

## Photoredox catalysis in the synthesis of $\gamma$ - and $\delta$ -lactams from *N*-alkenyl trichloro- and dichloroacetamides

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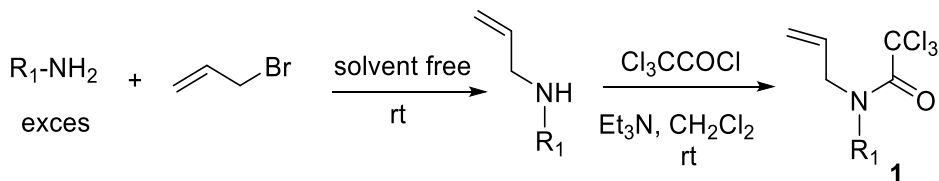
## 1. General information

Unless otherwise stated, all reactions were carried out under air atmosphere with commercially available solvents. Photocatalyzed reactions were carried out under air atmosphere in a 5 mL or a 10 mL glass vial using blue LED strip light ( $\lambda = 435\text{-}445\text{ nm}$ ). Reagents and solvents were used as received without further purification. All product mixtures were analyzed by thin-layer chromatography performed on  $\text{SiO}_2$  (Merck silica gel 60 F<sub>254</sub>) and the spots were located by UV light ( $\lambda = 254\text{ nm}$ ) and/or a 1%  $\text{KMnO}_4$  aqueous solution. Flash chromatography was carried out on  $\text{SiO}_2$  (Carlo Erba silica gel 60A, 35–70  $\mu$ ). Drying of organic extracts during the reaction workup was performed over anhydrous  $\text{Na}_2\text{SO}_4$  and solvent evaporation was accomplished with a rotatory evaporator.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Mercury 400, a Varian VNMRS 400, a Bruker 400 and a Bruker 500 spectrometers in  $\text{CDCl}_3$ . Chemical shifts are reported as  $\delta$  values (ppm) relative to internal  $\text{Me}_4\text{Si}$ ,  $^{13}\text{C}$  NMR spectra are referenced to the deuterated solvent signal ( $\text{CDCl}_3$ : 77.00 ppm). All NMR data assignments are supported by COSY and HSQC experiments. The following abbreviations (or combinations) were used to describe  $^1\text{H}$ -NMR multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextuplet, m = multiplet, b = broad. Infrared spectra were recorded on a Nicolet 320 FT-IR spectrophotometer. Melting points were recorded on a Gallenkamp melting point apparatus.

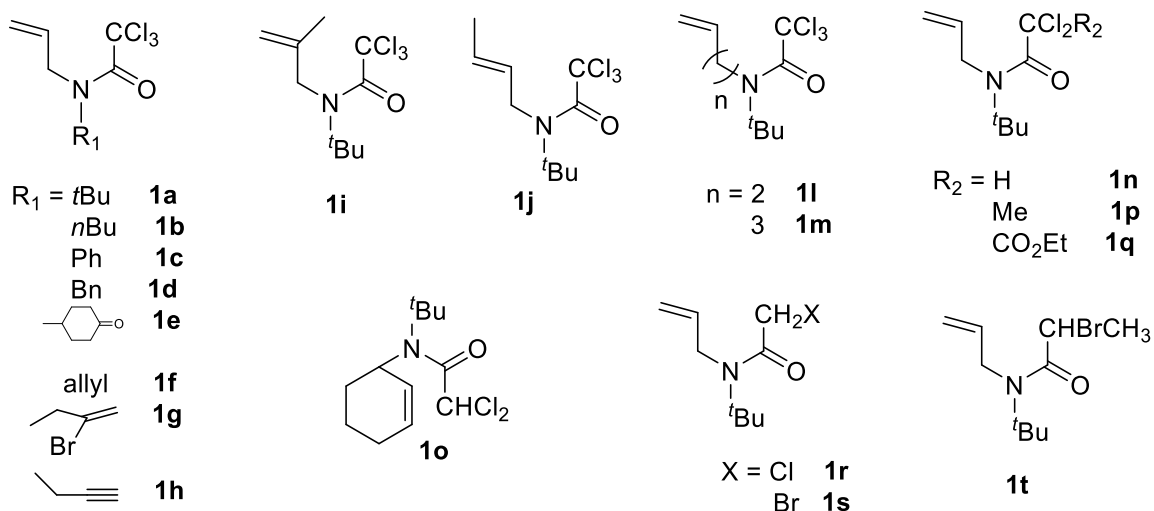
## 2. Preparation and characterization of amides 1a-1j and 1l-1t

Amides **1a-1j** and **1l-1t** were prepared using a two steps sequence: alkylation of the primary amine with the corresponding alkenyl bromide and further reaction of the secondary amine with the appropriate acetyl chloride derivative to yield the corresponding amide **1**.

- *Typical procedure for the preparation of 1a-1h*

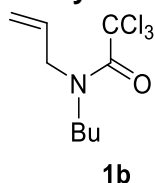


A mixture of allyl bromide (1 eq) and primary amine  $\text{R}_1\text{NH}_2$  (3-5 eq) was stirred at rt overnight the excess of amine was evaporated and the residue treated with trichloroacetyl chloride (1.5 eq) and triethylamine (2 eq) in dichloromethane at rt overnight. The mixture was then concentrated and purified by chromatography using a mixture of Hexane/EtOAc as eluent to provide trichloroacetamides **1** with acceptable to excellent yields.



The spectroscopy data of the following substrates was previously reported as it is indicated: **1a** see Li *et al.*<sup>1</sup>, for **1c**, **1d** and **1f** see Nagashima *et al.*<sup>2</sup>, for the preparation and spectra of **1e** see Diaba *et al.*<sup>3</sup>

#### N-Allyl-N-butyl-2,2,2-trichloroacetamide (**1b**)



Physical state: colorless oil

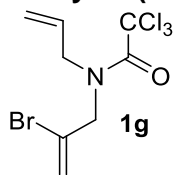
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 5.90-5.74 (m, 1H), 5.37-5.12 (m, 2H, CH<sub>2</sub>), 4.32 and 4.04 (2 br s, 2H), 3.64 and 3.37 (2 br s, 2H), 1.77-1.52 (m, 2H), 1.33 (sext, *J* = 7.3 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 160.1 (C=O), 132.4 and 131.6 (CH), 119.0 and 117.7 (CH<sub>2</sub>), 93.3 (CCl<sub>3</sub>), 52.3 and 50.7 (CH<sub>2</sub>), 49.3 and 47.9 (CH<sub>2</sub>), 29.8 and 28.3 (CH<sub>2</sub>), 19.8 (CH<sub>2</sub>), 13.8 (CH<sub>3</sub>); IR (NaCl) 3085, 3016, 2960, 2934, 2874, 1682, 1647 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>15</sub>Cl<sub>3</sub>NO 258.0214 [M+H]<sup>+</sup>, found 258.0215. Calcd. for C<sub>9</sub>H<sub>18</sub>Cl<sub>3</sub>N<sub>2</sub>O 275.0479 [M+NH<sub>4</sub>]<sup>+</sup>, found 275.0481. Calcd. for C<sub>9</sub>H<sub>14</sub>Cl<sub>3</sub>NNaO 280.0033 [M+Na]<sup>+</sup>, found 280.0037.

<sup>1</sup> Z. Zhang, L. Zhu, C. Li, *Chin. J. Chem.*, 2019, **37**, 452.

<sup>2</sup> Y. Motoyama, K. Kamo, A. Yuasa, H. Nagashima, *Chem. Commun.*, 2010, **46**, 2256.

<sup>3</sup> F. Diaba, J. A. Montiel, G. Serban, J. Bonjoch, *Org. Lett.*, 2015, **17**, 3860.

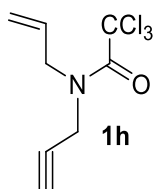
***N*-Allyl-*N*-(2-bromoallyl)-2,2,2-trichloroacetamide (1g)**



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.94-5.58 (m, 3H), 5.41-5.18 (m, 2H), 4.53 and 4.41 (2 br s, 2H), 4.29 and 4.06 (2 br s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.4 (C=O), 131.4 (CH), 126.3 (C), 120.0 ( $\text{CH}_2$ ), 118.7 ( $\text{CH}_2$ ), 92.6 ( $\text{CCl}_3$ ), 55.7 and 53.8 ( $\text{CH}_2$ ), 51.9 and 50.6 ( $\text{CH}_2$ ); IR (NaCl) 3085, 3016, 2987, 2931, 2875, 1682, 1642, 1629  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_8\text{H}_{10}\text{BrCl}_3\text{NO}$  319.9006  $[\text{M}+\text{H}]^+$ , found 319.9008. Calcd. for  $\text{C}_8\text{H}_{13}\text{BrCl}_3\text{N}_2\text{O}$  336.9271  $[\text{M}+\text{NH}_4]^+$ , found 336.9265.

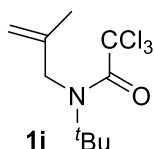
***N*-Allyl-2,2,2-trichloro-*N*-(prop-2-yn-1-yl)acetamide (1h)**



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.83 (ddt,  $J = 16.2, 9.9, 5.9$  Hz, 1H), 5.33 (br d,  $J = 13.3$  Hz, 2H), 4.45 (br s, 2H), 4.24 (br s, 2H), 2.30 (s, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.8 (C=O), 131.0 (CH), 120.1 and 119.0 ( $\text{CH}_2$ ), 92.5 ( $\text{CCl}_3$ ), 77.3 (C), 72.9 (CH), 51.6 and 50.3 ( $\text{CH}_2$ ), 38.9 and 36.8 ( $\text{CH}_2$ ); IR (NaCl) 3303, 3085, 3018, 2986, 2929, 2973, 2124, 1681, 1645  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_8\text{H}_9\text{Cl}_3\text{NO}$  239.9744  $[\text{M}+\text{H}]^+$ , found 239.9743.

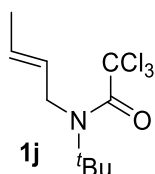
***N*-(*tert*-Butyl)-2,2,2-trichloro-*N*-(2-methylallyl)acetamide (1i)**



Physical state: white solid, m.p. 65-68  $^{\circ}\text{C}$

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.00 (br s, 1H) 4.95 (br s, 1H), 4.17 (br s, 2H), 1.70 (br s, 3H,  $\text{CH}_3$ ), 1.47 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.0 (C=O), 141.9 (C), 111.8 ( $\text{CH}_2$ ), 95.0 ( $\text{CCl}_3$ ), 61.2 (C), 51.8 ( $\text{CH}_2$ ), 27.4 ( $\text{CH}_3$ ), 20.0 ( $\text{CH}_3$ ); IR (NaCl): 3091, 2975, 2932 1685  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{17}\text{Cl}_3\text{NO}$  272.0370  $[\text{M}+\text{H}]^+$ , found, 272.0374. Calcd. for  $\text{C}_{10}\text{H}_{16}\text{Cl}_3\text{NNaO}$  294.0190  $[\text{M}+\text{Na}]^+$ , found, 294.0188.

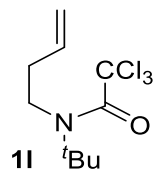
**(E)-N-(But-2-en-1-yl)-N-(tert-butyl)-2,2,2-trichloroacetamide (1j)**



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.67-5.47(m, 2H), 4.37 (br s, 2H), 1.71 (dm,  $J = 6.2$  Hz, 3H,  $\text{CH}_3$ ), 1.47 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.2 (C=O), 129.3 (CH), 127.7 (CH), 95.4 ( $\text{CCl}_3$ ), 61.0 (C), 48.7 ( $\text{CH}_2$ ), 28.1 ( $\text{CH}_3$ ), 17.8 ( $\text{CH}_3$ ); IR (NaCl) 2997, 2969, 2919, 2882, 2856, 1686, 1649  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{17}\text{Cl}_3\text{NO}$  272.0370  $[\text{M}+\text{H}]^+$ , found 272.0368. Calcd. for  $\text{C}_{10}\text{H}_{16}\text{Cl}_3\text{NNaO}$  294.0190  $[\text{M}+\text{Na}]^+$ , found 294.0187.

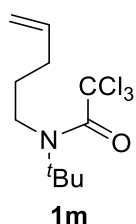
**N-(But-3-en-1-yl)-N-(tert-butyl)-2,2,2-trichloroacetamide<sup>4</sup> (1l)**



Physical state: white solid, m.p. 50-52 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.70 (ddt,  $J = 17.1, 10.3, 6.7$  Hz, 1H), 5.13-5.05 (m, 2H), 3.76 (brs, 2H), 2.48 (tdt,  $J = 8.3, 6.7, 1.3$  Hz, 2H), 1.51 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.9 (C=O), 133.5 (CH), 117.3 ( $\text{CH}_2$ ), 95.5 ( $\text{CCl}_3$ ), 60.1 (C), 46.0 ( $\text{CH}_2$ ), 35.7 ( $\text{CH}_2$ ), 28.1 ( $\text{CH}_3$ ); IR (NaCl) 3080, 2979, 2932, 1686, 1642  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{17}\text{Cl}_3\text{NO}$  272.0370  $[\text{M}+\text{H}]^+$ , found 272.0362. Calcd. for  $\text{C}_{10}\text{H}_{16}\text{Cl}_3\text{NNaO}$  294.0190  $[\text{M}+\text{Na}]^+$ , found 294.0194.

**N-(tert-Butyl)-2,2,2-trichloro-N-(pent-4-en-1-yl)acetamide (1m)**



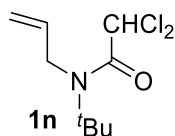
Physical state: white solid, m.p. 48-50 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.77 (ddt,  $J = 16.9, 10.2, 6.7$  Hz, 1H), 5.08-4.97 (m, 2H), 3.68 (br s, 2H), 2.04 (q,  $J = 7.3$  Hz, 2H), 1.83 (m, 2H), 1.49 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  160.0 (C=O), 136.9 (CH), 115.6 ( $\text{CH}_2$ ), 95.6 ( $\text{CCl}_3$ ), 60.1 (C), 46.7 ( $\text{CH}_2$ ),

<sup>4</sup> A. J. Clark, F. De Campo, R. J. Deeth, R. P. Filik, S. Gatard, N. A. Hunt, D. Lastécouères, G. H. Thomas, J. –B. Verlhac, H. Wongtap, *J. Chem. Soc., Perkin Trans. 1*, 2000, 671.

31.0 (CH<sub>2</sub>), 30.5 (CH<sub>2</sub>), 28.2 (CH<sub>3</sub>); IR (NaCl): 3078, 2977, 2928, 1685, 1641 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd for C<sub>11</sub>H<sub>19</sub>Cl<sub>3</sub>NO 286.0527 [M+H]<sup>+</sup>, found 286.0524. Calcd. for C<sub>11</sub>H<sub>18</sub>Cl<sub>3</sub>NNaO 308.0346 [M+Na]<sup>+</sup>, found 308.0349.

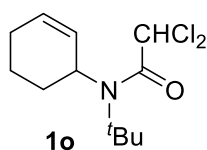
#### ***N*-Allyl-*N*-(*tert*-butyl)-2,2-dichloroacetamide (1n)**



Physical state: white solid, m.p. 48-51 °C

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.13 (s, 1H), 5.94 (ddt, *J* = 17.2, 10.5, 3.9 Hz, 1H), 5.35-5.21 (m, 2H), 4.05 (dt, *J* = 4.1, 2.1 Hz, 2H), 1.47 (s, 9H, *t*-Bu); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 164.6 (C=O), 135.1 (CH), 116.7 (CH<sub>2</sub>), 66.3 (CH<sub>2</sub>), 59.0 (C), 46.8 (CH<sub>2</sub>), 28.0 (CH<sub>3</sub>); IR (NaCl) 3088, 2978, 2932, 1678 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>16</sub>Cl<sub>2</sub>NO 224.0603 [M+H]<sup>+</sup>, found 224.0604. Calcd. for C<sub>9</sub>H<sub>10</sub>Cl<sub>2</sub>N<sub>2</sub>O 241.0869 [M+NH<sub>4</sub>]<sup>+</sup>, found 241.0873. Calcd. for C<sub>9</sub>H<sub>15</sub>Cl<sub>2</sub>NNaO 246.0423 [M+Na]<sup>+</sup>, found 246.0426.

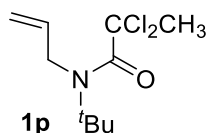
#### ***N*-(*tert*-Butyl)-2,2-dichloro-*N*-(cyclohex-2-en-1-yl)acetamide (1o)**



Physical state: white solid, m.p. 65–67 °C

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.47 (s, 1H), 5.91 (br s, 1H), 5.64 (br d, *J* = 10.3 Hz, 1H), 4.33 (s, 2H), 2.15-2.01 (m, 3H), 1.97-1.87 (m, 1H), 1.80-1.60 (m, 2H), 1.47 (s, 9H, *t*-Bu); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 167.1 (C=O), 130.9, 130.1, 66.6, 59.7, 52.8, 31.5, 28.6, 24.0, 22.6. HRMS (ESI-TOF) *m/z*: [M+H]<sup>+</sup> calcd. for C<sub>12</sub>H<sub>20</sub>Cl<sub>2</sub>NO 264.0916, found 264.0920.

#### ***N*-Allyl-*N*-(*tert*-butyl)-2,2-dichloropropanamide (1p)**

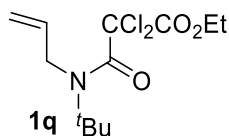


Physical state: colorless oil

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 5.97 (ddt, *J* = 17.4, 10.6, 5.4 Hz, 1H), 5.25-5.13 (m, 2H), 4.54 (br s, 2H), 2.30 (s, 3H), 1.46 (s, 9H, *t*-Bu); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.3 (C=O), 138.0 (CH), 115.9 (CH<sub>2</sub>), 82.0 (C), 59.8 (C), 49.1 (CH<sub>2</sub>), 37.3 (CH<sub>3</sub>), 28.2 (CH<sub>3</sub>);

IR (NaCl) 3082, 2980, 2943, 1665  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{18}\text{Cl}_2\text{NO}$  238.0760  $[\text{M}+\text{H}]^+$ , found 238.0764.

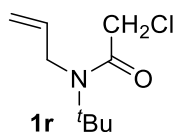
### Ethyl 3-(allyl(*tert*-butyl)amino)-2,2-dichloro-3-oxopropanoate (1q)



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.94 (ddt,  $J = 17.2, 10.5, 3.9$  Hz, 1H), 5.25-5.13 (m, 2H), 4.40 (q,  $J = 6.5$  Hz, 1H), 4.12 (ddt,  $J = 19.7, 3.9, 2.2$  Hz, 1H), 4.02 (ddt,  $J = 19.7, 4.0, 2.0$  Hz, 1H), 1.76 (d,  $J = 6.5$  Hz, 3H), 1.45 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.9 (C=O), 162.3 (C=O), 136.5 (CH), 116.5 ( $\text{CH}_2$ ), 81.8 ( $\text{CCl}_2$ ), 64.4 ( $\text{CH}_2$ ), 60.3 (C), 48.5 ( $\text{CH}_2$ ), 28.0 ( $\text{CH}_3$ ), 13.7 ( $\text{CH}_3$ ); IR (NaCl) 3088, 2981, 2934, 2975, 1769, 1735, 1663  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{12}\text{H}_{20}\text{Cl}_2\text{NO}_3$  296.0815  $[\text{M}+\text{H}]^+$ , found 296.0813.

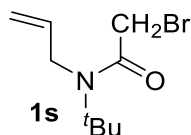
### *N*-Allyl-*N*-(*tert*-butyl)-2-chloroacetamide (1r)



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.88 (ddt,  $J = 17.1, 10.6, 4.2$  Hz, 1H), 5.28-5.17 (m, 2H), 4.02 (m, 4H), 1.46 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.1 (C=O), 135.5 (CH), 116.1 ( $\text{CH}_2$ ), 58.1 (C), 47.1 ( $\text{CH}_2$ ), 43.9 ( $\text{CH}_2$ ), 28.3 ( $\text{CH}_3$ ); IR (NaCl) 3087, 2977, 2930, 1658  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_9\text{H}_{17}\text{ClNO}$  190.0993  $[\text{M}+\text{H}]^+$ , found 190.0993. Calcd. for  $\text{C}_9\text{H}_{16}\text{ClNNaO}$  212.0813  $[\text{M}+\text{Na}]^+$ , found 212.0812.

### *N*-Allyl-2-bromo-*N*-(*tert*-butyl)acetamide (1s)

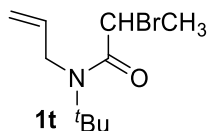


Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.90 (ddt,  $J = 17.1, 10.6, 4.2$  Hz, 1H), 5.27-5.18 (m, 2H), 4.06 (m, 2H), 3.78 (s, 2H), 1.45 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.5 (C=O), 135.7 (CH), 116.0 ( $\text{CH}_2$ ), 58.1 (C), 47.8 ( $\text{CH}_2$ ), 29.8 ( $\text{CH}_2$ ), 28.3 ( $\text{CH}_3$ ); IR (NaCl) 3086,

2966, 2929, 1652  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_9\text{H}_{17}\text{BrNO}$ : 234.0488  $[\text{M}+\text{H}]^+$ , found 234.0484. Calcd. for  $\text{C}_9\text{H}_{16}\text{BrNNaO}$ : 256.0307  $[\text{M}+\text{Na}]^+$ , found 256.0306.

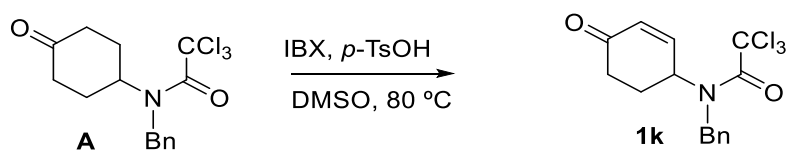
### ***N*-Allyl-2-bromo-*N*-(*tert*-butyl)propanamide (1t)**



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.87 (ddt,  $J = 17.4, 10.5, 5.3$  Hz, 1H), 5.23-5.16 (m, 2H), 4.35 (q,  $J = 7.1$  Hz, 2H), 4.23 (br s, 2H), 1.46 (s, 9H, *t*-Bu), 1.35 (t,  $J = 7.1$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2 (C=O), 136.3 (CH), 115.5 ( $\text{CH}_2$ ), 58.0 (C), 47.0 ( $\text{CH}_2$ ), 41.7 (CH), 28.3 ( $\text{CH}_3$ ), 22.1 ( $\text{CH}_3$ ); IR (NaCl) 3086, 2973, 2923, 1658  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{19}\text{BrNO}$  248.0645  $[\text{M}+\text{H}]^+$ , found 248.0642. Calcd. for  $\text{C}_{10}\text{H}_{18}\text{BrNNaO}$  270.0464  $[\text{M}+\text{Na}]^+$ , found 270.0485.

### **3. Preparation of 1k**



A mixture of **A**<sup>5</sup> (0.3 g, 0.86 mmol), IBX (293 mg, 0.1 mmol) and a catalytic amount of *p*-toluenesulfonic acid monohydrate in DMSO (3 mL) was heated to 80  $^\circ\text{C}$  overnight. The mixture was then partitioned between water and AcOEt. The organic layer was sequentially washed with aqueous saturated  $\text{NaHCO}_3$ , aqueous saturated  $\text{Na}_2\text{S}_2\text{O}_3$  then dried and concentrated. After chromatography using a mixture of Hexane/EtOAc as eluent, **1k** was isolated (180 mg, 60%).

Physical state: white foam

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52-7.07 (m, 5H), 6.85 (br d,  $J = 10.7$  Hz, 1H), 6.22-5.60 and 5.36-4.03 (m, 4H), 2.80-1.86 (m, 4H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  196.9 and 196.4 (C=O), 160.6 (C=O), 151.2, 149.3, 136.5, 135.0, 132.4, 128.9, 128.7, 127.3, 126.3, 93.5 and 92.9 ( $\text{CCl}_3$ ), 58.2, 57.2, 54.1, 49.3, 39.6, 36.7, 29.5, 28.8; IR (NaCl) 3088, 3063, 3032, 2959, 2879, 1681, 1604  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{15}\text{H}_{15}\text{Cl}_3\text{NO}_2$  346.0163

<sup>5</sup> J. Quirante, C. Escolano, F. Diaba, J. Bonjoch, *Heterocycles*, 1999, **50**, 731.

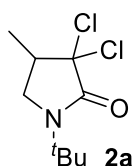


[M+H]<sup>+</sup>, found, 346.0165. Calcd. for C<sub>15</sub>H<sub>14</sub>Cl<sub>3</sub>NNaO<sub>2</sub> 367.9982 [M+Na]<sup>+</sup>, found 367.9980.

#### 4. Photocatalyzed synthesis and characterization of lactams **2** and **3**

A mixture of haloamide **1** (0.2 mmol), DIPEA<sup>6</sup> (0.18 mL, 1.0 mmol, 5 eq) and *fac*-Ir(ppy)<sub>3</sub> (1.3 mg, 0.002 mmol, 1 mol%) in THF/acetone (4 ml, 1:1) was stirred at rt under blue LED irradiation overnight. The mixture was then concentrated and purified by chromatography using a mixture of Hexane/EtOAc (1:0 to 1:1) as eluent, to provide the corresponding lactam **2**. In some cases lactams **3** were also isolated.

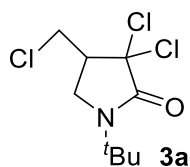
##### 1-(*tert*-Butyl)-3,3-dichloro-4-methylpyrrolidin-2-one (**2a**)



Physical state: white solid, m.p. 115–117 °C

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.47 (dd, *J* = 9.7, 6.9 Hz, 1H, H-5), 3.47 (dd, *J* = 9.7, 8.5 Hz, 1H, H-5), 2.67 (dq, *J* = 8.5, 6.6 Hz, 1H, H-4), 1.42 (s, 9H, *t*-Bu), 1.30 (d, *J* = 6.6 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.7 (C-2), 88.6 (C-3), 55.1 (C), 48.3 (C-5), 44.6 (C-4), 27.2 (CH<sub>3</sub>), 11.7 (CH<sub>3</sub>); IR (NaCl) 2977, 2937, 2881, 1705 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>16</sub>Cl<sub>2</sub>NO 224.0603 [M+H]<sup>+</sup>, found 224.0601. Calcd. for C<sub>9</sub>H<sub>15</sub>Cl<sub>2</sub>NNaO 246.0423 [M+Na]<sup>+</sup>, found 246.0420.

##### 1-(*tert*-Butyl)-3,3-dichloro-4-(chloromethyl)pyrrolidin-2-one (**3a**)



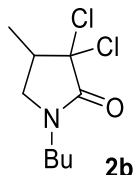
Physical state: white solid, m.p. 70–73 °C

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.99 (dd, *J* = 11.3, 4.2 Hz, 1H), 3.73 (d, *J* = 11.3 Hz, 1H), 3.70 (dd, *J* = 9.0, 5.0 Hz, 1H, H-5), 3.22 (dd, *J* = 10.0, 8.2 Hz, 1H, H-5), 3.05-2.93 (m, 1H, H-4), 1.44 (s, 9H, *t*-Bu); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.6 (C-2), 85.0 (C-3), 55.7 (C), 51.0 (C-4), 46.0 (C-5), 41.2 (CH<sub>2</sub>Cl), 27.2 (CH<sub>3</sub>); IR (NaCl) 3054, 2983, 2935, 1724

<sup>6</sup> With substrate **1a**, **1b**, **1i** and **1l** when DIPEA was replaced by K<sub>2</sub>CO<sub>3</sub>, the reaction was carried out using the same conditions.

cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>15</sub>Cl<sub>3</sub>NO 258.0214 [M+H]<sup>+</sup>, found 258.0205. Calcd. for C<sub>9</sub>H<sub>14</sub>Cl<sub>3</sub>NNaO 280.0033 [M+Na]<sup>+</sup>, found 280.0019.

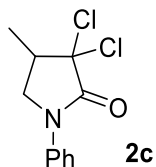
### 1-Butyl-3,3-dichloro-4-methylpyrrolidin-2-one (2b)



Physical state: colorless oil

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.44 (dt, *J* = 13.7, 7.4 Hz, 1H), 3.33 (dd, *J* = 9.8, 7.0 Hz, 1H, H-5), 3.26 (dt, *J* = 13.7, 7.0 Hz, 1H), 3.05 (dd, *J* = 9.8, 8.5 Hz, 1H, H-5), 2.78 (dq, *J* = 8.4, 6.7 Hz, 1H, H-4), 1.62-1.47 (m, 2H), 1.38-1.26 (m, 2H), 1.37-1.27 (m, 2H), 1.33 (d, *J* = 6.6 Hz, 3H, CH<sub>3</sub>), 0.94 (t, *J* = 7.3 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.9 (C=O), 87.4 (C-3), 50.0 (C-5), 45.4 (C-4), 43.4 (CH<sub>2</sub>), 29.0 (CH<sub>2</sub>), 19.8 (CH<sub>2</sub>), 13.7 (CH<sub>3</sub>), 11.8 (CH<sub>3</sub>); IR (NaCl) 2960, 2932, 2973, 1720 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>16</sub>Cl<sub>2</sub>NO 224.0603 [M+H]<sup>+</sup>, found 224.0604. Calcd. for C<sub>9</sub>H<sub>15</sub>Cl<sub>2</sub>NNaO 246.0423 [M+Na]<sup>+</sup>, found 246.0423.

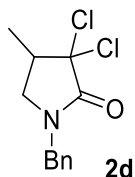
### 3,3-Dichloro-4-methyl-1-phenylpyrrolidin-2-one (2c)



Physical state: white solid, m.p. 111-114 °C

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.62 (m, 2H), 7.41 (m, 2H), 7.23 (m, 1H), 3.82 (dd, *J* = 9.6, 7.0 Hz, 1H, H-5), 3.54 (dd, *J* = 9.6, 8.9 Hz, 1H, H-5), 2.93 (dq, *J* = 8.9, 6.7 Hz, 1H, H-4), 1.43 (d, *J* = 6.6 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.5 (C-2), 138.3, 129.1, 125.8, 120.0 (Ar), 87.6 (C-3), 51.2 (C-5), 44.7 (C-4), 11.7 (CH<sub>3</sub>); IR (NaCl) 3115, 3045, 2982, 2938, 2882, 1714 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>11</sub>H<sub>12</sub>Cl<sub>2</sub>NO 244.0290 [M+H]<sup>+</sup>, found 244.0288. Calcd. for C<sub>11</sub>H<sub>11</sub>Cl<sub>2</sub>NNaO 266.0110 [M+Na]<sup>+</sup>, found 266.0108.

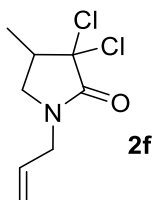
### 1-Benzyl-3,3-dichloro-4-methylpyrrolidin-2-one (2d)





2.15-2.07 (m, 2H), 2.02-1.83 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  208.0 (C=O), 165.7 (C-2), 83.5 (C-3), 51.6 (C-4), 50.1 (CH), 44.0 (C-5), 41.0 ( $\text{CH}_2$ ), 39.2 ( $\text{CH}_2$ ), 28.7 ( $\text{CH}_2$ ), 28.6 ( $\text{CH}_2$ ); IR (NaCl) 2976, 2968, 2918, 2886, 2851, 1718, 1701  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{11}\text{H}_{15}\text{Cl}_3\text{NO}_2$  298.0163  $[\text{M}+\text{H}]^+$ , found 298.0156. Calcd. for  $\text{C}_{11}\text{H}_{14}\text{Cl}_3\text{NNaO}_2$  319.9982  $[\text{M}+\text{Na}]^+$ , found 319.9970.

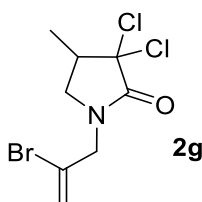
### 1-Allyl-3,3-dichloro-4-methylpyrrolidin-2-one (2f)



Physical state: white foam

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.80-5.65 (m, 1H), 5.32-5.19 (m, 2H,  $\text{CH}_2$ ), 3.95 (dm,  $J = 6.1$  Hz, 2H), 3.32 (dd,  $J = 9.8, 7.0$  Hz, 1H, H-5), 3.02 (dd,  $J = 9.8, 8.5$  Hz, 1H, H-5), 2.78 (m, 1H, H-4), 1.33 (d,  $J = 6.6$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8 (C-2), 130.9 (CH), 119.2 ( $\text{CH}_2$ ), 87.1 (C-3), 49.6 (C-5), 46.2 ( $\text{CH}_2$ ), 45.4 (C-4), 11.8 ( $\text{CH}_3$ ); IR (NaCl) 3084, 2980, 2936, 2880, 1723, 1645  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_8\text{H}_{12}\text{Cl}_2\text{NO}$  208.0290  $[\text{M}+\text{H}]^+$ , found 208.0288. Calcd. for  $\text{C}_8\text{H}_{11}\text{Cl}_2\text{NNaO}$  230.0110  $[\text{M}+\text{Na}]^+$ , found 230.0120.

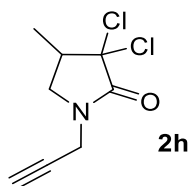
### 1-(2-Bromoallyl)-3,3-dichloro-4-methylpyrrolidin-2-one (2g)



Physical state: white solid, m.p. 80-82 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  5.84 (dt,  $J = 2.2, 1.4$  Hz, 1H), 5.67 (br d,  $J = 2.2$  Hz, 1H), 4.25 (d,  $J = 15.5$  Hz, 1H), 4.16 (d,  $J = 15.5$  Hz, 1H), 3.37 (dd,  $J = 9.7, 7.0$  Hz, 1H, H-5), 3.10 (dd,  $J = 9.7, 8.5$  Hz, 1H, H-5), 2.83 (dq,  $J = 8.5, 6.8$  Hz, 1H, H-4), 1.33 (d,  $J = 6.6$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.0 (C-2), 126.3 (C), 120.4 ( $\text{CH}_2$ ), 86.7 (C-3), 51.5 ( $\text{CH}_2$ ), 49.6 (C-5), 45.5 (C-4), 11.7 ( $\text{CH}_3$ ); IR (NaCl) 2982, 2938, 2922, 1715, 1629  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_8\text{H}_{11}\text{BrCl}_2\text{NO}$  285.9396  $[\text{M}+\text{H}]^+$ , found 285.9393. Calcd. for  $\text{C}_8\text{H}_{14}\text{BrCl}_2\text{N}_2\text{O}$  302.9661  $[\text{M}+\text{NH}_4]^+$ , found 302.9660. Calcd. for  $\text{C}_8\text{H}_{10}\text{BrCl}_2\text{NNaO}$  307.9215  $[\text{M}+\text{Na}]^+$ , found 307.9211.

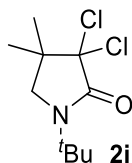
### 3,3-Dichloro-4-methyl-1-(prop-2-yn-1-yl)pyrrolidin-2-one (2h)



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.22 (dd,  $J = 17.6, 2.6$  Hz, 1H), 4.14 (dd,  $J = 17.6, 2.6$  Hz, 1H), 3.53 (dd,  $J = 9.7, 7.0$  Hz, 1H, H-5), 3.12 (dd,  $J = 9.7, 8.6$  Hz, 1H, H-5), 2.82 (dq,  $J = 8.6, 6.7$  Hz, 1H, H-4), 2.31 (t,  $J = 2.6$  Hz, 1H), 1.36 (d,  $J = 6.7$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.4 (C-2), 86.6 (C-3), 76.0 (C), 73.6 (CH), 49.1 (C-5), 45.3 (C-4), 33.3 ( $\text{CH}_2$ ), 11.7 ( $\text{CH}_3$ ); IR (NaCl) 3296, 2979, 2936, 2852, 2117, 1731  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_8\text{H}_{10}\text{Cl}_2\text{NO}$  206.0134  $[\text{M}+\text{H}]^+$ , found 206.0131. Calcd. for  $\text{C}_8\text{H}_9\text{Cl}_2\text{NNaO}$  227.9953  $[\text{M}+\text{Na}]^+$ , found 227.9951.

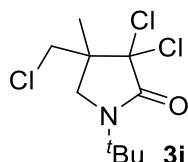
### 1-(tert-Butyl)-3,3-dichloro-4,4-dimethylpyrrolidin-2-one (2i)



Physical state: white solid, m.p. 104-106 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.20 (s, 2H, C-5), 1.41 (s, 9H, *t*-Bu), 1.27 (s, 6H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.2 (C-2), 93.1 (C-3), 54.9 (C), 54.7 (C-5), 44.5 (C-4), 27.1 ( $\text{CH}_3$ ), 22.6 ( $\text{CH}_3$ ); IR (NaCl) 2975, 2938, 2880, 1706  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{18}\text{Cl}_2\text{NO}$  238.0760  $[\text{M}+\text{H}]^+$ , found 238.0759. Calcd. for  $\text{C}_{10}\text{H}_{17}\text{Cl}_2\text{NNaO}$  260.0579  $[\text{M}+\text{Na}]^+$ , found 260.0579.

### 1-(tert-Butyl)-3,3-dichloro-4-(chloromethyl)-4-methylpyrrolidin-2-one (3i)

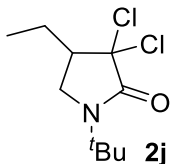


Physical state: amorphous solid

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.75 (d,  $J = 11.3$  Hz, 1H), 3.66 (d,  $J = 11.3$  Hz, 1H), 3.55 (d,  $J = 10.2$  Hz, 1H, H-5), 3.21 (d,  $J = 10.2$  Hz, 1H, H-5), 1.42 (s, 9H, *t*-Bu), 1.41 (s, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.3 (C-2), 90.7 (C-3), 55.4 (C), 50.9 ( $\text{CH}_2\text{Cl}$ ), 48.6

(C-4), 48.1 (C-5), 27.2 (CH<sub>3</sub>), 18.6 (CH<sub>3</sub>); IR (NaCl) 3055, 2986, 1721 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>10</sub>H<sub>17</sub>Cl<sub>3</sub>NO: 272.0370 [M+H]<sup>+</sup>, found 272.0373.

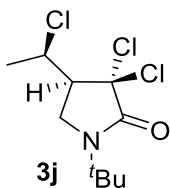
### 1-(*tert*-Butyl)-3,3-dichloro-4-ethylpyrrolidin-2-one (**2j**)



Physical state: colorless oil

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.49 (dd, *J* = 9.7, 7.0 Hz, 1H, H-5), 3.00 (dd, *J* = 9.7, 8.8 Hz, 1H, H-5), 2.50 (tdd, *J* = 8.7, 7.0, 5.4 Hz, 1H, H-4), 2.00-1.88 (m, 1H), 1.71-1.54 (m, 1H), 1.42 (s, 9H, *t*Bu), 1.07 (t, *J* = 7.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.9 (C=O), 88.0 (C-3), 55.2 (C), 50.8 (C-4), 47.0 (C-5), 27.2 (CH<sub>3</sub>), 20.9 (CH<sub>2</sub>), 11.2 (CH<sub>3</sub>); IR (NaCl) 2969, 2931, 2879, 1713 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>10</sub>H<sub>18</sub>Cl<sub>2</sub>NO 238.0760 [M+H]<sup>+</sup>, found 238.0756. Calcd. for C<sub>10</sub>H<sub>17</sub>Cl<sub>2</sub>NNaO 260.0579 [M+Na]<sup>+</sup>, found 260.0582.

### (*RS*)-1-(*tert*-Butyl)-3,3-dichloro-4-((*SR*)-1-chloroethyl)pyrrolidin-2-one<sup>7</sup> (**3j**)

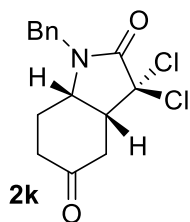


Physical state: white solid m.p. 70-73 °C

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.33 (dq, *J* = 9.9, 6.5 Hz, 1H), 3.74 (dd, *J* = 10.3, 7.2 Hz, 1H, H-5), 3.17 (dd, *J* = 10.3, 9.0 Hz, 1H, H-5), 2.81 (ddd, *J* = 9.9, 9.0, 7.2 Hz, 1H, H-4), 1.84 (d, *J* = 6.5 Hz, 3H, CH<sub>3</sub>), 1.44 (s, 9H, *t*Bu); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 165.7 (C=O), 84.8 (C-3), 56.9 (CH), 55.6 (C), 55.6 (C-4), 47.0 (C-5), 27.2 (CH<sub>3</sub>), 23.8 (CH<sub>3</sub>); IR (NaCl): 3054, 2986, 1719 cm<sup>-1</sup>; HRMS (ESI-TOF): calculated for C<sub>10</sub>H<sub>17</sub>Cl<sub>3</sub>NO: 272.0370 [M+H]<sup>+</sup>, found 272.0372. Calculated for C<sub>10</sub>H<sub>16</sub>Cl<sub>3</sub>NNaO: 294.0190 [M+Na]<sup>+</sup>, found 294.0195.

<sup>7</sup> For the relative configuration of **3i**: M. Pattarozzi, F. Roncaglia, L. Accorsi, A. F. Parsons, F. Ghelfi, *Tetrahedron*, 2010, **66**, 1357.

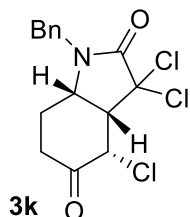
**(3aRS,7aSR)-1-Benzyl-3,3-dichlorohexahydro-2H-indole-2,5(3H)-dione (2k)**



Physical state: white foam

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41-7.25 (m, 5H, Ar), 5.05 (d,  $J = 15.0$  Hz, 1H), 4.17 (d,  $J = 15.0$  Hz, 1H), 3.81 (q,  $J = 6.9$  Hz, 1H, H-7a), 3.26 (q,  $J = 7.3$  Hz, 1H, H-3a), 2.77 (dd,  $J = 16.4, 7.4$  Hz, 1H, H-4), 2.68 (dd,  $J = 16.4, 7.7$  Hz, 1H, H-4), 2.36 (dt,  $J = 15.3, 5.1$  Hz, 1H), 2.24-2.03 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  206.5 (C-5), 166.4 (C-2), 134.7, 129.1, 128.4, 128.1, 85.3 (C-3), 52.4 (C-3a), 48.9 (C-7a), 45.7 ( $\text{CH}_2$ ), 38.5 (C-4), 35.6 (C-6), 24.4 (C-7); IR (NaCl): 3064, 3033, 2973, 2912, 2885, 1719  $\text{cm}^{-1}$ ; HRMS (ESI-TOF): calculated for  $\text{C}_{15}\text{H}_{16}\text{Cl}_2\text{NO}_2$ : 312.0553  $[\text{M}+\text{H}]^+$ , found 312.0552. Calculated for  $\text{C}_{15}\text{H}_{15}\text{Cl}_2\text{NNaO}_2$ : 334.0372  $[\text{M}+\text{Na}]^+$ , found 334.0375.

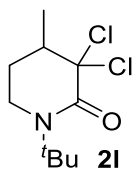
**(3aRS,4RS,7aRS)-1-Benzyl-3,3,4-trichlorohexahydro-2H-indole-2,5(3H)-dione (3k)**



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.32 (m, 3H, Ar), 7.27-7.24 (m, 2H, Ar), 4.94 (d,  $J = 14.9$  Hz, 1H,  $\text{CH}_2\text{Ar}$ ), 4.89 (d,  $J = 4.8$  Hz, 1H, H-4), 4.25 (d,  $J = 14.9$  Hz, 1H,  $\text{CH}_2\text{Ar}$ ), 3.81 (ddd,  $J = 11.3, 8.1, 5.1$  Hz, 1H, H-7a), 3.46 (dd,  $J = 8.2, 4.8$  Hz, 1H, H-3a), 2.71 (ddd,  $J = 16.9, 13.0, 6.0$  Hz, 1H, H-6ax), 2.45 (dt,  $J = 16.9, 4.3$  Hz, 1H, H-6eq), 2.27-2.18 (m, 1H, H-7eq), 2.08 (tdd,  $J = 13.5, 11.4, 5.1$  Hz, 1H, H-7ax);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  199.2 (C-5), 165.4 (C-2), 134.4, 129.2, 128.6, 128.1 (Ar), 82.6 (C-3), 57.3 (C-3a), 57.2 (C-4), 52.2 (C-7a), 46.2 ( $\text{CH}_2\text{Ar}$ ), 32.7 (C-6), 24.6 (C-7); IR (NaCl) 3062, 3031, 2922, 2851, 1727  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{15}\text{H}_{15}\text{Cl}_3\text{NO}_2$  346.0163  $[\text{M}+\text{H}]^+$ , found 346.0158.

