Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Synthesis and reactivity of hydroindole enelactams leading to densely functionalized scaffolds

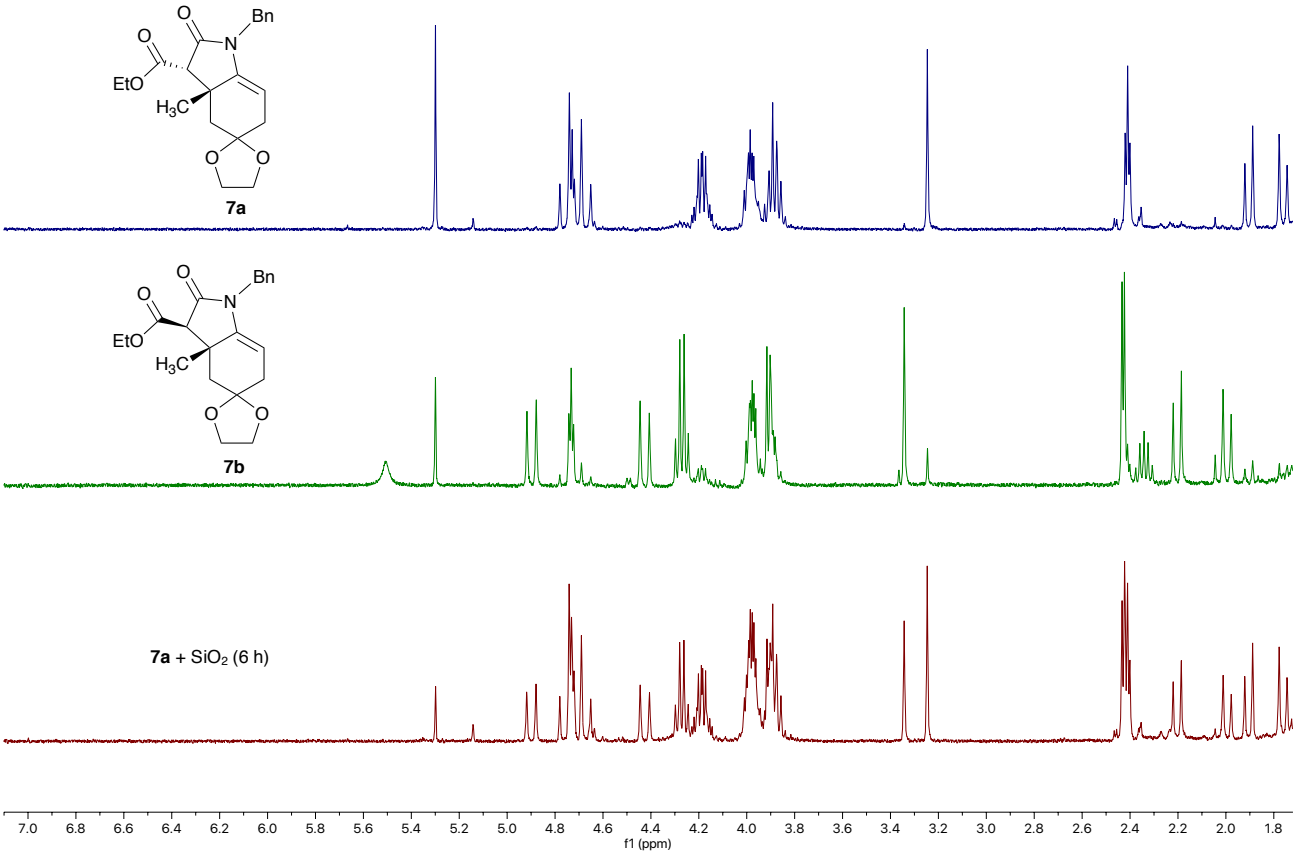
Clàudia Marquès, Faïza Diaba,* Jaume Roca, and Josep Bonjoch*

Laboratori de Química Orgànica, Facultat de Farmàcia, IBUB, Universitat de Barcelona, Av. Joan XXIII s/n, 08028-Barcelona, Spain

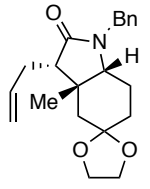
faiza.diaba@ub.edu, josep.bonjoch@ub.edu

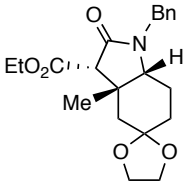
Contents

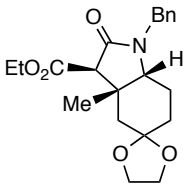
1. Partial epimerization of ester 7a to 7b: NMR Spectra

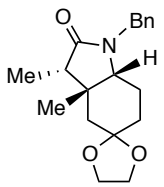


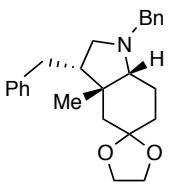
2. Table S-1. ^{13}C NMR data of Octahydroindoles: Lactams and Amines[a,b]

