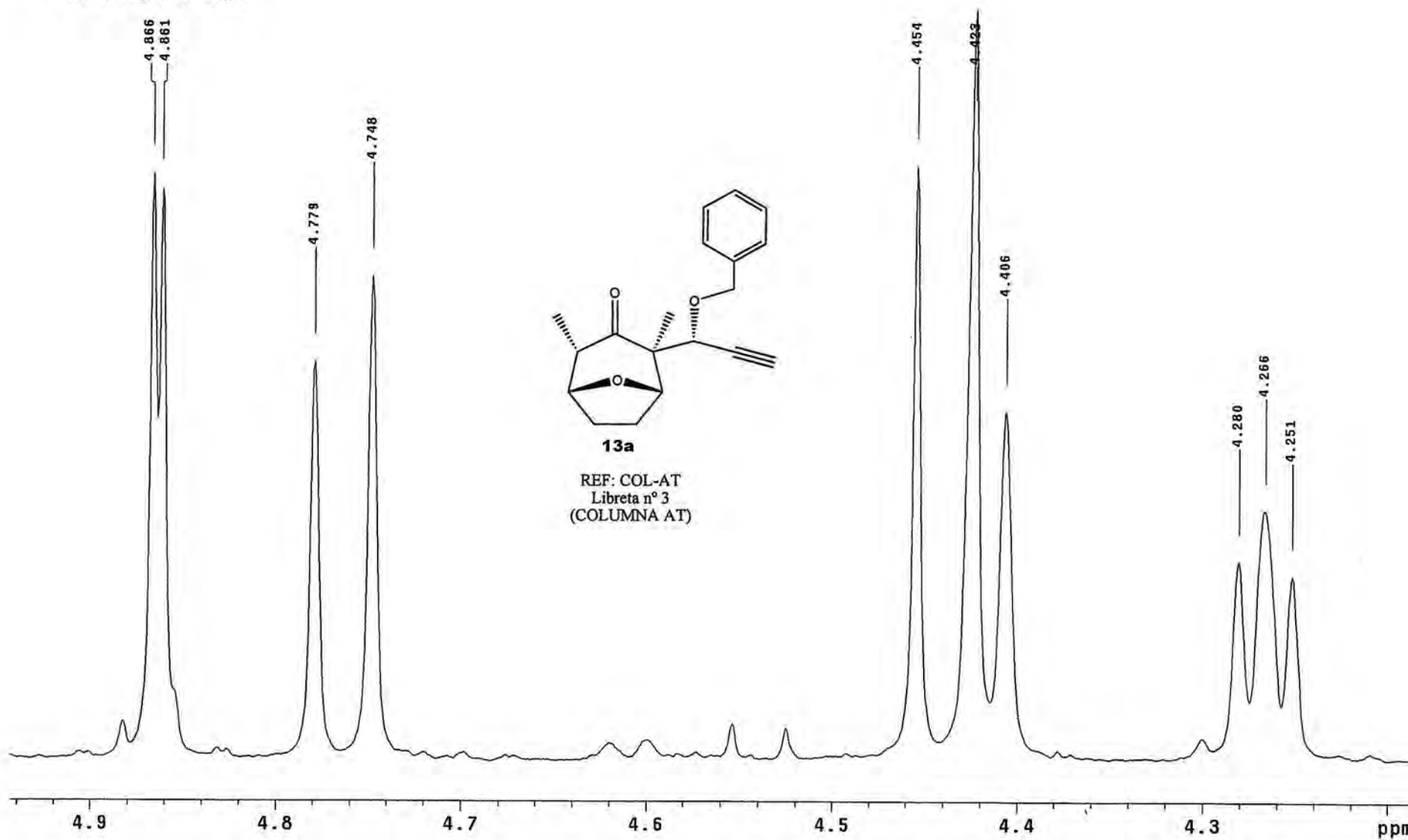
**13a**

REF: COL-AT  
Libreta nº 3  
(COLUMNNA AT)



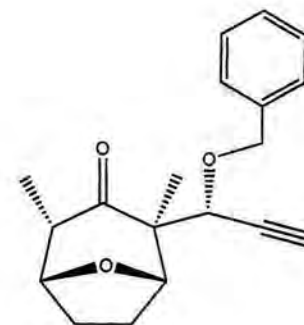
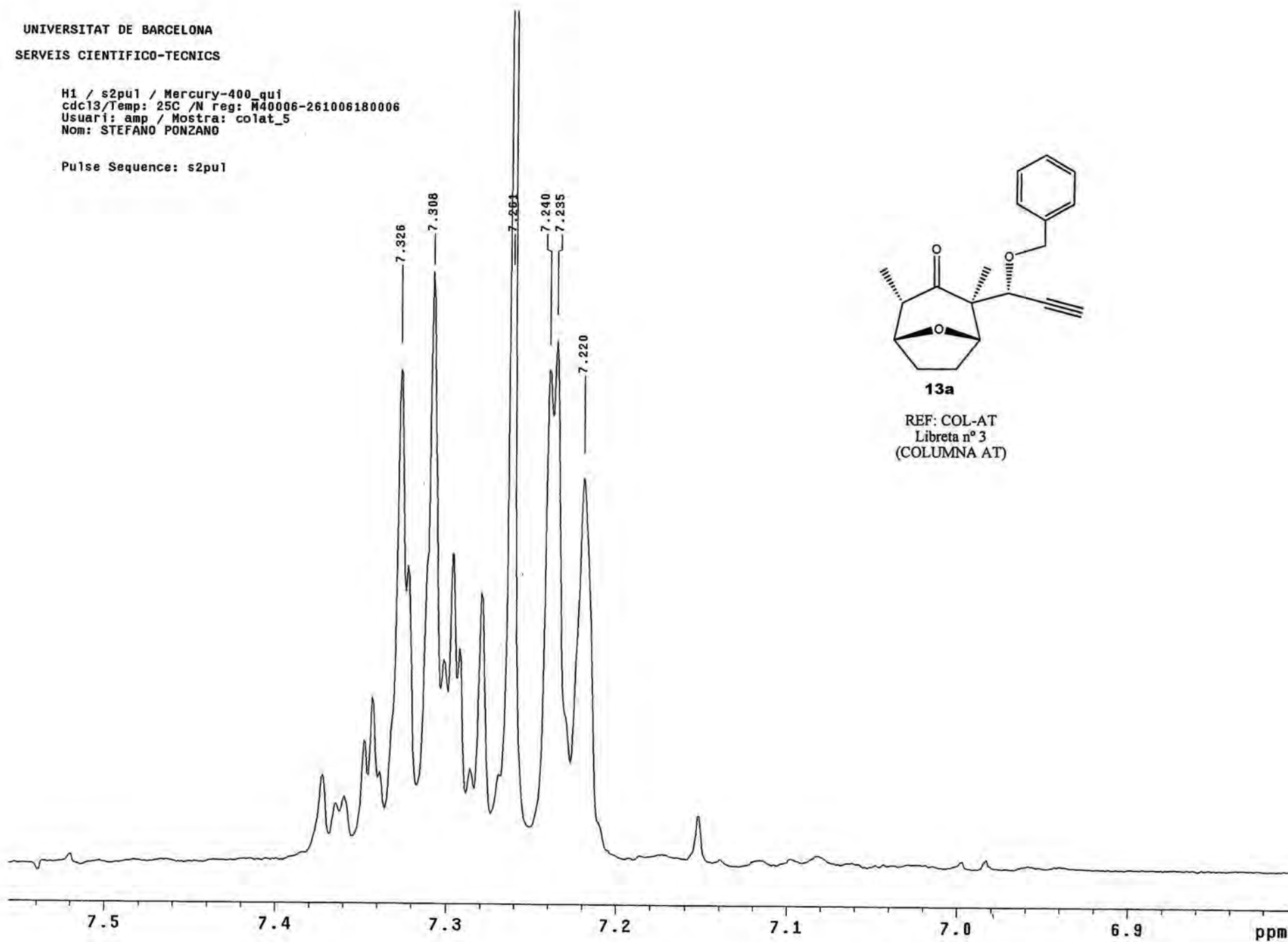
H1 / s2pu1 / Mercury-400.qui  
cdc13/Tmp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



H1 / s2pu1 / Mercury-400\_qul  
 cdc13/Temp: 25C /N reg: M40006-261006180006  
 Usuari: amp / Mostra: colat\_5  
 Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



**13a**

REF: COL-AT  
 Libreta nº 3  
 (COLUMNNA AT)

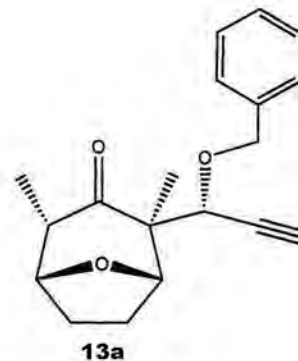
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pul / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

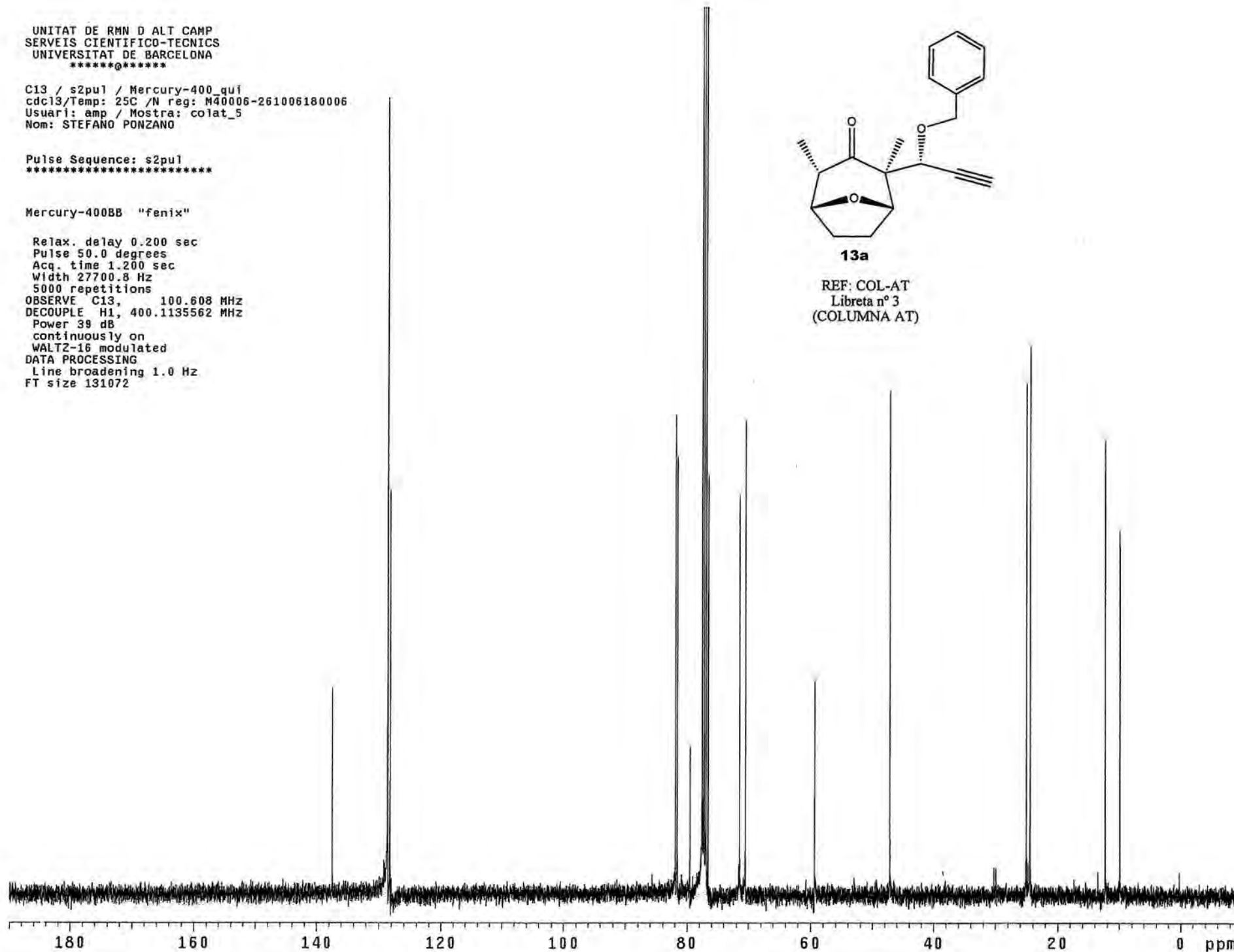
Pulse Sequence: s2pul  
\*\*\*\*\*

Mercury-400BB "fenix"

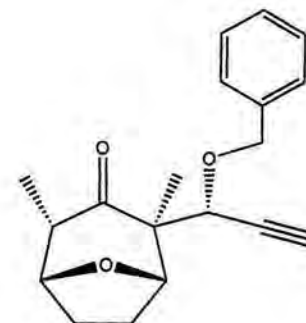
Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



REF: COL-AT  
Libreta nº 3  
(COLUMNNA AT)

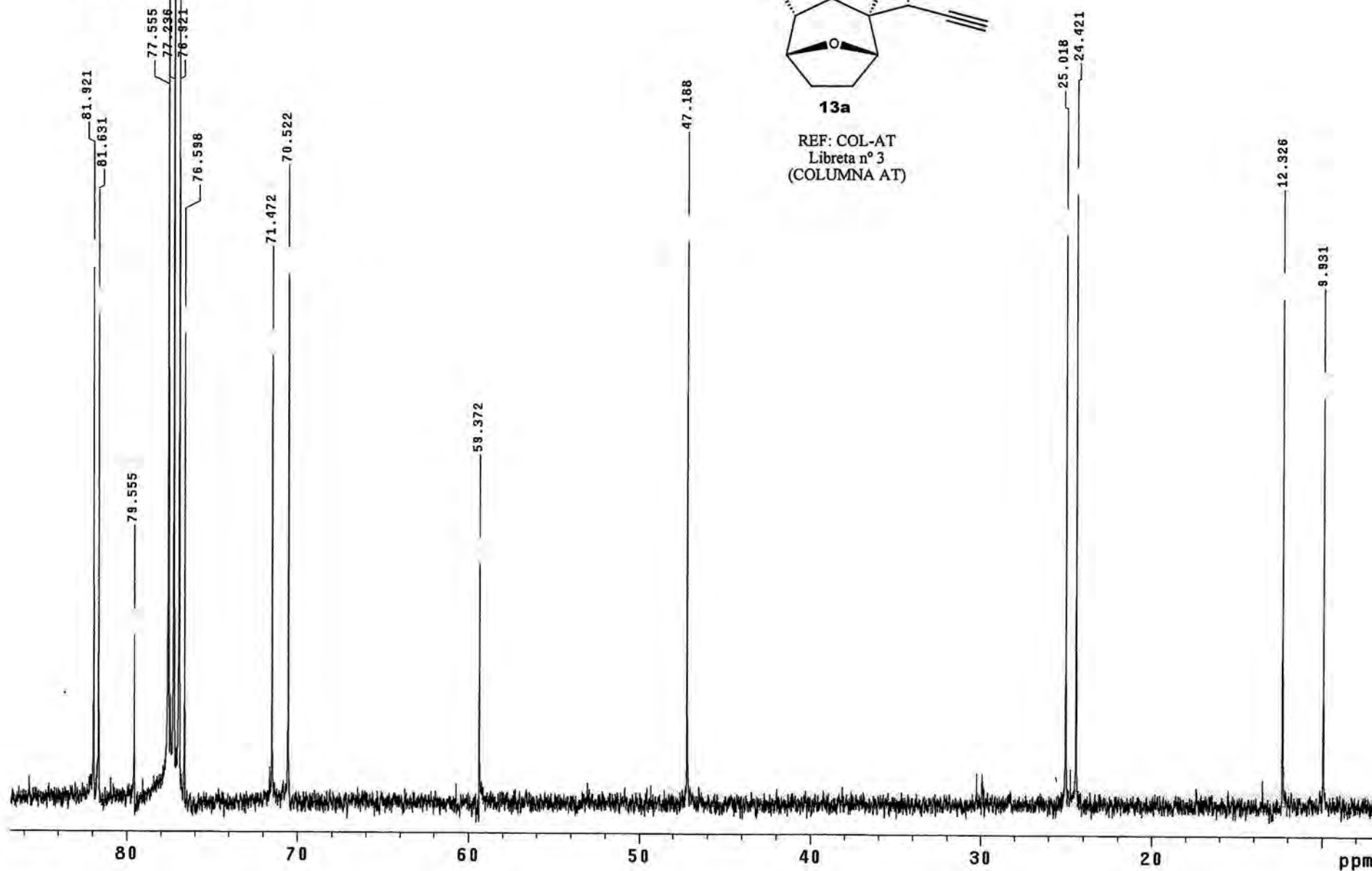


C13 / s2pul / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO  
Sist automatic  
Pulse Sequence: s2pul



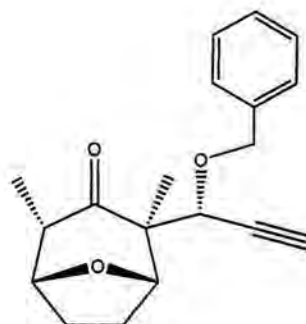
**13a**

REF: COL-AT  
Libreta n° 3  
(COLUMNNA AT)



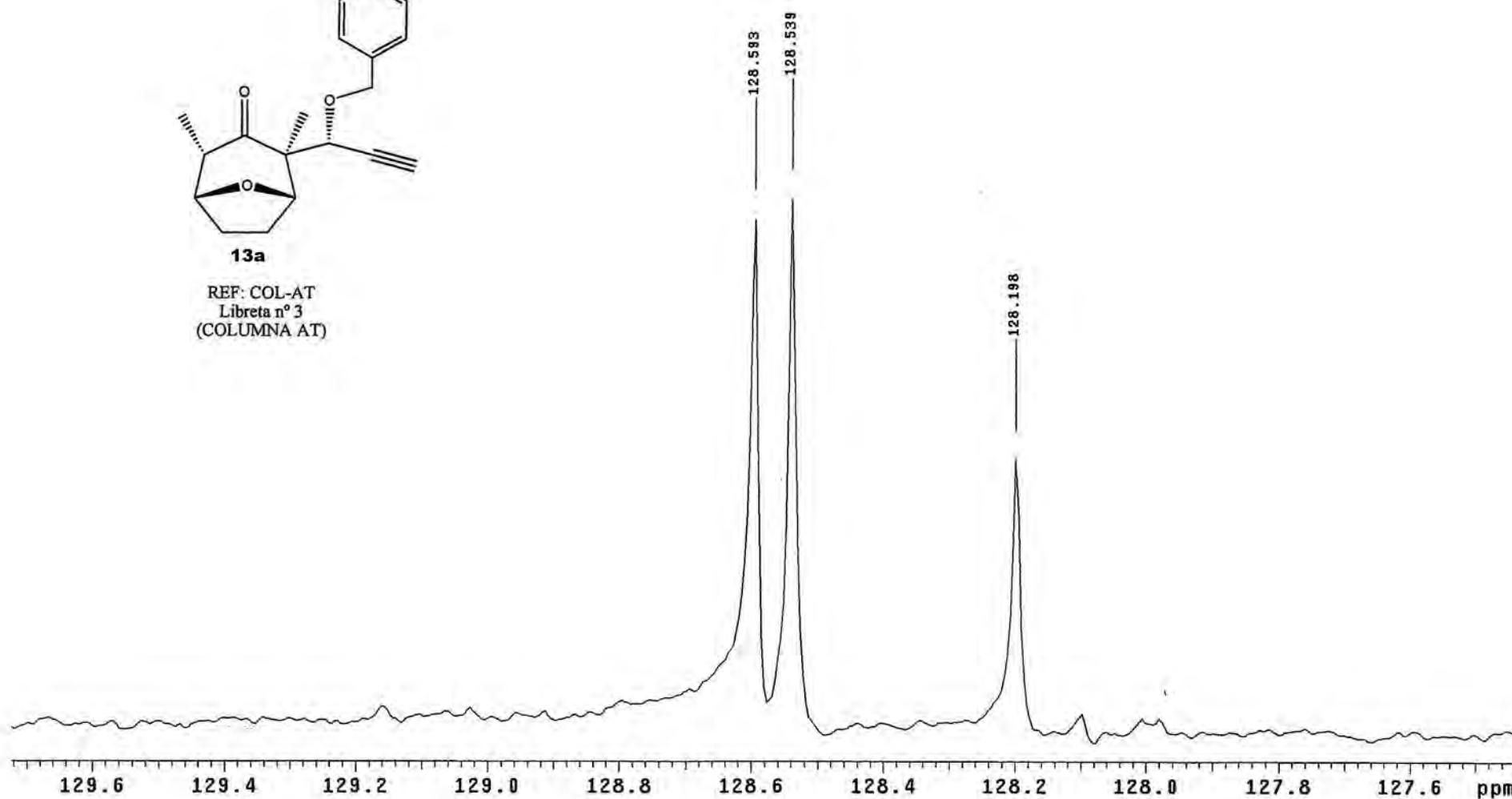
C13 / s2pul / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: s2pul



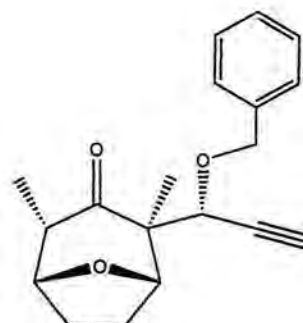
**13a**

REF: COL-AT  
Libreta n° 3  
(COLUMN AT)



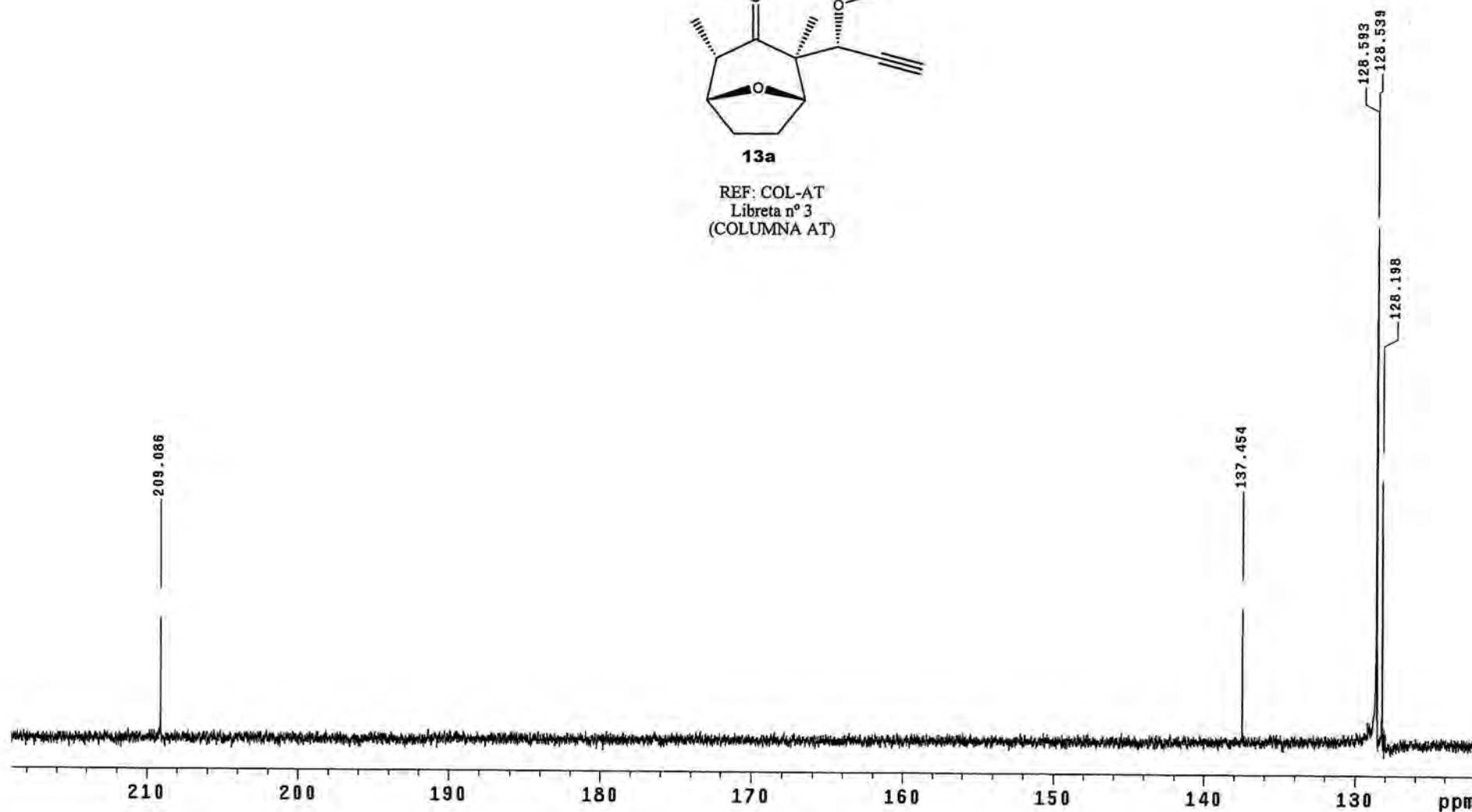
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



13a

REF: COL-AT  
Libreta n° 3  
(COLUMNNA AT)



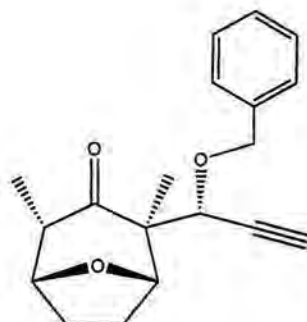
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gCOSY / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*

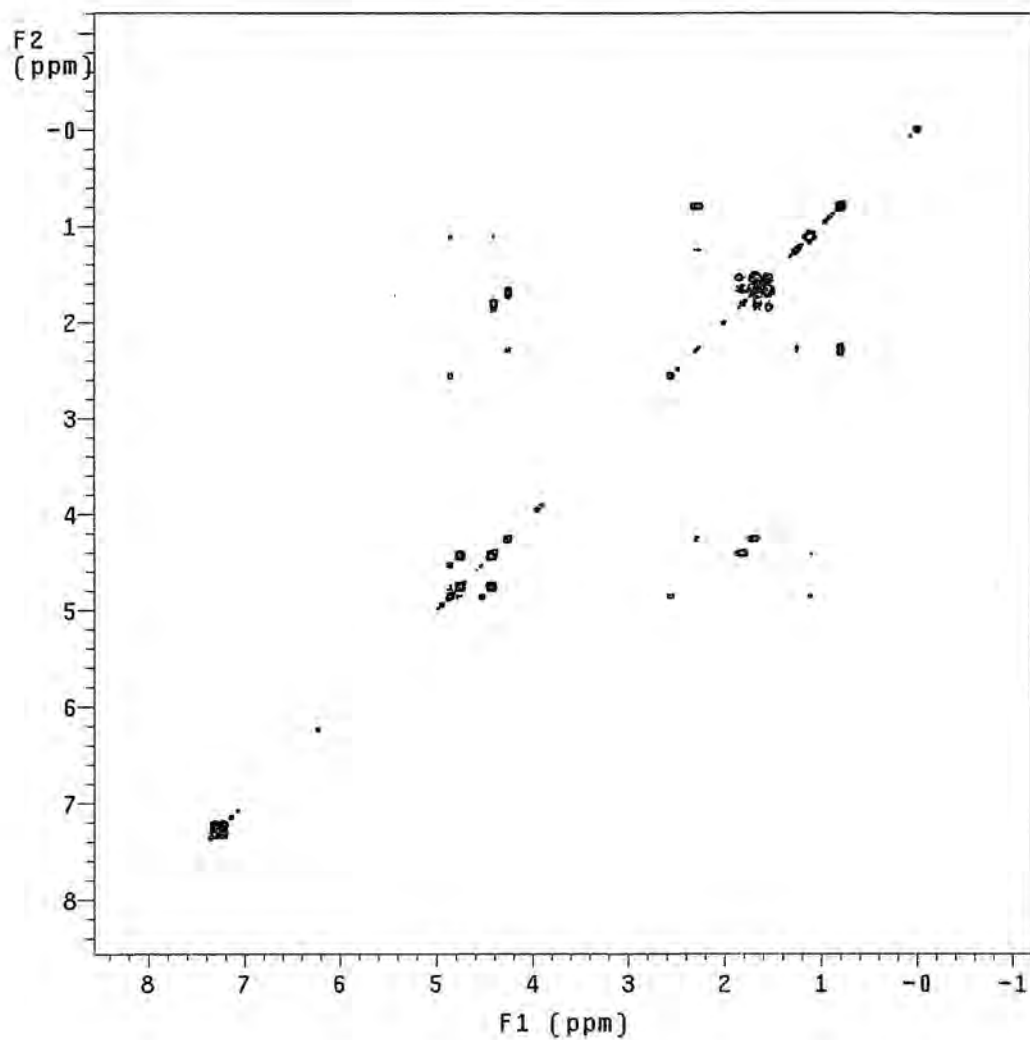
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3906.2 Hz  
2D Width 3906.2 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.066 sec  
FT size 2048 x 2048



**13a**

REF: COL-AT  
Libreta nº 3  
(COLUMN A T)



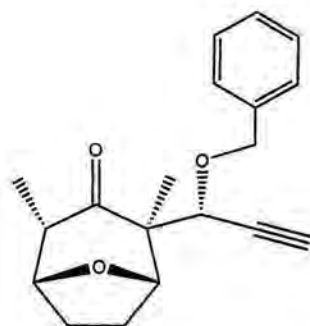
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TÈCNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gHSQC / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-261006180006  
Usuari: amp / Mostra: colat\_5  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*

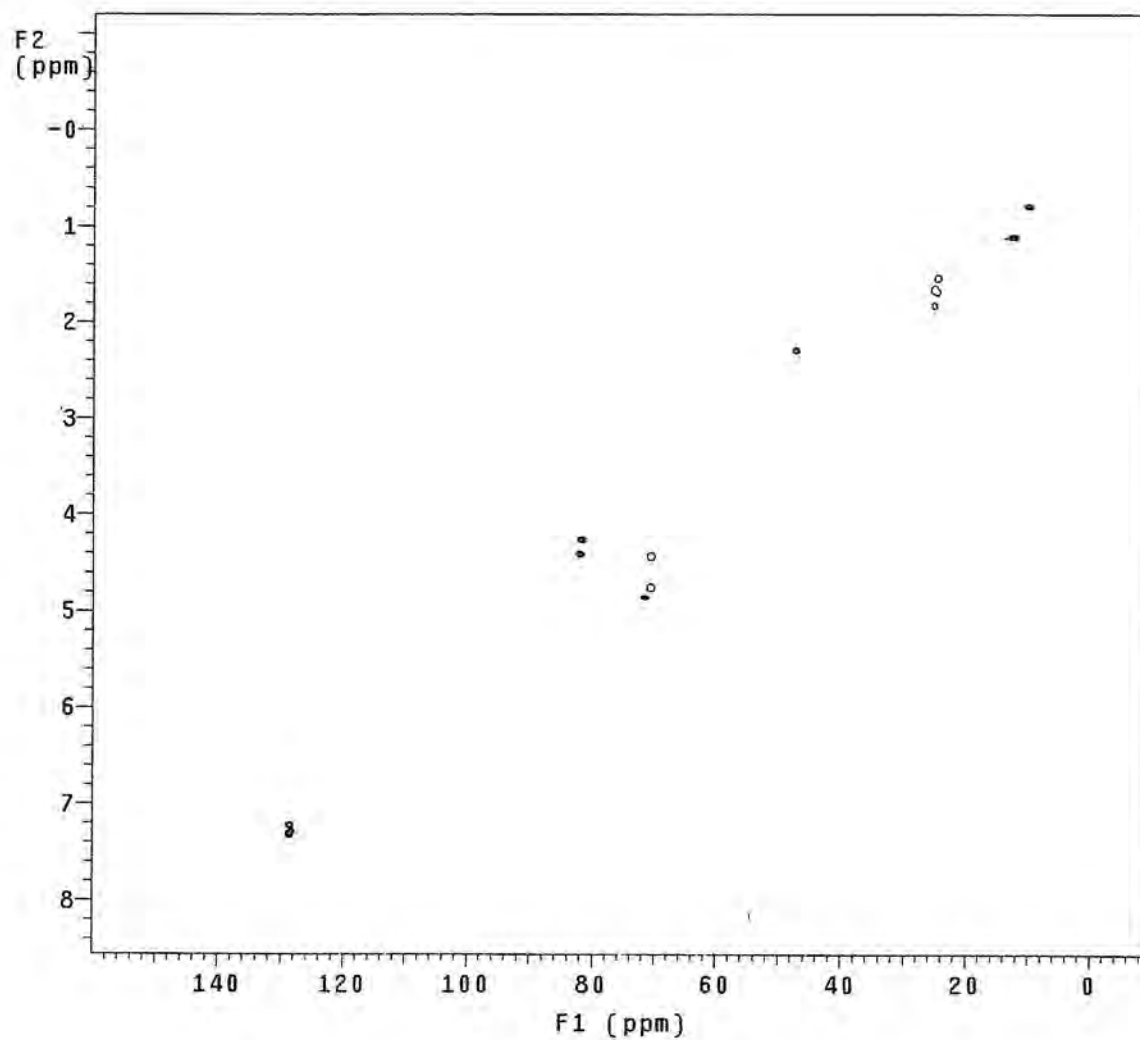
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.131 sec  
Width 3906.2 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048

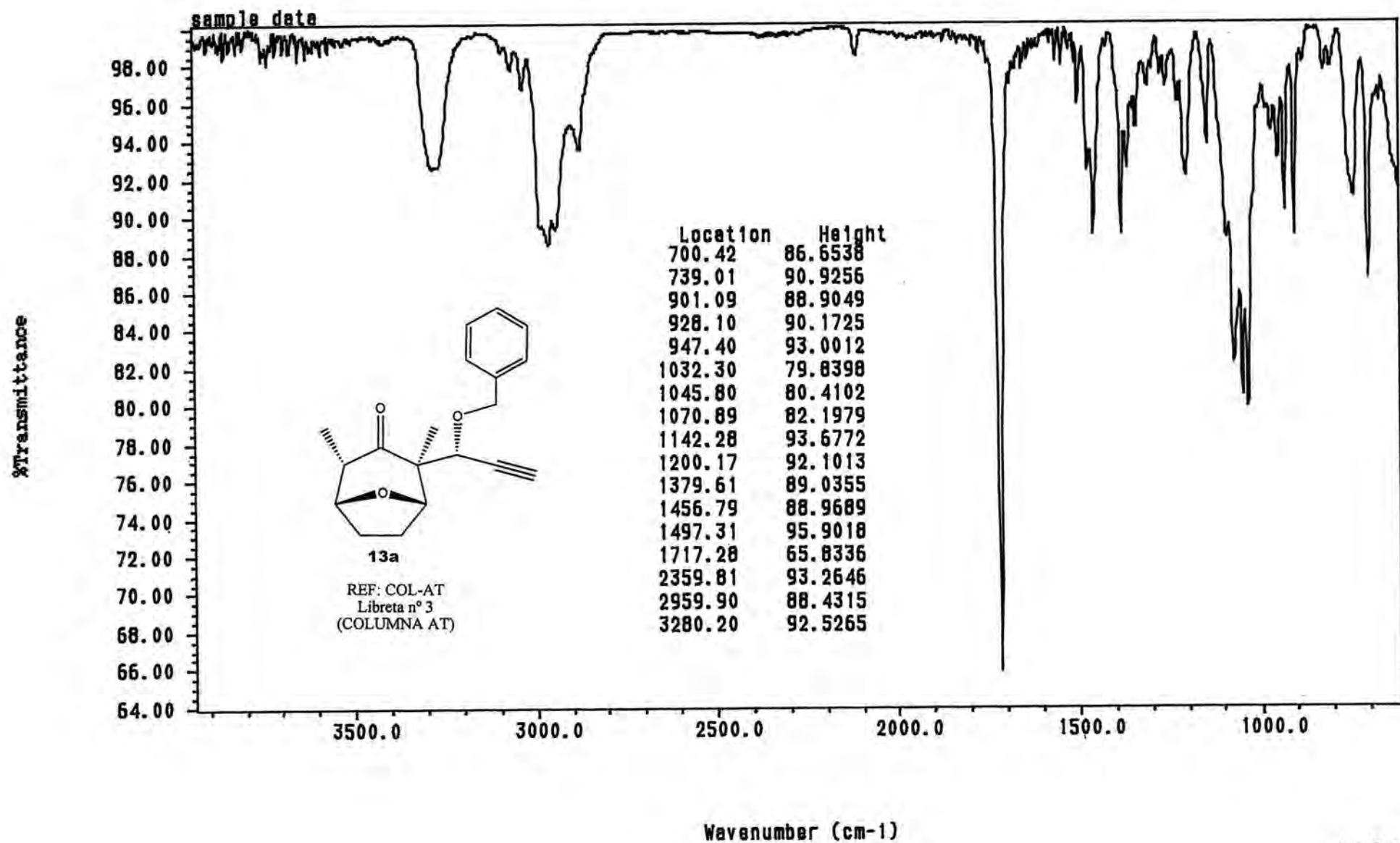


**13a**

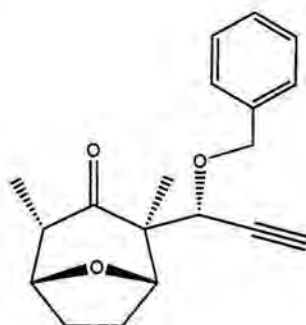
REF: COL-AT  
Libreta nº 3  
(COLUMN A T)



colAT\_5  
sample data



Nic/IR

**13a**

REF: COL-AT  
Libreta n° 3  
(COLUMN A T)

DEPCI4022#1-10 RT: 0.04-0.13 AV: 10

T: + c Full ms [ 60.00-800.00]

m/z= 60.0-448.1

Relative Abundance

| m/z   | Intensity  | Relative |
|-------|------------|----------|
| 107.9 | 226295.1   | 1.07     |
| 136.9 | 156478.3   | 0.74     |
| 191.0 | 131015.2   | 0.62     |
| 192.0 | 100037.6   | 0.47     |
| 193.0 | 112651.1   | 0.53     |
| 206.9 | 133175.9   | 0.63     |
| 210.0 | 123974.0   | 0.59     |
| 213.0 | 316831.7   | 1.50     |
| 239.0 | 78534.8    | 0.37     |
| 244.0 | 200264.7   | 0.95     |
| 279.0 | 247874.2   | 1.17     |
| 281.0 | 414556.3   | 1.96     |
| 282.1 | 78586.2    | 0.37     |
| 299.0 | 6526887.1  | 30.91    |
| 300.1 | 1189957.4  | 5.64     |
| 301.1 | 204652.3   | 0.97     |
| 314.0 | 123121.7   | 0.58     |
| 316.0 | 21113011.2 | 100.00   |
| 317.1 | 3658033.7  | 17.33    |
| 318.1 | 549285.5   | 2.60     |

299.0

317.1

318.1

319.2

90.9

107.9

136.9

154.0

206.9

213.0

244.0

255.0

281.0

368.1

406.1

422.1

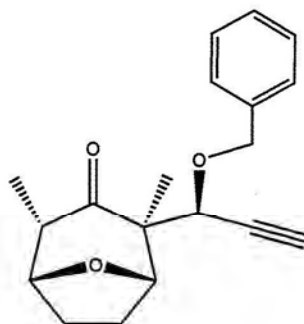
m/z

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

H1 / s2pu1 / Mercury-400\_gui  
cdcl3/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

FULL spectrum

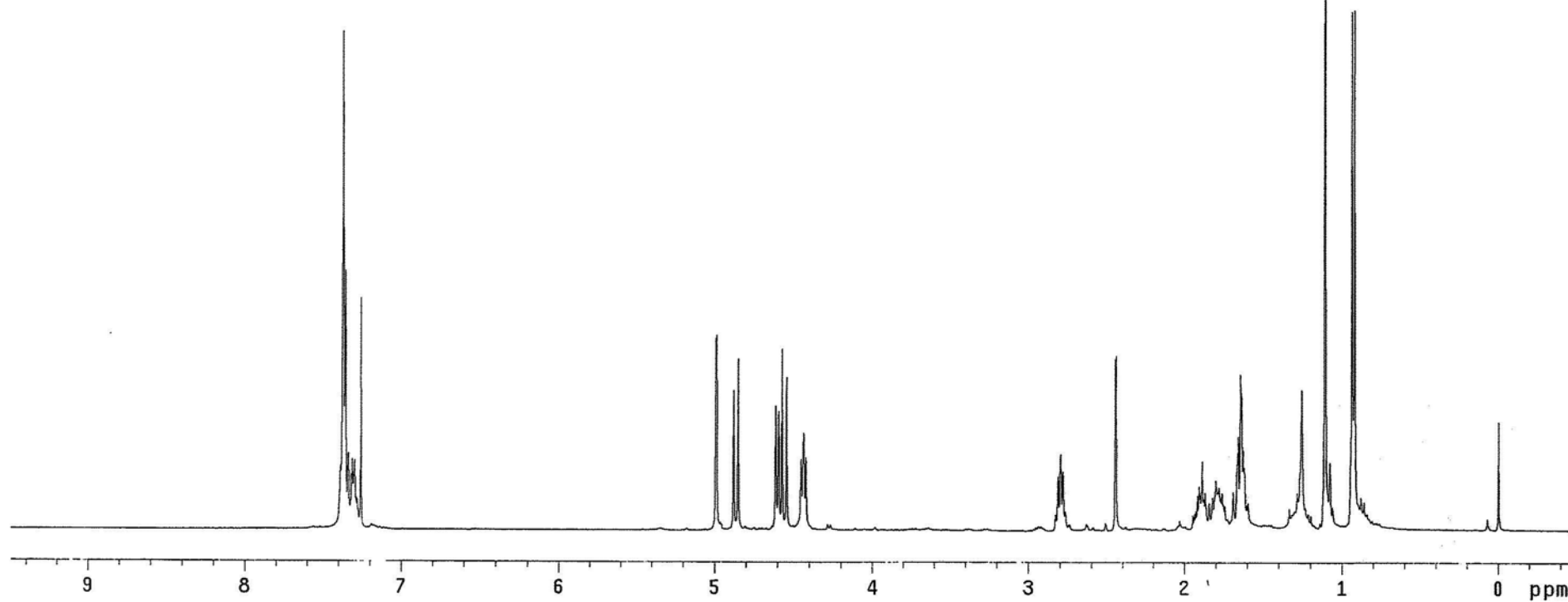
Pulse Sequence: s2pu1  
\*\*\*\*\*



**13b**

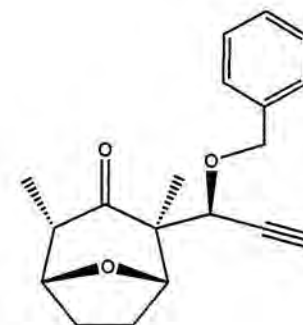
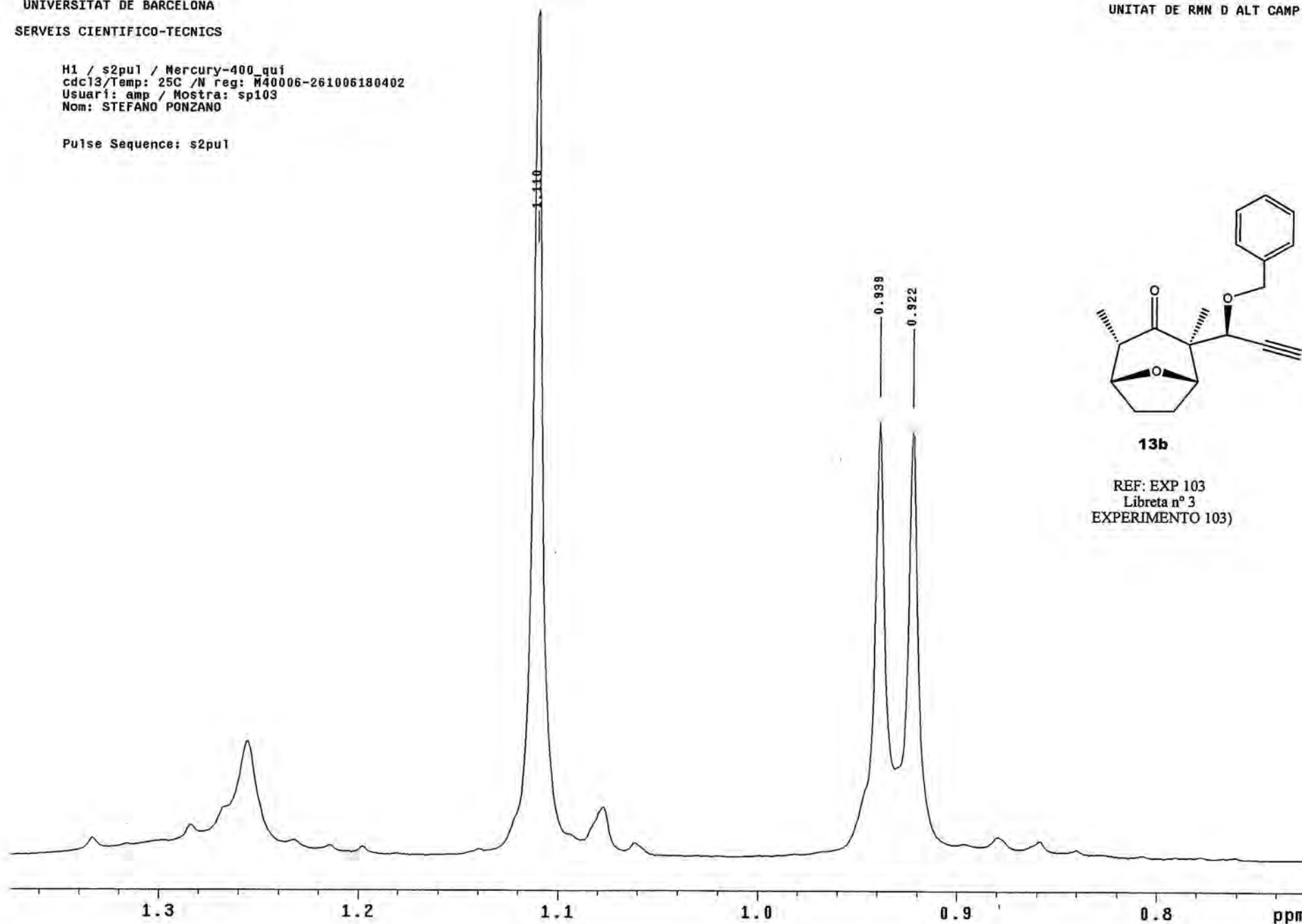
REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)

| INDEX | FREQUENCY | PPM   | HEIGHT |
|-------|-----------|-------|--------|
| 1     | 2950.928  | 7.375 | 79.5   |
| 2     | 2944.580  | 7.359 | 41.2   |
| 3     | 2904.785  | 7.260 | 36.8   |
| 4     | 1996.582  | 4.990 | 31.1   |
| 5     | 444.336   | 1.111 | 162.8  |
| 6     | 375.732   | 0.939 | 82.7   |
| 7     | 368.896   | 0.922 | 82.8   |



H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



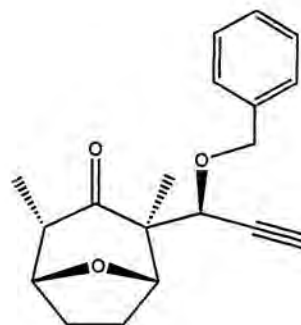
**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)

H1 / s2pu1 / Mercury-400\_qul  
cdc13/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

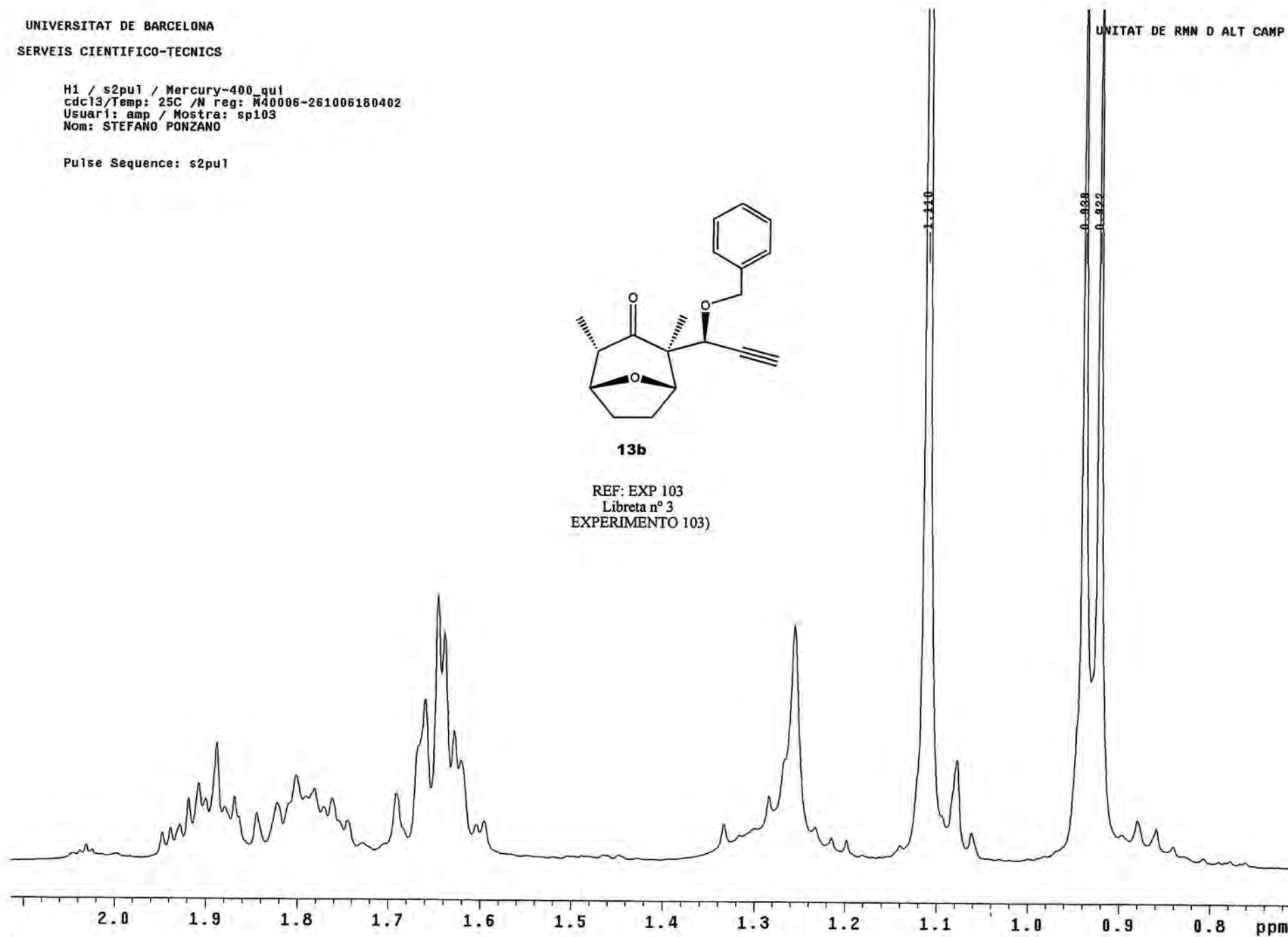
Pulse Sequence: s2pu1

UNITAT DE RMN D ALT CAMP



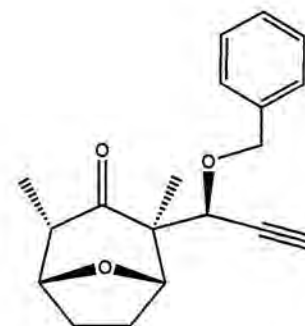
**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)



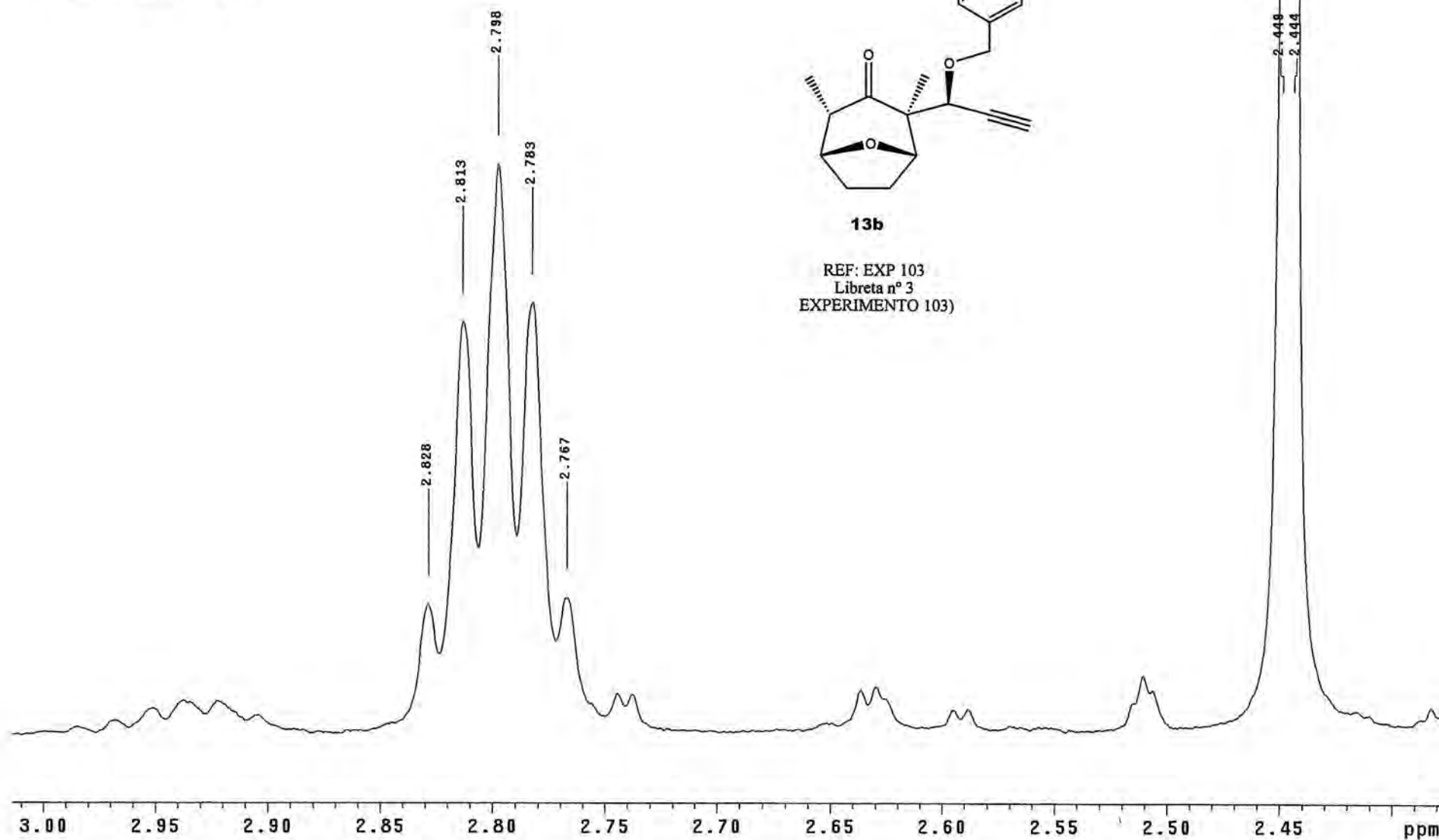
H1 / s2pu1 / Mercury-400\_qul  
cdc13/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



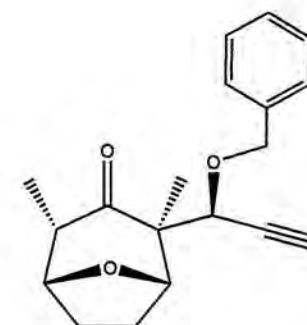
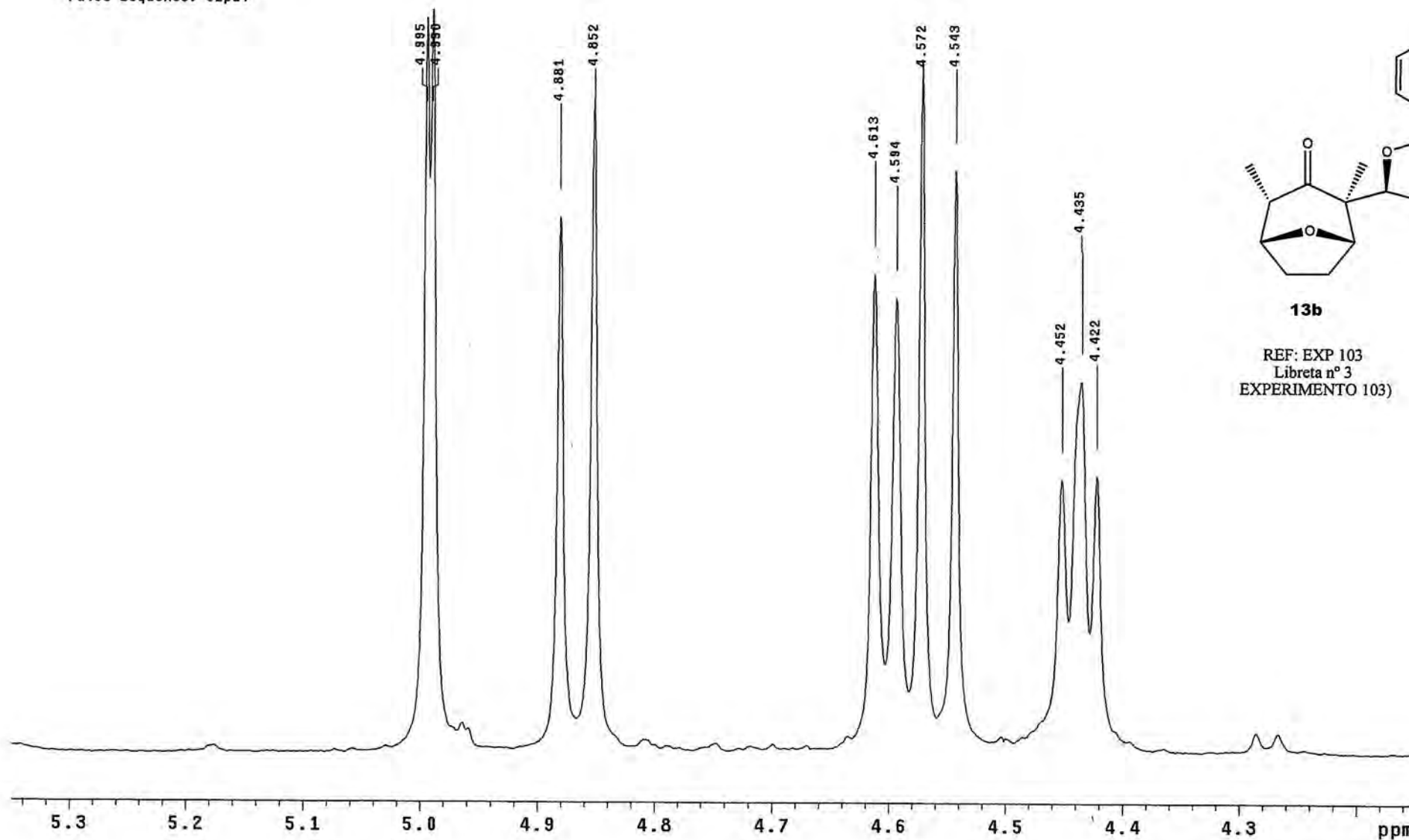
**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)