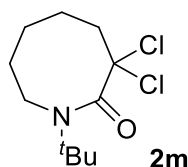
### 1-(*tert*-Butyl)-3,3-dichloro-4-methylpiperidin-2-one (2l)



Physical state: pale solid, m.p. 105-107 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.45 (ddd,  $J = 12.2, 5.5, 3.4$  Hz, 1H, H-6), 3.27 (dm,  $J = 12.2$  Hz, 1H, H-6), 2.49 (m, 1H, H-4), 1.95-1.80 (m, 2H,  $\text{CH}_2$ -5), 1.45 (s, 9H, *t*-Bu), 1.33 (d,  $J = 6.5$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  164.2 (C-2), 91.3 (C-3), 58.7 (C), 44.0 (C-4), 42.9 (C-6), 27.7 ( $\text{CH}_3$ ), 27.5 (C-5), 16.6 ( $\text{CH}_3$ ); IR (NaCl) 2980, 2935, 1662  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{10}\text{H}_{18}\text{Cl}_2\text{NO}$  238.0760  $[\text{M}+\text{H}]^+$ , found 238.0762. Calcd. for  $\text{C}_{10}\text{H}_{17}\text{Cl}_2\text{NNaO}$  260.0579  $[\text{M}+\text{Na}]^+$ , found 260.0577.

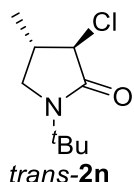
### 1-(*tert*-Butyl)-3,3-dichloroazocan-2-one (2m)



Physical state: colorless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.78 (t,  $J = 6.5$  Hz, 2H,  $\text{CH}_2$ -8), 2.96 (t,  $J = 6.4$  Hz, 2H,  $\text{CH}_2$ -4), 1.80 (m, 2H,  $\text{CH}_2$ -5), 1.69 (quint,  $J = 6.4$  Hz, 2H,  $\text{CH}_2$ -7), 1.54 (m, 2H  $\text{CH}_2$ -6), 1.46 (s, 9H, *t*-Bu);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.8 (C-2), 93.3 (C-3), 59.4 (C), 50.3 (C-4), 42.3 (C-8), 28.9 (C-7), 28.2 ( $\text{CH}_3$ ), 23.4 (C-5), 18.8 (C-6); IR (NaCl) 2962, 2935, 2865, 1668  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{11}\text{H}_{20}\text{Cl}_2\text{NO}$  252.0916  $[\text{M}+\text{H}]^+$ , found 252.0917.

### (3*RS*,4*SR*)-1-(*tert*-Butyl)-3-chloro-4-methylpyrrolidin-2-one (*trans*-2n, major diastereomer)



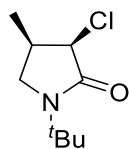
Physical state: white solid, m.p. 59-61 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.90 (d,  $J = 7.6$  Hz, 1H, H-3), 3.64 (dd,  $J = 9.7, 7.3$  Hz, 1H, H-5), 2.99 (dd,  $J = 9.7, 6.8$  Hz, 1H, H-5), 2.38 (quint,  $J = 7.1$  Hz, 1H, H-4), 1.41 (s, 9H, *t*-Bu), 1.20 (d,  $J = 6.8$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7 (C-2), 63.3 (C-3), 54.6 (C), 49.8 (C-5), 37.7 (C-4), 27.5 ( $\text{CH}_3$ ), 16.6 ( $\text{CH}_3$ ); IR (NaCl) 2961, 2924, 2852,



1686  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_9\text{H}_{17}\text{ClNO}$  190.0993  $[\text{M}+\text{H}]^+$ , found 190.0997.  
calcd. for  $\text{C}_9\text{H}_{16}\text{ClNNaO}$  212.0813  $[\text{M}+\text{Na}]^+$ , found 212.0819.

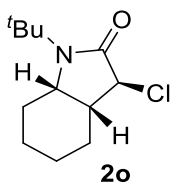
**(3*RS*,4*RS*)-1-(*tert*-Butyl)-3-chloro-4-methylpyrrolidin-2-one** (*cis*-**2n**, minor diastereomer)



*cis*-**2n**

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  4.24 (d,  $J = 6.1$  Hz, 1H, H-3), 3.47 (dd,  $J = 9.8, 6.8$  Hz, 1H, H-5), 3.14 (dd,  $J = 9.8, 8.0$  Hz, 1H, H-5), 2.51 (m, 1H, H-4), 1.40 (s, 9H, *t*-Bu), 1.16 (d,  $J = 6.7$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6 (C-2), 63.0 (C-3), 54.4 (C), 50.0 (C-5), 33.0 (C-4), 27.5 ( $\text{CH}_3$ ), 13.2 ( $\text{CH}_3$ ); IR (NaCl): 2961, 2924, 2852, 1686  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_9\text{H}_{17}\text{ClNO}$  190.0993  $[\text{M}+\text{H}]^+$ , found 190.0997.

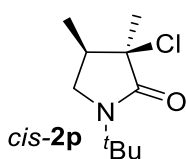
**(3*RS*,3*aSR*,7*aRS*)-1-(*tert*-Butyl)-3-chlorooctahydro-2H-indol-2-one** (**2o**)



Physical state: white solid, m.p. 130-132  $^\circ\text{C}$

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.38 (d,  $J = 12.0$  Hz, 1H, H-3), 3.62 (dt,  $J = 10.7, 6.4$  Hz, 1H, H-7a), 2.39 (dt,  $J = 12.0, 6.2$  Hz, 1H, H-3a), 2.16-2.01 (m, 2H), 1.73 (m, 1H), 1.68-1.49 (m, 2H), 1.43 (s, 9H, *t*-Bu), 1.35-1.05 (m, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.4 (C-2), 58.8 (C-3), 55.8 (C-7a), 54.8 (C), 44.1 (C-3a), 32.2 ( $\text{CH}_2$ ), 28.0 ( $\text{CH}_3$ ), 24.2 ( $\text{CH}_2$ ), 23.3 ( $\text{CH}_2$ ), 20.2 ( $\text{CH}_2$ ); IR (NaCl) 2985, 2956, 2939, 2863, 1692  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{12}\text{H}_{21}\text{ClNO}$  230.1306  $[\text{M}+\text{H}]^+$ , found 230.1302. Calcd. for  $\text{C}_{12}\text{H}_{20}\text{ClNNaO}$  252.1126  $[\text{M}+\text{Na}]^+$ , found 252.1122.

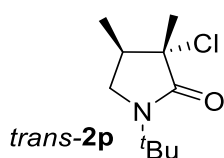
**(3*RS*,4*RS*)-1-(*tert*-Butyl)-3-chloro-3,4-dimethylpyrrolidin-2-one** (*cis*-**2p**, major diastereomer)



*cis*-**2p**

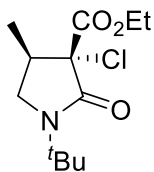
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.38 (dd,  $J = 9.7, 6.8$  Hz, 1H, H-5), 3.01 (t,  $J = 9.6$  Hz, H-5), 2.08 (m, 1H, H-4), 1.40 (s, 9H, t-Bu), 1.16 (d,  $J = 6.6$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  172.3 (C-2), 73.5 (C-3), 54.3 (C), 48.8 (C-5), 40.1 (C-4), 27.4 ( $\text{CH}_3$ ), 23.8 ( $\text{CH}_3$ ), 11.7 ( $\text{CH}_3$ ). IR of the mixture of diastereomers (NaCl) 2974, 2928, 2880, 1686  $\text{cm}^{-1}$ ; HRMS (ESI-TOF): calculated for  $\text{C}_{10}\text{H}_{19}\text{ClNO}$  204.1150  $[\text{M}+\text{H}]^+$ , found 204.1149. Calcd. for  $\text{C}_9\text{H}_{18}\text{ClNNaO}$  226.0969  $[\text{M}+\text{Na}]^+$ , found 226.0977.

**(3*RS*,4*SR*)-1-(*tert*-Butyl)-3-chloro-3,4-dimethylpyrrolidin-2-one** (*trans*-2p, minor diastereomer)



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.65 (dd,  $J = 9.7, 6.4$  Hz, 1H, H-5), 2.93 (dd,  $J = 9.7, 4.4$  Hz, 1H, H-5), 2.55 (m, 1H, H-4), 1.40 (s, 9H, t-Bu), 1.06 (d,  $J = 7.1$  Hz, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.8 (C-2), 71.9 (C-3), 54.3 (C), 49.4 (C-5), 40.6 (C-4), 27.3 ( $\text{CH}_3$ ), 21.0 ( $\text{CH}_3$ ), 14.2 ( $\text{CH}_3$ ); IR of the mixture of diastereomers (NaCl) 2974, 2928, 2880, 1686  $\text{cm}^{-1}$ ; HRMS (ESI-TOF): calculated for  $\text{C}_{10}\text{H}_{19}\text{ClNO}$  204.1150  $[\text{M}+\text{H}]^+$ , found 204.1149. Calcd. for  $\text{C}_9\text{H}_{18}\text{ClNNaO}$  226.0969  $[\text{M}+\text{Na}]^+$ , found 226.0977.

**Ethyl (3*RS*,4*SR*)-1-(*tert*-butyl)-3-chloro-4-methyl-2-oxopyrrolidine-3-carboxylate** (*trans*-2q)

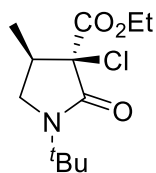


Physical state: white solid, m.p. 64-66 °C

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.29 (dq,  $J = 10.6, 7.1$  Hz, 1H), 4.24 (dq,  $J = 10.6, 7.1$  Hz, 1H), 3.54 (dd,  $J = 9.0, 7.9$  Hz, 1H, H-5), 3.16 (t,  $J = 9.2$  Hz, 1H, H-5), 2.76-2.66 (m, 1H, H-4), 1.43 (s, 9H, t-Bu), 1.31 (t,  $J = 7.1$  Hz, 3H), 1.12 (d,  $J = 7.1$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) 168.0 (C-2), 166.0 (CO), 74.7 (C-3), 62.8 ( $\text{CH}_2\text{O}$ ), 55.2 (C), 49.5 (C-5), 42.2 (C-4), 27.3 ( $\text{CH}_3$ ), 14.1 ( $\text{CH}_3$ ), 12.2 ( $\text{CH}_3$ ); IR (NaCl) 2980, 2936, 1757, 1704  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{12}\text{H}_{21}\text{ClNO}_3$  262.1204  $[\text{M}+\text{H}]^+$ , found 262.1205. Calcd. for  $\text{C}_{12}\text{H}_{20}\text{ClNNaO}_3$  284.1024  $[\text{M}+\text{Na}]^+$ , found 284.1025.

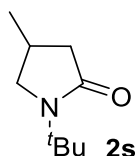
### Ethyl (3*RS*,4*RS*)-1-(*tert*-butyl)-3-chloro-4-methyl-2-oxopyrrolidine-3-carboxylate

(*cis*-2*q*, from a 1:1 mixture of diastereomers)



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.38-4.26 (m, 2H), 3.50 (dd, *J* = 8.9, 6.4 Hz, 1H, H-5), 3.06 (t, *J* = 8.9 Hz, 1H, H-5), 3.06-2.96 (m, 1H, H-4), 1.40 (s, 9H, *t*-Bu), 1.34 (t, *J* = 7.1 Hz, 3H), 1.13 (d, *J* = 6.4 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) 168.1 (C-2), 166.9 (CO), 75.1 (C-3), 63.1 (CH<sub>2</sub>O), 54.9 (C), 49.1 (C-5), 37.3 (C-4), 27.4 (CH<sub>3</sub>), 14.1 (CH<sub>3</sub>), 12.3 (CH<sub>3</sub>); IR of the mixture of diastereomers (NaCl) 2977, 2937, 2879, 1757, 1704 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>12</sub>H<sub>21</sub>ClNO<sub>3</sub> 262.1204 [M+H]<sup>+</sup>, found 262.1205. Calcd. for C<sub>12</sub>H<sub>20</sub>ClNNaO<sub>3</sub> 284.1024 [M+Na]<sup>+</sup>, found 284.1025.

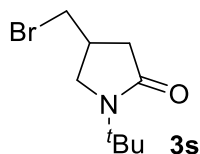
### 1-(*tert*-Butyl)-4-methylpyrrolidin-2-one (2*s*)



Physical state: colourless oil

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.58 (dd, *J* = 9.4, 7.5 Hz, 1H, H-5), 3.00 (dd, *J* = 9.4, 6.0 Hz, 1H, H-5), 2.53 (dd, *J* = 16.4, 8.3 Hz, 1H, H-3), 2.31 (m, 1H, H-4), 2.01 (dd, *J* = 16.4, 7.1 Hz, 1H, H-3), 1.39 (s, 9H, *t*-Bu), 1.09 (d, *J* = 6.7 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 175.1 (C-2), 53.9 (C), 53.3 (C-5), 41.4 (C-3), 27.7 (CH<sub>3</sub>), 26.1 (C-4), 19.6 (CH<sub>3</sub>); IR (NaCl) 2960, 2926, 2871, 1683 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>18</sub>NO 156.1383 [M+H]<sup>+</sup>, found 156.1385. Calcd. for C<sub>9</sub>H<sub>17</sub>NNaO 178.1202 [M+Na]<sup>+</sup>, found 178.1205.

### 4-(Bromomethyl)-1-(*tert*-butyl)pyrrolidin-2-one (3*s*)

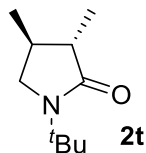


Physical state: colourless oil

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.64 (dd, *J* = 10.0, 7.7 Hz, 1H, H-5), 3.46 (dd, *J* = 10.1, 5.5 Hz, 1H), 3.37 (dd, *J* = 10.1, 7.7 Hz, 1H), 3.28 (dd, *J* = 10.0, 5.8 Hz, 1H, H-5), 2.67 (p, *J* = 6.6 Hz, 1H, H-4), 2.56 (dd, *J* = 16.6, 9.0 Hz, 1H, H-3), 2.22 (dd, *J* = 16.6, 6.8 Hz, 1H, H-3), 1.40 (s, 9H, *t*-Bu); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 173.3 (C-2), 54.3 (C), 50.4 (C-5),

38.5 (C-3), 35.9 (CH<sub>2</sub>Br), 33.3 (C-4), 27.7 (CH<sub>3</sub>); IR (NaCl) 2976, 2934, 2910, 2872, 1681 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>9</sub>H<sub>17</sub>BrNO 234.0488 [M+H]<sup>+</sup>, found 234.0489.

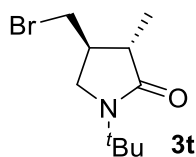
### (3*RS*,4*RS*)-1-(*tert*-Butyl)-3,4-dimethylpyrrolidin-2-one (2t)



Physical state: white foam

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.49 (dd, *J* = 9.4, 7.5 Hz, 1H, H-5), 2.87 (t, *J* = 9.1 Hz, 1H, H-5), 1.92 (dq, *J* = 9.9, 6.9 Hz, 1H, H-3), 1.86-1.74 (m, 1H), 1.38 (s, 9H, *t*-Bu), 1.13 (d, *J* = 6.9 Hz, 3H, CH<sub>3</sub>), 1.09 (d, *J* = 6.5 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 177.5 (C-2), 53.7 (C), 51.1 (C-5), 46.2 (C-3), 35.6 (C-4), 27.7 (CH<sub>3</sub>), 17.3 (CH<sub>3</sub>-4), 14.3 (CH<sub>3</sub>-3); IR (NaCl) 3012, 2963, 2922, 2868, 1681 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>10</sub>H<sub>20</sub>NO 170.1539 [M+H]<sup>+</sup>, found 170.1541. Calcd. for C<sub>10</sub>H<sub>19</sub>NNaO 192.1359 [M+Na]<sup>+</sup>, found 192.1367.

### (3*RS*,4*RS*)-4-(Bromomethyl)-1-(*tert*-butyl)-3-methylpyrrolidin-2-one (3t)

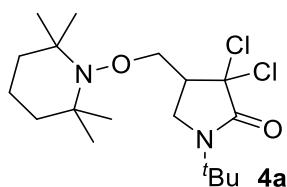


Physical state: white foam

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.60 (dd, *J* = 9.8, 7.6 Hz, 1H, H-5), 3.57 (dd, *J* = 10.2, 3.9 Hz, 1H), 3.35 (dd, *J* = 10.2, 8.2 Hz, 1H), 3.12 (dd, *J* = 9.8, 8.0 Hz, 1H, H-5), 2.26-2.14 (m, 2H, H-3 and H-4), 1.40 (s, 9H, *t*-Bu), 1.19 (d, *J* = 6.8 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 175.9 (C-2), 54.1 (C), 48.7 (C-5), 43.4 (CH), 42.4 (CH), 34.7 (CH<sub>2</sub>Br), 27.7 (CH<sub>3</sub>), 14.9 (CH<sub>3</sub>); IR (NaCl): 3012, 2963, 2922, 2868, 1681 cm<sup>-1</sup>; HRMS (ESI-TOF) calcd. for C<sub>10</sub>H<sub>19</sub>BrNO 248.0645 [M+H]<sup>+</sup>, found 248.0644. Calcd. for C<sub>10</sub>H<sub>18</sub>BrNNaO 270.0464 [M+Na]<sup>+</sup>, found 270.0462.

## 5. Radical trapping experiment

To a solution **1a** (46 mg, 0.18 mmol), DIPEA (0.16 mL, 0.92 mmol) and *fac*-Ir(ppy)<sub>3</sub> (1.2 mg, 0.0018 mmol, 1%) in THF/acetone (3 ml, 1:1) was added TEMPO (80 mg, 0.51 mmol) and the mixture was stirred at rt under blue LED irradiation overnight. The mixture was then concentrated and purified by chromatography using a mixture of Hexane/EtOAc (1:0 to 1:1) as eluent to provide **4a** (30 mg, 40%) and **1a** (16 mg, 35%).

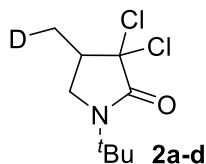


Physical state: colourless oil

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.21 (dd,  $J = 9.3, 5.1$  Hz, 1H), 3.92 (dd,  $J = 9.3, 8.2$  Hz, 1H), 3.58 (dd,  $J = 9.8, 6.8$  Hz, 1H, H-5), 3.25 (dd,  $J = 9.8, 7.8$  Hz, 1H, H-5), 2.92 (qd,  $J = 7.7, 5.1$  Hz, 1H, H-4), 1.50-1.41 (m, 5H), 1.43 (s, 9H, *t*-Bu), 1.38-1.29 (m, 2H), 1.22 (s, 3H,  $\text{CH}_3$ ), 1.16 (s, 3H,  $\text{CH}_3$ ), 1.11 (s, 3H,  $\text{CH}_3$ ), 1.10 (s, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.2 (C-2), 85.4 (C-3), 73.9 ( $\text{CH}_2\text{O}$ ), 59.9 (C), 55.3 (C), 48.4 (C-4), 45.4 (C-5), 39.4 ( $\text{CH}_2$ ), 33.2 ( $\text{CH}_3$ ), 33.0 ( $\text{CH}_3$ ), 27.2 ( $\text{CH}_3$ ), 20.1 ( $\text{CH}_3$ ), 17.0 ( $\text{CH}_2$ ); IR (NaCl) 3054, 2977, 2934, 1720  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_{18}\text{H}_{33}\text{Cl}_2\text{N}_2\text{O}_2$  379.1914  $[\text{M}+\text{H}]^+$ , found 379.1907. Calcd. for  $\text{C}_{18}\text{H}_{32}\text{Cl}_2\text{N}_2\text{NaO}_2$  401.1733  $[\text{M}+\text{Na}]^+$ , found 401.1729.

## 6. Amine free photocatalyzed reaction of 1a in acetone- $\text{d}_6$

A mixture of haloamide **1a** (52.2 mg, 0.2 mmol) and *fac*-Ir(ppy) $_3$  (1.3 mg, 0.002 mmol, 1%) in acetone- $\text{d}_6$  (4 ml) was stirred at rt under blue LED irradiation for 62 h. The mixture was then concentrated and purified by chromatography using a mixture of Hexane/EtOAc (1:0 to 1:1) as eluent to provide deuterated lactam **2a-d** (5 mg, 10%)<sup>8</sup> and **3a** (14 mg, 26%).



Physical state: white foam

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.46 (dd,  $J = 9.8, 6.8$  Hz, 1H, H-5), 3.01 (dd,  $J = 9.8, 8.6$  Hz, 1H, H-5), 2.66 (quintm,  $J = 6.8$  Hz, 1H, H-4), 1.42 (s, 9H, *t*-Bu), 1.28 (dt,  $J = 6.4, 2$  Hz,  $\text{CH}_2\text{D}$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.7 (C-2), 88.6 (C-3), 55.2 (C), 48.3 (C-5), 44.6 (C-4), 27.2 ( $\text{CH}_3$ ), 11.5 (t,  $J = 19.4$  Hz,  $\text{CH}_2\text{D}$ ); IR (NaCl) 2957, 2924, 2850, 1708  $\text{cm}^{-1}$ ; HRMS (ESI-TOF) calcd. for  $\text{C}_9\text{H}_{15}\text{DCl}_2\text{NO}$  225.0666  $[\text{M}+\text{H}]^+$ , found 225.0670. Calcd. for  $\text{C}_9\text{H}_{14}\text{DCl}_2\text{NNaO}$  247.0486  $[\text{M}+\text{Na}]^+$ , found 247.0491.

<sup>8</sup> The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **2a-d** is displayed in page S74 of this SI.

## 7. X-ray Crystallography data of 2o

CCDC 2096537 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif) or by emailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033

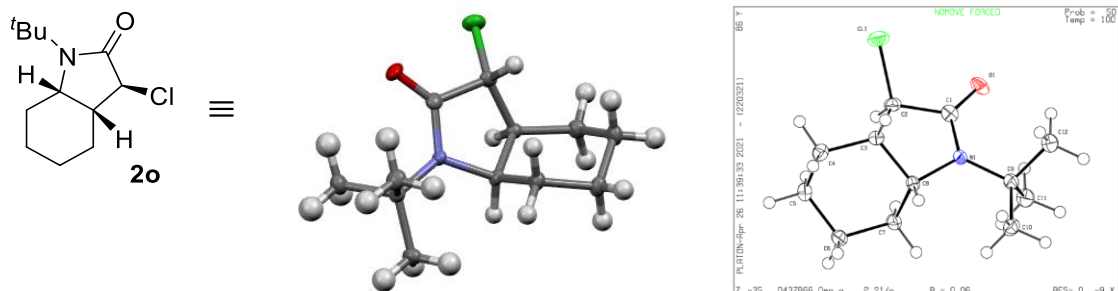


Table 1. Crystal data and structure refinement for D43ZB66\_0ma\_a.

Identification code	D43ZB66_0ma_a	
Empirical formula	C <sub>12</sub> H <sub>20</sub> Cl N O	
Formula weight	229.74	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 <sub>1</sub> /n	
Unit cell dimensions	a = 8.4389(15) Å	α = 90°.
	b = 16.182(3) Å	β = 91.763(7)°.
	c = 8.8628(17) Å	γ = 90°.
Volume	1209.7(4) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.261 Mg/m <sup>3</sup>	
Absorption coefficient	0.291 mm <sup>-1</sup>	
F(000)	496	
Crystal size	0.100 x 0.060 x 0.040 mm <sup>3</sup>	
Theta range for data collection	2.517 to 26.420°.	
Index ranges	-8 ≤ h ≤ 10, -20 ≤ k ≤ 20, -11 ≤ l ≤ 11	
Reflections collected	18662	
Independent reflections	2480 [R(int) = 0.1559]	
Completeness to theta = 25.242°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.4710	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	

Data / restraints / parameters	2480 / 0 / 140
Goodness-of-fit on $F^2$	1.034
Final R indices [ $I > 2\sigma(I)$ ]	R1 = 0.0582, wR2 = 0.0942
R indices (all data)	R1 = 0.1043, wR2 = 0.1131
Extinction coefficient	0.0052(9)
Largest diff. peak and hole	0.297 and -0.354 e.Å <sup>-3</sup>

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for D43ZB66\_0ma\_a.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

	x	y	z	$U(\text{eq})$
Cl(1)	2430(1)	6051(1)	4600(1)	28(1)
O(1)	1406(2)	4348(1)	3309(2)	23(1)
N(1)	2823(3)	3736(1)	5291(3)	16(1)
C(1)	2065(3)	4364(2)	4569(3)	18(1)
C(2)	2124(4)	5112(2)	5618(3)	18(1)
C(3)	3460(3)	4909(2)	6748(3)	16(1)
C(4)	3478(4)	5364(2)	8252(3)	21(1)
C(5)	2213(4)	5069(2)	9318(3)	23(1)
C(6)	2288(4)	4138(2)	9513(3)	21(1)
C(7)	2002(4)	3710(2)	7991(3)	19(1)
C(8)	3275(3)	3960(2)	6871(3)	16(1)
C(9)	2868(4)	2875(2)	4691(3)	18(1)
C(10)	3955(4)	2340(2)	5693(3)	22(1)
C(11)	1203(4)	2512(2)	4608(3)	23(1)
C(12)	3569(4)	2899(2)	3112(3)	25(1)



Table 3. Bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ] for D43ZB66\_0ma\_a.

---

Cl(1)-C(2)	1.790(3)
O(1)-C(1)	1.233(3)
N(1)-C(1)	1.352(4)
N(1)-C(8)	1.485(3)
N(1)-C(9)	1.492(4)
C(1)-C(2)	1.525(4)
C(2)-C(3)	1.520(4)
C(2)-H(2)	1.0000
C(3)-C(4)	1.523(4)
C(3)-C(8)	1.547(4)
C(3)-H(3)	1.0000
C(4)-C(5)	1.523(4)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.517(4)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.528(4)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.540(4)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-H(8)	1.0000
C(9)-C(11)	1.523(4)
C(9)-C(10)	1.526(4)
C(9)-C(12)	1.536(4)
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800

C(1)-N(1)-C(8)	111.5(2)
C(1)-N(1)-C(9)	123.4(2)
C(8)-N(1)-C(9)	123.8(2)
O(1)-C(1)-N(1)	127.3(3)
O(1)-C(1)-C(2)	125.1(3)
N(1)-C(1)-C(2)	107.6(2)
C(3)-C(2)-C(1)	103.9(2)
C(3)-C(2)-Cl(1)	113.6(2)
C(1)-C(2)-Cl(1)	111.6(2)
C(3)-C(2)-H(2)	109.2
C(1)-C(2)-H(2)	109.2
Cl(1)-C(2)-H(2)	109.2
C(2)-C(3)-C(4)	117.3(2)
C(2)-C(3)-C(8)	100.8(2)
C(4)-C(3)-C(8)	114.6(2)
C(2)-C(3)-H(3)	107.9
C(4)-C(3)-H(3)	107.9
C(8)-C(3)-H(3)	107.9
C(3)-C(4)-C(5)	113.7(2)
C(3)-C(4)-H(4A)	108.8
C(5)-C(4)-H(4A)	108.8
C(3)-C(4)-H(4B)	108.8
C(5)-C(4)-H(4B)	108.8
H(4A)-C(4)-H(4B)	107.7
C(6)-C(5)-C(4)	110.8(3)
C(6)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5A)	109.5
C(6)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	108.1
C(5)-C(6)-C(7)	110.1(2)
C(5)-C(6)-H(6A)	109.6
C(7)-C(6)-H(6A)	109.6
C(5)-C(6)-H(6B)	109.6
C(7)-C(6)-H(6B)	109.6
H(6A)-C(6)-H(6B)	108.1
C(6)-C(7)-C(8)	110.8(2)

C(6)-C(7)-H(7A)	109.5
C(8)-C(7)-H(7A)	109.5
C(6)-C(7)-H(7B)	109.5
C(8)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	108.1
N(1)-C(8)-C(7)	112.3(2)
N(1)-C(8)-C(3)	101.5(2)
C(7)-C(8)-C(3)	112.3(2)
N(1)-C(8)-H(8)	110.1
C(7)-C(8)-H(8)	110.1
C(3)-C(8)-H(8)	110.1
N(1)-C(9)-C(11)	110.1(2)
N(1)-C(9)-C(10)	110.1(2)
C(11)-C(9)-C(10)	110.3(3)
N(1)-C(9)-C(12)	108.4(2)
C(11)-C(9)-C(12)	110.3(2)
C(10)-C(9)-C(12)	107.6(3)
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(9)-C(11)-H(11A)	109.5
C(9)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(9)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(9)-C(12)-H(12A)	109.5
C(9)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(9)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5

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Symmetry transformations used to generate equivalent atoms:

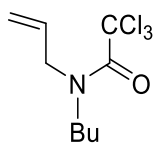
Table 4. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for D43ZB66\_0ma\_a. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^2 U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Cl(1)	34(1)	21(1)	29(1)	9(1)	-1(1)	4(1)
O(1)	22(1)	32(1)	14(1)	2(1)	-4(1)	6(1)
N(1)	19(1)	16(1)	12(1)	-1(1)	-3(1)	1(1)
C(1)	13(2)	24(2)	19(2)	0(1)	2(1)	2(1)
C(2)	19(2)	16(2)	18(2)	4(1)	4(1)	1(1)
C(3)	15(2)	16(2)	16(2)	2(1)	0(1)	0(1)
C(4)	24(2)	17(2)	22(2)	-1(1)	0(1)	-2(1)
C(5)	30(2)	21(2)	19(2)	-5(1)	2(1)	-2(1)
C(6)	25(2)	21(2)	16(2)	0(1)	1(1)	-5(1)
C(7)	23(2)	19(2)	15(2)	-1(1)	-1(1)	-3(1)
C(8)	18(2)	17(2)	12(1)	-1(1)	-4(1)	-2(1)
C(9)	20(2)	19(2)	15(2)	-1(1)	-2(1)	1(1)
C(10)	26(2)	16(2)	24(2)	-1(1)	-3(1)	3(1)
C(11)	23(2)	24(2)	22(2)	0(1)	-3(1)	-5(1)
C(12)	29(2)	23(2)	22(2)	-2(1)	5(1)	2(1)

Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^{-3}$ ) for D43ZB66\_0ma\_a.

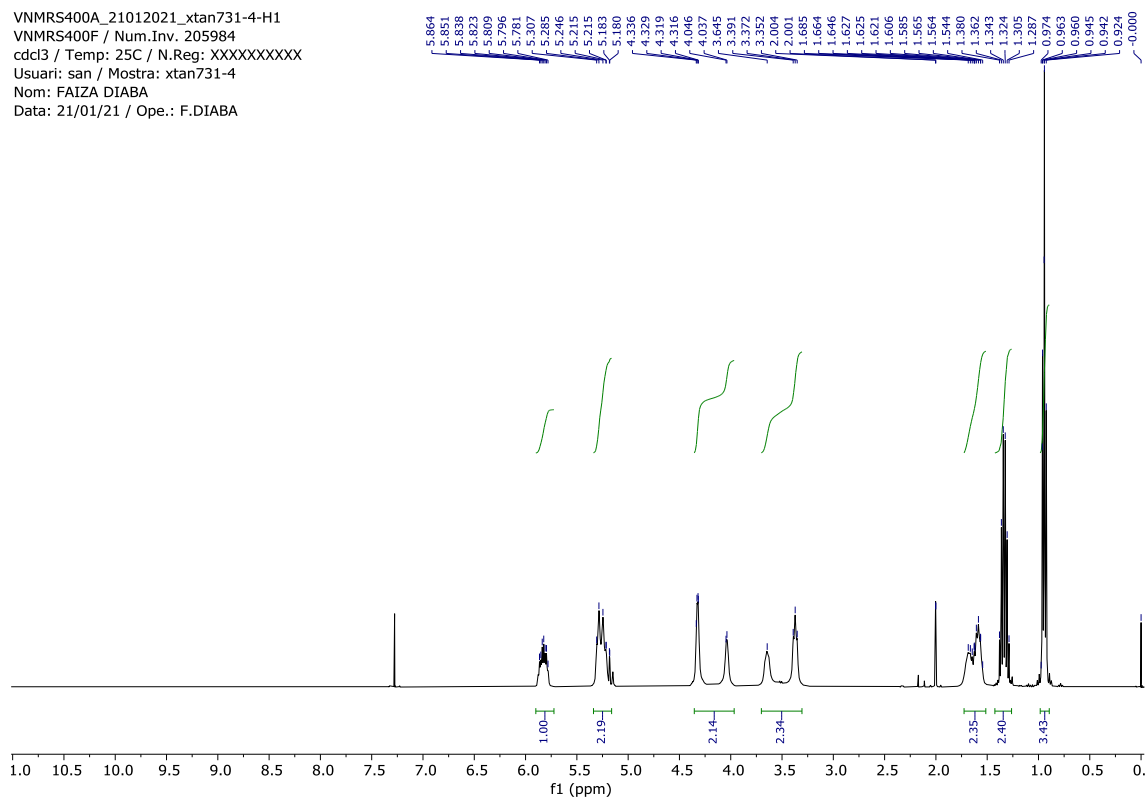
	x	y	z	U(eq)
H(2)	1104	5150	6160	21
H(3)	4486	5024	6254	19
H(4A)	3322	5961	8057	25
H(4B)	4533	5293	8754	25
H(5A)	2374	5339	10312	28
H(5B)	1152	5228	8907	28
H(6A)	1475	3959	10225	25
H(6B)	3342	3979	9941	25
H(7A)	941	3864	7573	23
H(7B)	2023	3104	8135	23
H(8)	4315	3702	7168	19
H(10A)	4000	1780	5274	33
H(10B)	3540	2317	6713	33
H(10C)	5022	2579	5735	33
H(11A)	510	2870	3985	35
H(11B)	790	2474	5627	35
H(11C)	1237	1960	4157	35
H(12A)	3636	2335	2714	37
H(12B)	4632	3143	3178	37
H(12C)	2886	3233	2439	37

## 8. <sup>1</sup>H and <sup>13</sup>C NMR spectra for new compounds

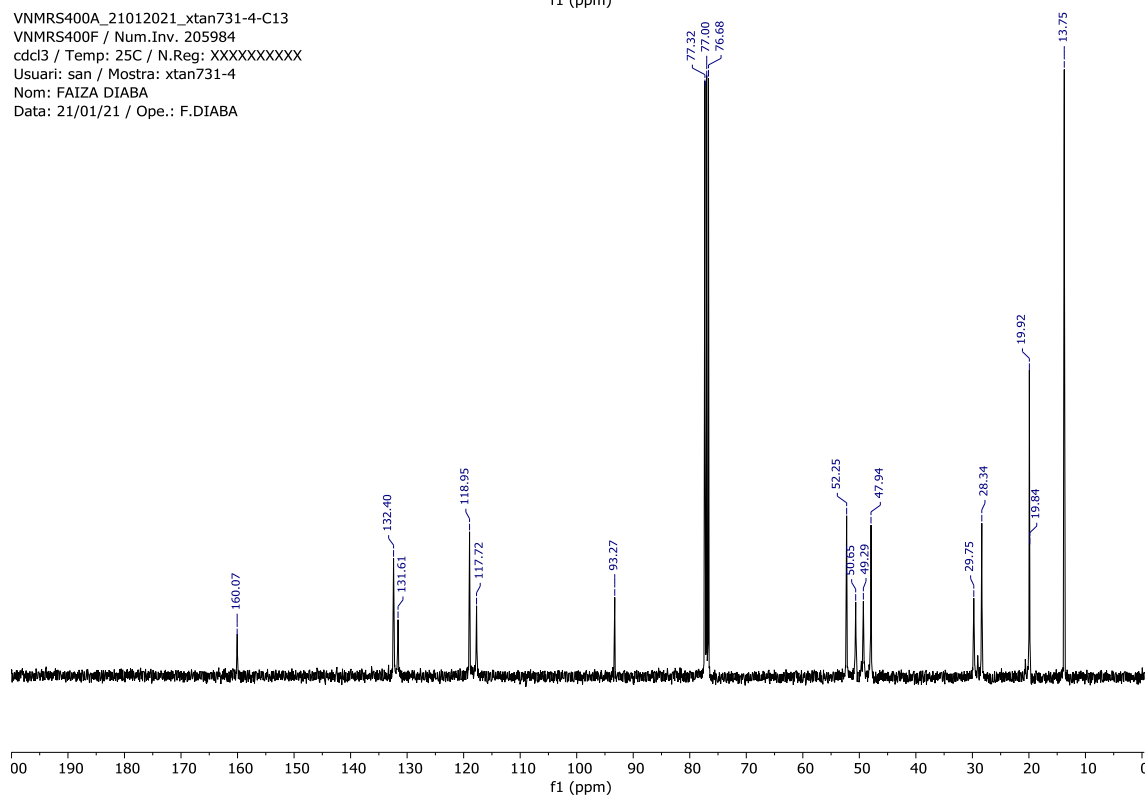


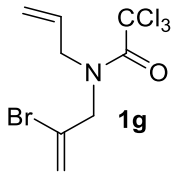
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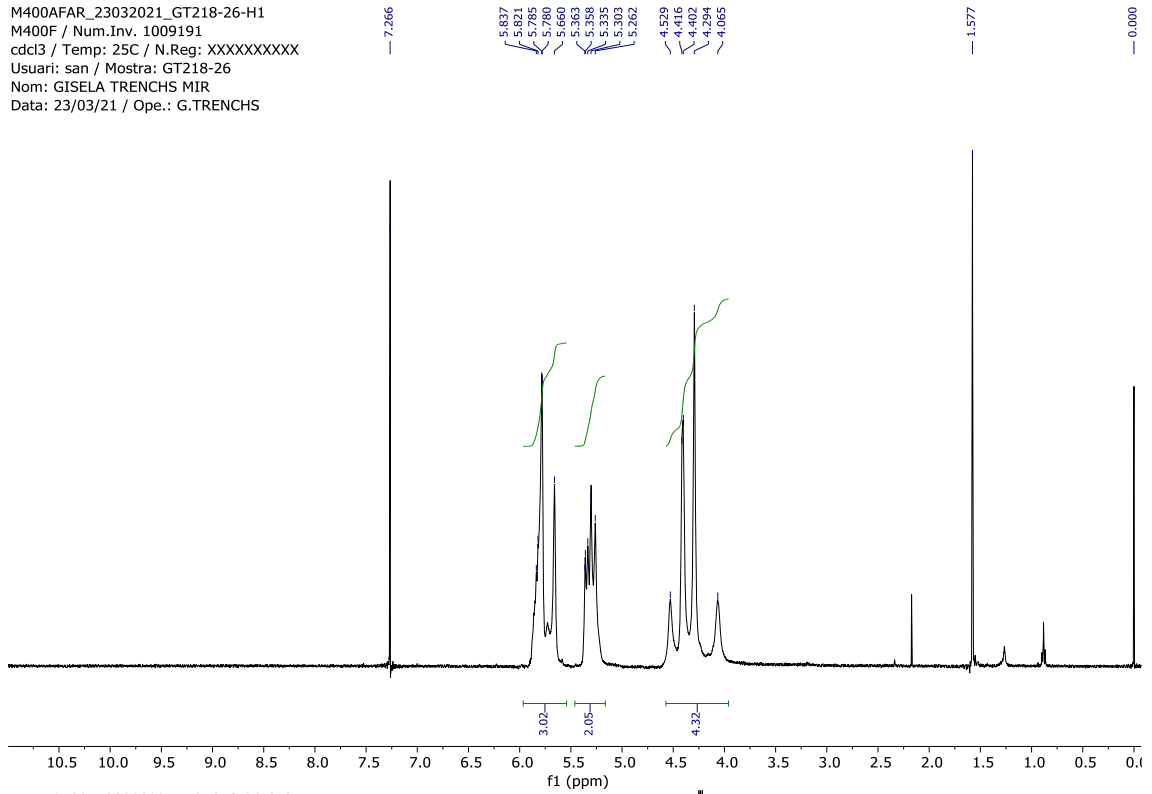


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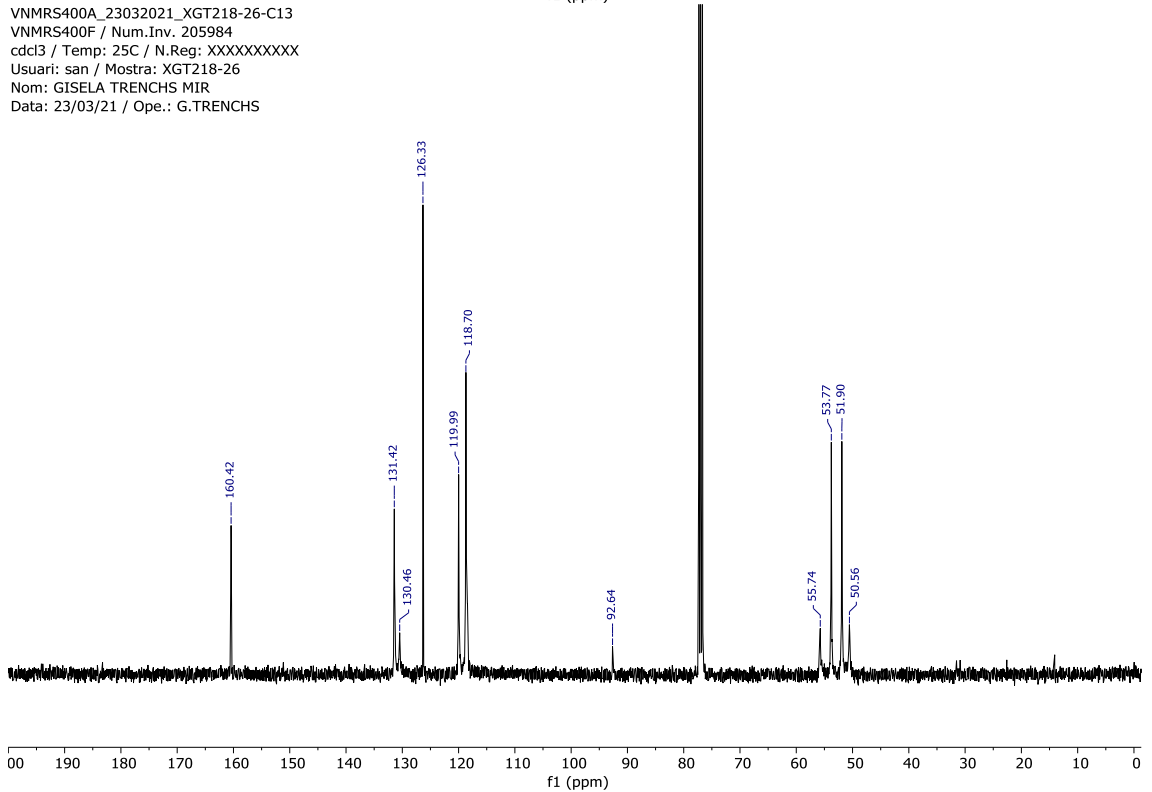