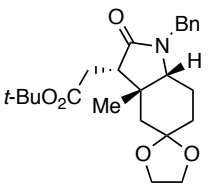

c-4a

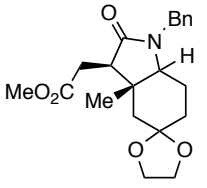

c-18a

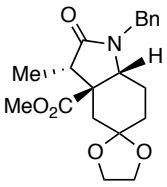

c-18b

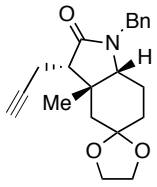

c-20a

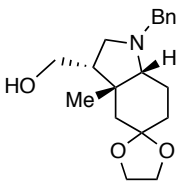

c-21a

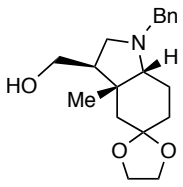

c-22a

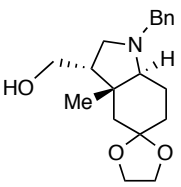

c-22b (7a-Hβ)
t-22b (7a-Hα)

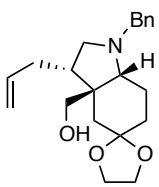

c-23a

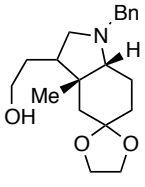

c-24a


c-26a


c-26b


t-26a


c-27a


c-28a (3-Hβ)
c-28b (3-Hα)

	c-4a	c-18a	c-18b	c-20a	c-21a	c-22a	c-22b	t-22b	c-23a	c-24a	c-26a	c-26b	t-26a	c-27a	c-28a	c-28b
C-2	177.1	167.6	168.5	177.8	176.9	176.1	174.4	176.1	173.5	175.5	55.1	55.7	56.5	57.0	57.1	58.2
C-3	55.4	61.0	60.8	50.4	53.7	52.2	45.5	52.2	46.5	53.8	51.0	48.4	48.4	44.8	45.8	41.4
C-3a	41.8	42.0	..41.2	41.1	41.8	41.1	40.4	43.4	51.2	41.6	43.0	43.3	43.2	47.9	43.6	43.1
C-4	36.9	37.0	42.0	36.5	36.9	37.1	40.8	42.1	33.2	36.2	36.5	44.0	41.1	35.0	36.6	43.5
C-5	108.3	107.6	107.4	108.2	108.1	107.9	107.6	108.3	107.3	107.9	109.6	109.3	110.1	109.8	110.1	109.3
C-6	28.8	28.5	29.1	28.7	28.5	28.7	31.0	34.6	29.4	28.6	28.6	29.0	34.3	28.5	28.8	29.6
C-7	20.6	20.7	21.4	20.6	20.3	20.4	24.3	20.4	21.4	20.2	21.4	21.6	21.7	23.0	21.4	22.5
C-7a	59.5	59.5	59.0	59.5	59.2	59.3	60.4	64.1	54.5	59.3	68.5	66.7	68.6	69.6	68.5	67.5
Me	23.2	23.2	20.3	21.9	22.7	22.4	23.5	13.8	---	22.9	23.9	19.9	22.6	---	23.2	---
NCH_2	44.1	44.1	44.1	43.8	44.0	44.0	44.5	44.6	44.1	44.0	57.9	57.7	58.2	57.9	58.3	58.0

[a] All NMR spectroscopic data assignments are supported by gCOSY and gHSQC experiments. 1D and 2D NMR spectra were recorded on a Varian Mercury 400 or Varian VNMRS 400 spectrometer in $CDCl_3$. [b] References for previously reported compounds: **c-4a** (G. Coussanes and J. Bonjoch, *Org. Lett.* 2017, **19**, 878–881); **c-22b**, **t-22b** and **c-28b** (A. Cordero-Vargas, B. Bradshaw and J. Bonjoch, *Tetrahedron* **2008**, *64*, 8134–8140); **c-28a** (S. Jansana, G. Coussanes, J. Puig, F. Diaba and J. Bonjoch, *Helv. Chim. Acta* **2019**, *102*, e1900188).

3. Experimental section

General Methods. All reactions were carried out under an argon atmosphere with dry solvents under anhydrous conditions. All product mixtures were analyzed by thin-layer chromatography using TLC silica gel plates with a fluorescent indicator. Analytical thin-layer chromatography was performed on SiO_2 (Merck silica gel 60 F_{254}), and the spots were located by UV light (λ 254 nm) and/or a 1% $KMnO_4$ aqueous solution or hexachloroplatinate reagent. Chromatography was carried out on SiO_2 (Carlo Erba silica gel 60A, 35–70 μ), unless otherwise noted. Drying of organic extracts during the reaction workup was performed over anhydrous Na_2SO_4. Solvent evaporation was accomplished with a rotatory evaporator. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ solution. Chemical shifts of ^1H and ^{13}C NMR spectra are reported in ppm downfield (δ) from Me_4Si. All NMR spectroscopic data assignments are supported by gCOSY and gHSQC experiments. 1D and 2D NMR spectra were recorded on a Varian Mercury 400 or Varian VNMRS 400 spectrometer in $CDCl_3$. The signal due to residual $CHCl_3$ appearing at δ_H 7.26 and the central resonance of the $CDCl_3$ triplet at δ_C 77.0 were used to reference ^1H and ^{13}C NMR spectra, respectively. Infrared spectra were recorded on a Nicolet 320 FT-IR spectrophotometer. The high-resolution ESI mass spectra were obtained on an Agilent LC/MSD-TOF mass spectrometer.