H1 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

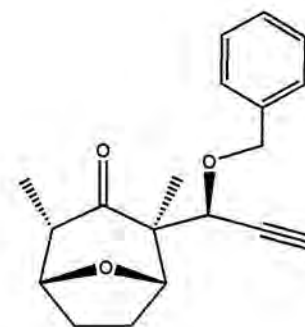
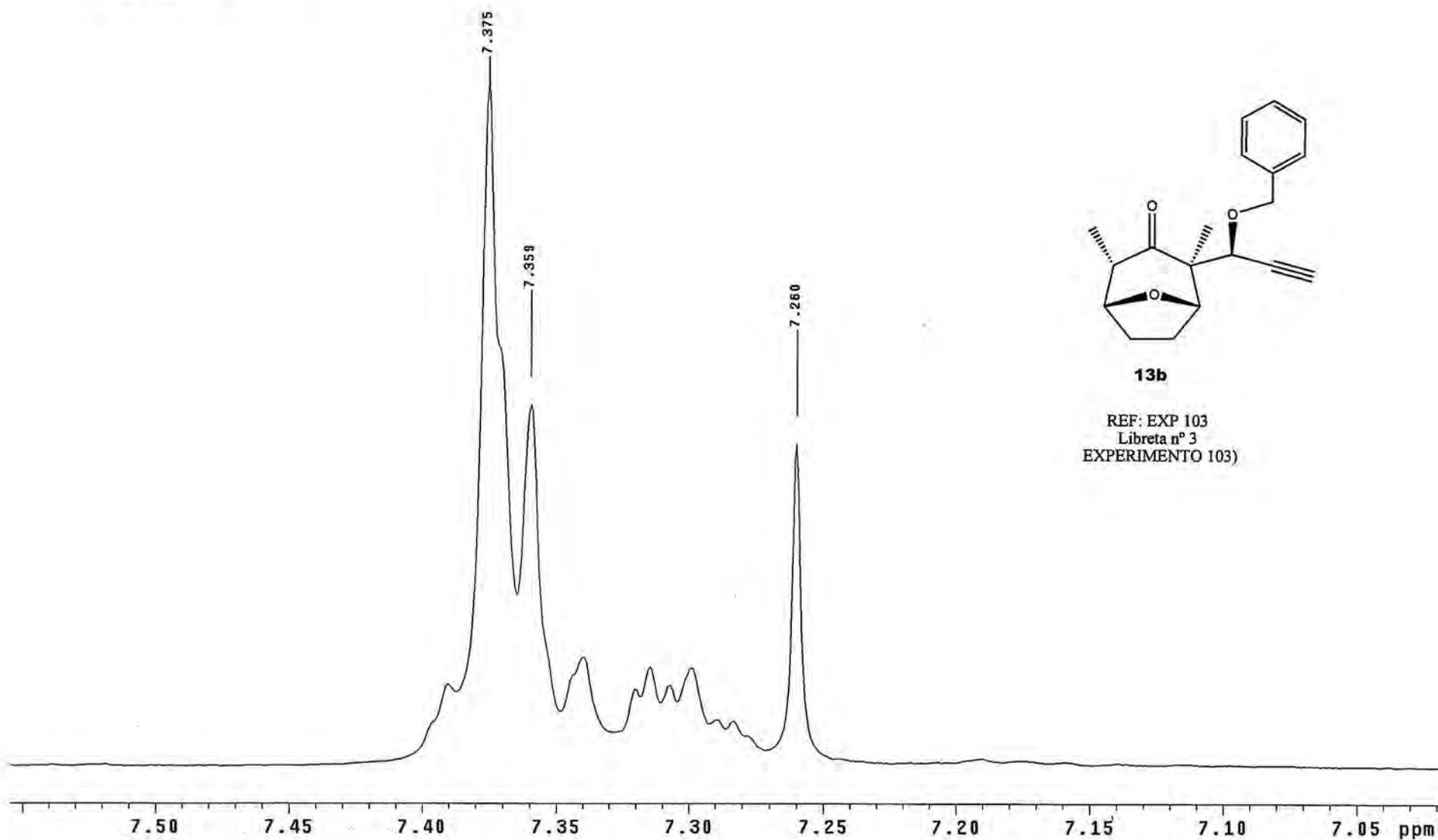


**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)

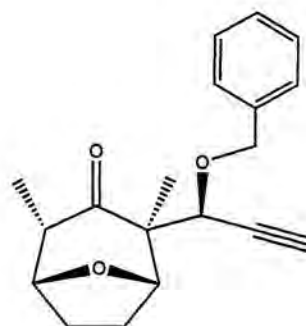
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TÈCNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*

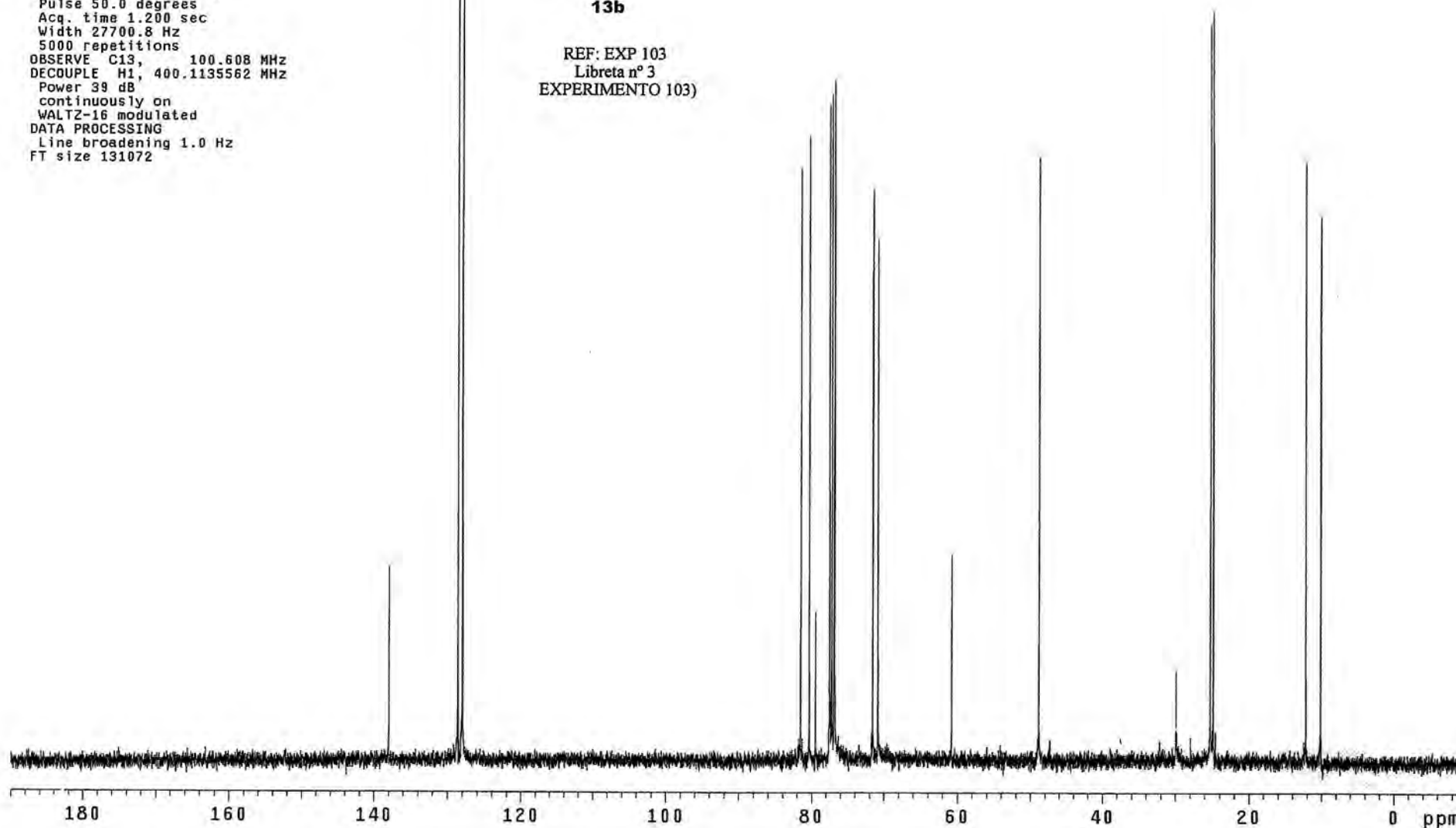
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)



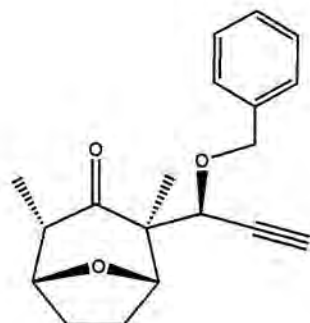
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gCOSY / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*@\*\*\*\*\*

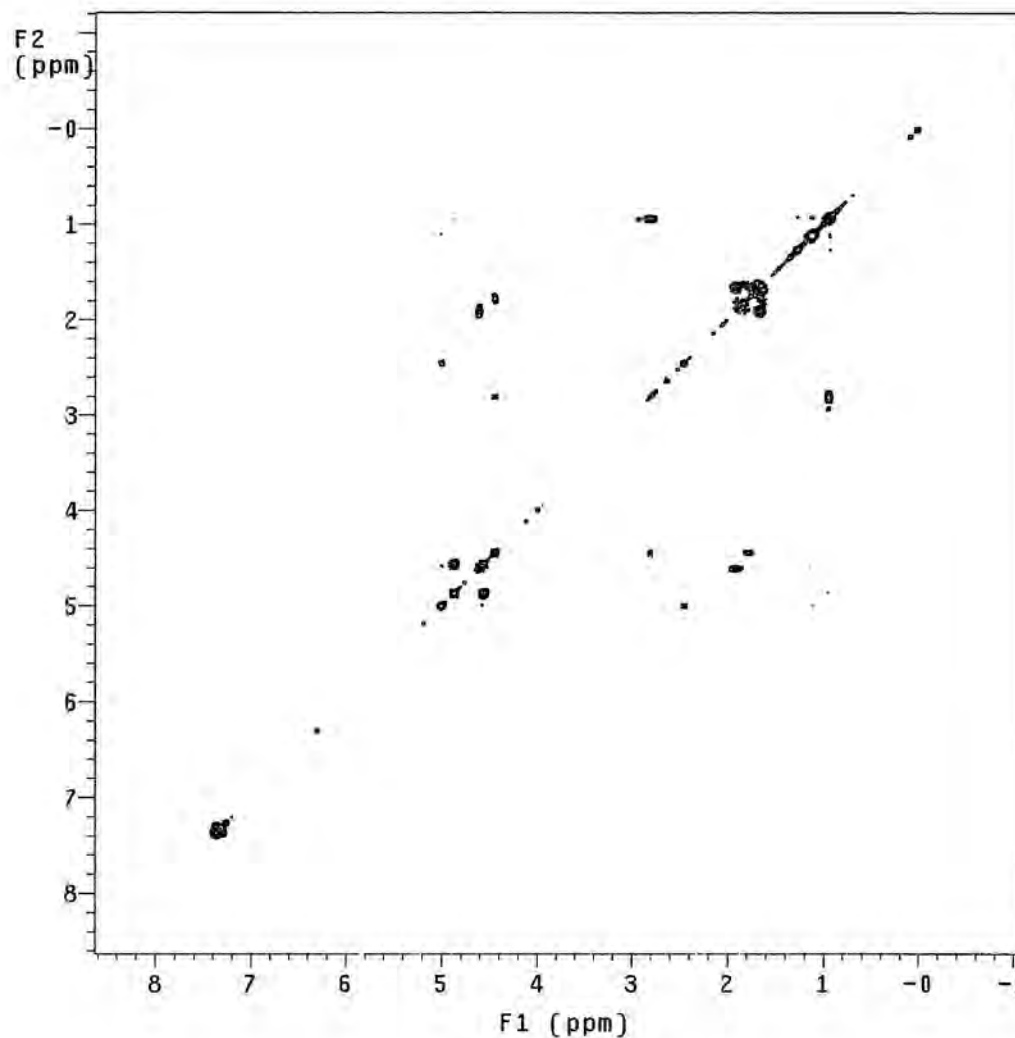
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3937.0 Hz  
2D Width 3937.0 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.065 sec  
FT size 2048 x 2048



**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)



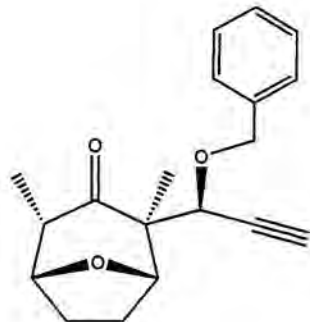
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gHSQC / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-261006180402  
Usuari: amp / Mostra: sp103  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*

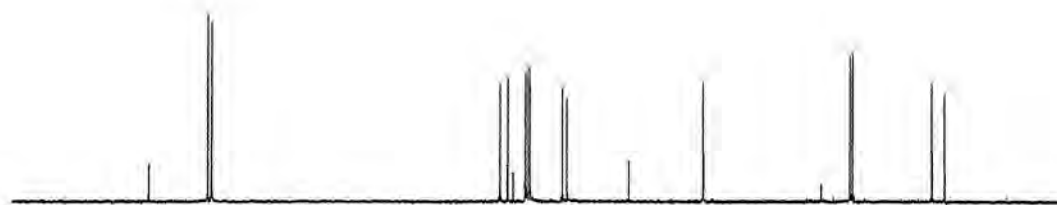
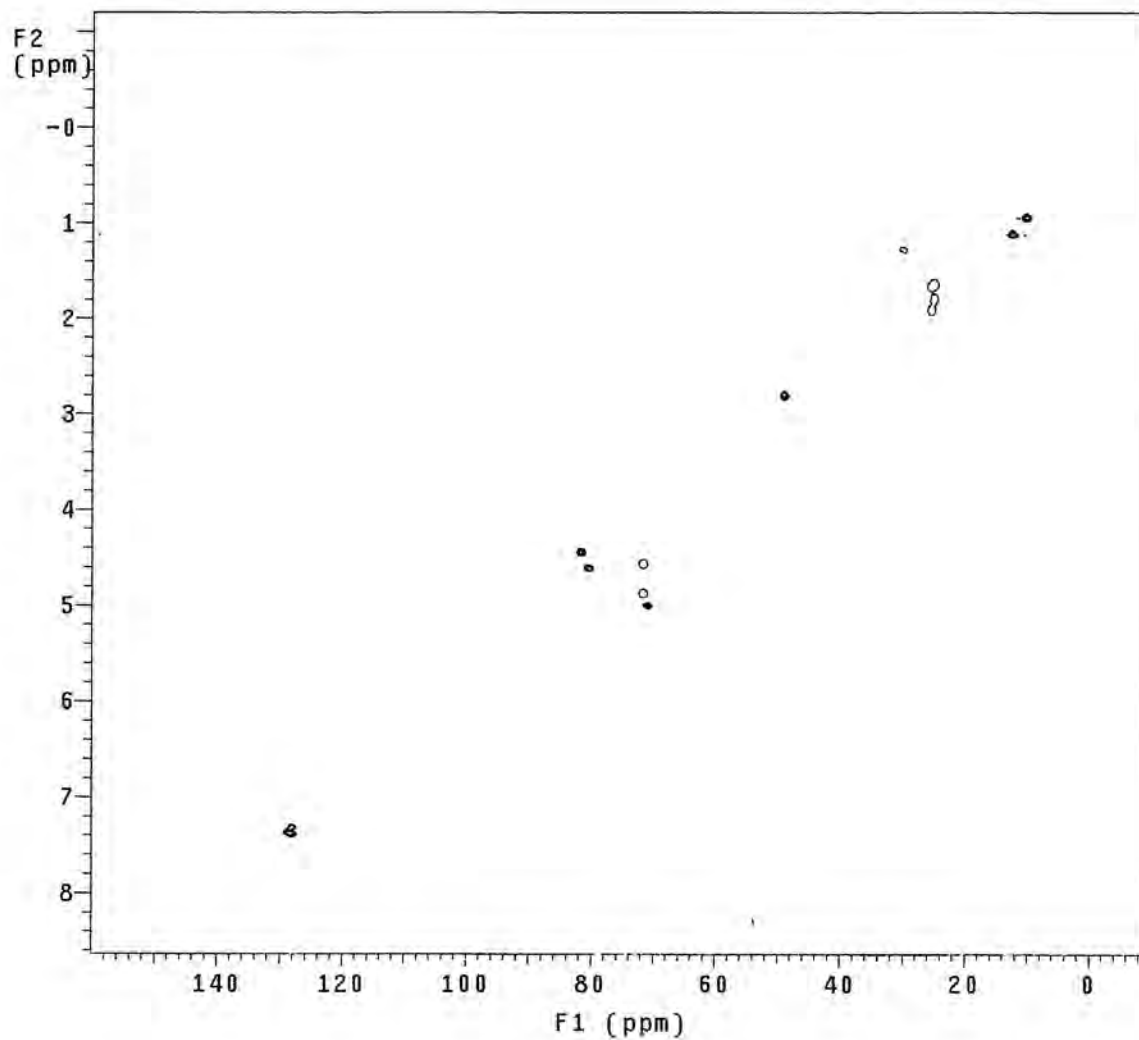
Mercury-400BB "fenix"

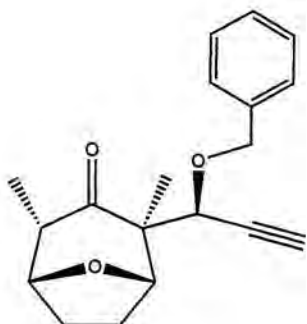
Relax. delay 1.000 sec  
Acq. time 0.130 sec  
Width 3937.0 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048



**13b**

REF: EXP 103  
Libreta nº 3  
EXPERIMENTO 103)

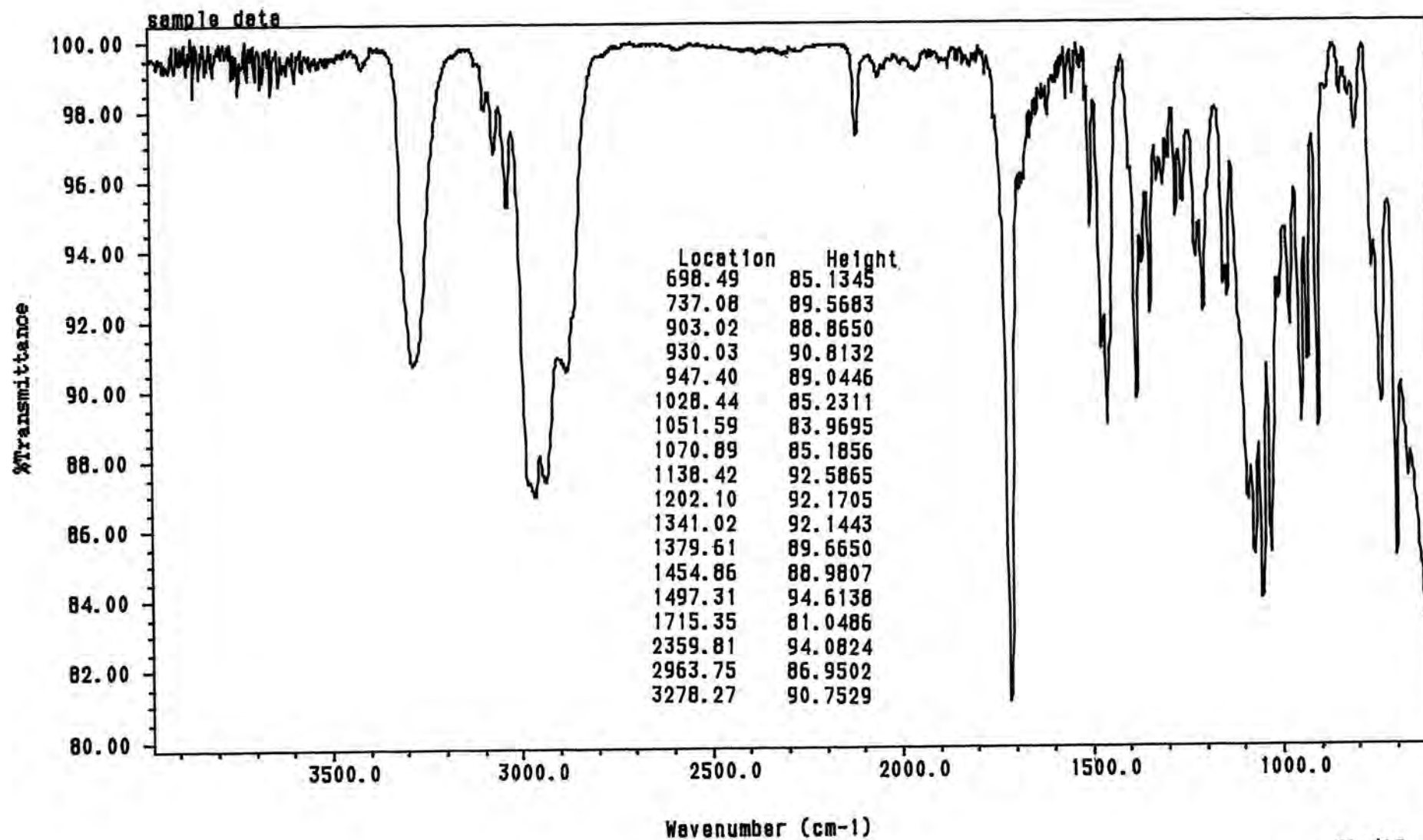


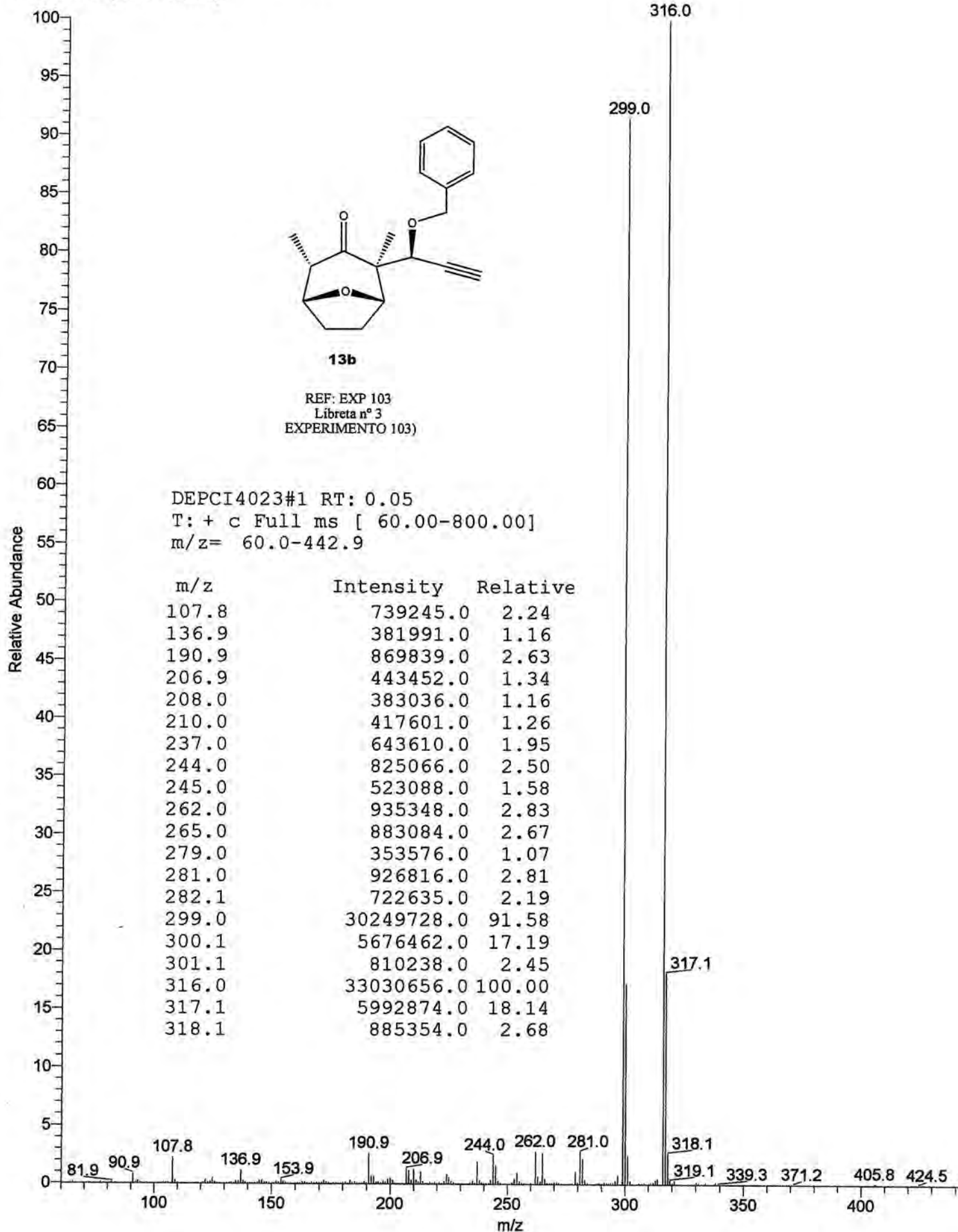


13b

REF: EXP 103  
Libreta n° 3  
EXPERIMENTO 103)

sp103  
sample data

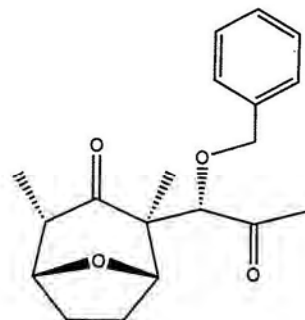




UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TÈCNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N meg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

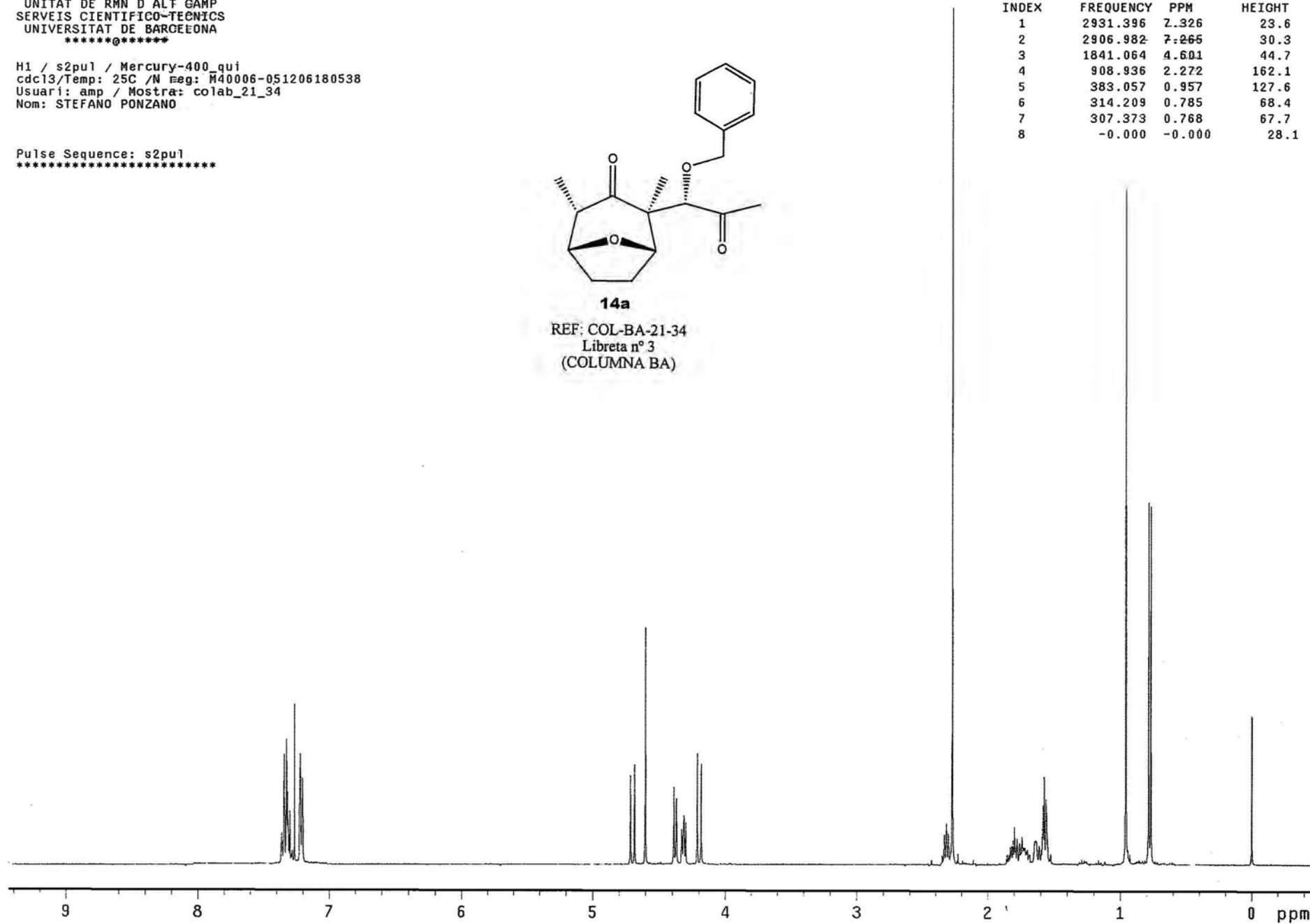
Pulse Sequence: s2pu1  
\*\*\*\*\*



**14a**

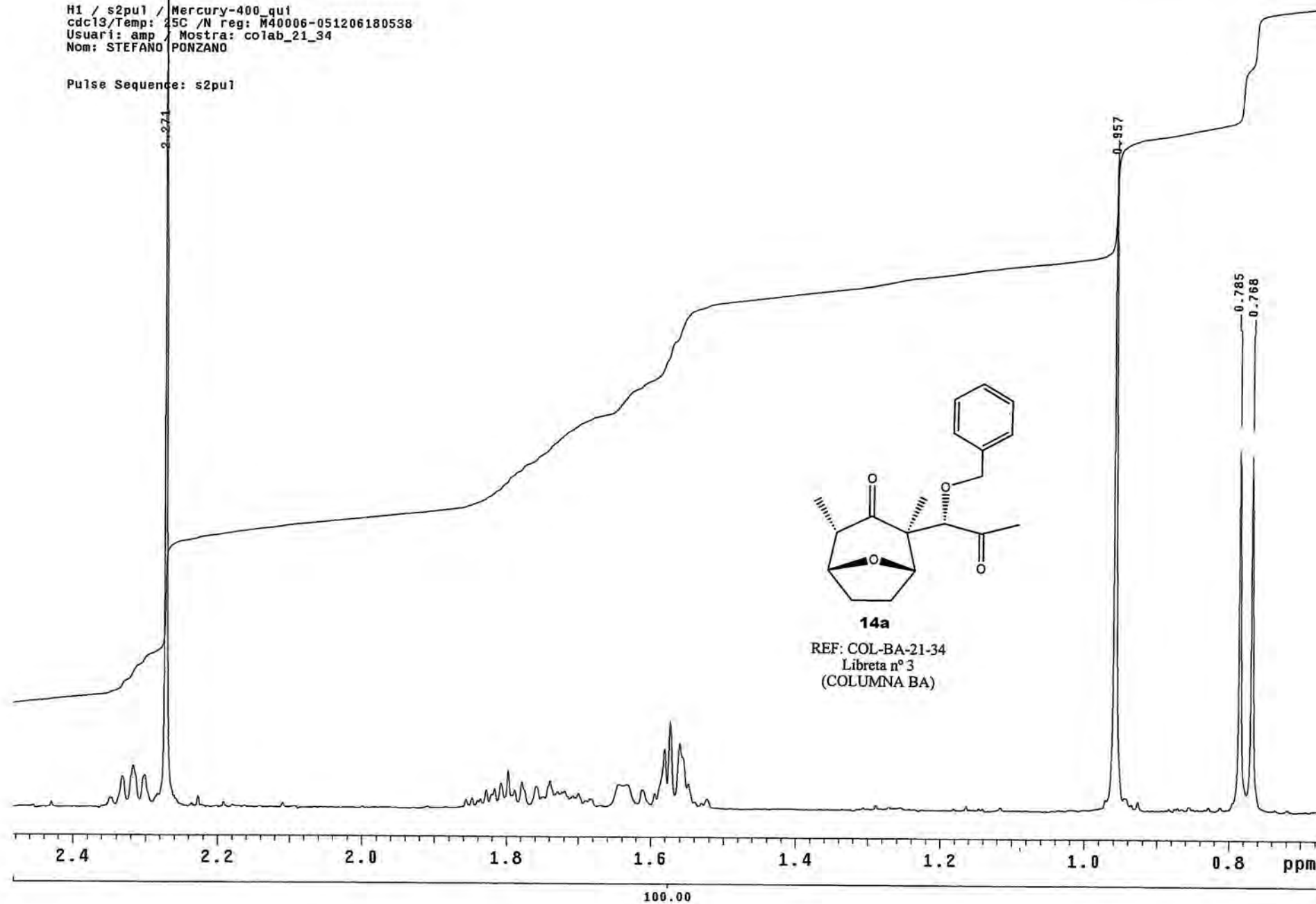
REF: COL-BA-21-34  
Libreta nº 3  
(COLUMNA BA)

| INDEX | FREQUENCY | PPM    | HEIGHT |
|-------|-----------|--------|--------|
| 1     | 2931.396  | 7.326  | 23.6   |
| 2     | 2906.982  | 7.265  | 30.3   |
| 3     | 1841.064  | 4.601  | 44.7   |
| 4     | 908.936   | 2.272  | 162.1  |
| 5     | 383.057   | 0.957  | 127.6  |
| 6     | 314.209   | 0.785  | 68.4   |
| 7     | 307.373   | 0.768  | 67.7   |
| 8     | -0.000    | -0.000 | 28.1   |



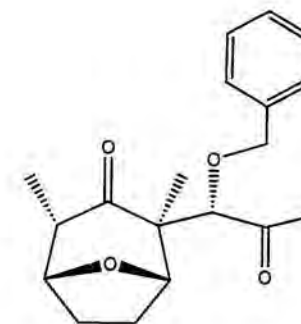
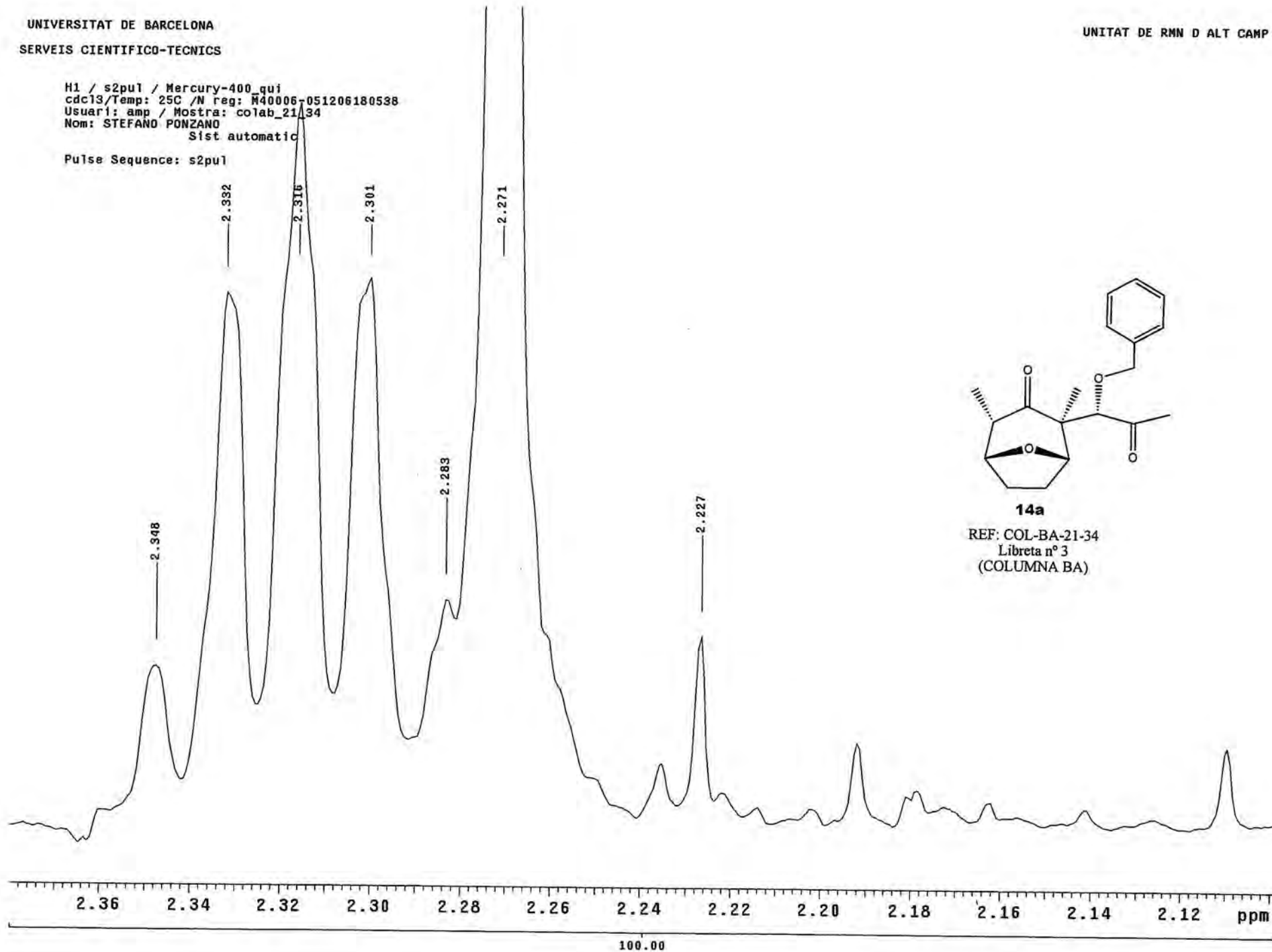
H1 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



H1 / s2pul / Mercury-400 qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21/34  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pul

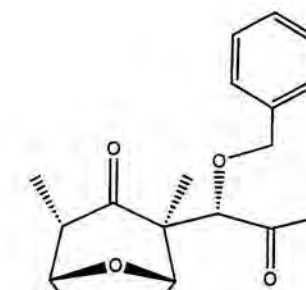
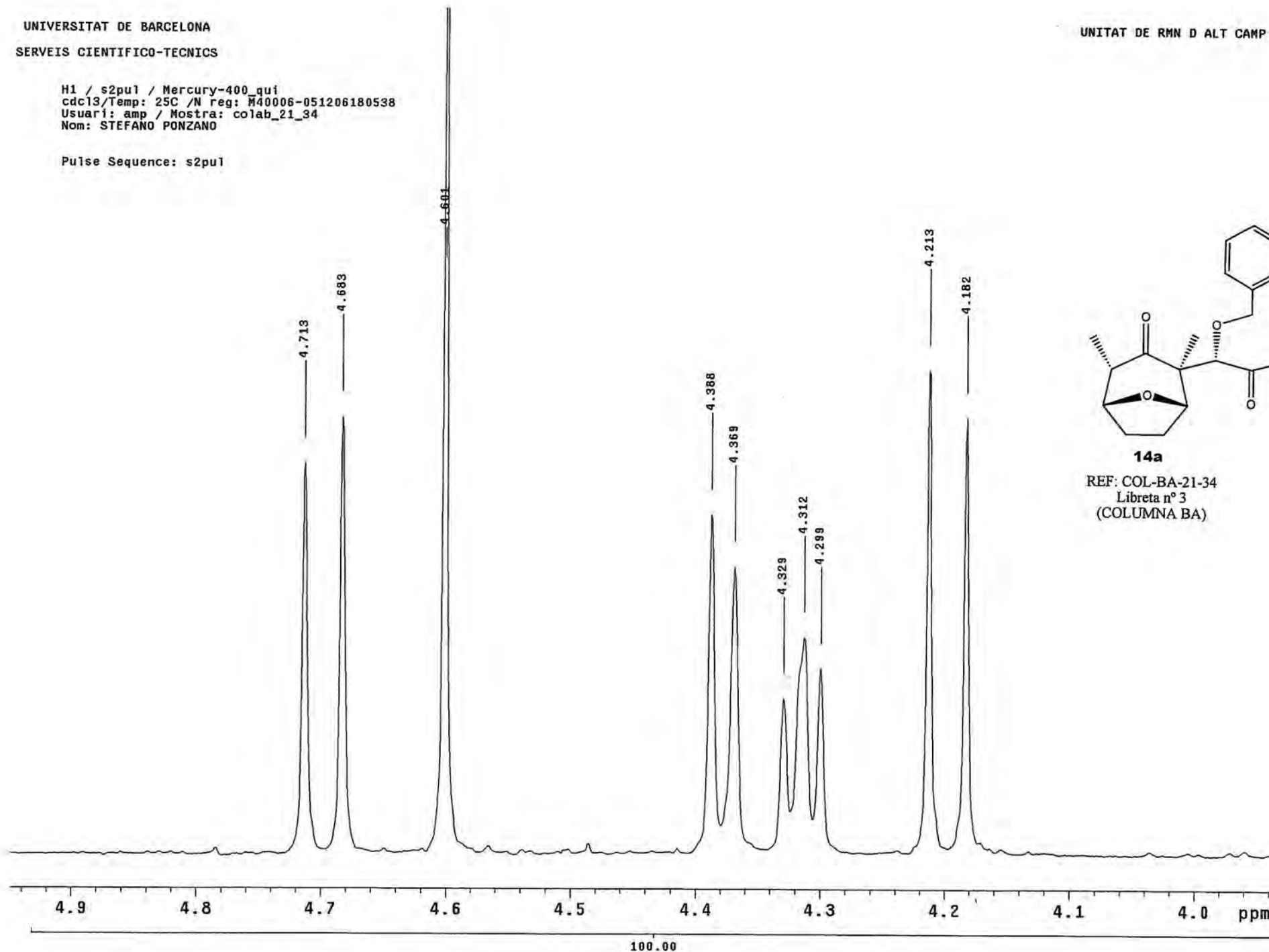


**14a**

REF: COL-BA-21-34  
Libreta nº 3  
(COLUMN BA)

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

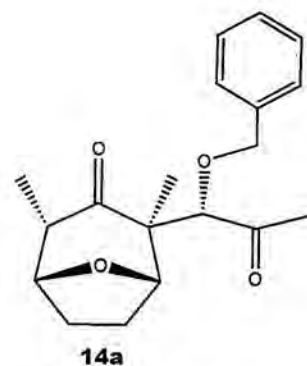
Pulse Sequence: s2pu1

**14a**

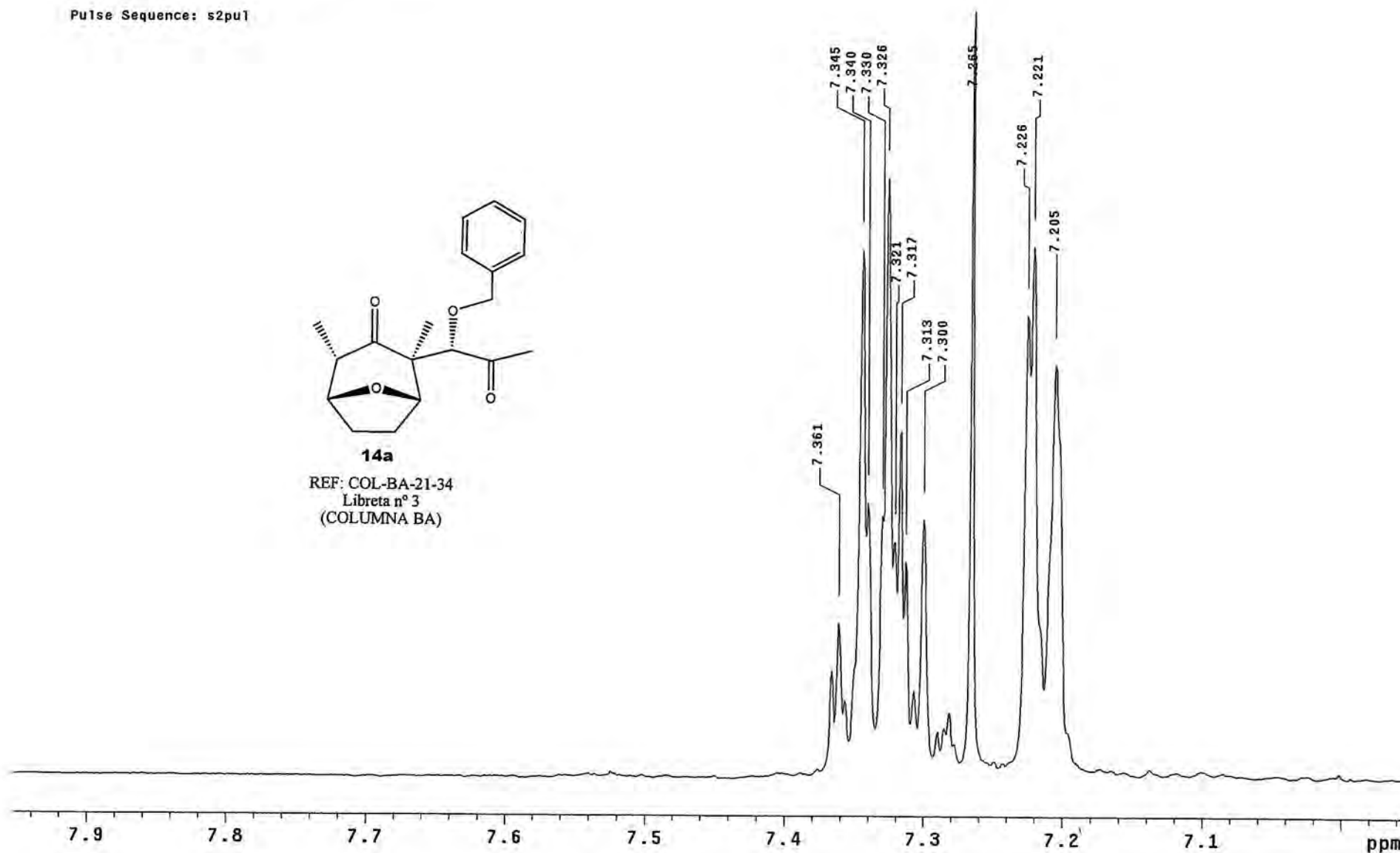
REF: COL-BA-21-34  
Libreta n° 3  
(COLUMN BA)

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



REF: COL-BA-21-34  
Libreta n° 3  
(COLUMNNA BA)



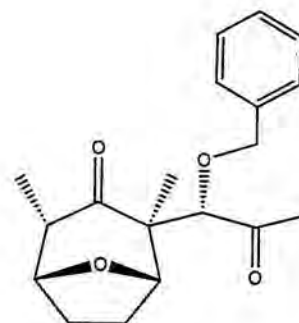
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pu1 / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*

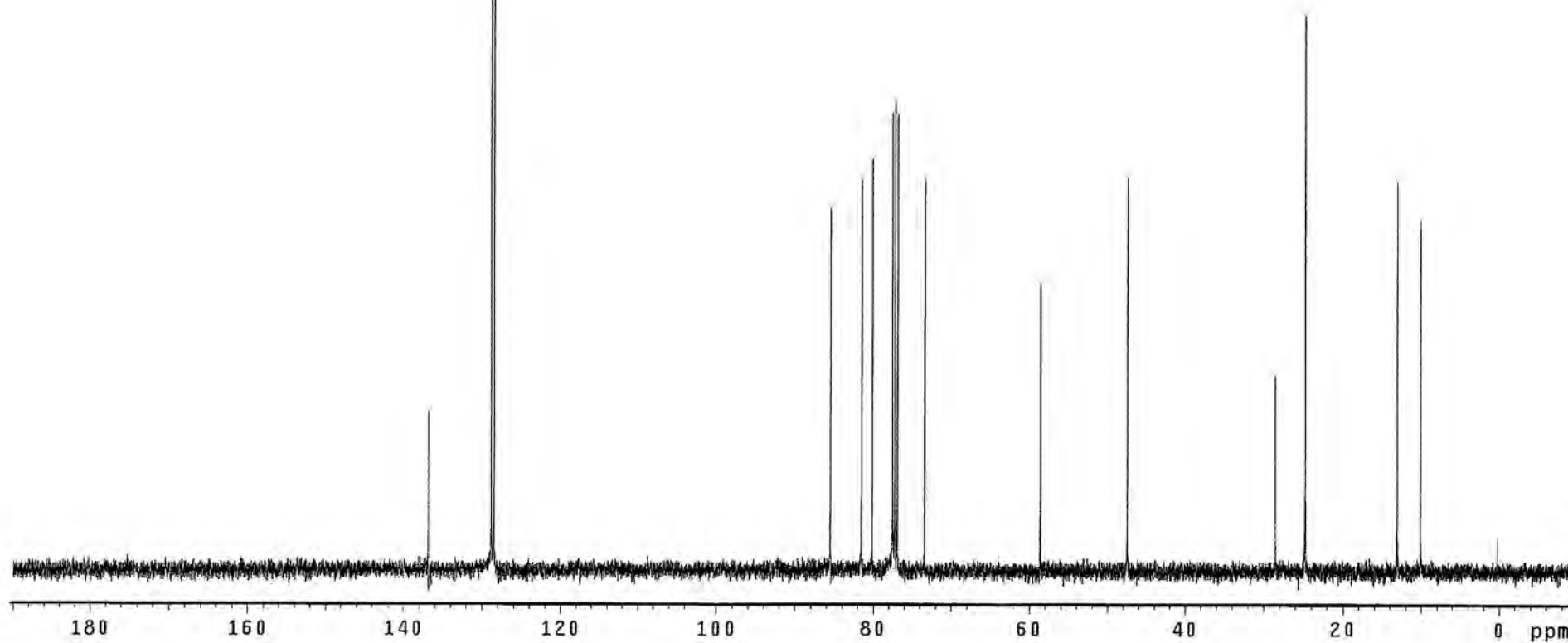
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



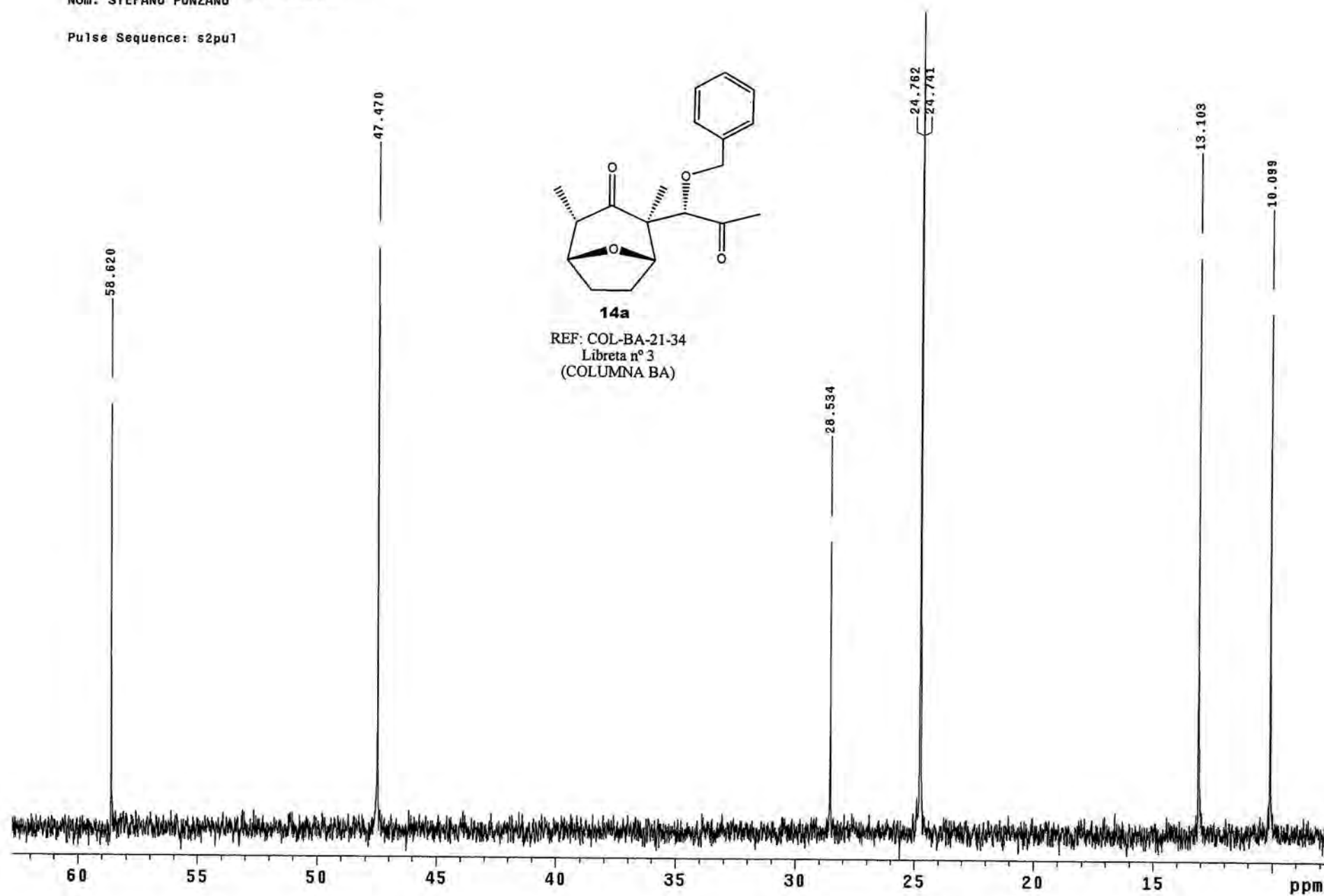
**14a**

REF: COL-BA-21-34  
Libreta nº 3  
(COLUMN BA)



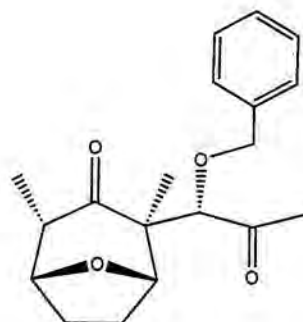
C13 / s2pu1 / Mercury-400\_gu1  
cdc13/Tmp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



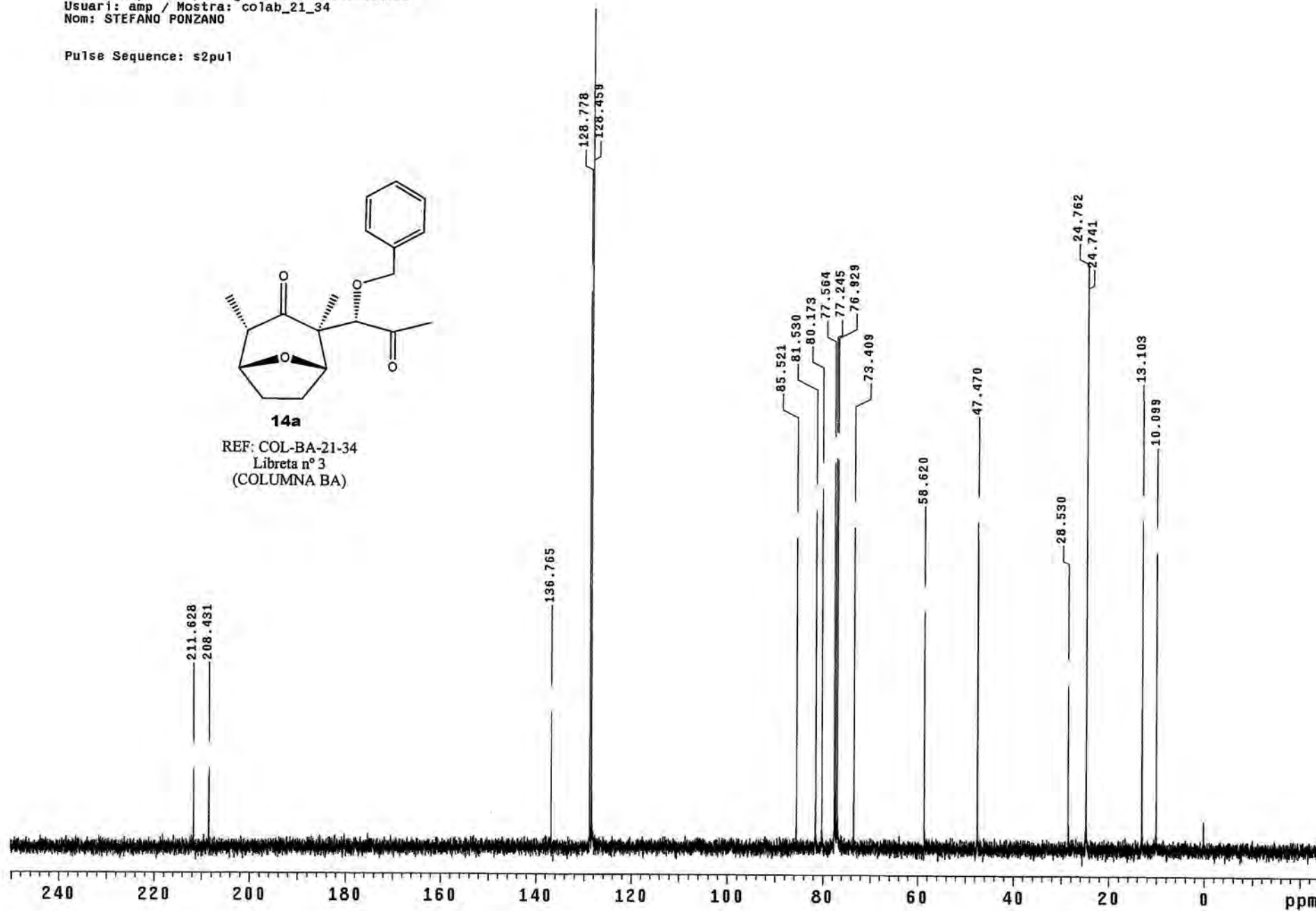
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