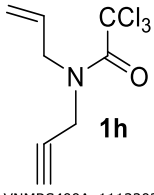


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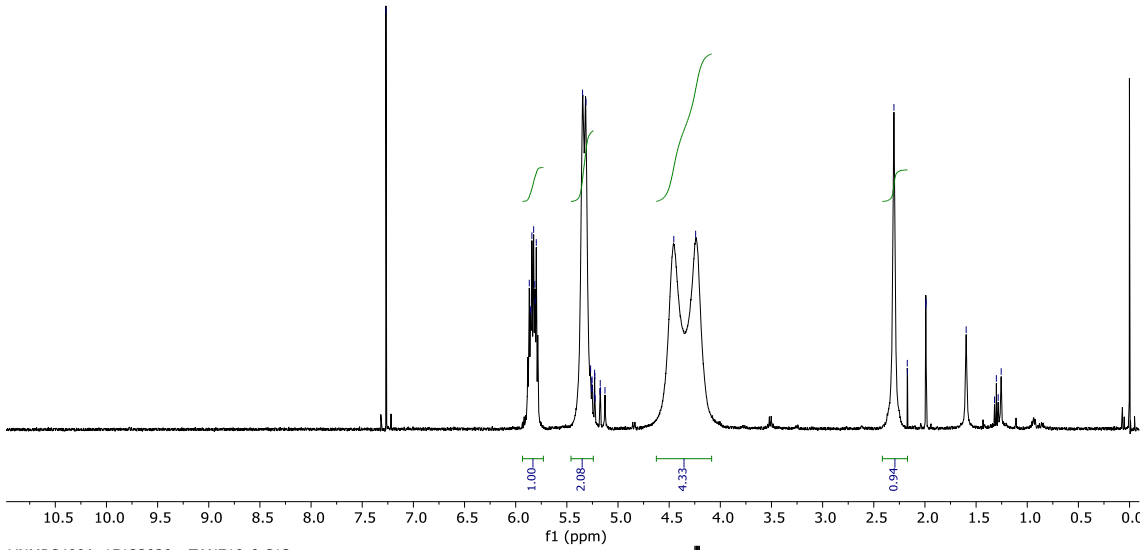
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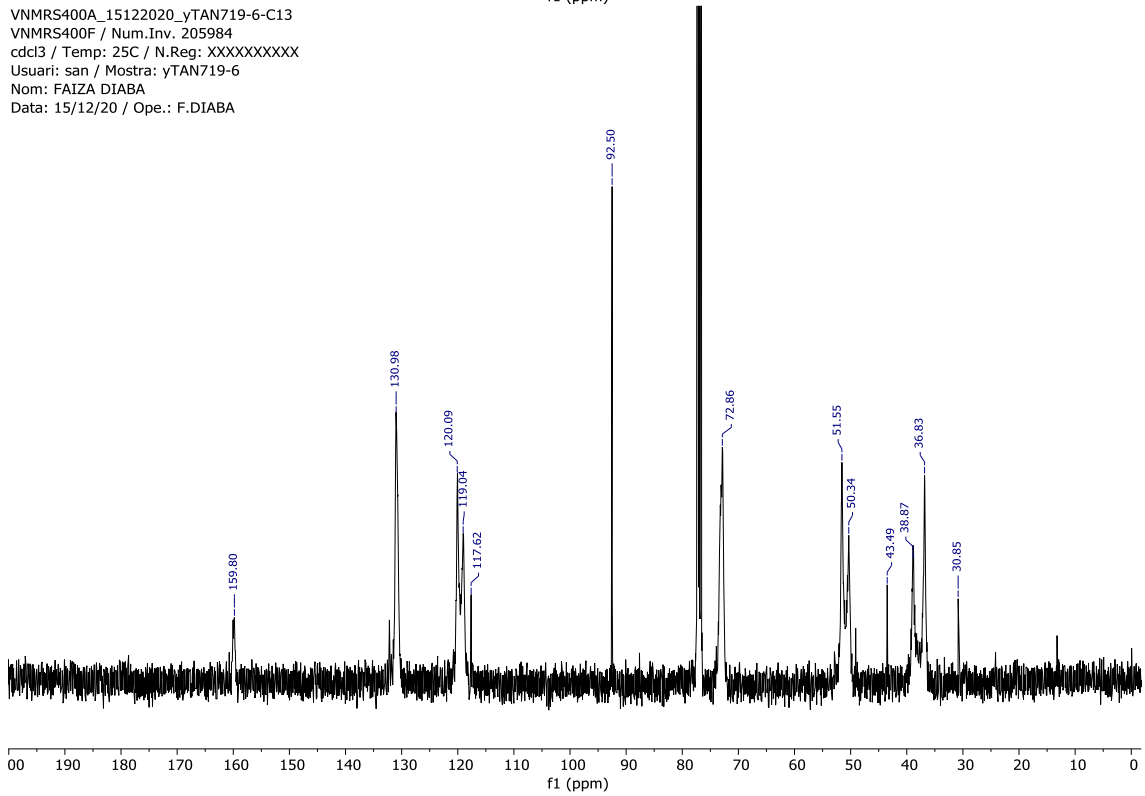


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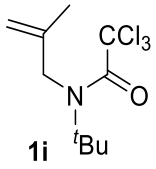
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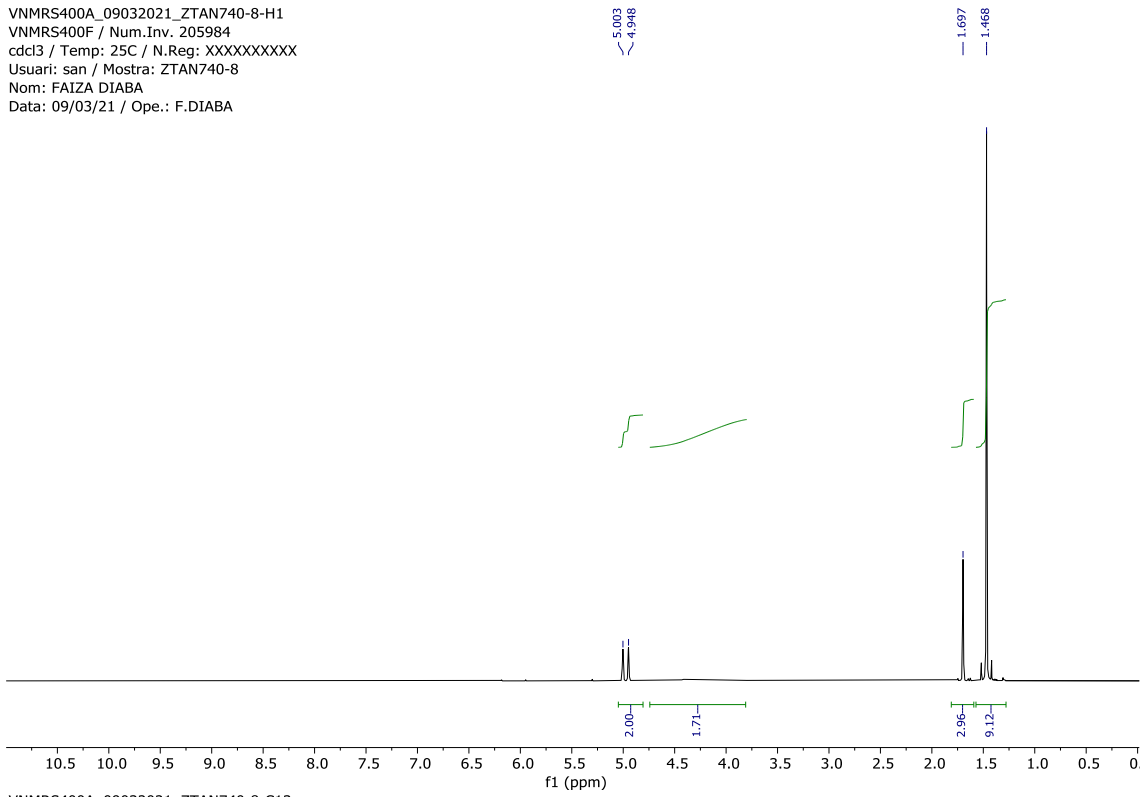
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 VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: yTAN719-6  
 Nom: FAIZA DIABA  
 Data: 15/12/20 / Ope.: F.DIABA



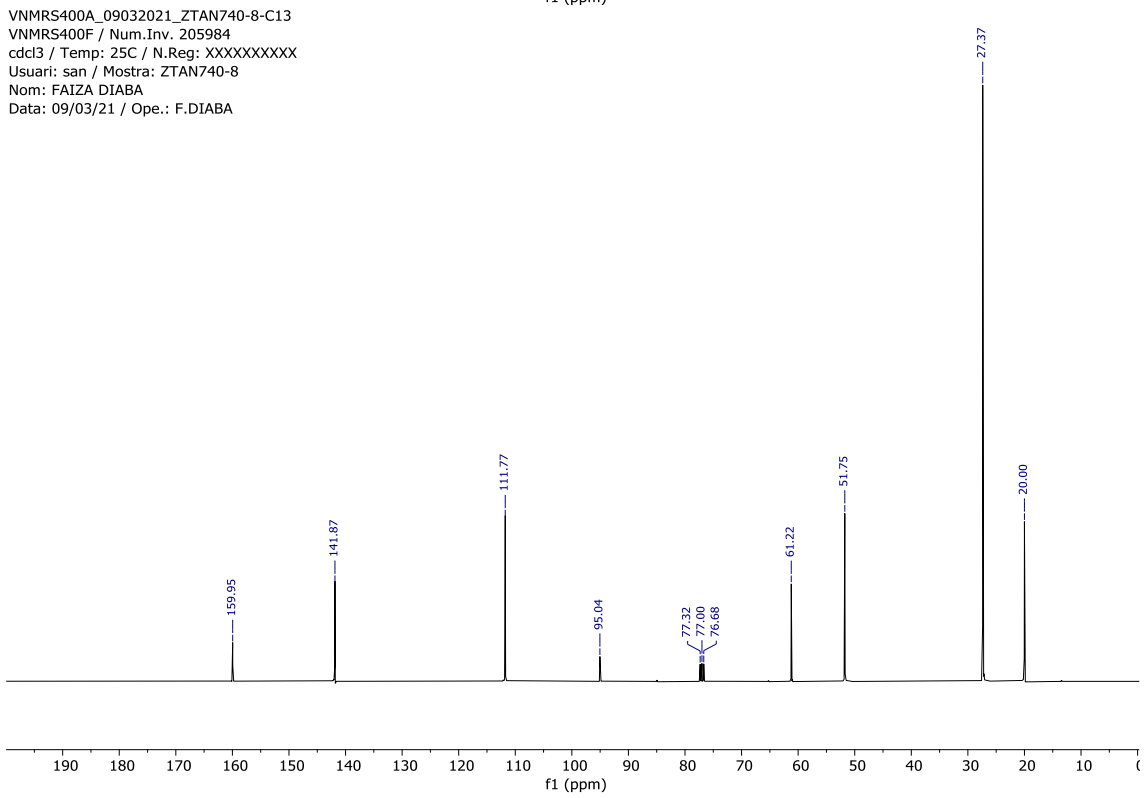


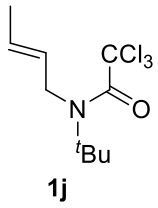


VNMRS400A\_09032021\_ZTAN740-8-H1  
 VNMRS400F / Num.Inv. 205984  
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 Usuari: san / Mostra: ZTAN740-8  
 Nom: FAIZA DIABA  
 Data: 09/03/21 / Ope.: F.DIABA

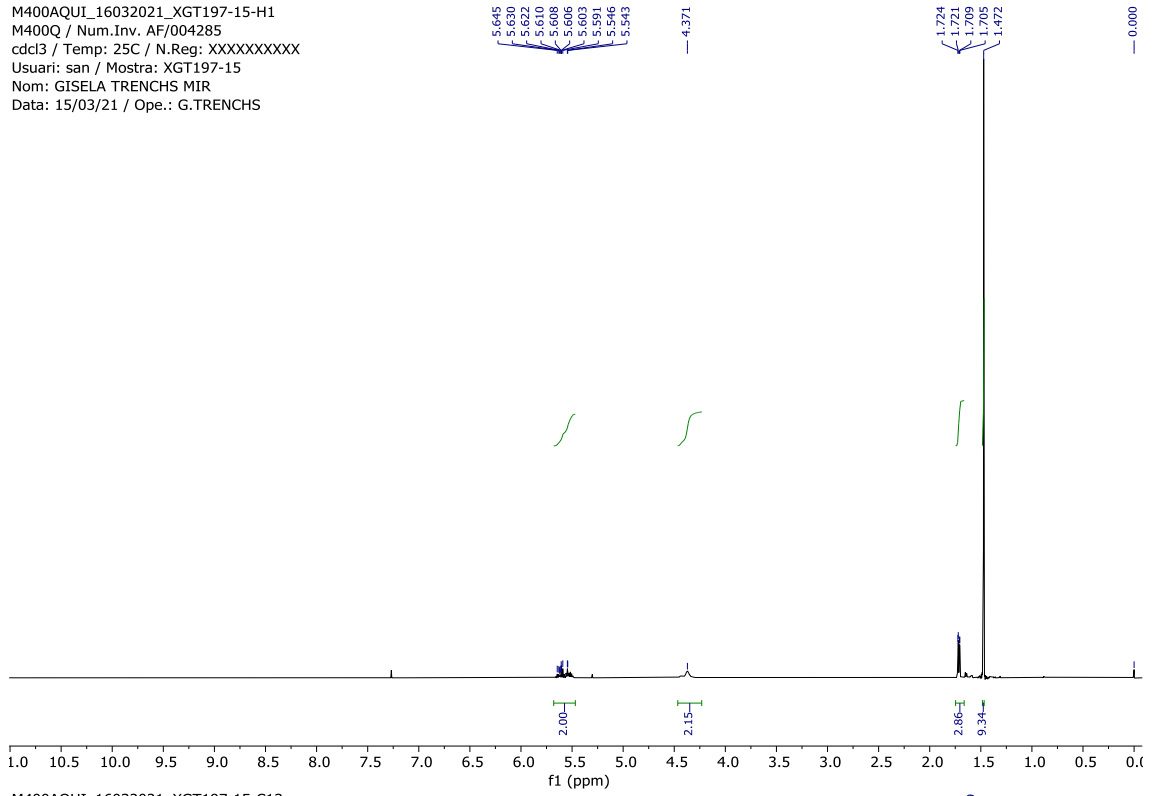


VNMRS400A\_09032021\_ZTAN740-8-C13  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: ZTAN740-8  
 Nom: FAIZA DIABA  
 Data: 09/03/21 / Ope.: F.DIABA

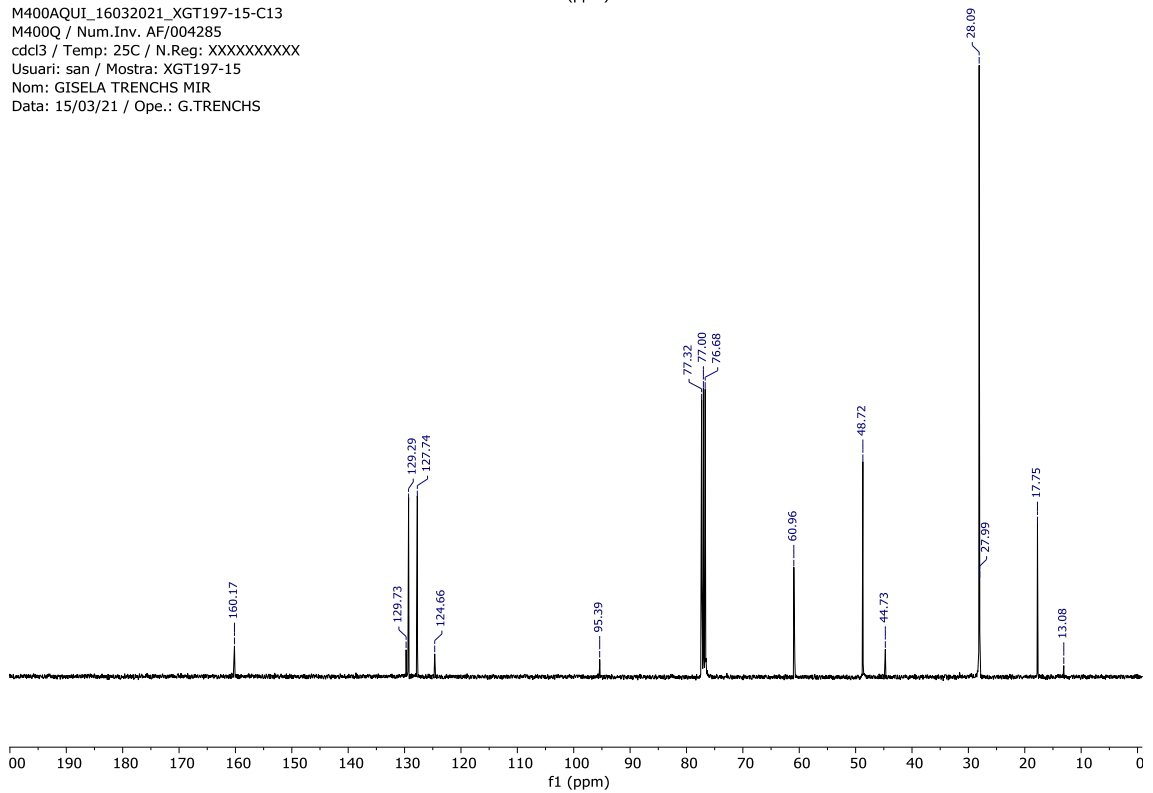


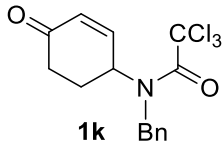


M400AQUI\_16032021\_XGT197-15-H1  
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Usuari: san / Mostra: XGT197-15  
Nom: GISELA TRENCHS MIR  
Data: 15/03/21 / Ope.: G.TRENCHS

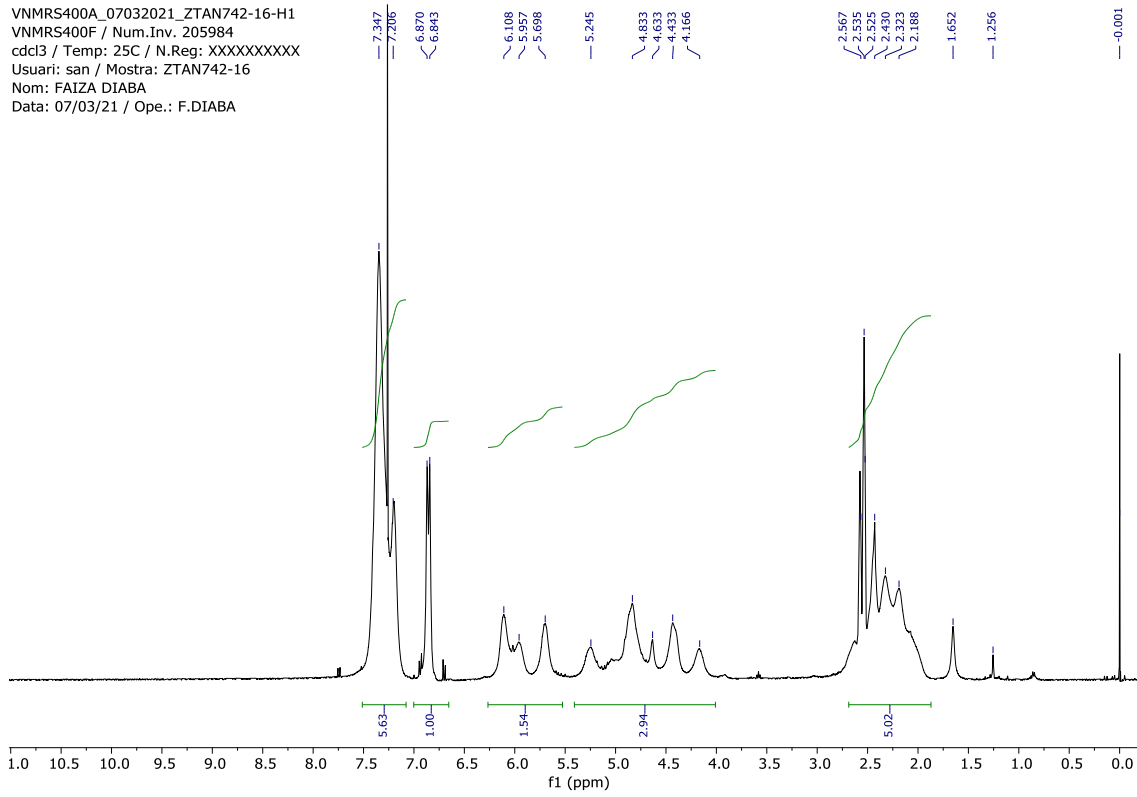


M400AQUI\_16032021\_XGT197-15-C13  
M400Q / Num.Inv. AF/004285  
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
Usuari: san / Mostra: XGT197-15  
Nom: GISELA TRENCHS MIR  
Data: 15/03/21 / Ope.: G.TRENCHS

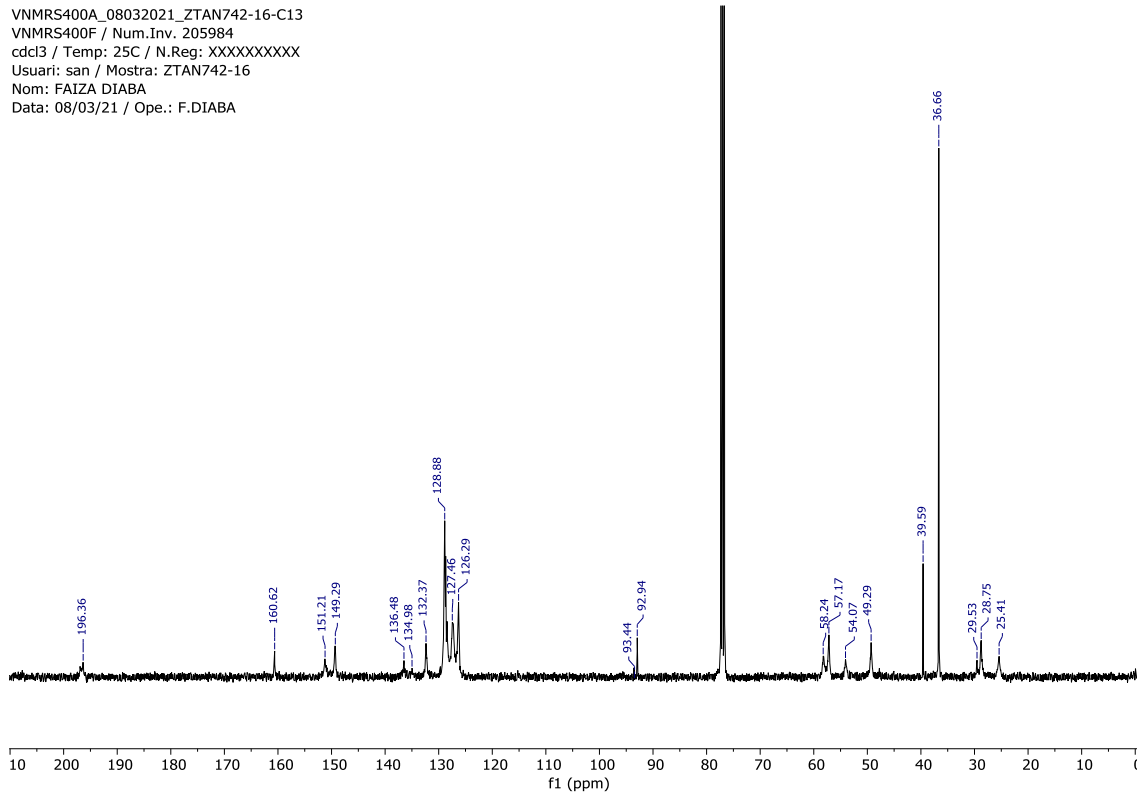


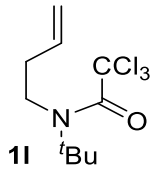


VNMRS400A\_07032021\_ZTAN742-16-H1  
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 Nom: FAIZA DIABA  
 Data: 07/03/21 / Ope.: F.DIABA

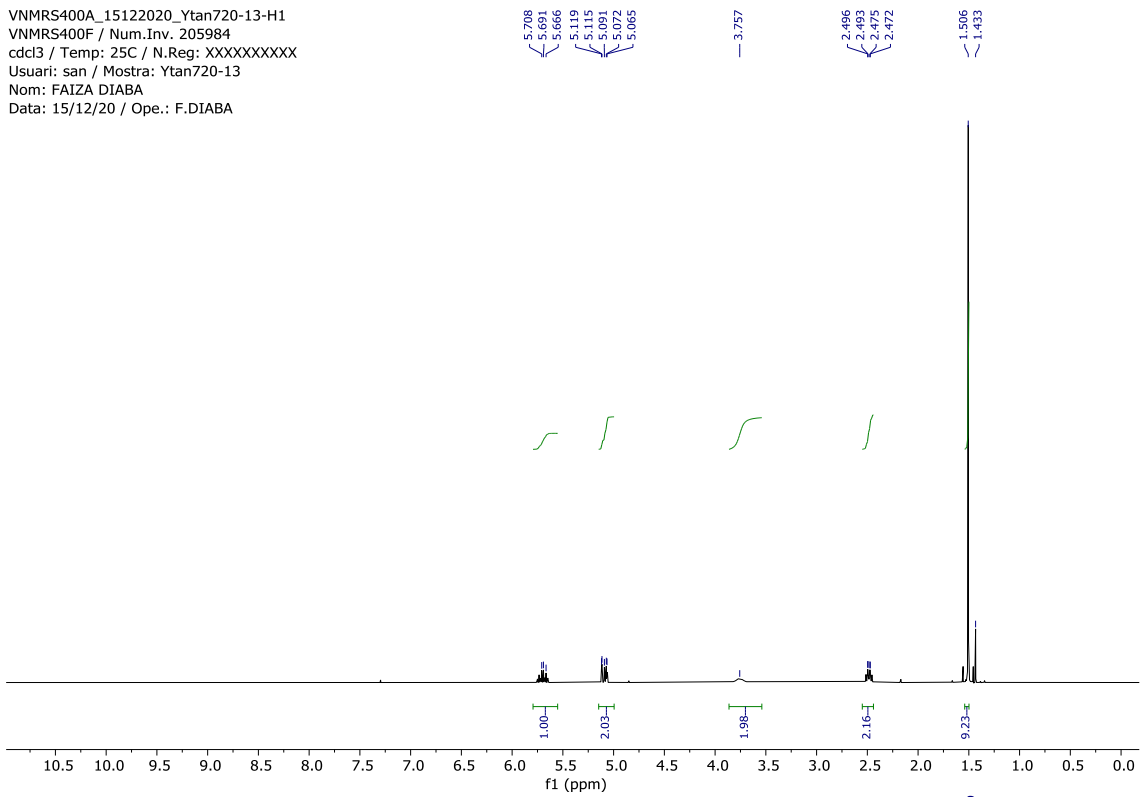


VNMRS400A\_08032021\_ZTAN742-16-C13  
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 Usuari: san / Mostra: ZTAN742-16  
 Nom: FAIZA DIABA  
 Data: 08/03/21 / Ope.: F.DIABA

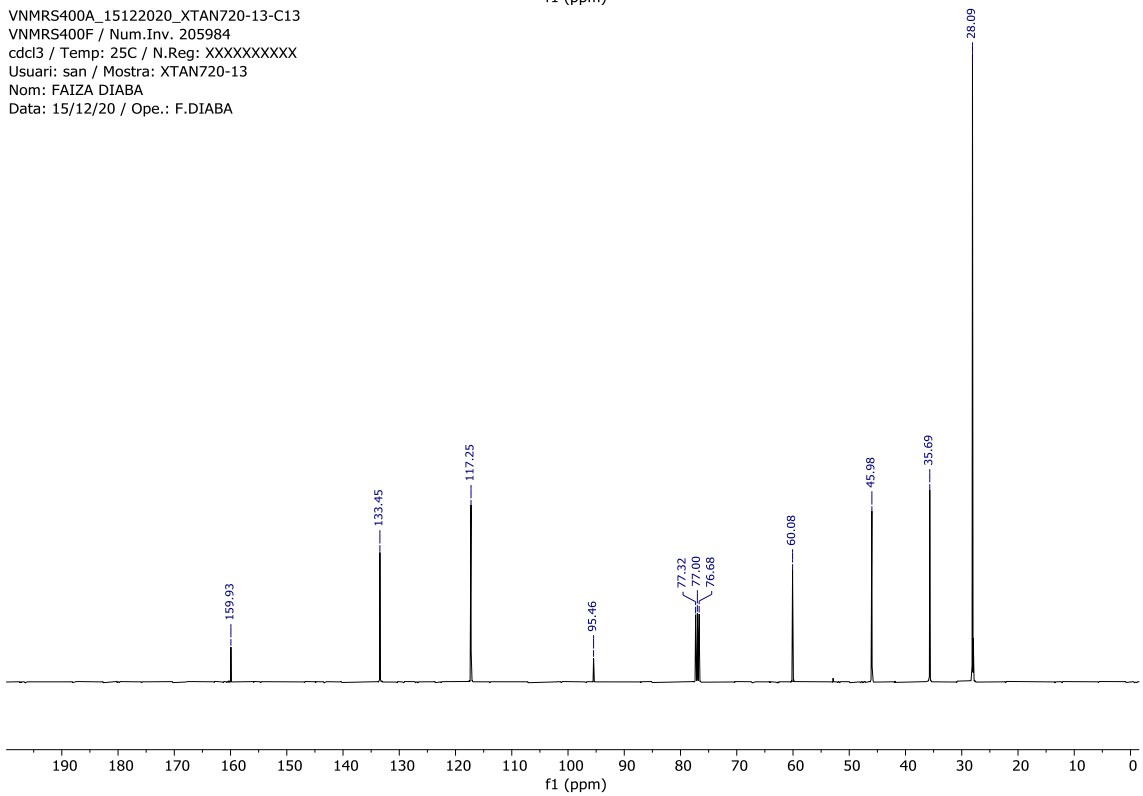


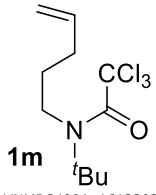


VNMRS400A\_15122020\_Ytan720-13-H1  
 VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: Ytan720-13  
 Nom: FAIZA DIABA  
 Data: 15/12/20 / Ope.: F.DIABA

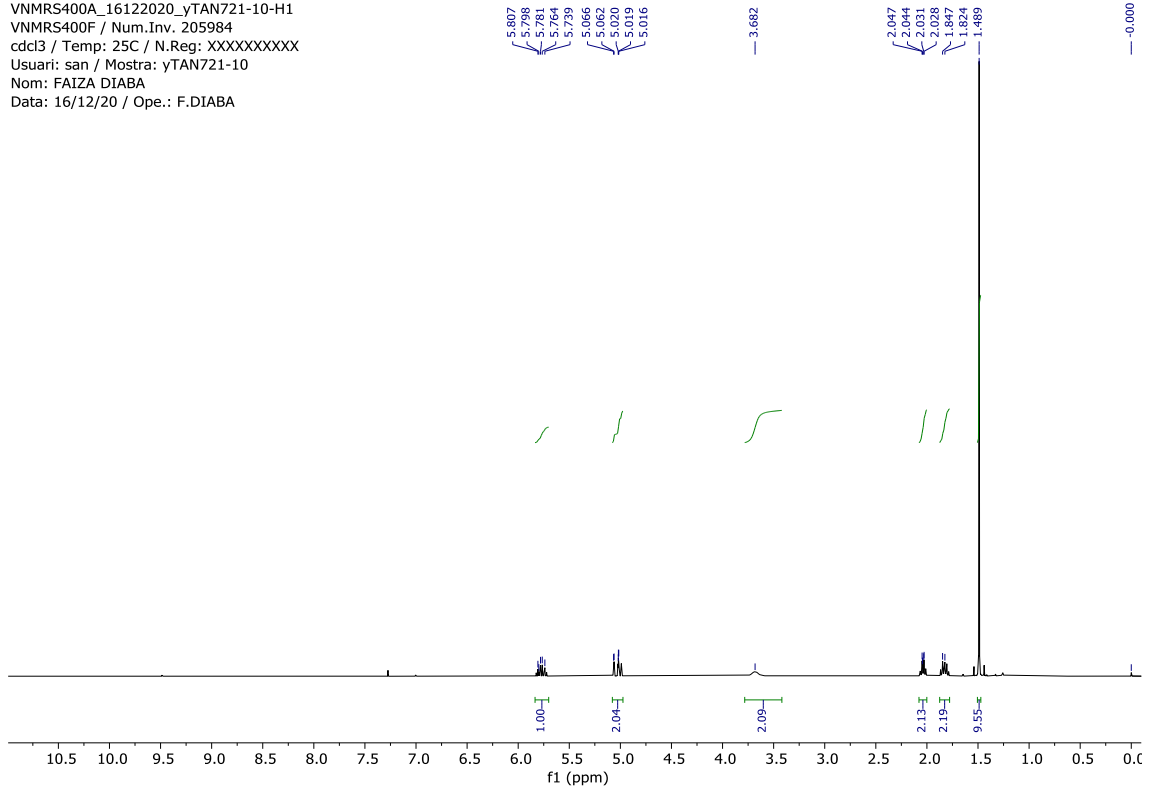


VNMRS400A\_15122020\_XTAN720-13-C13  
 VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Nom: FAIZA DIABA  
 Data: 15/12/20 / Ope.: F.DIABA

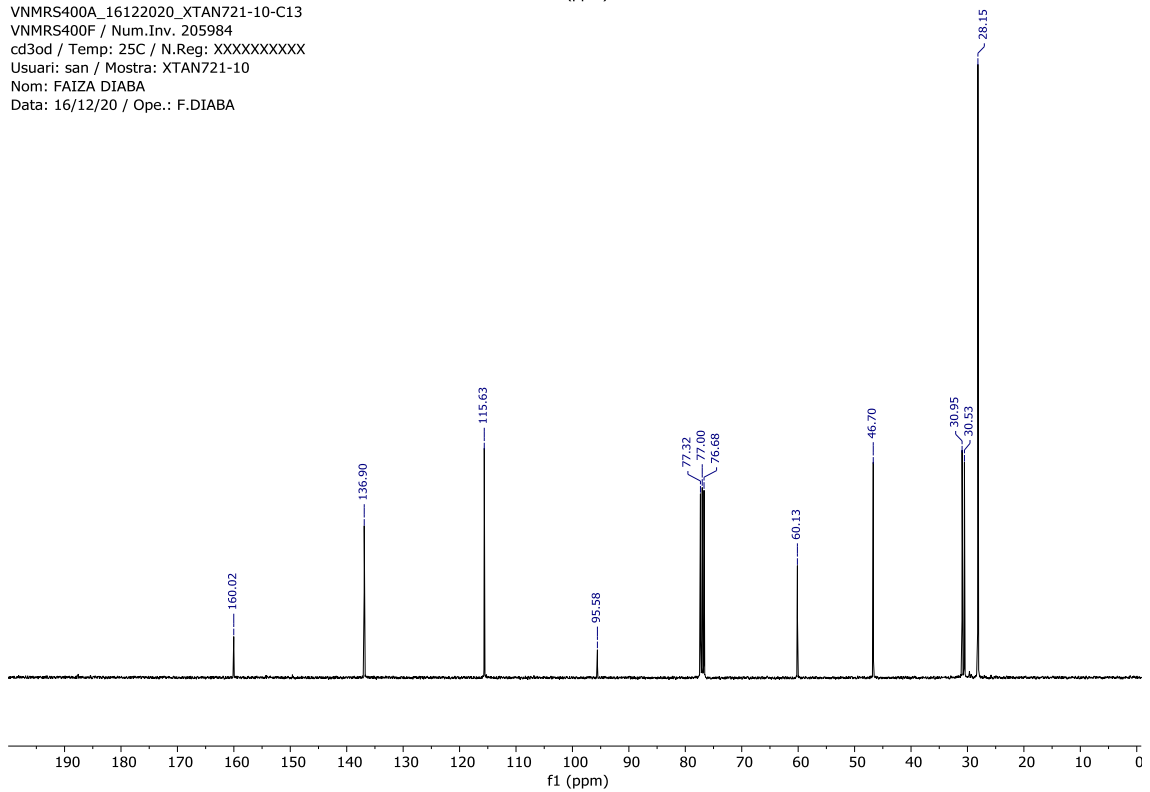


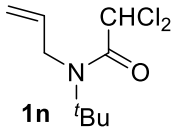


VNMRS400A\_16122020\_yTAN721-10-H1  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: yTAN721-10  
 Nom: FAIZA DIABA  
 Data: 16/12/20 / Ope.: F.DIABA

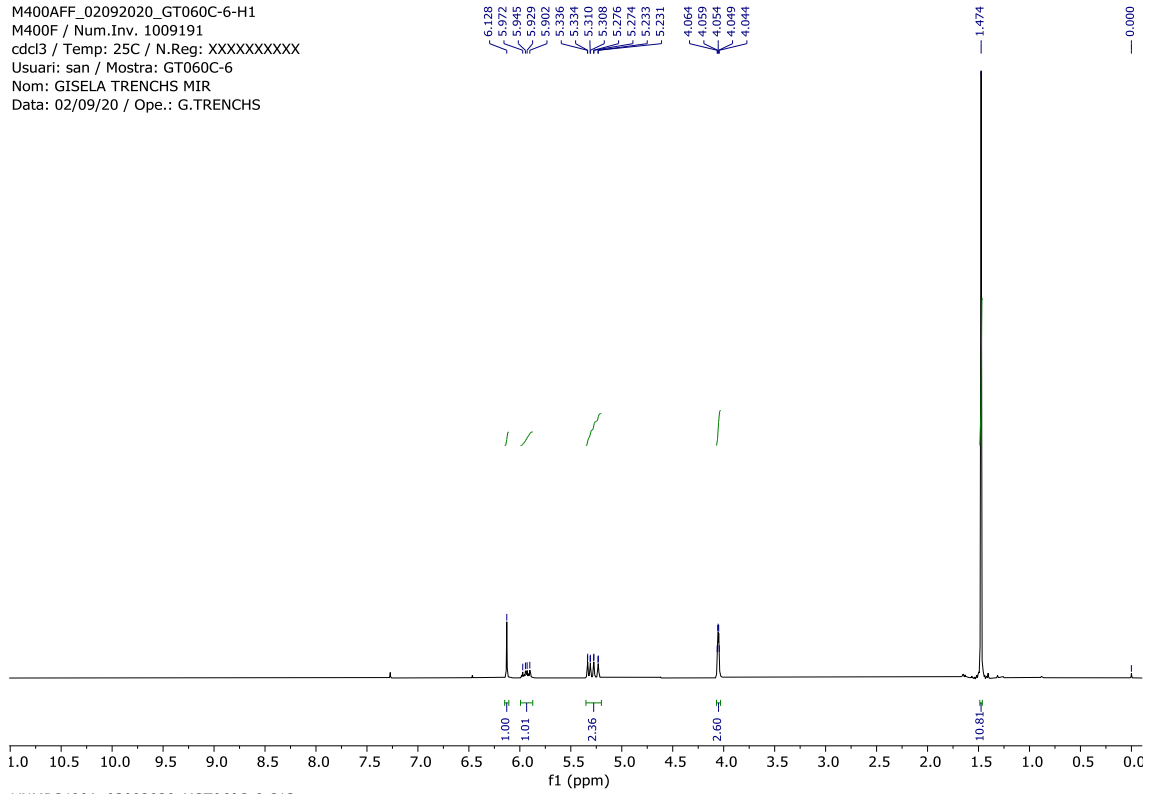


VNMRS400A\_16122020\_XTAN721-10-C13  
 VNMRS400F / Num.Inv. 205984  
 cd3od / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: XTAN721-10  
 Nom: FAIZA DIABA  
 Data: 16/12/20 / Ope.: F.DIABA

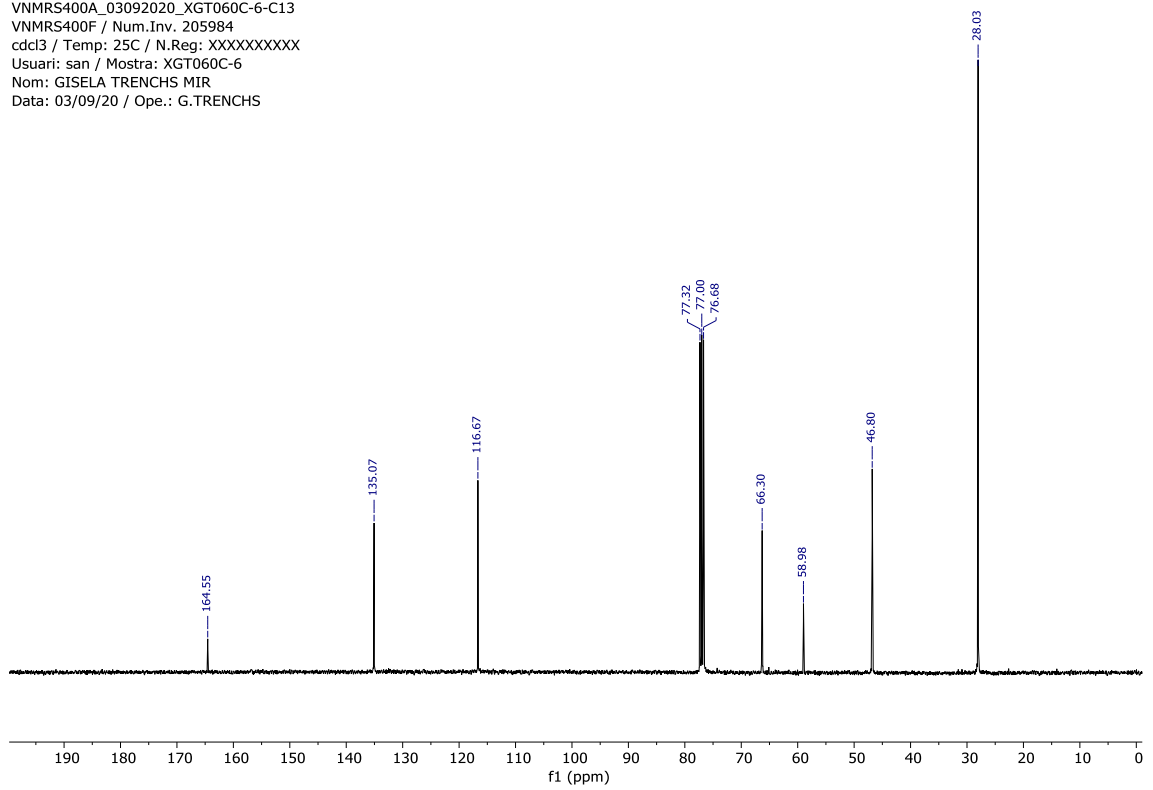


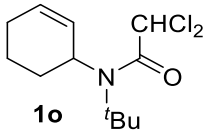


M400AFF\_02092020\_GT060C-6-H1  
 M400F / Num.Inv. 1009191  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: GT060C-6  
 Nom: GISELA TRENCHS MIR  
 Data: 02/09/20 / Ope.: G.TRENCHS