1-Benzyl-3a-methyl-1,3a,4,6-tetrahydro-2*H*-indole-2,5(3*H*)-dione monoethylene acetal (**3**). 2-Methylcyclohexane-1,4-dione monoethylene acetal (**1**) was prepared on a 30 mmol scale according to Danishefsky's protocol[4] (LHMDS, MeI, THF) to achieve 3.7 g of the methylated ketone **1**. Spectroscopic properties matched those previously described.[5] Lactam **3** was prepared from trichloroacetamide **2** (Scheme 3) on a 15 mmol scale according to the procedure previously reported,[1] obtaining 3.45 g (77%) of lactam **3**. ^1H NMR (400 MHz, $CDCl_3$): δ 7.34–7.18 (m, 5H, Ph), 4.70 (t, *J* = 4.2 Hz, 1H, H-7), 4.85 and 4.43 (2d, *J* = 15.4 Hz, 1H each, CH_2Ph), 4.01–3.95 and 3.93–3.84; (2 m, 2H each, OCH_2), 2.42 (d, *J* = 4.2 Hz, 2H, H-6), 2.38 (d, *J* = 4.4 Hz, 2H, H-3), 2.04 and 1.86 (2 d, *J* = 13.2 Hz, 1H each, H-4), 1.28 (s, 3H, Me); ^{13}C NMR (101 MHz, $CDCl_3$): δ 174.3 (C-2), 145.7 (C-7a), 136.5, 128.8, 127.5, and 127.4 (Ph), 108.9 (C-5), 95.1 (C-7), 64.6 and 63.9 (OCH_2), 47.1 (C-3), 43.7 (C-4), 43.6 (CH_2Ph), 37.5 (C-3a), 35.4 (C-6), 25.9 (Me). Spectral data are in accordance with published values.[1]

(3*RS*,3a*SR*)-3-Allyl-1-benzyl-3a-methyl-1,3a,4,6-tetrahydro-2*H*-indole-2,5(3*H*)-dione monoethylene acetal (4a). According to the general procedure, compound **4a** was synthesized from lactam **3** (55 mg, 0.18 mmol), LDA in THF (1 M, 0.24 mL), and allyl bromide (33 μL) in THF (0.6 mL). Purification by chromatography (SiO_2, hexane/EtOAc 9:1 $\rightarrow$ 4:1) gave **4a** (128 mg, 70%) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.30-7.20 (m, 5H, Ph), 5.90-5.80 (m, 1H, =CH), 5.10 (d, *J* = 18 Hz, 1H, $=CH_2$ cis), 5.06 (d, *J* = 10 Hz, 1H, $=CH_2$ trans), 4.70 (t, *J* = 3.8 Hz, 1H, H-7), 4.70 and 4.55 (2 dd, *J* =15.3 Hz, 1H each, CH_2Ph), 4.00-3.85 (m, 4H, OCH_2), 2.39 (d, *J* = 3.8 Hz, 2H, H-6), 2.35-2.26 (m, 3H, H-3 and 3-CH_2), 2.00 and 1.74 (2 d, *J* = 13.2 Hz, 1H each, H-4), 1.30 (s, 3H, Me); ^{13}C NMR (101 MHz, $CDCl_3$): δ 176.9 (C-2), 144.5 (C-7a), 136.4 (Ph), 135.6 (=CH), 128.6, 127.6, and 127.3 (Ph), 117.2 ($=CH_2$), 108.9 (C-5), 95.9 (C-7), 64.7 and 63.9 (OCH_2), 54.4 (C-3), 43.7 (CH_2Ph), 40.7 (C-3a), 38.1 (C-4), 35.3 (C-6), 33.2 (CH_2-C-3), 28.0 (Me). Spectral data are in accordance with published values.[1]