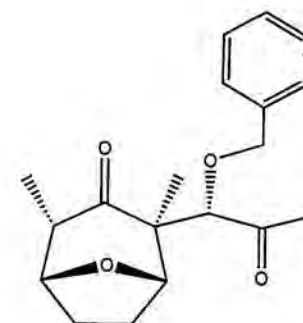
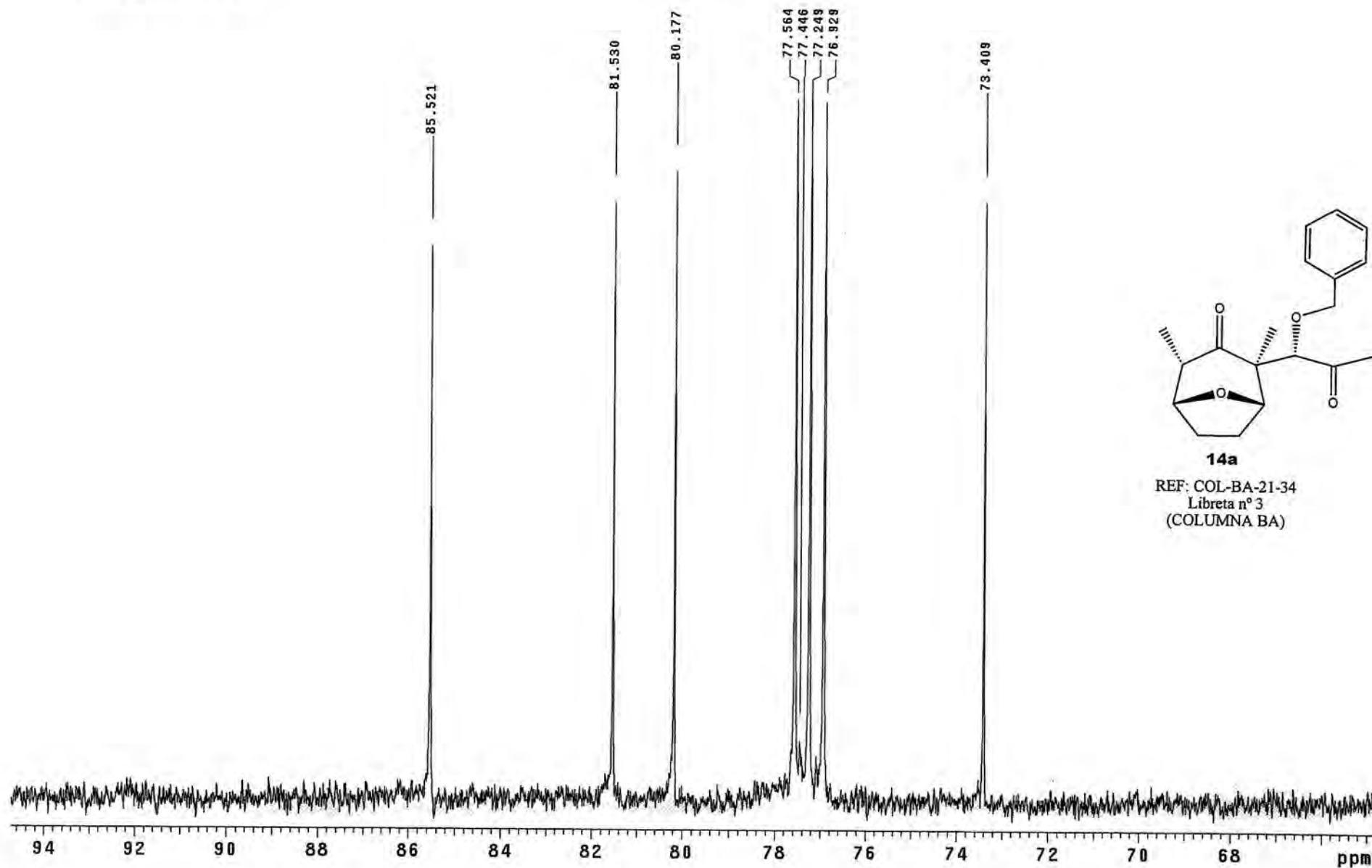
**14a**

REF: COL-BA-21-34  
Libreta n° 3  
(COLUMNBA BA)



C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

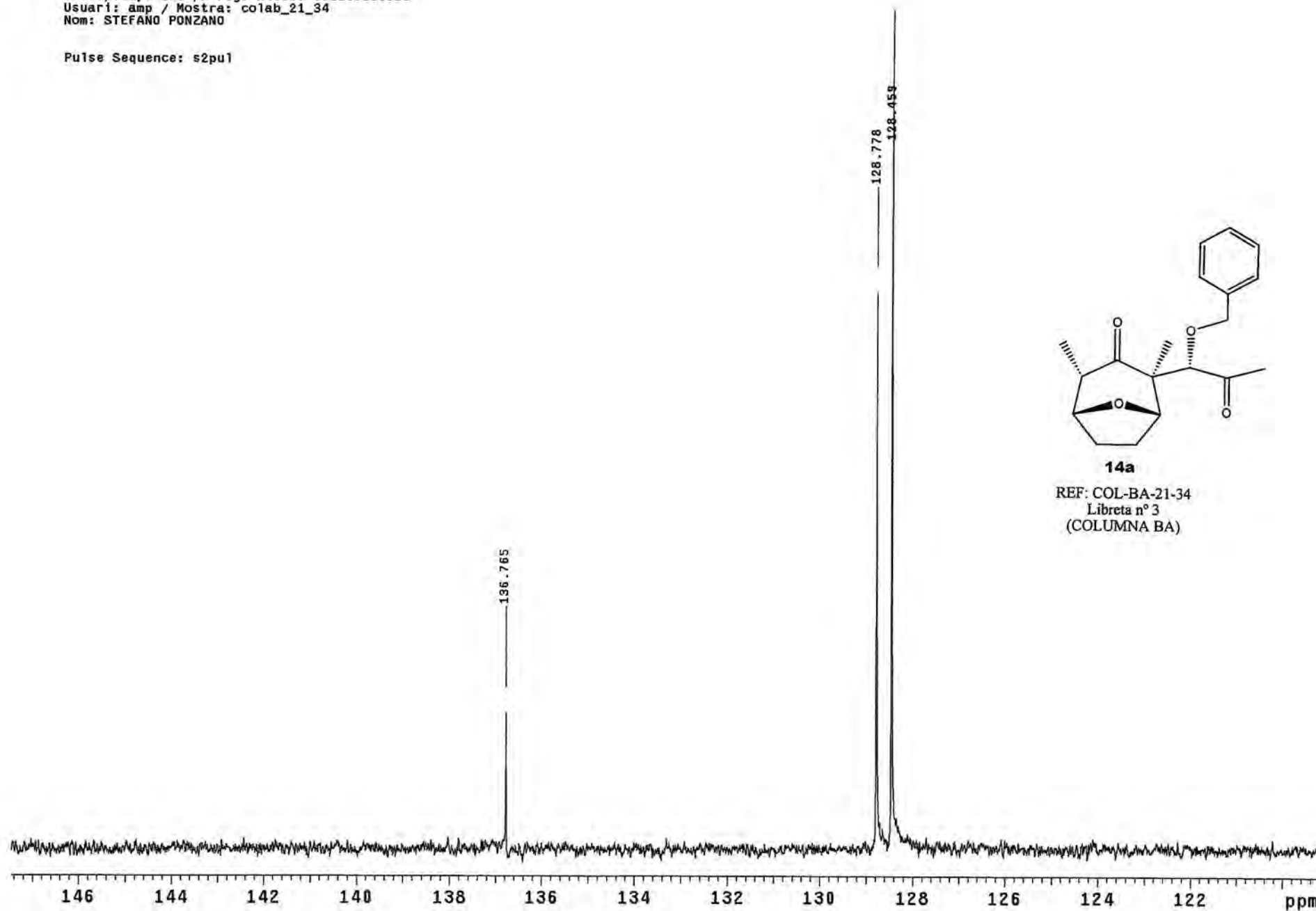


**14a**

REF: COL-BA-21-34  
Libreta n° 3  
(COLUMN BA)

C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



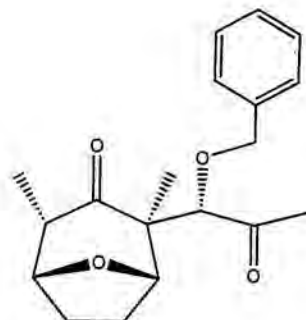
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFIC-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gCOSY / Mercury-400\_qui  
cdc13/Temp: 25C /N. reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*

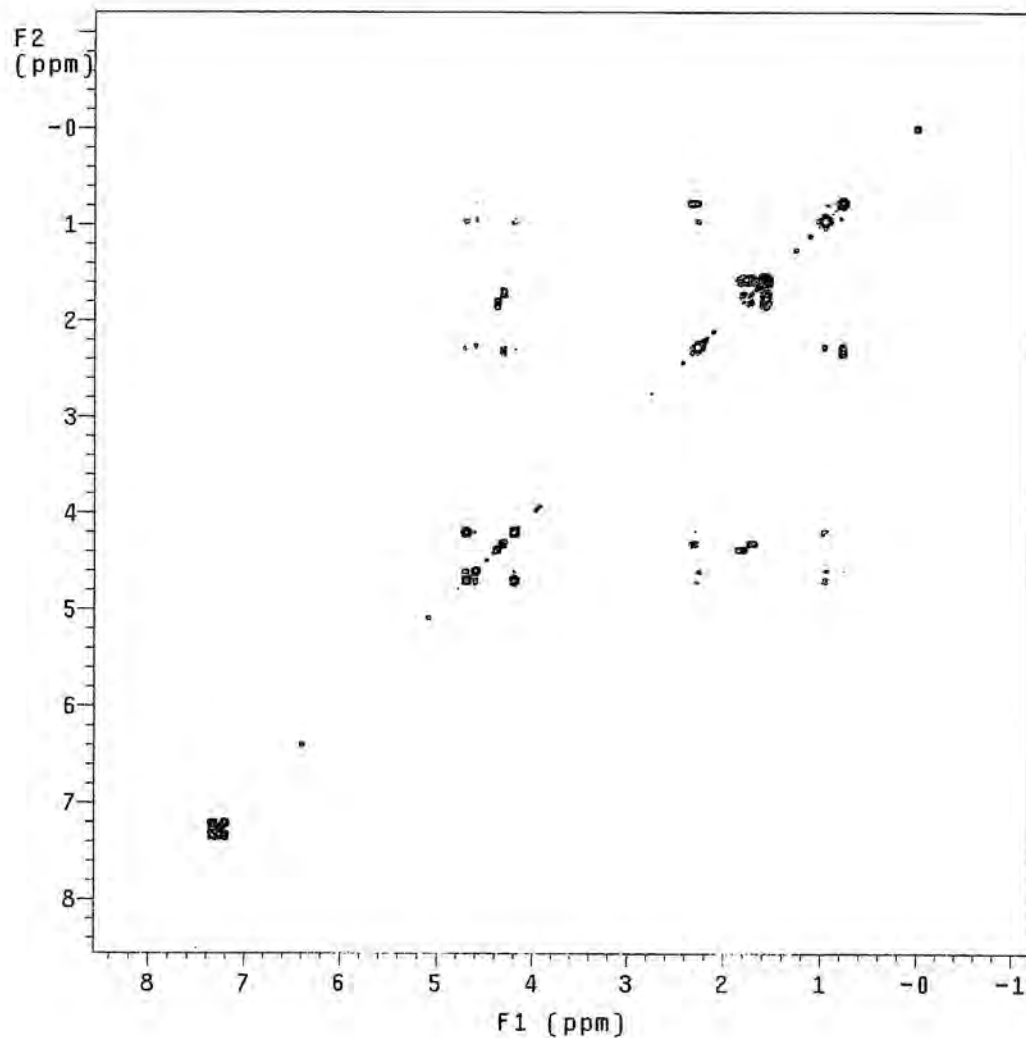
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3906.2 Hz  
2D Width 3906.2 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.066 sec  
FT size 2048 x 2048



**14a**

REF: COL-BA-21-34  
Libreta nº 3  
(COLUMN BA)



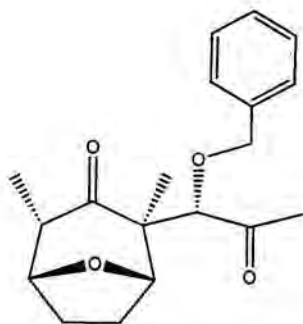
UNITAT DE RMN D'ALT-CAMP  
SERVEIS CIENTÍFICO-TÈCNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*g\*\*\*\*\*

H1 / gHSQC / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: colab\_21\_34  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*g\*\*\*\*\*

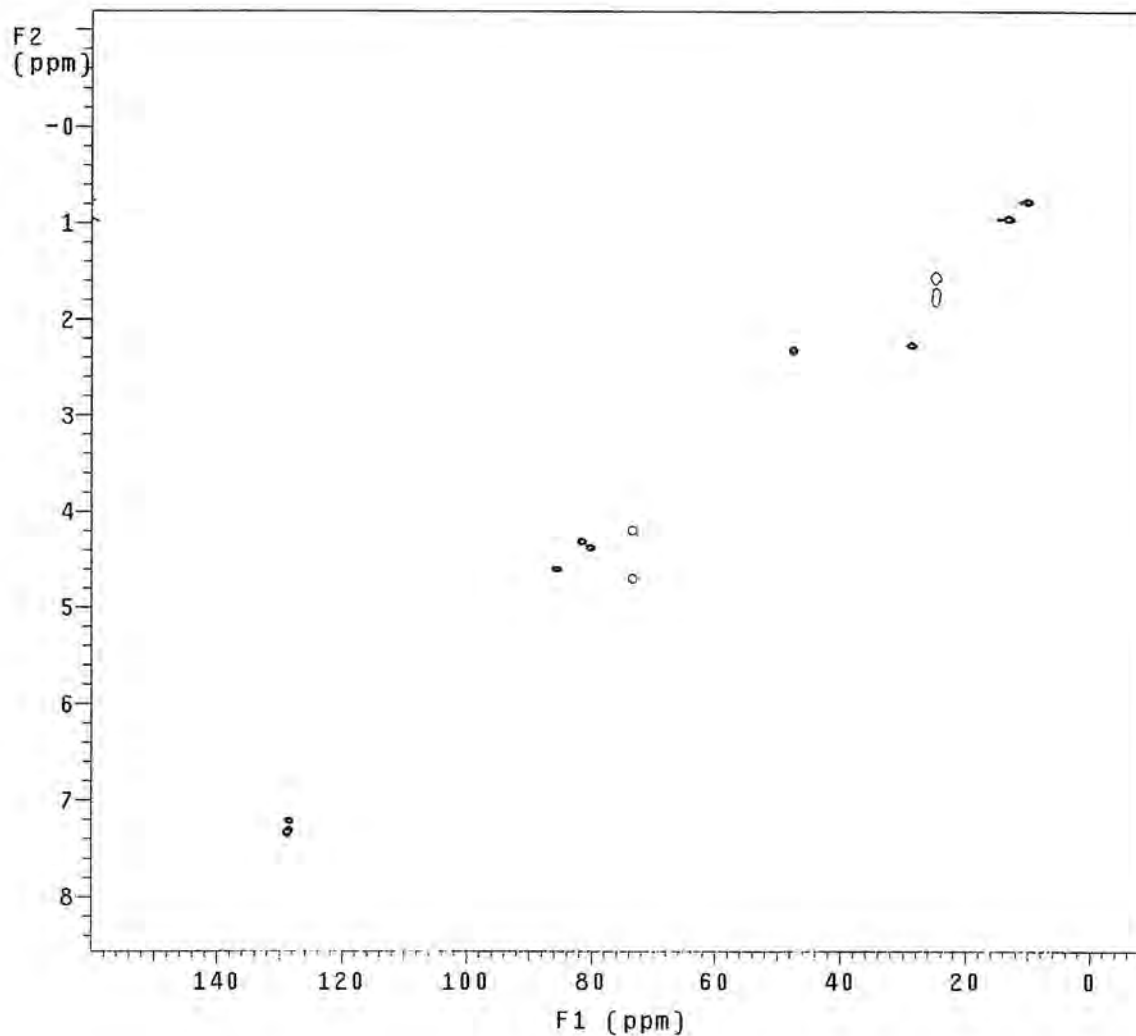
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.131 sec  
Width 3906.2 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048

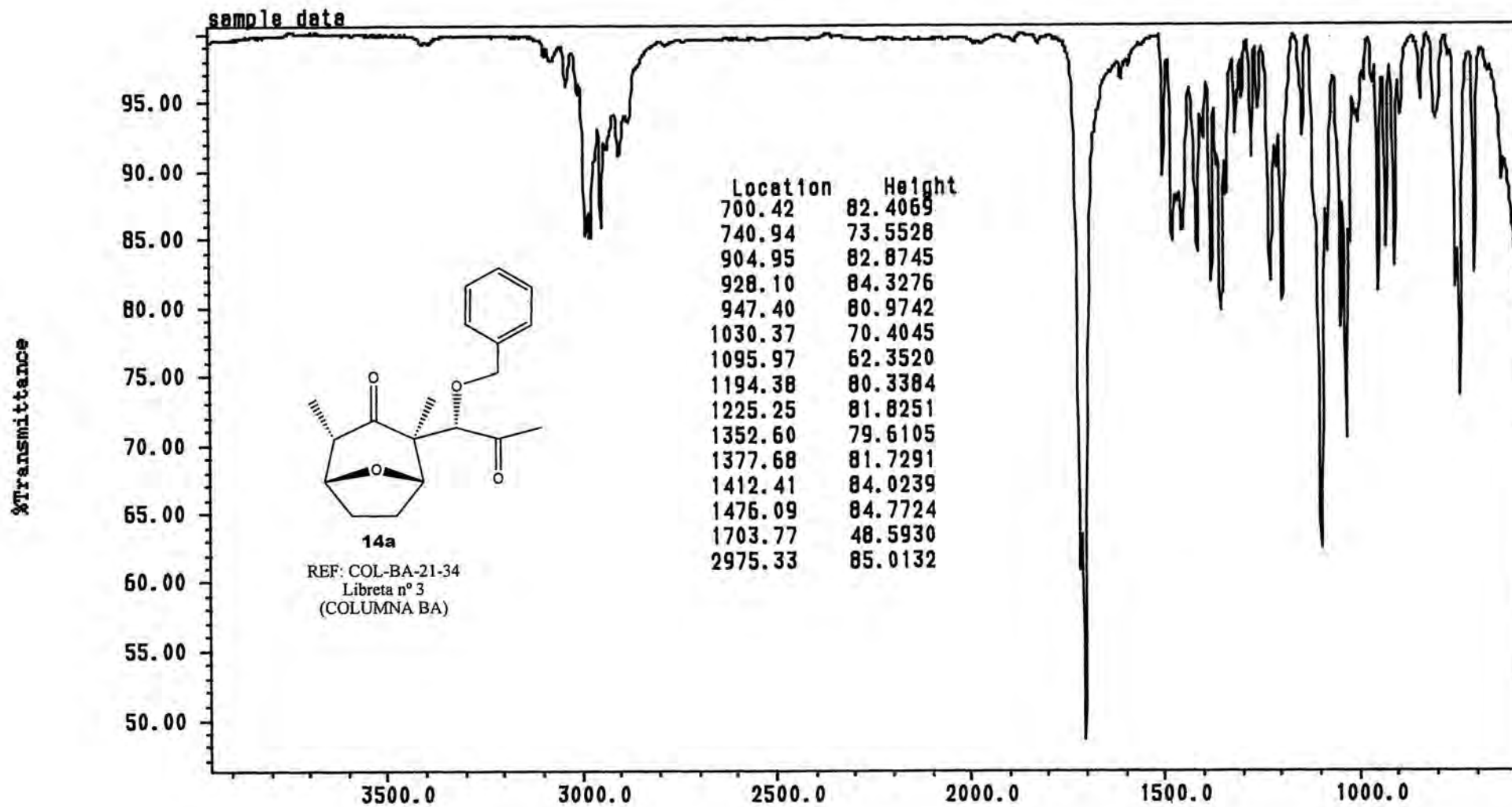


**14a**

REF: COL-BA-21-34  
Libreta nº 3  
(COLUMN BA)

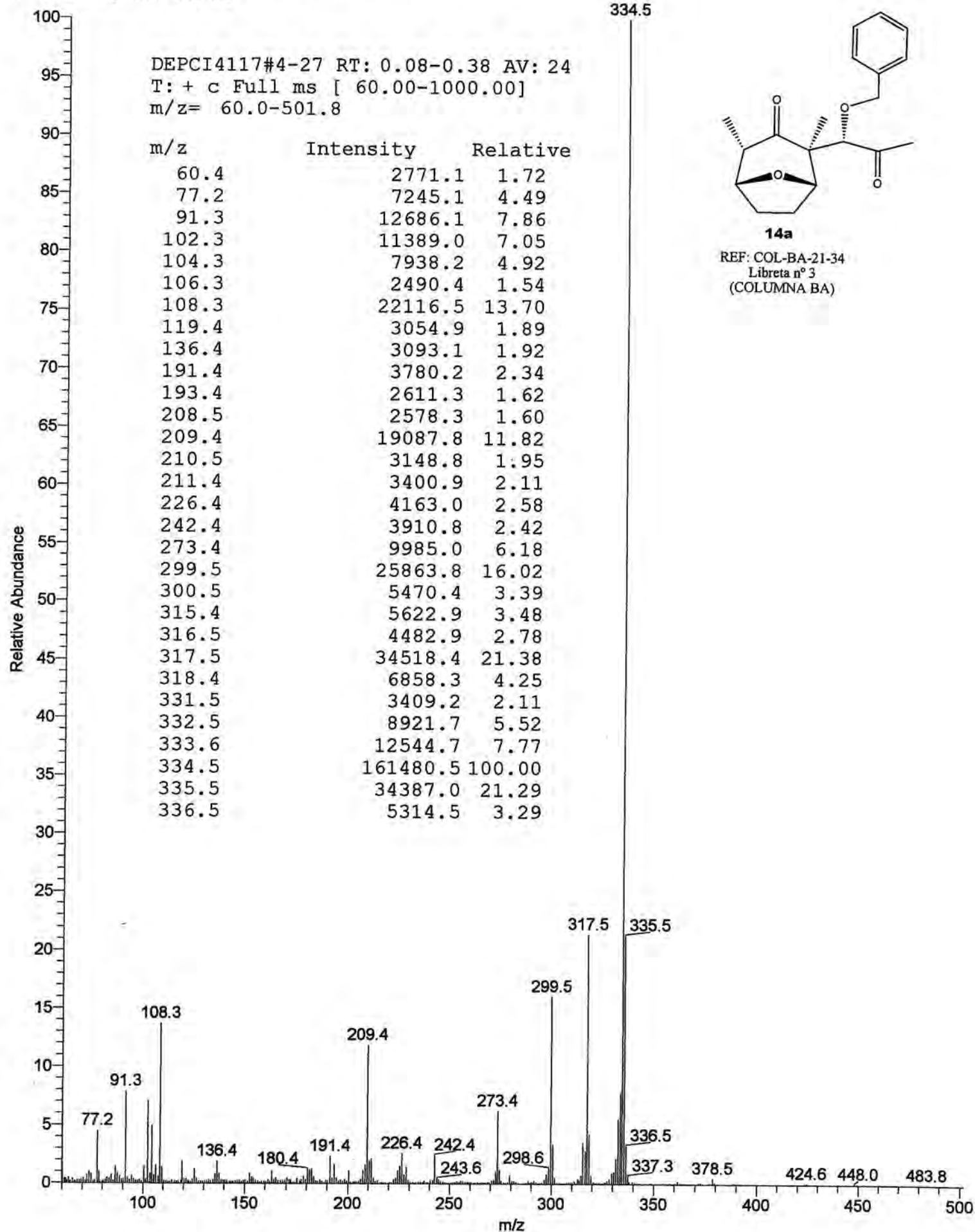


colBA21\_34  
sample data



Wavenumber (cm-1)

N1c/IR



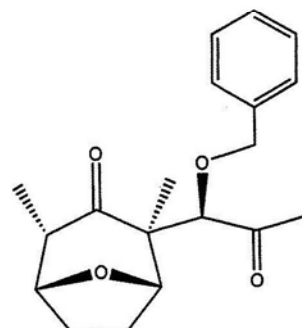
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / s2pul / Mercury-400\_gui  
cdcl3/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pul  
\*\*\*\*\*

Mercury-400BB "fenix"

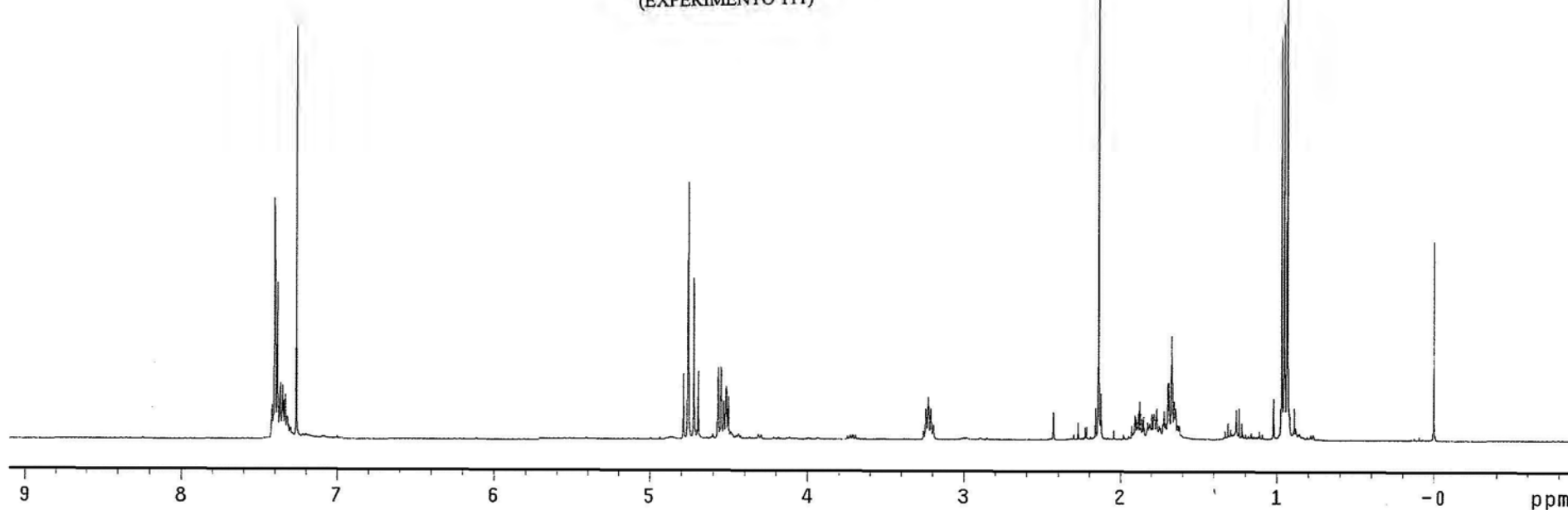
Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.498 sec  
Width 4180.6 Hz  
8 repetitions  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 32768



**14b**

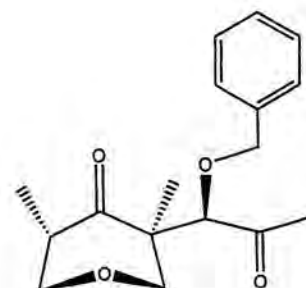
REF: EXP 111  
Libreta n° 3  
(EXPERIMENTO 111)

| INDEX | FREQUENCY | PPM    | HEIGHT |
|-------|-----------|--------|--------|
| 1     | 2962.195  | 7.403  | 38.1   |
| 2     | 2955.051  | 7.386  | 24.7   |
| 3     | 2905.804  | 7.262  | 65.4   |
| 4     | 1904.032  | 4.759  | 28.3   |
| 5     | 1902.245  | 4.754  | 40.8   |
| 6     | 1888.977  | 4.721  | 25.6   |
| 7     | 1827.482  | 4.567  | 11.5   |
| 8     | 1819.827  | 4.548  | 11.6   |
| 9     | 856.585   | 2.141  | 151.2  |
| 10    | 666.998   | 1.667  | 16.6   |
| 11    | 387.849   | 0.969  | 63.7   |
| 12    | 381.215   | 0.953  | 66.3   |
| 13    | 373.815   | 0.934  | 126.7  |
| 14    | -0.000    | -0.000 | 31.6   |



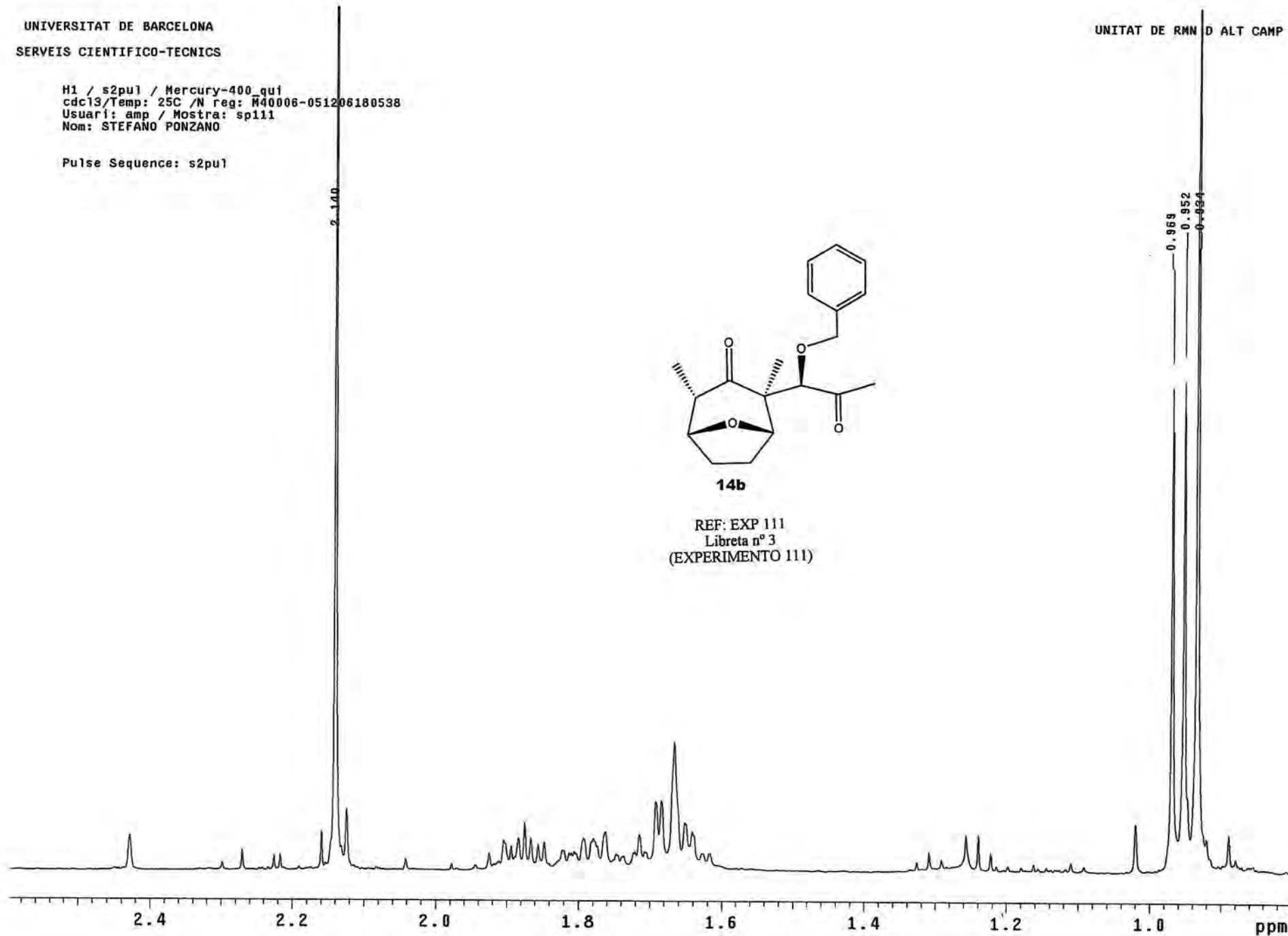
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: N40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



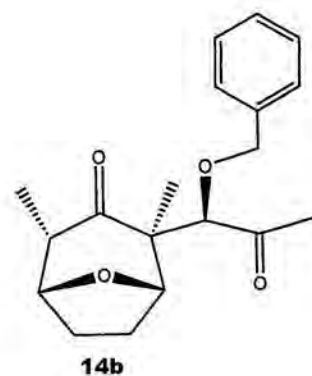
**14b**

REF: EXP 111  
Libreta n° 3  
(EXPERIMENTO 111)

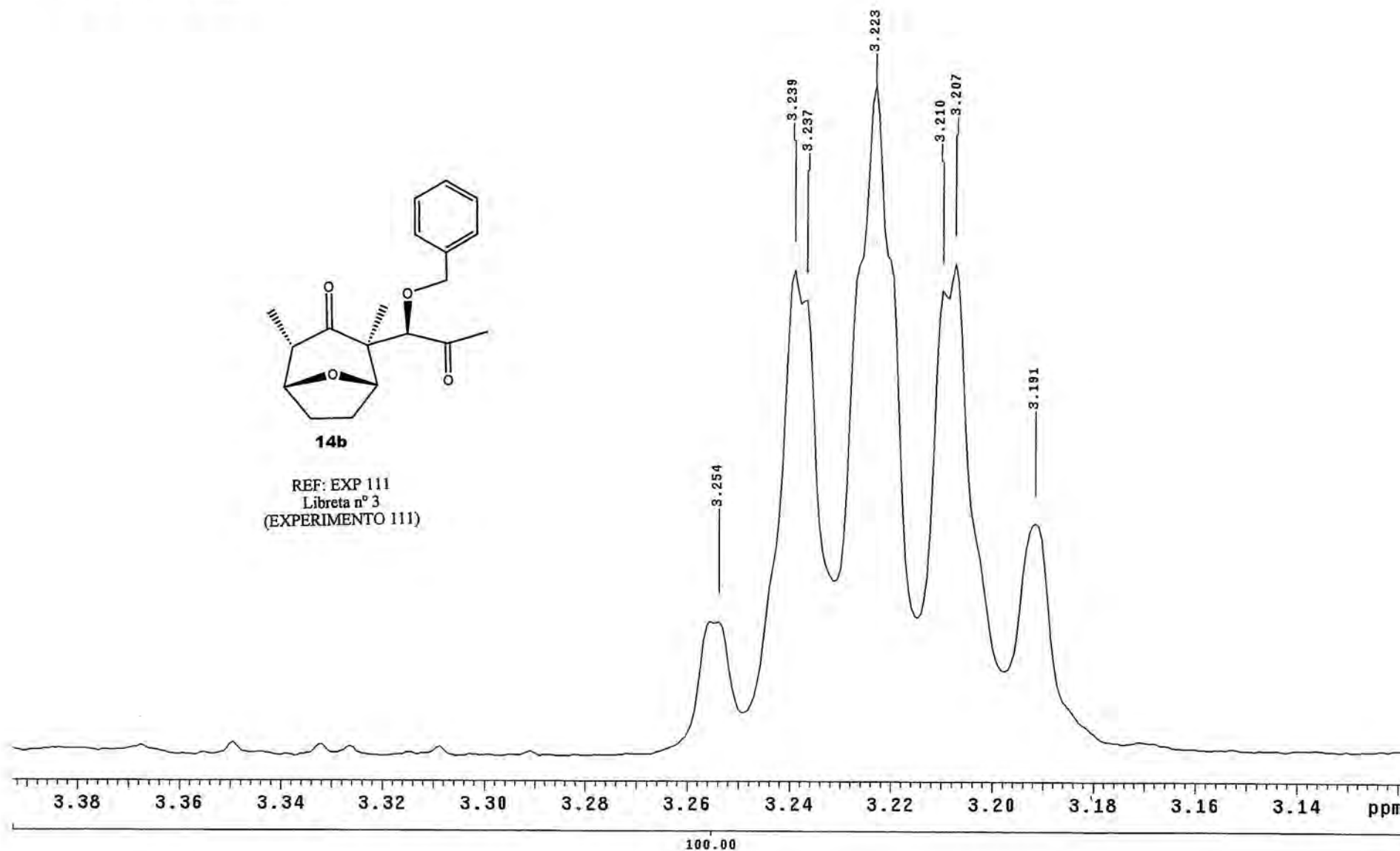


H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

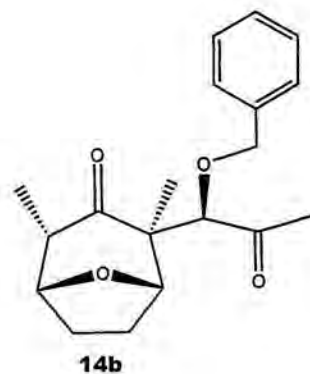


REF: EXP 111  
Libreta nº 3  
(EXPERIMENTO 111)

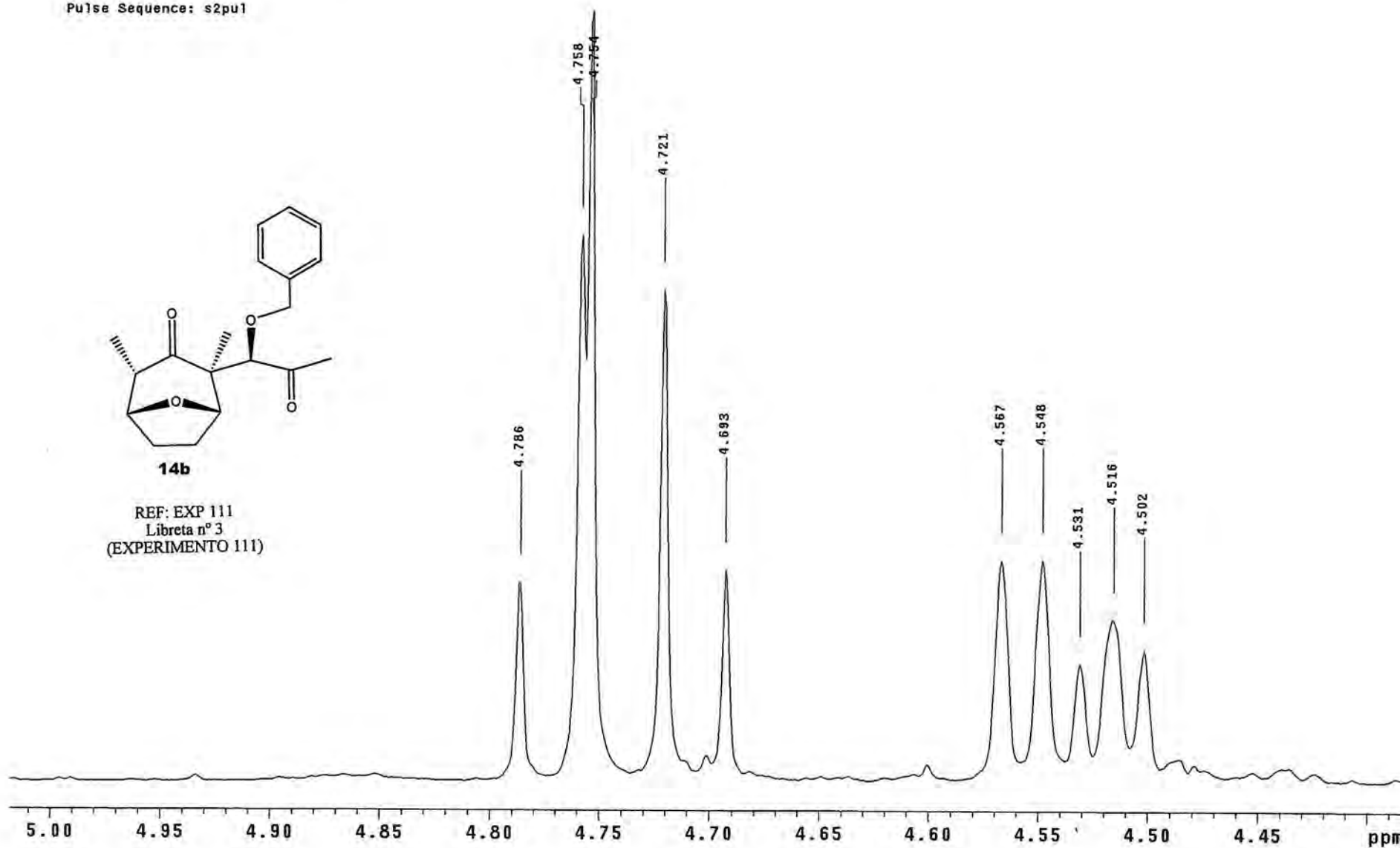


H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

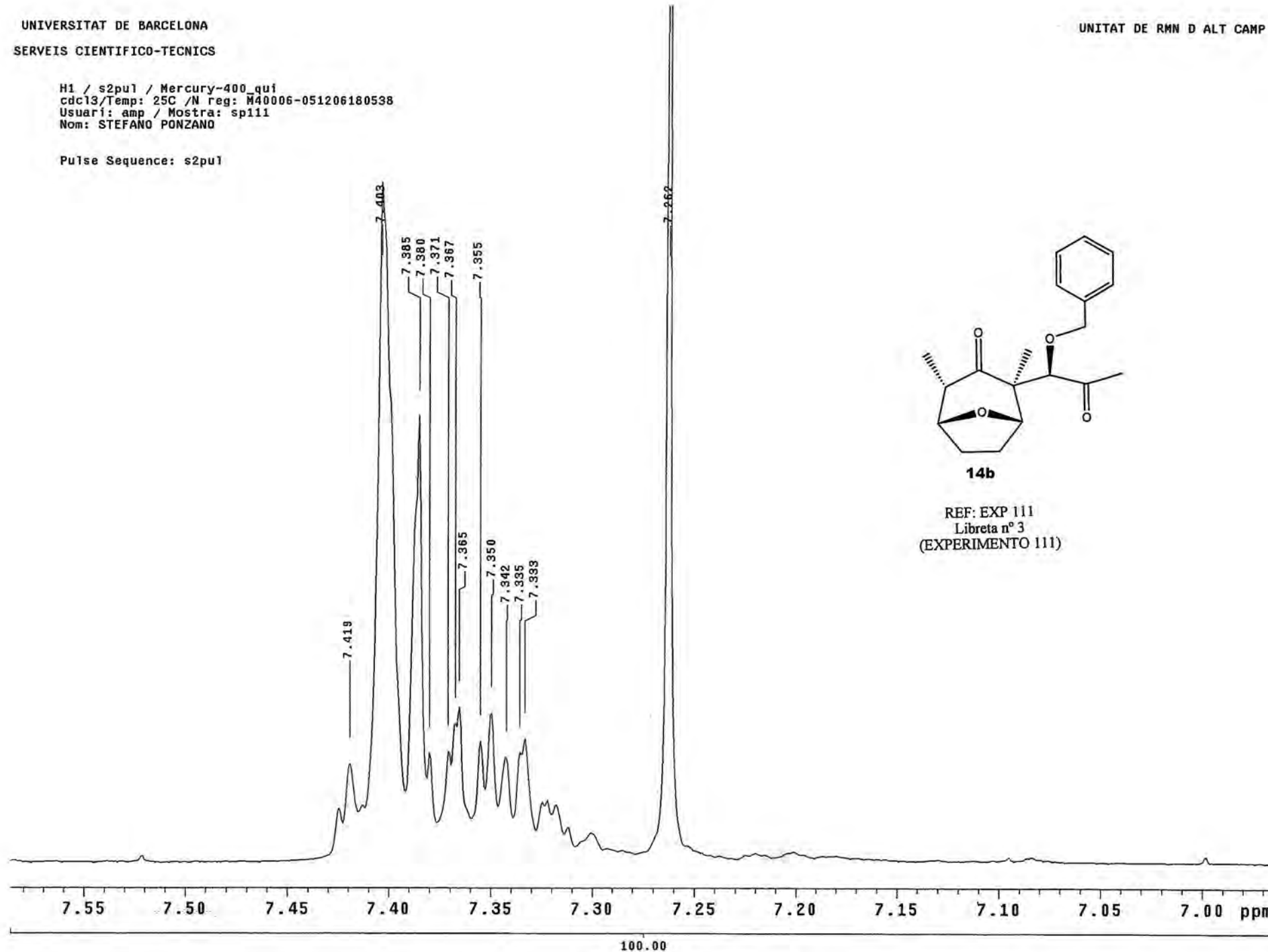


REF: EXP 111  
Libreta nº 3  
(EXPERIMENTO 111)



H1 / s2pu1 / Mercury-400.ql  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



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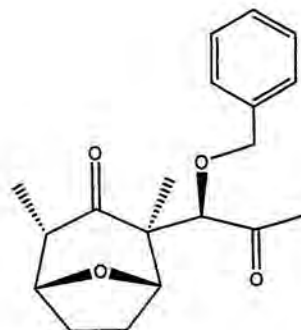
C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

\*\*\*\*\*

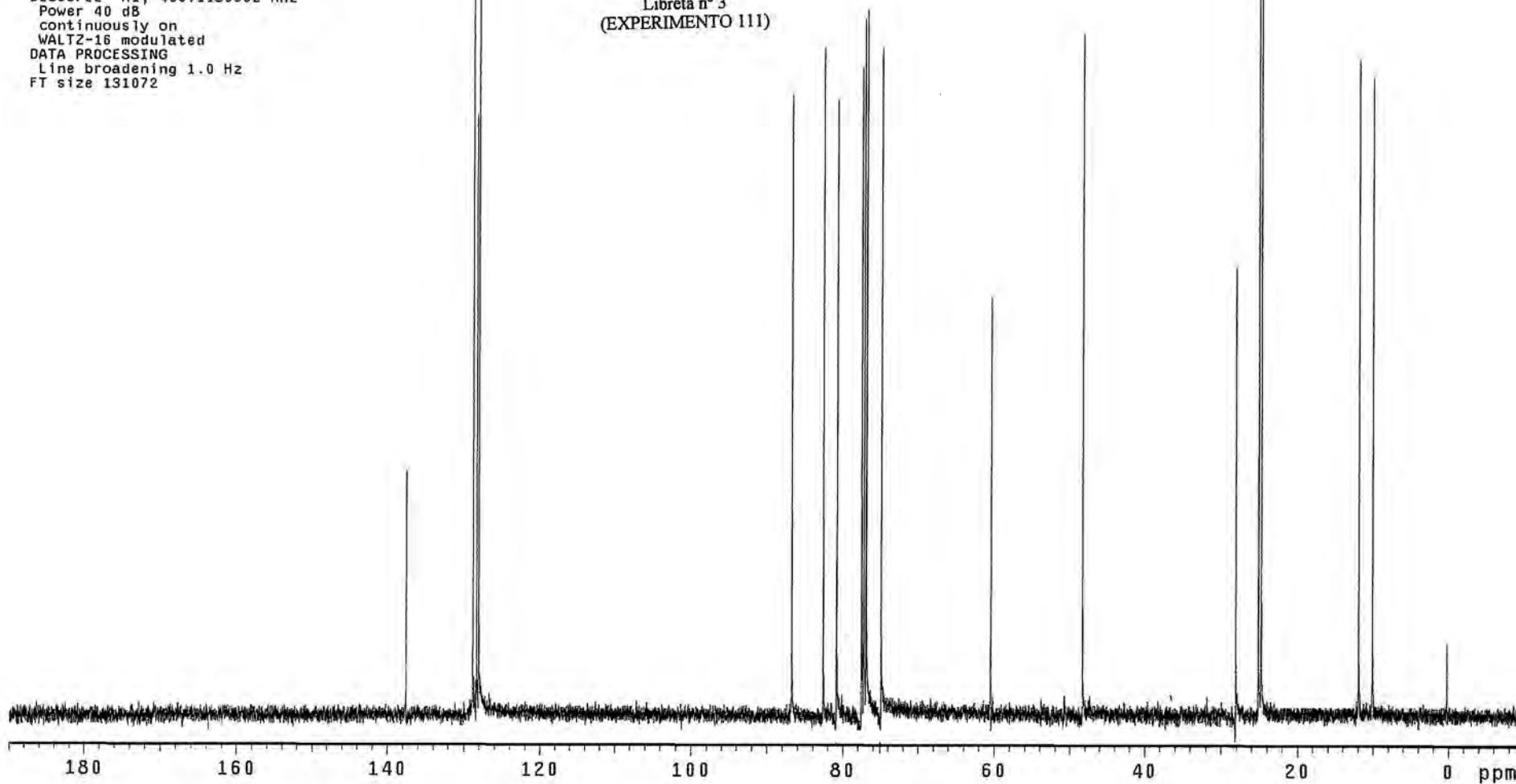
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 40 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



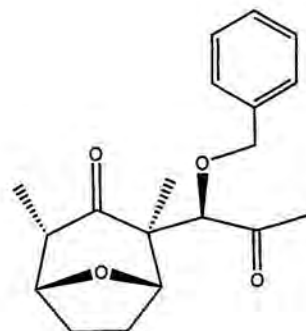
**14b**

REF: EXP 111  
Libreta n° 3  
(EXPERIMENTO 111)



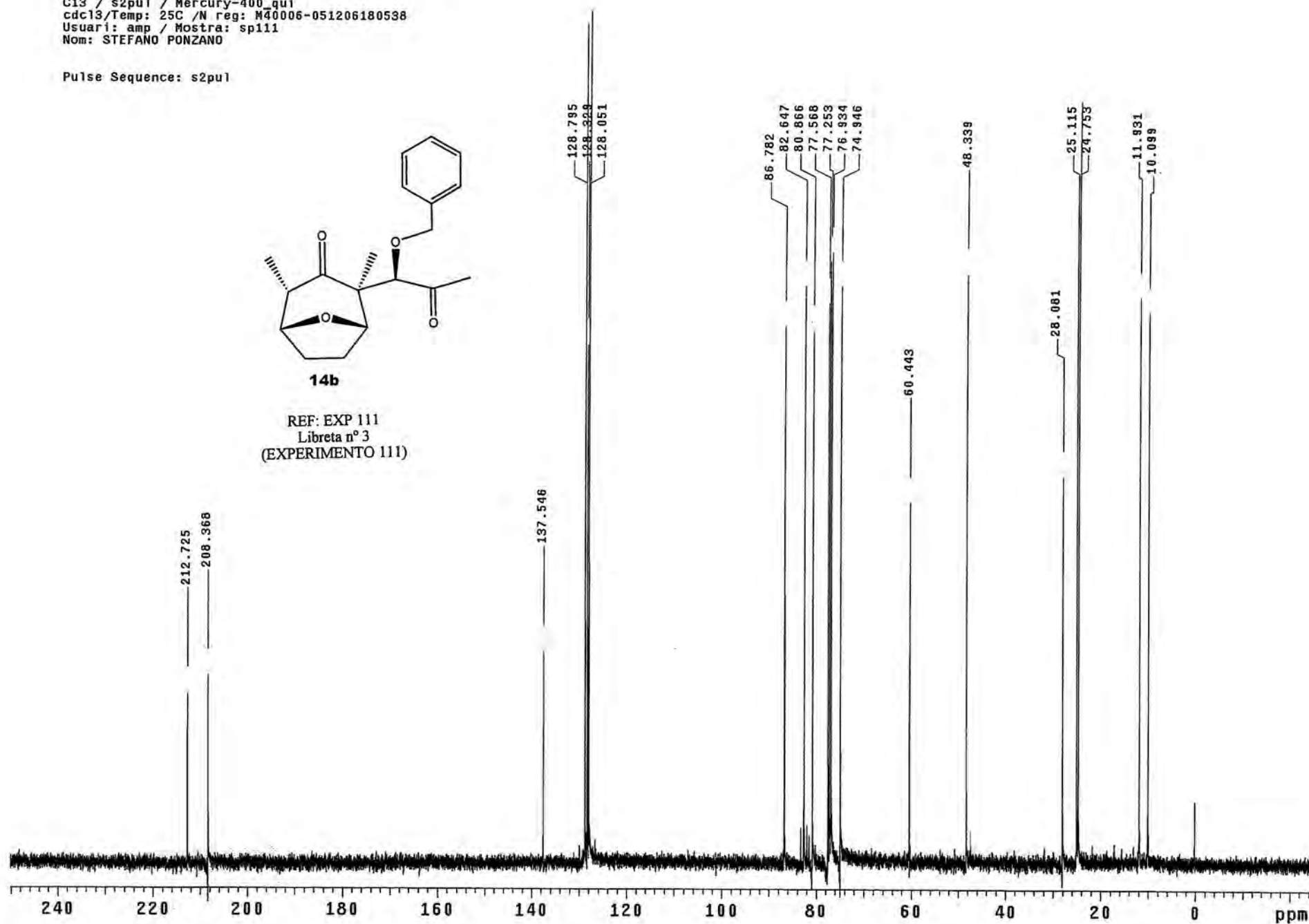
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



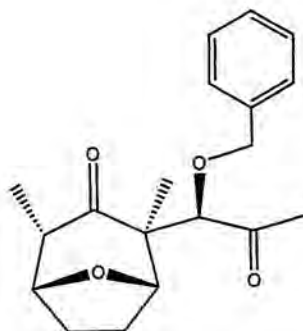
**14b**

REF: EXP 111  
Libreta nº 3  
(EXPERIMENTO 111)



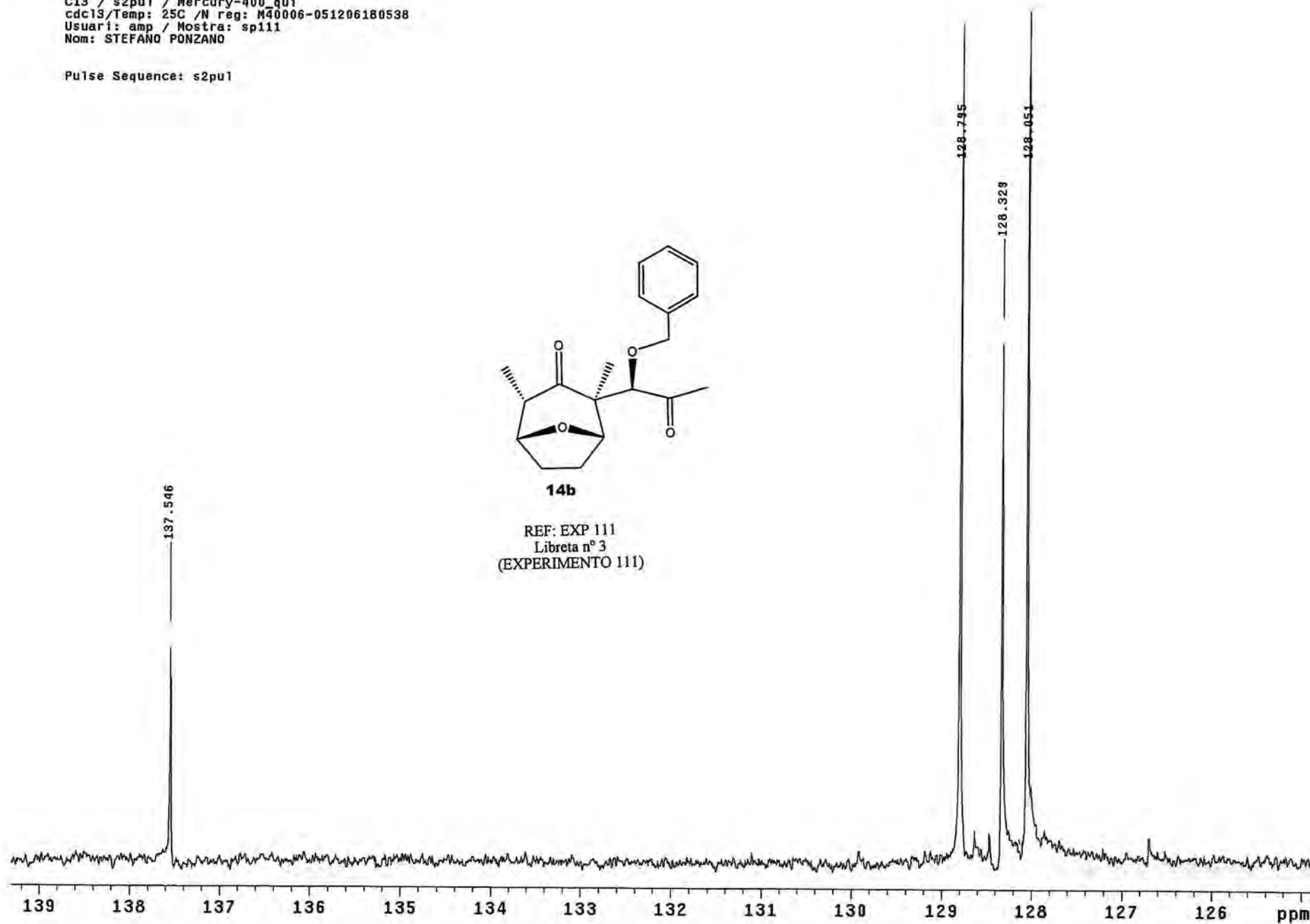
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



**14b**

REF: EXP 111  
Libreta nº 3  
(EXPERIMENTO 111)



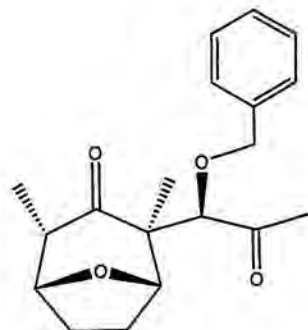
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICOS-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gCOSY / Mercury-400 qui  
cdc13/Temp: 25C /N Freq: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*

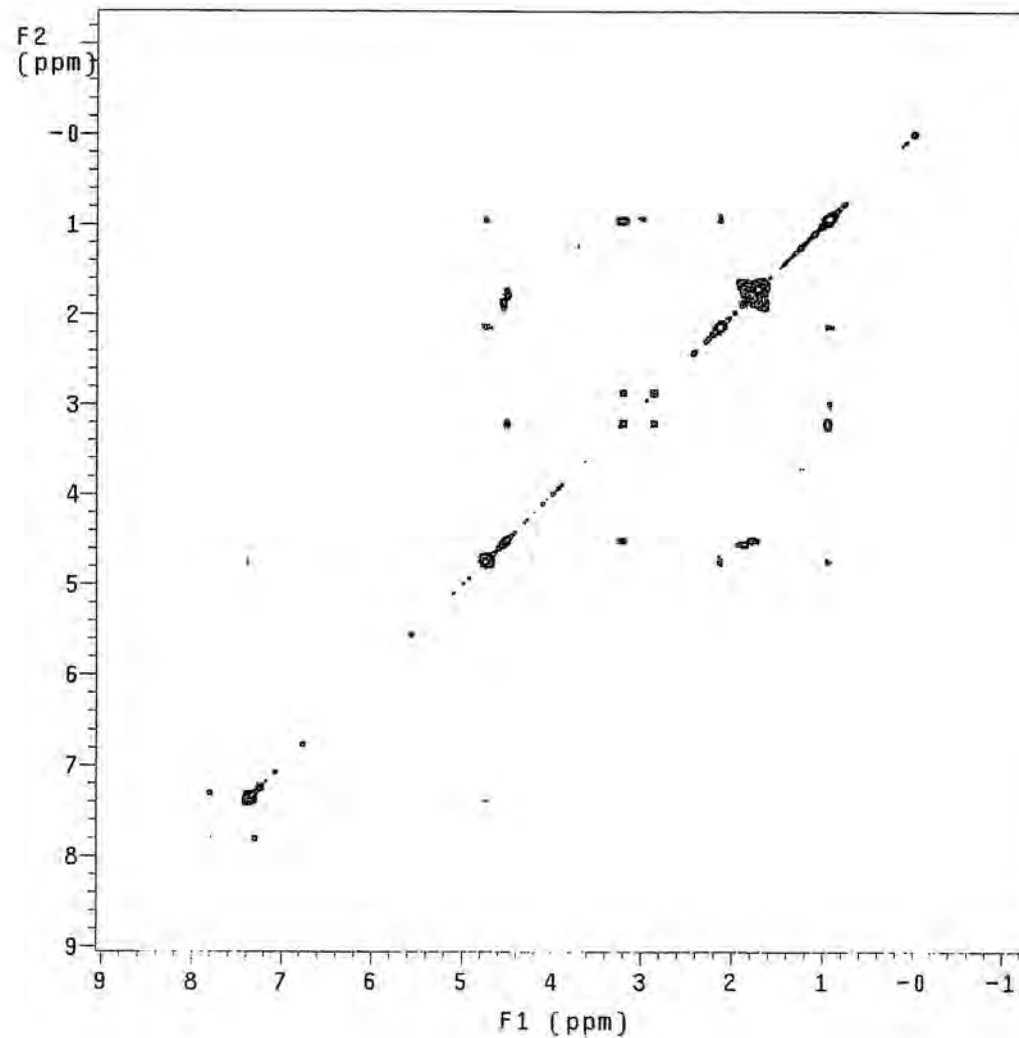
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 4166.7 Hz  
2D Width 4166.7 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.061 sec  
FT size 2048 x 2048