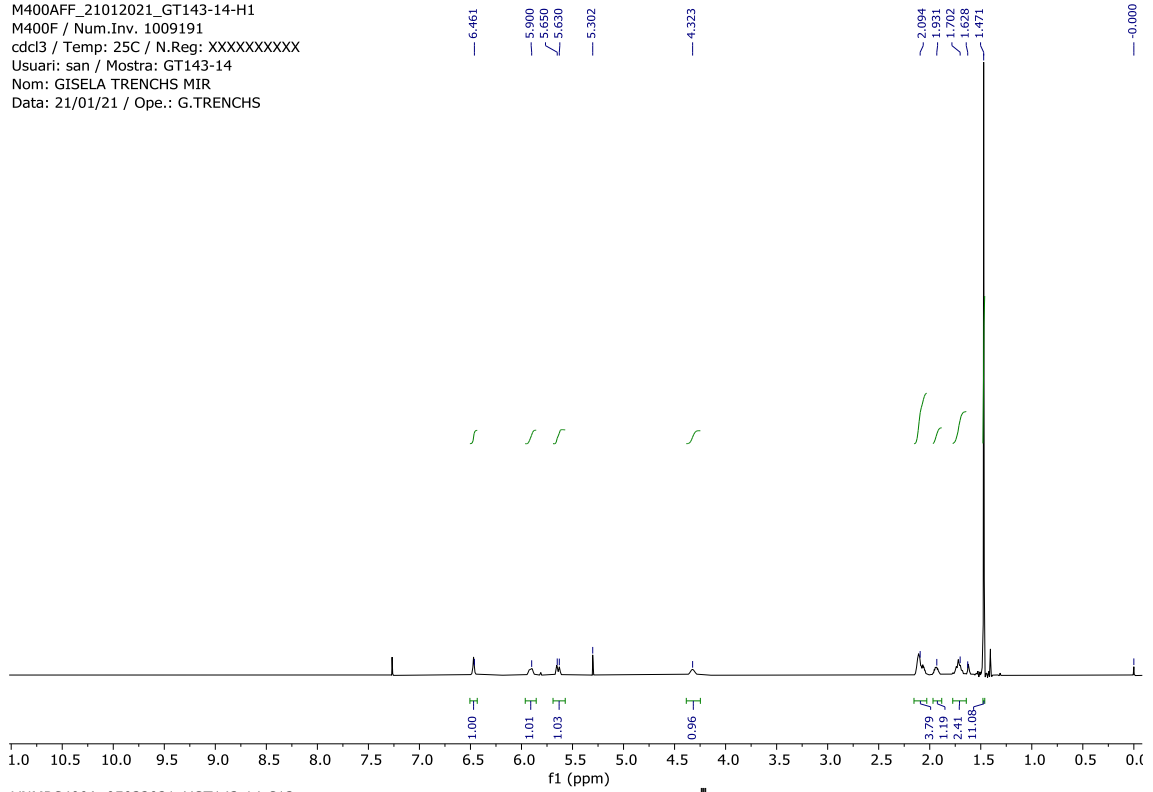


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 Usuari: san / Mostra: XGT060C-6  
 Nom: GISELA TRENCHS MIR  
 Data: 03/09/20 / Ope.: G.TRENCHS

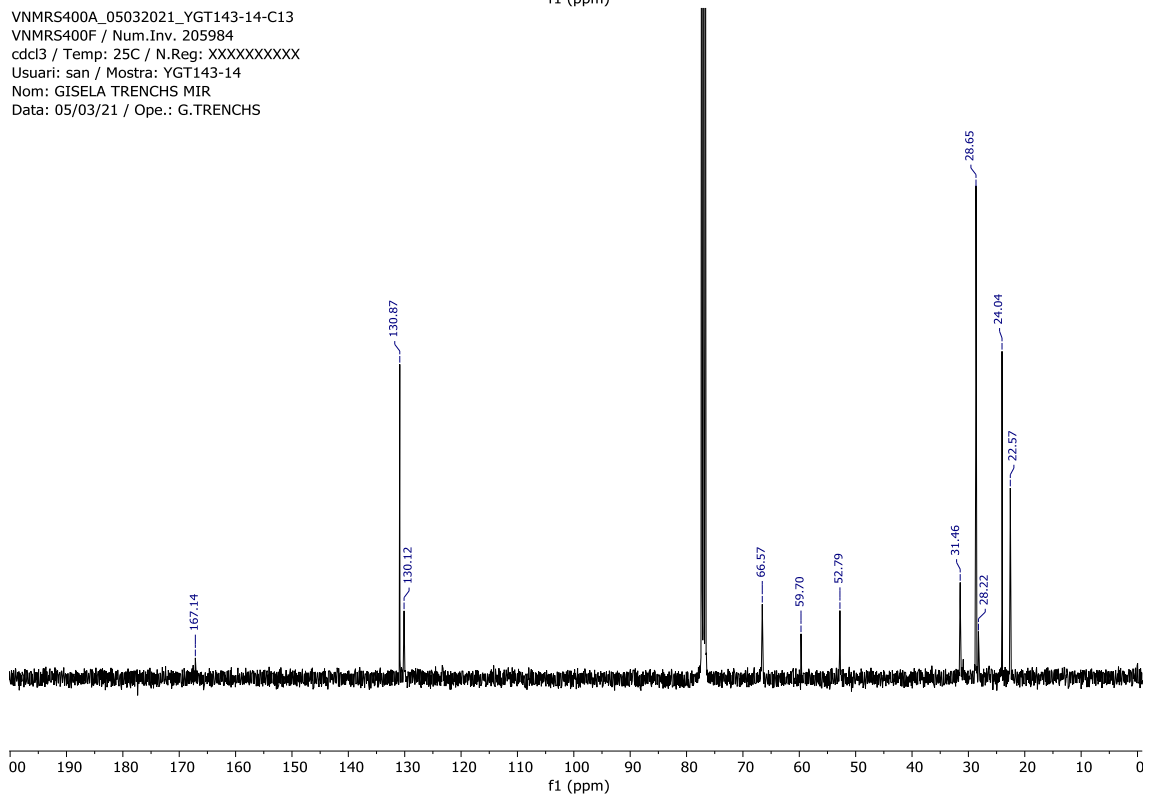


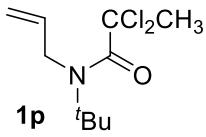


M400AFF\_21012021\_GT143-14-H1  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: GT143-14  
 Nom: GISELA TRENCHS MIR  
 Data: 21/01/21 / Ope.: G.TRENCHS

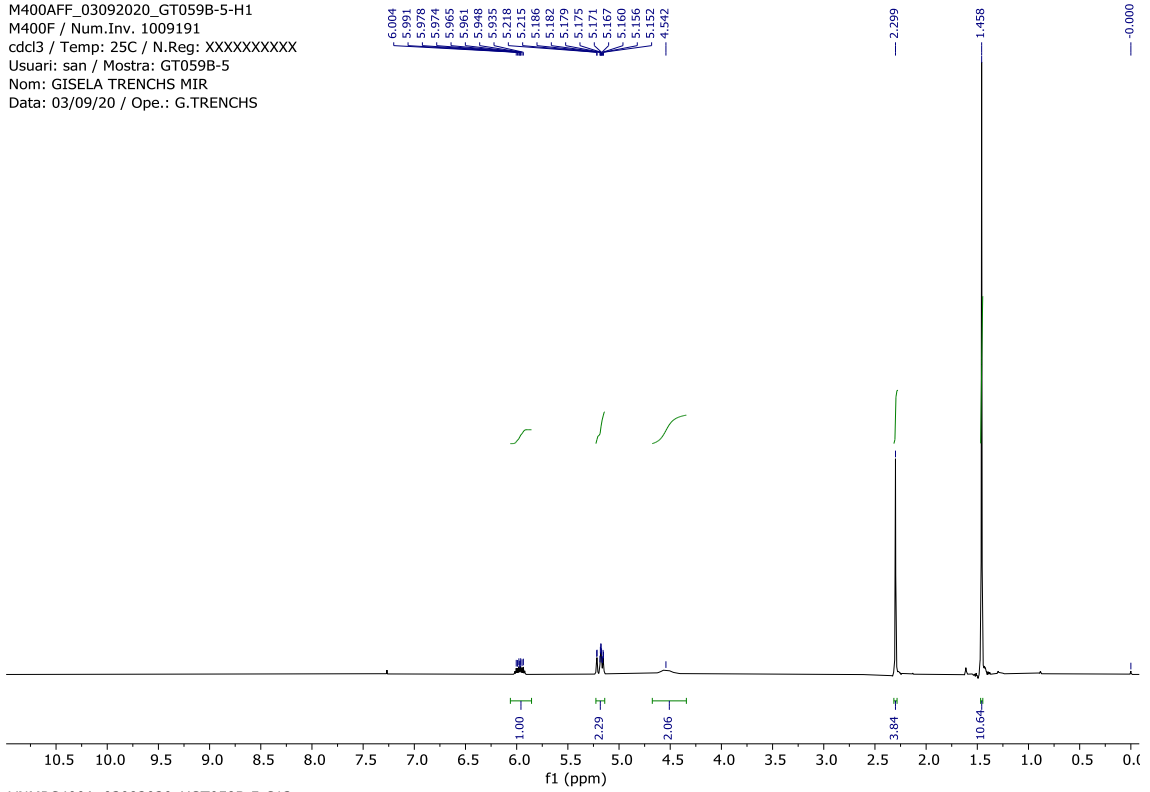


VNMRS400A\_05032021\_YGT143-14-C13  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: YGT143-14  
 Nom: GISELA TRENCHS MIR  
 Data: 05/03/21 / Ope.: G.TRENCHS

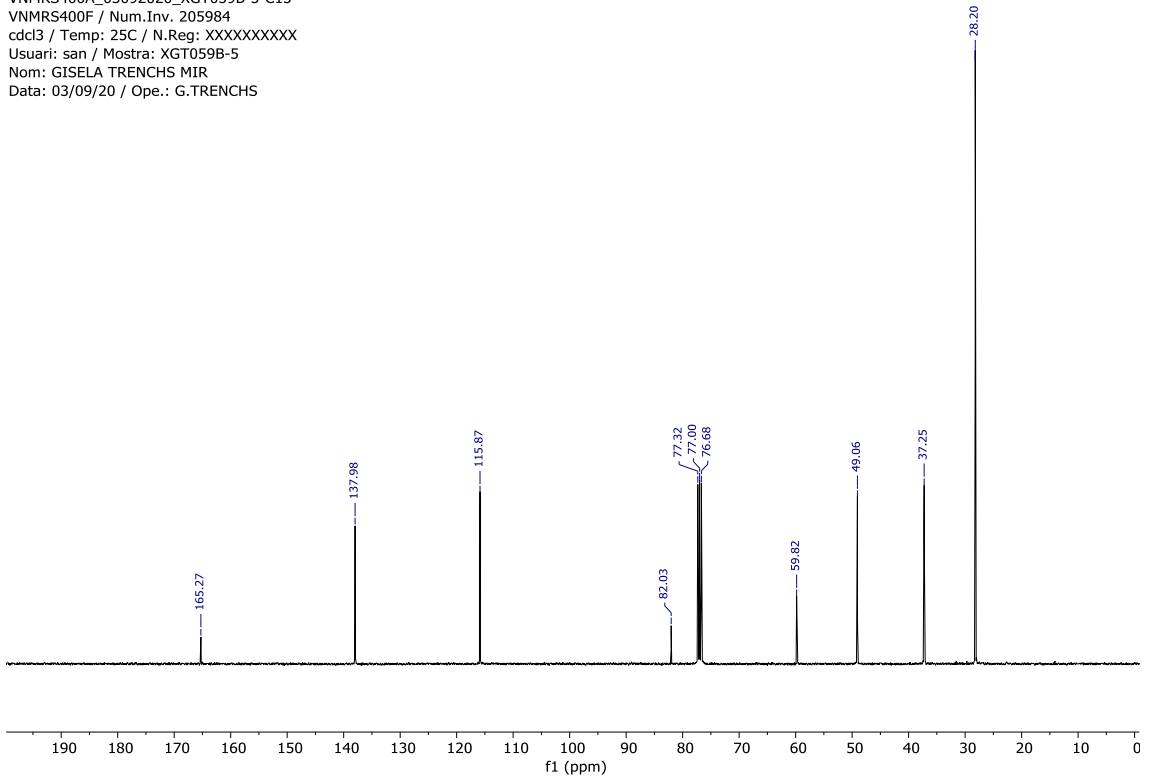




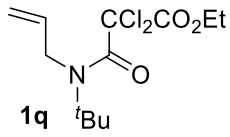
M400AFF\_03092020\_GT059B-5-H1  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: GT059B-5  
 Nom: GISELA TRENCHS MIR  
 Data: 03/09/20 / Ope.: G.TRENCHS



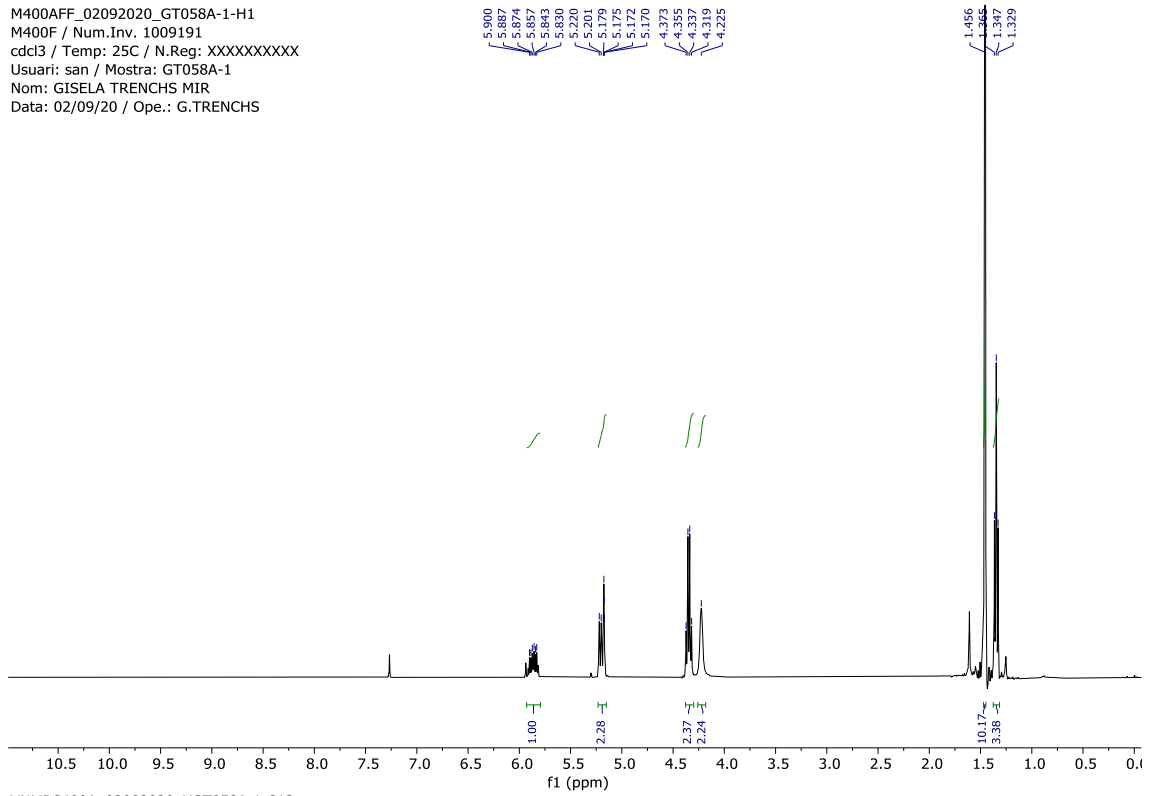
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Nom: GISELA TRENCHS MIR  
 Data: 03/09/20 / Ope.: G.TRENCHS



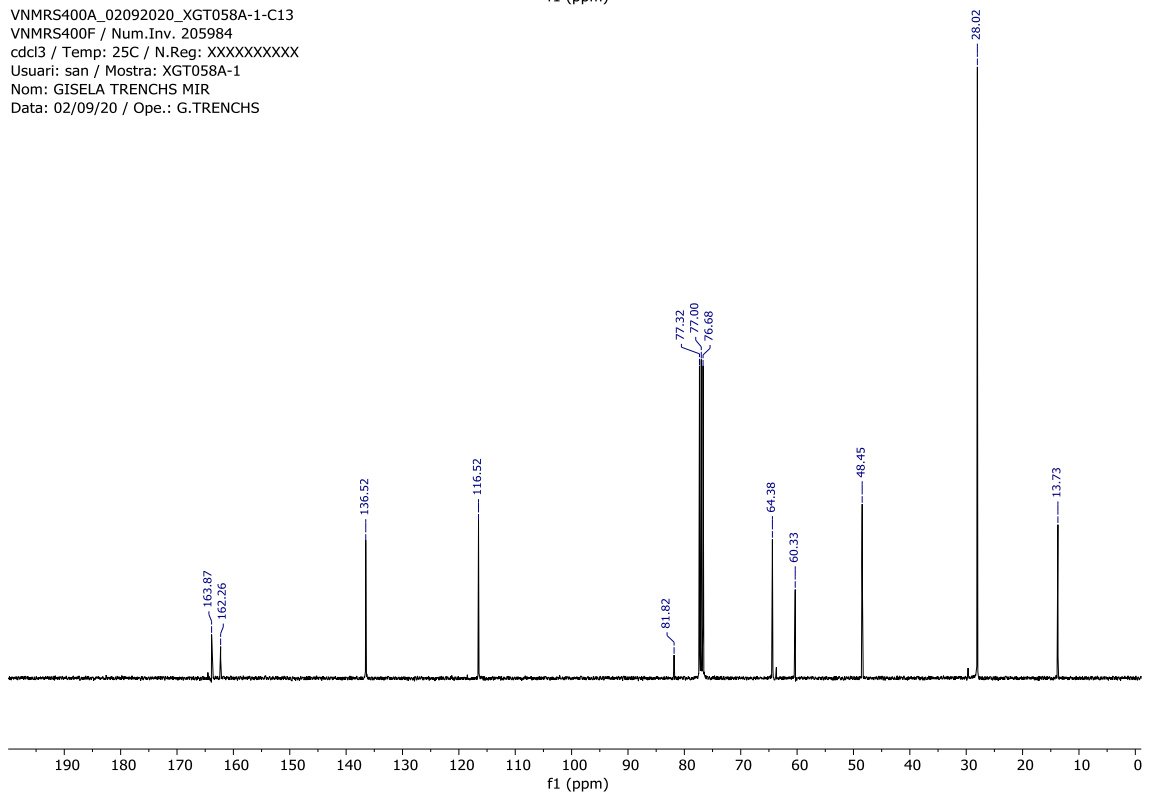


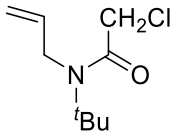


M400AFF\_02092020\_GT058A-1-H1  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: GT058A-1  
 Nom: GISELA TRENCHS MIR  
 Data: 02/09/20 / Ope.: G.TRENCHS



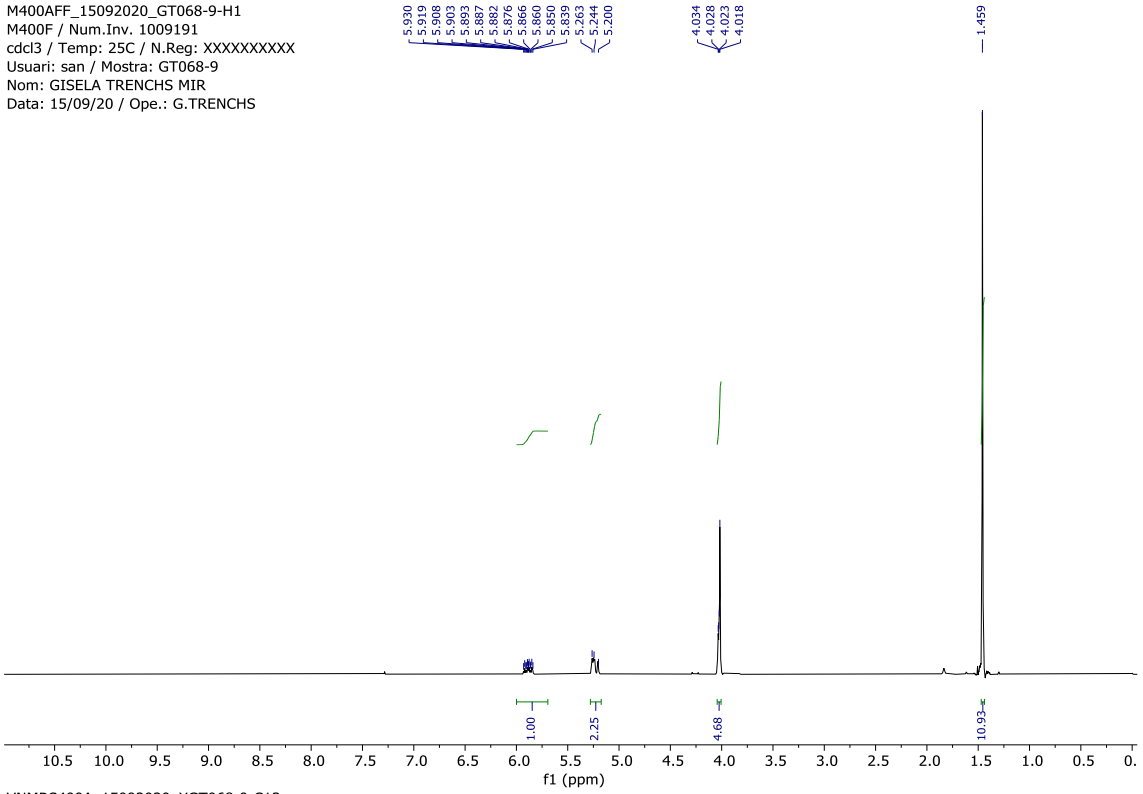
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 VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Nom: GISELA TRENCHS MIR  
 Data: 02/09/20 / Ope.: G.TRENCHS



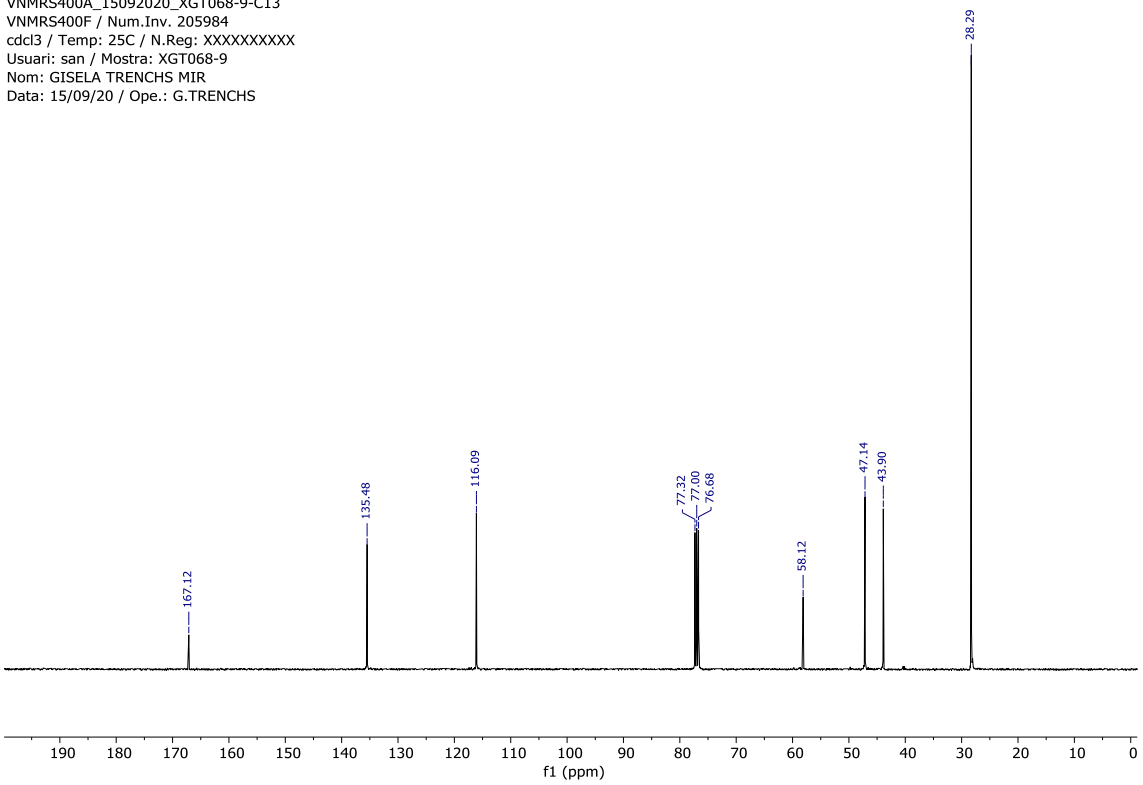


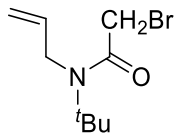
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M400AFF\_15092020\_GT068-9-H1  
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cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
Usuari: san / Mostra: GT068-9  
Nom: GISELA TRENCHS MIR  
Data: 15/09/20 / Ope.: G.TRENCHS



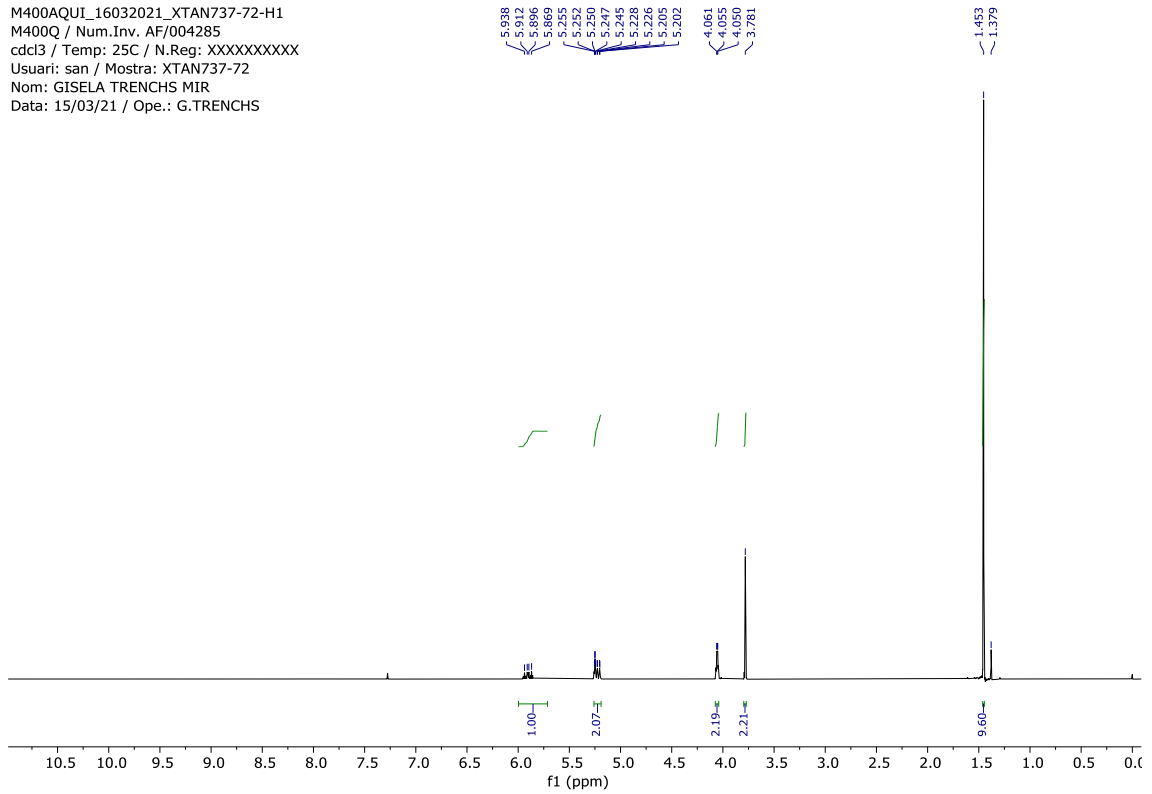
VNMRS400A\_15092020\_XGT068-9-C13  
VNMRS400F / Num.Inv. 205984  
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
Usuari: san / Mostra: XGT068-9  
Nom: GISELA TRENCHS MIR  
Data: 15/09/20 / Ope.: G.TRENCHS



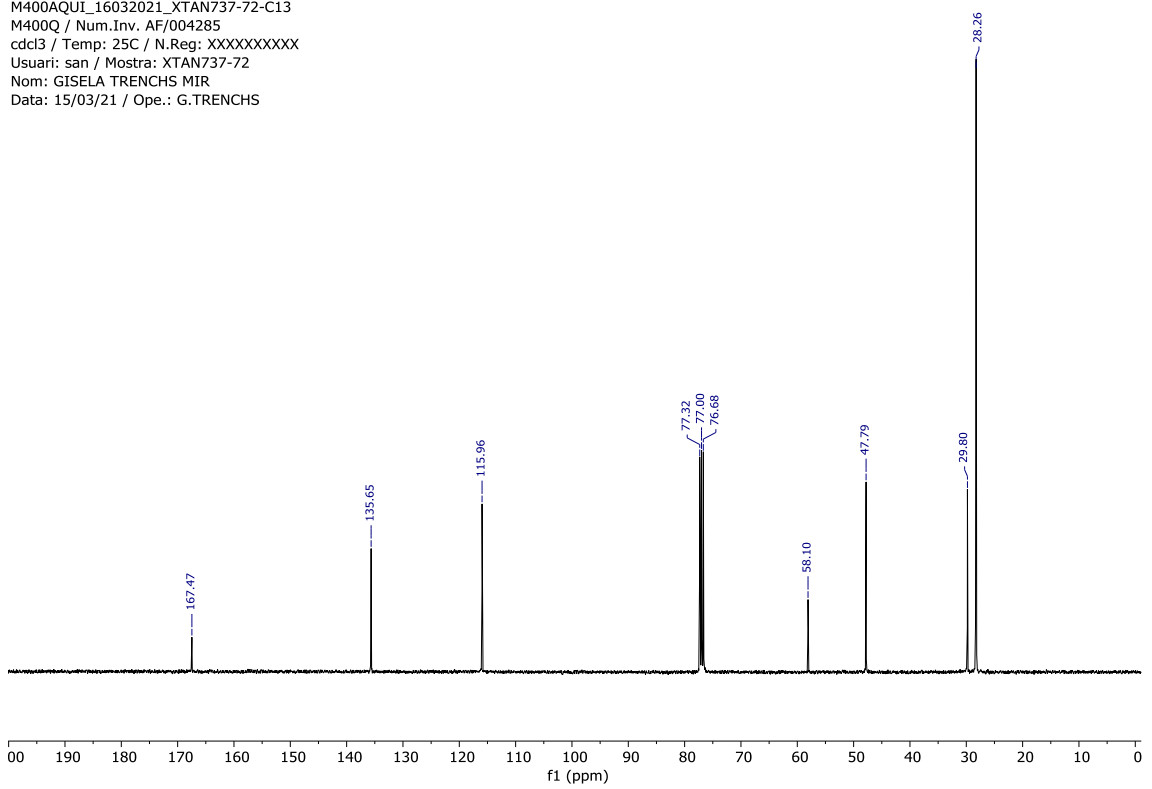


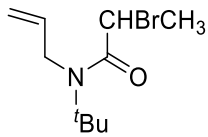
**1s**

M400AQUI\_16032021\_XTAN737-72-H1  
M400Q / Num.Inv. AF/004285  
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
Usuari: san / Mostra: XTAN737-72  
Nom: GISELA TRENCHS MIR  
Data: 15/03/21 / Ope.: G.TRENCHS



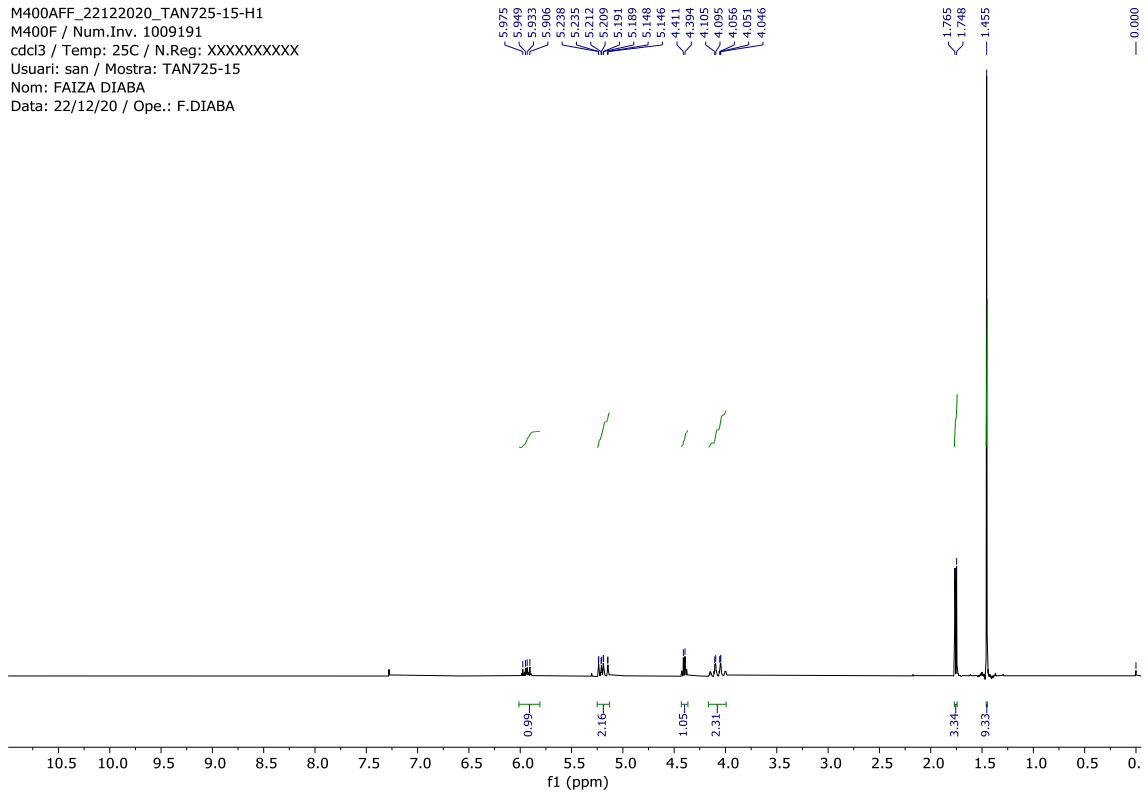
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cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
Usuari: san / Mostra: XTAN737-72  
Nom: GISELA TRENCHS MIR  
Data: 15/03/21 / Ope.: G.TRENCHS



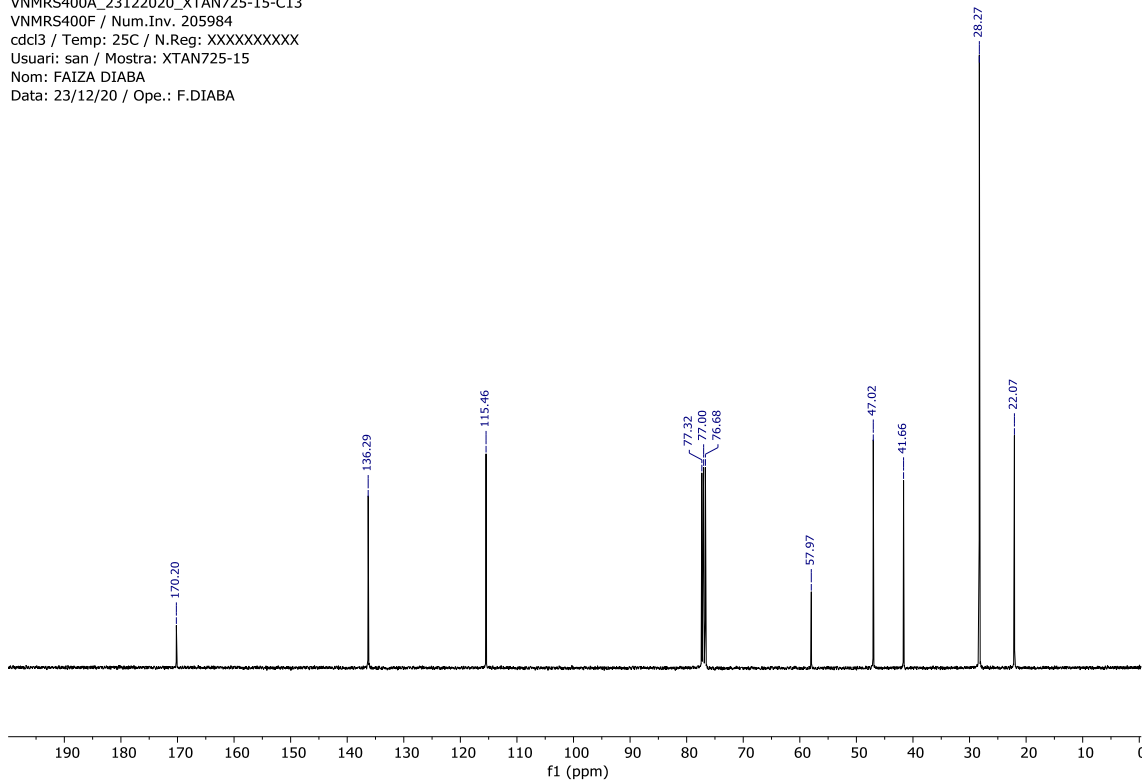


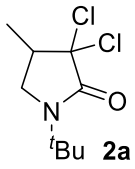
**1t**

M400AFF\_22122020\_TAN725-15-H1  
 M400F / Num.Inv. 1009191  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: TAN725-15  
 Nom: FAIZA DIABA  
 Data: 22/12/20 / Ope.: F.DIABA

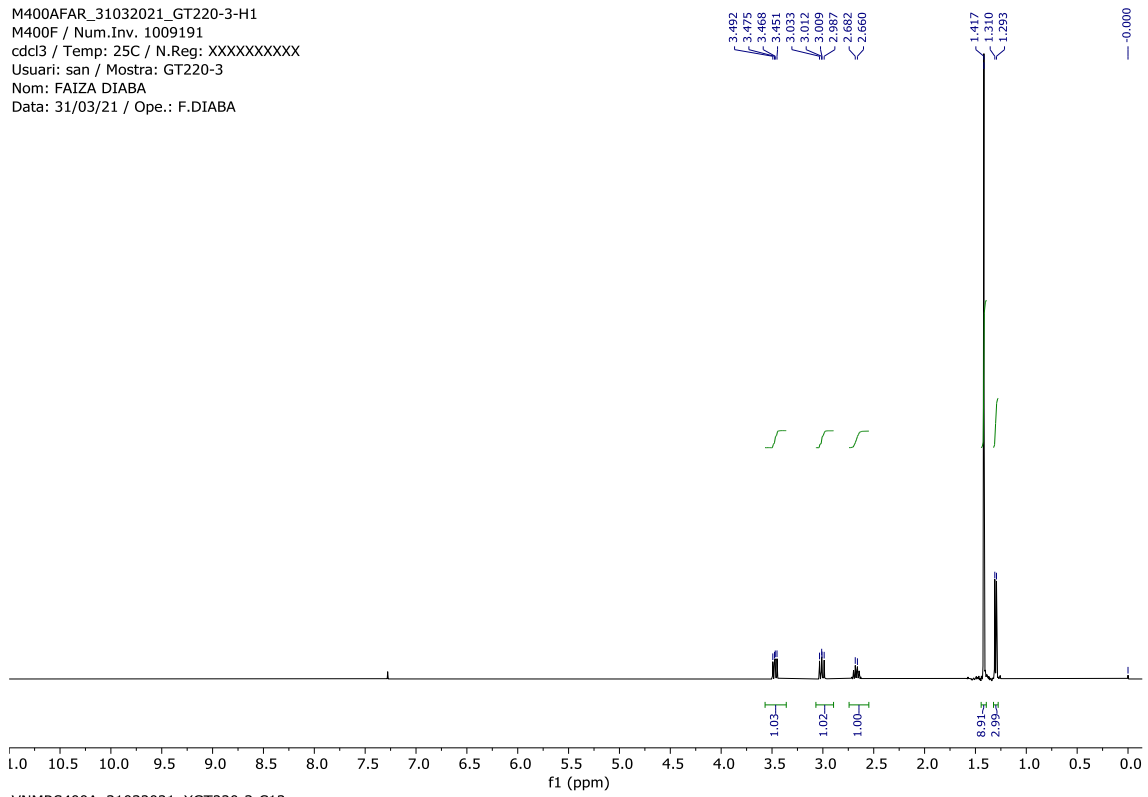


VNMRS400A\_23122020\_XTAN725-15-C13  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: XTAN725-15  
 Nom: FAIZA DIABA  
 Data: 23/12/20 / Ope.: F.DIABA

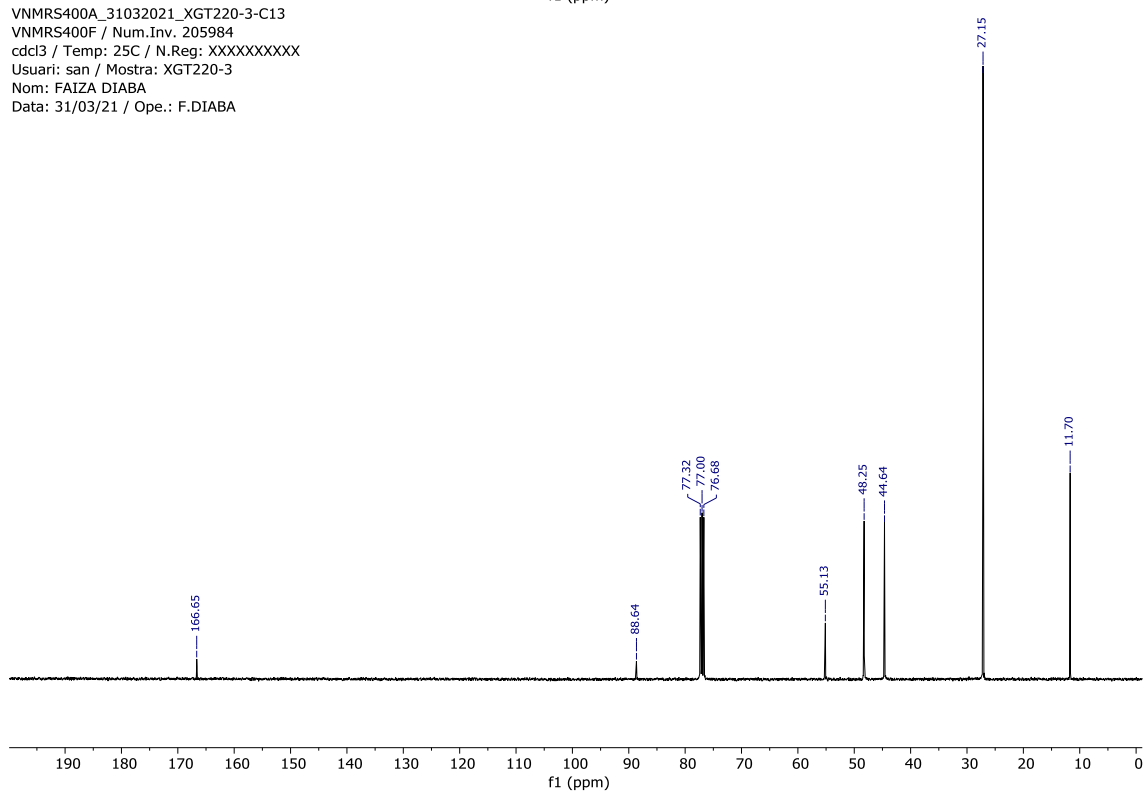


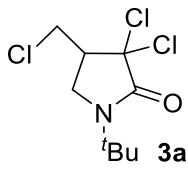


M400AFAR\_31032021\_GT220-3-H1  
 M400F / Num.Inv. 1009191  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: GT220-3  
 Nom: FAIZA DIABA  
 Data: 31/03/21 / Ope.: F.DIABA