Evolution of 7b to compound 19. On exposure to air or in the chromatographic column using SiO_2, enlactam **7b** partially evolved to hydroxylated octahydroindole **19**: FTIR (neat, cm^{-1}) 3404, 3087, 3063, 3030, 2978, 2960, 2938, 2888, 1742, 1694 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.39–7.20 (m, 5H, ArH), 4.81 (s, 1H, OH), 4.73 and 4.34 (2d, *J* = 15.2 Hz, 1H each, CH_2Ph), 4.20-4.32 (m, 2H, CH_2), 3.96–3.73 (m, 4H, OCH_2), 3.12 (s, 1H, H-3), 2.02–1.99 (m, 2H, H-7), 1.75 (dd, *J* = 14.0, 3.1 Hz, H-4), 1.48 (d, *J* = 14.0, 1H, H-4), 1.33 (t, *J* = 7.2 Hz, 3H, CH_3), 1.26 (s, 3H, CH_3), 1.24 (m, 1H, H-6eq), 0.77 (m, 1H, H-6ax). ^{13}CNMR (101 MHz, $CDCl_3$): δ 172.1 (CO), 171.2 (CO), 138.1, 128.6, 128.3, and 127.5 (Ph), 107.3 (C-5), 92.3 (C-7a), 64.5 and 63.8 (OCH_2), 62.7 (CH_2), 61.5 (C-3), 45.1 (C-4), 44.5 (C-3a), 43.3 (CH_2Ph), 30.2 (C-6), 28.1 (C-7), 16.1 (Me), 14.1 (CH_3). HRMS (ESI) *m*/*z*: $[M+H]^+$ calcd for $C_{21}H_{28}NO_6$ 390.1911, found 390.1911.

Octahydroindole c-4a was available from enamide **4a** by sodium cyanoborohydride reduction in acetic acid according to the previously reported procedure.[1] ^1H NMR (400 MHz, $CDCl_3$): δ 7.30–7.19 (m, 5H, Ph), 5.97 (m, 1H, =CH), 5.11 (d, *J* = 17.0 Hz, 1H, $=CH_2$), 5.03 (d, *J* = 10.0 Hz, 1H, $=CH_2$), 5.02 and 3.92 (2 d, *J* = 15.2 Hz, 1H each, CH_2Ph), 3.92–3.87 (m, 4H, OCH_2), 3.03 (t, *J* = 3.2 Hz, 1H, H-7a), 2.64 (m, 1H, H-1'), 2.14 (m, 2H, H-3 and H-1'), 1.92 (dq, *J* = 15.2, 3.6 Hz, 1H, H-7eq), 1.82 (tt, *J* = 10.8, 3.6 Hz, 1H, H-7ax), 1.55 and 1.44 (2 d, *J* =13.2 Hz, 1H each, H-4), 1.44 and 1.29 (2 m, 1H each, H-6), 1.23 (s, 3H, Me). ^{13}C NMR (101 MHz, $CDCl_3$): δ 177.1 (C-2), 137.8 (*ipso*-Ph), 136.9 (=CH), 128.9, 128.2, and 127.7 (Ph), 116.1 ($=CH_2$), 108.3 (C-5), 64.8 and 63.9 (OCH_2), 59.5 (C-7a), 55.4 (C-3), 44.1 (CH_2Ph), 41.8 (C-3a), 36.9 (C-4), 29.2 (CH_2), 28,8 (C-6), 23.2 (Me), 20.6 (C-7).

Hydrogenation of 7a in MeOH. To a solution of **7a** (100 mg, 0.27 mmol) in MeOH (10 mL) was added Pd/C (40 mg, 40% w/w) and the mixture was stirred under an atmosphere of H_2 at room temperature overnight. The mixture was filtered through a short celite pad, concentrated and purified by chromatography (EtOAc in hexane from 0% to 40%) to provide a 1:8 mixture of **c-18a** and **c-18b** (74 mg, 73%) and finally **25** (20 mg, 18%). **(3*RS*,3a*SR*,7a*SR*) Ethyl 1-Benzyl-3-(hydroxymethyl)-3a-methyl-2,5-dioxooctahydro-1*H*-indole-3-carboxylate ethylene acetal (25)**. FTIR (neat, cm^{-1}) 3475, 2942, 2889, 1716, 1682 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.39–7.27 (m, 5H, PhH), 5.17 (d, *J* = 15.4 Hz, 1H, CH_2Ph), 4.30–4.12 (m, 3H, 1H-CH_2OH, OEt), 3.98–3.88 (m, 5H, 1H-CH_2OH, OCH_2), 3.89 (d, *J* = 14.8 Hz, 1H, CH_2Ph), 3.39 (t, *J* = 3.4 Hz, 1H, H-7a), 2.00 (dq, *J* = 15.2, 3.2 Hz, 1H, H-7eq), 1.82 (tt, *J* = 15.2, 3.6 Hz, 1H, H-7ax), 1.75 and 1.64 (2d, *J* = 13.8 Hz, 1H each, H-4), 1.63–1.54 (m, 1H, H-6), 1.39 (td, *J* = 13.8, 4.1 Hz, 1H, H-6), 1.21 (s, 3H, CH_3), 1.19 (t, *J* = 7.2 Hz, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$): δ 174.1 (CO), 170.5 (C-2), 135.7, 128.5, 128.4, and 127.7 (Ph), 107.6 (C-5), 64.7 (OCH_2), 64.1 (C-3), 63.8 (OCH_2), 61.8 (CH_2OH), 61.6 (CH_2), 58.1 (C-7a), 44.5 (C-3a), 44.1 (CH_2Ph), 37.4 (C-4), 29.0 (C-6), 20.3 (C-7), 19.2 (Me), 14.2 (CH_3). HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{22}H_{30}NO_6$ 404.2068; found 404.2073.