14b

REF: EXP 111  
Libreta nº 3  
(EXPERIMENTO 111)

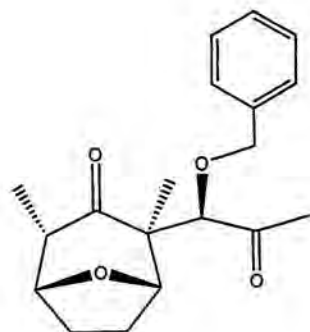


H1 / gHSQC / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-051206180538  
Usuari: amp / Mostra: sp111  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*g\*\*\*\*\*

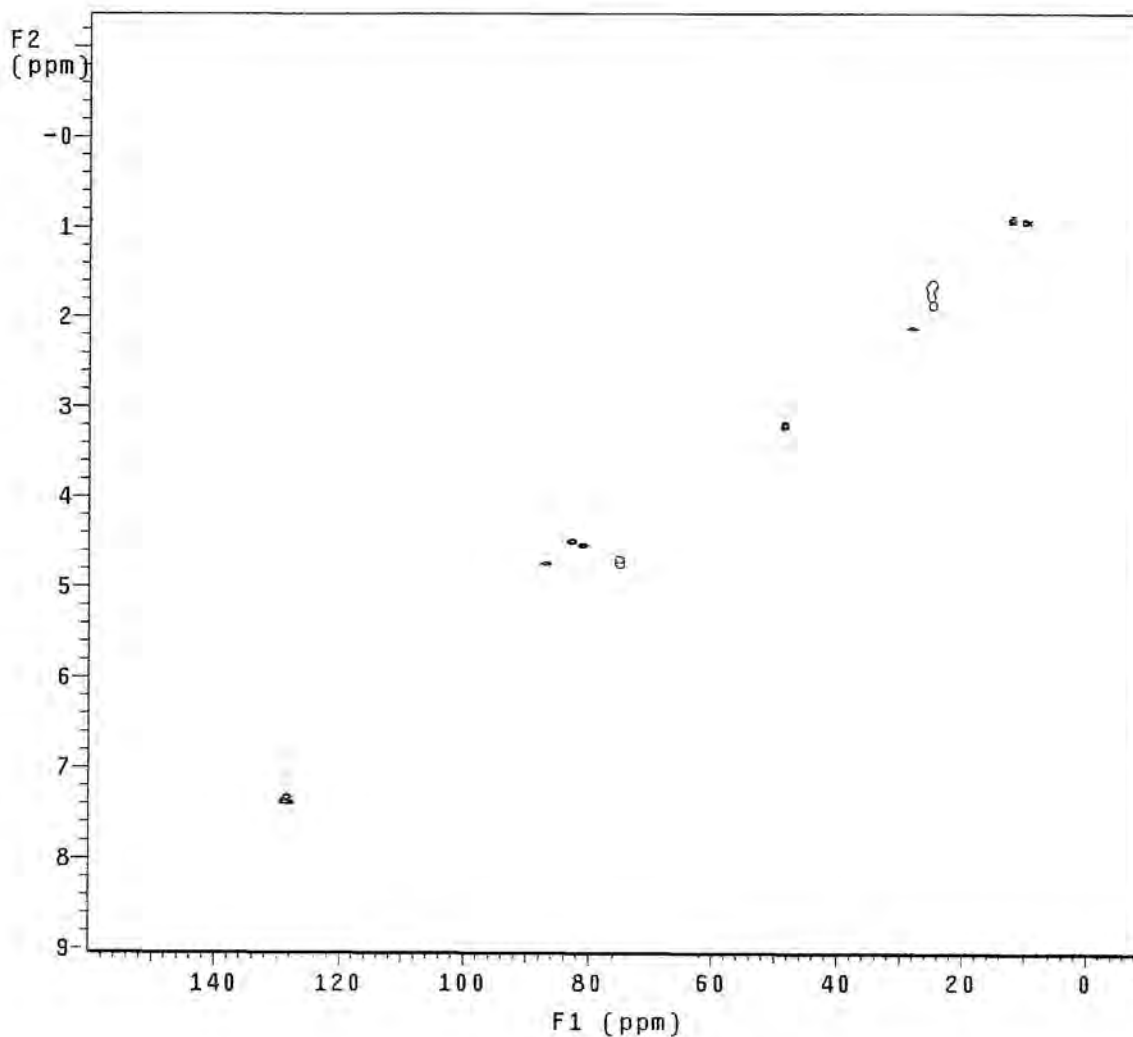
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.246 sec  
Width 4166.7 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 45 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.113 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 2048 x 2048

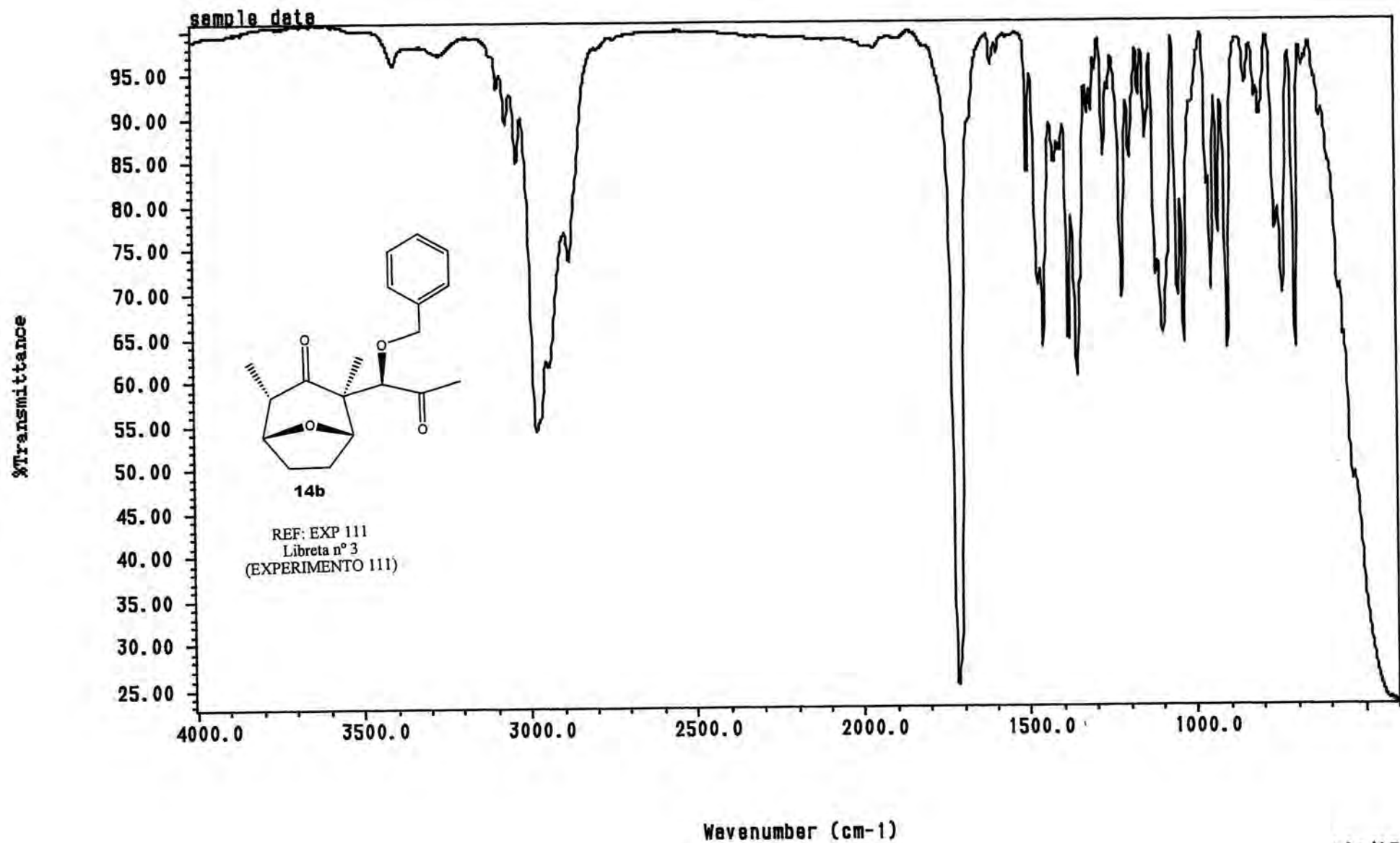


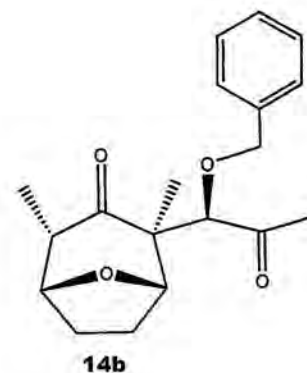
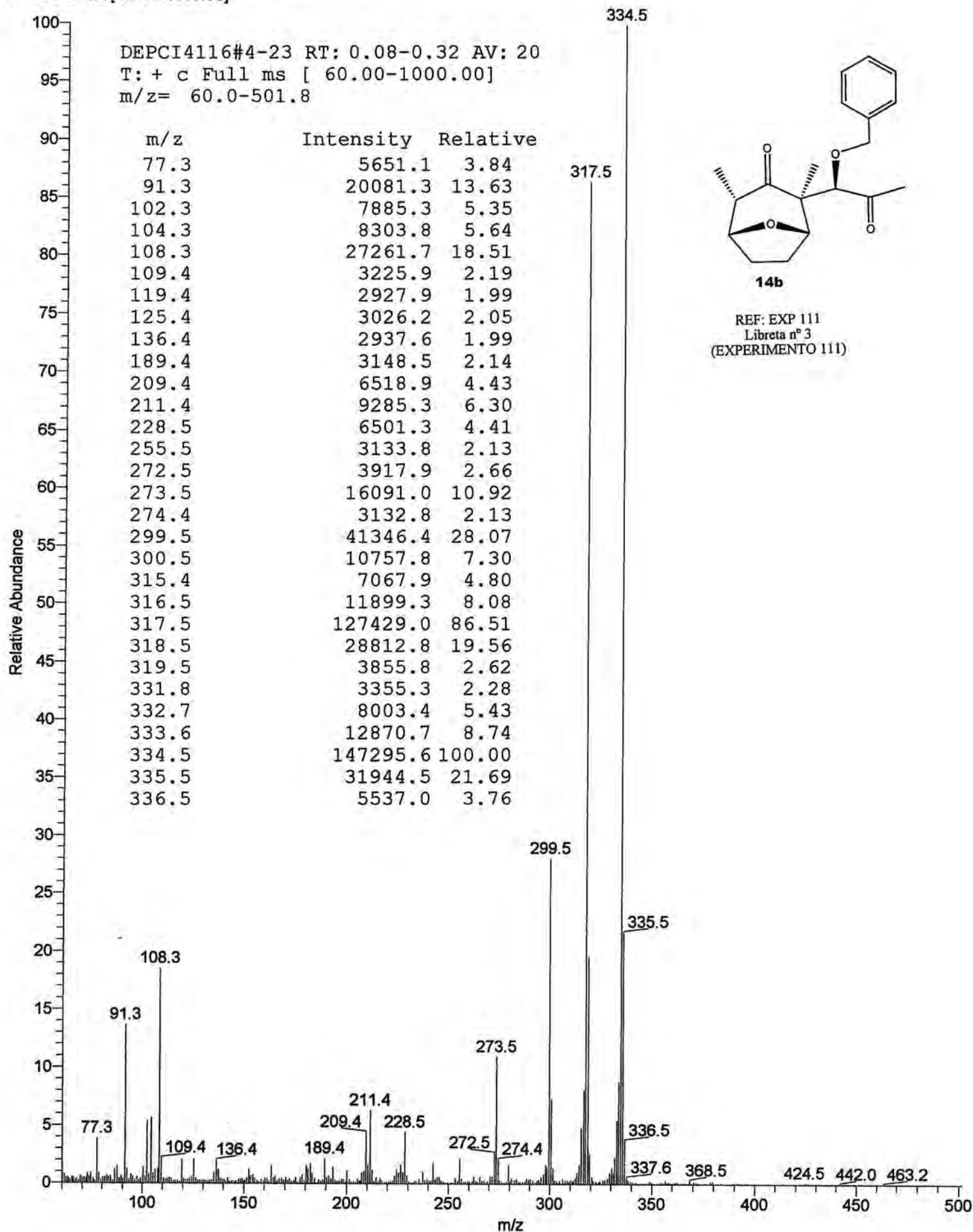
14b

REF: EXP 111  
Libreta nº 3  
(EXPERIMENTO 111)



sp111  
sample data



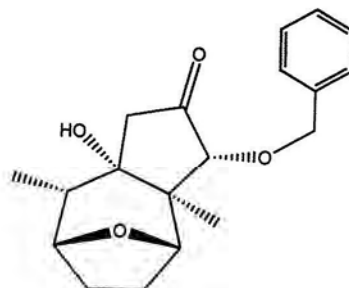


REF: EXP 111  
Libreta n° 3  
(EXPERIMENTO 111)

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

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

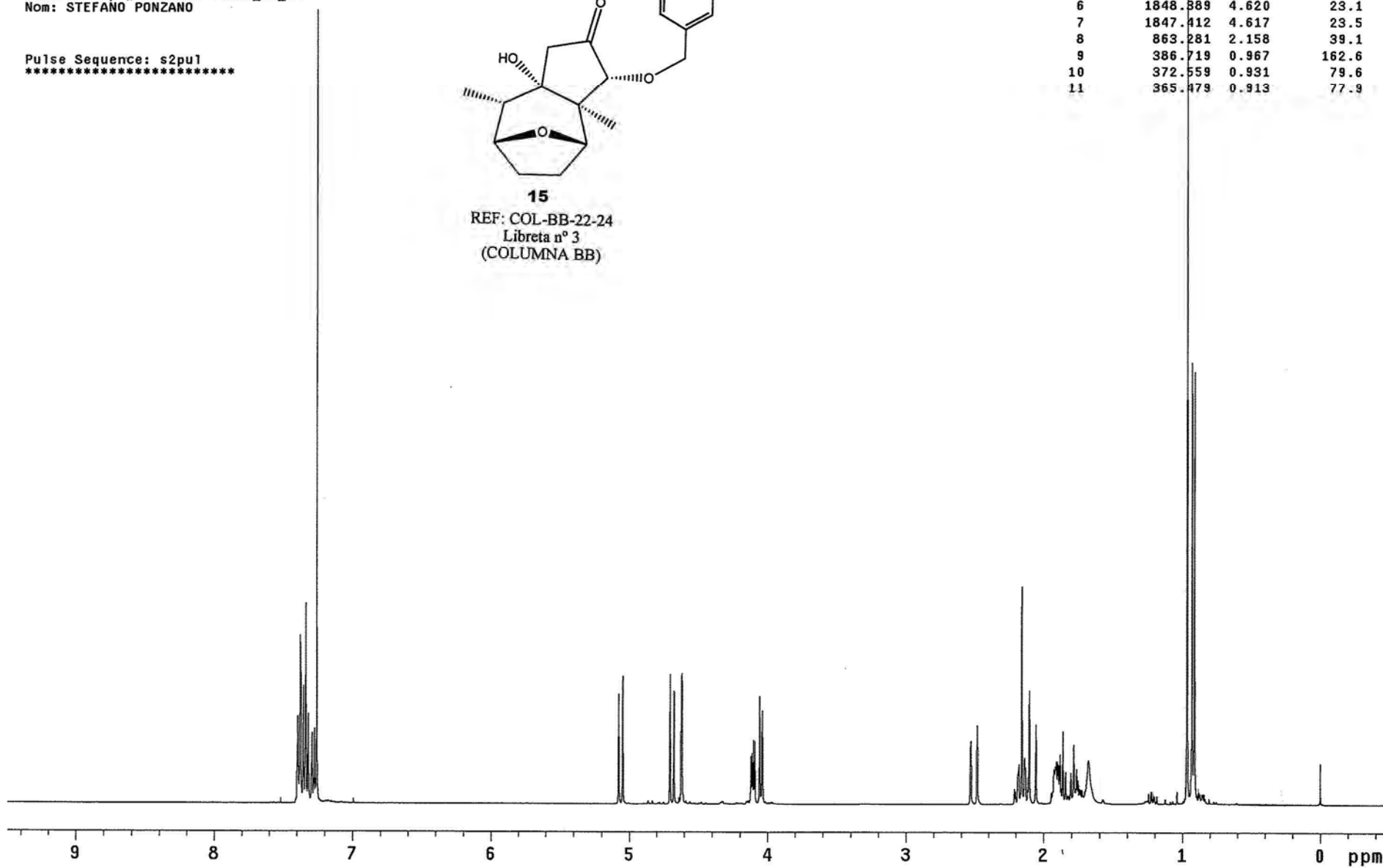
Pulse Sequence: s2pu1  
\*\*\*\*\*



**15**

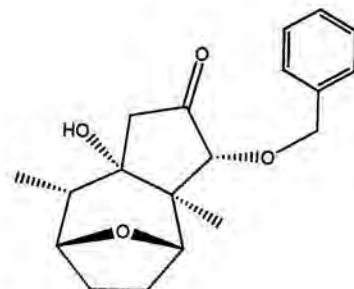
REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)

| INDEX | FREQUENCY | PPM   | HEIGHT |
|-------|-----------|-------|--------|
| 1     | 2951.172  | 7.376 | 30.3   |
| 2     | 2935.303  | 7.336 | 35.9   |
| 3     | 2903.309  | 7.257 | 143.5  |
| 4     | 2020.264  | 5.049 | 23.0   |
| 5     | 1881.336  | 4.703 | 23.4   |
| 6     | 1848.389  | 4.620 | 23.1   |
| 7     | 1847.412  | 4.617 | 23.5   |
| 8     | 863.281   | 2.158 | 39.1   |
| 9     | 386.719   | 0.967 | 162.6  |
| 10    | 372.559   | 0.931 | 79.6   |
| 11    | 365.479   | 0.913 | 77.9   |



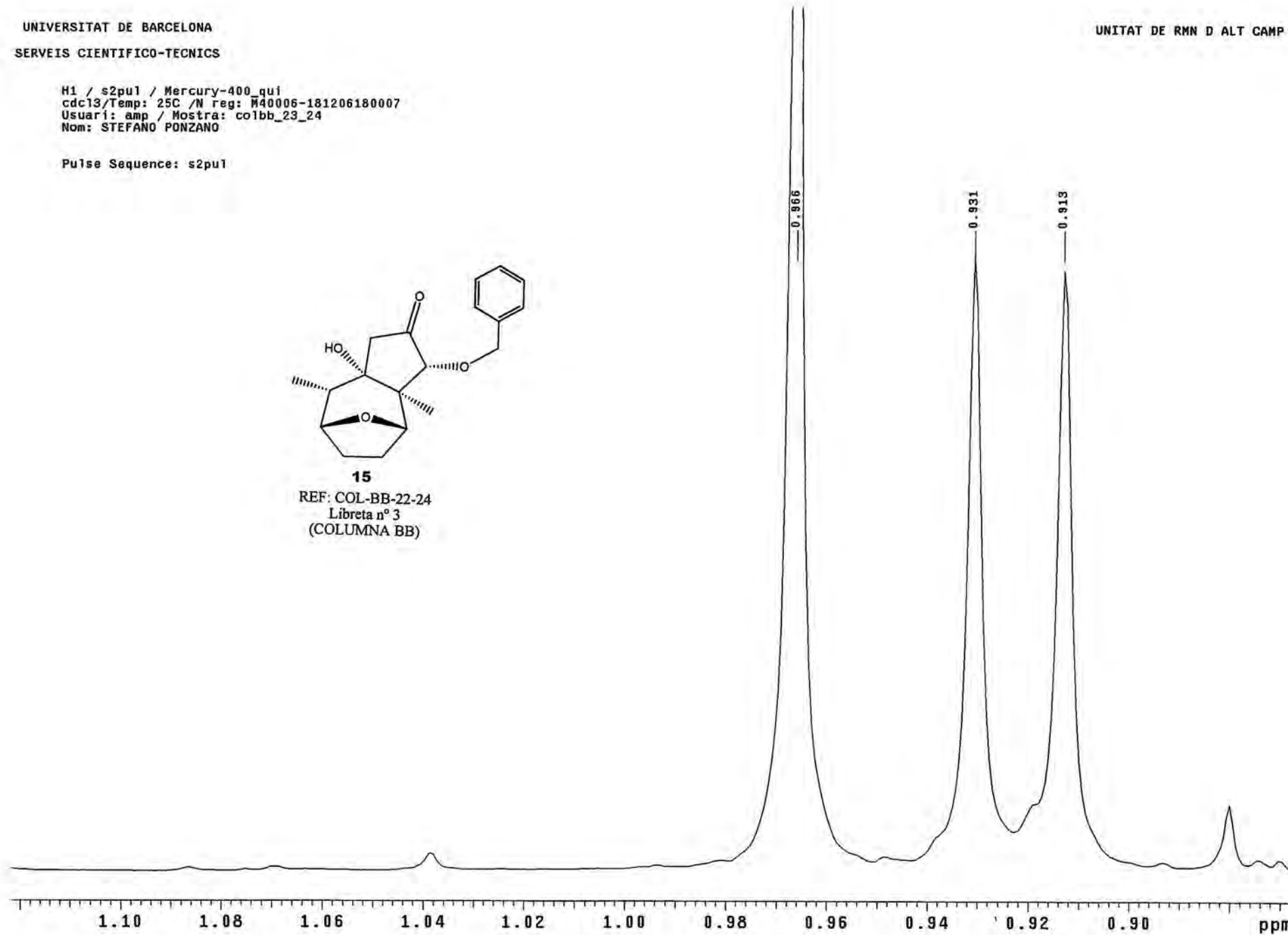
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



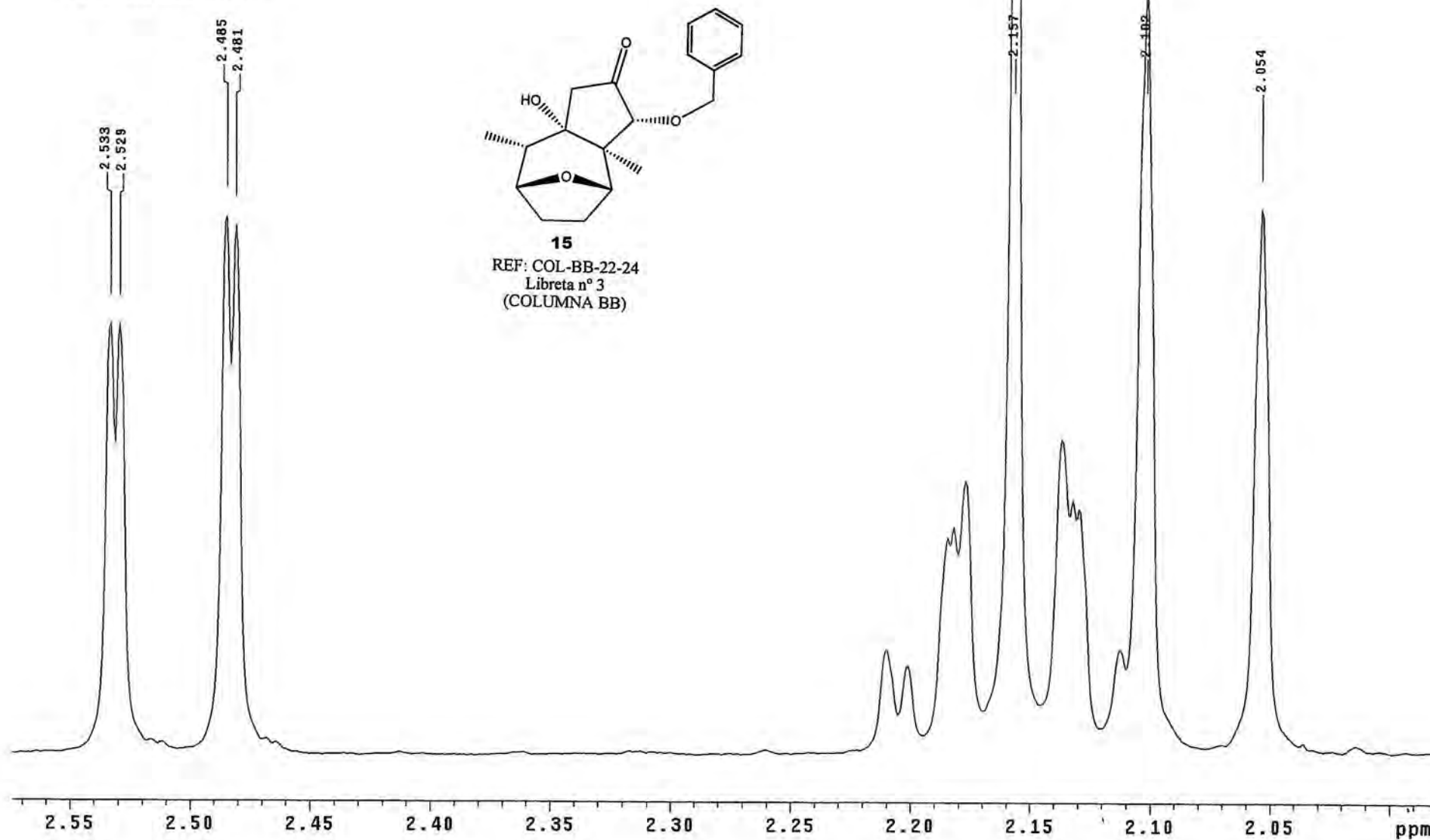
**15**

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNNA BB)



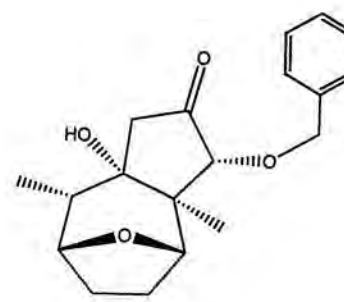
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



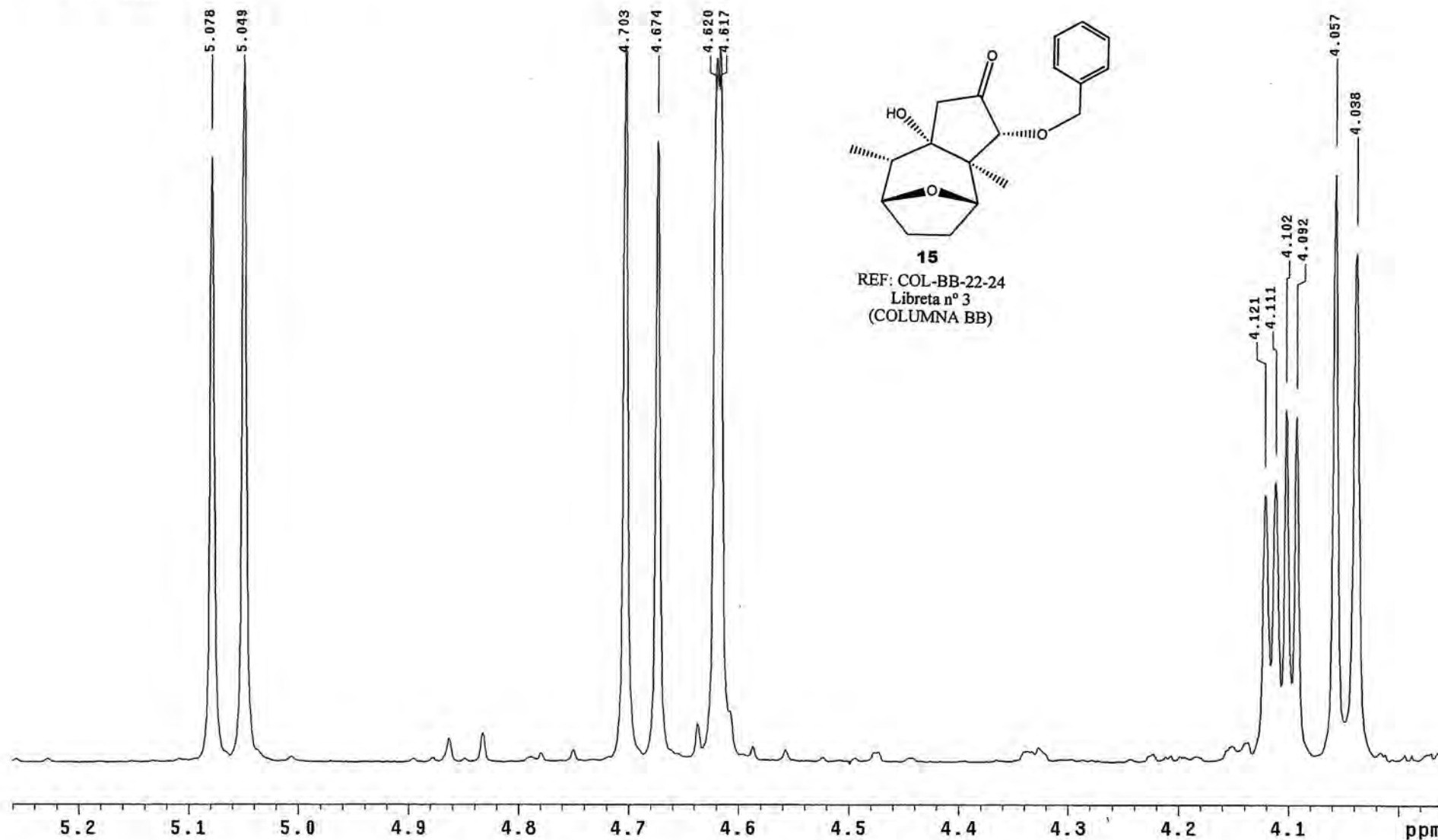
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



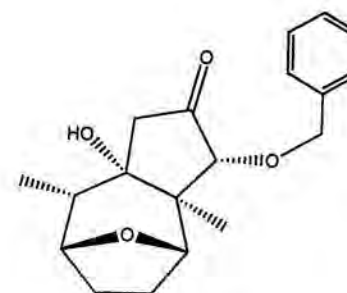
**15**

REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)



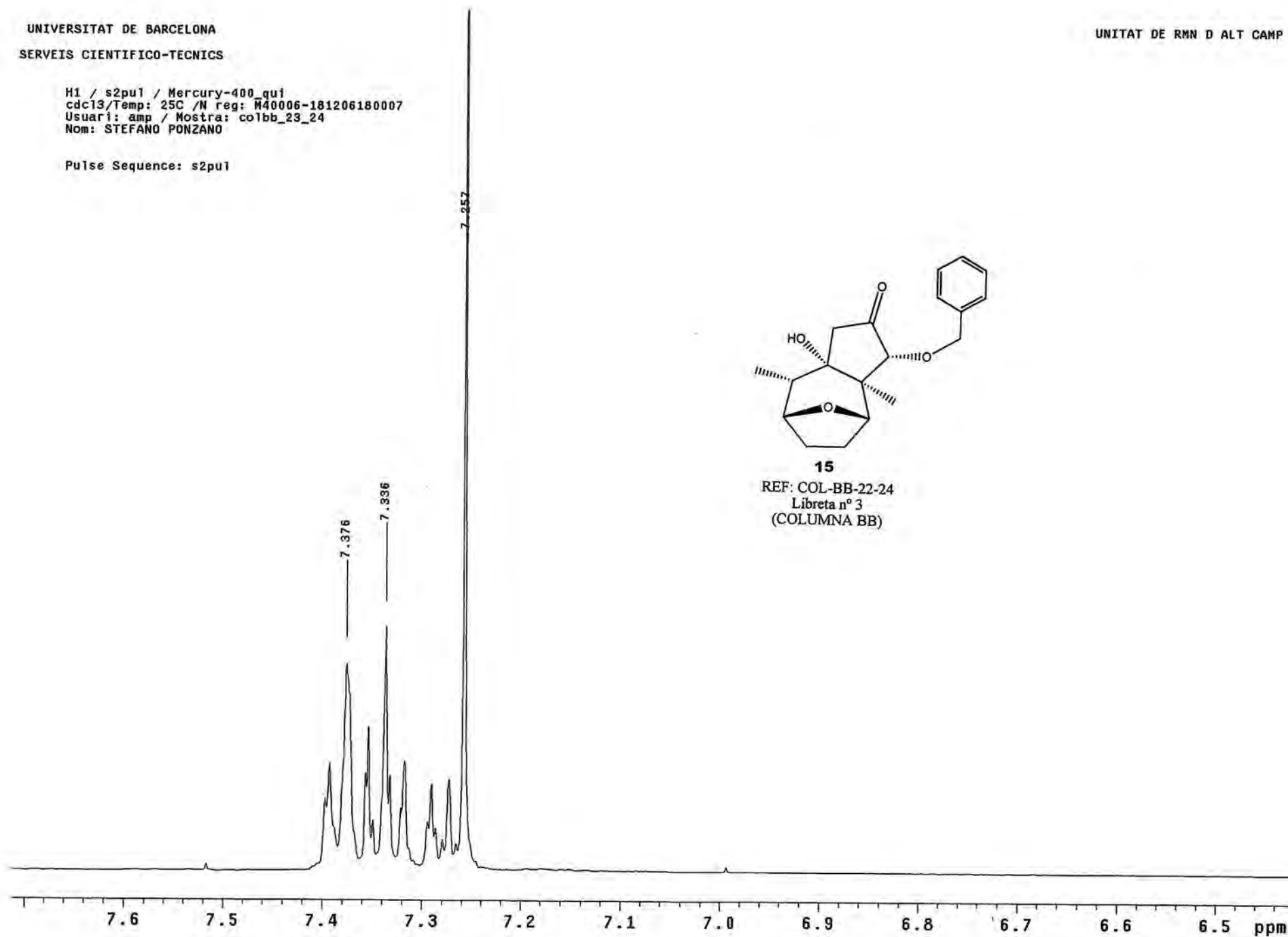
H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



**15**

REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)



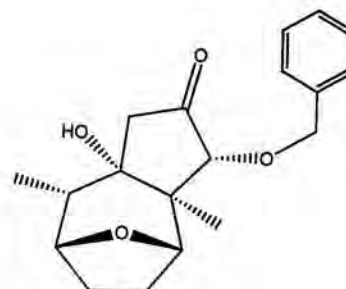
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pul / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pul  
\*\*\*\*\*

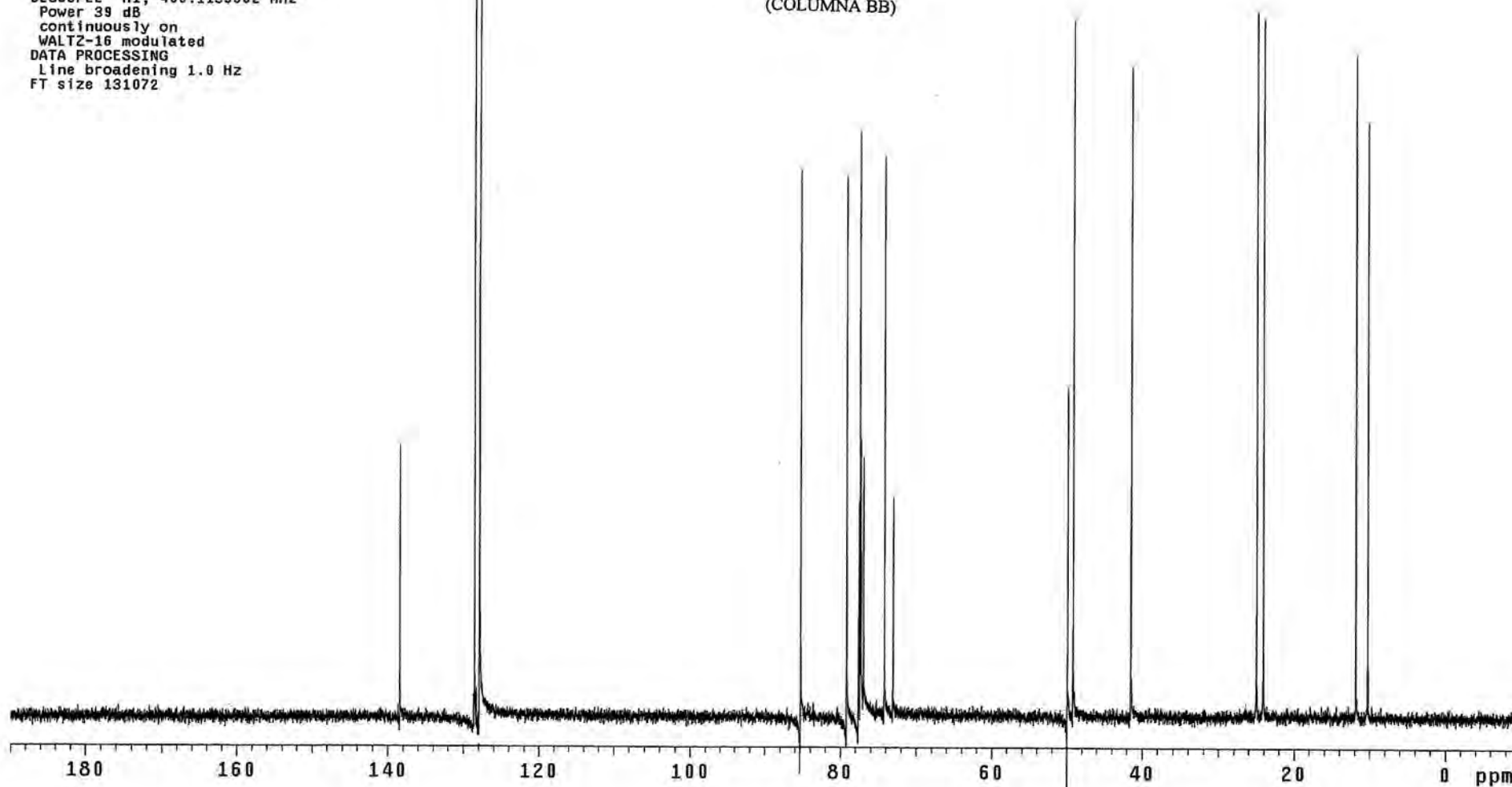
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



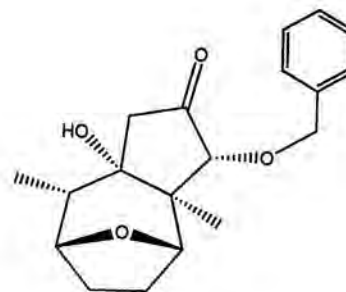
15

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMN BB)



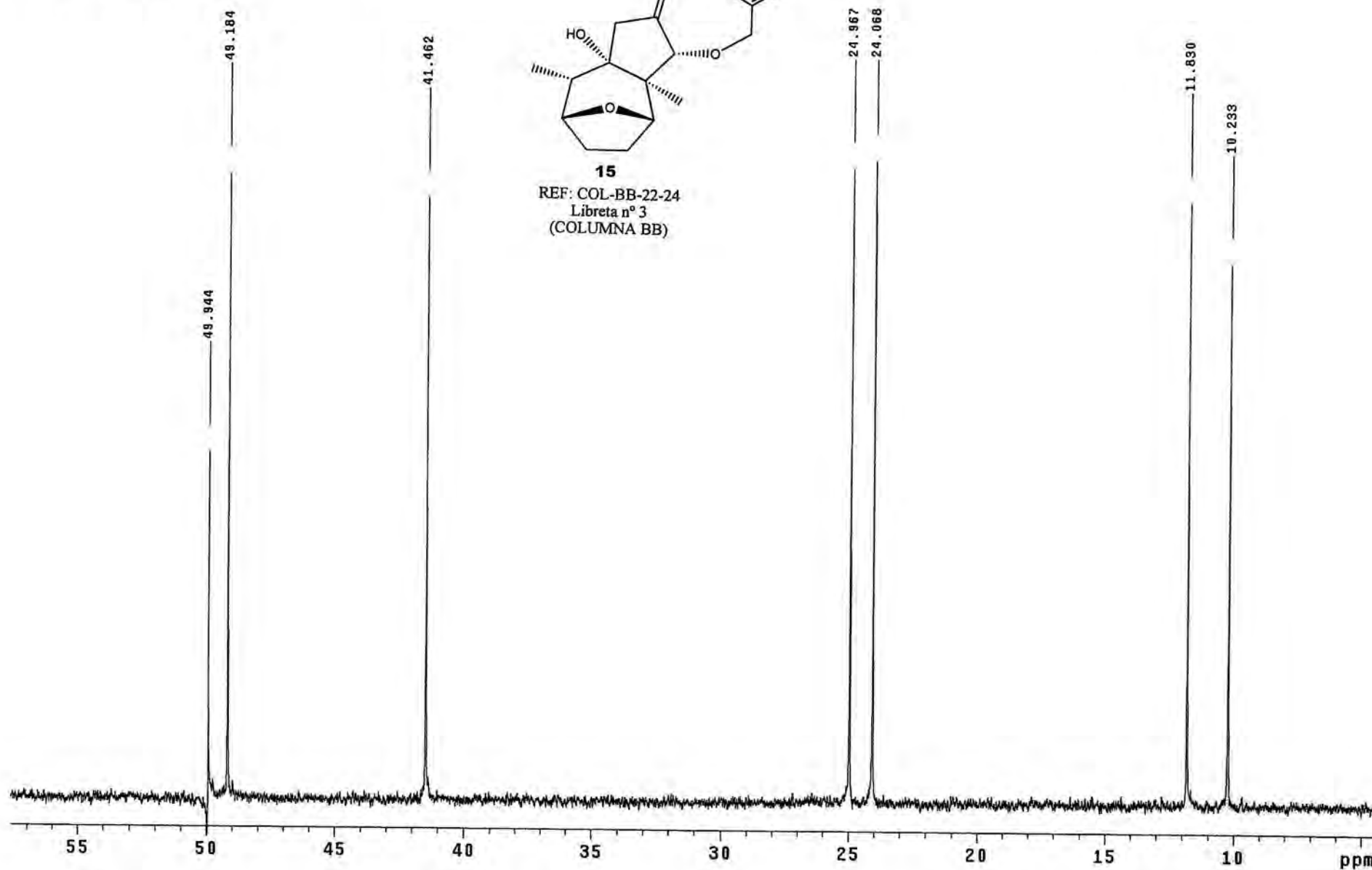
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



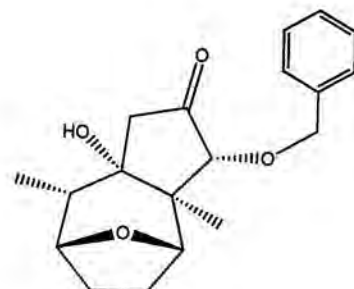
**15**

REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)



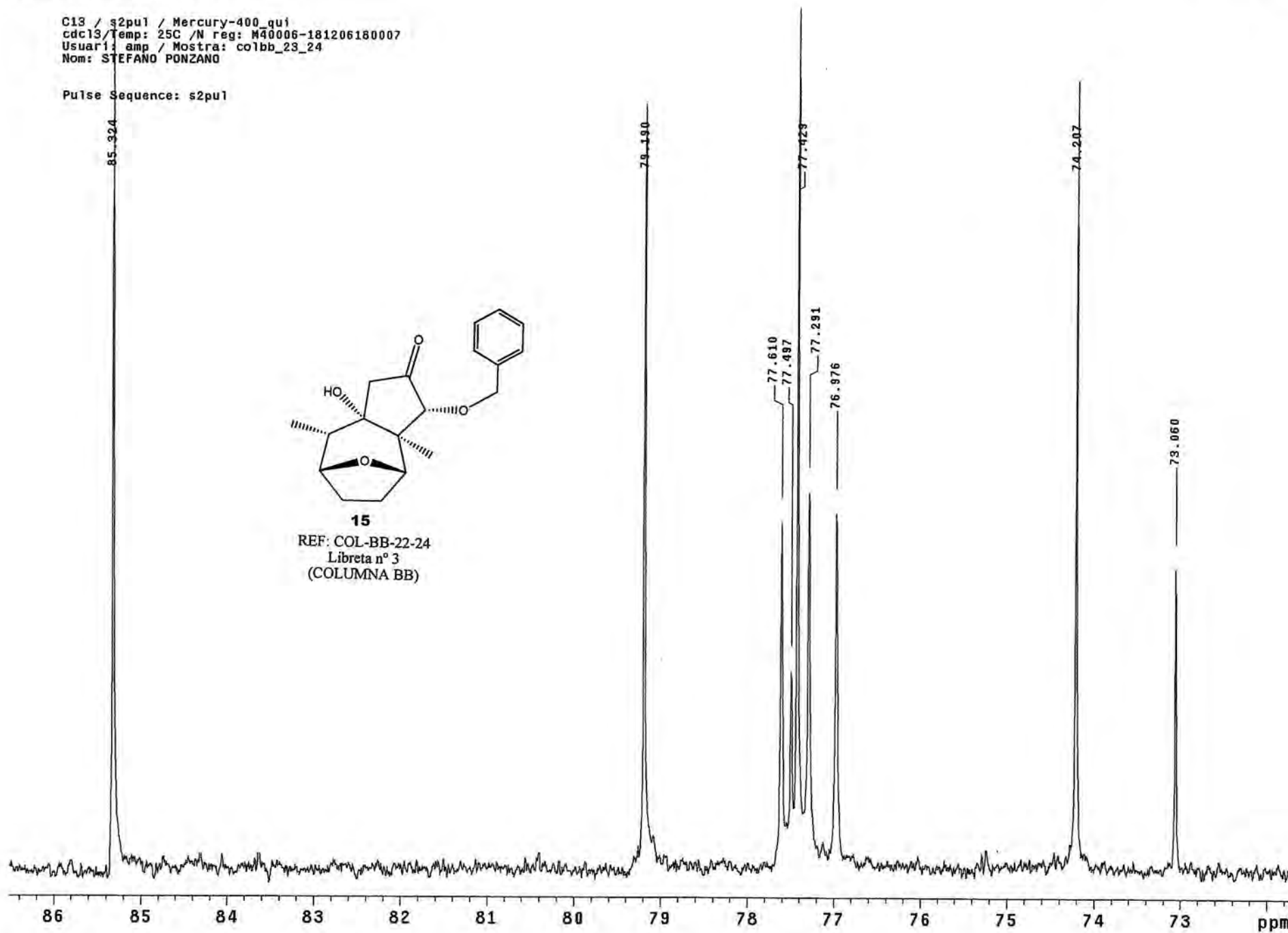
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



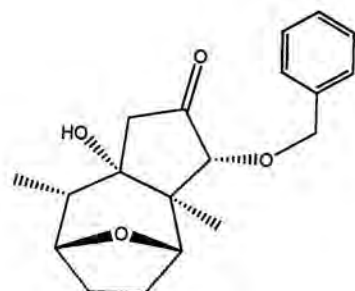
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REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNNA BB)



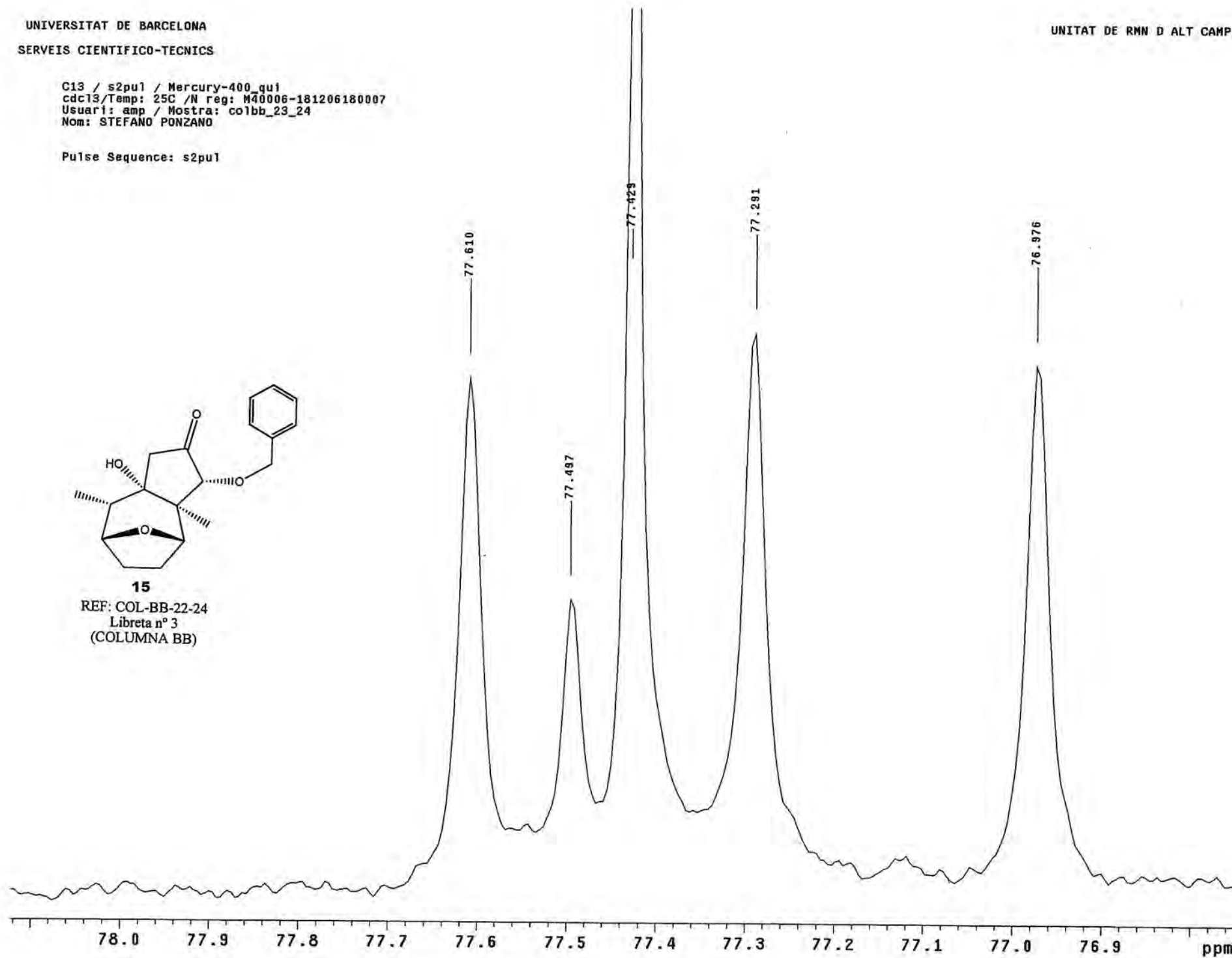
C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



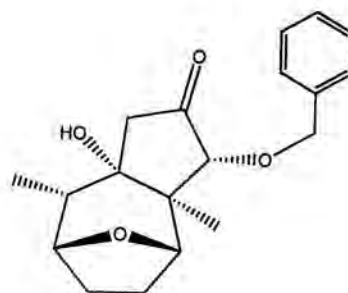
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REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNNA BB)



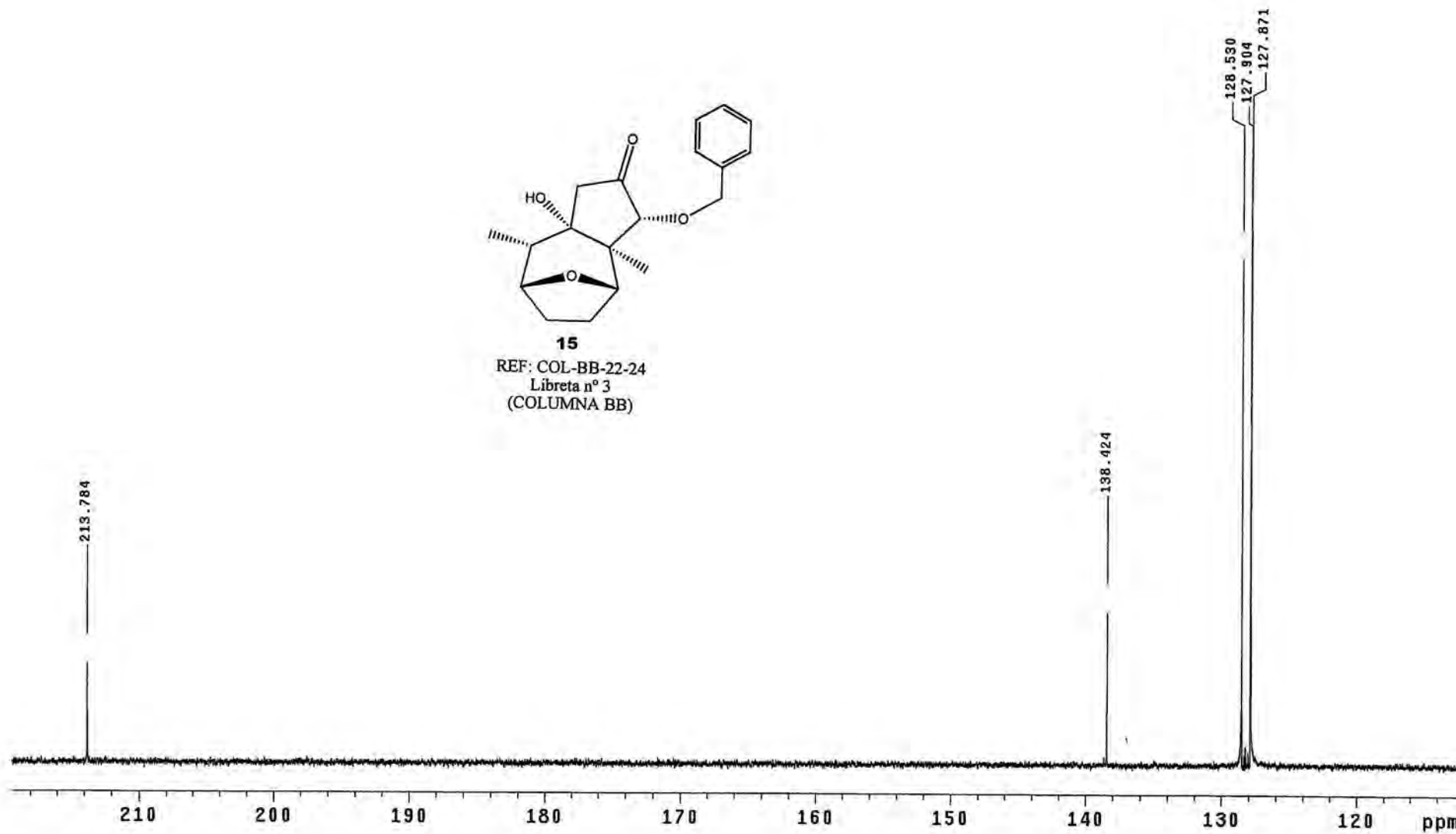
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



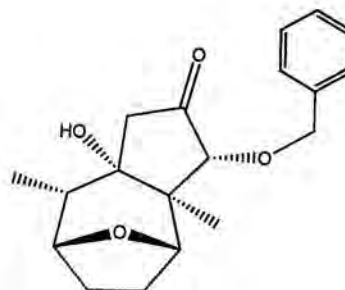
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REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNA BB)



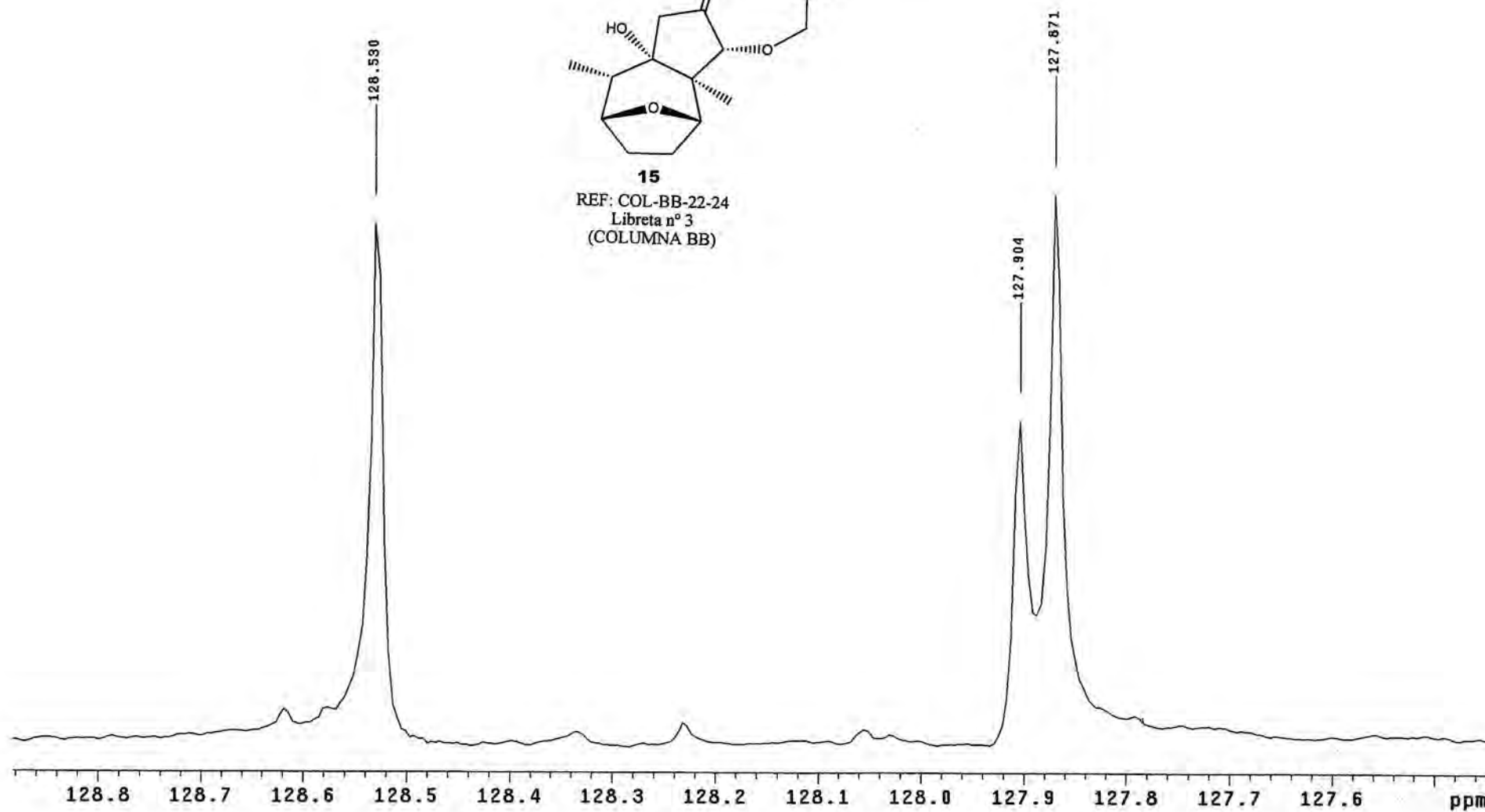
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cdcl3/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