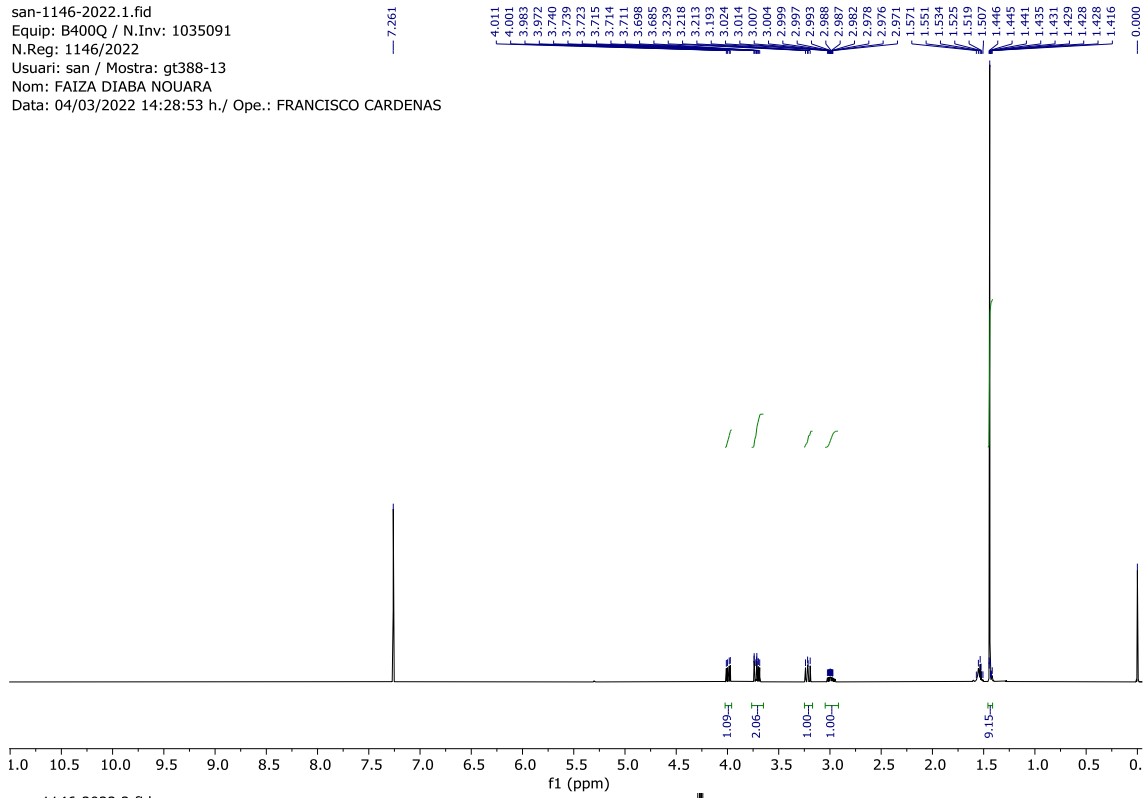


VNMRS400A\_31032021\_XGT220-3-C13  
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 Usuari: san / Mostra: XGT220-3  
 Nom: FAIZA DIABA  
 Data: 31/03/21 / Ope.: F.DIABA

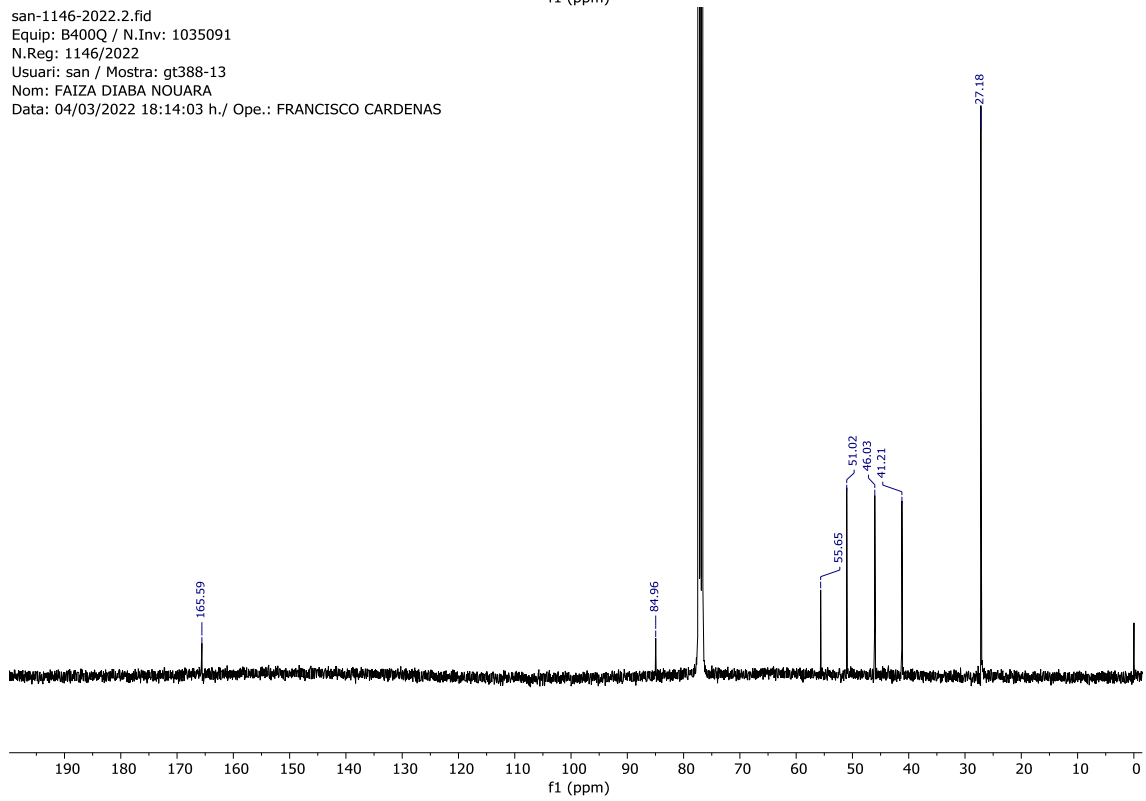


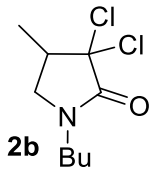


san-1146-2022.1.fid  
 Equip: B400Q / N.Inv: 1035091  
 N.Reg: 1146/2022  
 Usuari: san / Mostra: gt388-13  
 Nom: FAIZA DIABA NOUARA  
 Data: 04/03/2022 14:28:53 h./ Ope.: FRANCISCO CARDENAS

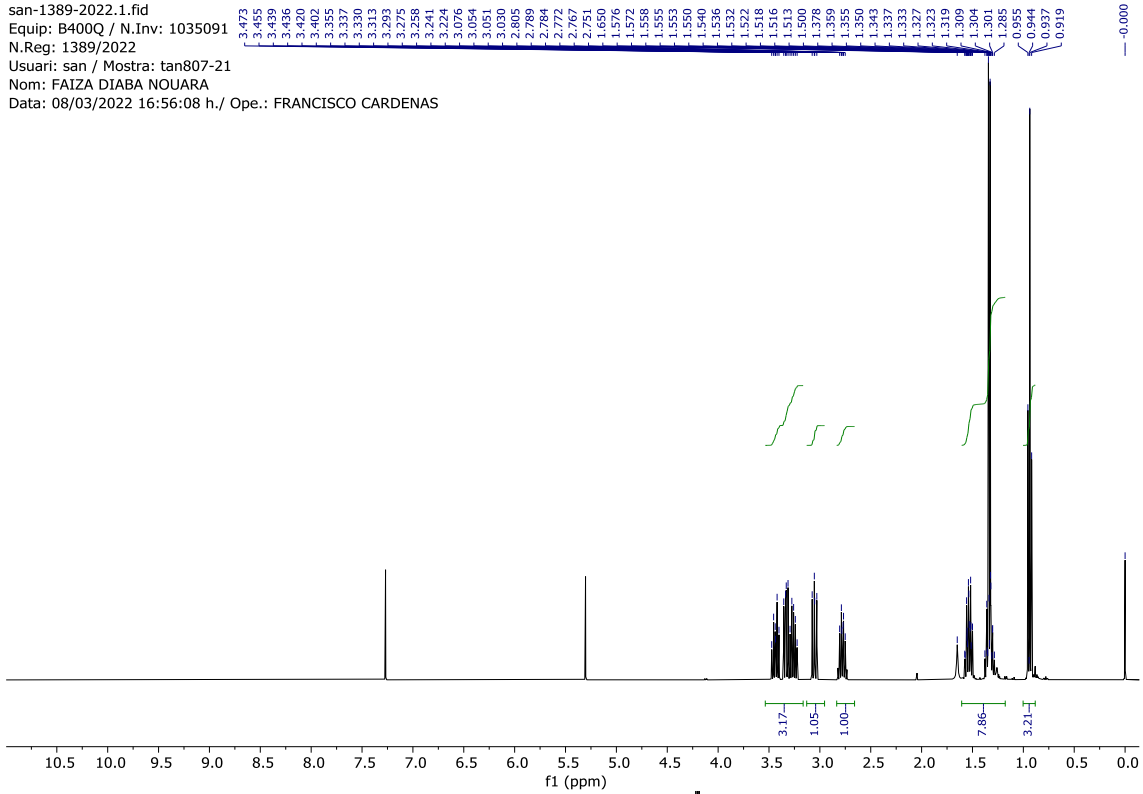


san-1146-2022.2.fid  
 Equip: B400Q / N.Inv: 1035091  
 N.Reg: 1146/2022  
 Usuari: san / Mostra: gt388-13  
 Nom: FAIZA DIABA NOUARA  
 Data: 04/03/2022 18:14:03 h./ Ope.: FRANCISCO CARDENAS

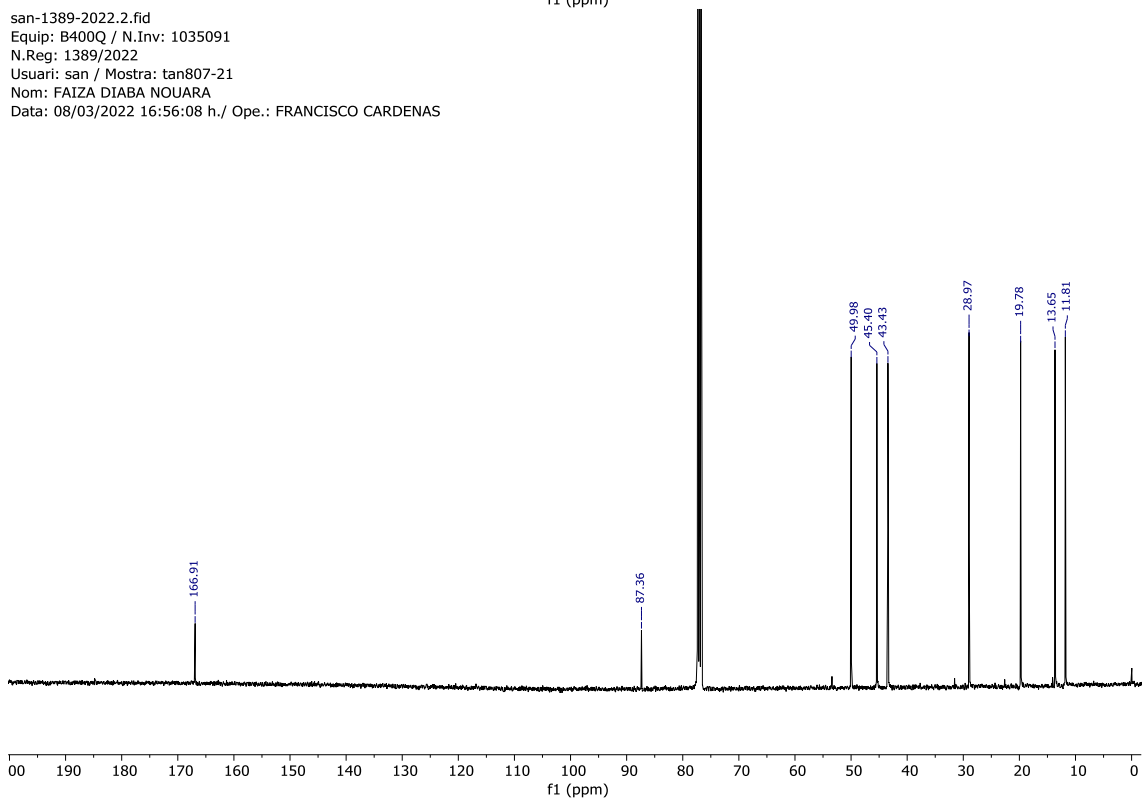


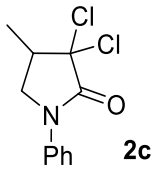


san-1389-2022.1.fid  
 Equip: B400Q / N.Inv: 1035091  
 N.Reg: 1389/2022  
 Usuari: san / Mostra: tan807-21  
 Nom: FAIZA DIABA NOUARA  
 Data: 08/03/2022 16:56:08 h./ Ope.: FRANCISCO CARDENAS

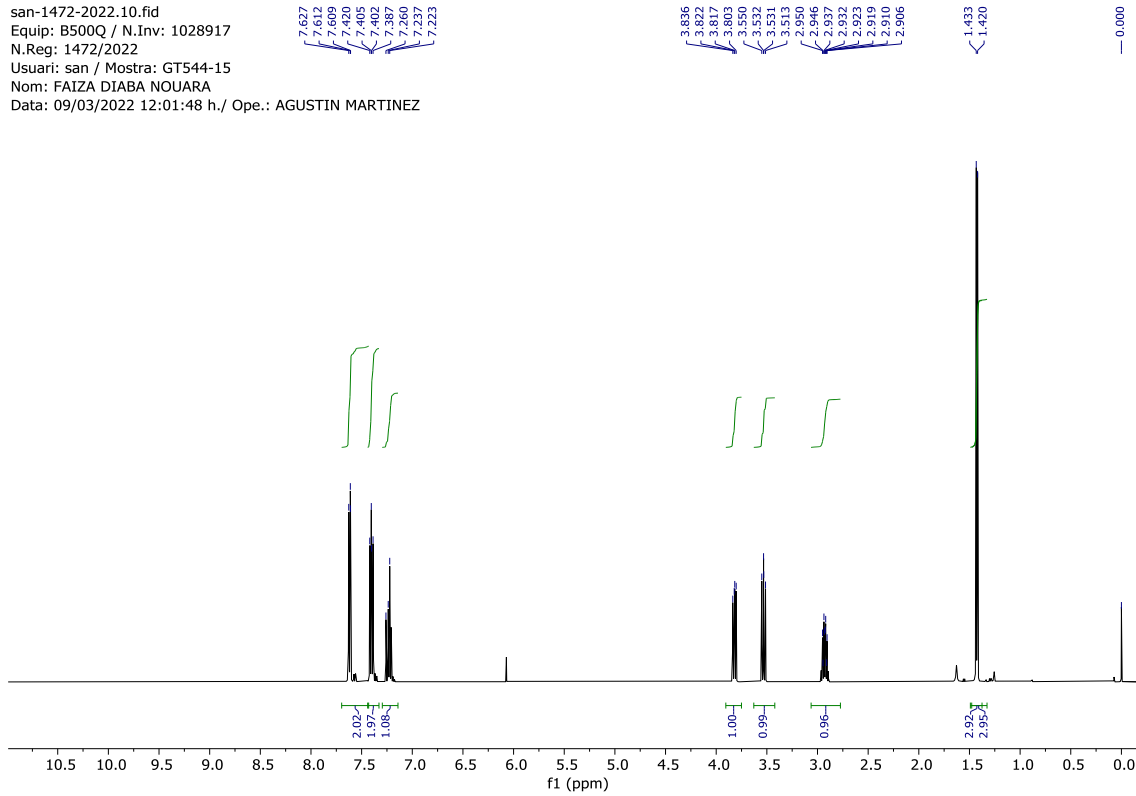


san-1389-2022.2.fid  
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 N.Reg: 1389/2022  
 Usuari: san / Mostra: tan807-21  
 Nom: FAIZA DIABA NOUARA  
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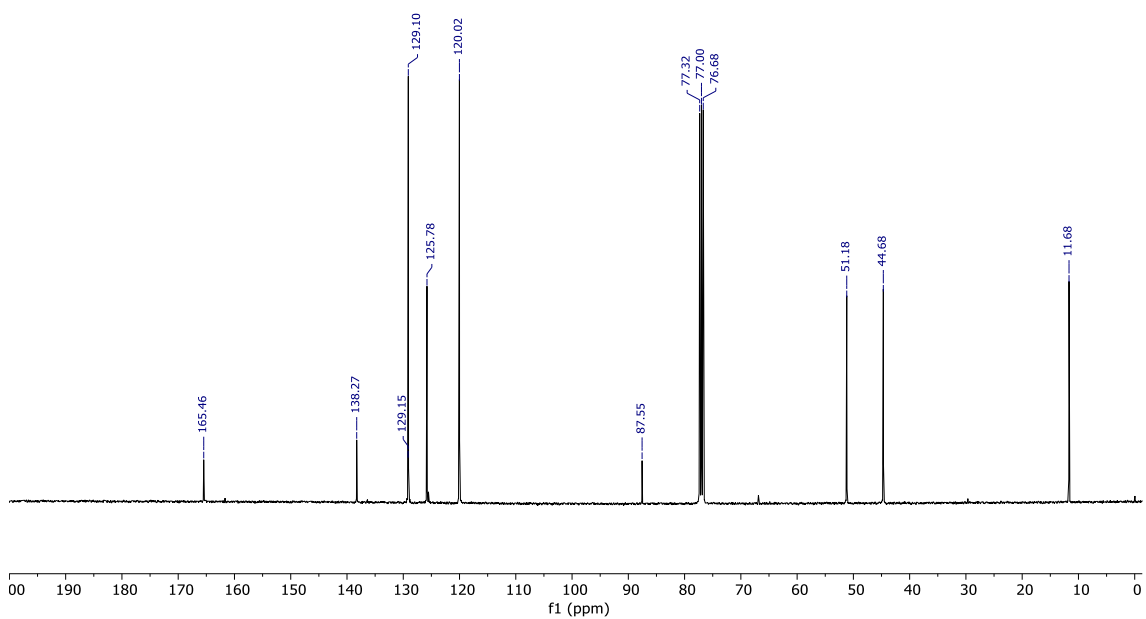




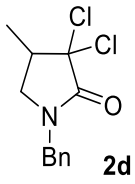
san-1472-2022.10.fid  
 Equip: B500Q / N.Inv: 1028917  
 N.Reg: 1472/2022  
 Usuari: san / Mostra: GT544-15  
 Nom: FAIZA DIABA NOUARA  
 Data: 09/03/2022 12:01:48 h./ Ope.: AGUSTIN MARTINEZ



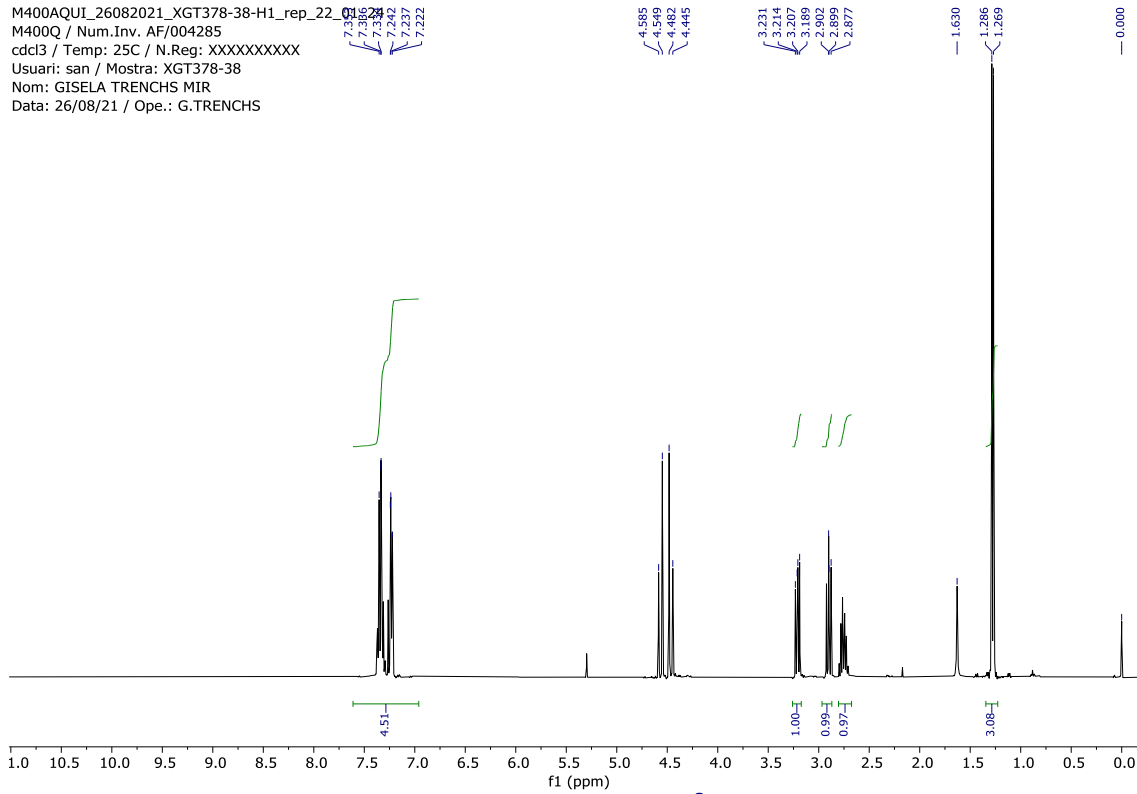
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 N.Reg: 1472/2022  
 Usuari: san / Mostra: GT544-15  
 Nom: FAIZA DIABA NOUARA  
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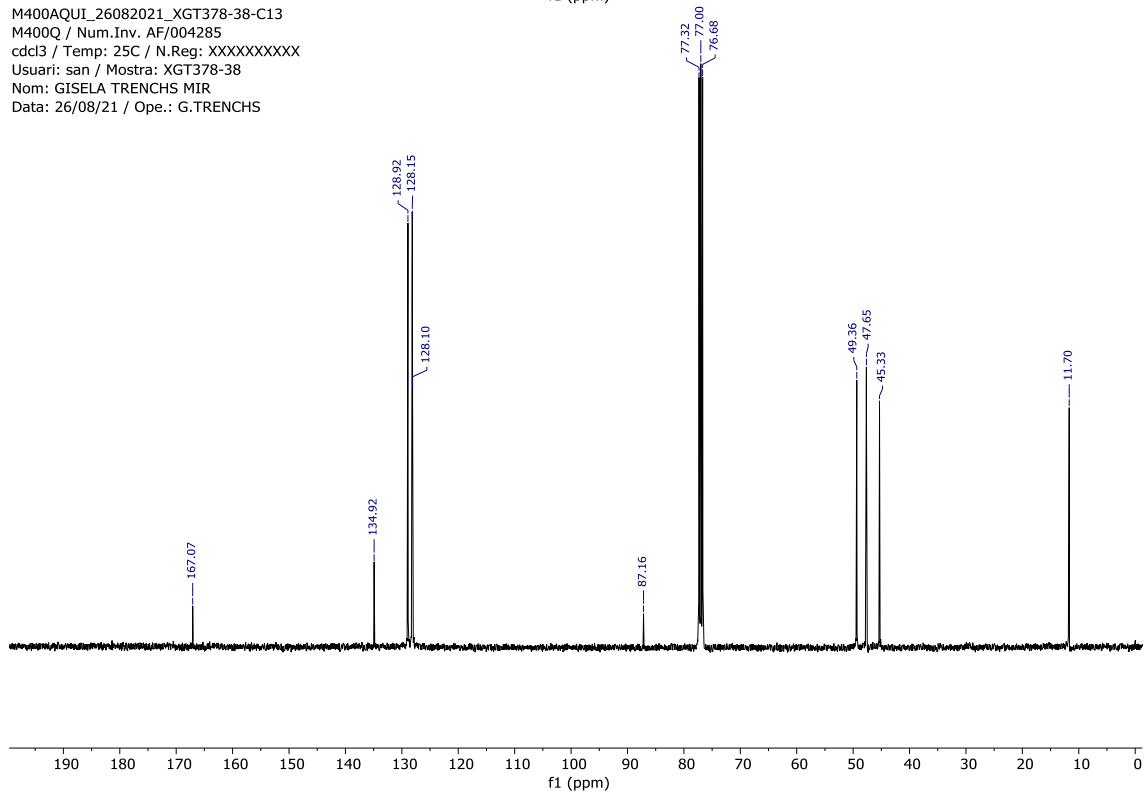


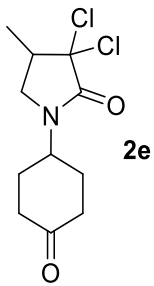


M400AQUI\_26082021\_XGT378-38-H1\_rep\_22\_03  
 M400Q / Num.Inv. AF/004285  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: XGT378-38  
 Nom: GISELA TRENCHS MIR  
 Data: 26/08/21 / Ope.: G.TRENCHS

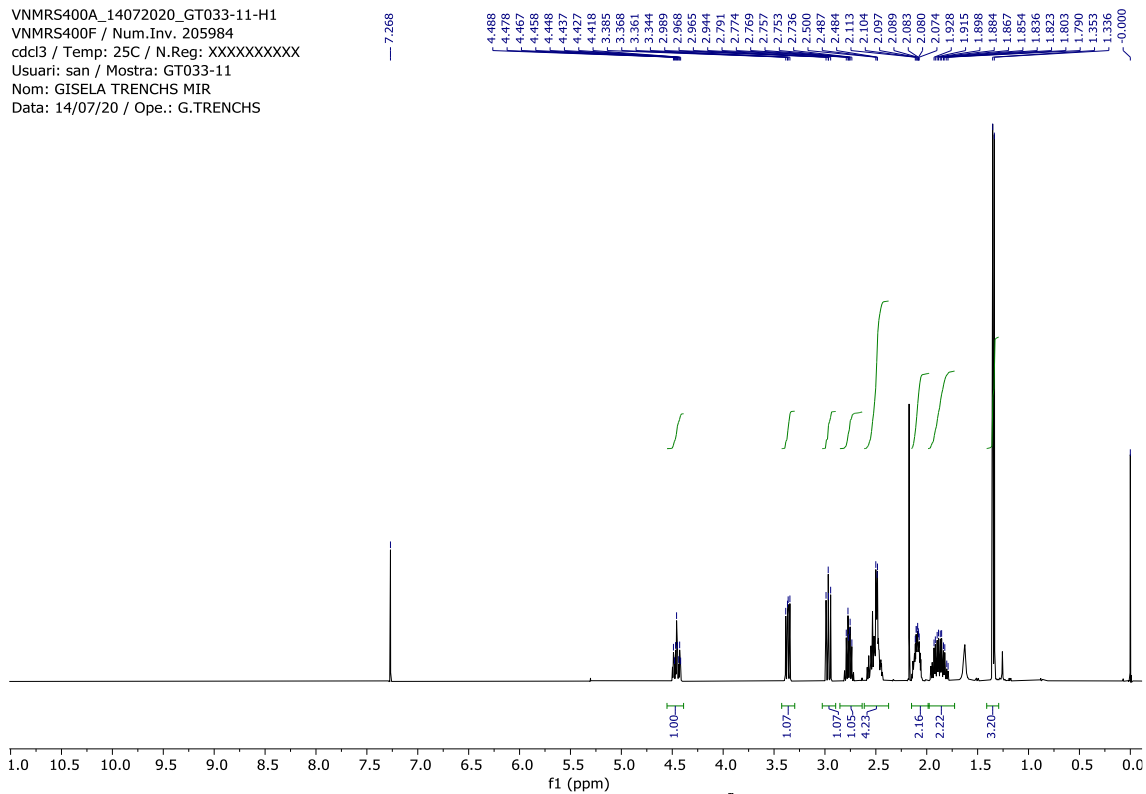


M400AQUI\_26082021\_XGT378-38-C13  
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 Nom: GISELA TRENCHS MIR  
 Data: 26/08/21 / Ope.: G.TRENCHS

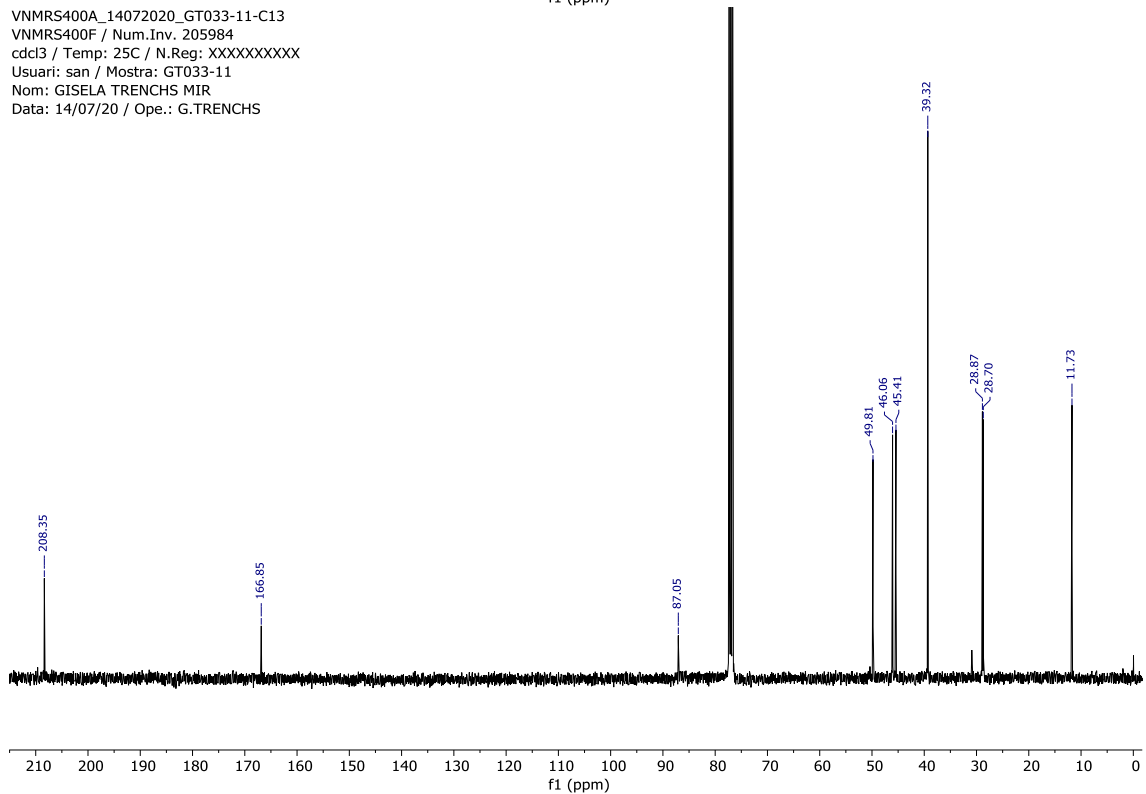


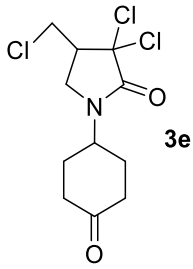


VNMRS400A\_14072020\_GT033-11-H1  
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 Usuari: san / Mostra: GT033-11  
 Nom: GISELA TRENCHS MIR  
 Data: 14/07/20 / Ope.: G.TRENCHS



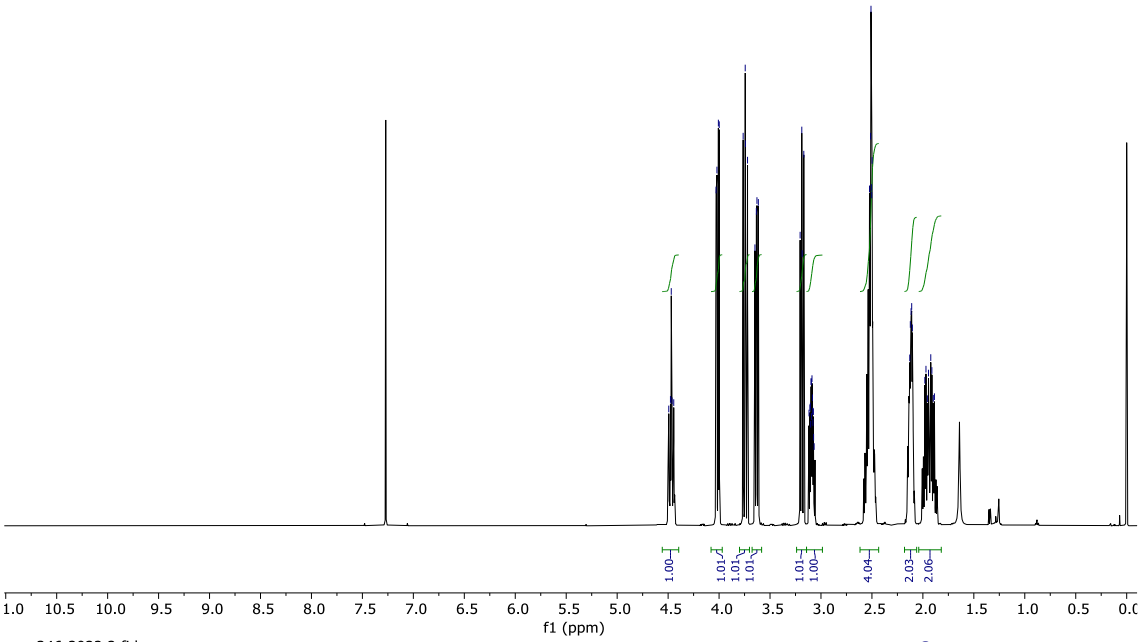
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 Usuari: san / Mostra: GT033-11  
 Nom: GISELA TRENCHS MIR  
 Data: 14/07/20 / Ope.: G.TRENCHS



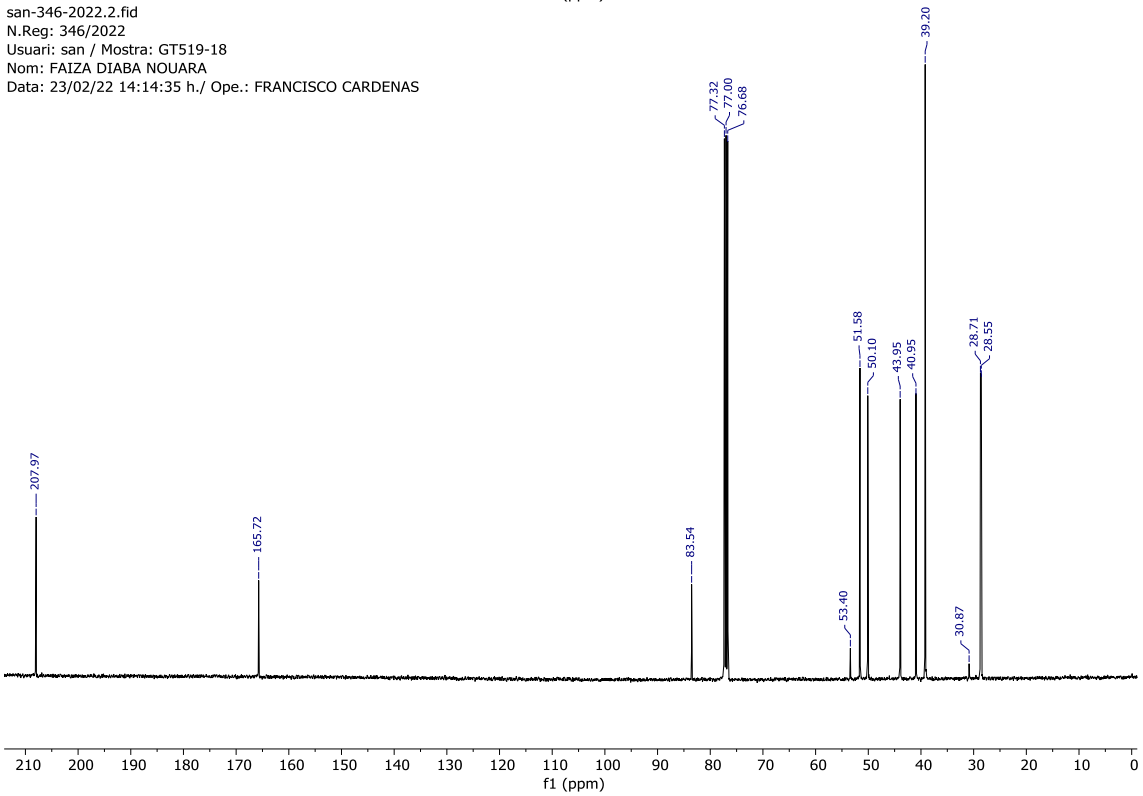


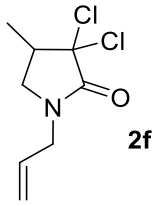
962-2022\_B500QA\_03032022\_GT519-18.1.fid  
 Equip: B500Q / N.Inv: 1028917  
 N.Reg: 962/2022  
 Usuari: san / Mostra: GT519-18  
 Nom: GISELA TRENCHS MIR  
 Data: 03/03/2022 13:21:47 h./ Ope.: ALBERT GALLEN

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4.461  
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4.007  
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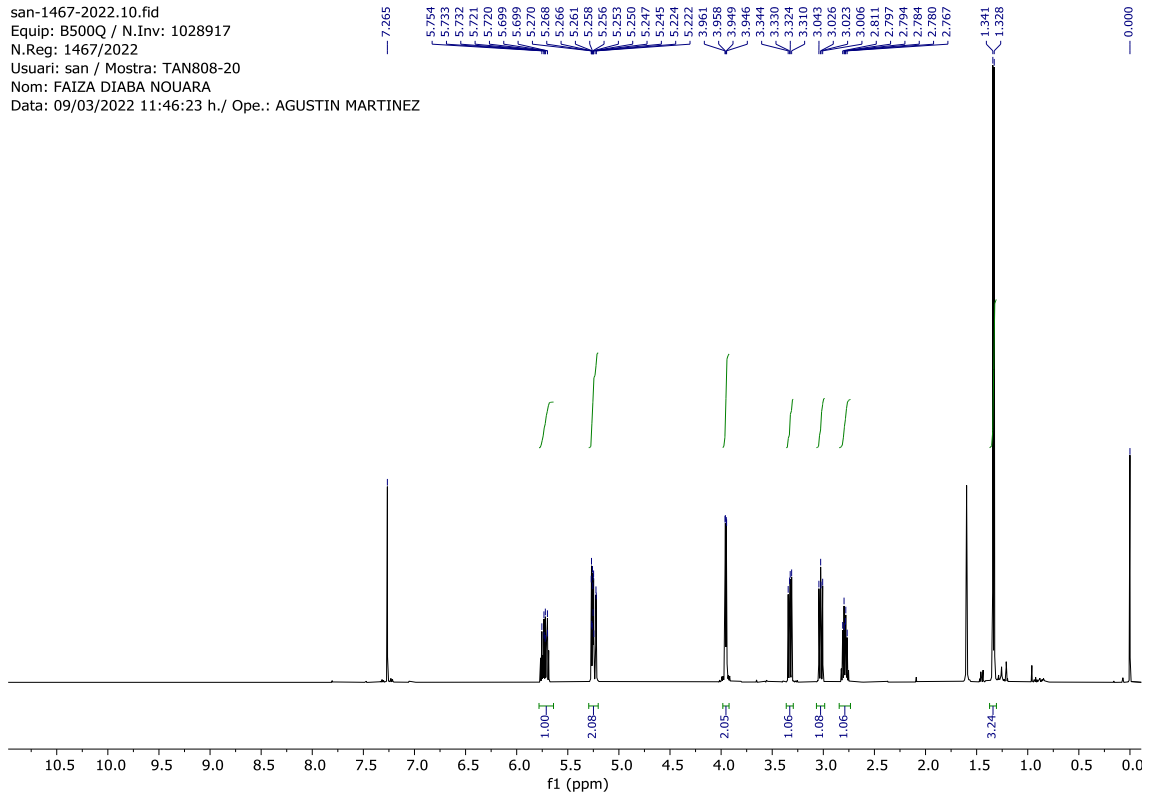


san-346-2022.2.fid  
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 Usuari: san / Mostra: GT519-18  
 Nom: FAIZA DIABA NOUARA  
 Data: 23/02/22 14:14:35 h./ Ope.: FRANCISCO CARDENAS

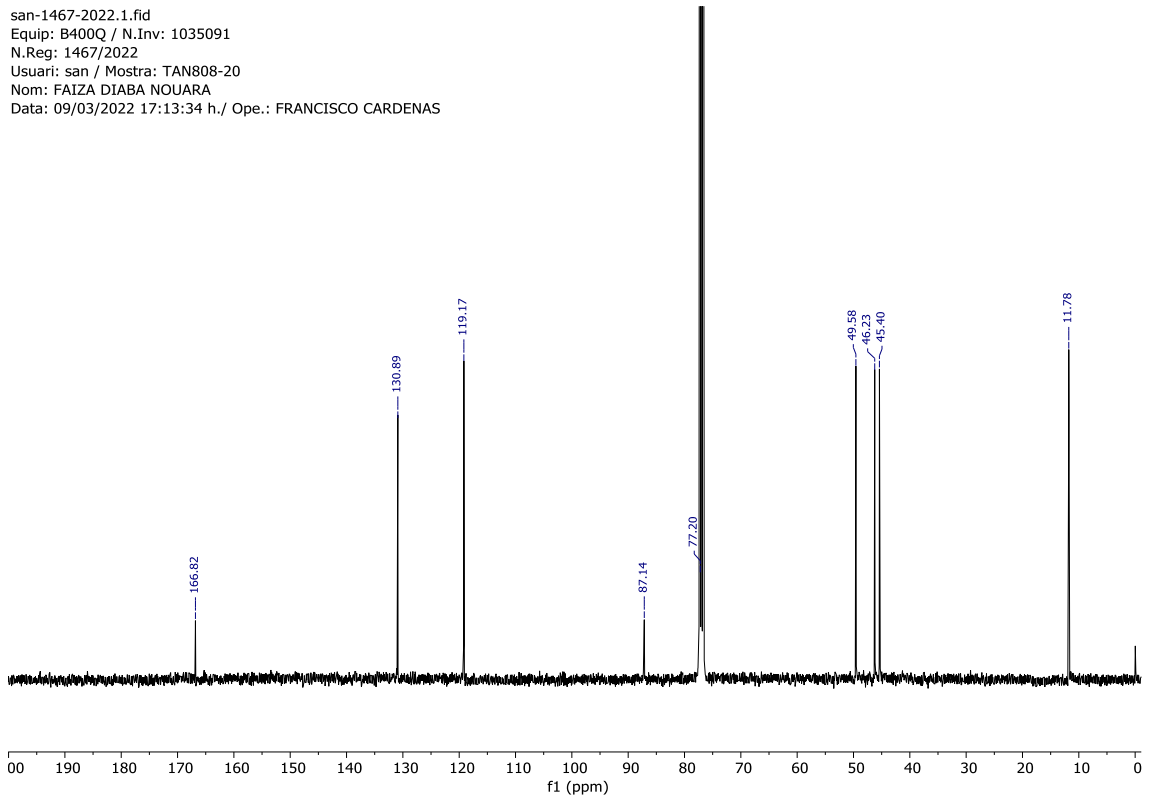


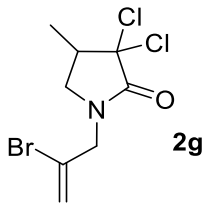


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 N.Reg: 1467/2022  
 Usuari: san / Mostra: TAN808-20  
 Nom: FAIZA DIABA NOUARA  
 Data: 09/03/2022 11:46:23 h./ Ope.: AGUSTIN MARTINEZ