Reduction of 7a with $NaCNBH_3$. To a solution of **7a** (100 mg, 0.27 mmol) in AcOH (1 mL) was added $NaCNBH_3$ (34 mg, 0.54 mmol) portionwise and the mixture was stirred at room temperature for 62 h. Water was added and the mixture was extracted with CH_2Cl_2 (3×15 mL). The organic layer was washed with saturated aqueous Na_2CO_3 (2×15 mL), dried, concentrated, and purified by chromatography (SiO_2, EtOAc in hexane from 20% to 100%) to give a 1:1.2 mixture of **c-18a** and **c-18b** (70 mg, 67%). Finally, **7a** (10 mg) was recovered. For their NMR data, see Experimental in the Article.

4. References

1. Coussanes G.; Bonjoch, *Org. Lett.* 2017, **19**, 878−881.

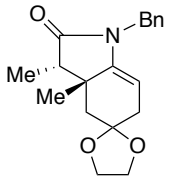
2. A. Cordero-Vargas, B. Bradshaw, J. Bonjoch, *Tetrahedron* 2008, **64**, 8134−8140.

3. S. Jansana, G. Coussanes, J. Puig, F. Diaba, J. Bonjoch, *Helv. Chim. Acta* 2019, **102**, e1900188.

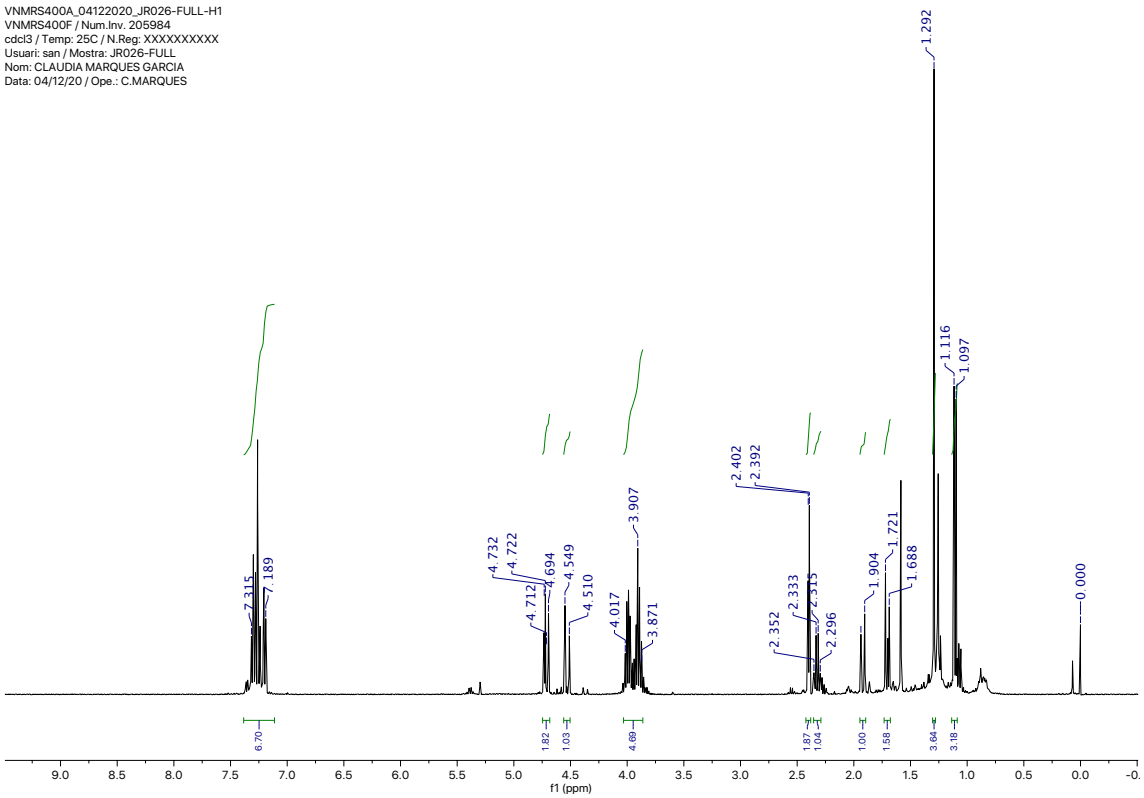
4. Cho, Y. S.; Carcache, D. A.; Tian, Y.; Li, Y.-M.; Danishefsky, S. J. *J. Am. Chem. Soc.* **2004**, *126*, 14358-14359.