**15**

REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)



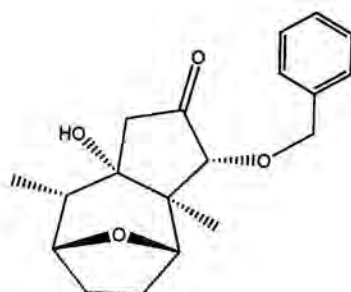
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / gCOSY / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40006-181206180007  
Usuari: amp / Mostra: colbb\_23\_24  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*

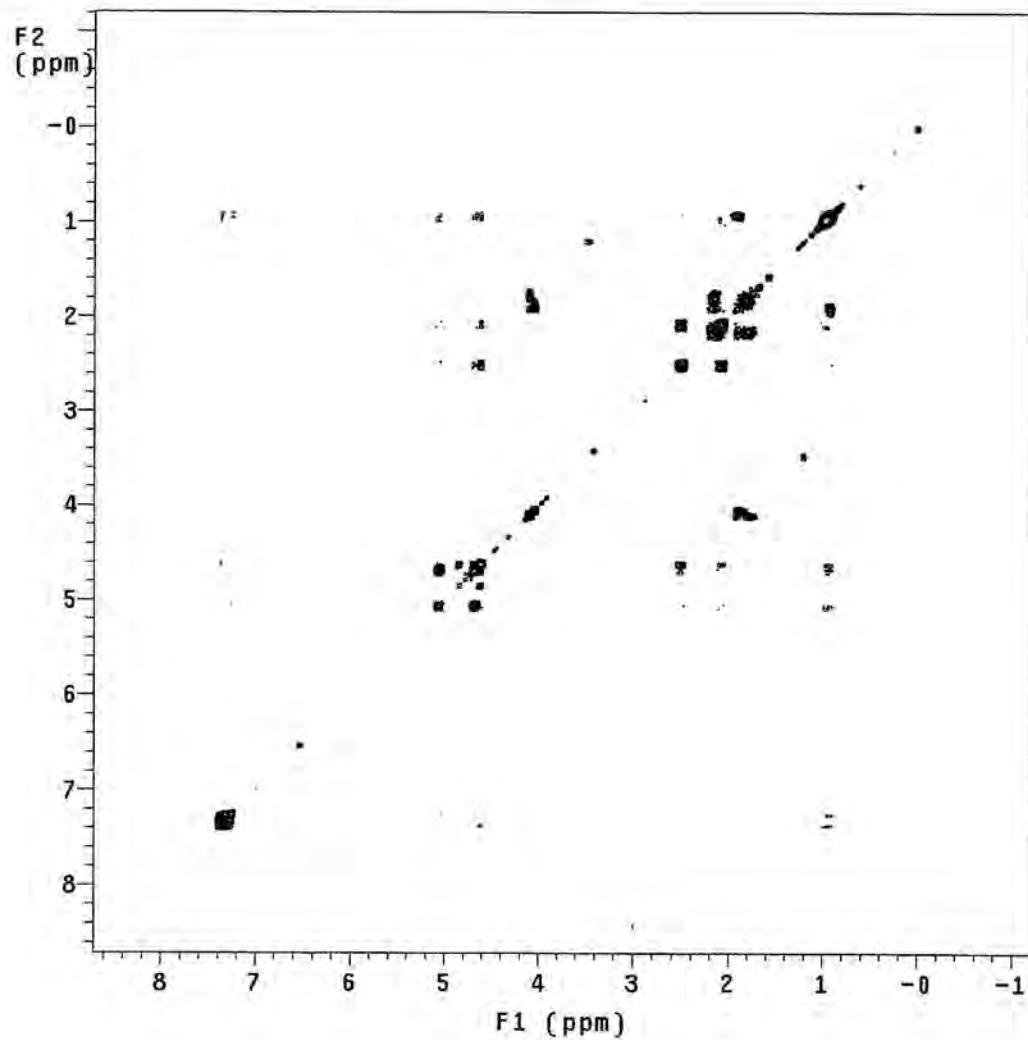
Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3968.3 Hz  
2D Width 3968.3 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.065 sec  
FT size 2048 x 2048



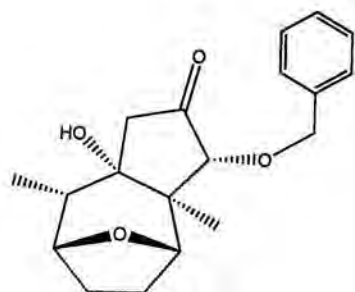
15

REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)



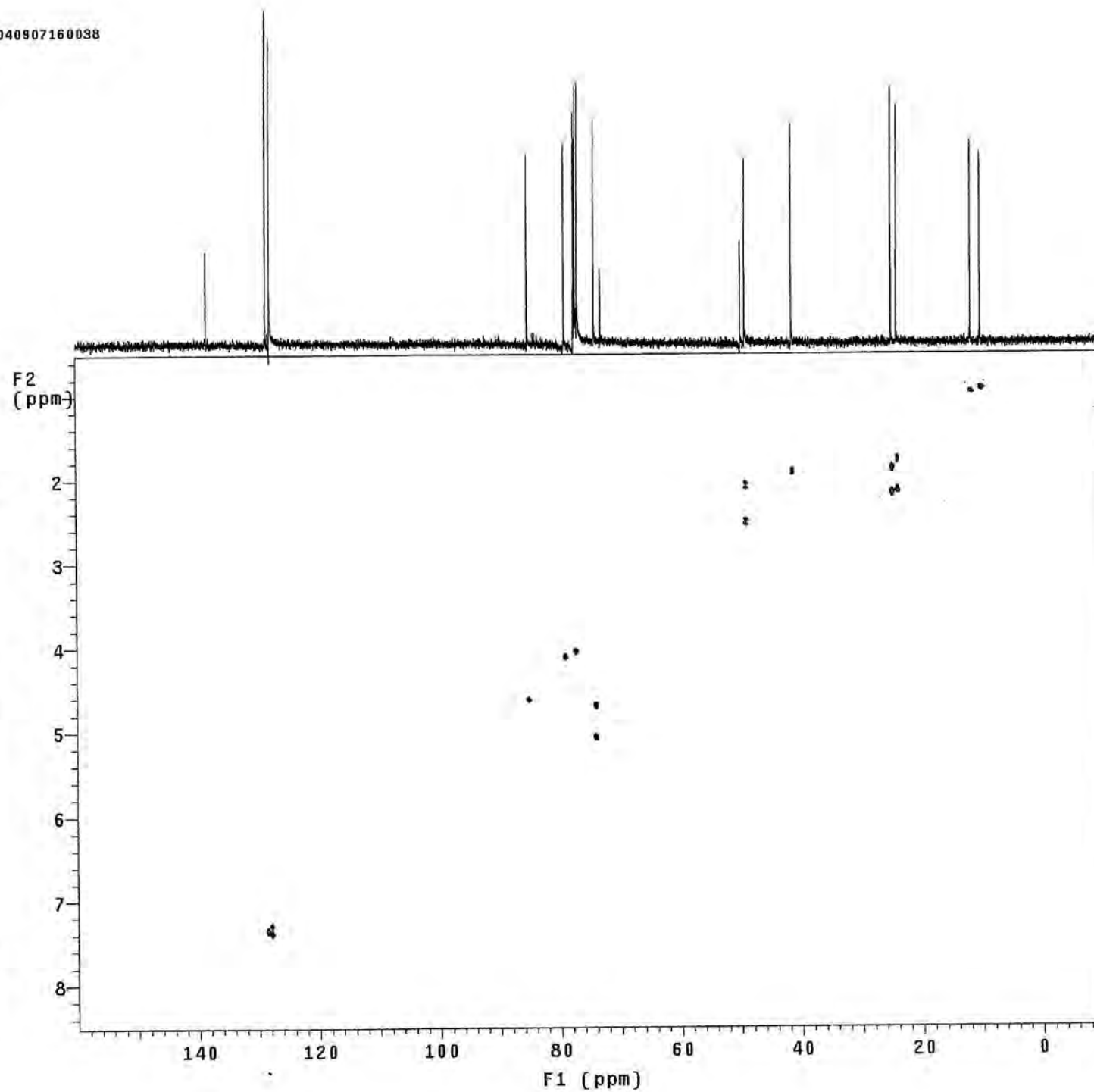
H1 / gHSQC / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-040907160038  
Usuari: amp / Mostra: w15\_20  
Nom: JOAN\_ANTONI RUIZ RINCON

Pulse Sequence: gHSQC

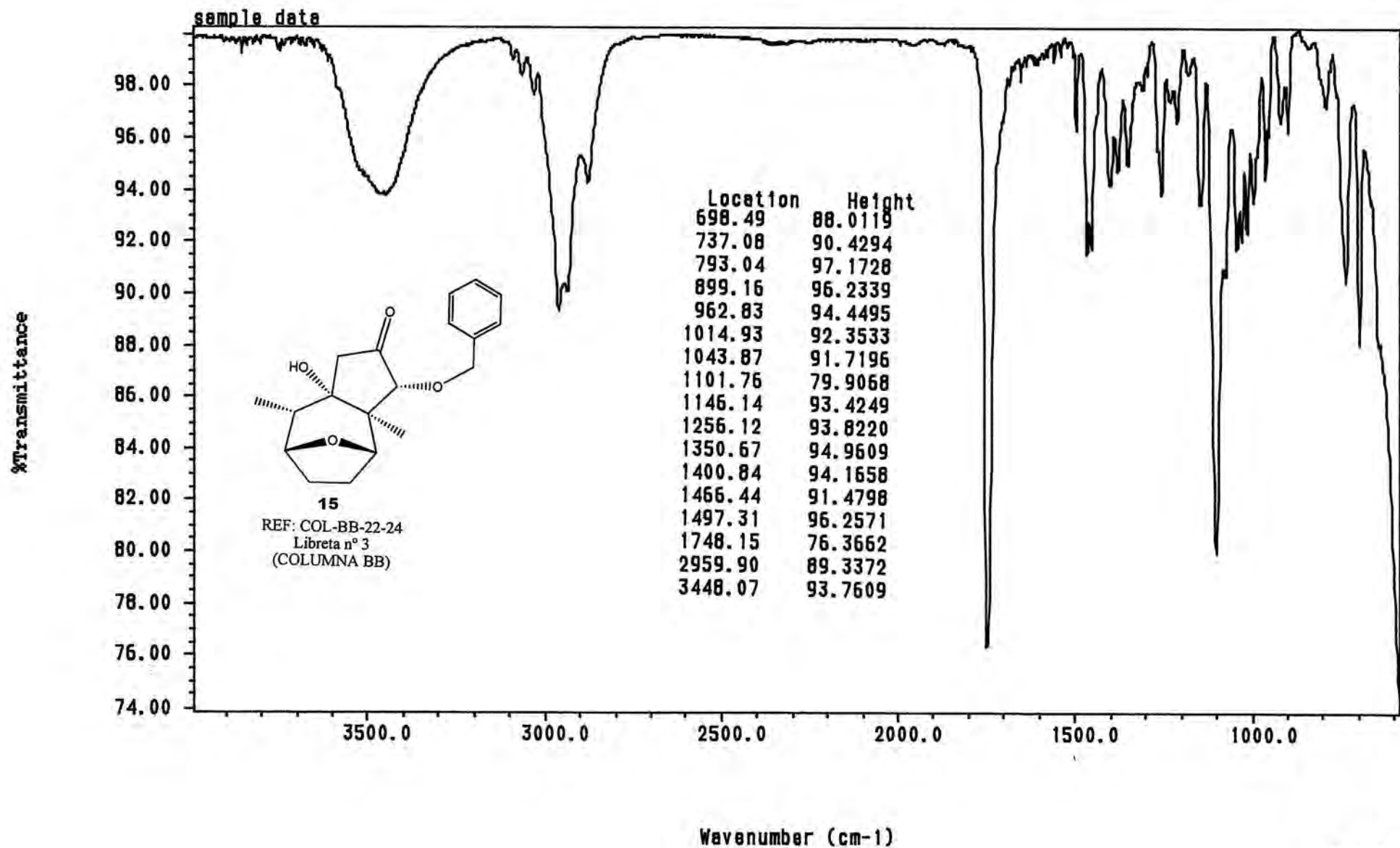


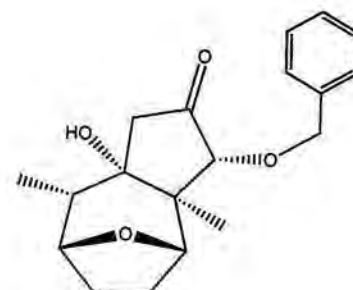
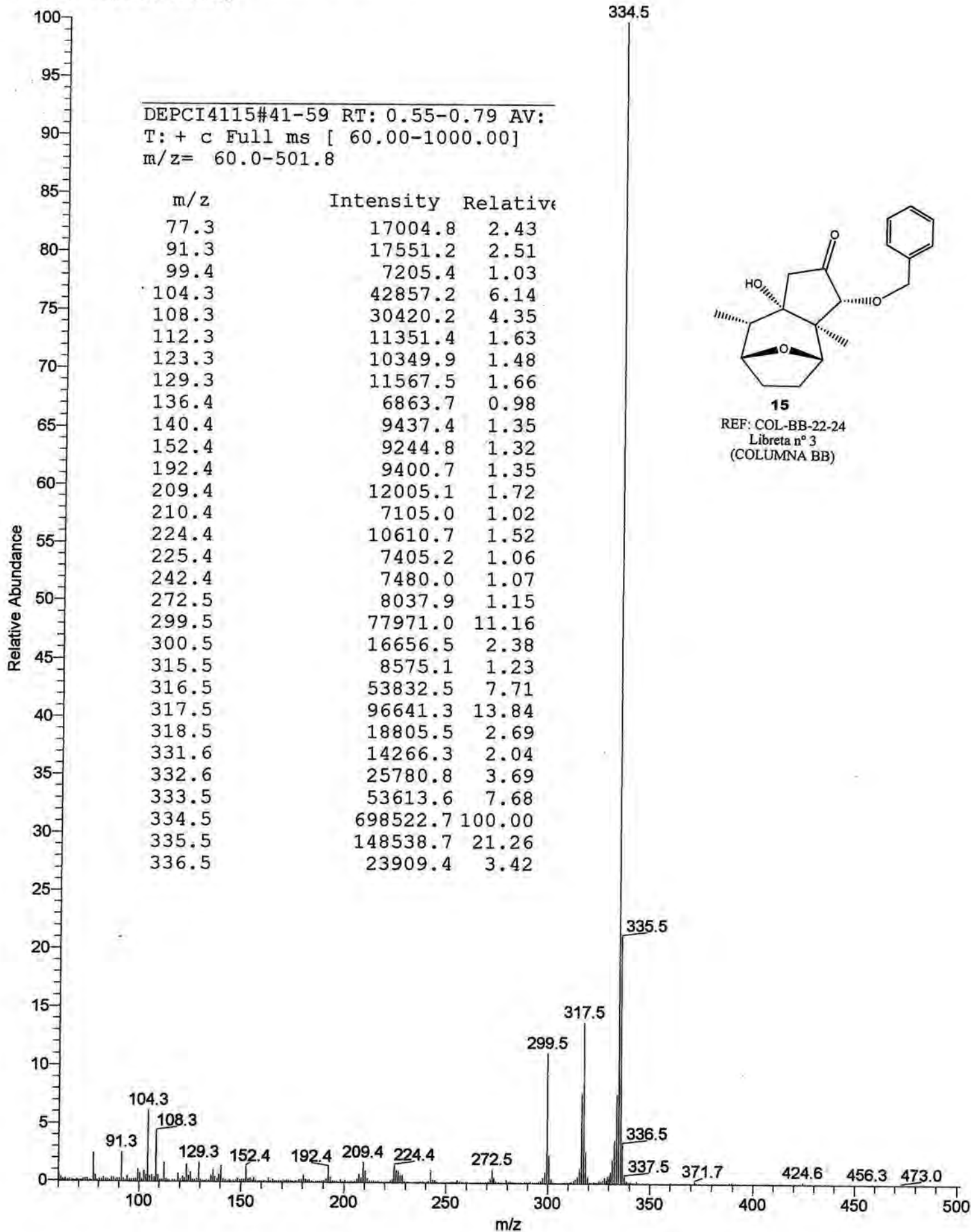
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REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNA BB)

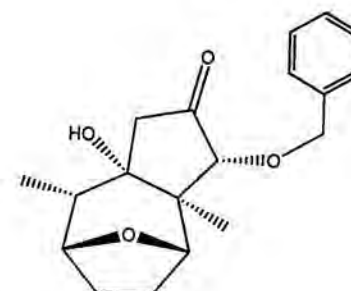
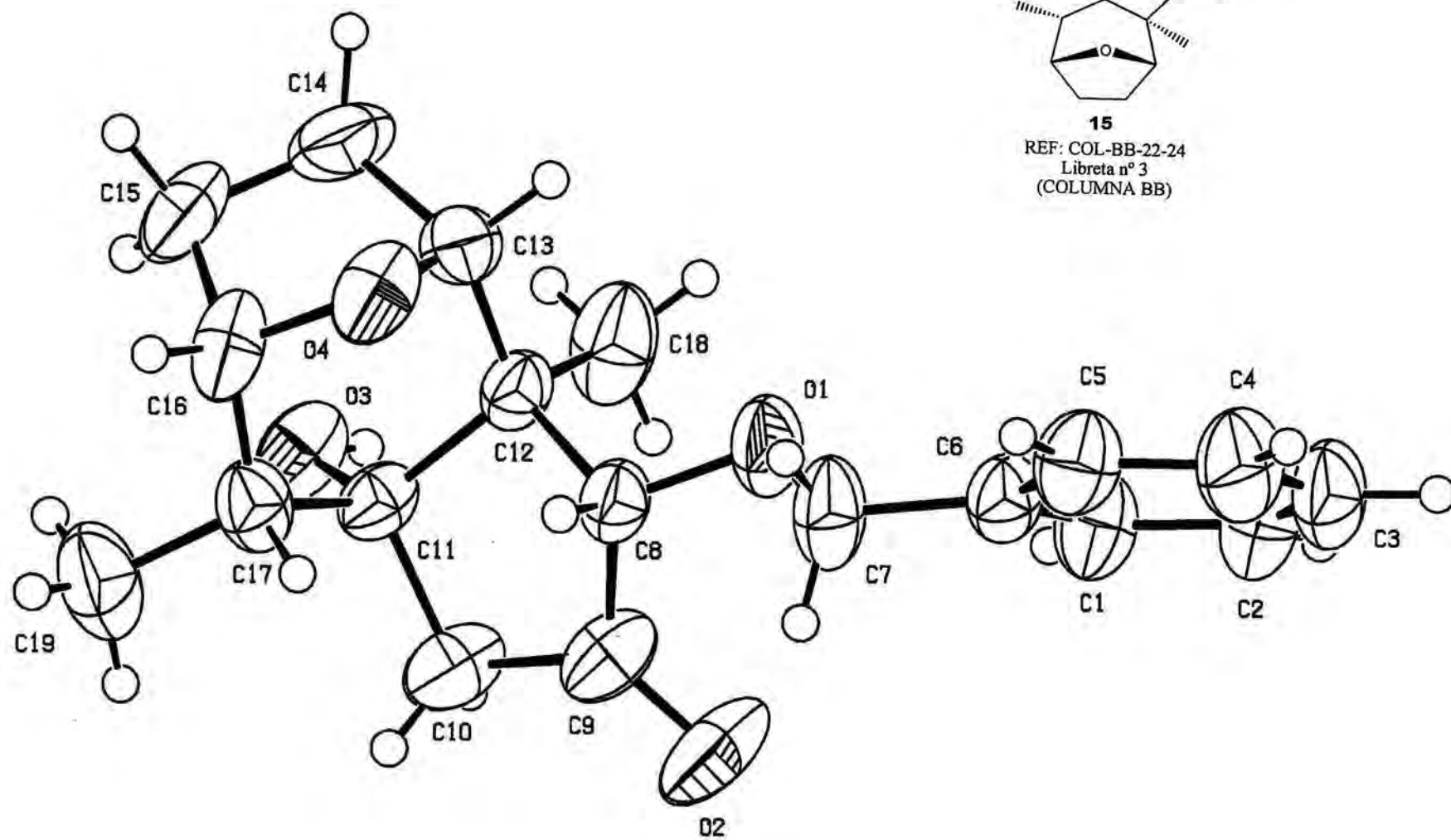


colBB23\_24  
sample data



**15**

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMN BB)

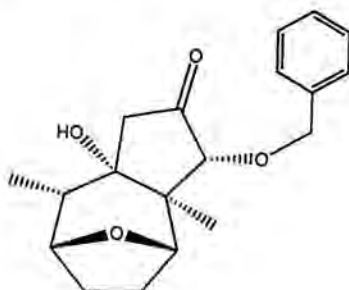


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REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNNA BB)

Table 1. Crystal data and structure refinement for monlc71.

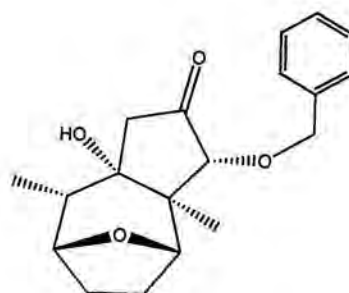
|                                   |                                                                                                    |
|-----------------------------------|----------------------------------------------------------------------------------------------------|
| Identification code               | monlc71                                                                                            |
| Empirical formula                 | C <sub>19</sub> H <sub>24</sub> O <sub>4</sub>                                                     |
| Formula weight                    | 316.38                                                                                             |
| Temperature                       | 293(2) K                                                                                           |
| Wavelength                        | 0.71073 Å                                                                                          |
| Crystal system, space group       | Monoclinic, Cc                                                                                     |
| Unit cell dimensions              | a = 20.125(9) Å    α = 90 °.<br>b = 6.419(2) Å    β = 121.76(4) °.<br>c = 15.359(9) Å    γ = 90 °. |
| Volume                            | 1687.0(14) Å <sup>3</sup>                                                                          |
| Z, Calculated density             | 4, 1.246 Mg/m <sup>3</sup>                                                                         |
| Absorption coefficient            | 0.086 mm <sup>-1</sup>                                                                             |
| F(000)                            | 680                                                                                                |
| Crystal size                      | 0.2 x 0.1 x 0.1 mm                                                                                 |
| Theta range for data collection   | 2.38 to 30.01 °.                                                                                   |
| Limiting indices                  | -27<=h<=28, -9<=k<=8, -14<=l<=21                                                                   |
| Reflections collected / unique    | 4689 / 2450 [R(int) = 0.0658]                                                                      |
| Completeness to theta = 30.01     | 99.8 %                                                                                             |
| Absorption correction             | None                                                                                               |
| Refinement method                 | Full-matrix least-squares on F <sup>2</sup>                                                        |
| Data / restraints / parameters    | 2450 / 2 / 210                                                                                     |
| Goodness-of-fit on F <sup>2</sup> | 1.005                                                                                              |
| Final R indices [I>2σ(I)]         | R1 = 0.0508, wR2 = 0.1034                                                                          |
| R indices (all data)              | R1 = 0.1440, wR2 = 0.1290                                                                          |
| Absolute structure parameter      | -10(10)                                                                                            |
| Largest diff. peak and hole       | 0.131 and -0.175 e.Å <sup>-3</sup>                                                                 |



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Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for monlc71. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

|       | x       | y       | z        | U(eq) |
|-------|---------|---------|----------|-------|
| O(1)  | 2652(1) | 6154(4) | 200(2)   | 62(1) |
| O(2)  | 1819(2) | 9493(5) | 412(2)   | 95(1) |
| O(3)  | 4244(1) | 9674(4) | 3445(2)  | 60(1) |
| O(4)  | 3827(2) | 3924(4) | 2772(2)  | 63(1) |
| C(1)  | 1659(2) | 6653(7) | -1991(3) | 64(1) |
| C(2)  | 1323(2) | 6616(7) | -3052(3) | 70(1) |
| C(3)  | 997(2)  | 4845(8) | -3586(3) | 71(1) |
| C(4)  | 1003(3) | 3093(8) | -3089(3) | 76(1) |
| C(5)  | 1355(2) | 3100(7) | -2030(3) | 66(1) |
| C(6)  | 1663(2) | 4924(6) | -1480(3) | 52(1) |
| C(7)  | 1951(2) | 4931(7) | -359(3)  | 66(1) |
| C(8)  | 2793(2) | 6748(6) | 1162(2)  | 47(1) |
| C(9)  | 2381(2) | 8717(6) | 1156(3)  | 62(1) |
| C(10) | 2821(2) | 9660(6) | 2209(3)  | 66(1) |
| C(11) | 3586(2) | 8370(5) | 2808(2)  | 45(1) |
| C(12) | 3662(2) | 7257(6) | 1960(2)  | 46(1) |
| C(13) | 4154(2) | 5297(6) | 2362(3)  | 57(1) |
| C(14) | 4995(2) | 5663(8) | 3279(3)  | 82(1) |
| C(15) | 4909(3) | 5395(7) | 4203(3)  | 75(1) |
| C(16) | 4043(3) | 4903(6) | 3733(3)  | 64(1) |
| C(17) | 3519(2) | 6776(6) | 3504(3)  | 55(1) |
| C(18) | 3975(2) | 8718(8) | 1476(3)  | 80(1) |
| C(19) | 3628(3) | 7728(9) | 4481(3)  | 86(2) |

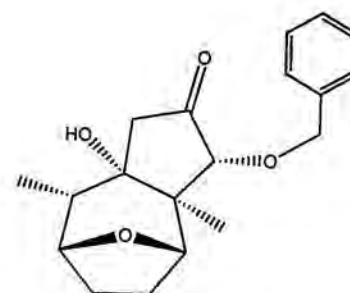


**15**

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNNA BB)

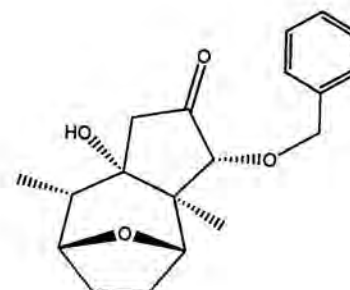
Table 3. Bond lengths [Å] and angles [°] for monlc71.

|                  |          |
|------------------|----------|
| O(1)-C(8)        | 1.403(4) |
| O(1)-C(7)        | 1.438(4) |
| O(2)-C(9)        | 1.214(4) |
| O(3)-C(11)       | 1.431(4) |
| O(3)-H(3)        | 0.8200   |
| O(4)-C(13)       | 1.430(5) |
| O(4)-C(16)       | 1.444(5) |
| C(1)-C(6)        | 1.357(5) |
| C(1)-C(2)        | 1.398(5) |
| C(1)-H(1)        | 0.9300   |
| C(2)-C(3)        | 1.352(6) |
| C(2)-H(2)        | 0.9300   |
| C(3)-C(4)        | 1.356(7) |
| C(3)-H(3A)       | 0.9300   |
| C(4)-C(5)        | 1.391(6) |
| C(4)-H(4)        | 0.9300   |
| C(5)-C(6)        | 1.385(5) |
| C(5)-H(5)        | 0.9300   |
| C(6)-C(7)        | 1.498(5) |
| C(7)-H(7A)       | 0.9700   |
| C(7)-H(7B)       | 0.9700   |
| C(8)-C(9)        | 1.509(5) |
| C(8)-C(12)       | 1.552(5) |
| C(8)-H(8)        | 0.9800   |
| C(9)-C(10)       | 1.502(6) |
| C(10)-C(11)      | 1.553(5) |
| C(10)-H(10A)     | 0.9700   |
| C(10)-H(10B)     | 0.9700   |
| C(11)-C(17)      | 1.535(5) |
| C(11)-C(12)      | 1.562(5) |
| C(12)-C(13)      | 1.518(5) |
| C(12)-C(18)      | 1.525(5) |
| C(13)-C(14)      | 1.547(5) |
| C(13)-H(13)      | 0.9800   |
| C(14)-C(15)      | 1.526(7) |
| C(14)-H(14A)     | 0.9700   |
| C(14)-H(14B)     | 0.9700   |
| C(15)-C(16)      | 1.528(7) |
| C(15)-H(15A)     | 0.9700   |
| C(15)-H(15B)     | 0.9700   |
| C(16)-C(17)      | 1.513(6) |
| C(16)-H(16)      | 0.9800   |
| C(17)-C(19)      | 1.525(5) |
| C(17)-H(17)      | 0.9800   |
| C(18)-H(18A)     | 0.9600   |
| C(18)-H(18B)     | 0.9600   |
| C(18)-H(18C)     | 0.9600   |
| C(19)-H(19A)     | 0.9600   |
| C(19)-H(19B)     | 0.9600   |
| C(19)-H(19C)     | 0.9600   |
| C(8)-O(1)-C(7)   | 111.6(3) |
| C(11)-O(3)-H(3)  | 109.5    |
| C(13)-O(4)-C(16) | 102.8(3) |
| C(6)-C(1)-C(2)   | 120.9(4) |
| C(6)-C(1)-H(1)   | 119.6    |
| C(2)-C(1)-H(1)   | 119.6    |

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REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)

|                     |          |
|---------------------|----------|
| C(3)-C(2)-C(1)      | 120.2(4) |
| C(3)-C(2)-H(2)      | 119.9    |
| C(1)-C(2)-H(2)      | 119.9    |
| C(2)-C(3)-C(4)      | 119.9(4) |
| C(2)-C(3)-H(3A)     | 120.1    |
| C(4)-C(3)-H(3A)     | 120.1    |
| C(3)-C(4)-C(5)      | 120.3(4) |
| C(3)-C(4)-H(4)      | 119.9    |
| C(5)-C(4)-H(4)      | 119.9    |
| C(6)-C(5)-C(4)      | 120.3(4) |
| C(6)-C(5)-H(5)      | 119.8    |
| C(4)-C(5)-H(5)      | 119.8    |
| C(1)-C(6)-C(5)      | 118.3(3) |
| C(1)-C(6)-C(7)      | 122.9(3) |
| C(5)-C(6)-C(7)      | 118.8(4) |
| O(1)-C(7)-C(6)      | 110.7(3) |
| O(1)-C(7)-H(7A)     | 109.5    |
| C(6)-C(7)-H(7A)     | 109.5    |
| O(1)-C(7)-H(7B)     | 109.5    |
| C(6)-C(7)-H(7B)     | 109.5    |
| H(7A)-C(7)-H(7B)    | 108.1    |
| O(1)-C(8)-C(9)      | 114.6(3) |
| O(1)-C(8)-C(12)     | 114.1(3) |
| C(9)-C(8)-C(12)     | 102.4(3) |
| O(1)-C(8)-H(8)      | 108.5    |
| C(9)-C(8)-H(8)      | 108.5    |
| C(12)-C(8)-H(8)     | 108.5    |
| O(2)-C(9)-C(10)     | 125.2(4) |
| O(2)-C(9)-C(8)      | 125.9(4) |
| C(10)-C(9)-C(8)     | 108.7(3) |
| C(9)-C(10)-C(11)    | 105.2(3) |
| C(9)-C(10)-H(10A)   | 110.7    |
| C(11)-C(10)-H(10A)  | 110.7    |
| C(9)-C(10)-H(10B)   | 110.7    |
| C(11)-C(10)-H(10B)  | 110.7    |
| H(10A)-C(10)-H(10B) | 108.8    |
| O(3)-C(11)-C(17)    | 107.6(3) |
| O(3)-C(11)-C(10)    | 111.5(3) |
| C(17)-C(11)-C(10)   | 110.2(3) |
| O(3)-C(11)-C(12)    | 112.1(3) |
| C(17)-C(11)-C(12)   | 111.0(3) |
| C(10)-C(11)-C(12)   | 104.5(3) |
| C(13)-C(12)-C(18)   | 111.0(3) |
| C(13)-C(12)-C(8)    | 111.7(3) |
| C(18)-C(12)-C(8)    | 109.1(3) |
| C(13)-C(12)-C(11)   | 111.7(3) |
| C(18)-C(12)-C(11)   | 111.9(3) |
| C(8)-C(12)-C(11)    | 101.0(3) |
| O(4)-C(13)-C(12)    | 109.7(3) |
| O(4)-C(13)-C(14)    | 103.2(3) |
| C(12)-C(13)-C(14)   | 114.3(3) |
| O(4)-C(13)-H(13)    | 109.8    |
| C(12)-C(13)-H(13)   | 109.8    |
| C(14)-C(13)-H(13)   | 109.8    |
| C(15)-C(14)-C(13)   | 103.0(4) |
| C(15)-C(14)-H(14A)  | 111.2    |
| C(13)-C(14)-H(14A)  | 111.2    |
| C(15)-C(14)-H(14B)  | 111.2    |
| C(13)-C(14)-H(14B)  | 111.2    |
| H(14A)-C(14)-H(14B) | 109.1    |
| C(14)-C(15)-C(16)   | 104.1(3) |



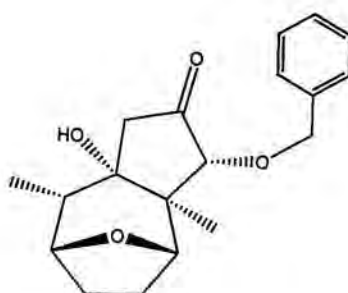
**15**

REF: COL-BB-22-24  
Libreta nº 3  
(COLUMNNA BB)

|                     |          |
|---------------------|----------|
| C(14)-C(15)-H(15A)  | 110.9    |
| C(16)-C(15)-H(15A)  | 110.9    |
| C(14)-C(15)-H(15B)  | 110.9    |
| C(16)-C(15)-H(15B)  | 110.9    |
| H(15A)-C(15)-H(15B) | 109.0    |
| O(4)-C(16)-C(17)    | 108.1(3) |
| O(4)-C(16)-C(15)    | 103.1(4) |
| C(17)-C(16)-C(15)   | 115.3(3) |
| O(4)-C(16)-H(16)    | 110.0    |
| C(17)-C(16)-H(16)   | 110.0    |
| C(15)-C(16)-H(16)   | 110.0    |
| C(16)-C(17)-C(19)   | 111.6(3) |
| C(16)-C(17)-C(11)   | 112.7(3) |
| C(19)-C(17)-C(11)   | 113.4(3) |
| C(16)-C(17)-H(17)   | 106.1    |
| C(19)-C(17)-H(17)   | 106.1    |
| C(11)-C(17)-H(17)   | 106.1    |
| C(12)-C(18)-H(18A)  | 109.5    |
| C(12)-C(18)-H(18B)  | 109.5    |
| H(18A)-C(18)-H(18B) | 109.5    |
| C(12)-C(18)-H(18C)  | 109.5    |
| H(18A)-C(18)-H(18C) | 109.5    |
| H(18B)-C(18)-H(18C) | 109.5    |
| C(17)-C(19)-H(19A)  | 109.5    |
| C(17)-C(19)-H(19B)  | 109.5    |
| H(19A)-C(19)-H(19B) | 109.5    |
| C(17)-C(19)-H(19C)  | 109.5    |
| H(19A)-C(19)-H(19C) | 109.5    |
| H(19B)-C(19)-H(19C) | 109.5    |

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Symmetry transformations used to generate equivalent atoms:

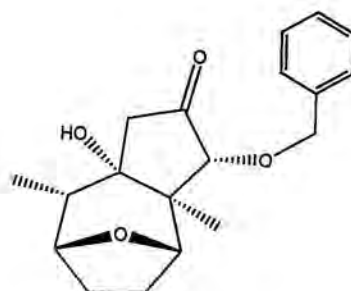


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REF: COL-BB-22-24  
 Libreta nº 3  
 (COLUMNNA BB)

Table 5. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for monlc71.  
The anisotropic displacement factor exponent takes the form:  
 $-2 \pi^2 [ h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12} ]$

|       | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|-------|----------|----------|----------|----------|----------|----------|
| O(1)  | 51(1)    | 90(2)    | 37(1)    | -8(1)    | 17(1)    | -18(1)   |
| O(2)  | 58(2)    | 92(2)    | 76(2)    | 12(2)    | -5(2)    | 18(2)    |
| O(3)  | 53(2)    | 43(1)    | 58(2)    | -4(1)    | 11(1)    | -11(1)   |
| O(4)  | 72(2)    | 35(1)    | 53(2)    | -5(1)    | 12(1)    | 3(1)     |
| C(1)  | 67(2)    | 58(2)    | 49(2)    | -11(2)   | 17(2)    | -17(2)   |
| C(2)  | 67(2)    | 80(3)    | 47(2)    | 6(2)     | 20(2)    | -15(2)   |
| C(3)  | 60(2)    | 102(4)   | 39(2)    | -9(2)    | 18(2)    | -18(2)   |
| C(4)  | 73(3)    | 84(3)    | 53(2)    | -24(2)   | 20(2)    | -22(2)   |
| C(5)  | 61(2)    | 68(3)    | 56(2)    | -5(2)    | 22(2)    | -15(2)   |
| C(6)  | 44(2)    | 62(2)    | 42(2)    | -5(2)    | 17(2)    | -9(2)    |
| C(7)  | 63(2)    | 84(3)    | 38(2)    | -10(2)   | 18(2)    | -27(2)   |
| C(8)  | 43(2)    | 56(2)    | 36(2)    | 0(2)     | 16(1)    | -3(2)    |
| C(9)  | 43(2)    | 56(2)    | 62(2)    | 5(2)     | 12(2)    | -3(2)    |
| C(10) | 49(2)    | 52(2)    | 70(3)    | -7(2)    | 12(2)    | 7(2)     |
| C(11) | 38(2)    | 38(2)    | 45(2)    | -5(2)    | 12(1)    | -1(2)    |
| C(12) | 36(2)    | 55(2)    | 39(2)    | 1(2)     | 14(1)    | -3(2)    |
| C(13) | 49(2)    | 66(2)    | 48(2)    | -14(2)   | 19(2)    | 6(2)     |
| C(14) | 41(2)    | 99(3)    | 75(3)    | -16(3)   | 10(2)    | 19(2)    |
| C(15) | 67(3)    | 64(3)    | 50(2)    | 2(2)     | 0(2)     | 21(2)    |
| C(16) | 77(3)    | 52(2)    | 38(2)    | 3(2)     | 14(2)    | -5(2)    |
| C(17) | 55(2)    | 63(2)    | 44(2)    | -13(2)   | 24(2)    | -12(2)   |
| C(18) | 69(3)    | 104(4)   | 57(2)    | 11(3)    | 25(2)    | -27(3)   |
| C(19) | 90(3)    | 114(4)   | 56(2)    | -22(3)   | 41(2)    | -20(3)   |

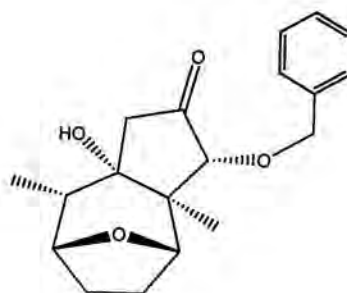


15

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMN A BB)

Table 6. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for monlc71.

|        | x    | y     | z     | U(eq) |
|--------|------|-------|-------|-------|
| H(3)   | 4295 | 10534 | 3090  | 90    |
| H(1)   | 1882 | 7878  | -1631 | 77    |
| H(2)   | 1323 | 7814  | -3393 | 84    |
| H(3A)  | 770  | 4828  | -4293 | 85    |
| H(4)   | 771  | 1882  | -3457 | 92    |
| H(5)   | 1383 | 1874  | -1690 | 79    |
| H(7A)  | 1551 | 5501  | -257  | 79    |
| H(7B)  | 2057 | 3513  | -102  | 79    |
| H(8)   | 2626 | 5612  | 1430  | 57    |
| H(10A) | 2519 | 9561  | 2533  | 79    |
| H(10B) | 2936 | 11115 | 2176  | 79    |
| H(13)  | 4172 | 4601  | 1806  | 69    |
| H(14A) | 5355 | 4646  | 3287  | 98    |
| H(14B) | 5177 | 7052  | 3260  | 98    |
| H(15A) | 5053 | 6662  | 4606  | 90    |
| H(15B) | 5234 | 4261  | 4636  | 90    |
| H(16)  | 3977 | 3919  | 4170  | 76    |
| H(17)  | 2982 | 6250  | 3115  | 66    |
| H(18A) | 3995 | 7996  | 942   | 121   |
| H(18B) | 4491 | 9172  | 1988  | 121   |
| H(18C) | 3637 | 9906  | 1192  | 121   |
| H(19A) | 3519 | 6697  | 4841  | 128   |
| H(19B) | 3277 | 8883  | 4308  | 128   |
| H(19C) | 4157 | 8204  | 4908  | 128   |



15

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNA BB)

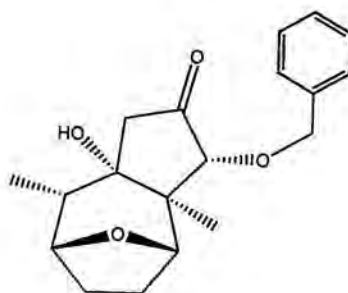
Table 7. Hydrogen bonds for monlc71 [Å and °].

| D    | --H    | ..A    |           | d(D-H) | d(H...A) | d(D...A) | <(DHA) |
|------|--------|--------|-----------|--------|----------|----------|--------|
| O(3) | --H(3) | ..O(4) | [1565.01] | 0.82   | 2.32     | 2.881(4) | 126    |

Translation of ARU-code to Equivalent Position Code

[ 1565. ] = x,1+y,z

|   |       |   |       |       |       |       |   |
|---|-------|---|-------|-------|-------|-------|---|
| - | C(8)  | - | O(1)  | C(9)  | C(12) | H(8)  | S |
| - | C(12) | - | C(8)  | C(11) | C(13) | C(18) | R |
| - | C(13) | - | O(4)  | C(12) | C(14) | H(13) | R |
| - | C(16) | - | O(4)  | C(15) | C(17) | H(16) | S |
| - | C(17) | - | C(11) | C(16) | C(19) | H(17) | R |



**15**

REF: COL-BB-22-24  
Libreta n° 3  
(COLUMNNA BB)

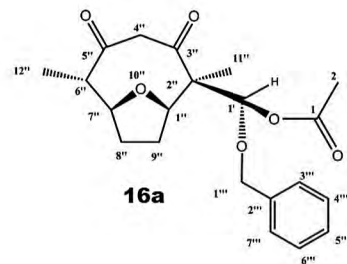
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

H1 / s2pu1 / Mercury-400 qui  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

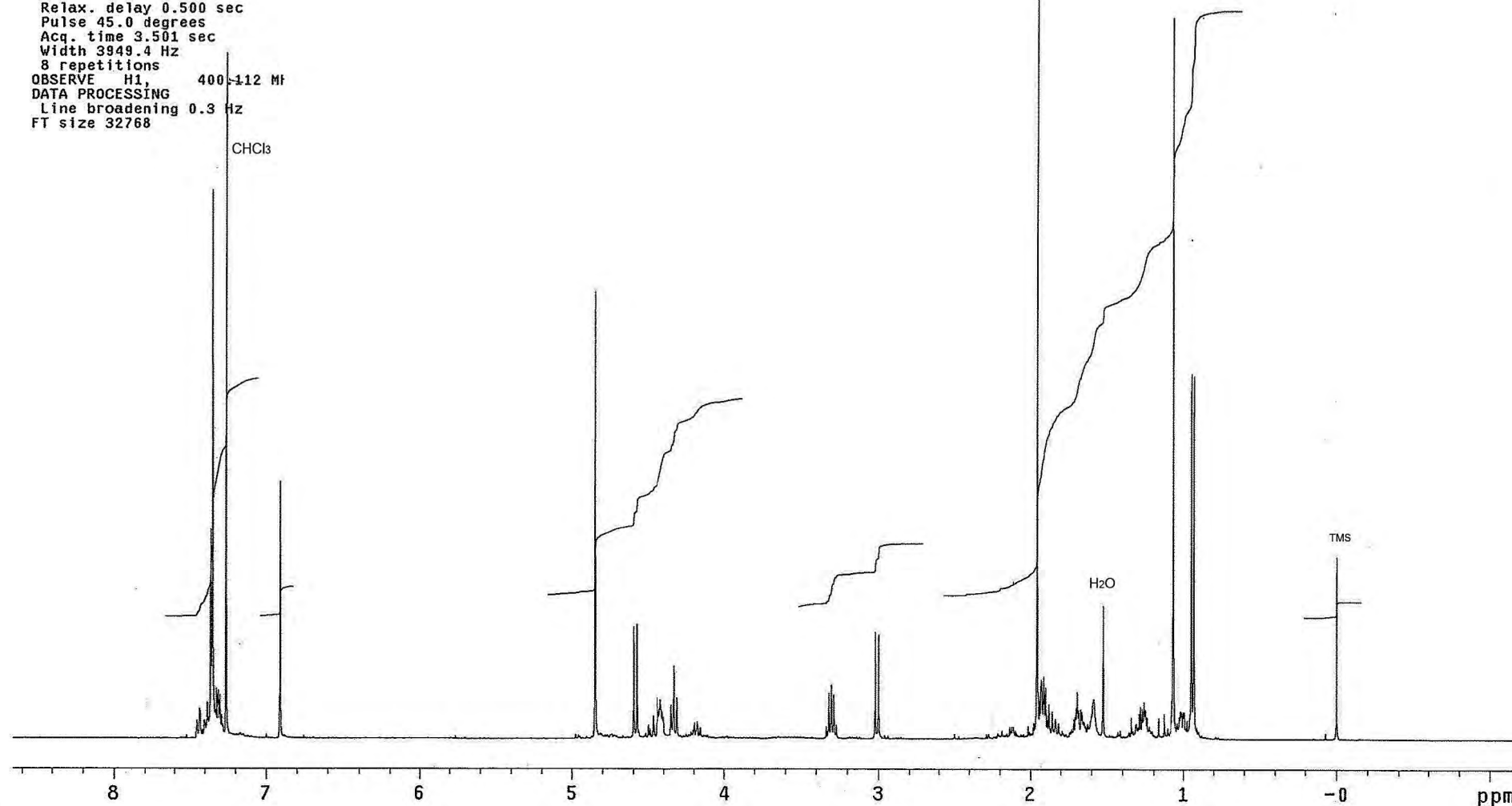
Pulse Sequence: s2pu1  
\*\*\*\*\*@\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.501 sec  
Width 3949.4 Hz  
8 repetitions  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 32768

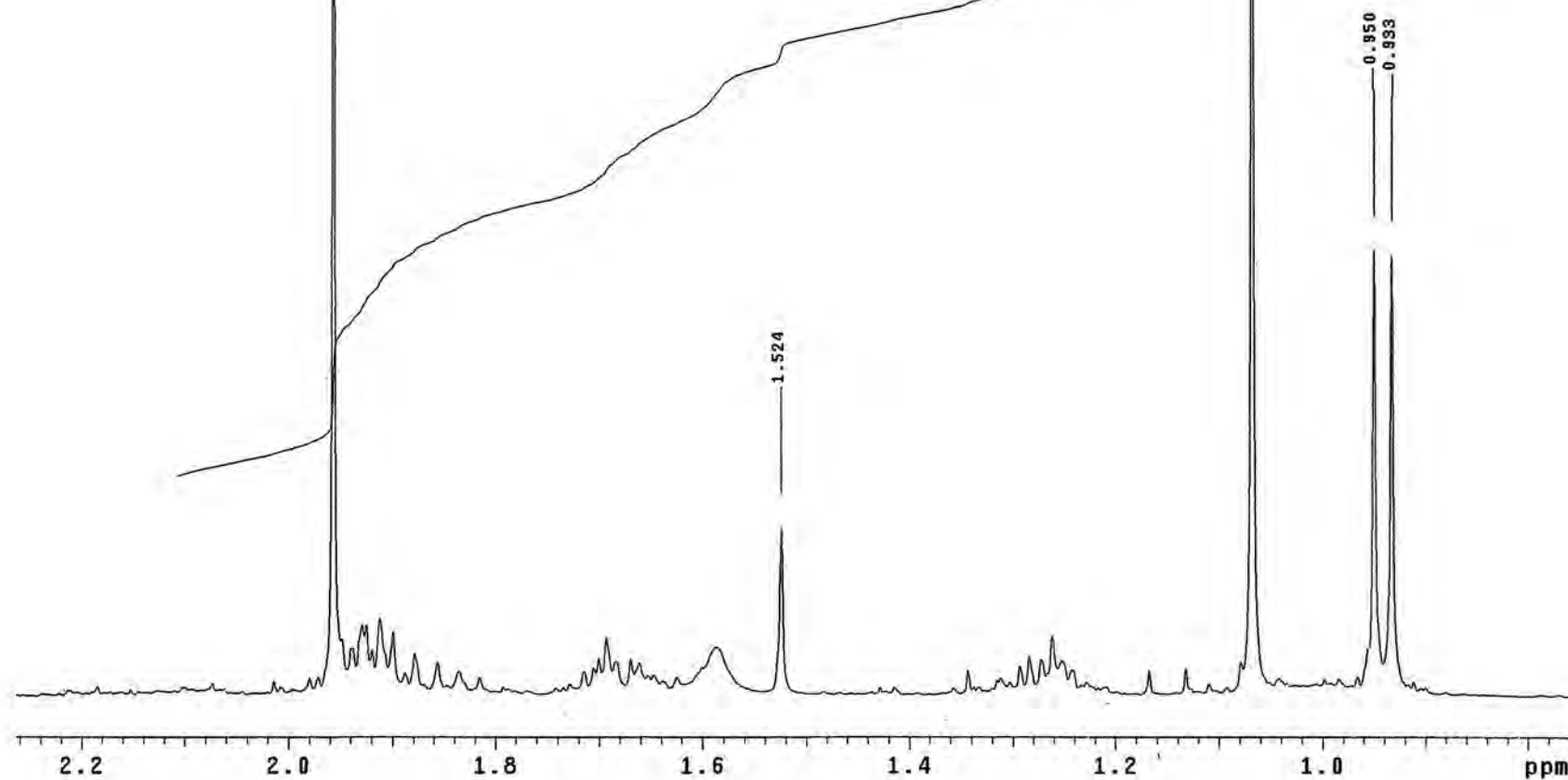
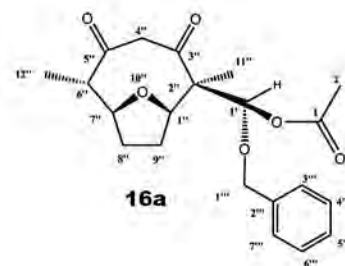


| INDEX | FREQUENCY | PPM    | HEIGHT |
|-------|-----------|--------|--------|
| 1     | 2945.694  | 7.362  | 34.4   |
| 2     | 2941.355  | 7.351  | 90.4   |
| 3     | 2905.919  | 7.263  | 112.8  |
| 4     | 2764.661  | 6.910  | 42.2   |
| 5     | 1940.253  | 4.849  | 73.6   |
| 6     | 846.345   | 2.115  | 25.9   |
| 7     | 783.188   | 1.957  | 151.2  |
| 8     | 427.632   | 1.069  | 118.8  |
| 9     | 380.385   | 0.951  | 60.2   |
| 10    | 373.635   | 0.934  | 59.8   |
| 11    | -0.000    | -0.000 | 29.9   |



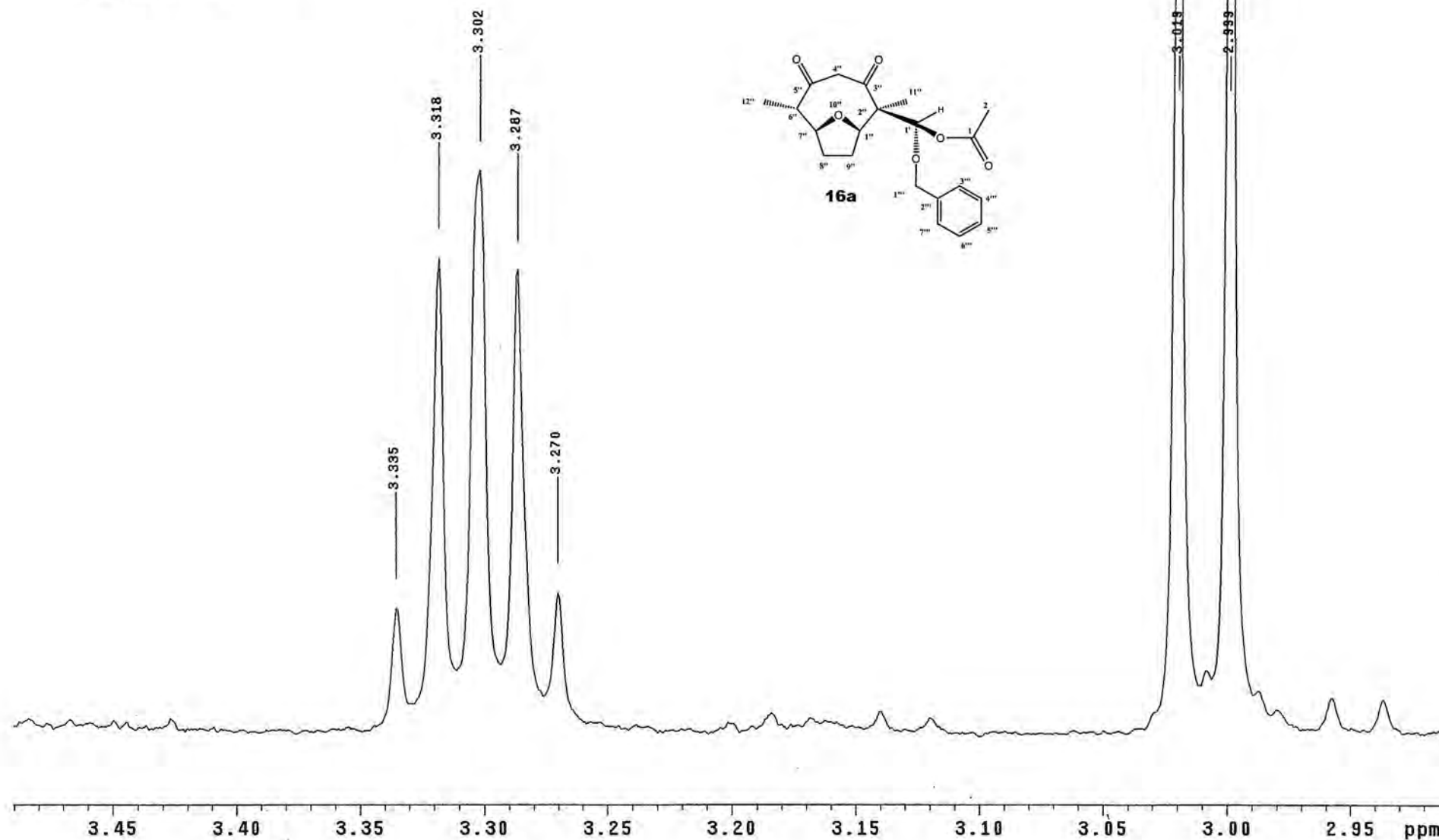
H1 / s2pu1 / Mercury-400.ql  
cdcl3/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

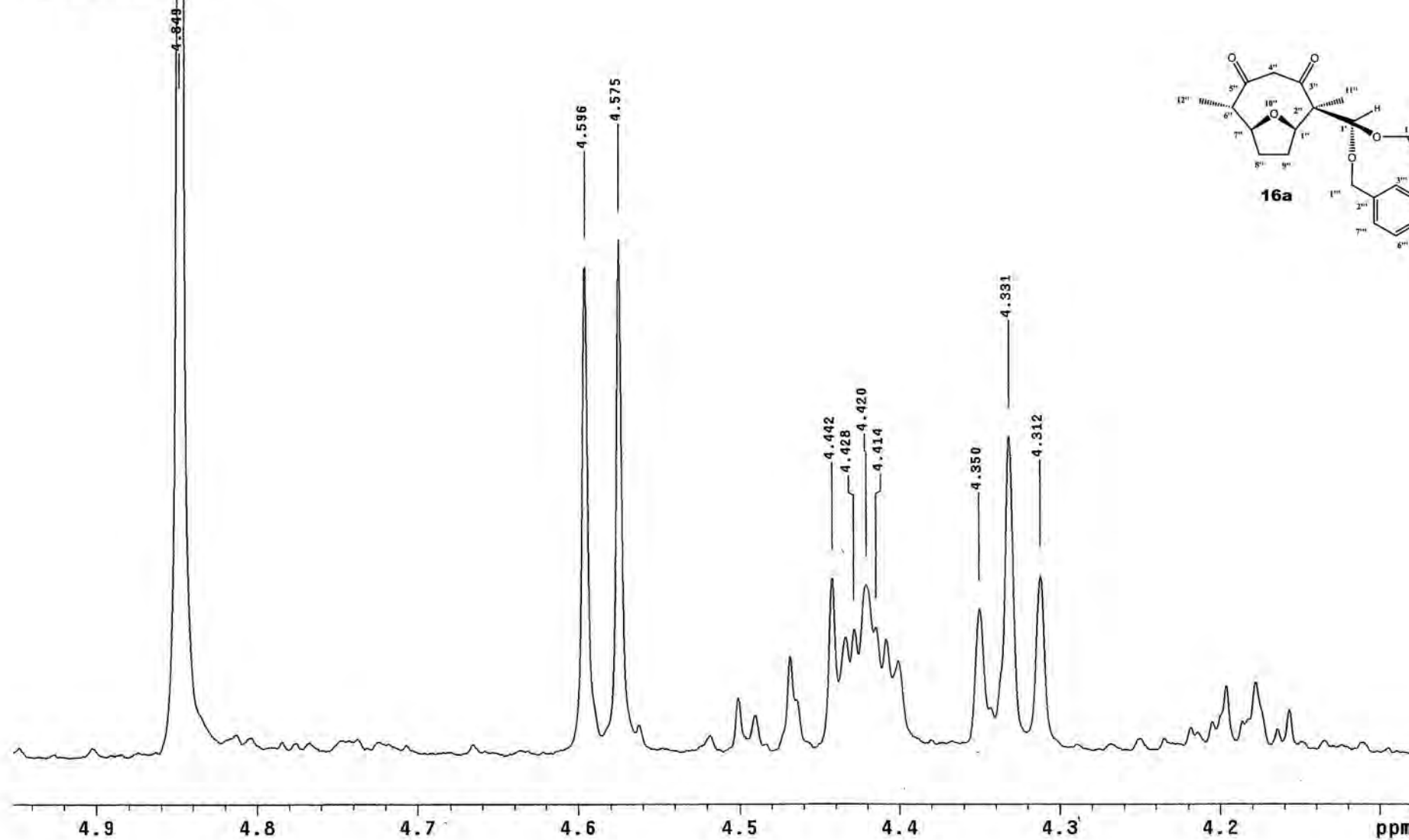
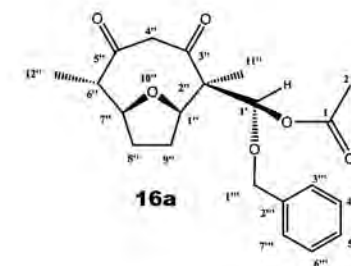