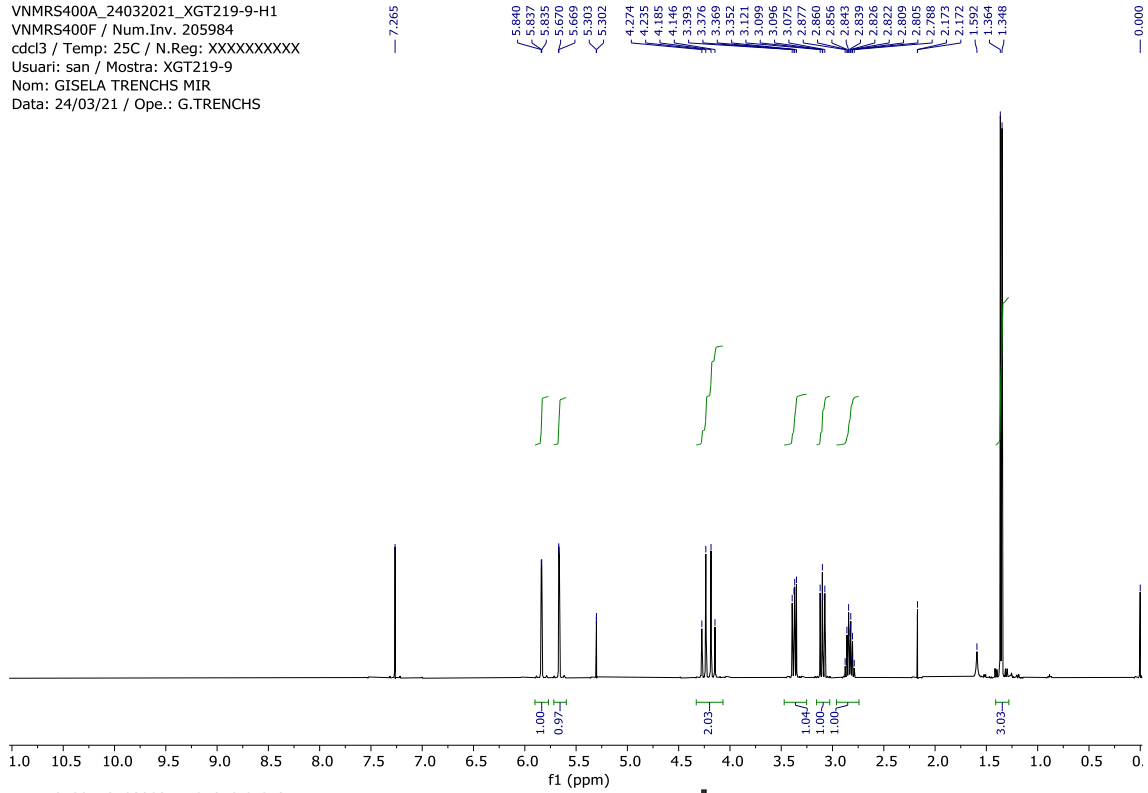


san-1467-2022.1.fid  
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 N.Reg: 1467/2022  
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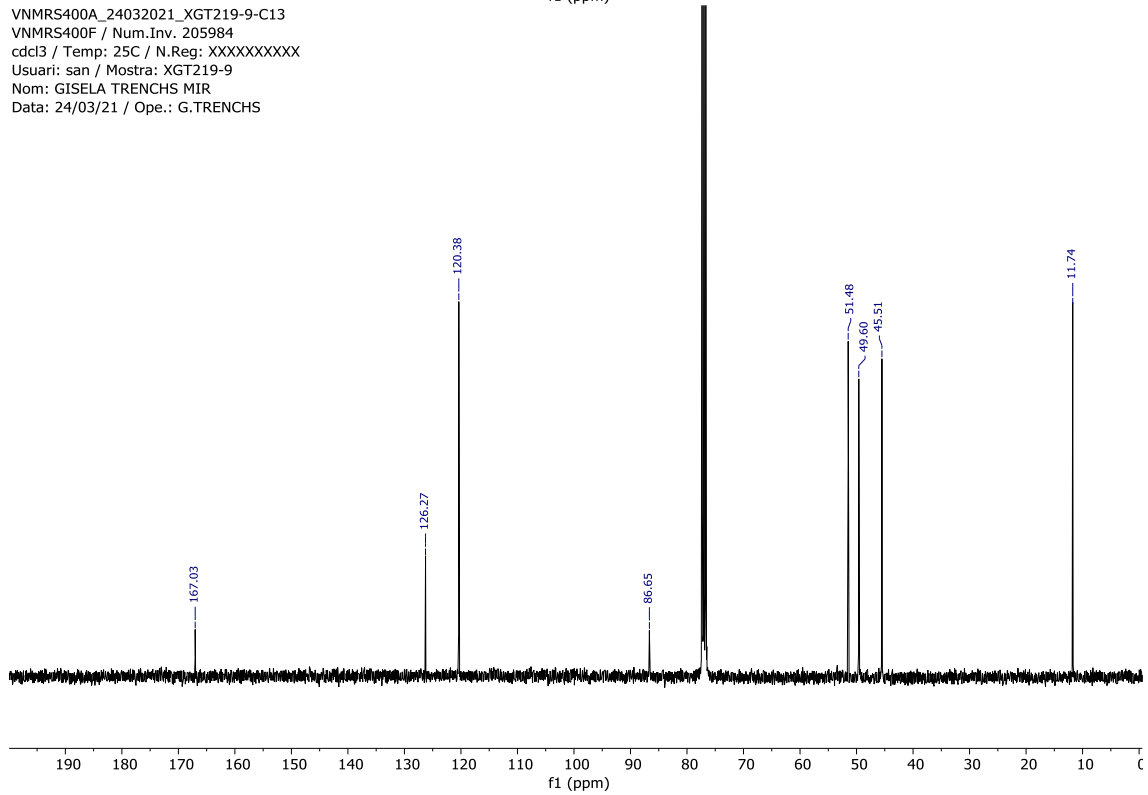


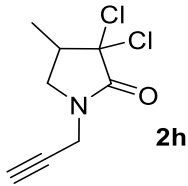


VNMRS400A\_24032021\_XGT219-9-H1  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: XGT219-9  
 Nom: GISELA TRENCHS MIR  
 Data: 24/03/21 / Ope.: G.TRENCHS

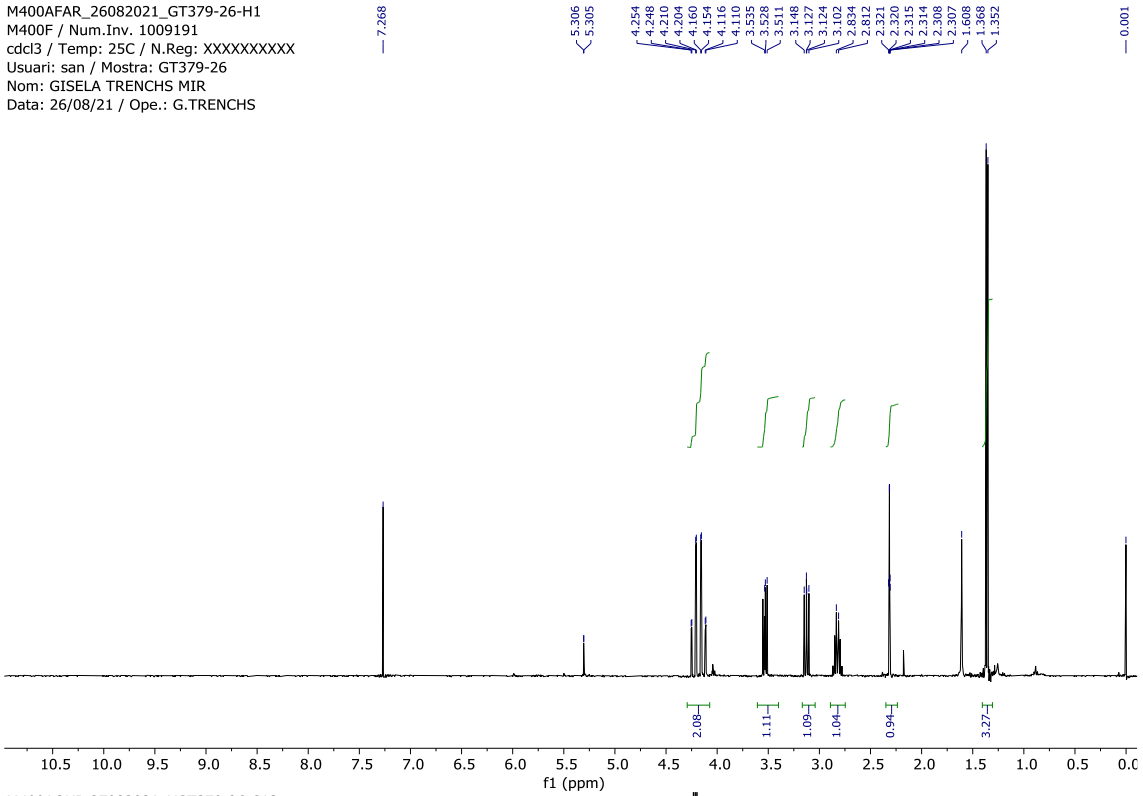


VNMRS400A\_24032021\_XGT219-9-C13  
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 Nom: GISELA TRENCHS MIR  
 Data: 24/03/21 / Ope.: G.TRENCHS

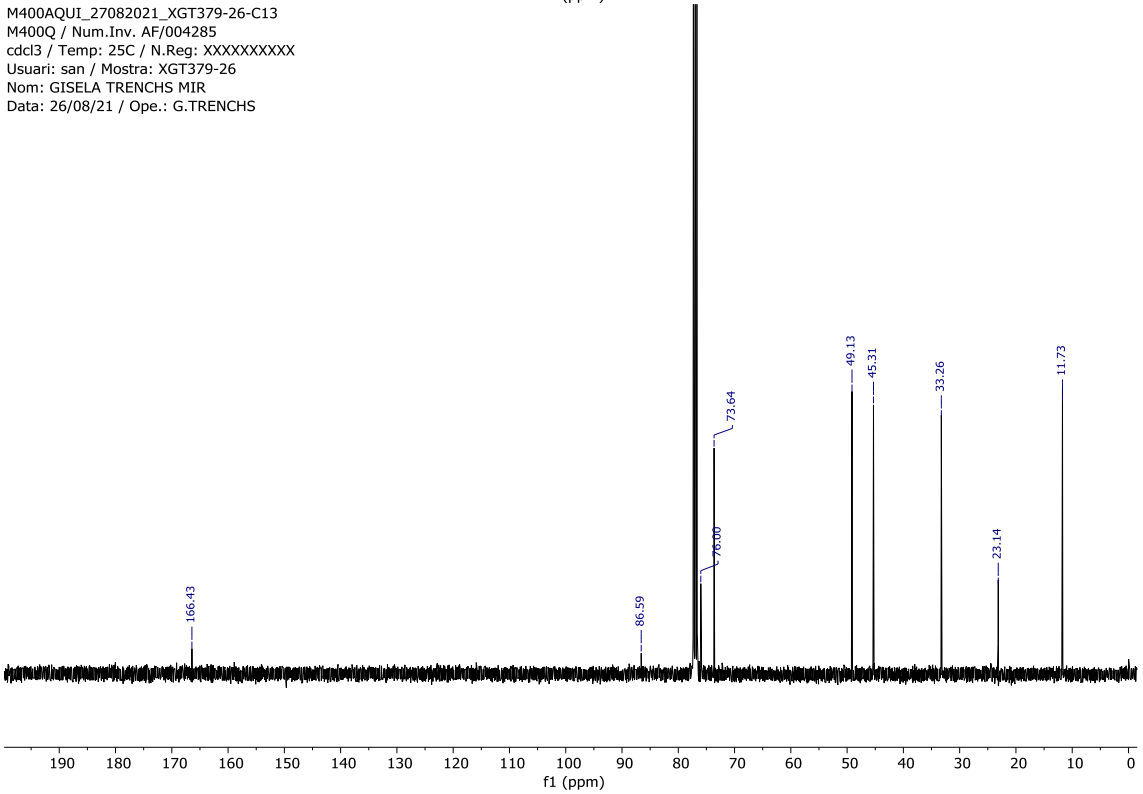


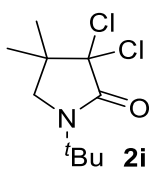


M400AFAR\_26082021\_GT379-26-H1  
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 Usuari: san / Mostra: GT379-26  
 Nom: GISELA TRENCHS MIR  
 Data: 26/08/21 / Ope.: G.TRENCHS

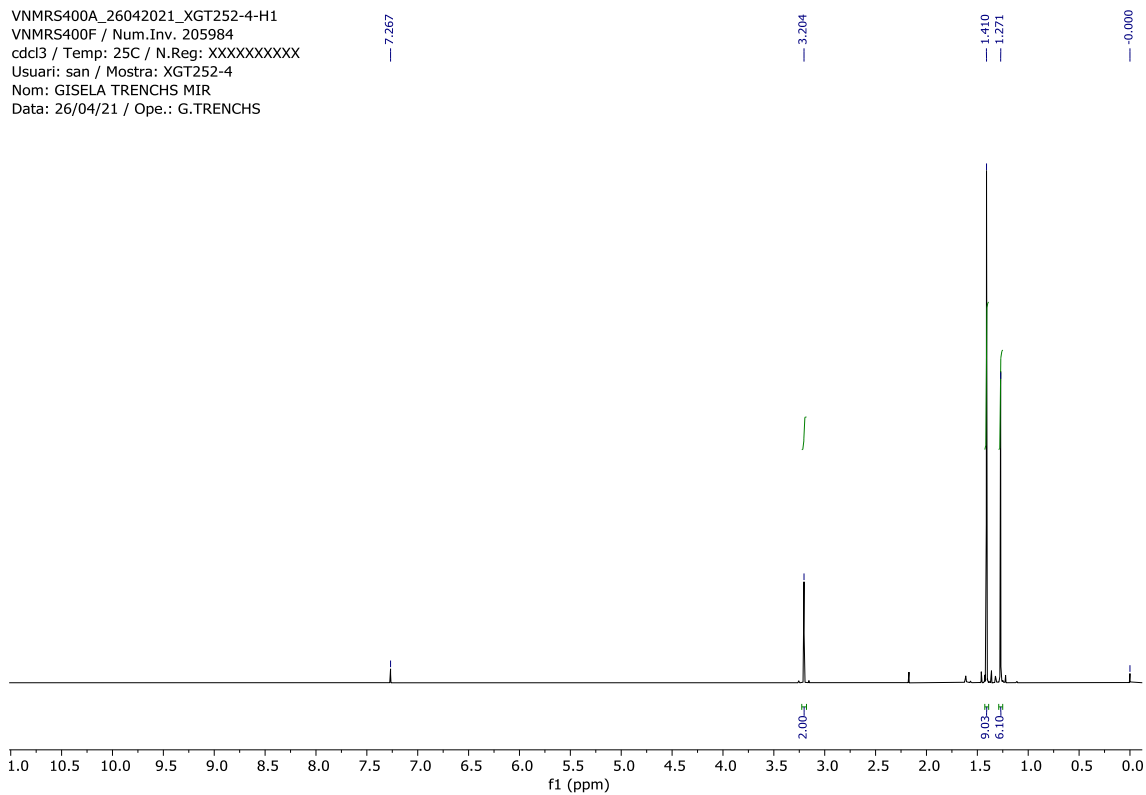


M400AQUI\_27082021\_XGT379-26-C13  
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 Nom: GISELA TRENCHS MIR  
 Data: 26/08/21 / Ope.: G.TRENCHS

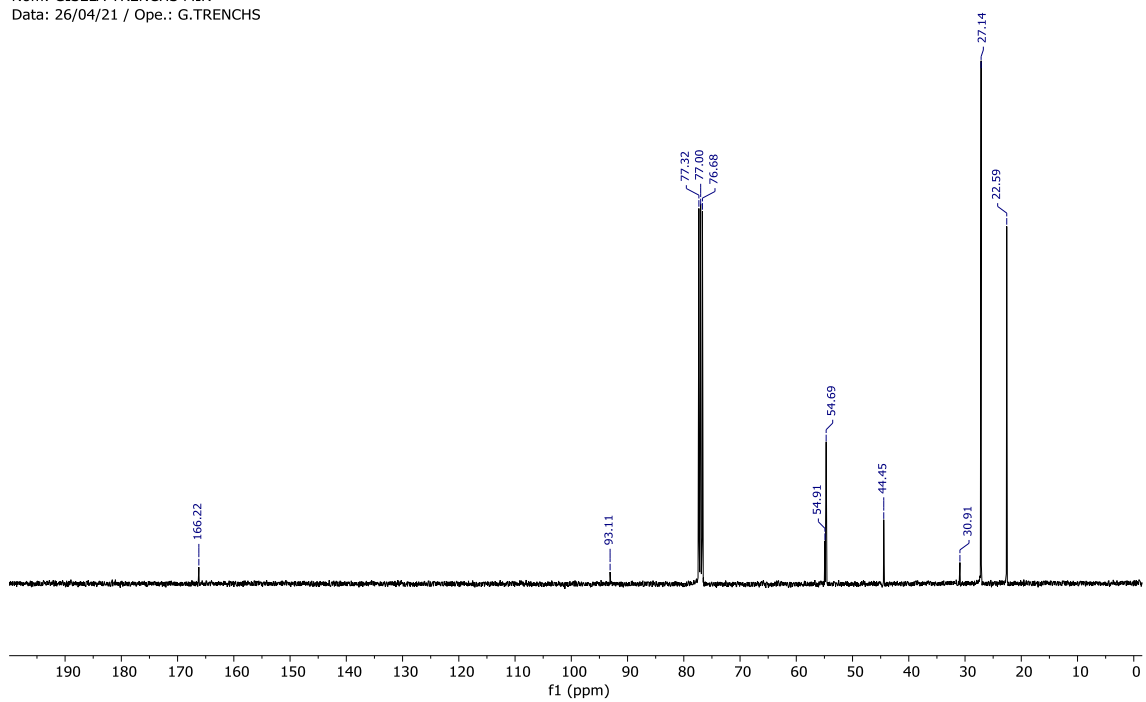


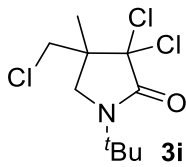


VNMRS400A\_26042021\_XGT252-4-H1  
 VNMRS400F / Num.Inv. 205984  
 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
 Usuari: san / Mostra: XGT252-4  
 Nom: GISELA TRENCHS MIR  
 Data: 26/04/21 / Ope.: G.TRENCHS

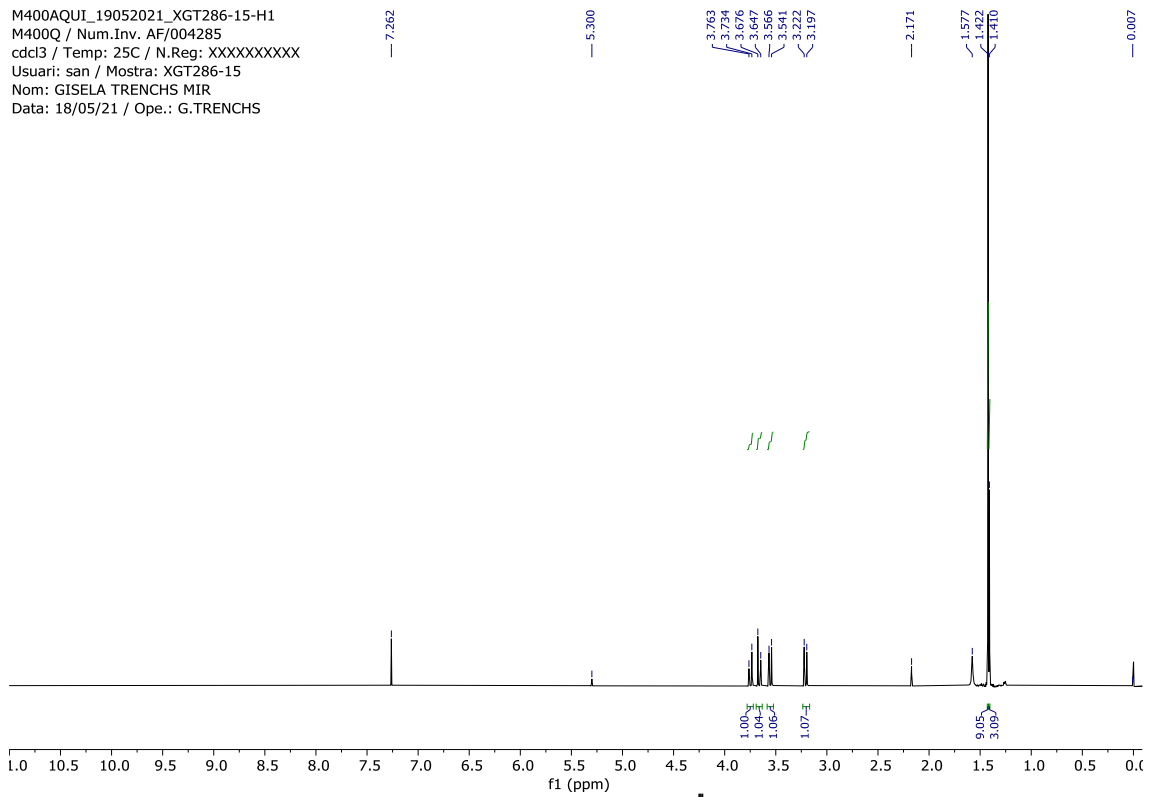


VNMRS400A\_26042021\_XGT252-4-C13  
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 Data: 26/04/21 / Ope.: G.TRENCHS

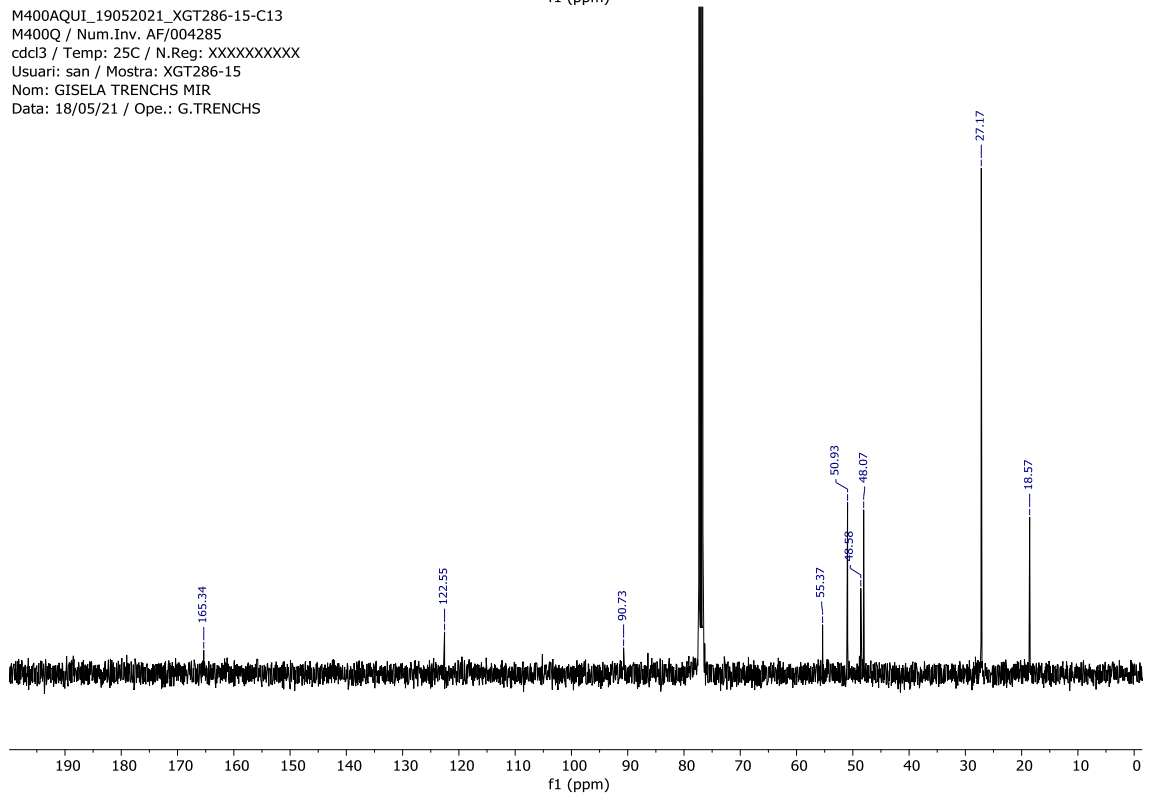




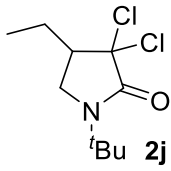
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 M400Q / Num.Inv. AF/004285  
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 Nom: GISELA TRENCHS MIR  
 Data: 18/05/21 / Ope.: G.TRENCHS



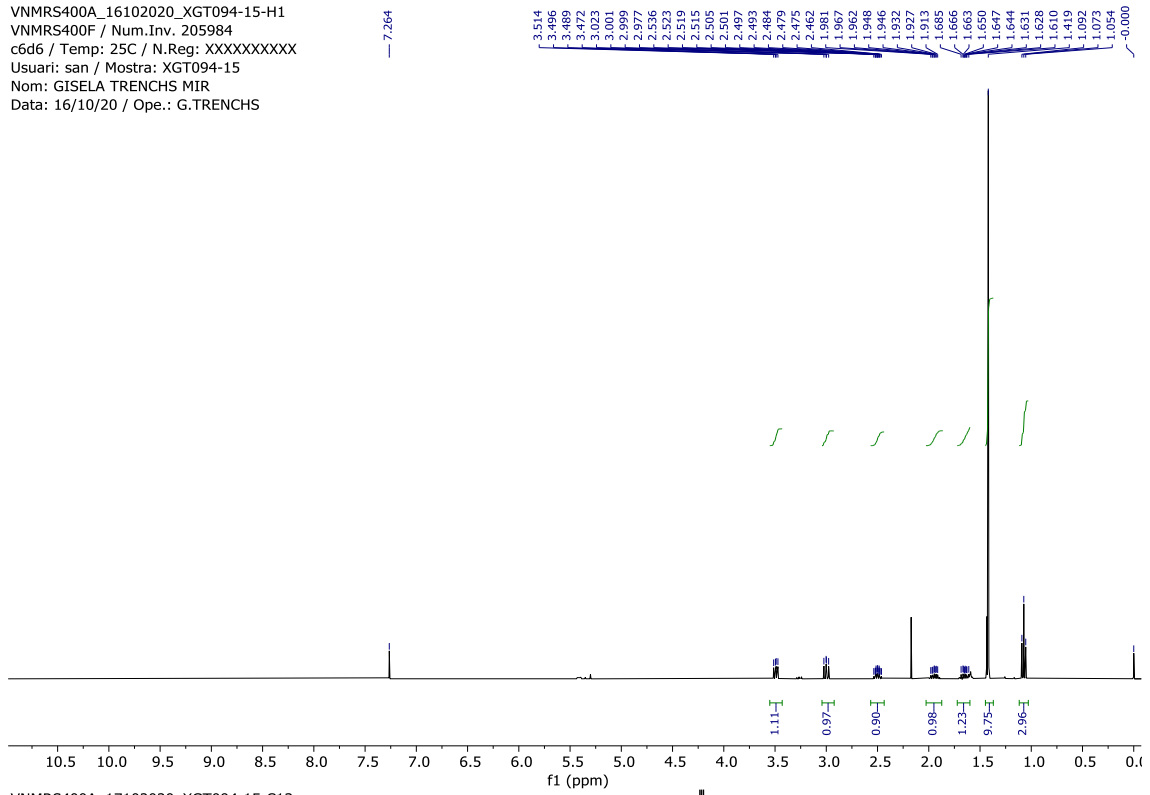
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Nom: GISELA TRENCHS MIR  
 Data: 18/05/21 / Ope.: G.TRENCHS



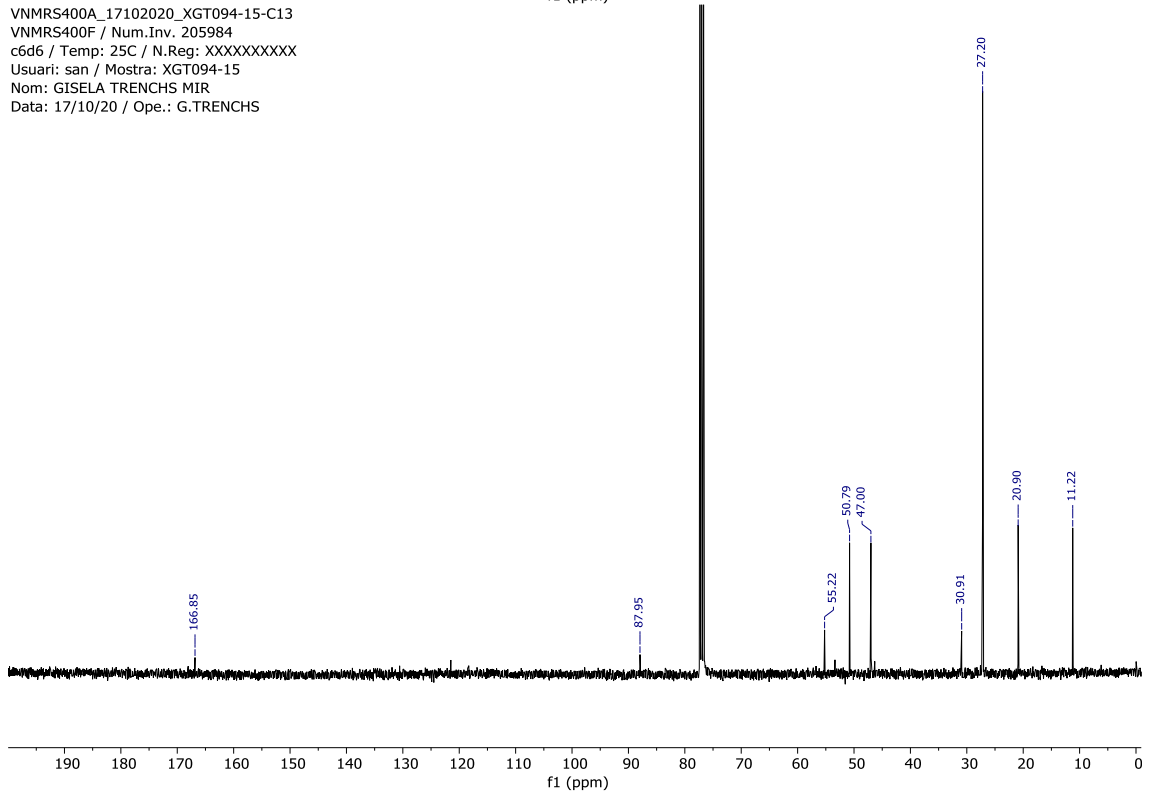


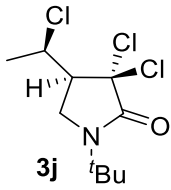


VNMRS400A\_16102020\_XGT094-15-H1  
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 Usuari: san / Mostra: XGT094-15  
 Nom: GISELA TRENCHS MIR  
 Data: 16/10/20 / Ope.: G.TRENCHS

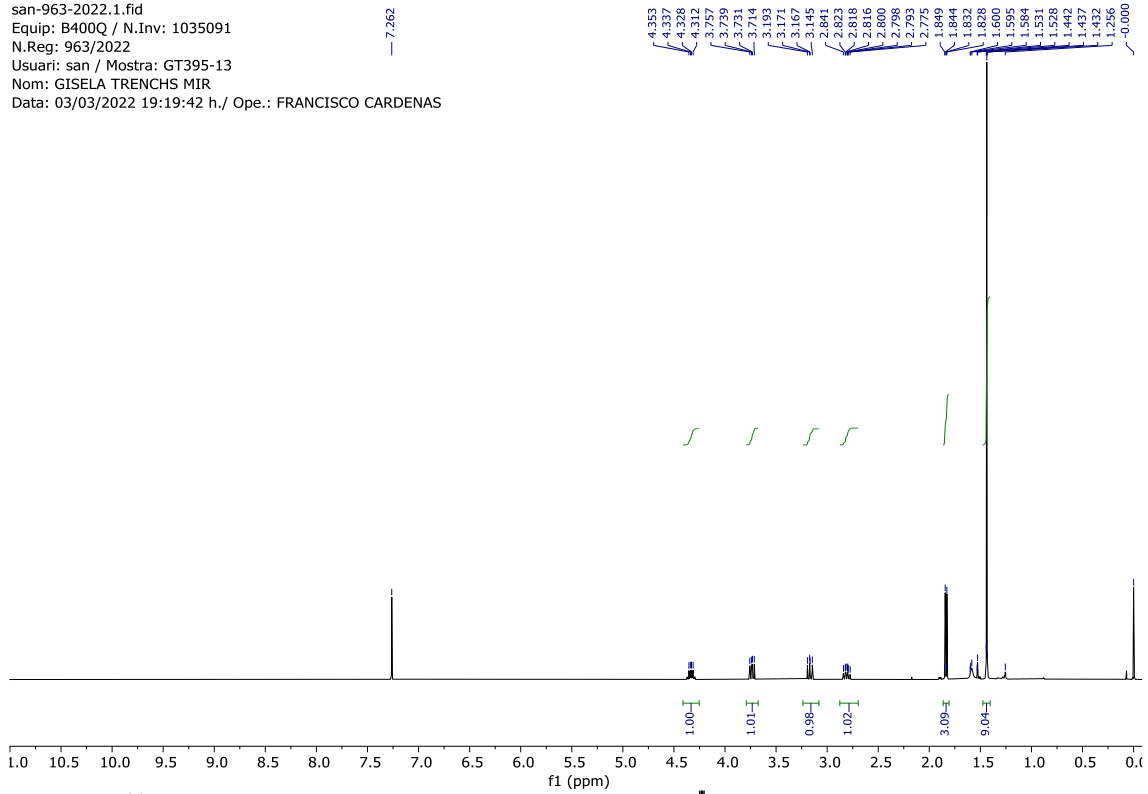


VNMRS400A\_17102020\_XGT094-15-C13  
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 Data: 17/10/20 / Ope.: G.TRENCHS

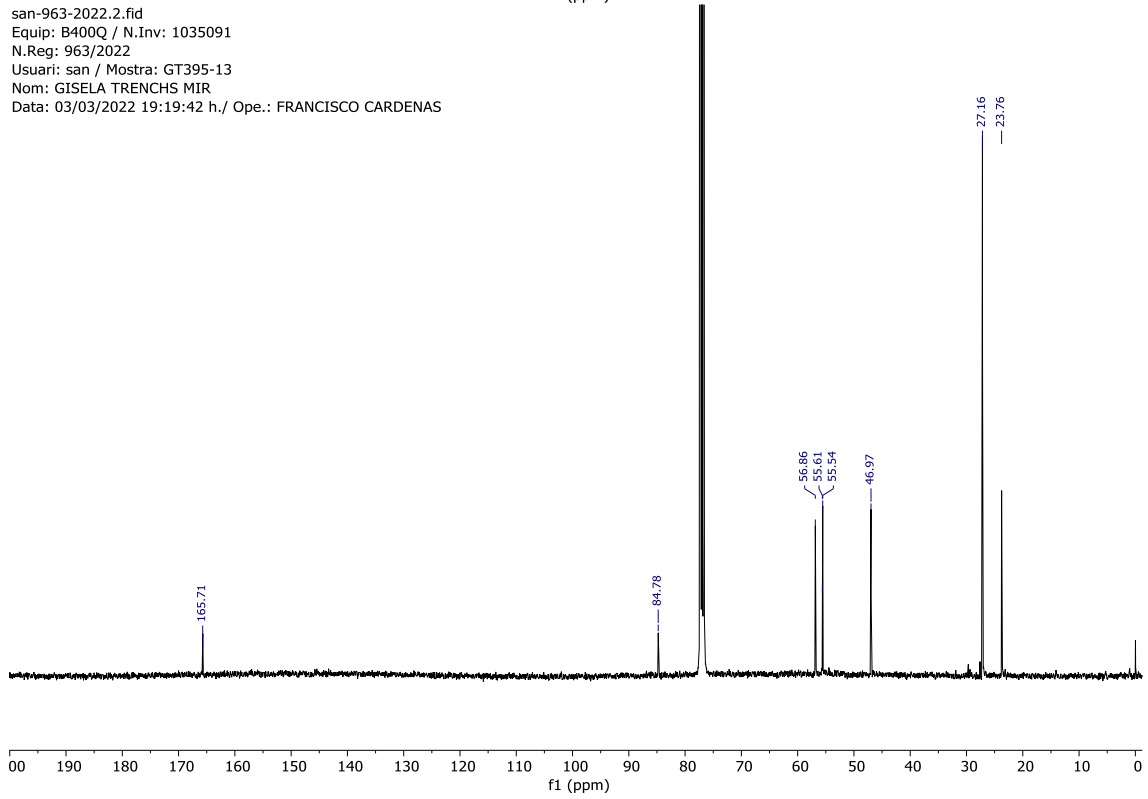


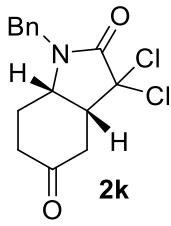


san-963-2022.1.fid  
 Equip: B400Q / N.Inv: 1035091  
 N.Reg: 963/2022  
 Usuari: san / Mostra: GT395-13  
 Nom: GISELA TRENCHS MIR  
 Data: 03/03/2022 19:19:42 h./ Ope.: FRANCISCO CARDENAS

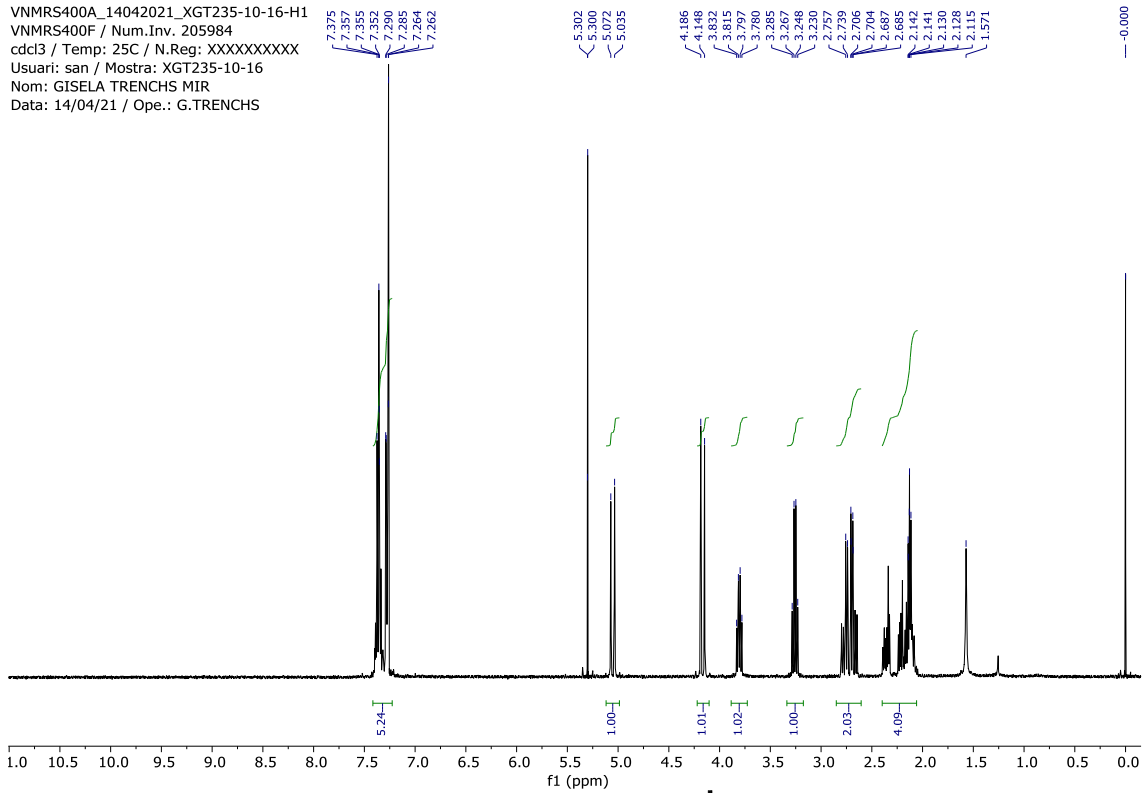


san-963-2022.2.fid  
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 N.Reg: 963/2022  
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 Data: 03/03/2022 19:19:42 h./ Ope.: FRANCISCO CARDENAS

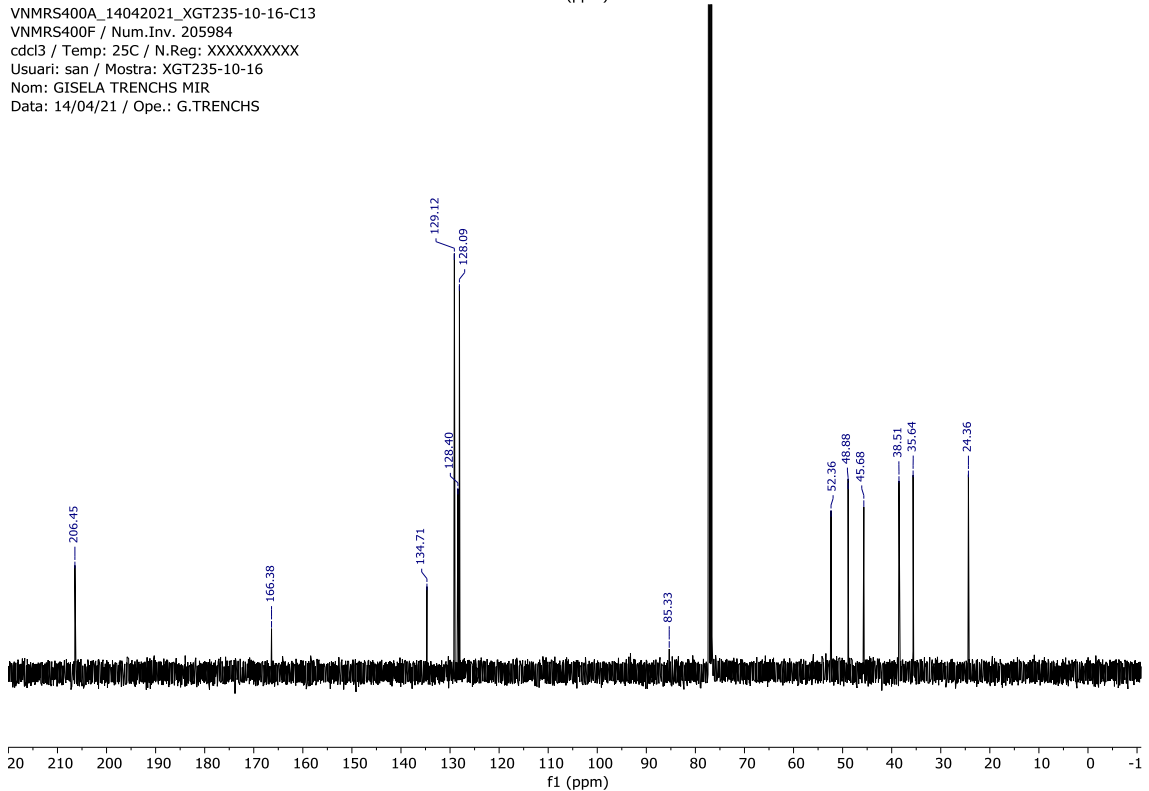


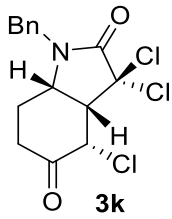


VNMRS400A\_14042021\_XGT235-10-16-H1  
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 Nom: GISELA TRENCHS MIR  
 Data: 14/04/21 / Ope.: G.TRENCHS