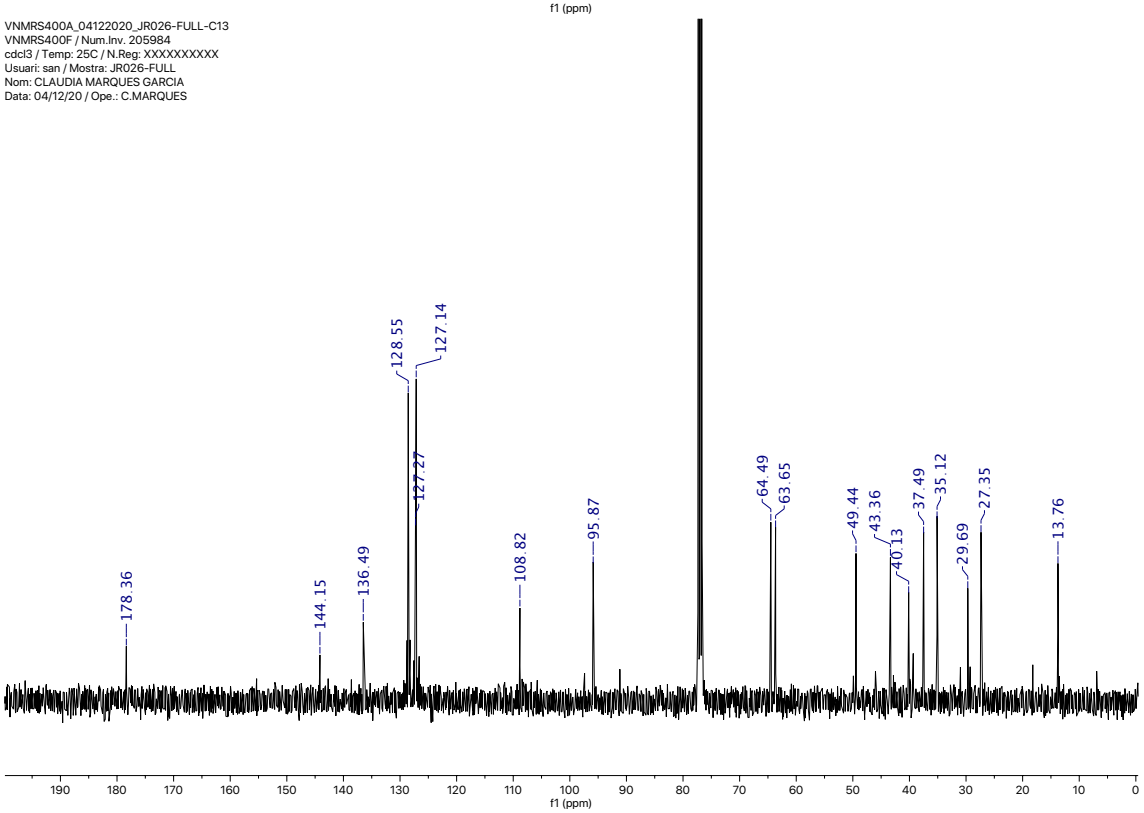
5. G. Revial, I. Jabin, M. Redolfi, M.; Pfau, *Tetrahedron: Asymmetry* 2001, **12**, 1683−1688.

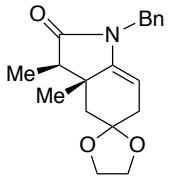
5. ^{1}H- and ^{13}C-NMR Spectra of new compounds