H1 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C / N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO  
Sist automatic  
Pulse Sequence: s2pu1



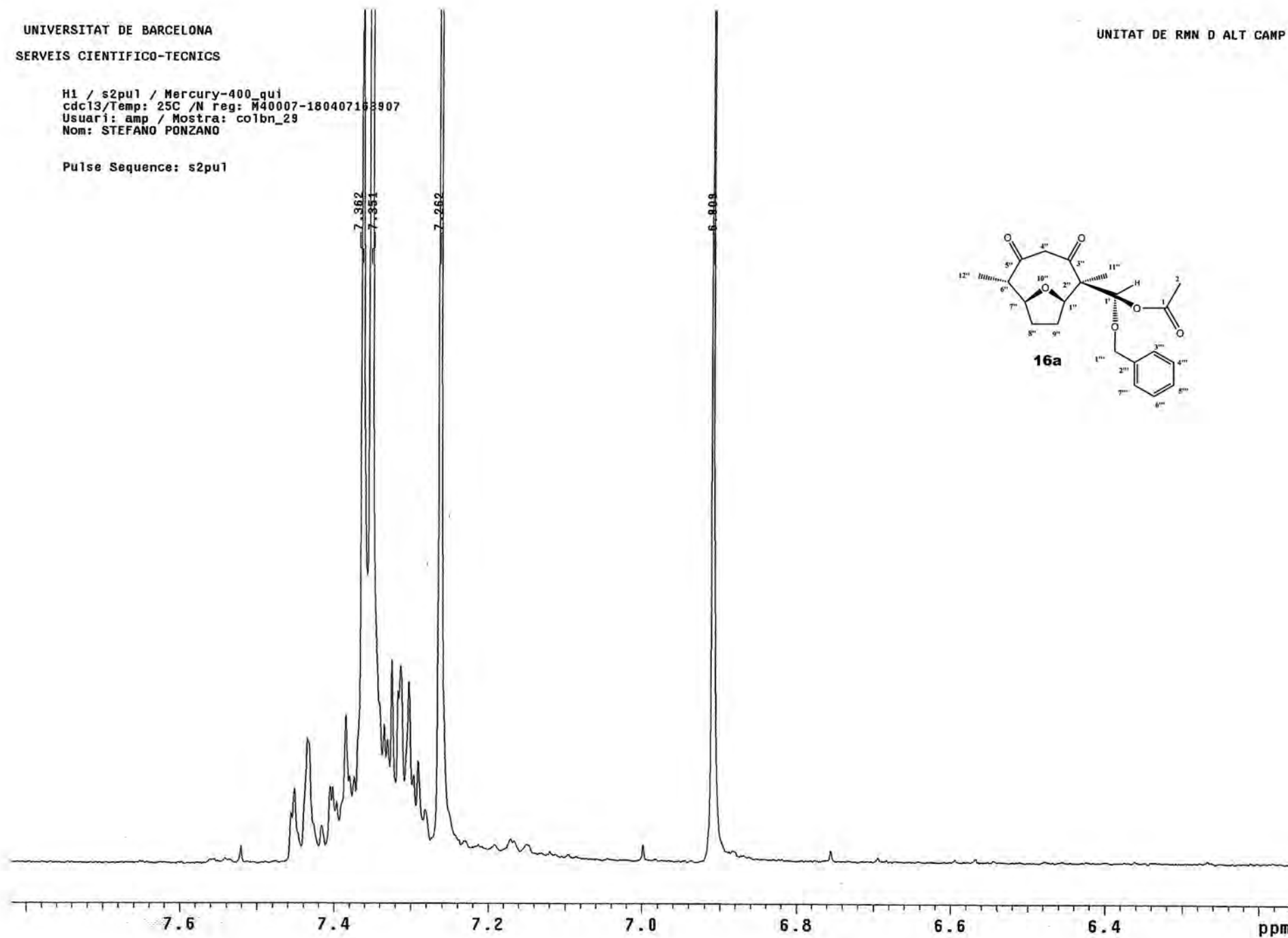
UNIVERSITAT DE BARCELONA

SERVEIS CIENTIFICO-TECNICS

H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407188907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

UNITAT DE RMN D ALT CAMP



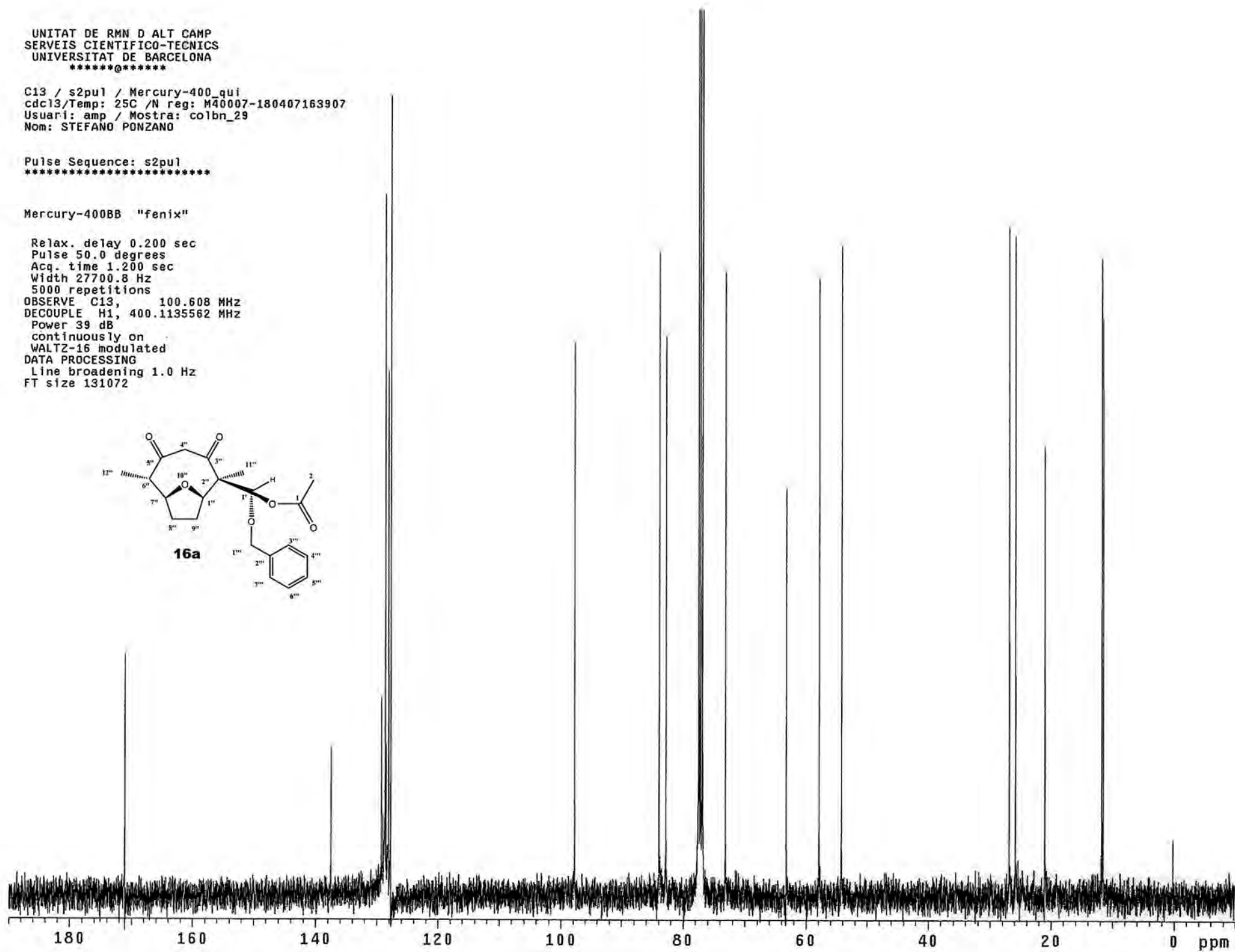
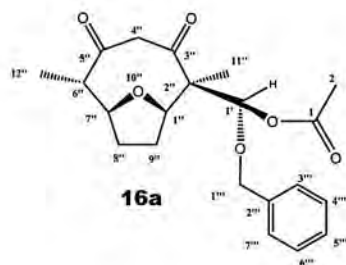
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFIC0-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

C13 / s2pul / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pul  
\*\*\*\*\*

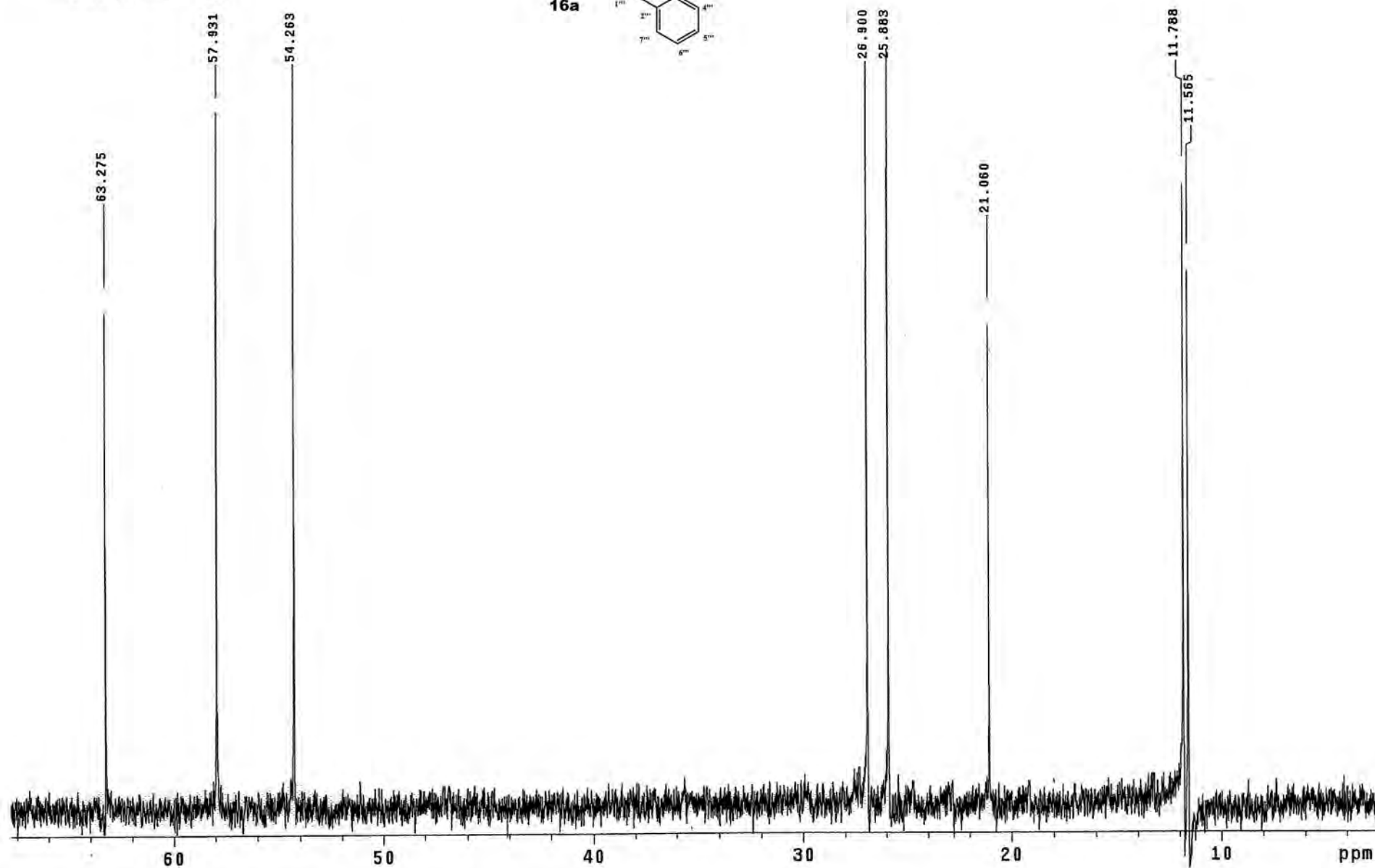
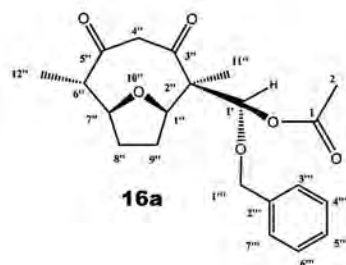
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
5000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072



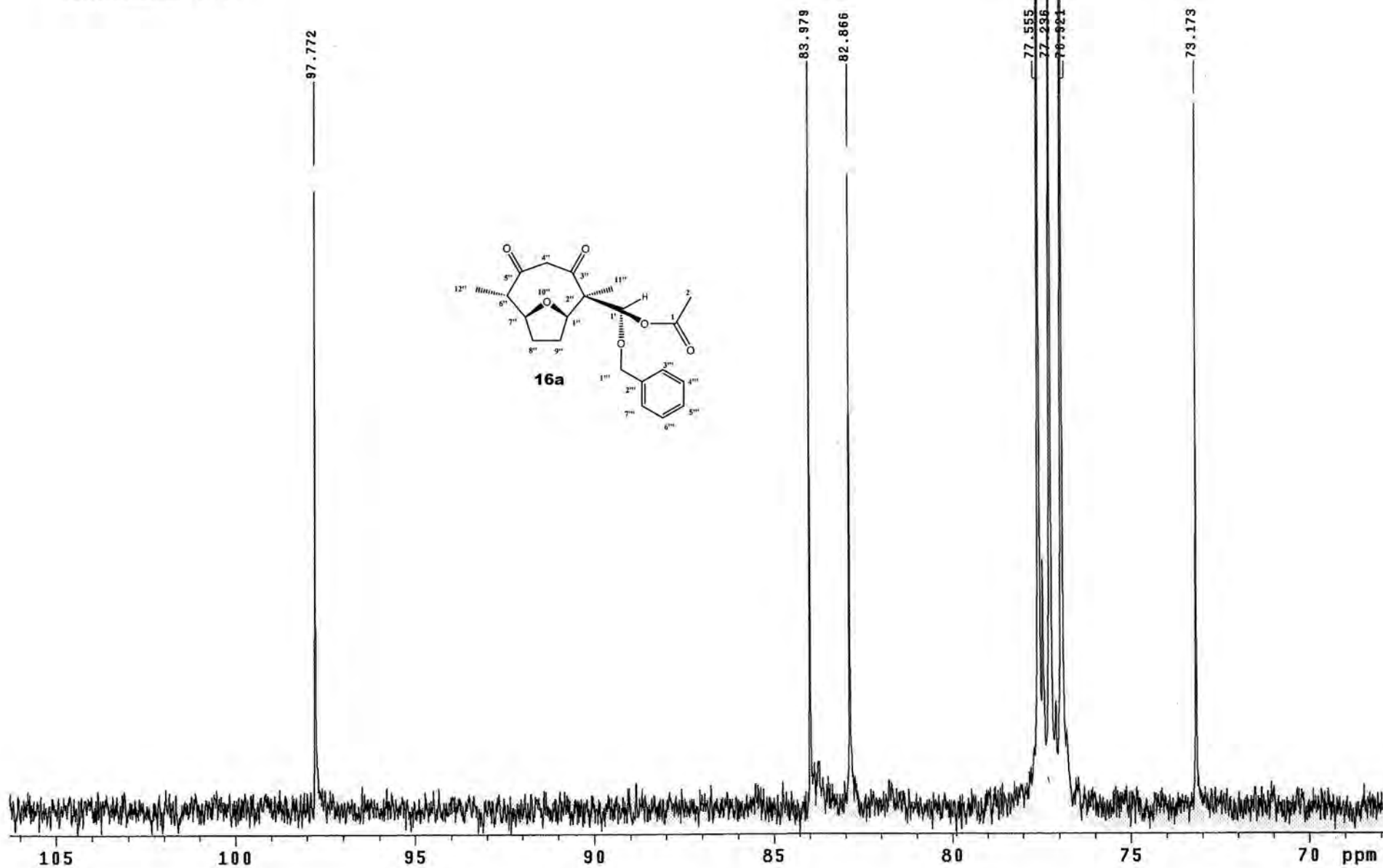
C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



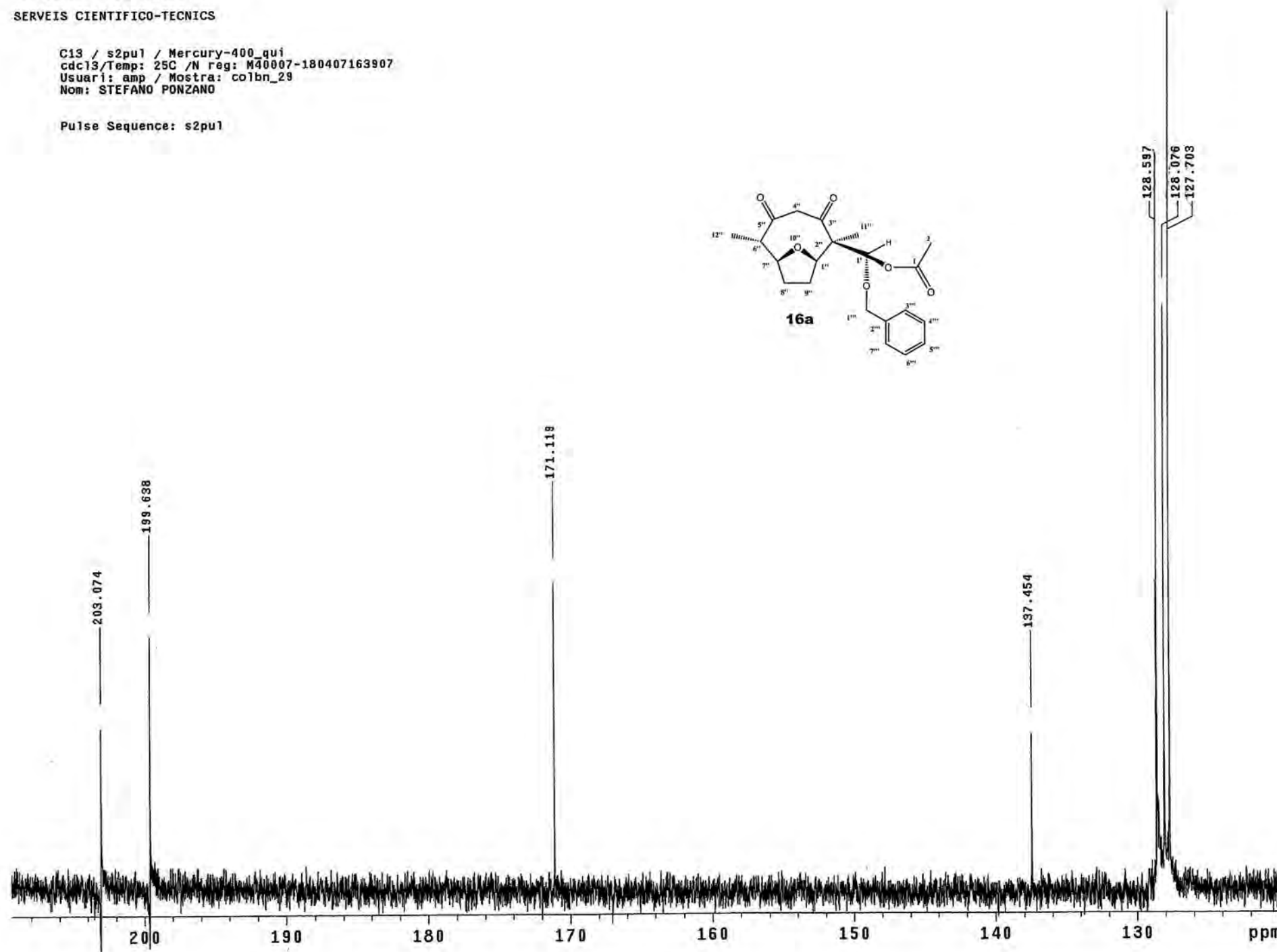
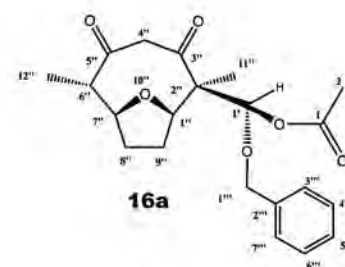
C13 / s2pu1 / Mercury-400\_qu1  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1



C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1

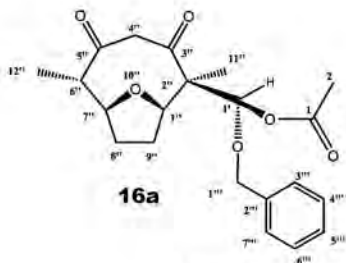


H1 / gC0SY / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

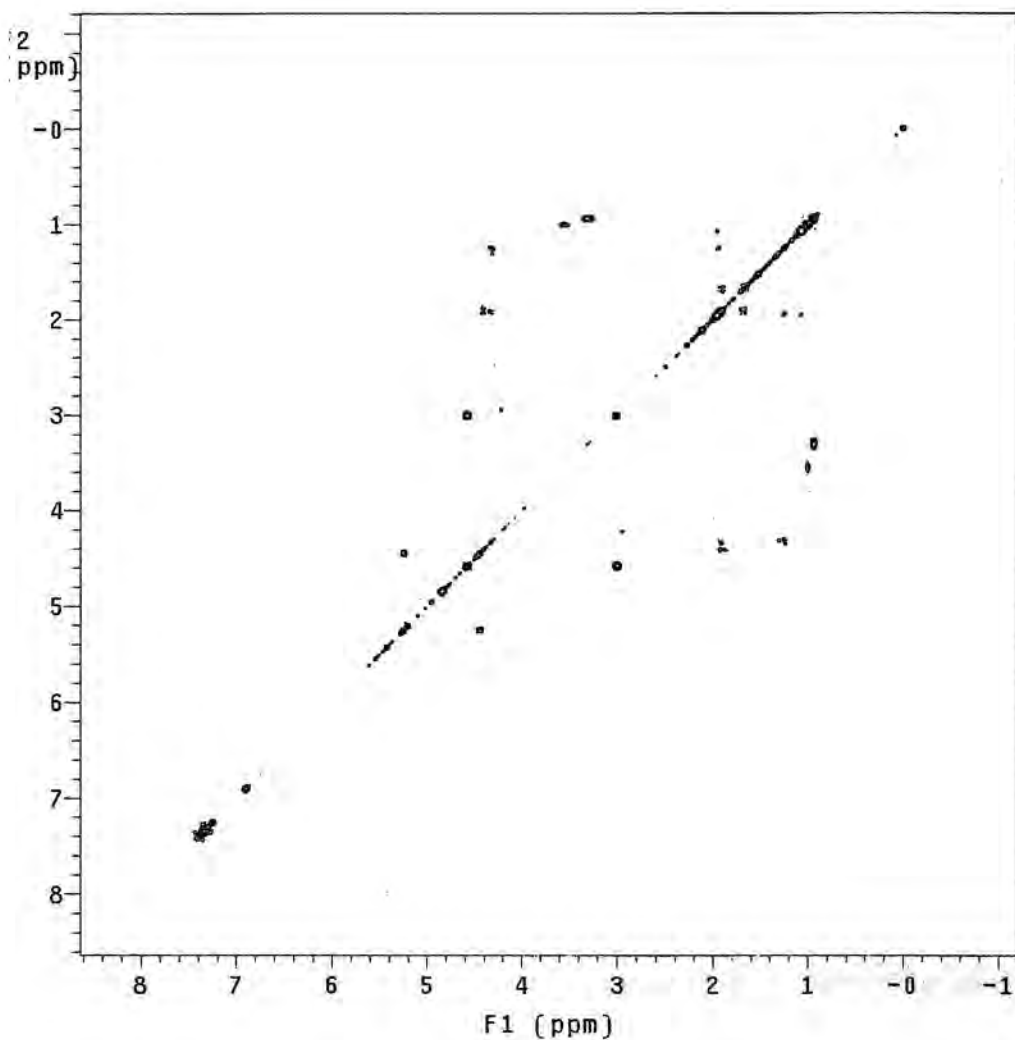
Pulse Sequence: gC0SY  
\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3937.0 Hz  
2D Width 3937.0 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.065 sec  
FT size 2048 x 2048



COL BN-29  
Libreta n° 3  
(COLUMN BN)



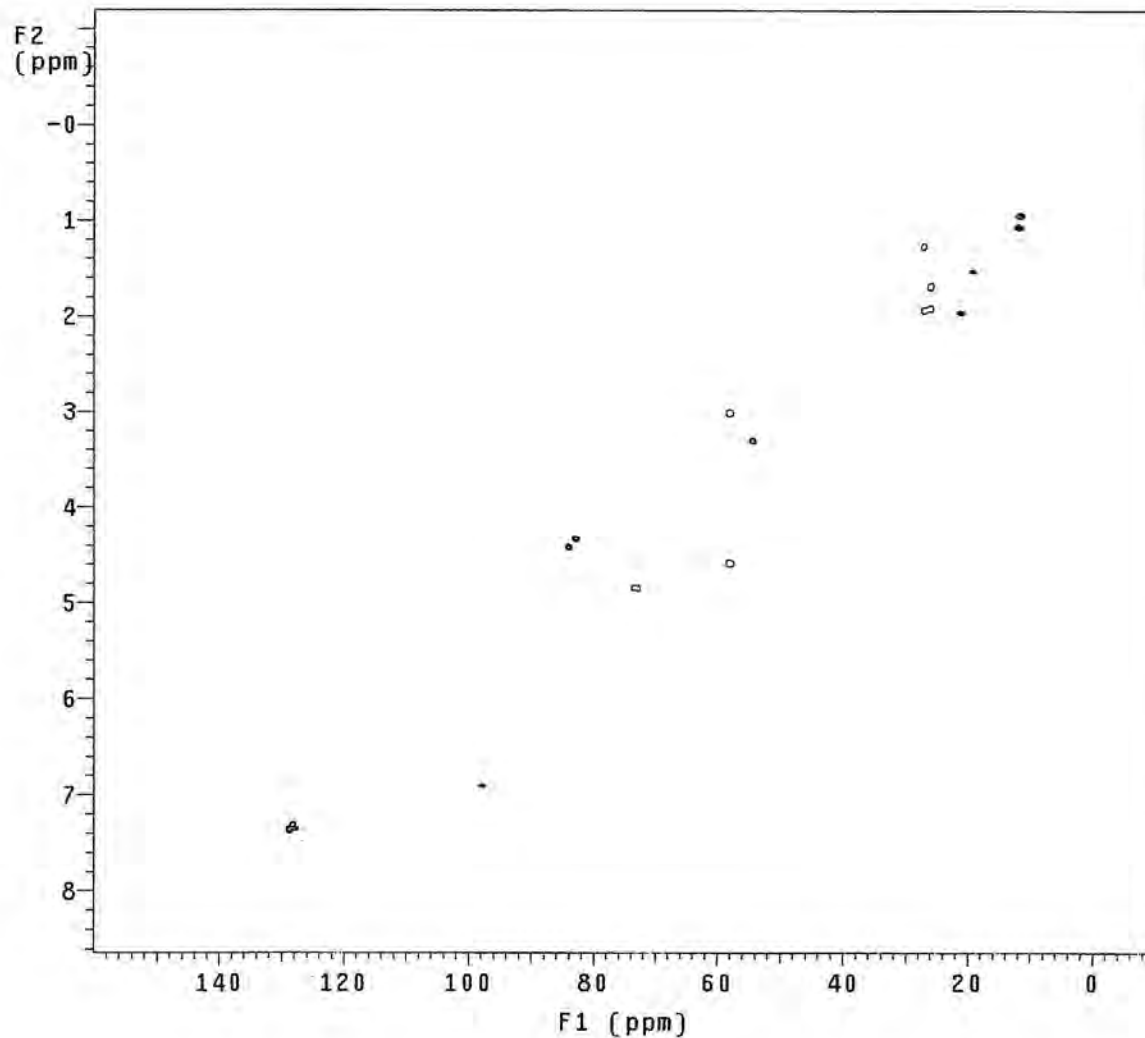
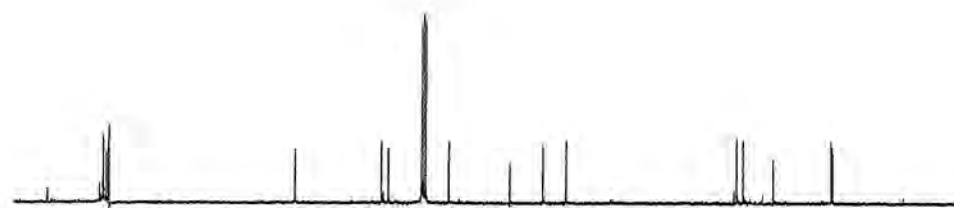
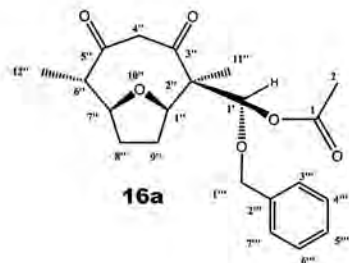
UNITAT DE RMN D ALT CAMP  
SERVEIS CIENTIFICO-TECNICS  
UNIVERSITAT DE BARCELONA  
\*\*\*\*\*@\*\*\*\*\*

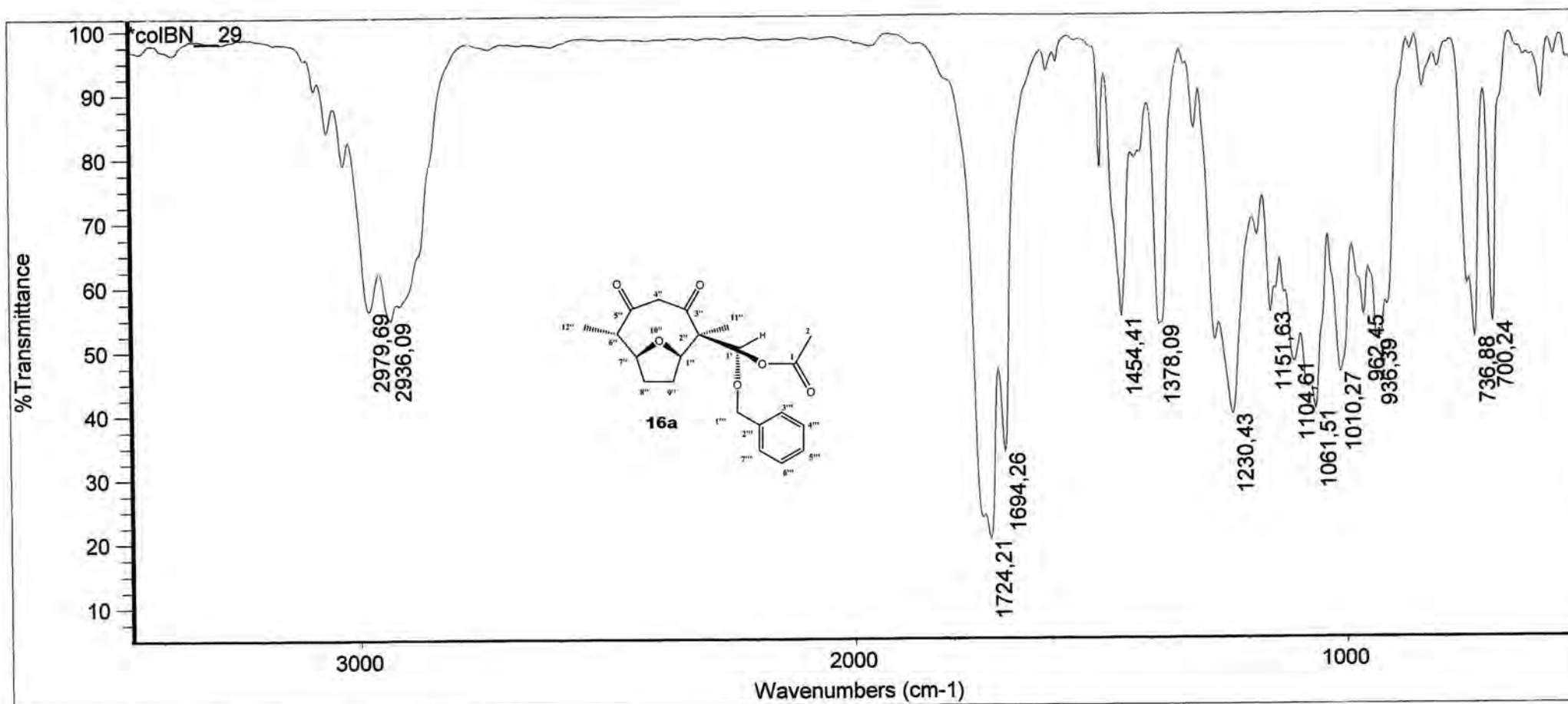
H1 / gHSQC / Mercury-400\_qui  
cdcl3/Temp: 25C /N reg: H40007-180407163907  
Usuari: amp / Mostra: colbn\_29  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.130 sec  
Width 3937.0 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 44 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048





# FIND PEAKS:

Spectrum:

\*colBN\_29

Region:

3465,27

524,29

Absolute threshold: 61,754

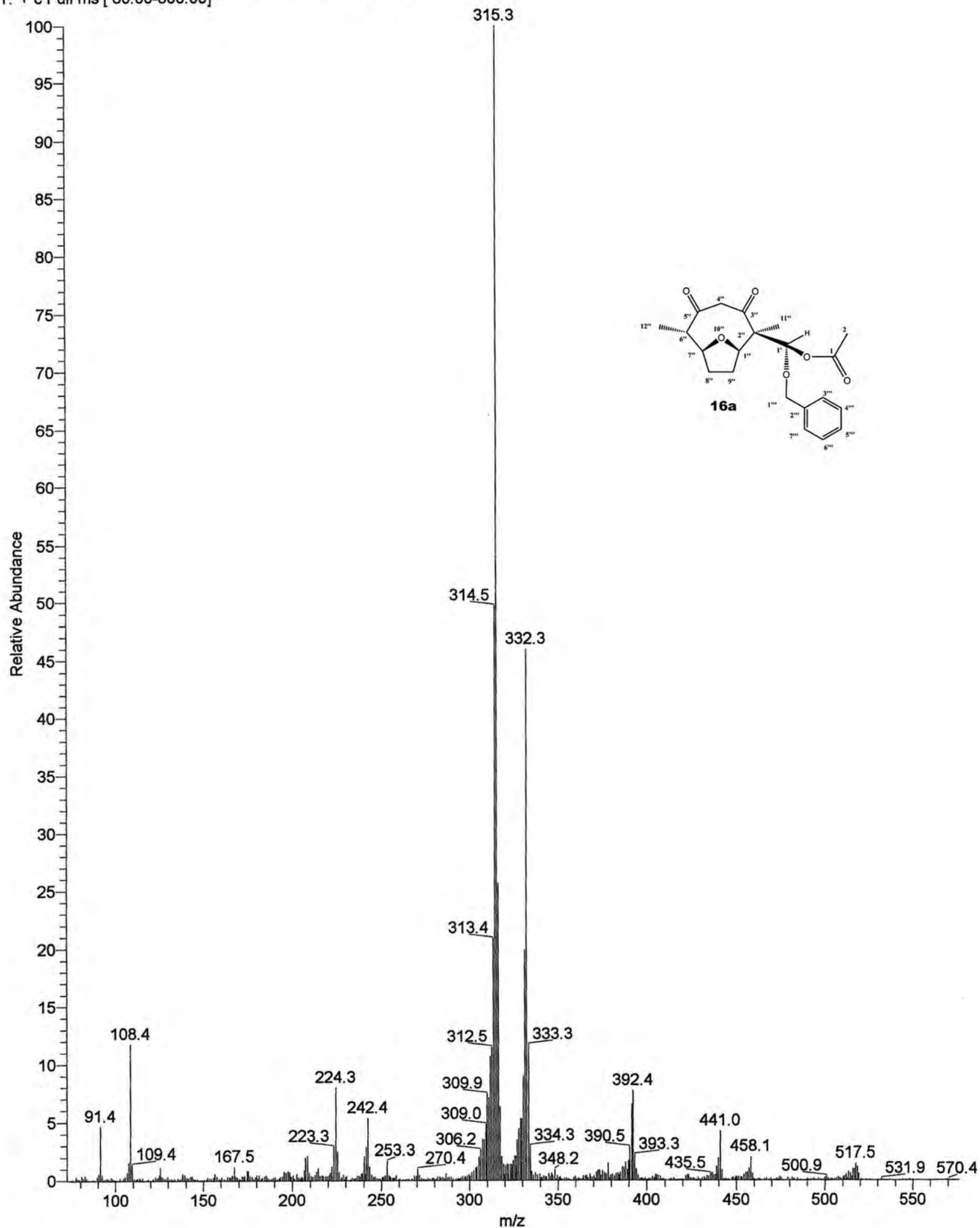
Sensitivity:

50

Peak list:

|           |         |            |        |
|-----------|---------|------------|--------|
| Position: | 1724,21 | Intensity: | 20,388 |
| Position: | 1694,26 | Intensity: | 33,893 |
| Position: | 1230,43 | Intensity: | 39,691 |
| Position: | 1061,51 | Intensity: | 40,392 |
| Position: | 1010,27 | Intensity: | 46,124 |
| Position: | 1104,61 | Intensity: | 47,809 |

|           |         |            |        |
|-----------|---------|------------|--------|
| Position: | 936,39  | Intensity: | 51,239 |
| Position: | 736,88  | Intensity: | 51,508 |
| Position: | 1378,09 | Intensity: | 53,650 |
| Position: | 700,24  | Intensity: | 53,821 |
| Position: | 2936,09 | Intensity: | 54,808 |
| Position: | 962,45  | Intensity: | 54,941 |
| Position: | 1454,41 | Intensity: | 54,980 |
| Position: | 1151,63 | Intensity: | 55,549 |
| Position: | 2979,69 | Intensity: | 56,296 |

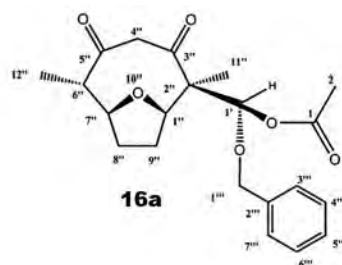


DEPCI4392#53-79 RT: 0.57-0.84 AV: 27

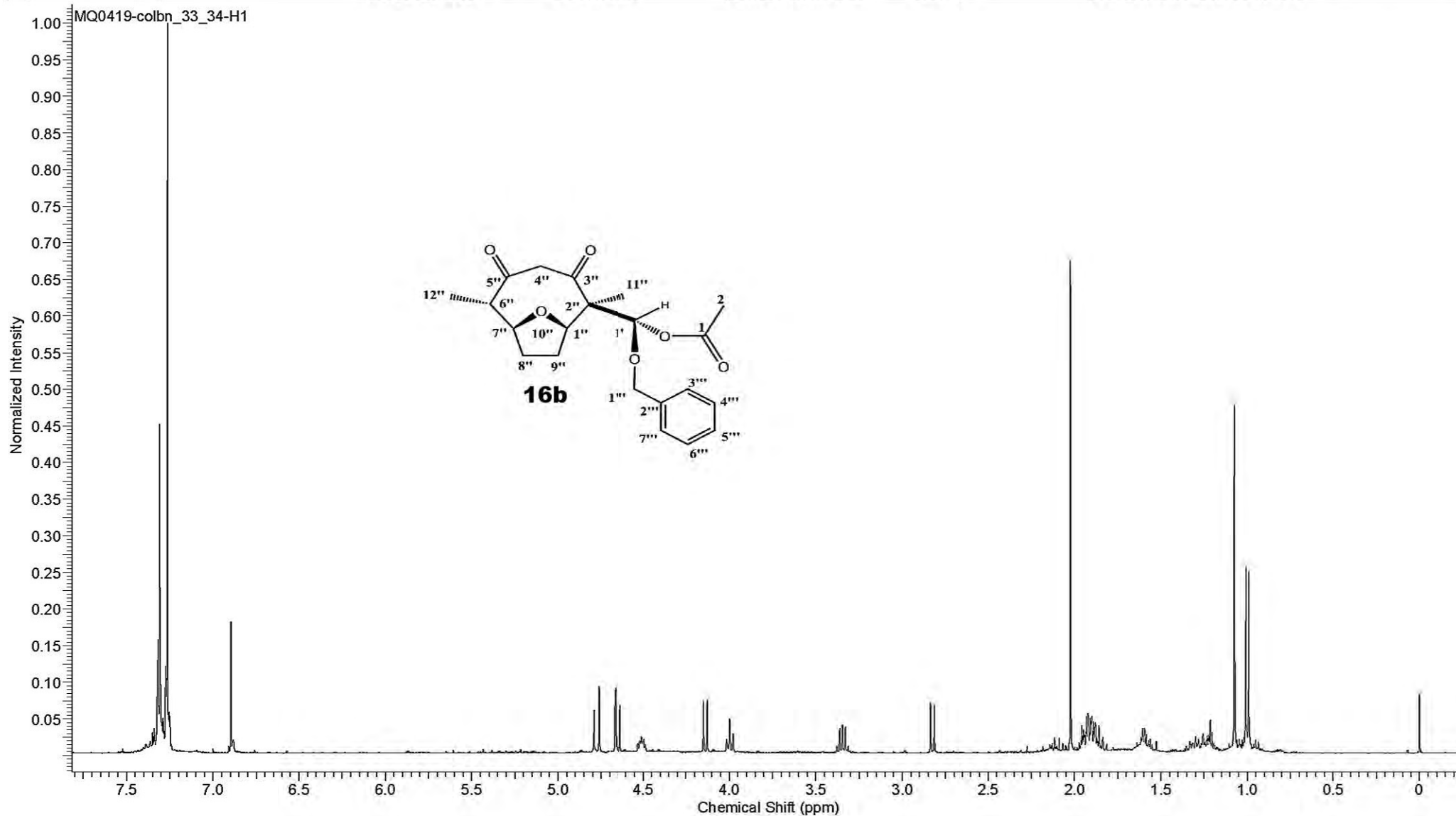
T: + c Full ms [ 60.00-800.00]

m/z= 72.3-576.0

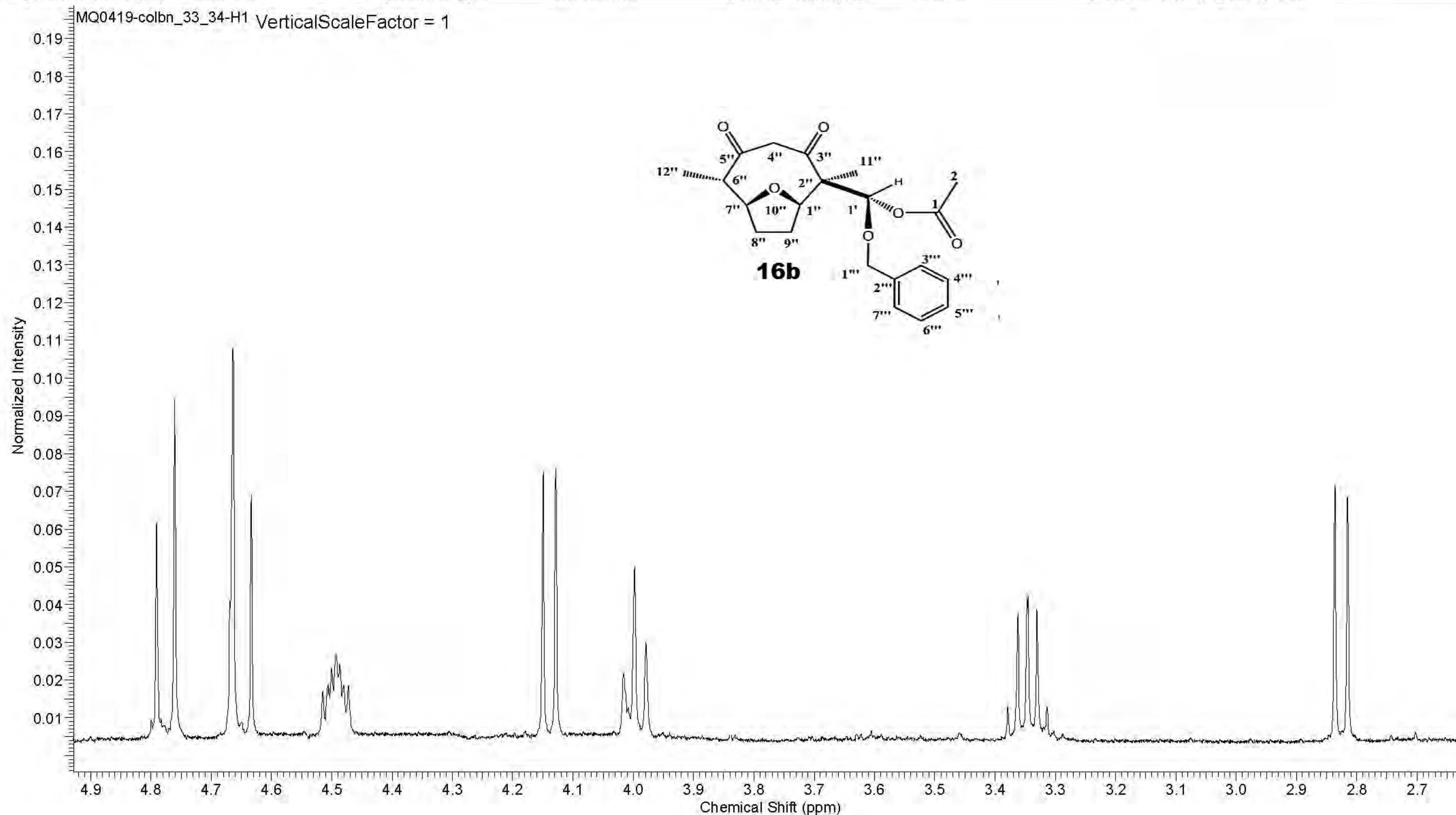
| m/z   | Intensity | Relative |
|-------|-----------|----------|
| 91.4  | 4251.4    | 4.62     |
| 107.6 | 1456.1    | 1.58     |
| 108.4 | 10812.9   | 11.75    |
| 207.4 | 1810.3    | 1.97     |
| 208.4 | 1989.2    | 2.16     |
| 223.3 | 2655.8    | 2.89     |
| 224.3 | 7331.6    | 7.97     |
| 225.3 | 2266.0    | 2.46     |
| 240.3 | 1926.1    | 2.09     |
| 241.6 | 2625.5    | 2.85     |
| 242.4 | 4903.0    | 5.33     |
| 253.3 | 1551.7    | 1.69     |
| 305.1 | 1834.7    | 1.99     |
| 306.2 | 2391.4    | 2.60     |
| 307.3 | 3238.9    | 3.52     |
| 308.2 | 3179.8    | 3.46     |
| 309.0 | 4431.6    | 4.82     |
| 309.9 | 6850.4    | 7.45     |
| 310.8 | 6514.5    | 7.08     |
| 311.7 | 9814.9    | 10.67    |
| 312.5 | 10561.9   | 11.48    |
| 313.4 | 19212.5   | 20.88    |
| 314.5 | 45715.9   | 49.69    |
| 315.3 | 91998.2   | 100.00   |
| 316.3 | 23602.9   | 25.66    |
| 317.2 | 5817.5    | 6.32     |
| 318.1 | 1922.0    | 2.09     |
| 324.7 | 1547.3    | 1.68     |
| 325.4 | 1939.8    | 2.11     |
| 326.1 | 1920.0    | 2.09     |
| 326.9 | 3173.3    | 3.45     |
| 327.8 | 4056.7    | 4.41     |
| 328.7 | 4880.4    | 5.30     |
| 329.4 | 4841.9    | 5.26     |
| 330.3 | 8247.5    | 8.96     |
| 331.4 | 18312.7   | 19.91    |
| 332.3 | 42294.2   | 45.97    |
| 333.3 | 10758.0   | 11.69    |
| 334.3 | 2741.2    | 2.98     |
| 378.3 | 1339.3    | 1.46     |
| 388.2 | 1441.9    | 1.57     |
| 389.6 | 1549.7    | 1.68     |
| 390.5 | 2622.6    | 2.85     |
| 391.5 | 5975.4    | 6.50     |
| 392.4 | 7065.4    | 7.68     |
| 393.3 | 2009.1    | 2.18     |
| 439.9 | 1739.2    | 1.89     |
| 441.0 | 3858.1    | 4.19     |
| 458.1 | 1841.3    | 2.00     |
| 517.5 | 1313.7    | 1.43     |



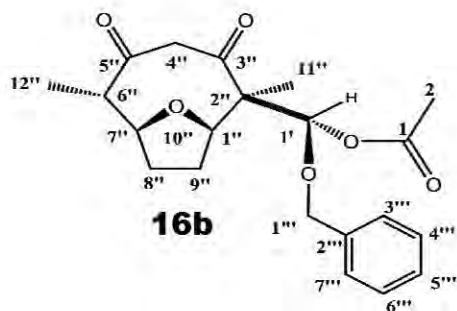
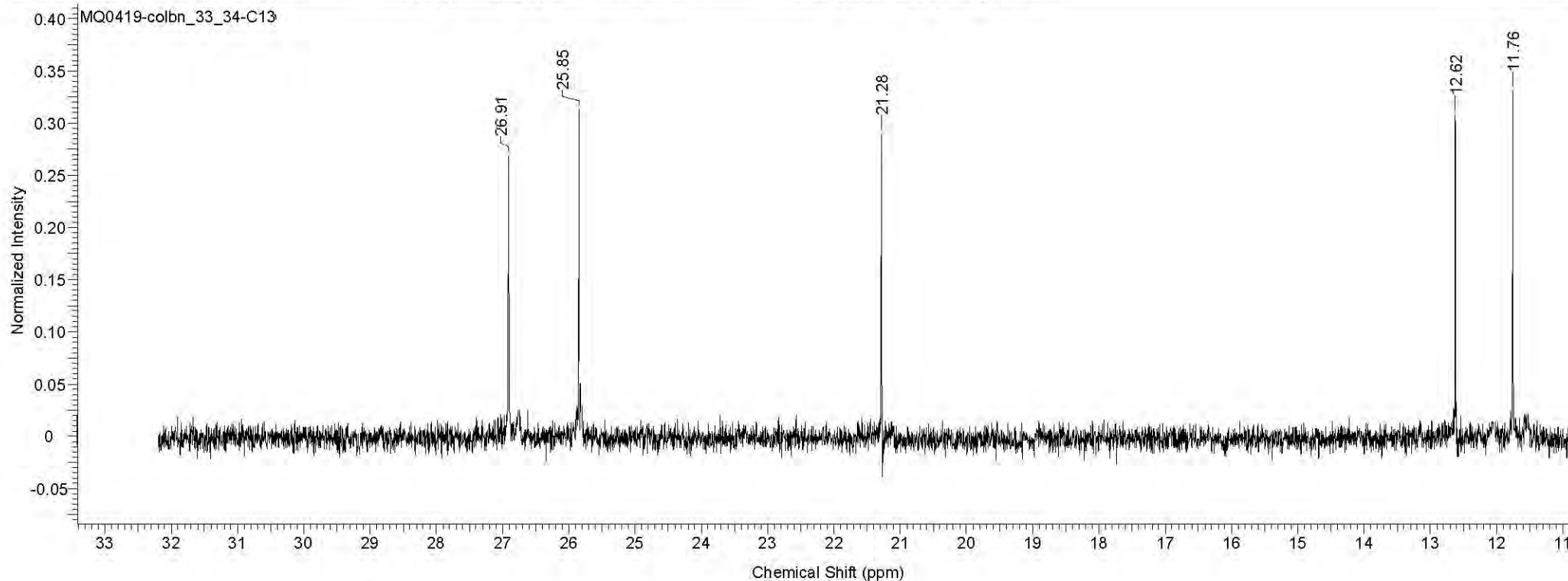
|                                                                                         |           |                                   |          |                               |              |
|-----------------------------------------------------------------------------------------|-----------|-----------------------------------|----------|-------------------------------|--------------|
| <b>Acquisition Time (sec)</b> 3.4998                                                    |           |                                   |          |                               |              |
| <b>Comment</b> H1 / s2pul / Mercury-400 qui cdc13/Temp: 25C /N reg: M40007-190407170747 |           | Usuari: amp / Mostra: colbn_33_34 |          | Nom: STEFANO PONZANO          |              |
|                                                                                         |           | <b>File Name</b>                  |          | MQ0419-colbn_33_34-H1.fid\fid |              |
| <b>Frequency (MHz)</b>                                                                  | 400.11    | <b>Nucleus</b>                    | 1H       | <b>Number of Transients</b>   | 8            |
| <b>Points Count</b>                                                                     | 16384     | <b>Pulse Sequence</b>             | s2pul    | <b>Receiver Gain</b>          | 32.00        |
| <b>Spectrum Offset (Hz)</b>                                                             | 1802.4902 | <b>Spectrum Type</b>              | STANDARD | <b>Sweep Width (Hz)</b>       | 4000.00      |
|                                                                                         |           |                                   |          | <b>Original Points Count</b>  | 13999        |
|                                                                                         |           |                                   |          | <b>Solvent</b>                | CHLOROFORM-d |
|                                                                                         |           |                                   |          | <b>Temperature (degree C)</b> | 25.0         |



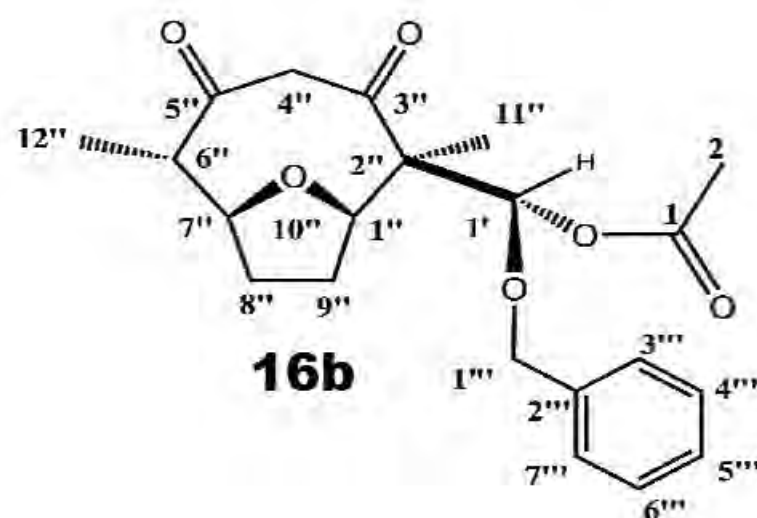
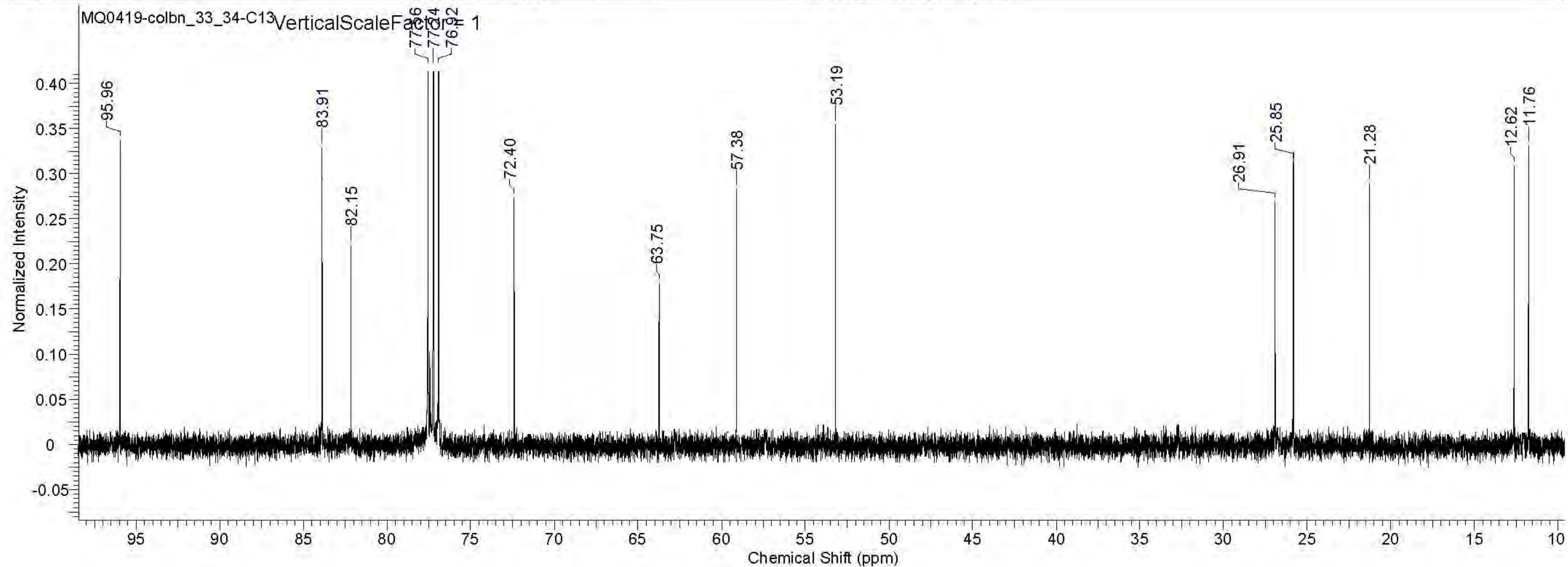
|                                                                                                                                                |           |                       |             |                                 |                                    |
|------------------------------------------------------------------------------------------------------------------------------------------------|-----------|-----------------------|-------------|---------------------------------|------------------------------------|
| <b>Acquisition Time (sec)</b> 3.4998                                                                                                           |           |                       |             |                                 |                                    |
| <b>Comment</b> H1 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |           |                       |             |                                 |                                    |
|                                                                                                                                                |           | <b>Date Stamp</b>     | Apr 19 2007 | <b>File Name</b>                | MQ0419-colbn_33_34-H1.fid\fid      |
| <b>Frequency (MHz)</b>                                                                                                                         | 400.11    | <b>Nucleus</b>        | 1H          | <b>Number of Transients</b> 8   | <b>Original Points Count</b> 13999 |
| <b>Points Count</b>                                                                                                                            | 16384     | <b>Pulse Sequence</b> | s2pul       | <b>Receiver Gain</b> 32.00      | <b>Solvent</b> CHLOROFORM-d        |
| <b>Spectrum Offset (Hz)</b>                                                                                                                    | 1802.4902 | <b>Spectrum Type</b>  | STANDARD    | <b>Sweep Width (Hz)</b> 4000.00 | <b>Temperature (degree C)</b> 25.0 |