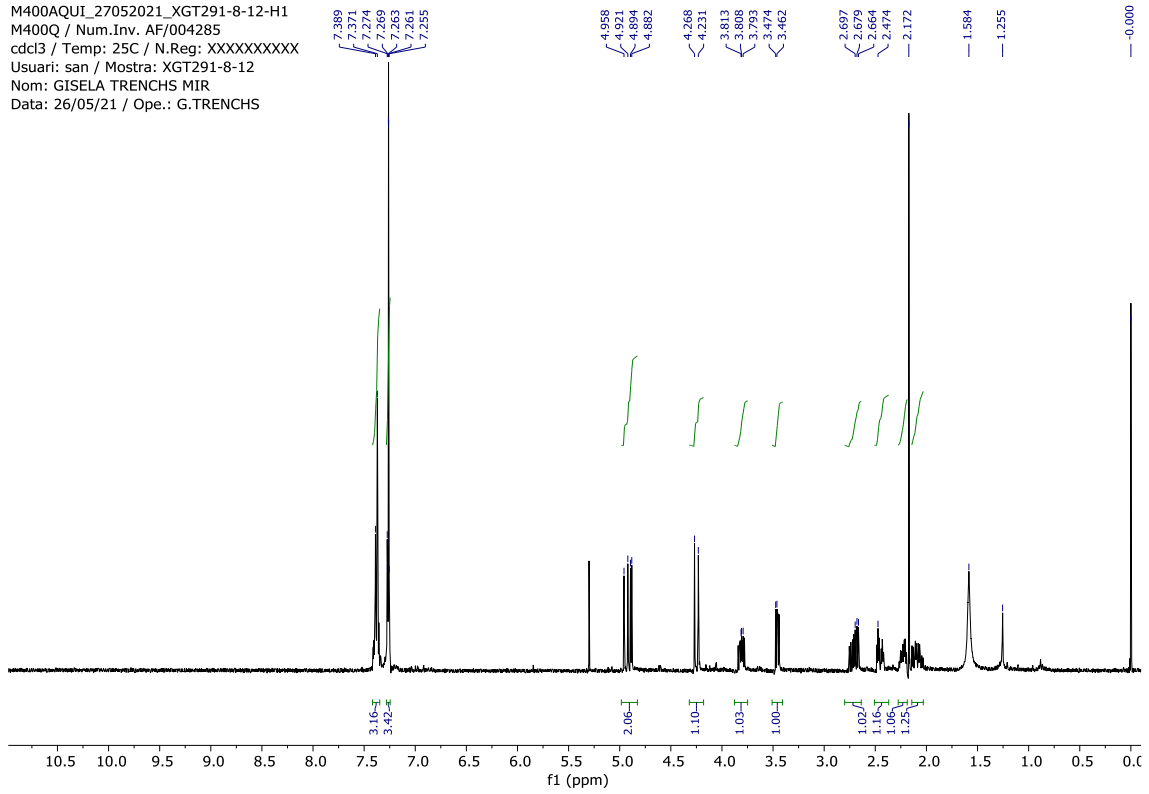


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 Nom: GISELA TRENCHS MIR  
 Data: 14/04/21 / Ope.: G.TRENCHS

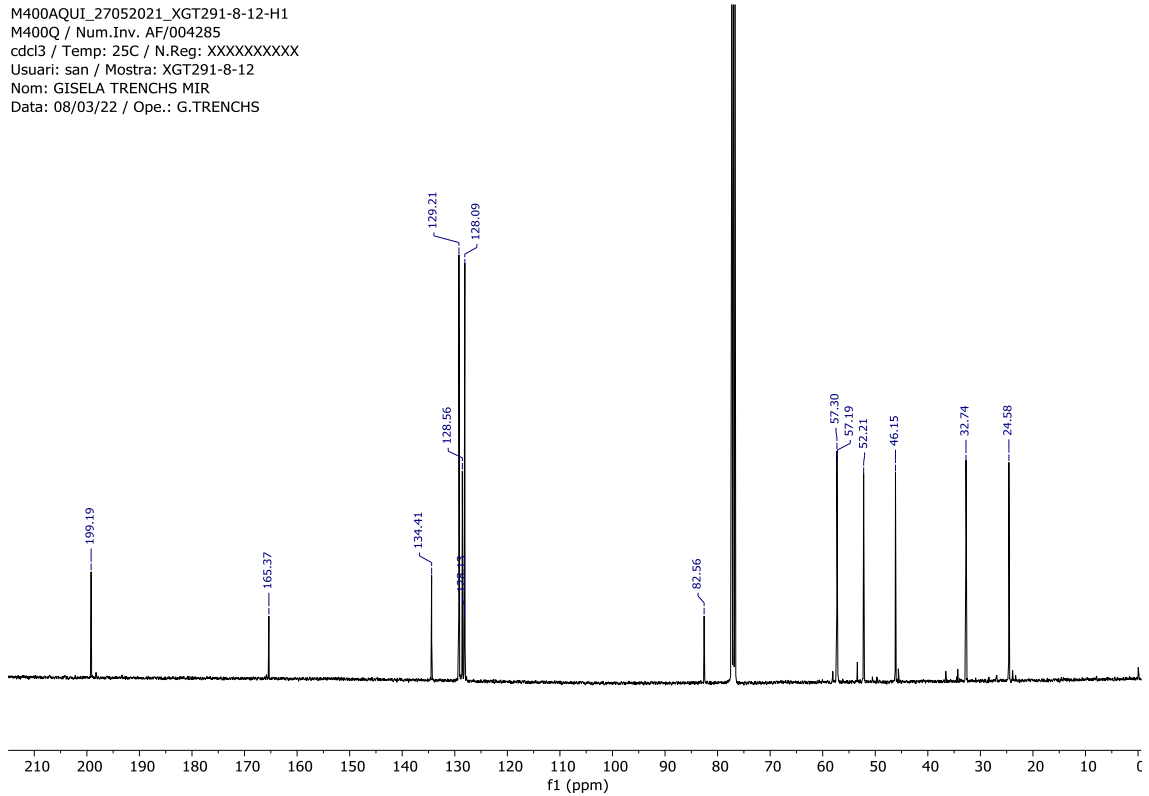


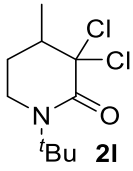


M400AQUI\_27052021\_XGT291-8-12-H1  
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 Nom: GISELA TRENCHS MIR  
 Data: 26/05/21 / Ope.: G.TRENCHS

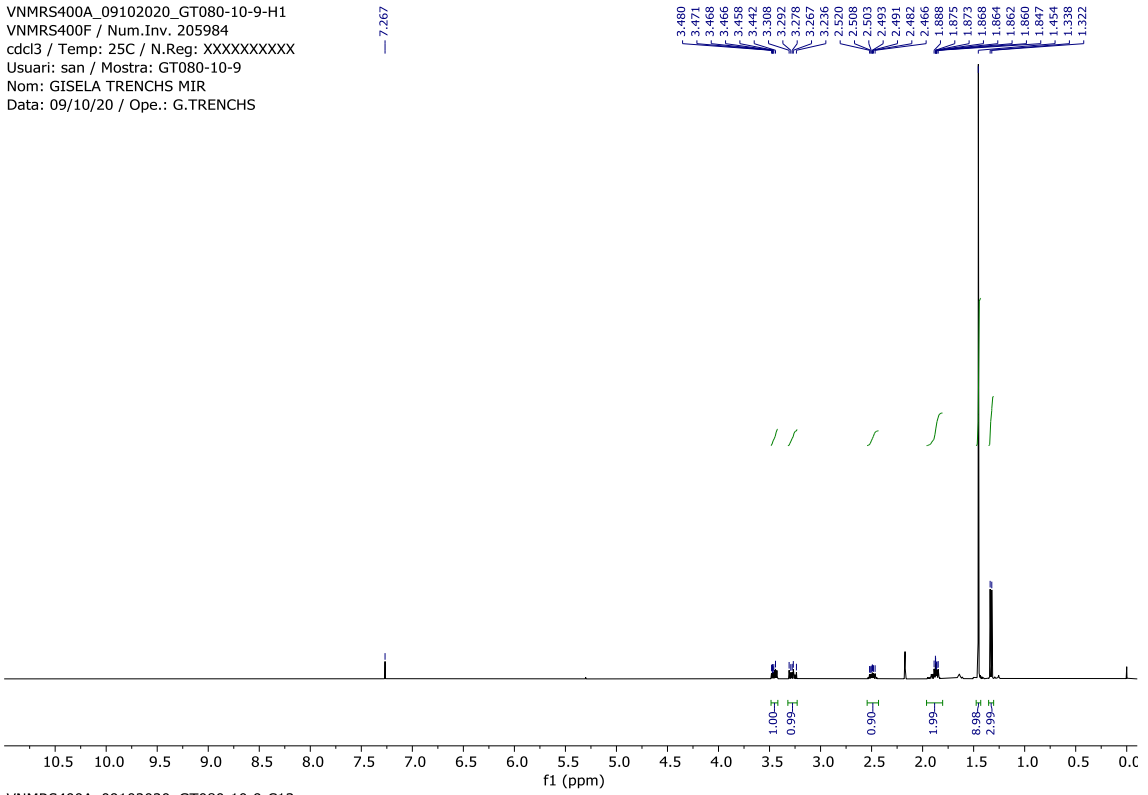


M400AQUI\_27052021\_XGT291-8-12-H1  
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 Nom: GISELA TRENCHS MIR  
 Data: 08/03/22 / Ope.: G.TRENCHS

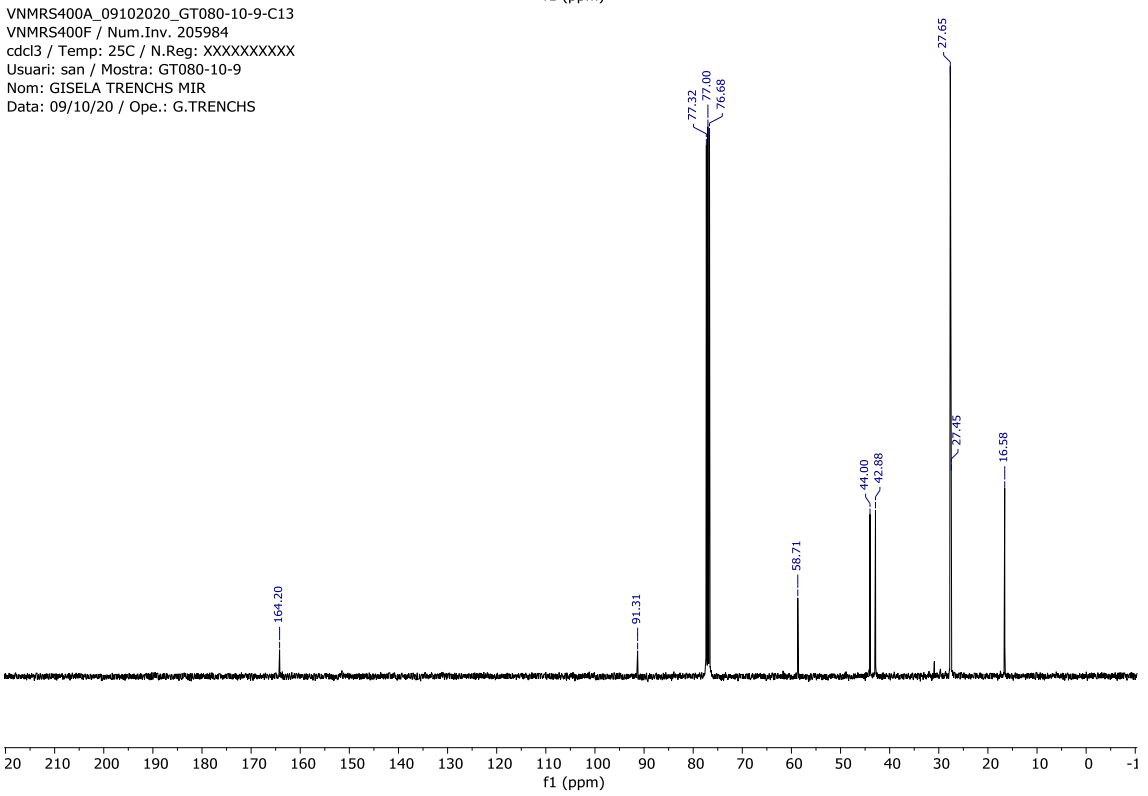


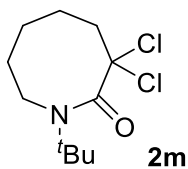


VNMRS400A\_09102020\_GT080-10-9-H1  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Nom: GISELA TRENCHS MIR  
 Data: 09/10/20 / Ope.: G.TRENCHS

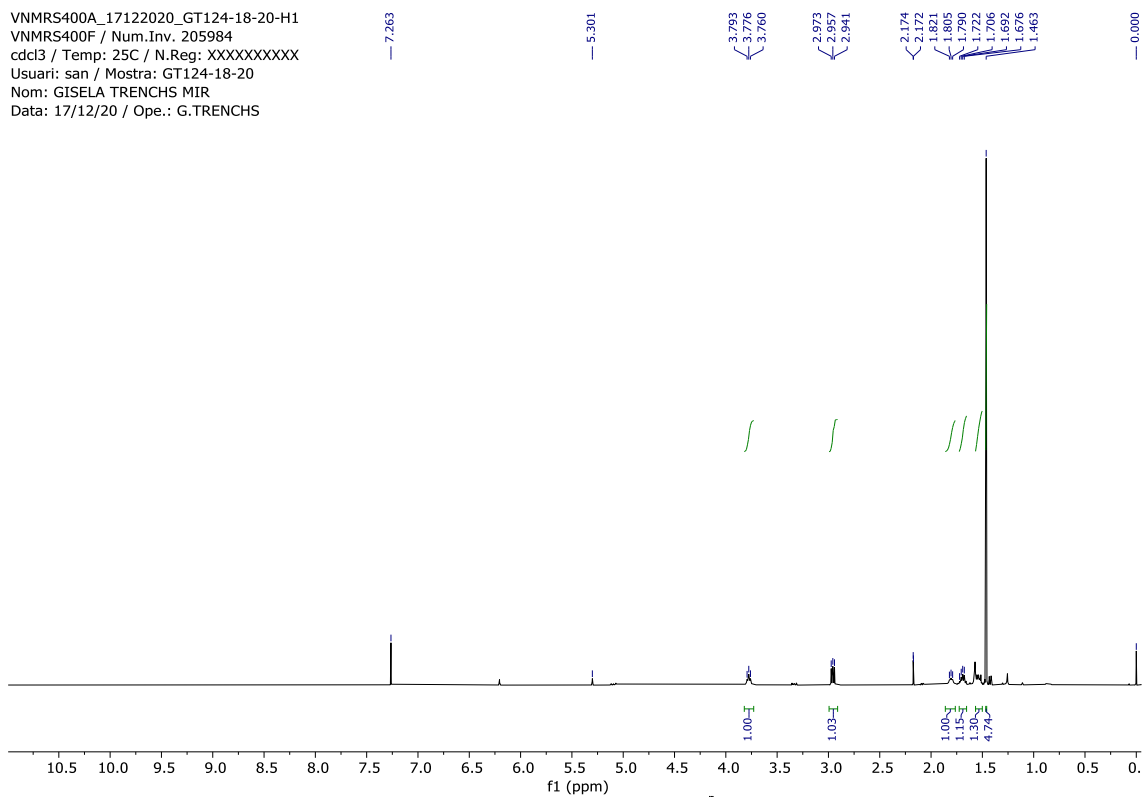


VNMRS400A\_09102020\_GT080-10-9-C13  
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 Nom: GISELA TRENCHS MIR  
 Data: 09/10/20 / Ope.: G.TRENCHS

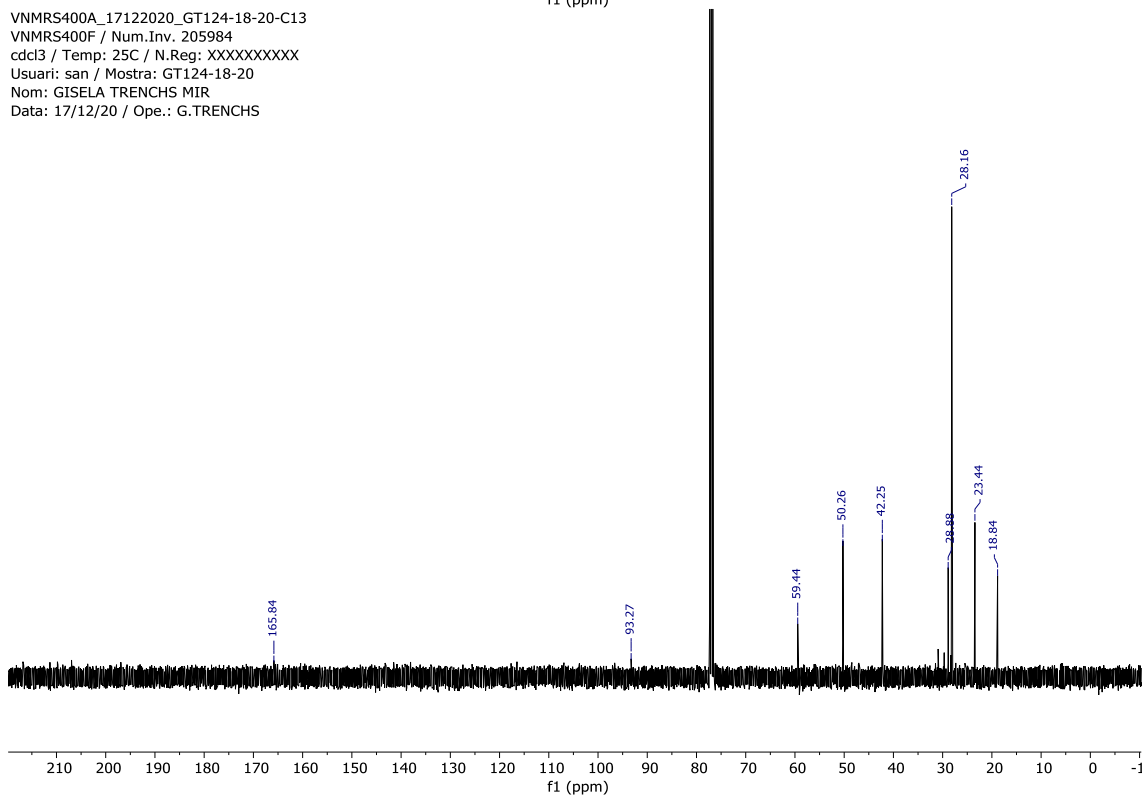


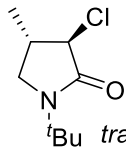


VNMRS400A\_17122020\_GT124-18-20-H1  
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 Usuari: san / Mostra: GT124-18-20  
 Nom: GISELA TRENCHS MIR  
 Data: 17/12/20 / Ope.: G.TRENCHS



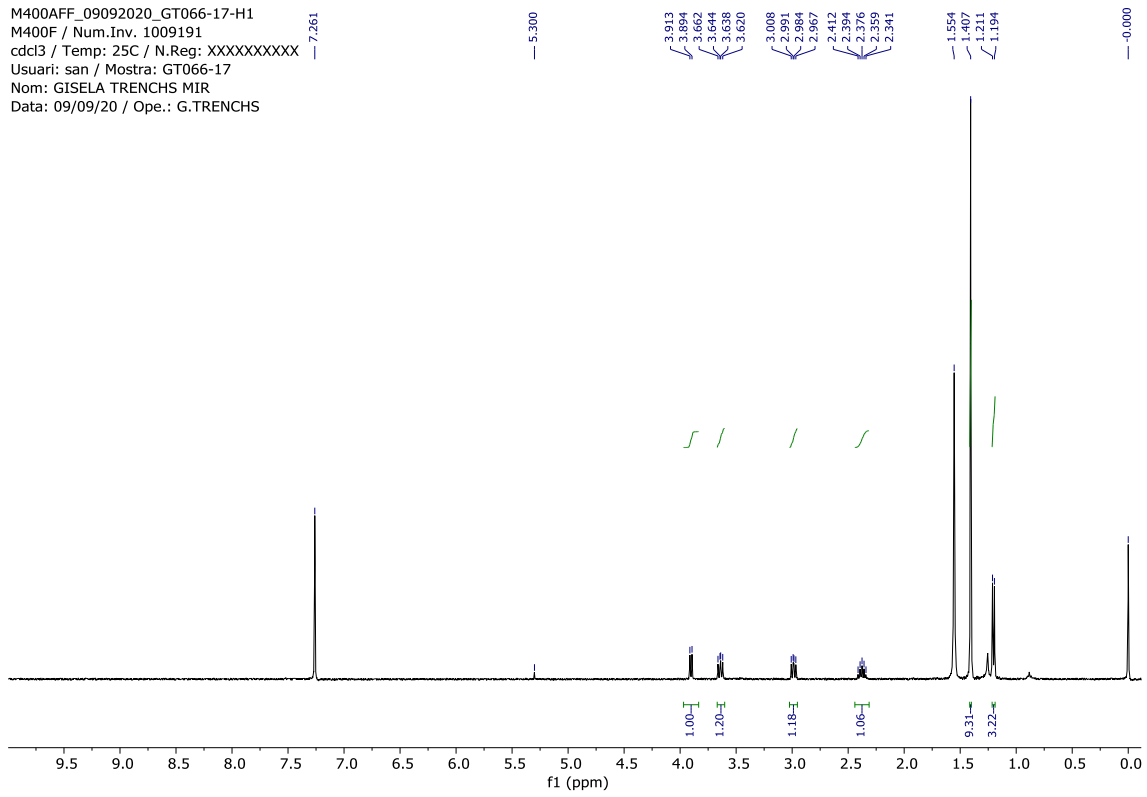
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Data: 17/12/20 / Ope.: G.TRENCHS



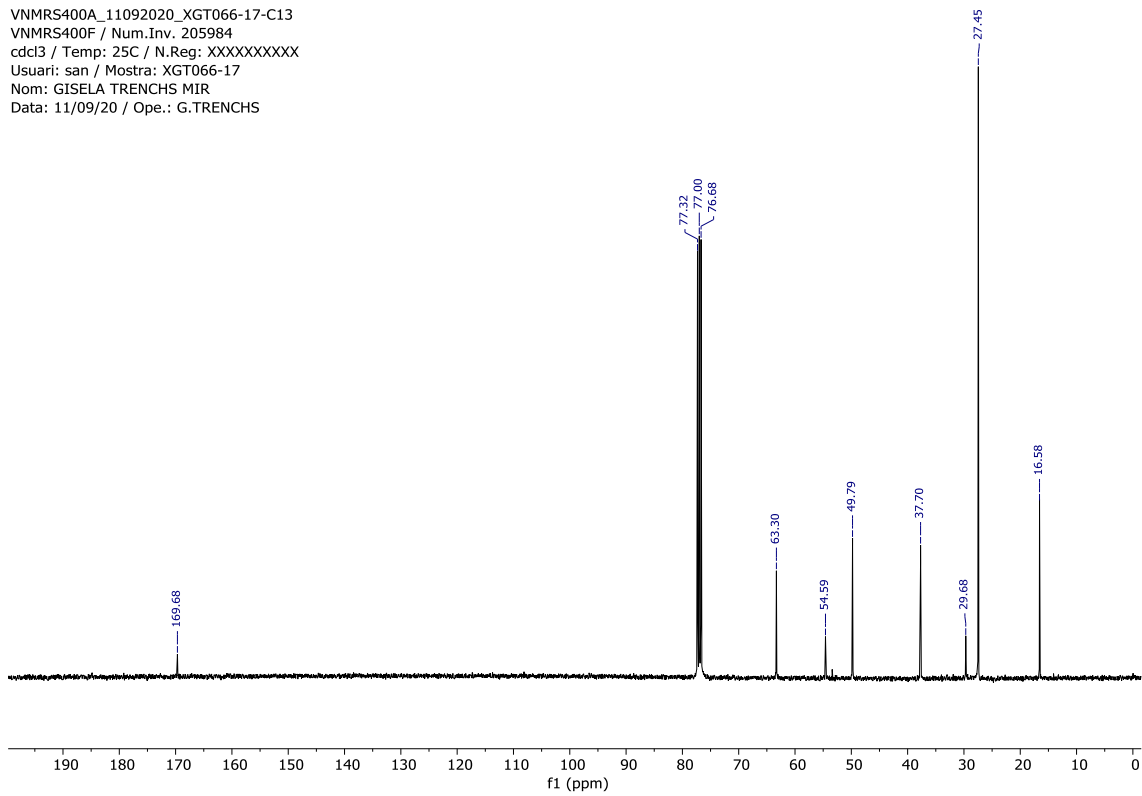


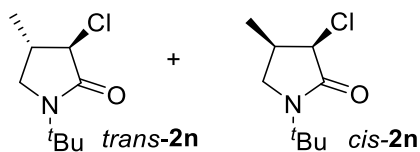
**trans-2n, major diastereomer**

M400AFF\_09092020\_GT066-17-H1  
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 Usuari: san / Mostra: GT066-17  
 Nom: GISELA TRENCHS MIR  
 Data: 09/09/20 / Ope.: G.TRENCHS

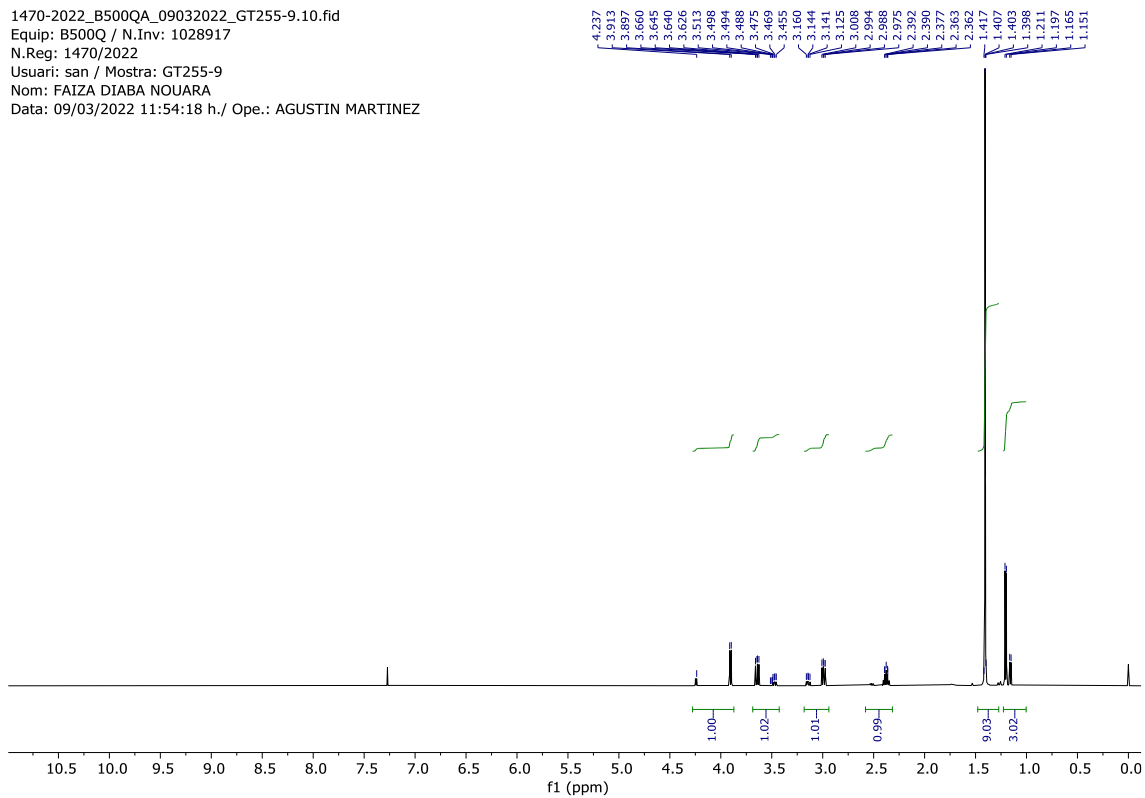


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 Nom: GISELA TRENCHS MIR  
 Data: 11/09/20 / Ope.: G.TRENCHS

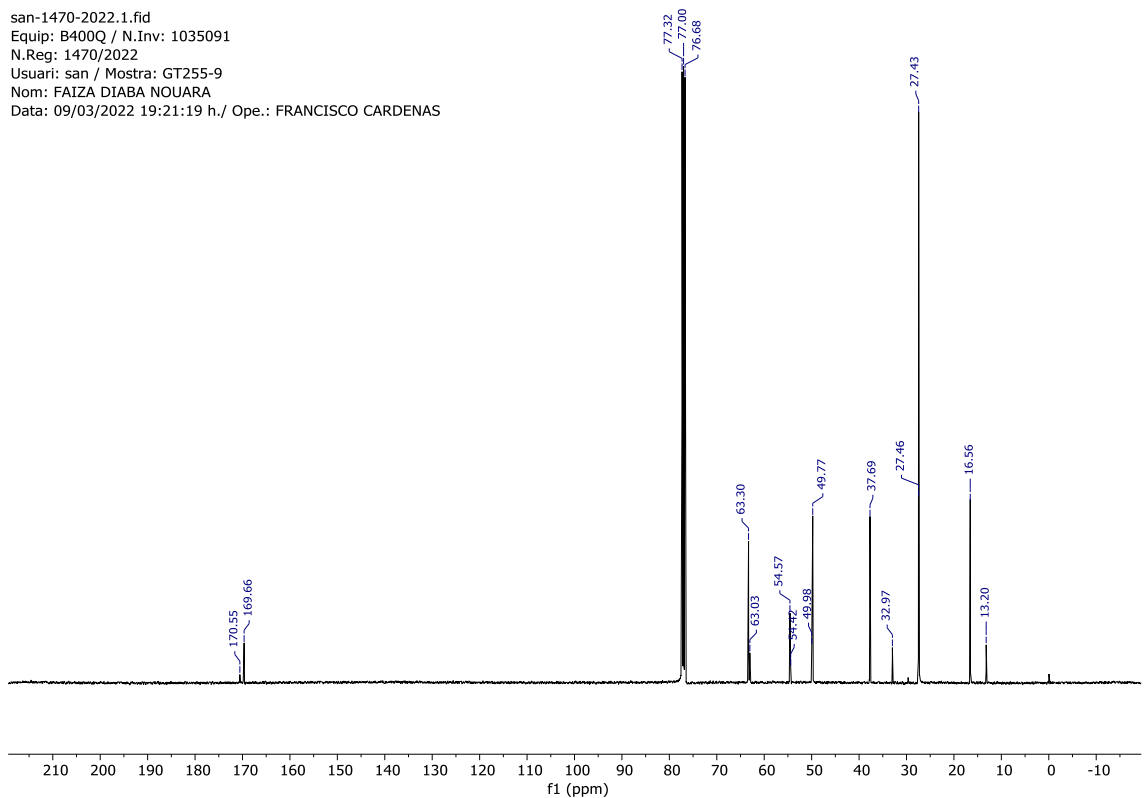




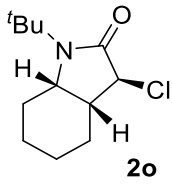
1470-2022\_B500QA\_09032022\_GT255-9.10.fid  
 Equip: B500Q / N.Inv: 1028917  
 N.Reg: 1470/2022  
 Usuari: san / Mostra: GT255-9  
 Nom: FAIZA DIABA NOUARA  
 Data: 09/03/2022 11:54:18 h./ Ope.: AGUSTIN MARTINEZ



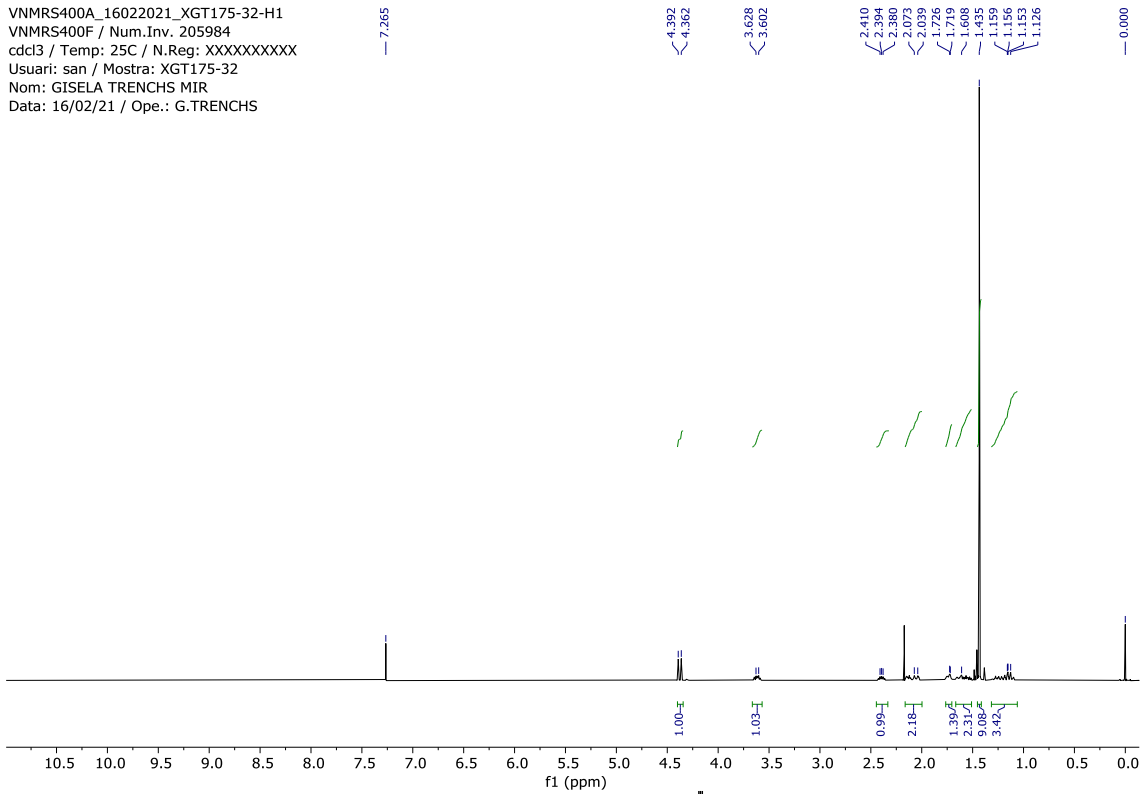
san-1470-2022.1.fid  
 Equip: B400Q / N.Inv: 1035091  
 N.Reg: 1470/2022  
 Usuari: san / Mostra: GT255-9  
 Nom: FAIZA DIABA NOUARA  
 Data: 09/03/2022 19:21:19 h./ Ope.: FRANCISCO CARDENAS



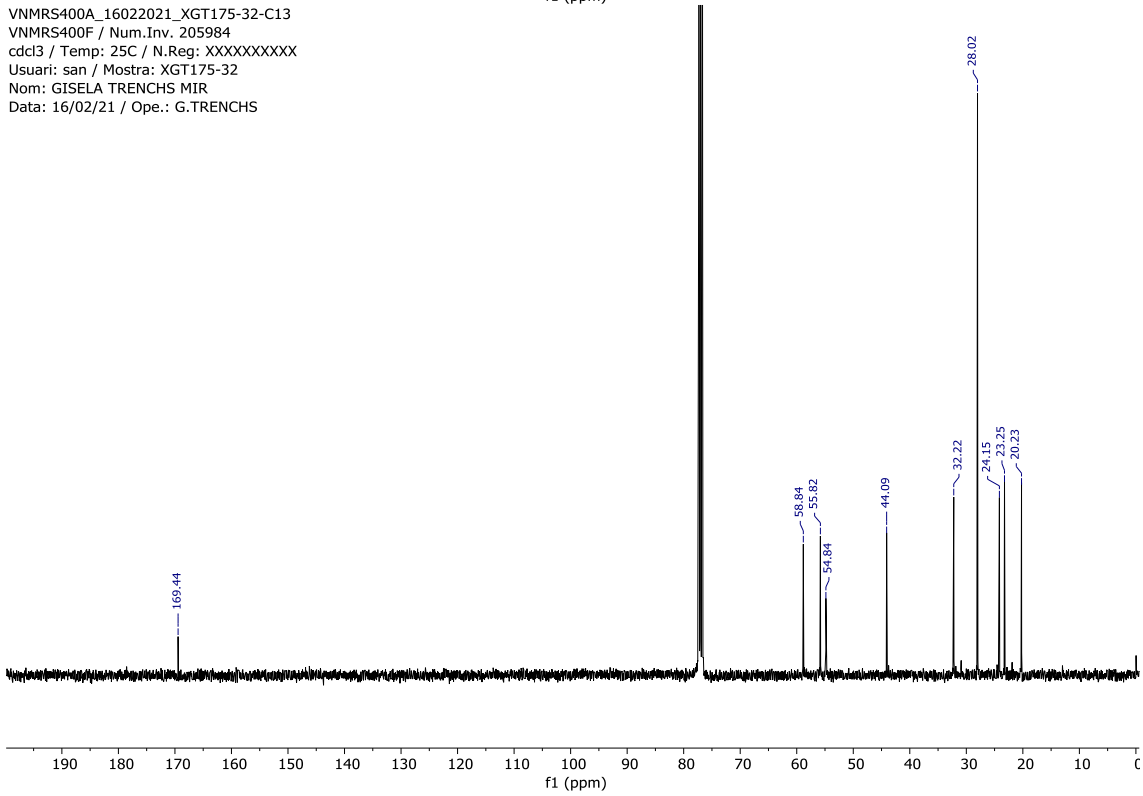


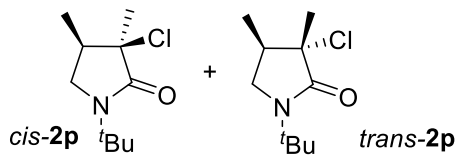


VNMRS400A\_16022021\_XGT175-32-H1  
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 Usuari: san / Mostra: XGT175-32  
 Nom: GISELA TRENCHS MIR  
 Data: 16/02/21 / Ope.: G.TRENCHS

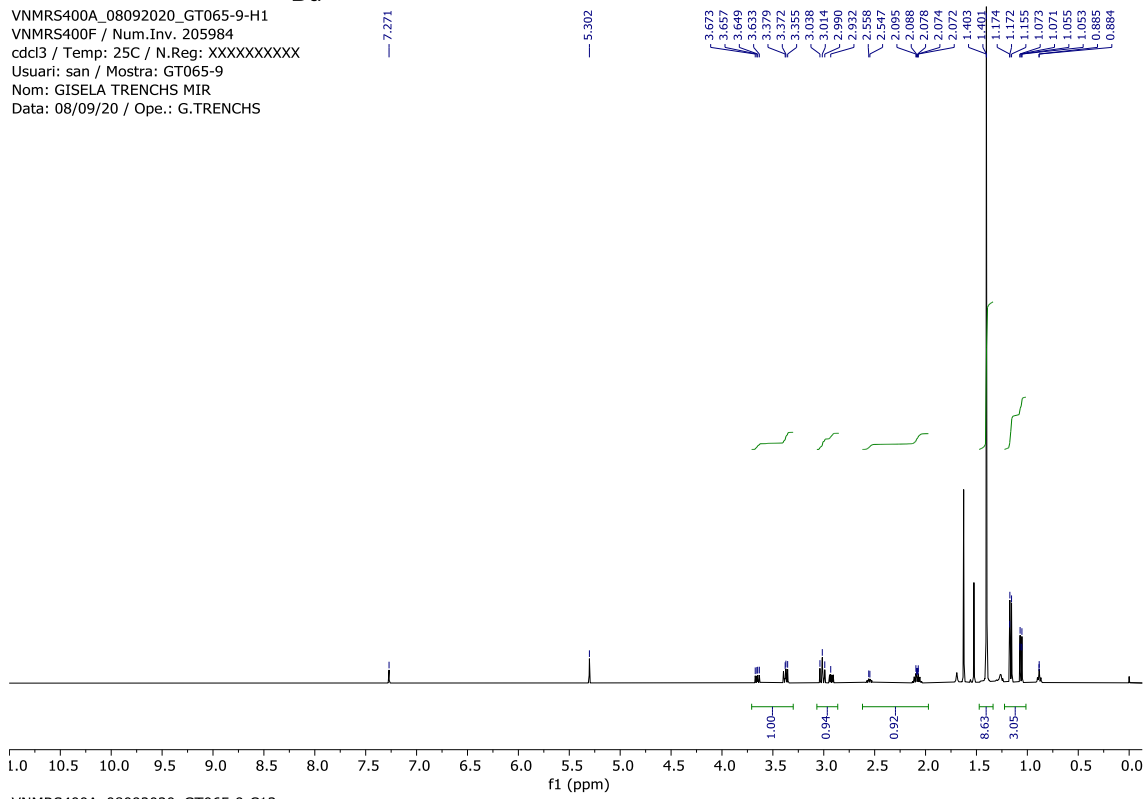


VNMRS400A\_16022021\_XGT175-32-C13  
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 cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX  
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 Nom: GISELA TRENCHS MIR  
 Data: 16/02/21 / Ope.: G.TRENCHS

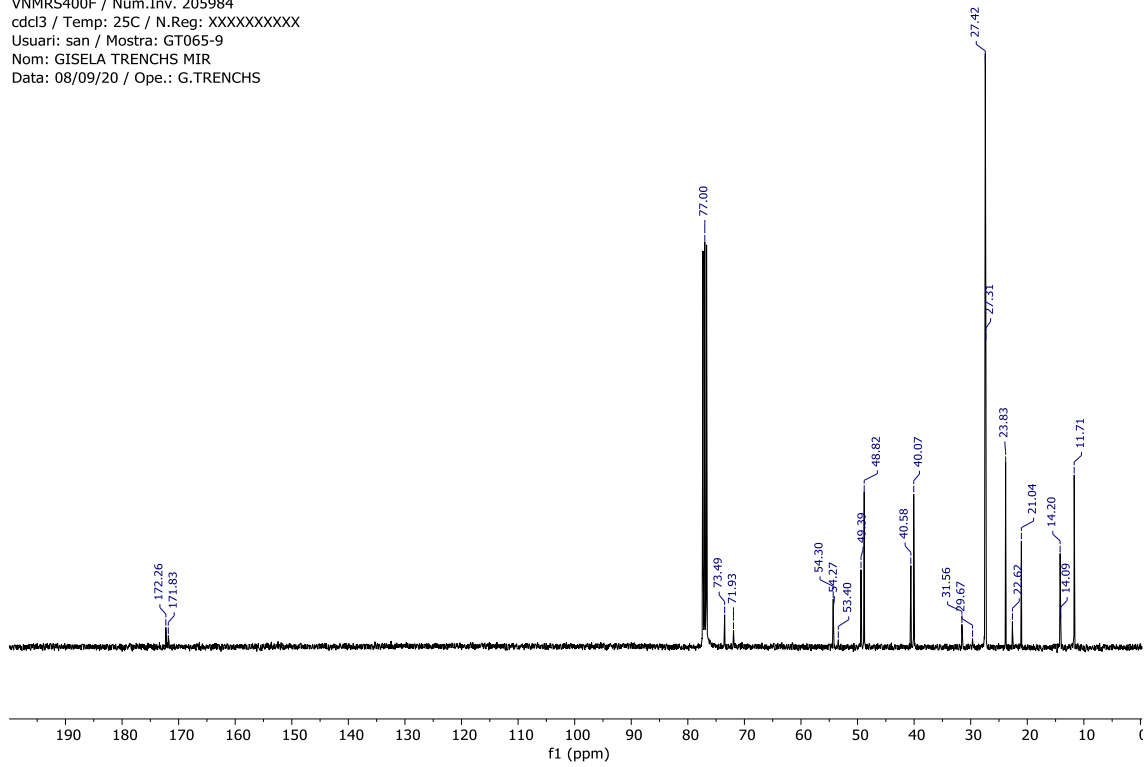


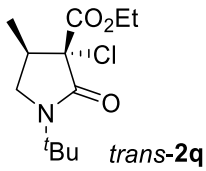


VNMRS400A\_08092020\_GT065-9-H1  
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 Data: 08/09/20 / Ope.: G.TRENCHS