5a

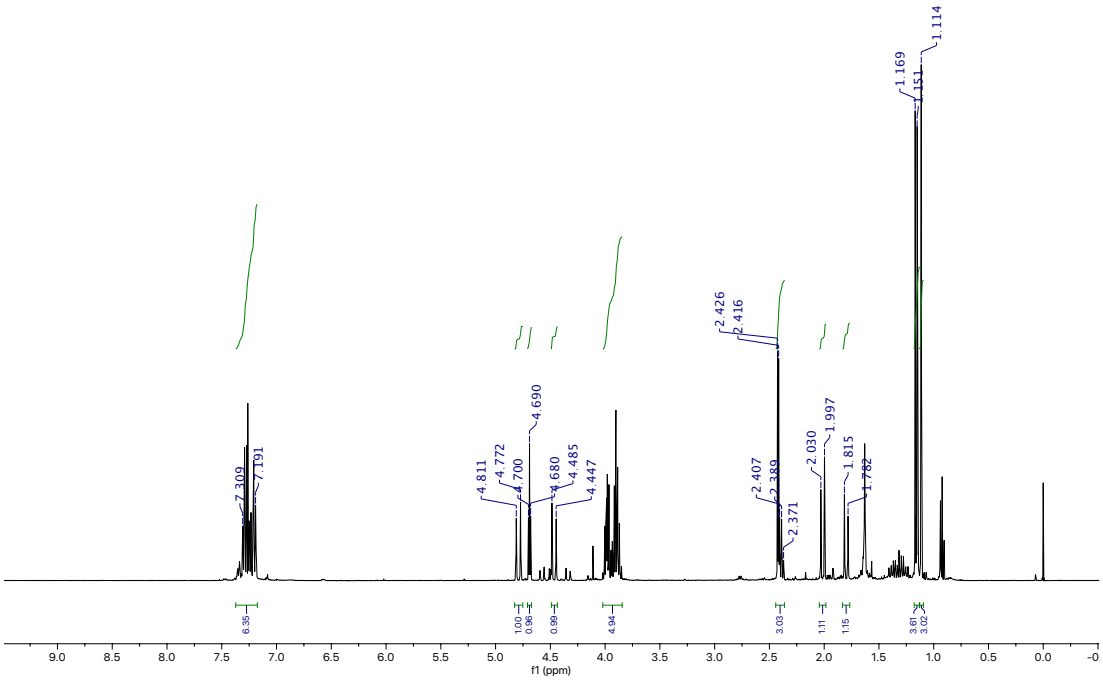




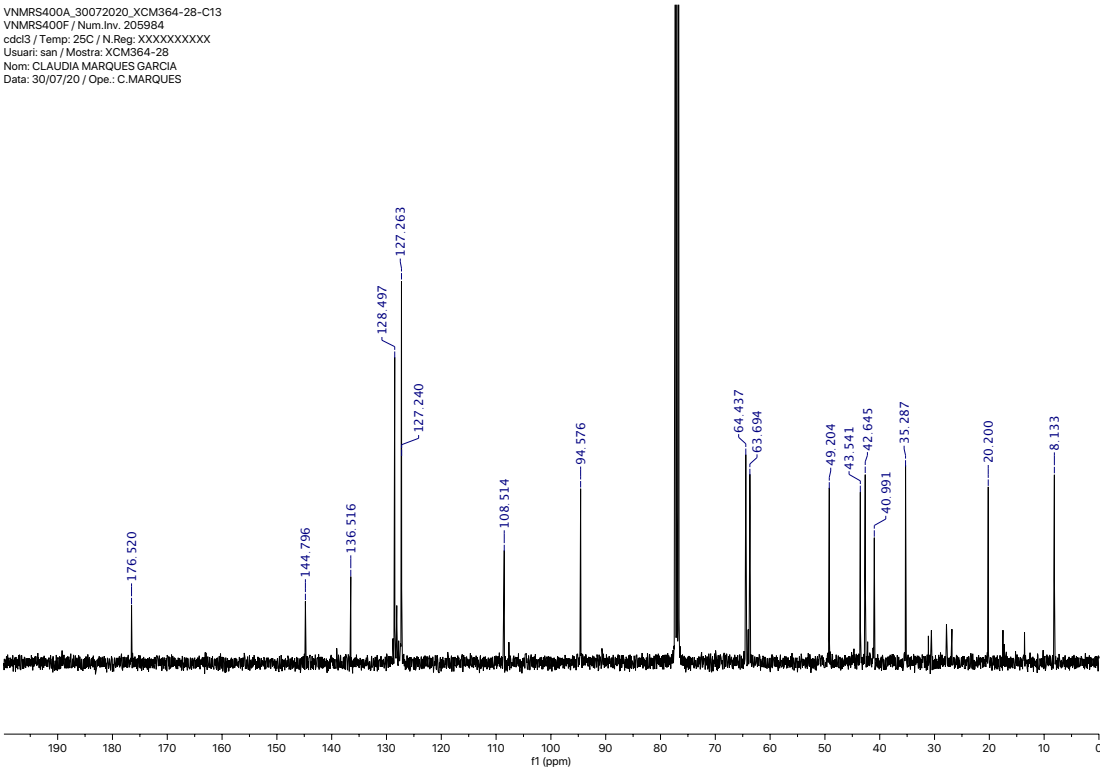


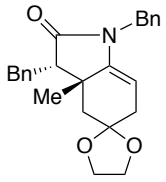
5b

VNMRS400A_30072020_XCM364-28-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM364-28
Nom: CLAUDIA MARQUES GARCIA
Data: 30/07/20 / Ope.: C.MARQUES