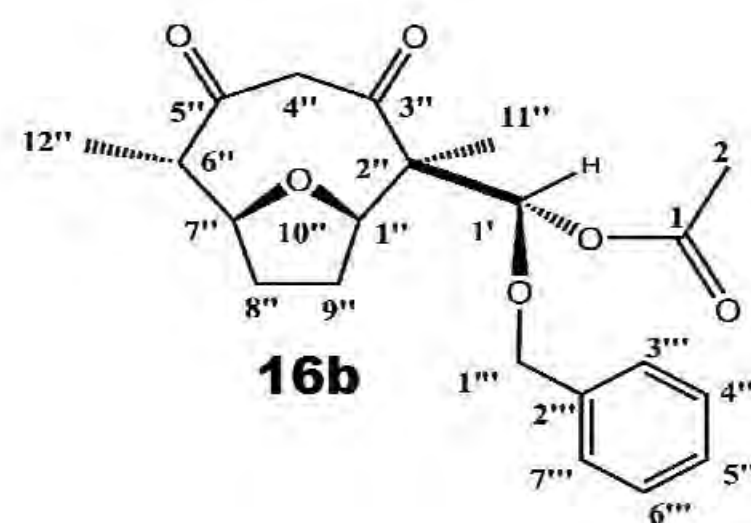
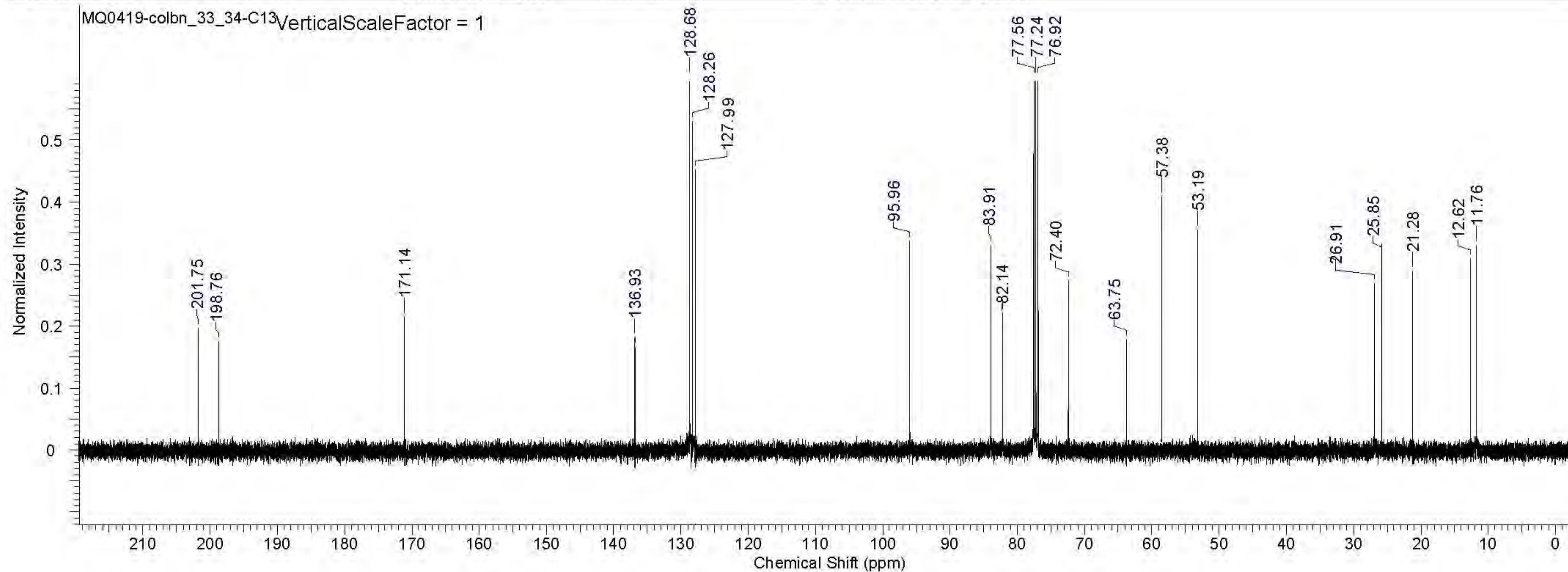
|                                                                                          |          |                                                        |                                |
|------------------------------------------------------------------------------------------|----------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |          |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |          | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |          | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                   | 100.62   | <b>Nucleus</b>                                         | 13C                            |
| <b>Original Points Count</b>                                                             | 33241    | <b>Points Count</b>                                    | 65536                          |
| <b>Receiver Gain</b>                                                                     | 39.00    | <b>Solvent</b>                                         | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                     | STANDARD | <b>Sweep Width (Hz)</b>                                | 27700.83                       |
|                                                                                          |          | <b>Number of Transients</b>                            | 4000                           |
|                                                                                          |          | <b>Pulse Sequence</b>                                  | s2pul                          |
|                                                                                          |          | <b>Spectrum Offset (Hz)</b>                            | 11318.4121                     |
|                                                                                          |          | <b>Temperature (degree C)</b>                          | 25.000                         |



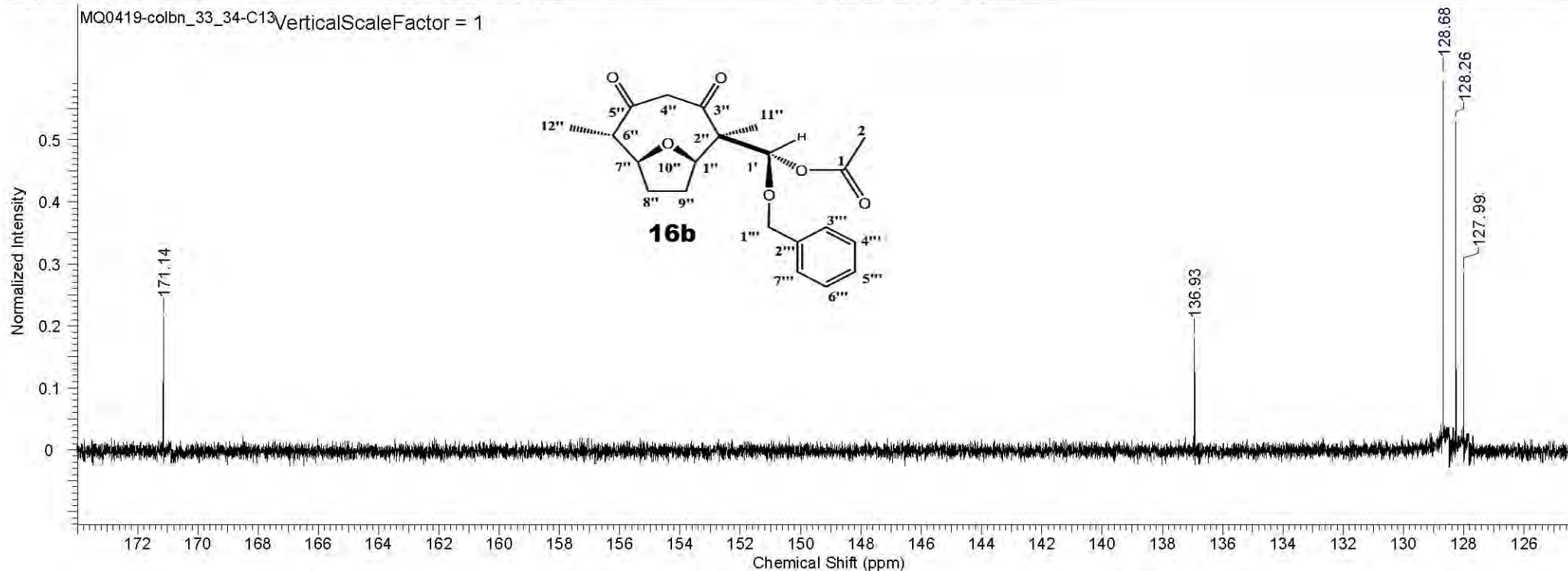
|                                                                                          |          |                                                        |                                |
|------------------------------------------------------------------------------------------|----------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |          |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |          | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |          | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                   | 100.62   | <b>Nucleus</b>                                         | 13C                            |
| <b>Original Points Count</b>                                                             | 33241    | <b>Points Count</b>                                    | 65536                          |
| <b>Receiver Gain</b>                                                                     | 39.00    | <b>Solvent</b>                                         | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                     | STANDARD | <b>Sweep Width (Hz)</b>                                | 27700.83                       |
|                                                                                          |          | <b>Number of Transients</b>                            | 4000                           |
|                                                                                          |          | <b>Pulse Sequence</b>                                  | s2pul                          |
|                                                                                          |          | <b>Spectrum Offset (Hz)</b>                            | 11318.4121                     |
|                                                                                          |          | <b>Temperature (degree C)</b>                          | 25.0                           |



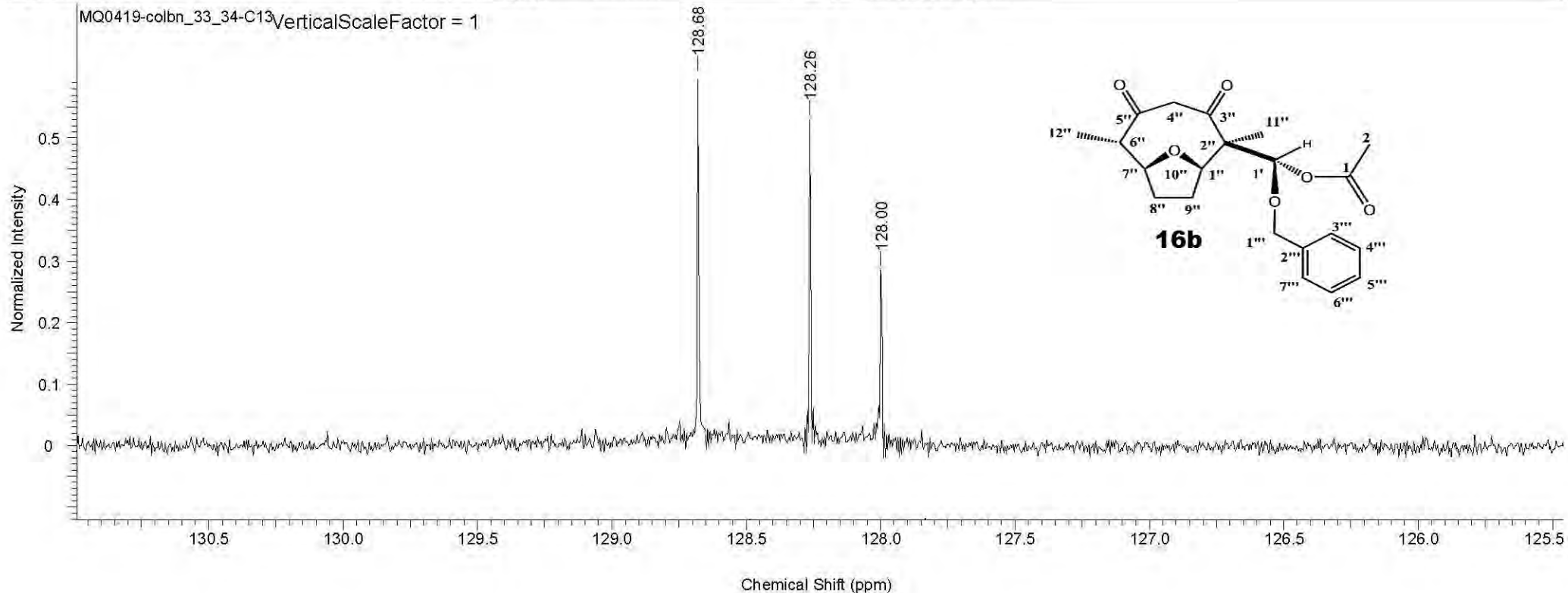
|                                                                                          |          |                                                        |                                |
|------------------------------------------------------------------------------------------|----------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |          |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |          | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |          | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                   | 100.62   | <b>Nucleus</b>                                         | 13C                            |
| <b>Original Points Count</b>                                                             | 33241    | <b>Points Count</b>                                    | 65536                          |
| <b>Receiver Gain</b>                                                                     | 39.00    | <b>Solvent</b>                                         | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                     | STANDARD | <b>Sweep Width (Hz)</b>                                | 27700.83                       |
|                                                                                          |          | <b>Number of Transients</b>                            | 4000                           |
|                                                                                          |          | <b>Pulse Sequence</b>                                  | s2pul                          |
|                                                                                          |          | <b>Spectrum Offset (Hz)</b>                            | 11318.4121                     |
|                                                                                          |          | <b>Temperature (degree C)</b>                          | 25.000                         |



|                                                                                          |                                  |                                                        |                                |
|------------------------------------------------------------------------------------------|----------------------------------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |                                  |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |                                  | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |                                  | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b> 100.62                                                            | <b>Nucleus</b> 13C               | <b>Number of Transients</b> 4000                       |                                |
| <b>Original Points Count</b> 33241                                                       | <b>Points Count</b> 65536        | <b>Pulse Sequence</b> s2pul                            |                                |
| <b>Receiver Gain</b> 39.00                                                               | <b>Solvent</b> CHLOROFORM-d      | <b>Spectrum Offset (Hz)</b> 11318.4121                 |                                |
| <b>Spectrum Type</b> STANDARD                                                            | <b>Sweep Width (Hz)</b> 27700.83 | <b>Temperature (degree C)</b> 25.000                   |                                |



|                                                                                          |                                  |                                                                                       |  |  |
|------------------------------------------------------------------------------------------|----------------------------------|---------------------------------------------------------------------------------------|--|--|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |                                  |                                                                                       |  |  |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |                                  | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO Data:19/04/07 / Sist automatic |  |  |
|                                                                                          |                                  | <b>File Name</b> MQ0419-colbn_33_34-C13.fid\fid                                       |  |  |
| <b>Frequency (MHz)</b> 100.62                                                            | <b>Nucleus</b> 13C               | <b>Number of Transients</b> 4000                                                      |  |  |
| <b>Original Points Count</b> 33241                                                       | <b>Points Count</b> 65536        | <b>Pulse Sequence</b> s2pul                                                           |  |  |
| <b>Receiver Gain</b> 39.00                                                               | <b>Solvent</b> CHLOROFORM-d      | <b>Spectrum Offset (Hz)</b> 11318.4121                                                |  |  |
| <b>Spectrum Type</b> STANDARD                                                            | <b>Sweep Width (Hz)</b> 27700.83 | <b>Temperature (degree C)</b> 25.0                                                    |  |  |

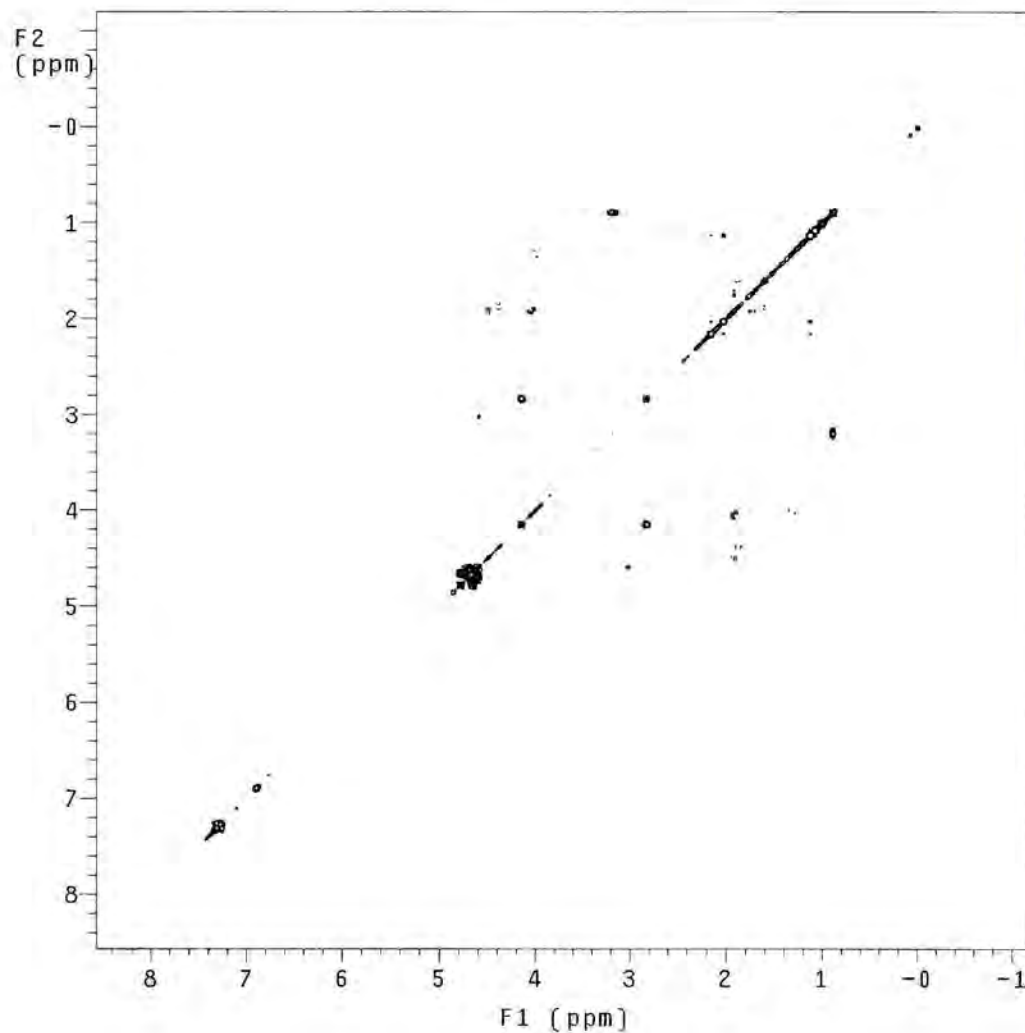
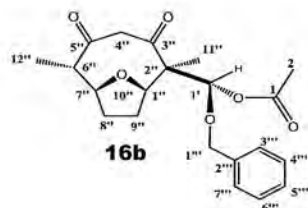


H1 / gCOSY / Mercury-400\_qui  
 cdc13/Temp: 25C /N reg: M40007-190407170747  
 Usuari: amp / Mostra: colbn\_33\_34  
 Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
 \*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
 Acq. time 0.150 sec  
 Width 3906.2 Hz  
 2D Width 3906.2 Hz  
 2 repetitions  
 256 increments  
 OBSERVE H1, 400.112 MHz  
 DATA PROCESSING  
 Sq. sine bell 0.075 sec  
 F1 DATA PROCESSING  
 Sq. sine bell 0.066 sec  
 FT size 2048 x 2048

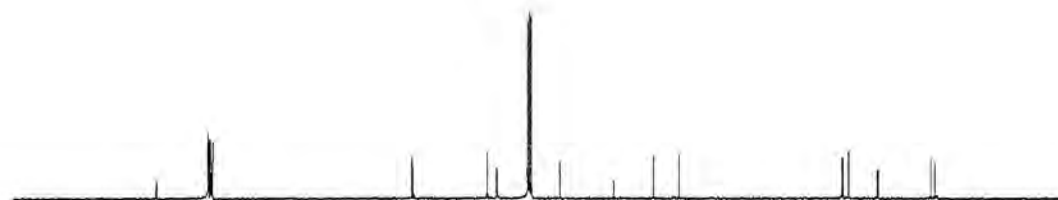
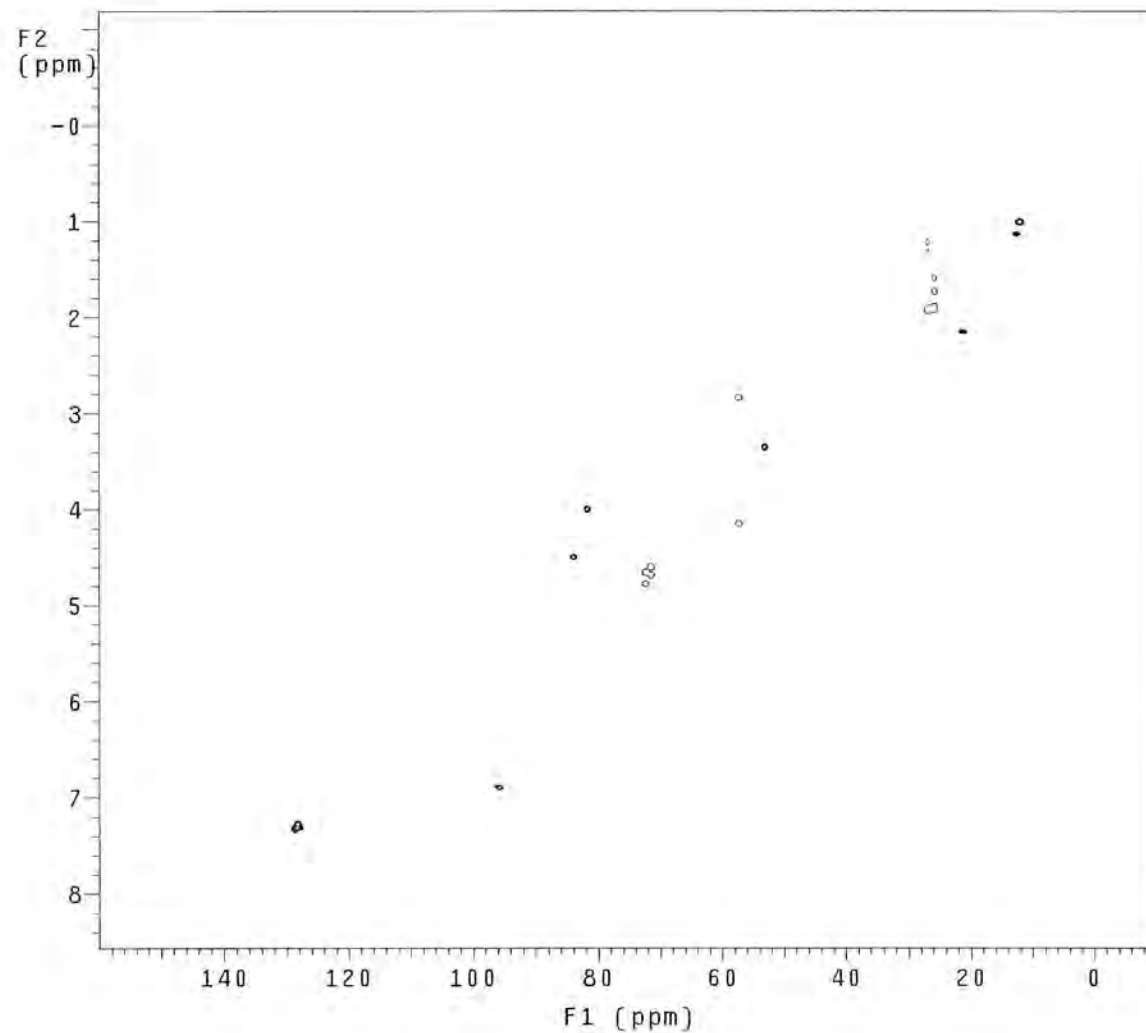
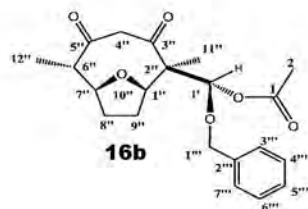


H1 / gHSQC / Mercury-400\_qui  
 cdc13/Temp: 25C /N reg: M40007-190407170747  
 Usuari: amp / Mostra: colbn\_33\_34  
 Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
 \*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
 Acq. time 0.131 sec  
 Width 3906.2 Hz  
 2D Width 17094.0 Hz  
 4 repetitions  
 2 x 128 increments  
 OBSERVE H1, 400.112 MHz  
 DECOUPLE C13, 100.6156477 MHz  
 Power 44 dB  
 on during acquisition  
 off during delay  
 GARP-1 modulated  
 DATA PROCESSING  
 Gauss apodization 0.061 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.007 sec  
 FT size 1024 x 2048

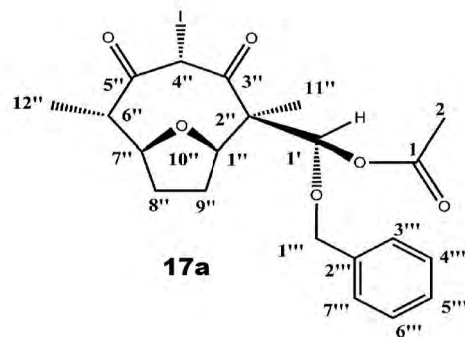


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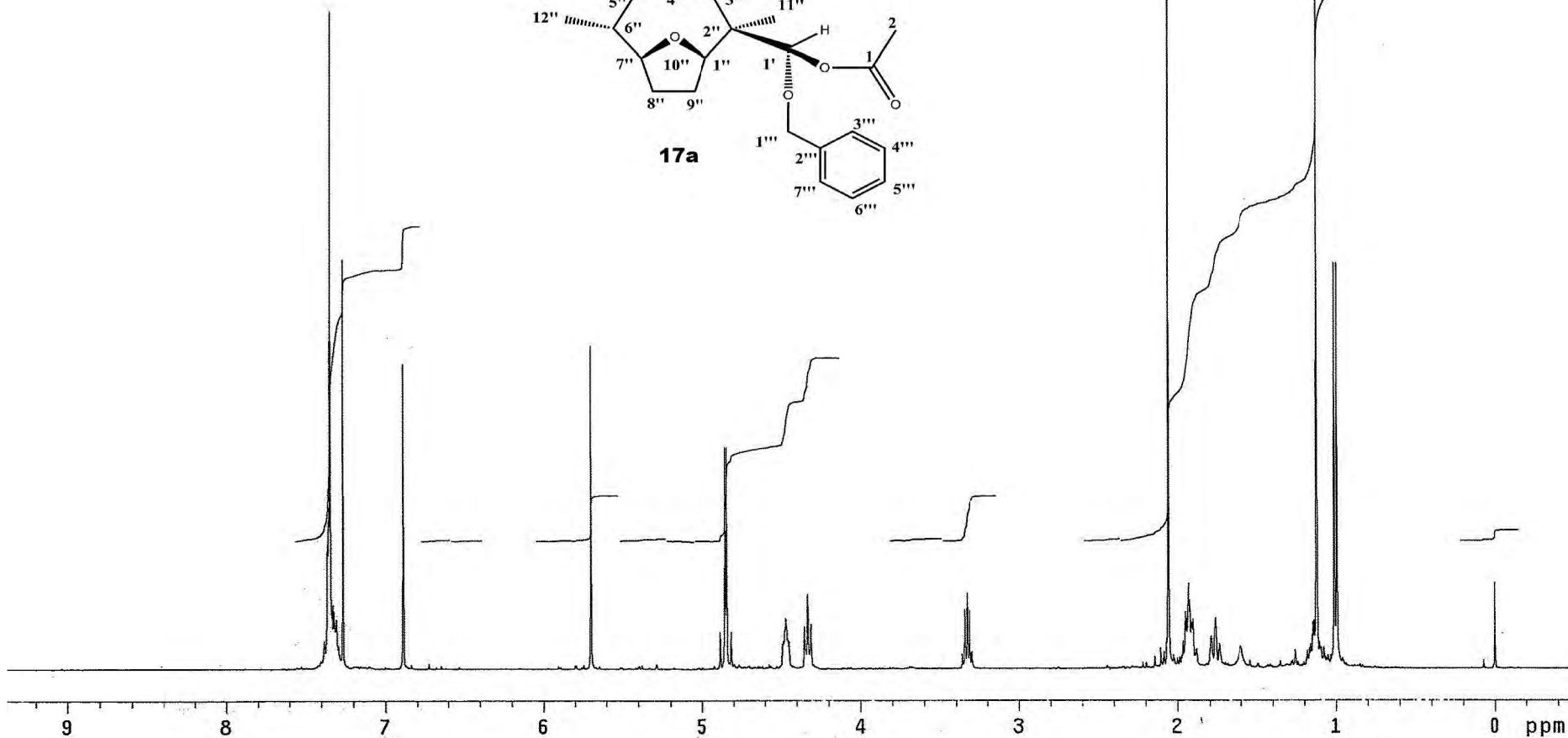
H1 / s2pul / Mercury-400.ql  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO

Pulse Sequence: s2pul  
\*\*\*\*\*

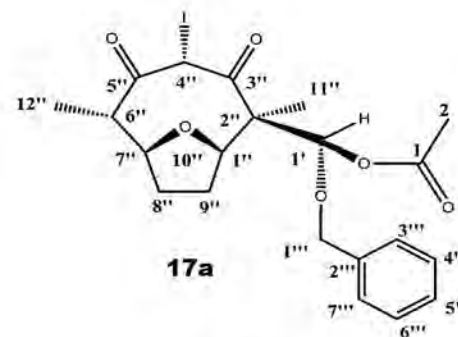
COL BN-23  
Libreta nº 3  
(COLUMN BN)



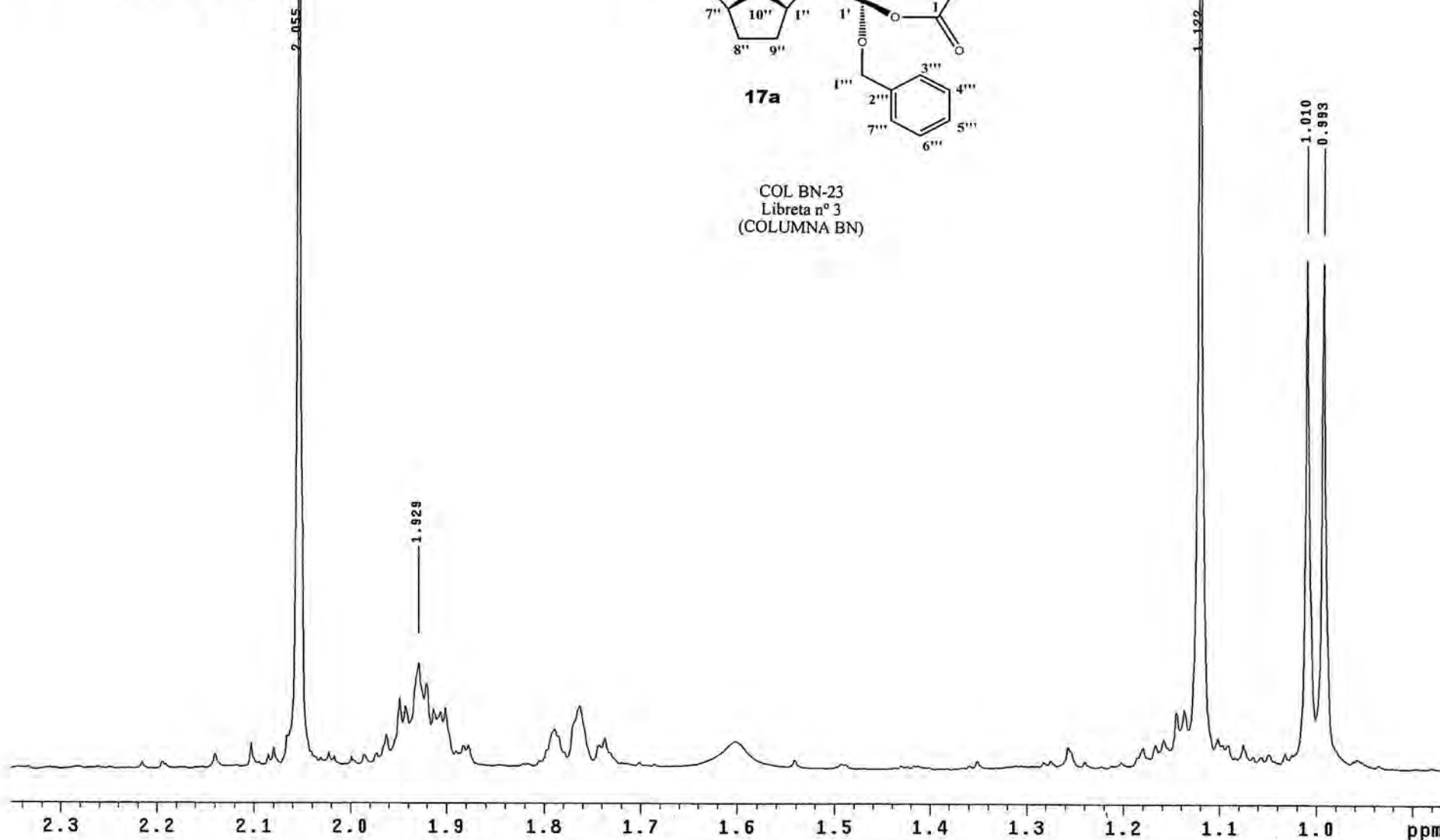
| INDEX | FREQUENCY | PPM   | HEIGHT |
|-------|-----------|-------|--------|
| 1     | 2945.068  | 7.361 | 19.1   |
| 2     | 2943.848  | 7.358 | 20.3   |
| 3     | 2942.139  | 7.353 | 25.1   |
| 4     | 2939.209  | 7.346 | 103.1  |
| 5     | 2906.250  | 7.264 | 64.2   |
| 6     | 2755.859  | 6.888 | 47.7   |
| 7     | 2279.541  | 5.697 | 50.7   |
| 8     | 1942.383  | 4.855 | 34.6   |
| 9     | 1937.988  | 4.844 | 34.5   |
| 10    | 822.266   | 2.055 | 162.0  |
| 11    | 448.975   | 1.122 | 126.5  |
| 12    | 404.297   | 1.010 | 63.7   |
| 13    | 397.461   | 0.993 | 63.6   |



H1 / s2pu1 / Mercury-400\_gui  
cdcl3/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Sist automatic  
Pulse Sequence: s2pu1

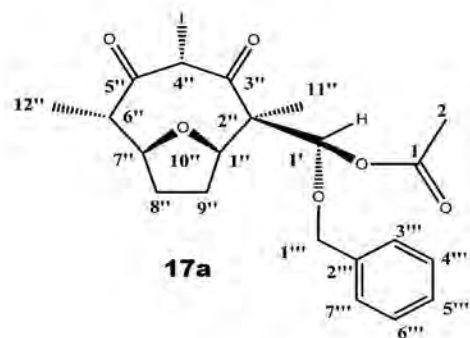


COL BN-23  
Libreta n° 3  
(COLUMNNA BN)

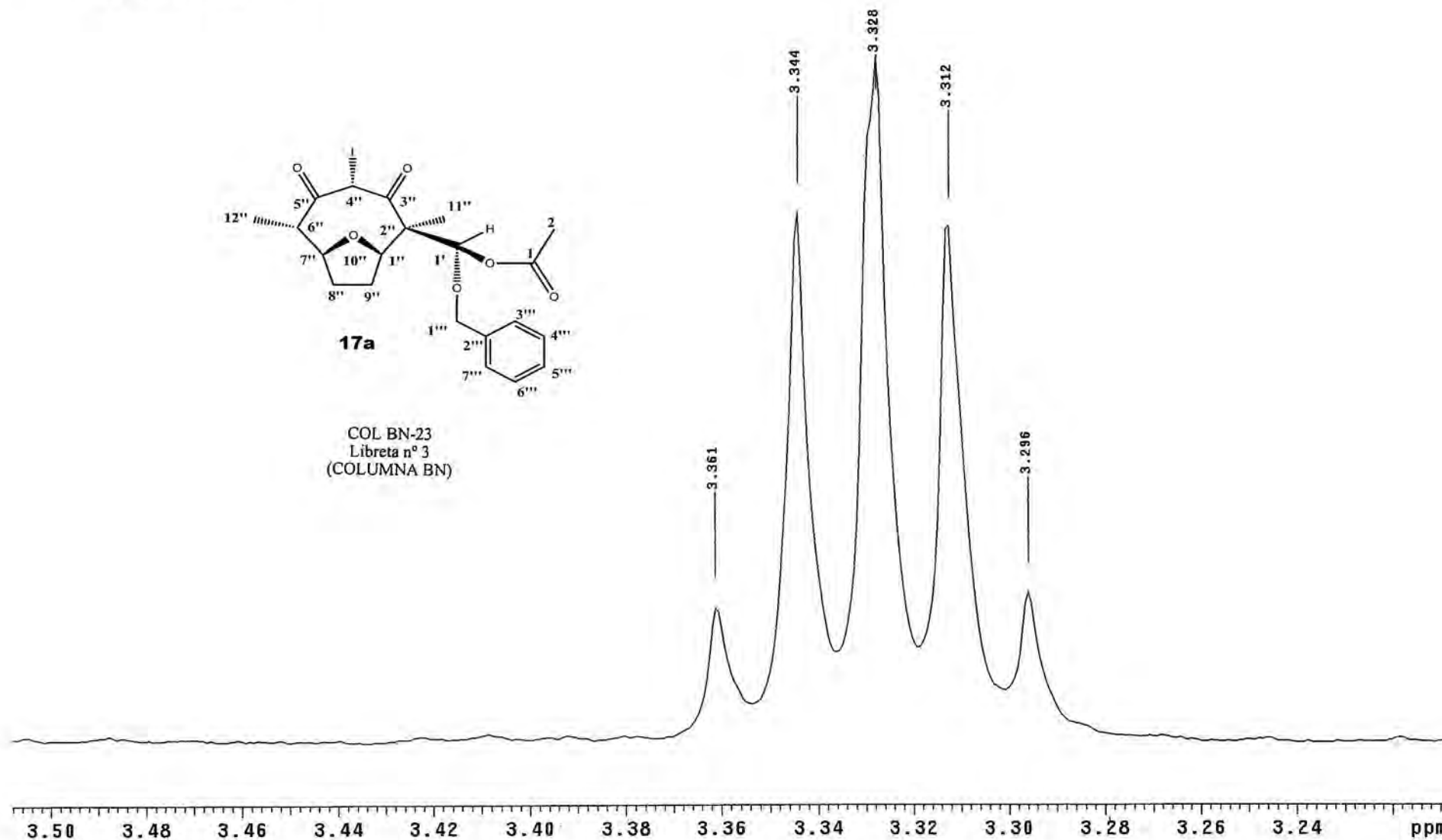


H1 / s2pu1 / Mercury-400.qui  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Data:18/04/07 / Sist automatic

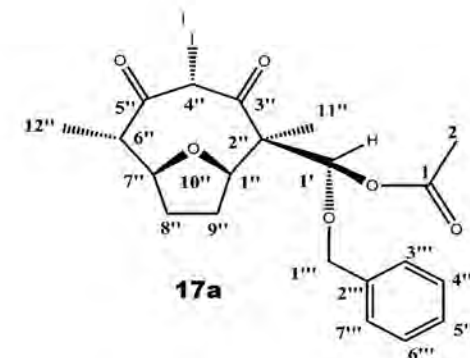
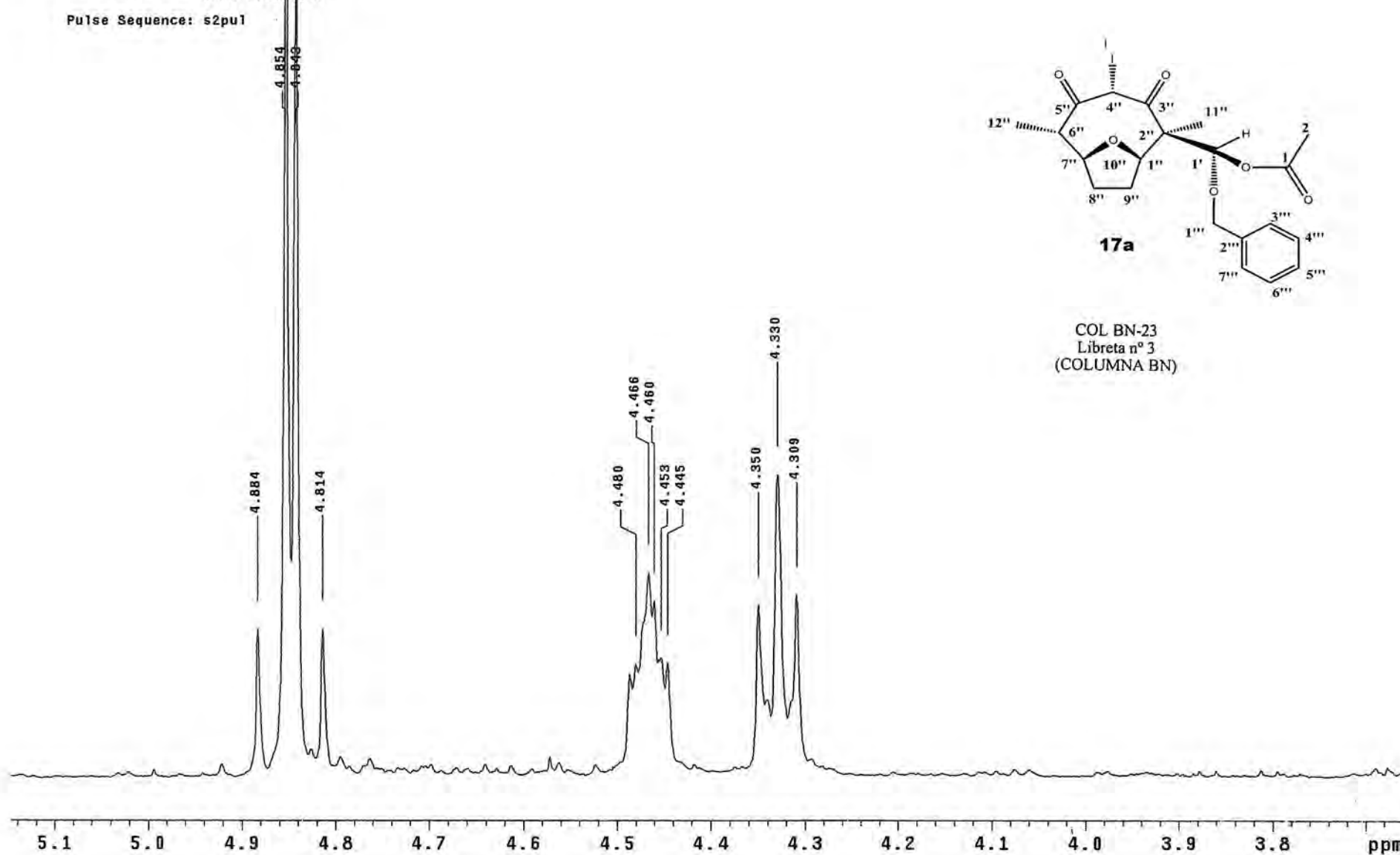
Pulse Sequence: s2pu1



COL BN-23  
Libreta n° 3  
(COLUMNA BN)



H1 / s2pu1 / Mercury-400  
cdcl3/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Sist automatic  
Pulse Sequence: s2pu1

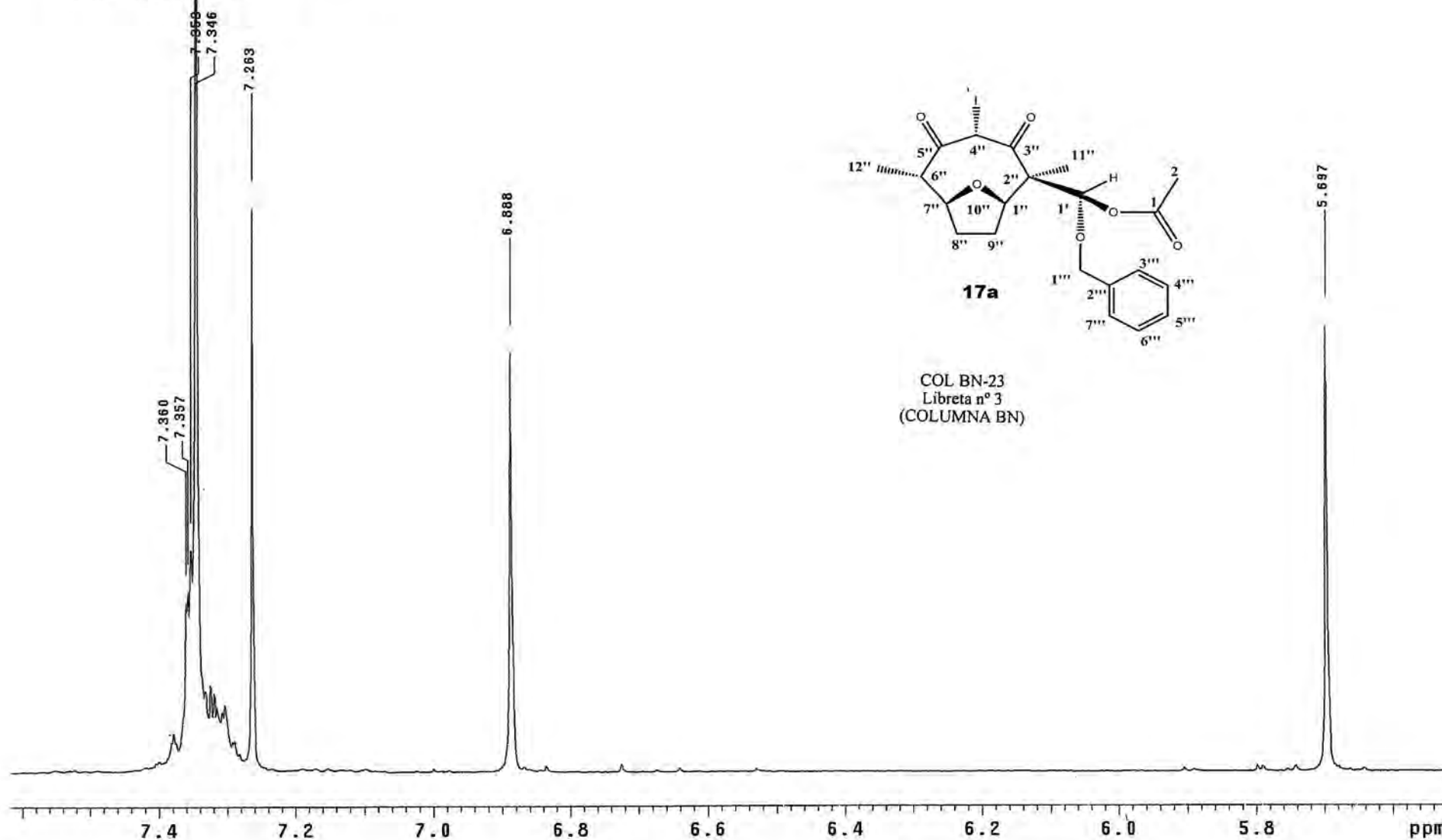


COL BN-23  
Libreta nº 3  
(COLUMNA BN)

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H1 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pu1



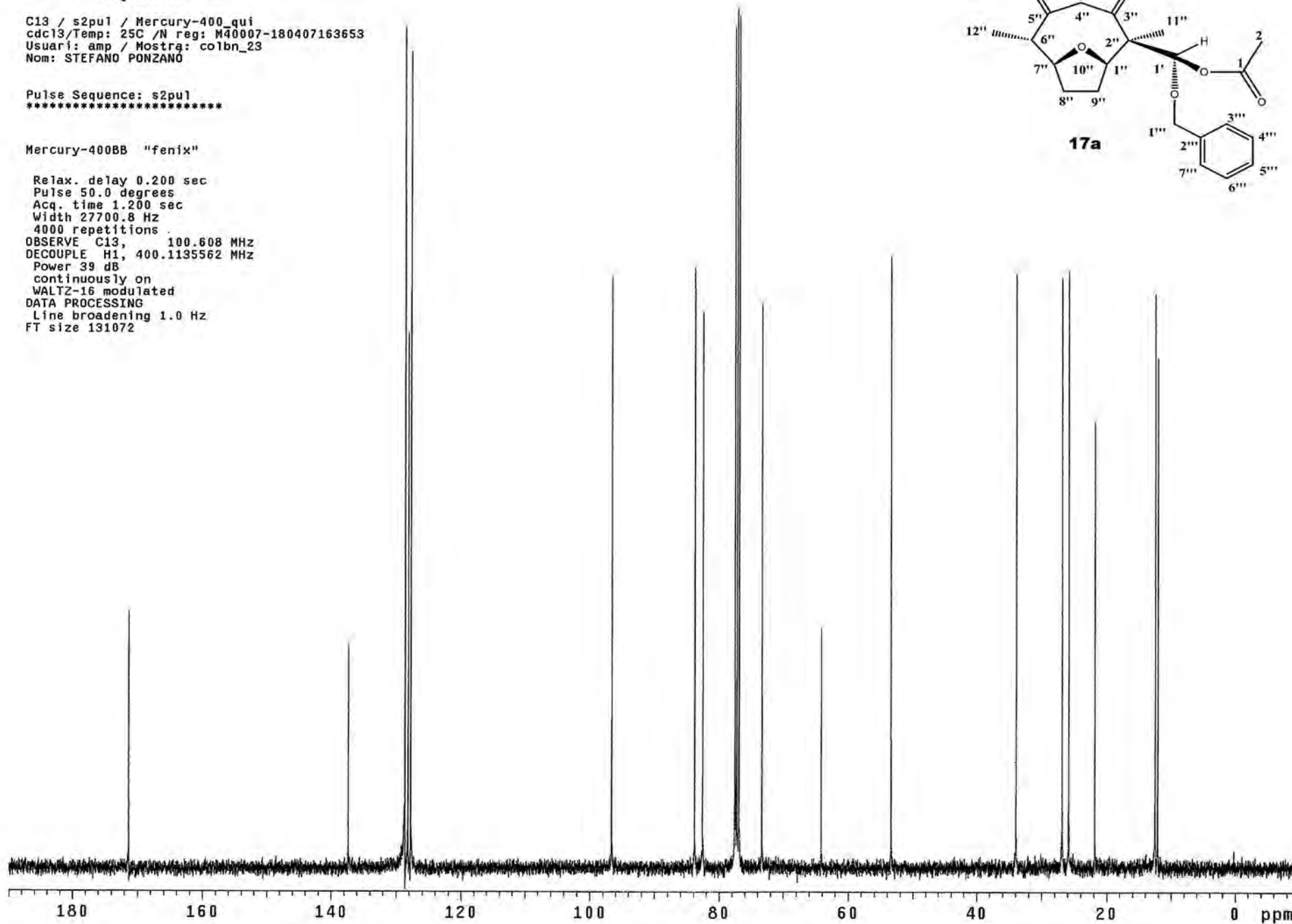
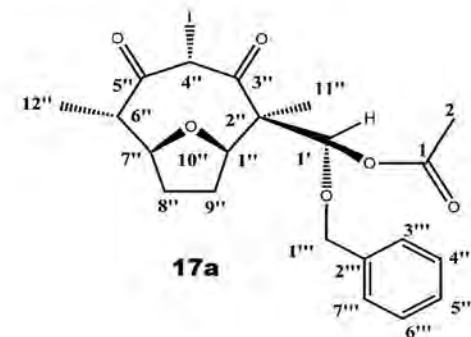
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UNIVERSITAT DE BARCELONA  
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C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO

Pulse Sequence: s2pu1  
\*\*\*\*\*@\*\*\*\*\*

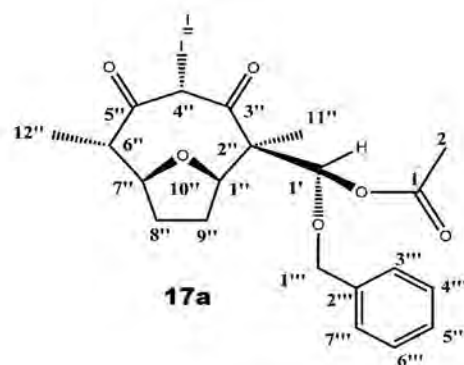
Mercury-400BB "fenix"

Relax. delay 0.200 sec  
Pulse 50.0 degrees  
Acq. time 1.200 sec  
Width 27700.8 Hz  
4000 repetitions  
OBSERVE C13, 100.608 MHz  
DECOUPLE H1, 400.1135562 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072

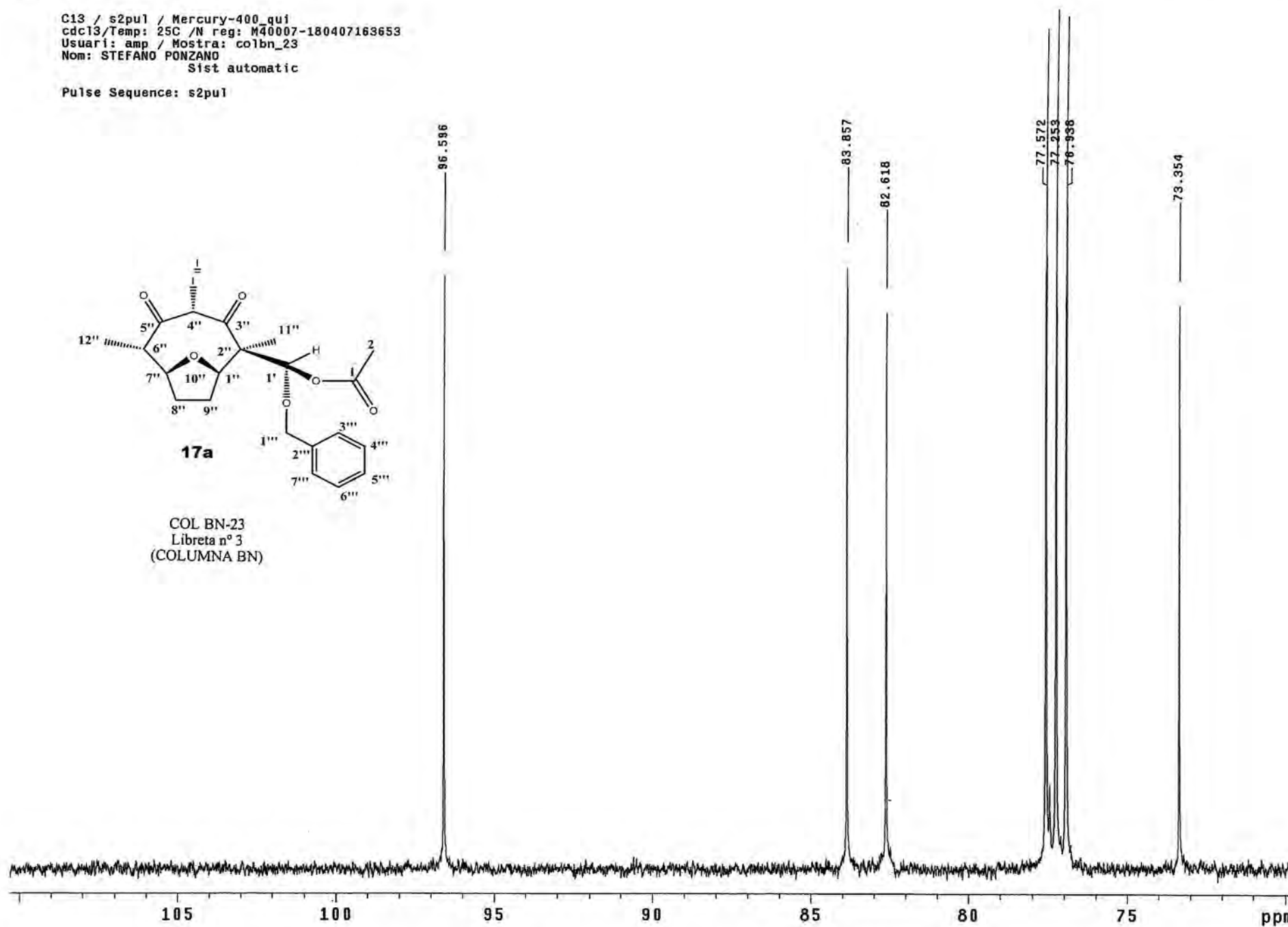


C13 / s2pul / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pul

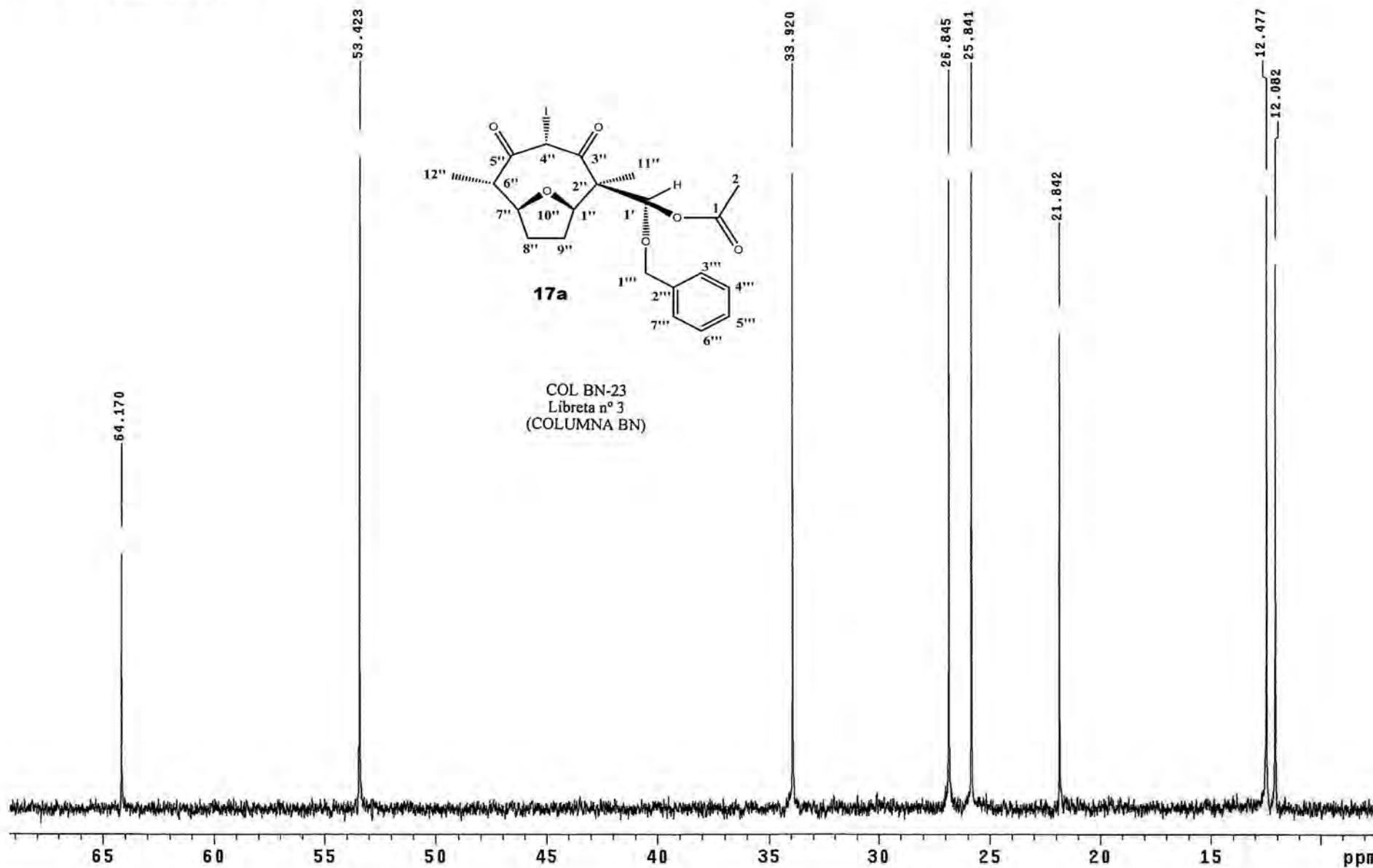


COL BN-23  
Libreta n° 3  
(COLUMNA BN)



C13 / s2pu1 / Mercury-400\_gui  
cdc13/Temp: 25C /N reg: M40007-180407183853  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Sist automatic