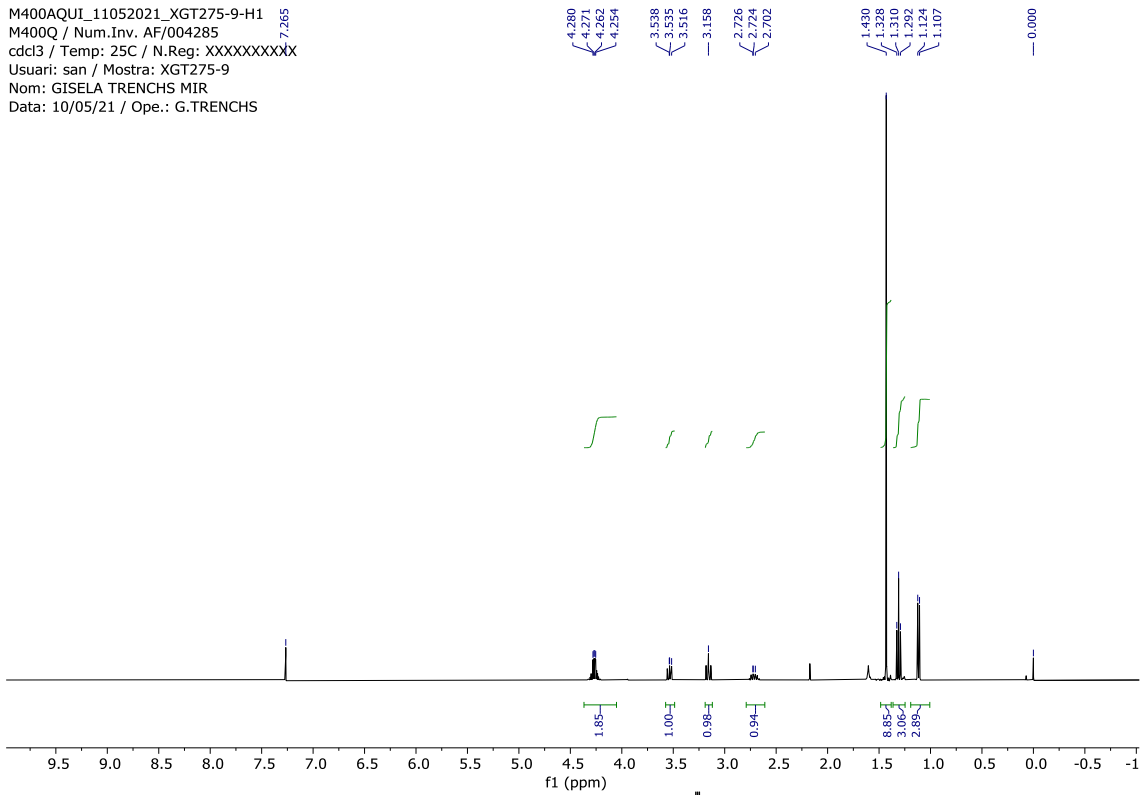


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 Data: 08/09/20 / Ope.: G.TRENCHS

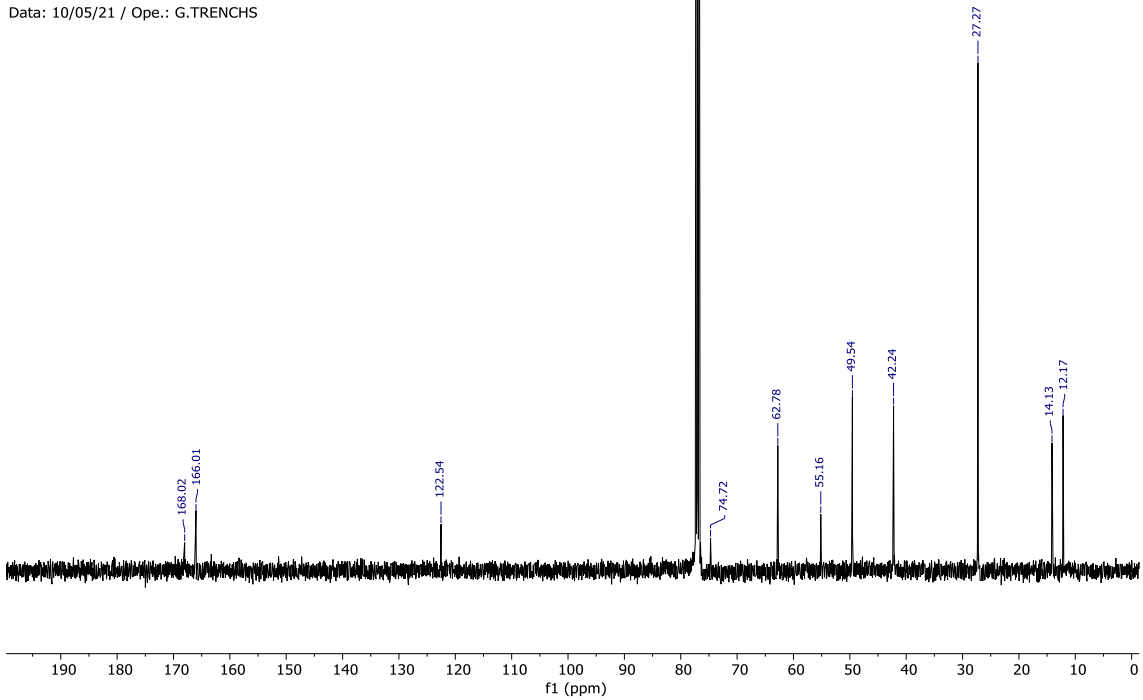


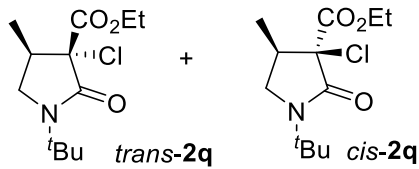


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 Data: 10/05/21 / Ope.: G.TRENCHS

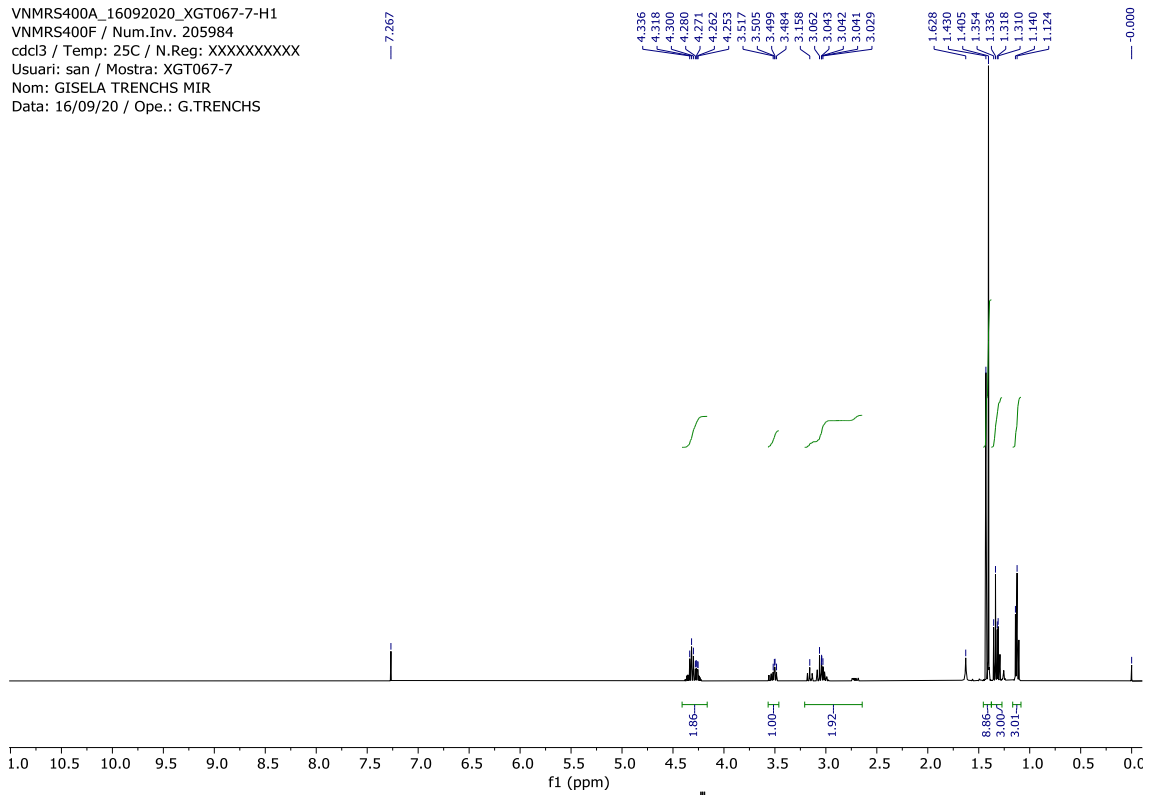


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 Data: 10/05/21 / Ope.: G.TRENCHS

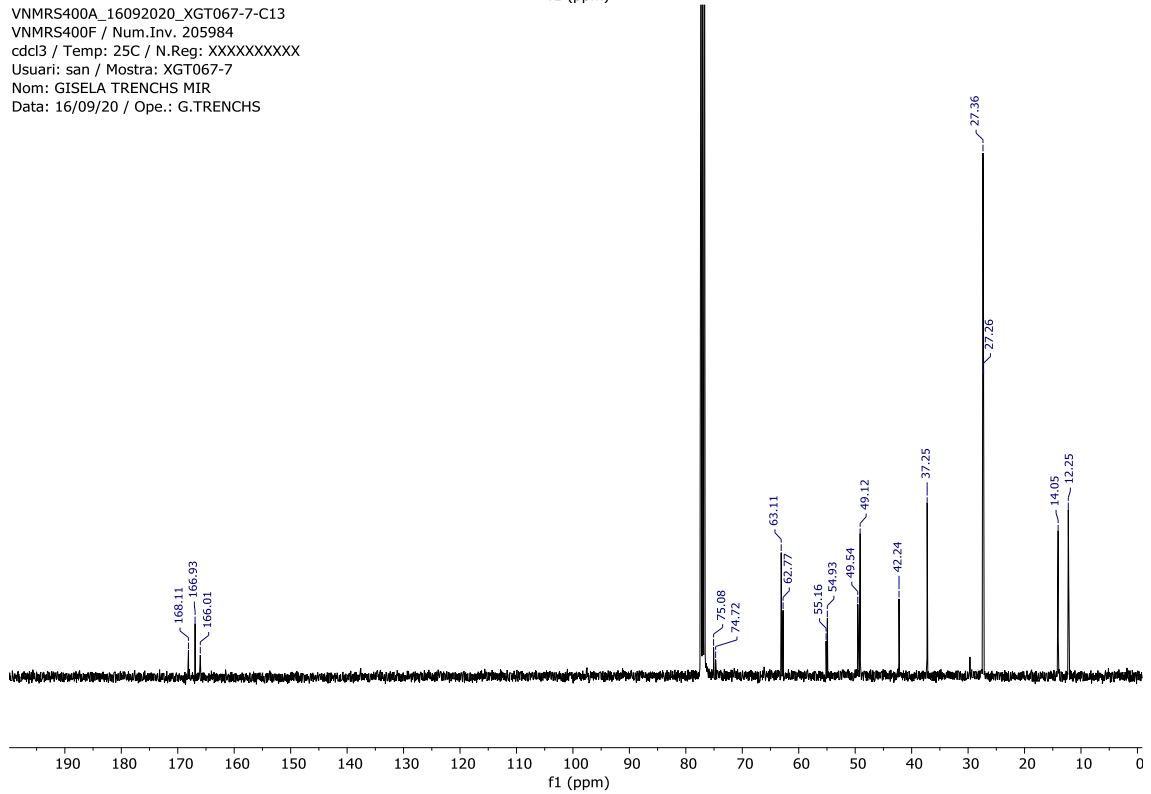


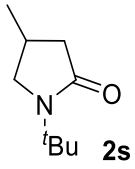


VNMR5400A\_16092020\_XGT067-7-H1  
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 Data: 16/09/20 / Ope.: G.TRENCHS

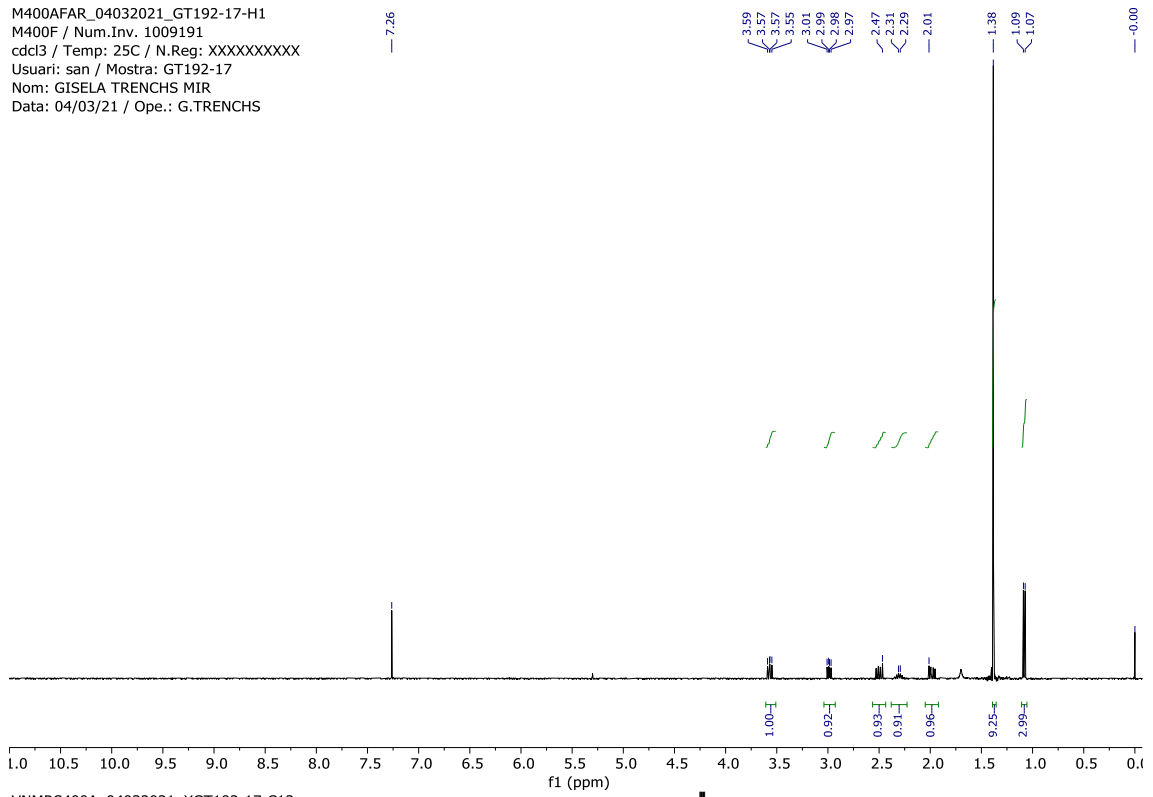


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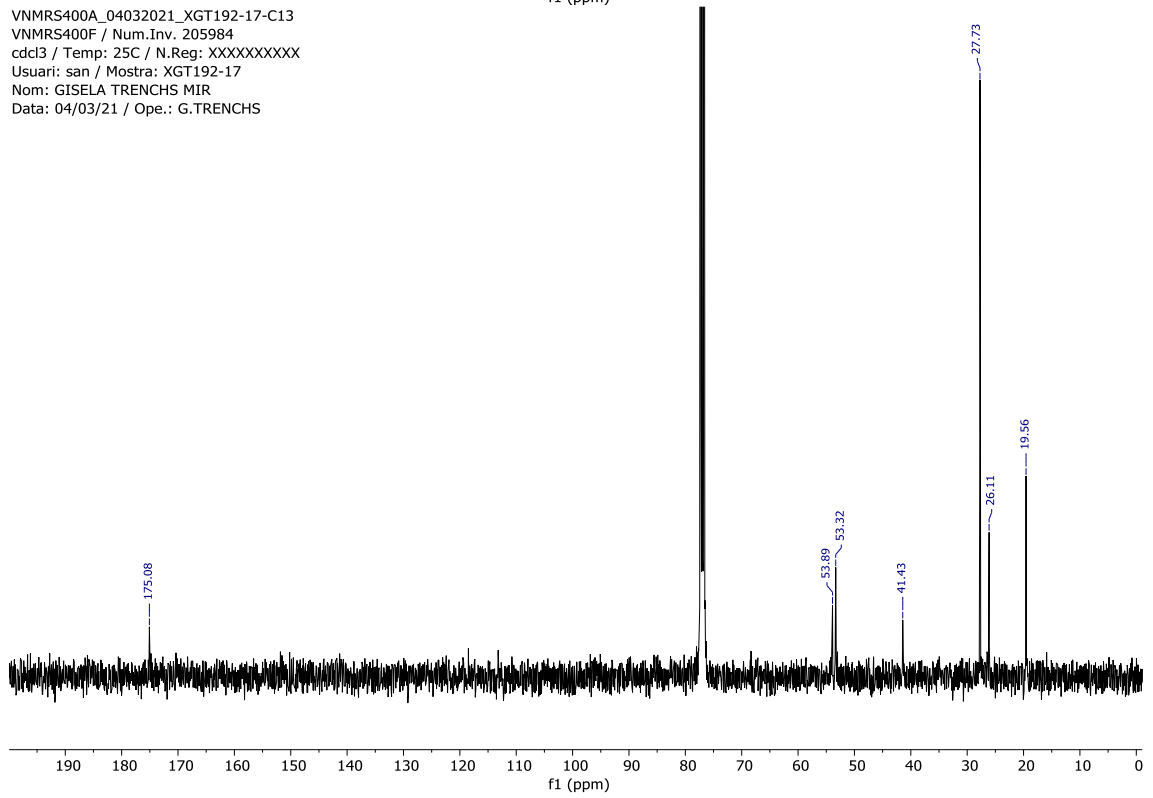


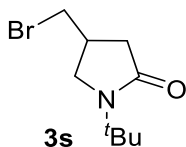


M400AFAR\_04032021\_GT192-17-H1  
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 Data: 04/03/21 / Ope.: G.TRENCHS

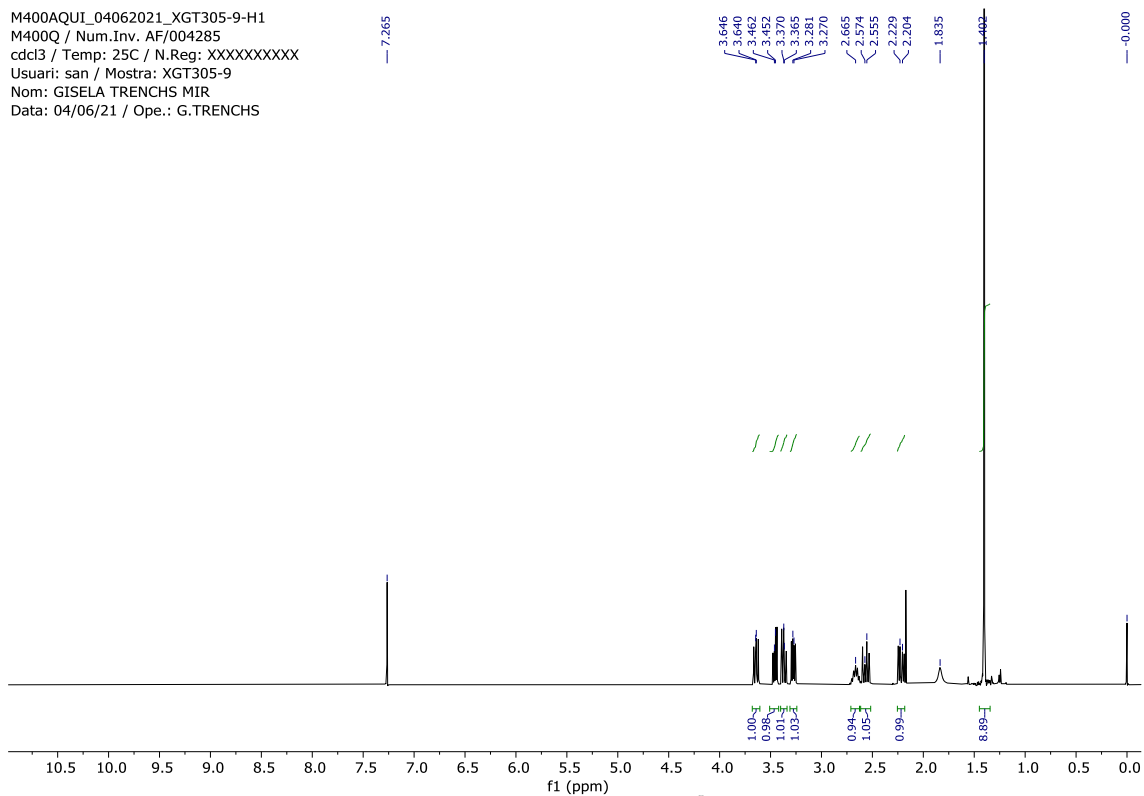


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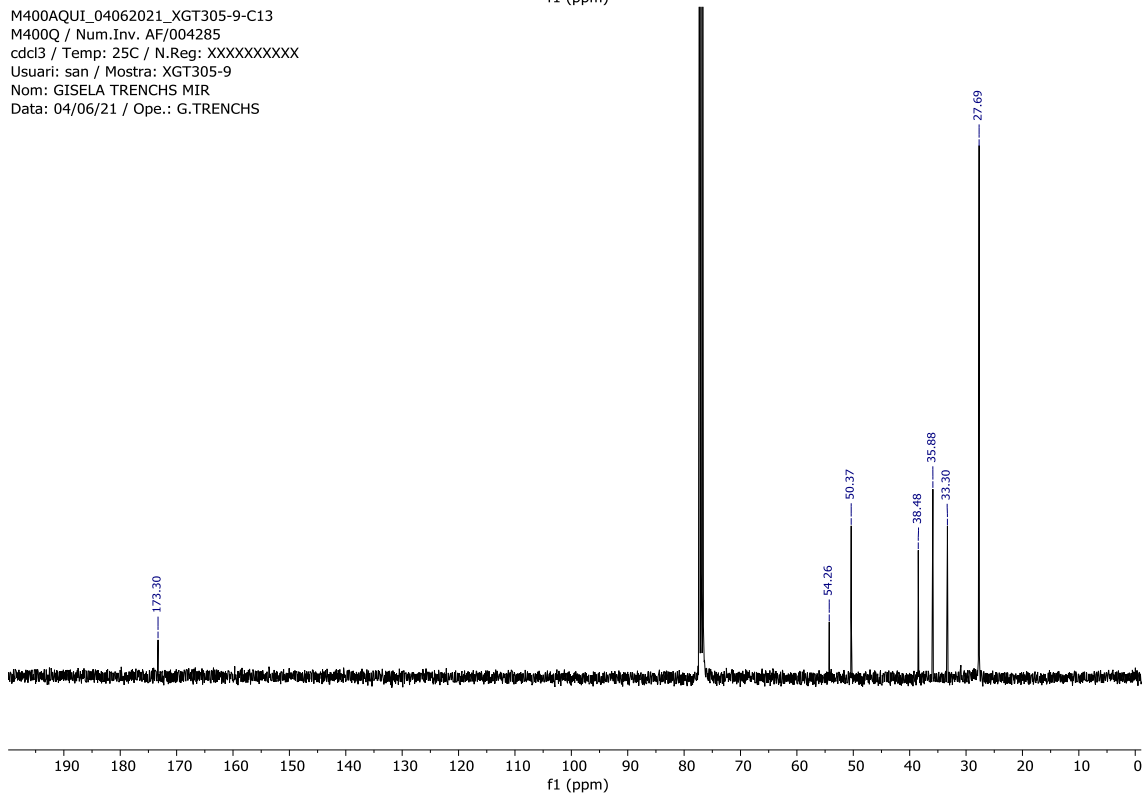


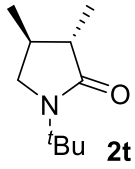


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Nom: GISELA TRENCHS MIR  
Data: 04/06/21 / Ope.: G.TRENCHS

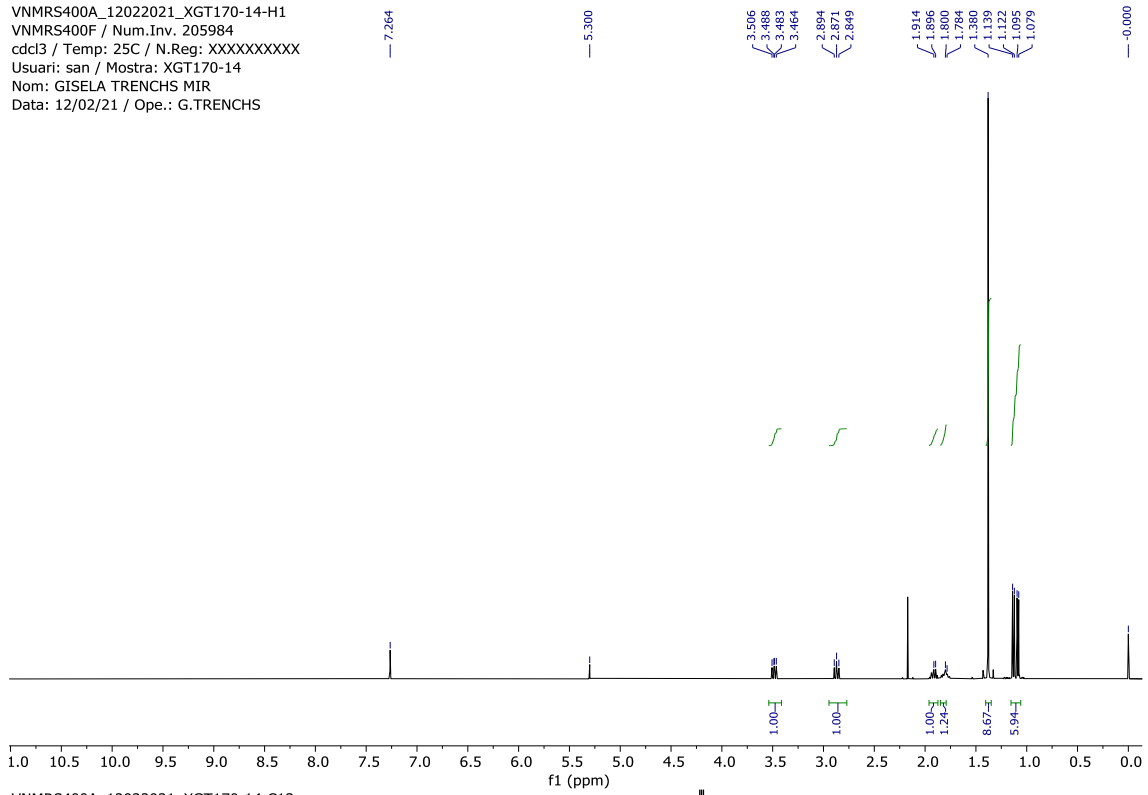


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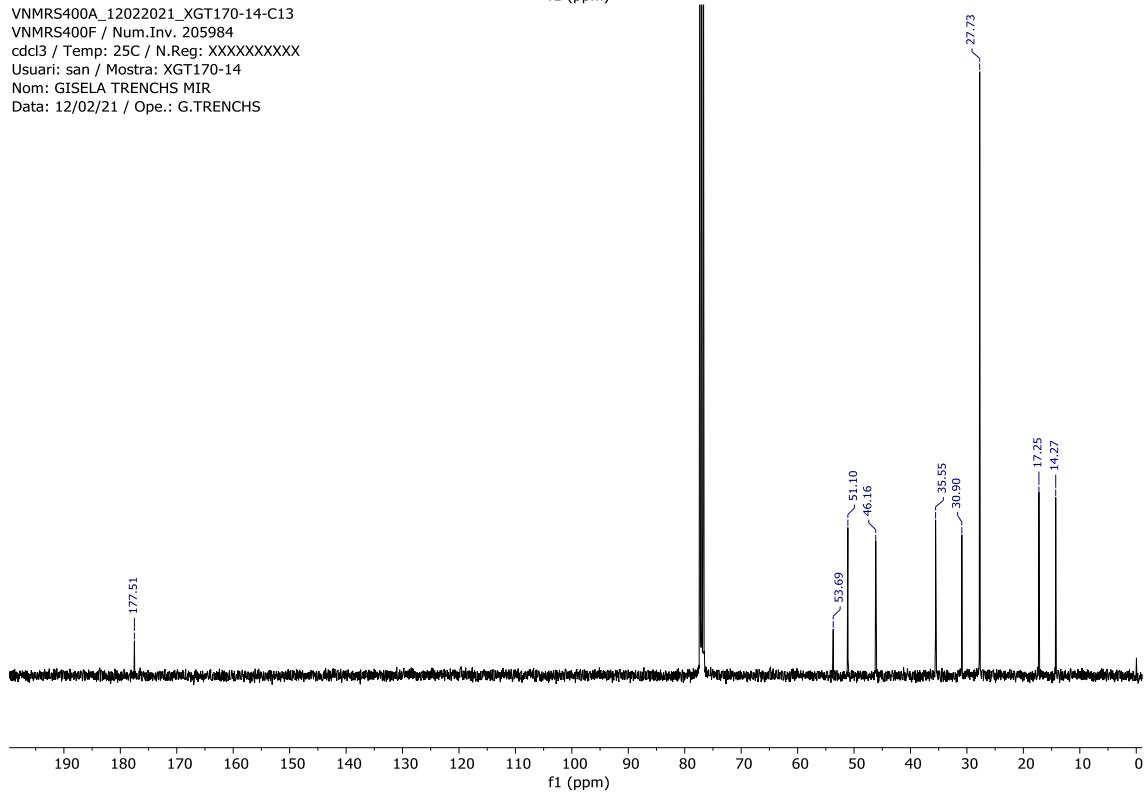


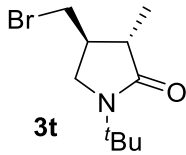


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 Data: 12/02/21 / Ope.: G.TRENCHS

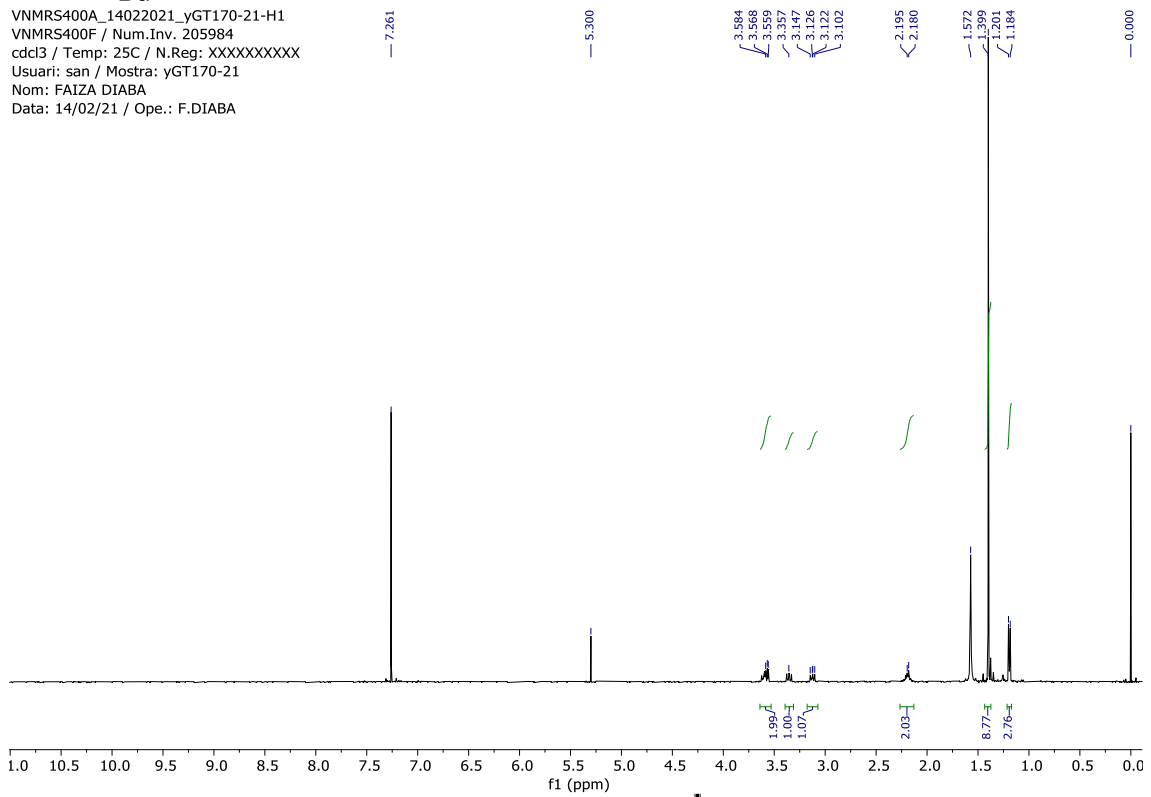


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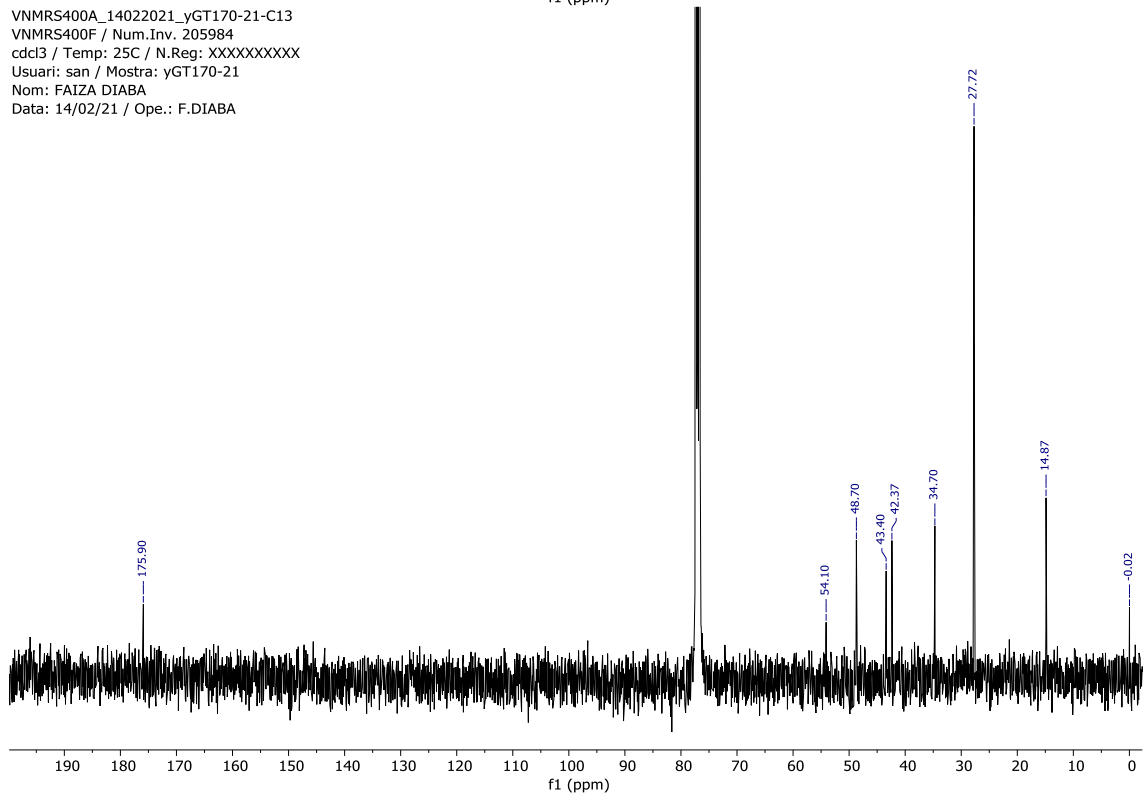




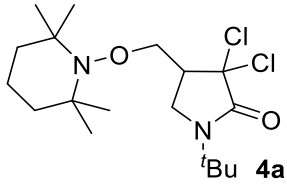
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 Nom: FAIZA DIABA  
 Data: 14/02/21 / Ope.: F.DIABA



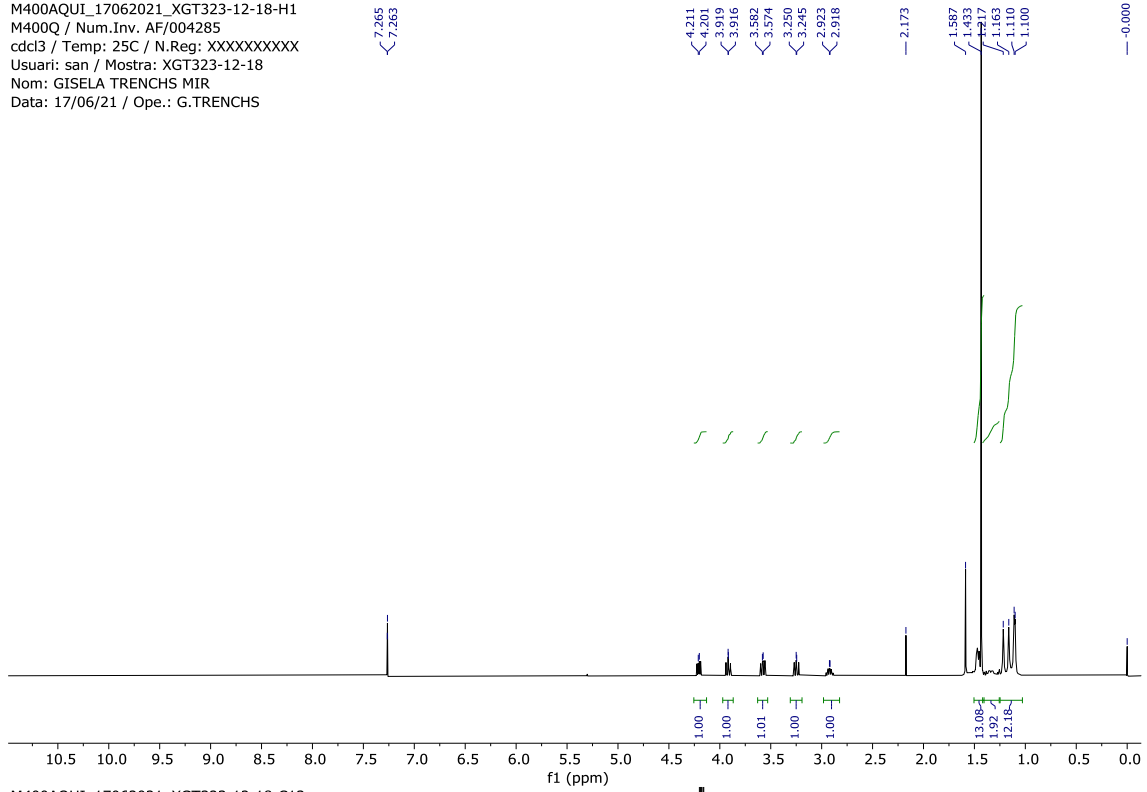
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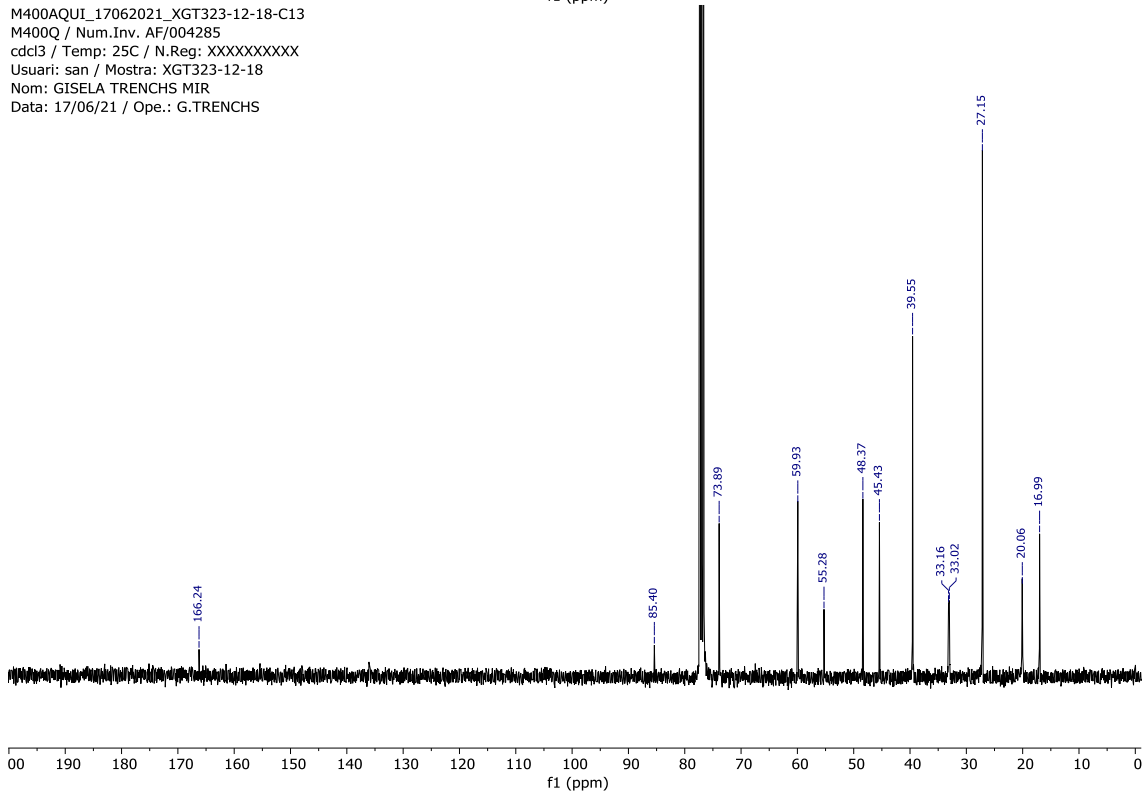


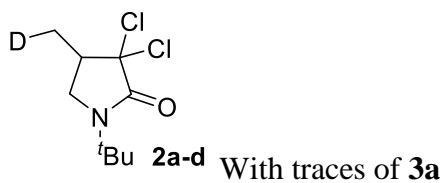


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 Nom: GISELA TRENCHS MIR  
 Data: 17/06/21 / Ope.: G.TRENCHS

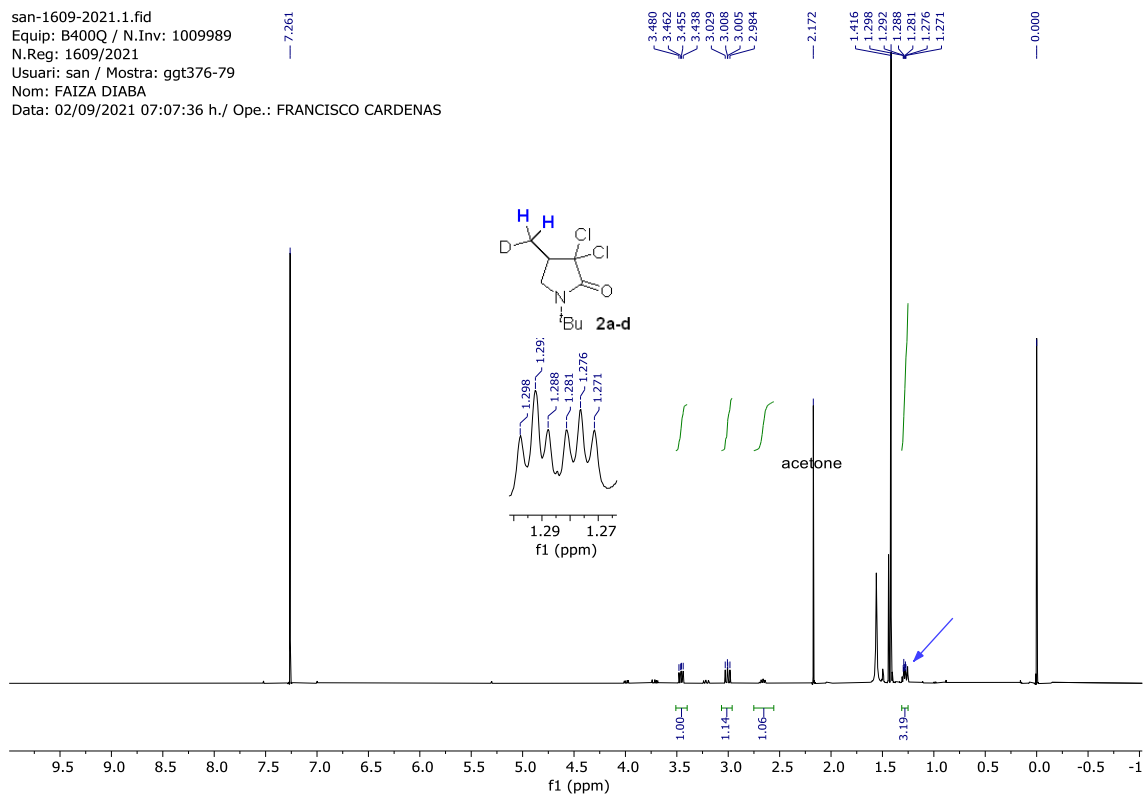


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 Nom: GISELA TRENCHS MIR  
 Data: 17/06/21 / Ope.: G.TRENCHS





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