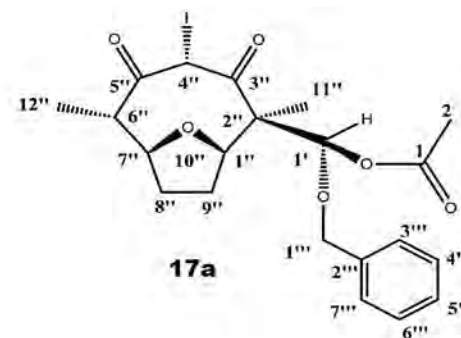
Pulse Sequence: s2pu1



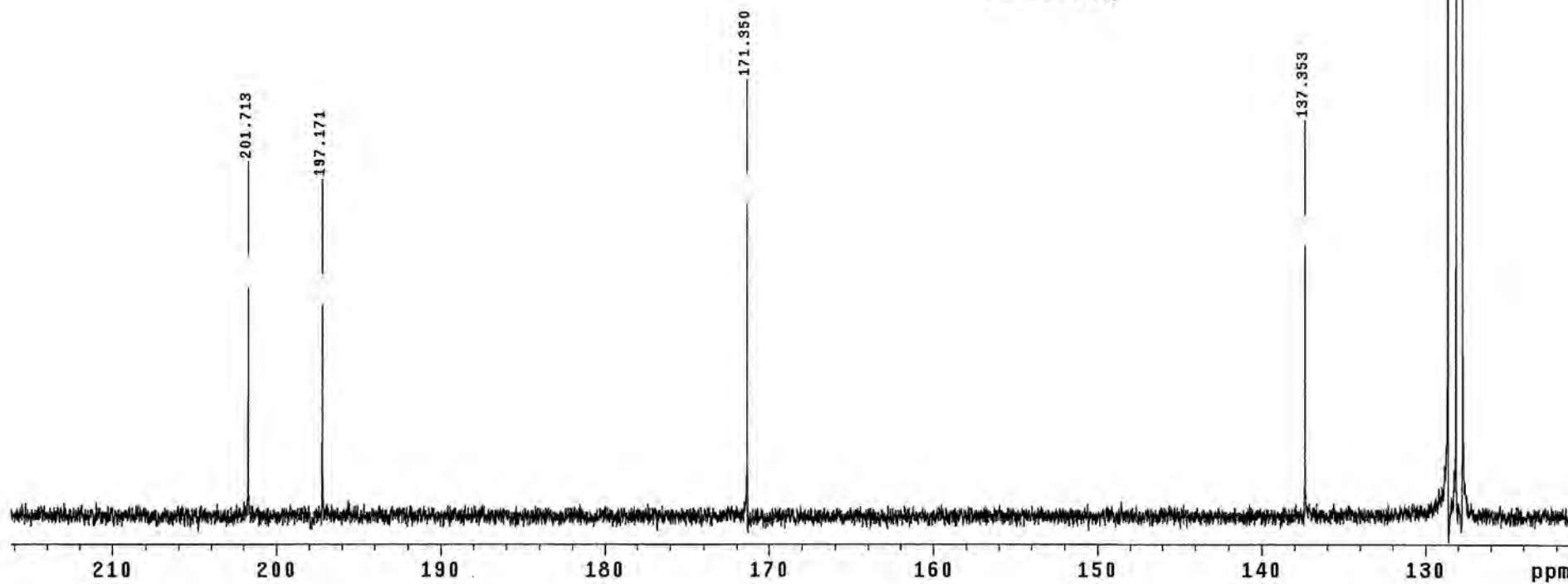
## SERVEIS CIENTIFICO-TECNICS

C13 / s2pu1 / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO  
Sist automatic

Pulse Sequence: s2pu1



COL BN-23  
Libreta nº 3  
(COLUMNA BN)



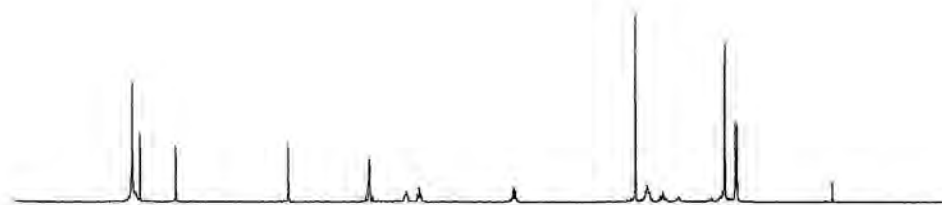
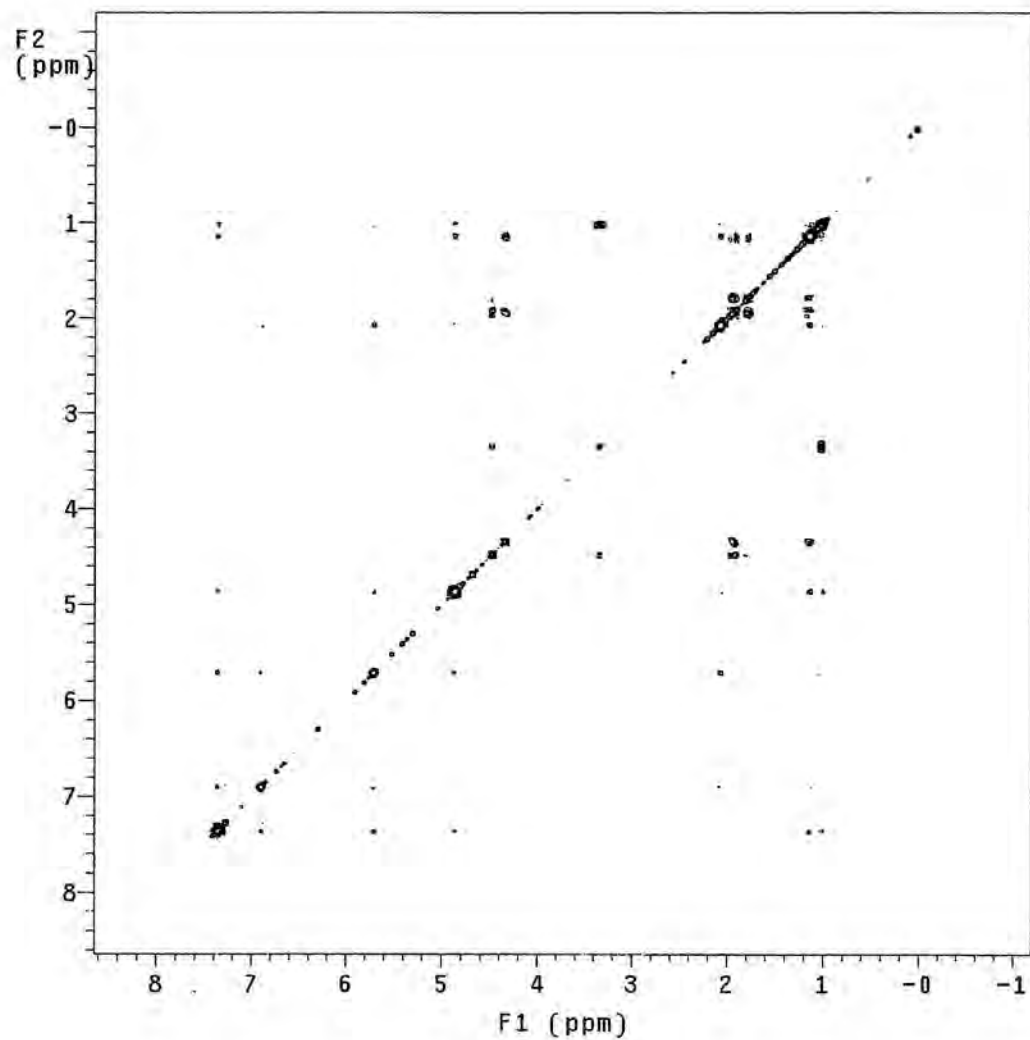
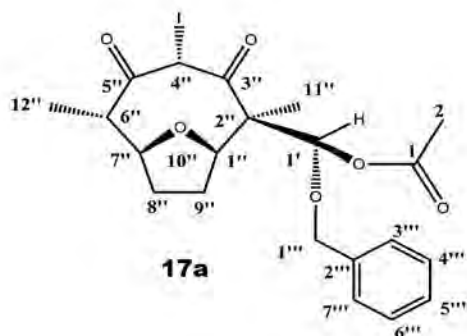
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SERVEIS CIENTÍFICO-TECNICS  
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H1 / gCOSY / Mercury-400\_qu1  
cdcl3/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3937.0 Hz  
2D Width 3937.0 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.065 sec  
FT size 2048 x 2048



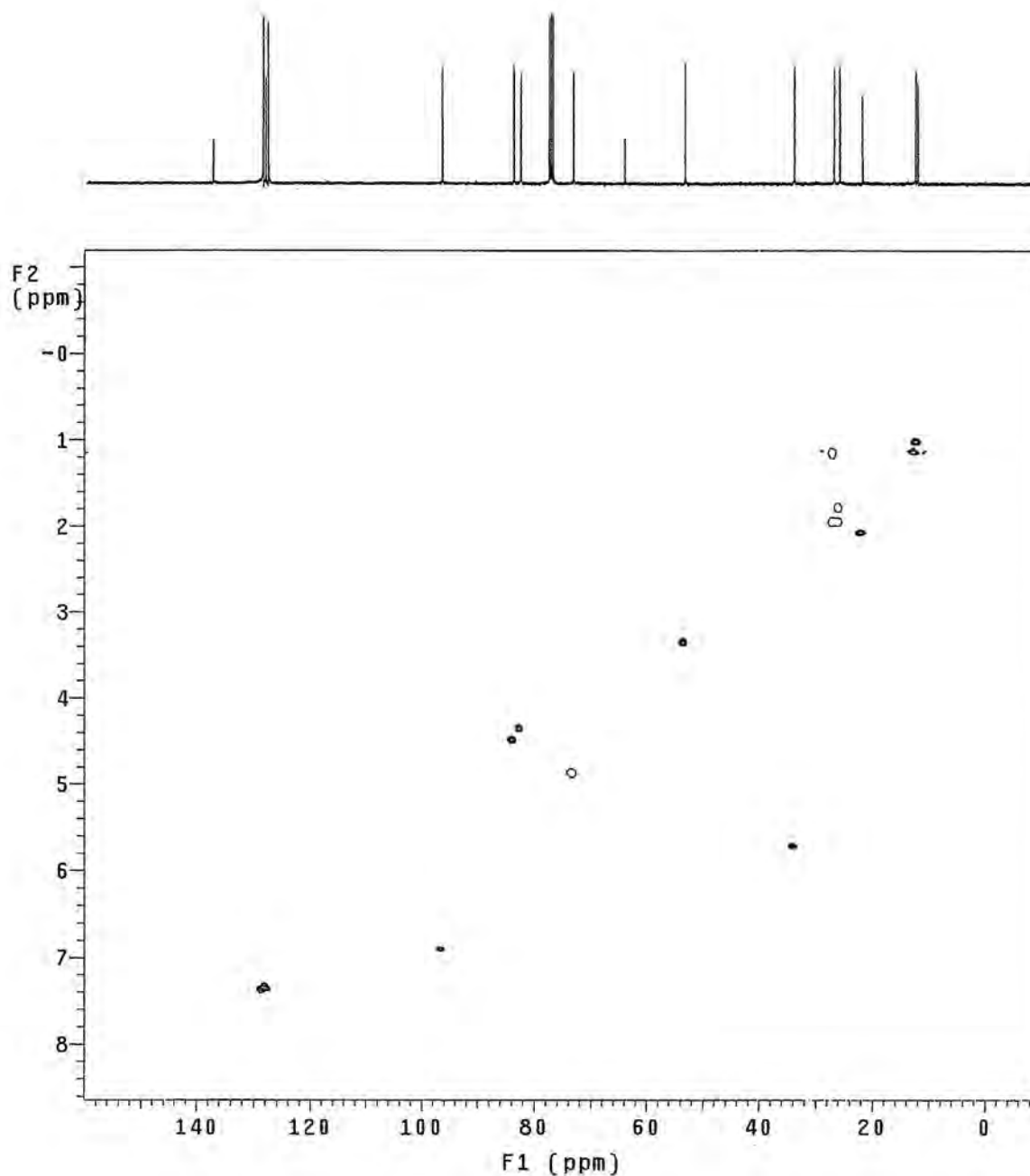
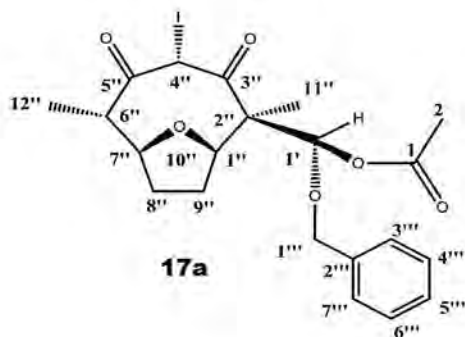
UNITAT DE RMN D'ALT CAMP  
SERVEIS CIENTÍFICO-TECNICS  
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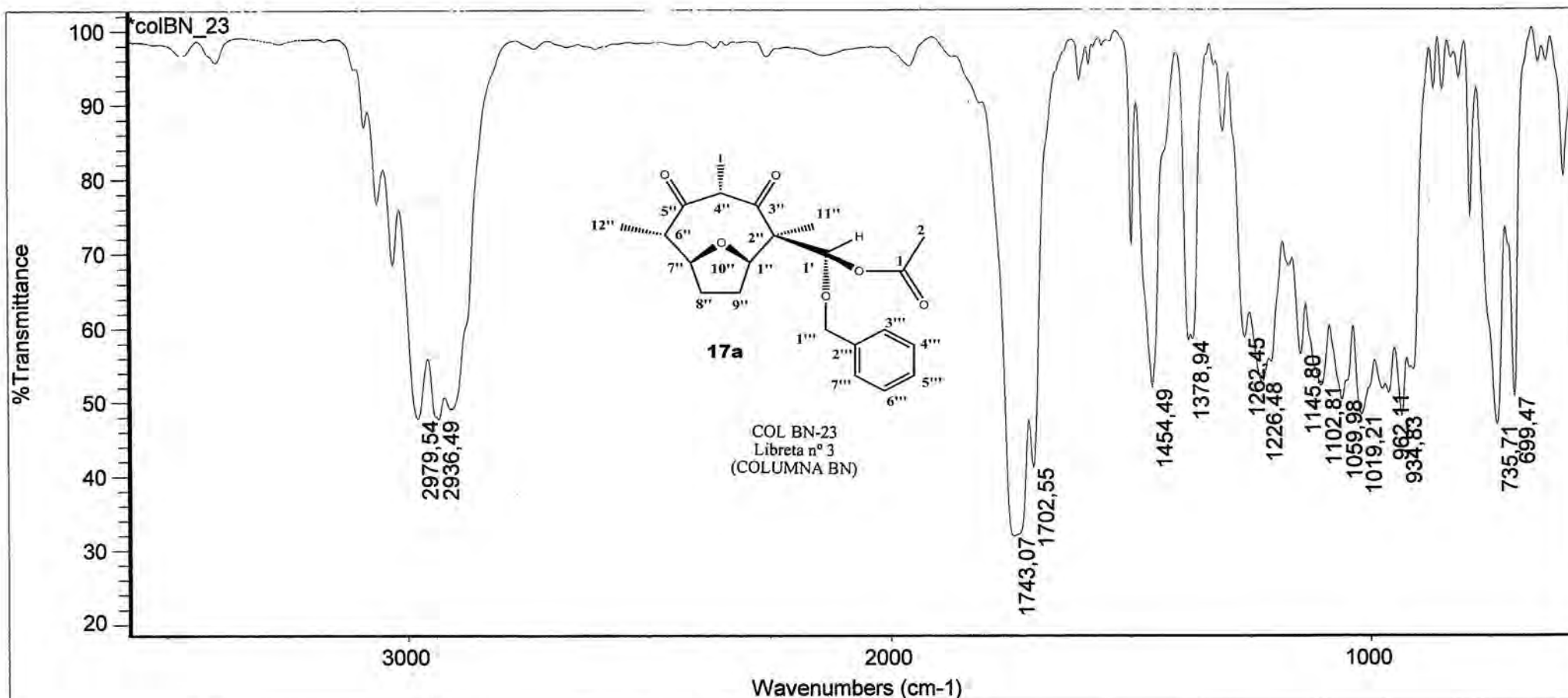
H1 / gHSQC / Mercury-400. qui  
cdc13/Temp: 25C /N reg: M40007-180407163653  
Usuari: amp / Mostra: colbn\_23  
Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.130 sec  
Width 3937.0 Hz  
2D Width 17094.0 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 400.112 MHz  
DECOUPLE C13, 100.6156477 MHz  
Power 44 dB  
on during acquisition  
off during delay  
GARP-1 modulated  
DATA PROCESSING  
Gauss apodization 0.060 sec  
F1 DATA PROCESSING  
Gauss apodization 0.007 sec  
FT size 1024 x 2048



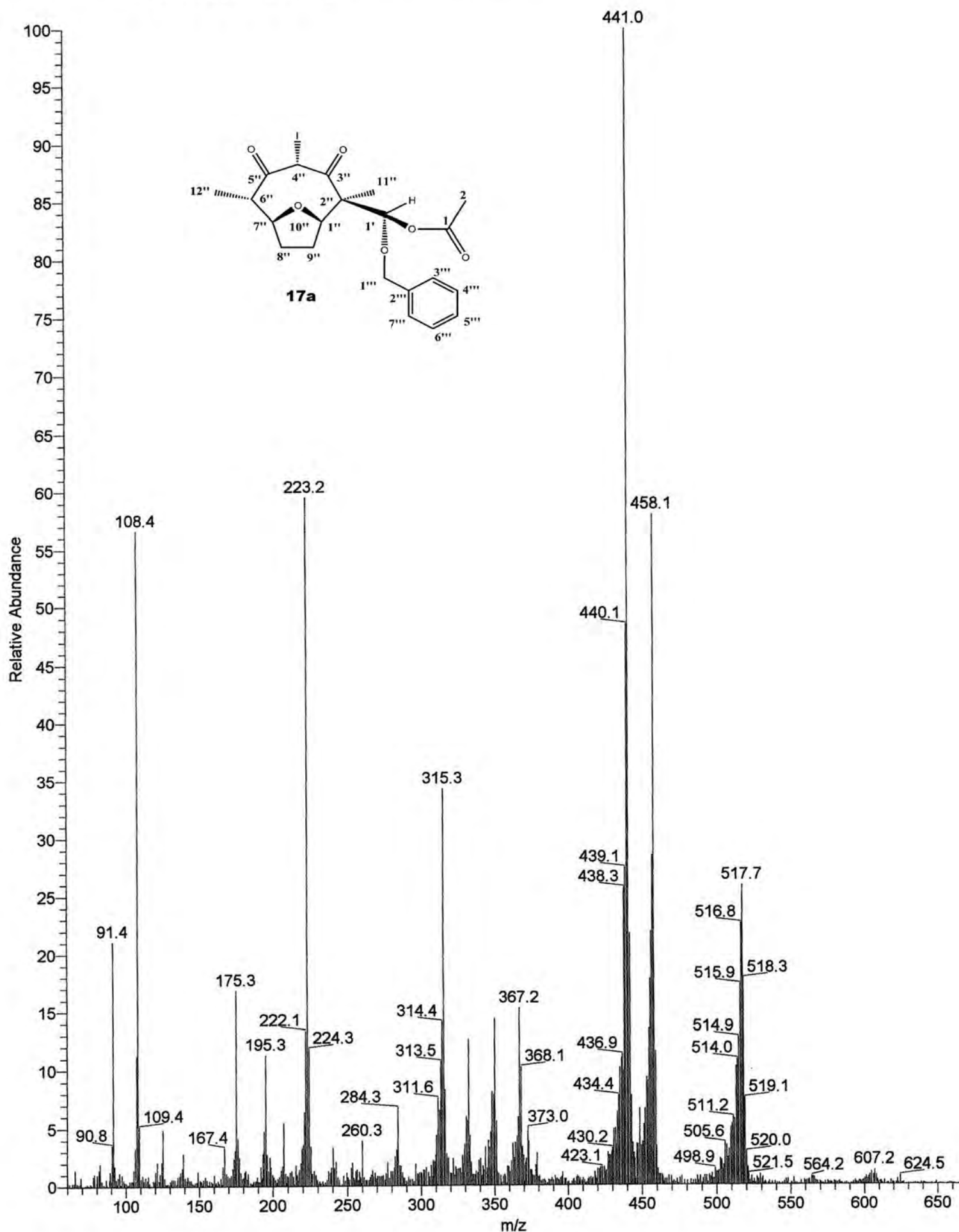
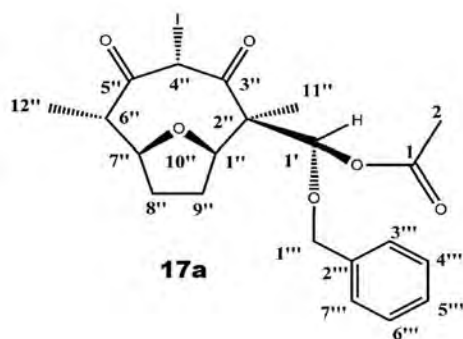


# FIND PEAKS:

Spectrum: \*colBN\_23  
Region: 3579,10 563,18  
Absolute threshold: 66,094  
Sensitivity: 50

## Peak list:

|           |         |            |        |           |         |            |        |
|-----------|---------|------------|--------|-----------|---------|------------|--------|
| Position: | 1743,07 | Intensity: | 31,924 | Position: | 1019,21 | Intensity: | 48,318 |
| Position: | 1702,55 | Intensity: | 41,171 | Position: | 1059,98 | Intensity: | 50,266 |
| Position: | 735,71  | Intensity: | 46,949 | Position: | 699,47  | Intensity: | 50,722 |
| Position: | 2936,49 | Intensity: | 47,664 | Position: | 962,11  | Intensity: | 51,209 |
| Position: | 2979,54 | Intensity: | 47,698 | Position: | 1454,49 | Intensity: | 51,881 |
| Position: | 934,83  | Intensity: | 48,101 | Position: | 1102,81 | Intensity: | 52,205 |
|           |         |            |        | Position: | 1226,48 | Intensity: | 52,750 |
|           |         |            |        | Position: | 1145,80 | Intensity: | 56,440 |
|           |         |            |        | Position: | 1378,94 | Intensity: | 58,393 |
|           |         |            |        | Position: | 1262,45 | Intensity: | 58,797 |



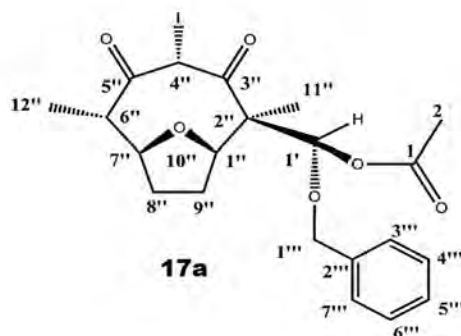
| m/z   | Intensity | Relative |
|-------|-----------|----------|
| 91.4  | 5036.8    | 21.10    |
| 107.7 | 2668.5    | 11.18    |
| 108.4 | 13511.0   | 56.61    |
| 175.3 | 4022.4    | 16.85    |
| 195.3 | 2686.6    | 11.26    |
| 222.1 | 3189.9    | 13.36    |
| 223.2 | 14203.6   | 59.51    |
| 224.3 | 2822.2    | 11.82    |
| 284.3 | 1615.1    | 6.77     |
| 311.6 | 1811.2    | 7.59     |
| 312.6 | 1554.4    | 6.51     |
| 313.5 | 2556.9    | 10.71    |
| 314.4 | 3369.1    | 14.12    |
| 315.3 | 8181.2    | 34.28    |
| 316.3 | 1979.6    | 8.29     |
| 332.3 | 3002.9    | 12.58    |
| 348.3 | 1925.0    | 8.07     |
| 349.2 | 1877.1    | 7.86     |
| 350.1 | 3426.9    | 14.36    |
| 367.2 | 3640.5    | 15.25    |
| 368.1 | 2435.6    | 10.20    |
| 434.4 | 1850.8    | 7.75     |
| 435.2 | 2418.8    | 10.13    |
| 436.1 | 2614.3    | 10.95    |
| 436.9 | 2688.4    | 11.26    |
| 437.5 | 2354.5    | 9.86     |
| 438.3 | 6124.6    | 25.66    |
| 439.1 | 6546.0    | 27.43    |
| 440.1 | 11561.9   | 48.44    |
| 441.0 | 23867.7   | 100.00   |
| 442.1 | 5179.4    | 21.70    |
| 442.9 | 1854.4    | 7.77     |
| 448.5 | 1577.8    | 6.61     |
| 452.1 | 1582.6    | 6.63     |
| 453.0 | 2218.0    | 9.29     |
| 453.9 | 2139.6    | 8.96     |
| 454.8 | 3213.2    | 13.46    |
| 455.7 | 4238.8    | 17.76    |
| 456.6 | 5218.9    | 21.87    |
| 457.3 | 6796.7    | 28.48    |
| 458.1 | 13833.8   | 57.96    |
| 459.2 | 2736.5    | 11.47    |
| 513.1 | 2431.8    | 10.19    |
| 514.0 | 2591.0    | 10.86    |
| 514.9 | 3035.1    | 12.72    |
| 515.9 | 4129.1    | 17.30    |
| 516.8 | 5390.9    | 22.59    |
| 517.7 | 6173.2    | 25.86    |
| 518.3 | 4246.5    | 17.79    |
| 519.1 | 1788.6    | 7.49     |

Ref: COL BN\_23 (EM352) Stefano Ponzano DEPCI/NH3+

DEPCI4391#53-86 RT: 0.57-0.91 AV: 34

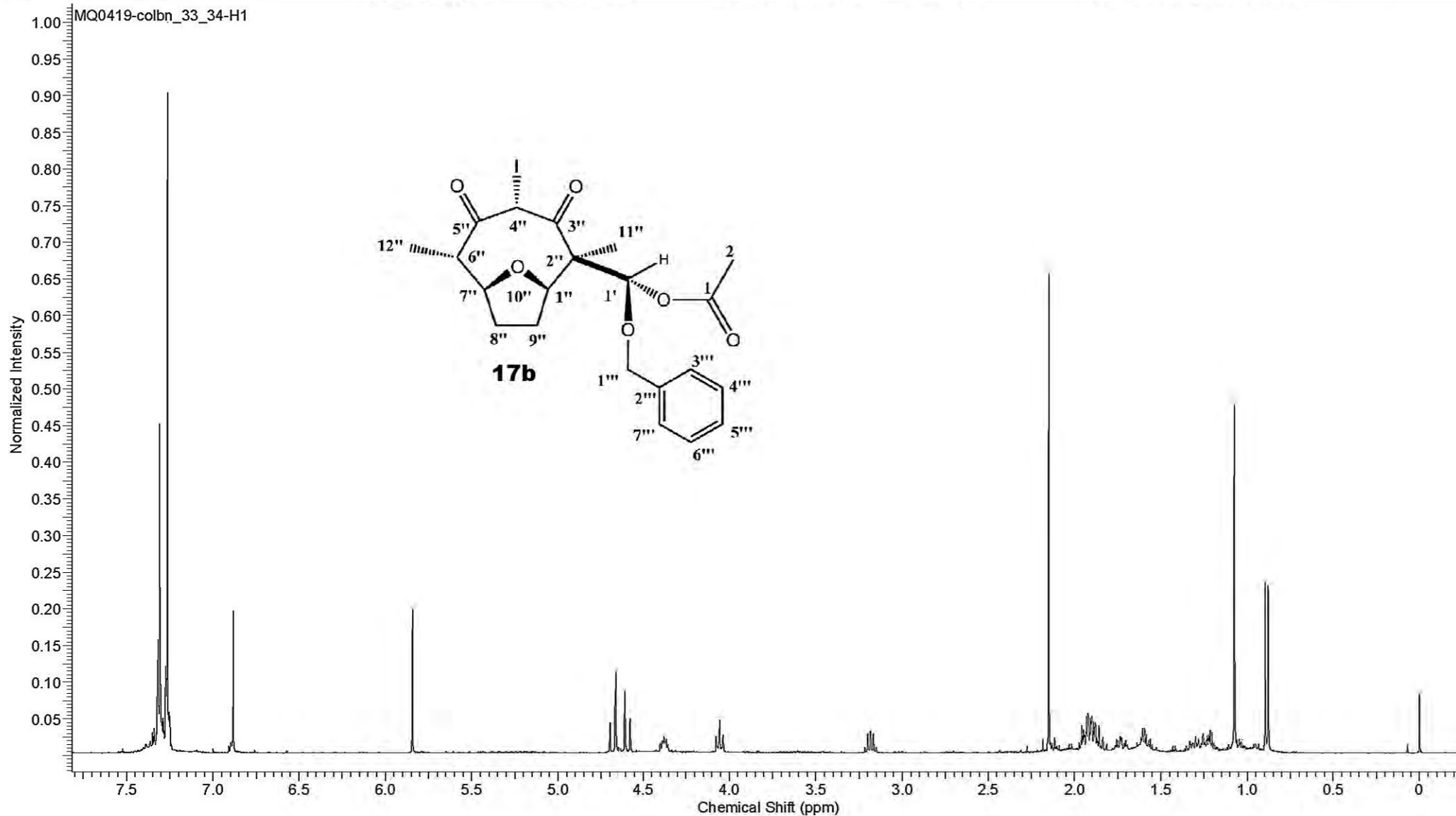
T: + c Full ms [ 60.00-800.00]

m/z= 60.0-665.8

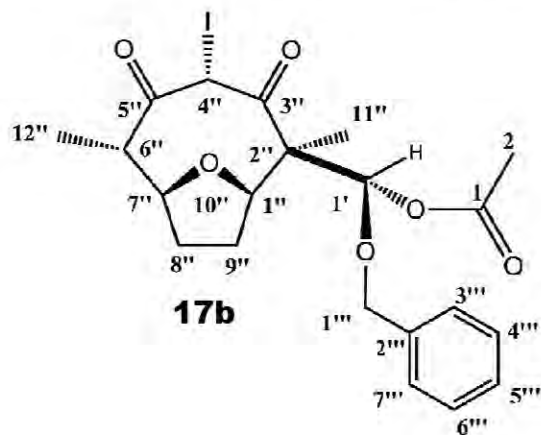
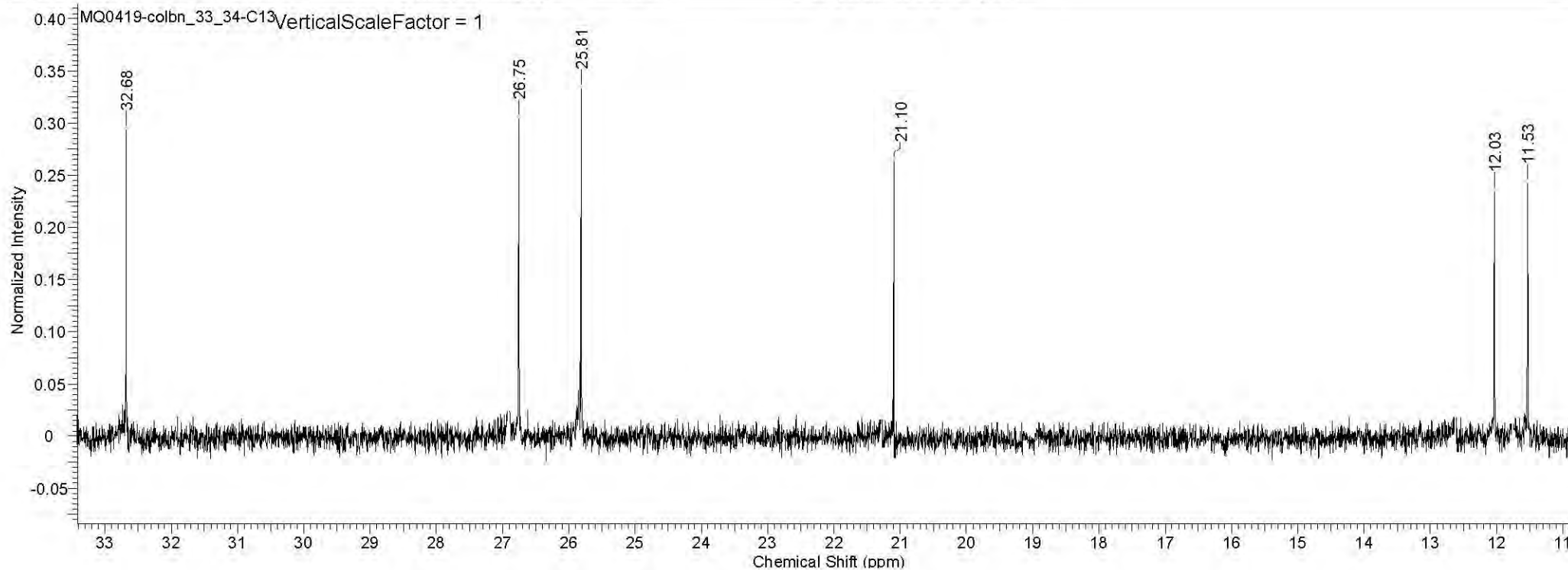


COL BN-23  
Libreta n° 3  
(COLUMNNA BN)

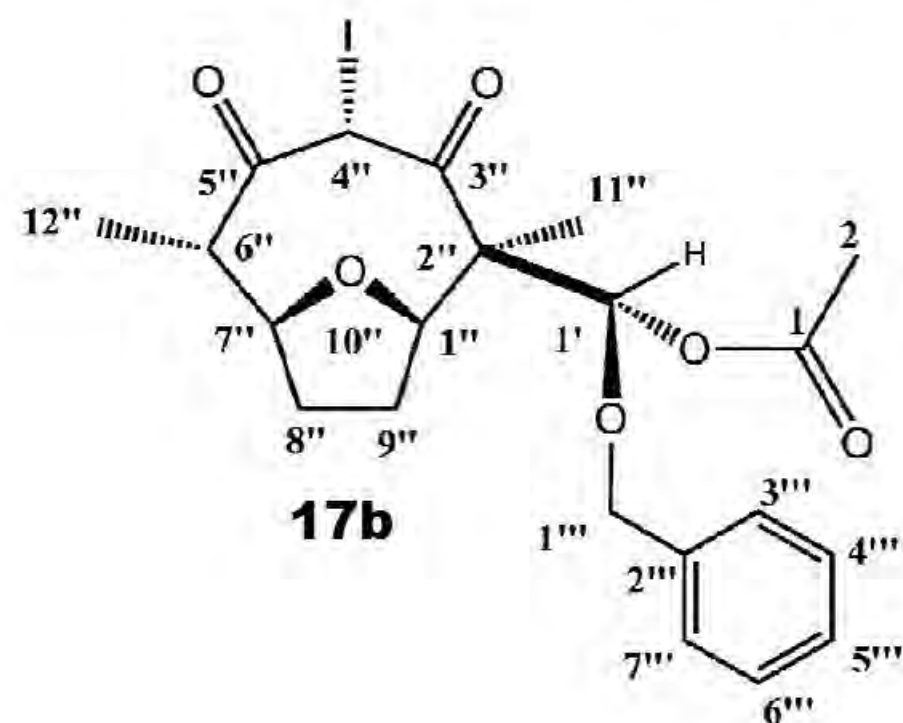
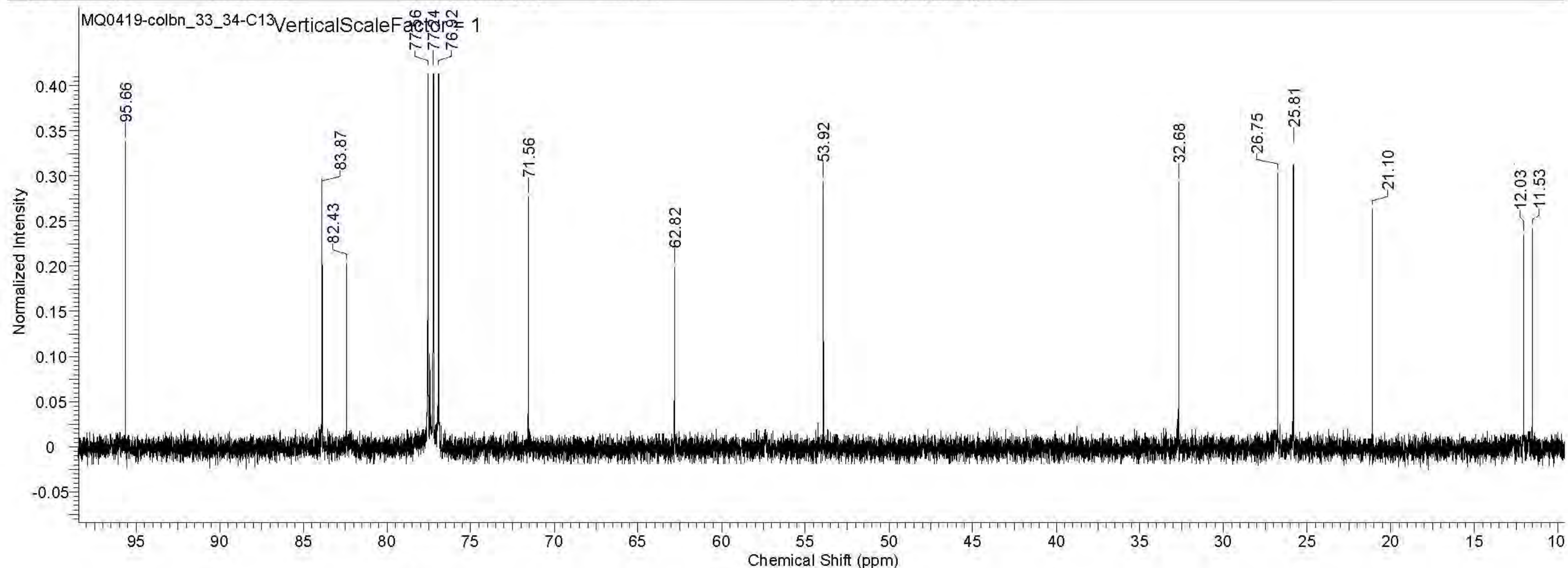
|                                                                                         |                               |                                                        |                                    |  |
|-----------------------------------------------------------------------------------------|-------------------------------|--------------------------------------------------------|------------------------------------|--|
| <b>Acquisition Time (sec)</b> 3.4998                                                    |                               |                                                        |                                    |  |
| <b>Comment</b> H1 / s2pul / Mercury-400 qui cdc13/Temp: 25C /N reg: M40007-190407170747 |                               | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                    |  |
|                                                                                         |                               | <b>File Name</b> MQ0419-colbn_33_34-H1.fid\fid         |                                    |  |
| <b>Frequency (MHz)</b> 400.11                                                           | <b>Nucleus</b> 1H             | <b>Number of Transients</b> 8                          | <b>Original Points Count</b> 13999 |  |
| <b>Points Count</b> 16384                                                               | <b>Pulse Sequence</b> s2pul   | <b>Receiver Gain</b> 32.00                             | <b>Solvent</b> CHLOROFORM-d        |  |
| <b>Spectrum Offset (Hz)</b> 1802.4902                                                   | <b>Spectrum Type</b> STANDARD | <b>Sweep Width (Hz)</b> 4000.00                        | <b>Temperature (degree C)</b> 25.0 |  |



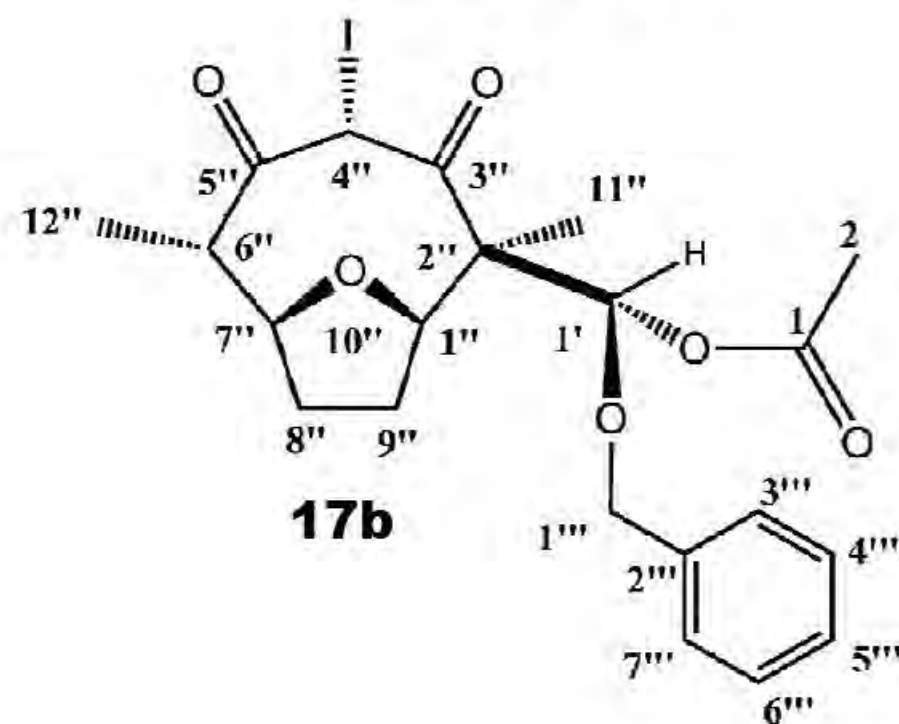
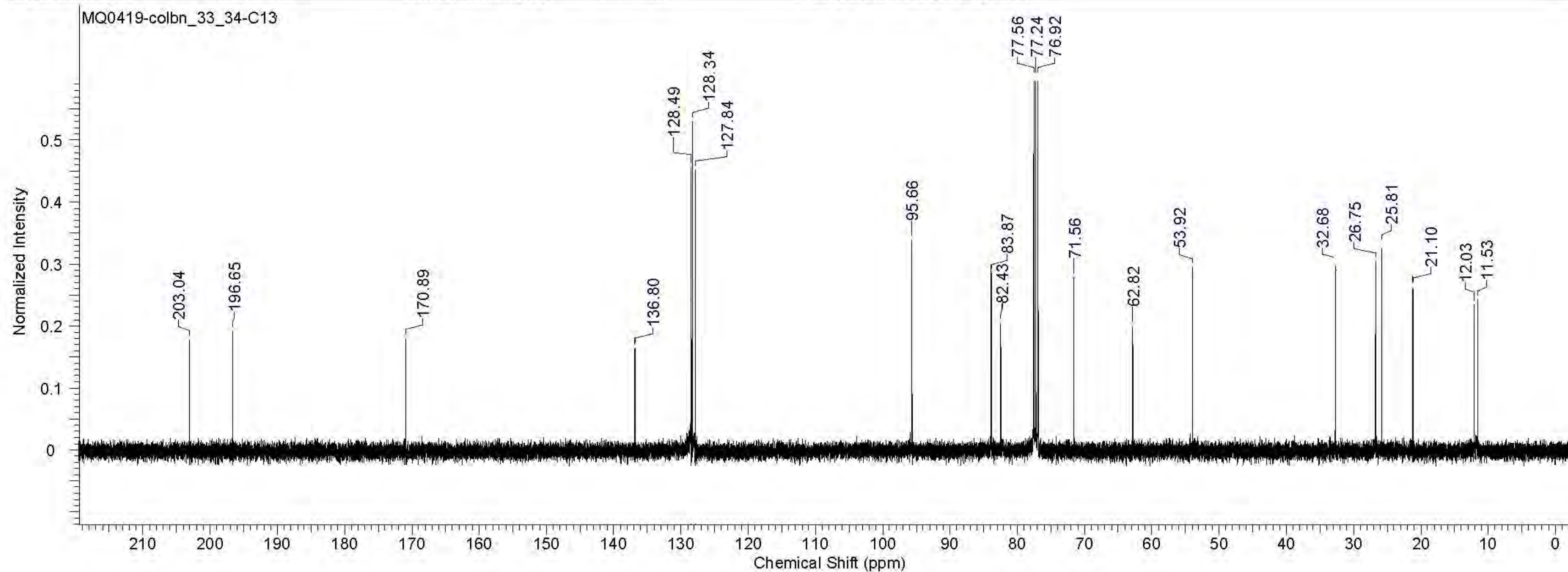
|                                                                                          |          |                                                        |                                |
|------------------------------------------------------------------------------------------|----------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |          |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |          | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |          | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                   | 100.62   | <b>Nucleus</b>                                         | 13C                            |
| <b>Original Points Count</b>                                                             | 33241    | <b>Points Count</b>                                    | 65536                          |
| <b>Receiver Gain</b>                                                                     | 39.00    | <b>Solvent</b>                                         | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                     | STANDARD | <b>Sweep Width (Hz)</b>                                | 27700.83                       |
|                                                                                          |          | <b>Number of Transients</b>                            | 4000                           |
|                                                                                          |          | <b>Pulse Sequence</b>                                  | s2pul                          |
|                                                                                          |          | <b>Spectrum Offset (Hz)</b>                            | 11318.4121                     |
|                                                                                          |          | <b>Temperature (degree C)</b>                          | 25.0                           |



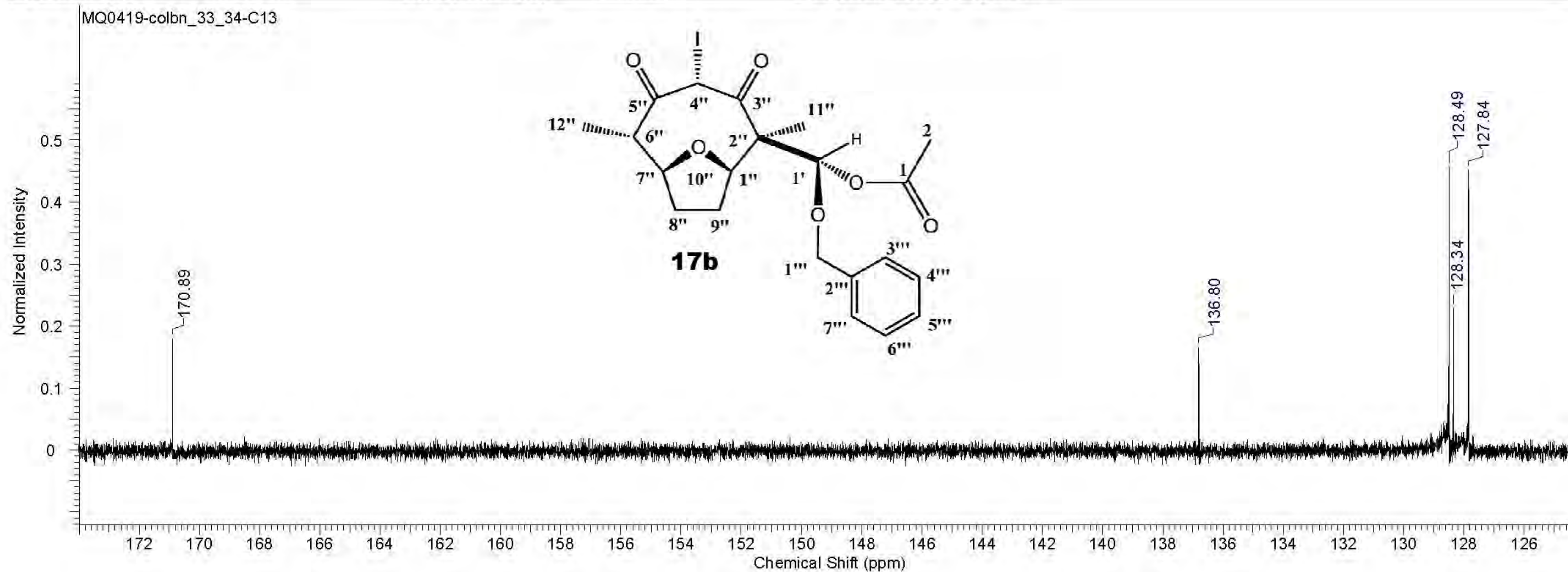
|                                                                                                                                                 |          |                               |                                |
|-------------------------------------------------------------------------------------------------------------------------------------------------|----------|-------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                                                                            |          |                               |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |          |                               |                                |
|                                                                                                                                                 |          | <b>File Name</b>              | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                                                                          | 100.62   | <b>Nucleus</b>                | 13C                            |
| <b>Original Points Count</b>                                                                                                                    | 33241    | <b>Points Count</b>           | 65536                          |
| <b>Receiver Gain</b>                                                                                                                            | 39.00    | <b>Solvent</b>                | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                                                                            | STANDARD | <b>Sweep Width (Hz)</b>       | 27700.83                       |
|                                                                                                                                                 |          | <b>Number of Transients</b>   | 4000                           |
|                                                                                                                                                 |          | <b>Pulse Sequence</b>         | s2pul                          |
|                                                                                                                                                 |          | <b>Spectrum Offset (Hz)</b>   | 11318.4121                     |
|                                                                                                                                                 |          | <b>Temperature (degree C)</b> | 25.0                           |



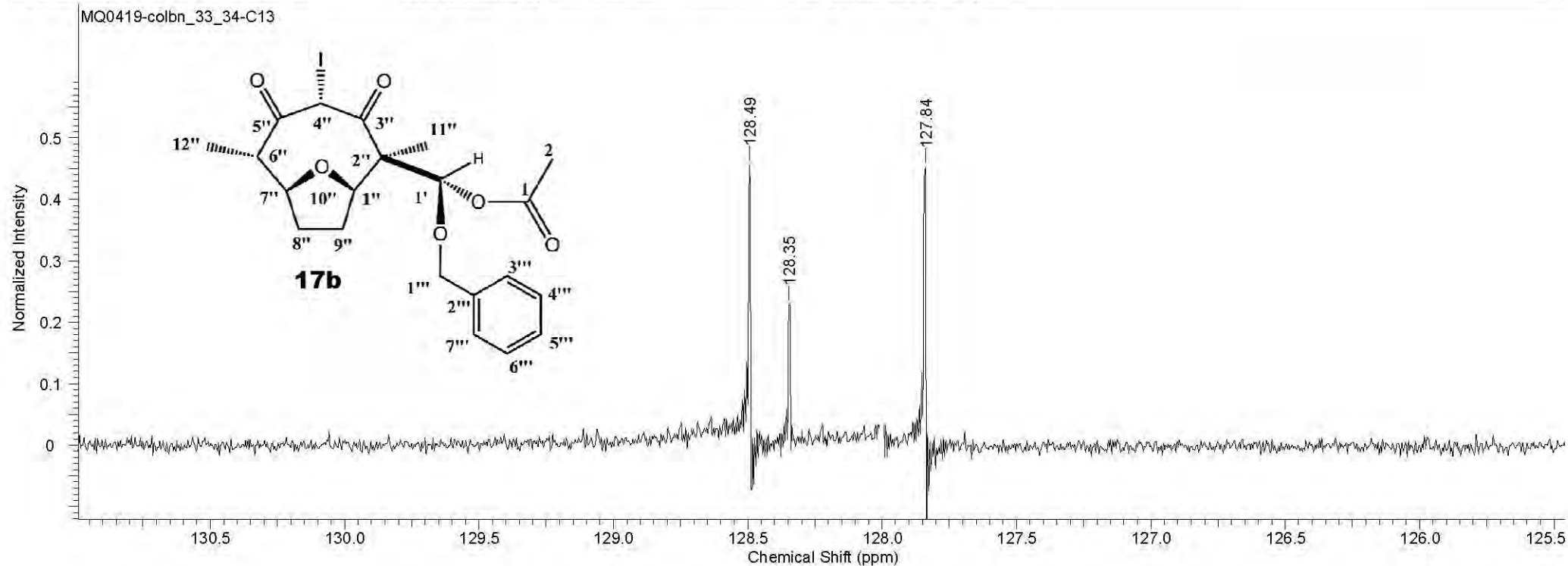
|                                                                                          |          |                                                        |                                |
|------------------------------------------------------------------------------------------|----------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |          |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |          | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |          | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                   | 100.62   | <b>Nucleus</b>                                         | 13C                            |
| <b>Original Points Count</b>                                                             | 33241    | <b>Points Count</b>                                    | 65536                          |
| <b>Receiver Gain</b>                                                                     | 39.00    | <b>Solvent</b>                                         | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                     | STANDARD | <b>Sweep Width (Hz)</b>                                | 27700.83                       |
|                                                                                          |          | <b>Number of Transients</b>                            | 4000                           |
|                                                                                          |          | <b>Pulse Sequence</b>                                  | s2pul                          |
|                                                                                          |          | <b>Spectrum Offset (Hz)</b>                            | 11318.4121                     |
|                                                                                          |          | <b>Temperature (degree C)</b>                          | 25.000                         |



|                                                                                          |          |                                                        |                                |
|------------------------------------------------------------------------------------------|----------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |          |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |          | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |          | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b>                                                                   | 100.62   | <b>Nucleus</b>                                         | 13C                            |
| <b>Original Points Count</b>                                                             | 33241    | <b>Points Count</b>                                    | 65536                          |
| <b>Receiver Gain</b>                                                                     | 39.00    | <b>Solvent</b>                                         | CHLOROFORM-d                   |
| <b>Spectrum Type</b>                                                                     | STANDARD | <b>Sweep Width (Hz)</b>                                | 27700.83                       |
|                                                                                          |          | <b>Number of Transients</b>                            | 4000                           |
|                                                                                          |          | <b>Pulse Sequence</b>                                  | s2pul                          |
|                                                                                          |          | <b>Spectrum Offset (Hz)</b>                            | 11318.4121                     |
|                                                                                          |          | <b>Temperature (degree C)</b>                          | 25.000                         |



|                                                                                          |                                  |                                                        |                                |
|------------------------------------------------------------------------------------------|----------------------------------|--------------------------------------------------------|--------------------------------|
| <b>Acquisition Time (sec)</b> 1.2000                                                     |                                  |                                                        |                                |
| <b>Comment</b> C13 / s2pul / Mercury-400 qui cdcl3/Temp: 25C /N reg: M40007-190407170747 |                                  | Usuari: amp / Mostra: colbn_33_34 Nom: STEFANO PONZANO |                                |
|                                                                                          |                                  | <b>File Name</b>                                       | MQ0419-colbn_33_34-C13.fid\fid |
| <b>Frequency (MHz)</b> 100.62                                                            | <b>Nucleus</b> 13C               | <b>Number of Transients</b> 4000                       |                                |
| <b>Original Points Count</b> 33241                                                       | <b>Points Count</b> 65536        | <b>Pulse Sequence</b> s2pul                            |                                |
| <b>Receiver Gain</b> 39.00                                                               | <b>Solvent</b> CHLOROFORM-d      | <b>Spectrum Offset (Hz)</b> 11318.4121                 |                                |
| <b>Spectrum Type</b> STANDARD                                                            | <b>Sweep Width (Hz)</b> 27700.83 | <b>Temperature (degree C)</b> 25.0                     |                                |

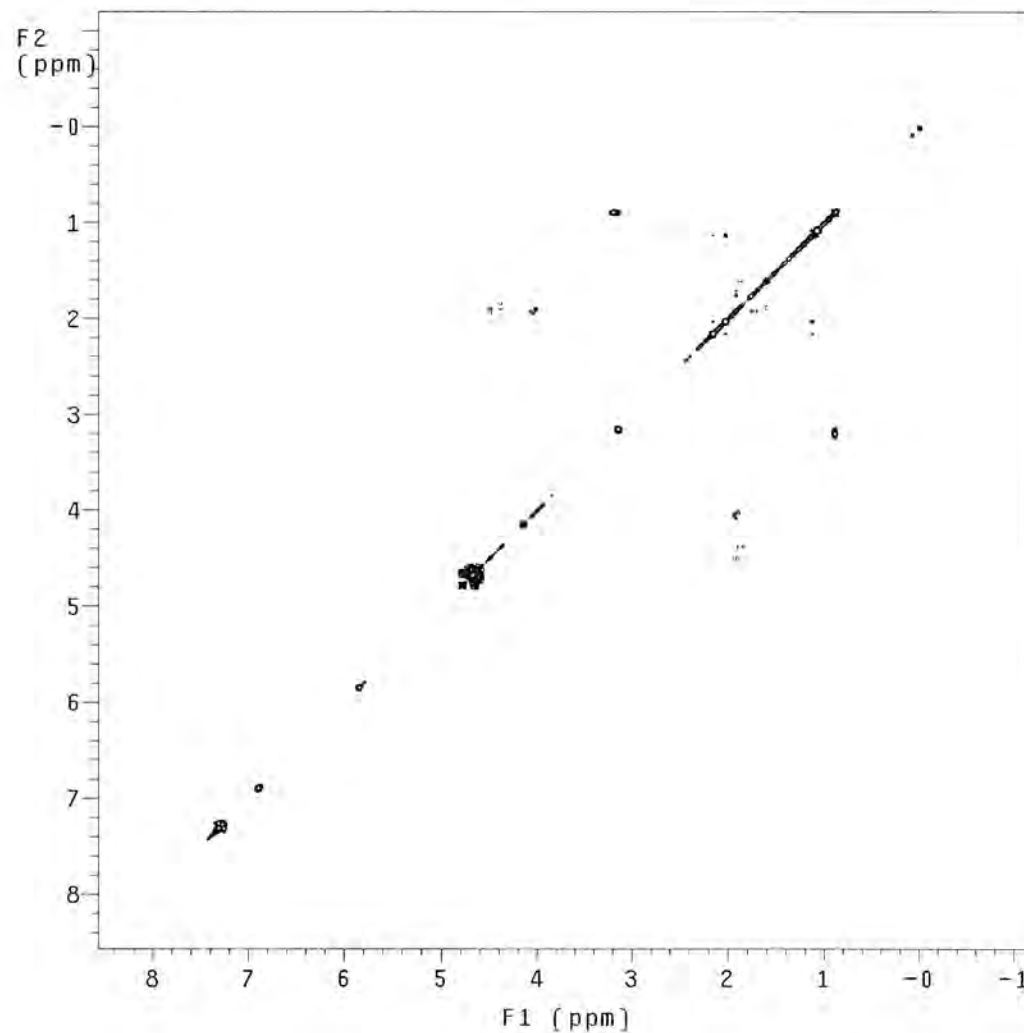
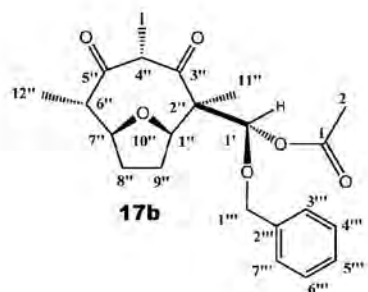


H1 / gCOSY / Mercury-400\_qui  
cdc13/Temp: 25C /N reg: M40007-190407170747  
Usuari: amp / Mostra: colbn\_33\_34  
Nom: STEFANO PONZANO

Pulse Sequence: gCOSY  
\*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 3906.2 Hz  
2D Width 3906.2 Hz  
2 repetitions  
256 increments  
OBSERVE H1, 400.112 MHz  
DATA PROCESSING  
Sq. sine bell 0.075 sec  
F1 DATA PROCESSING  
Sq. sine bell 0.066 sec  
FT size 2048 x 2048



H1 / gHSQC / Mercury-400\_qui  
 cdc13/Temp: 25C /N reg: M40007-190407170747  
 Usuari: amp / Mostra: colbn\_33\_34  
 Nom: STEFANO PONZANO

Pulse Sequence: gHSQC  
 \*\*\*\*\*

Mercury-400BB "fenix"

Relax. delay 1.000 sec  
 Acq. time 0.131 sec  
 Width 3906.2 Hz  
 2D Width 17094.0 Hz  
 4 repetitions  
 2 x 128 increments  
 OBSERVE H1, 400.112 MHz  
 DECOUPLE C13, 100.6156477 MHz  
 Power 44 dB  
 on during acquisition  
 off during delay  
 GARP-1 modulated  
 DATA PROCESSING  
 Gauss apodization 0.061 sec  
 F1 DATA PROCESSING  
 Gauss apodization 0.007 sec  
 FT size 1024 x 2048