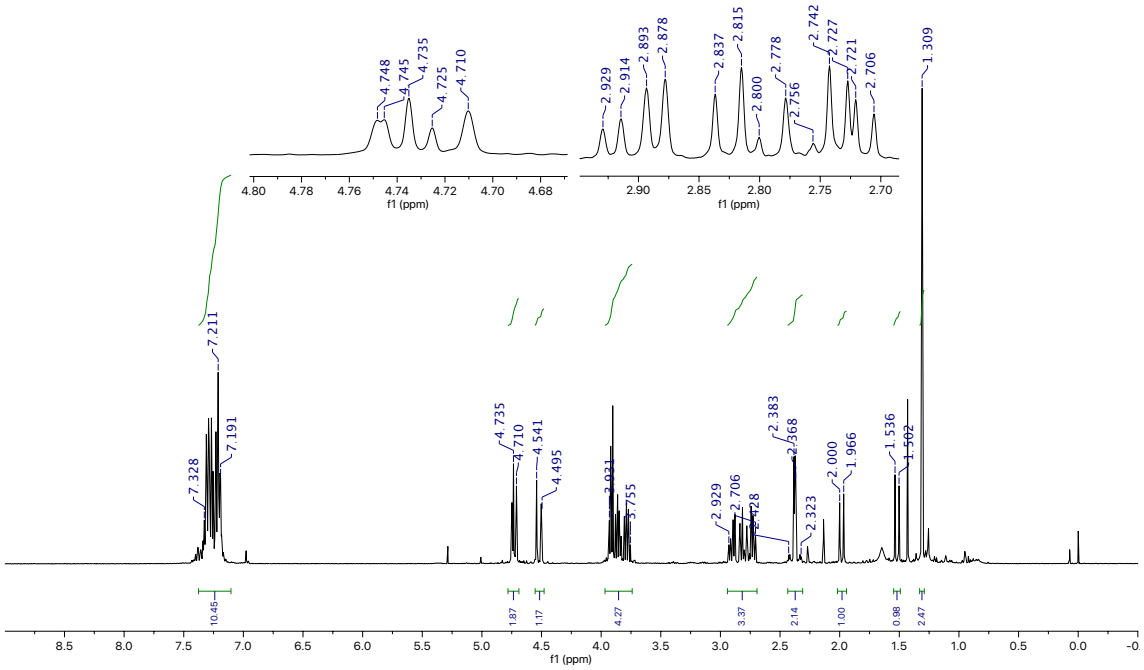
VNMRS400A_30072020_XCM364-28-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM364-28
Nom: CLAUDIA MARQUES GARCIA
Data: 30/07/20 / Ope.: C.MARQUES



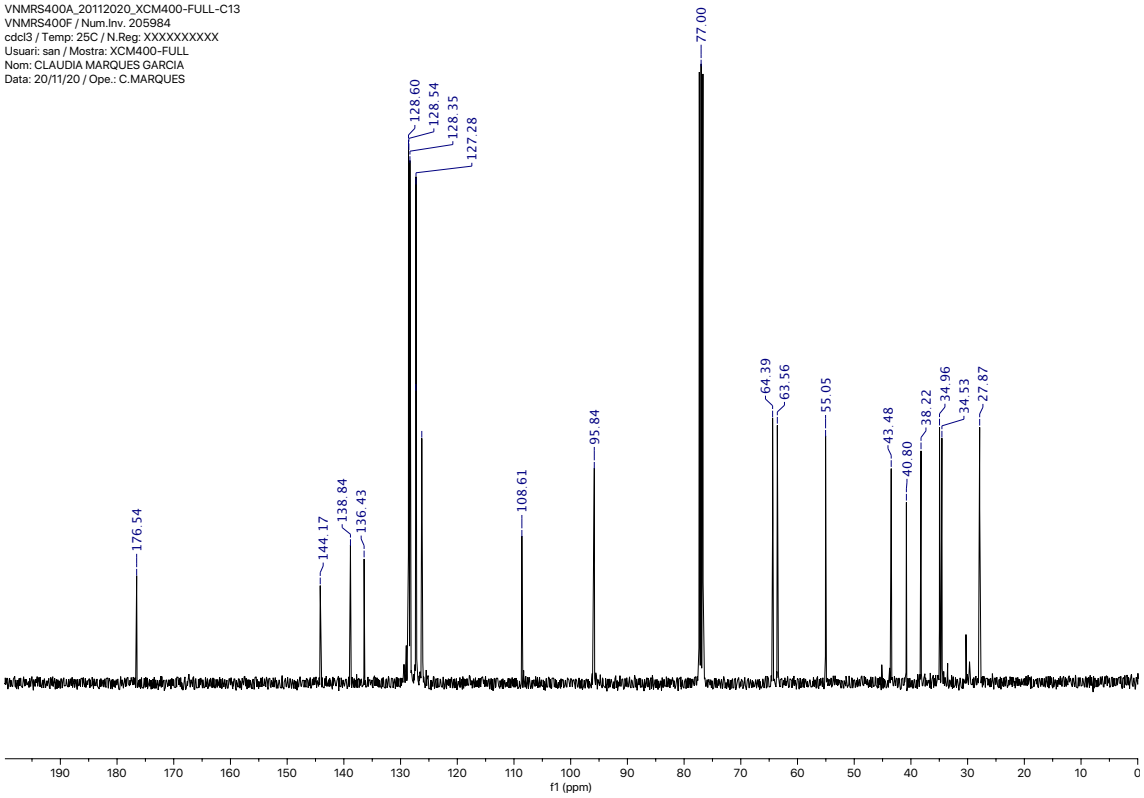


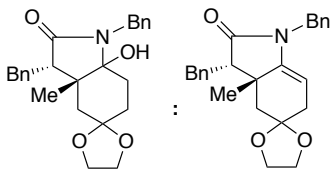
6a

VNMRS400A_20112020_XCM400-FULL-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM400-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 20/11/20 / Ope.: C.MARQUES

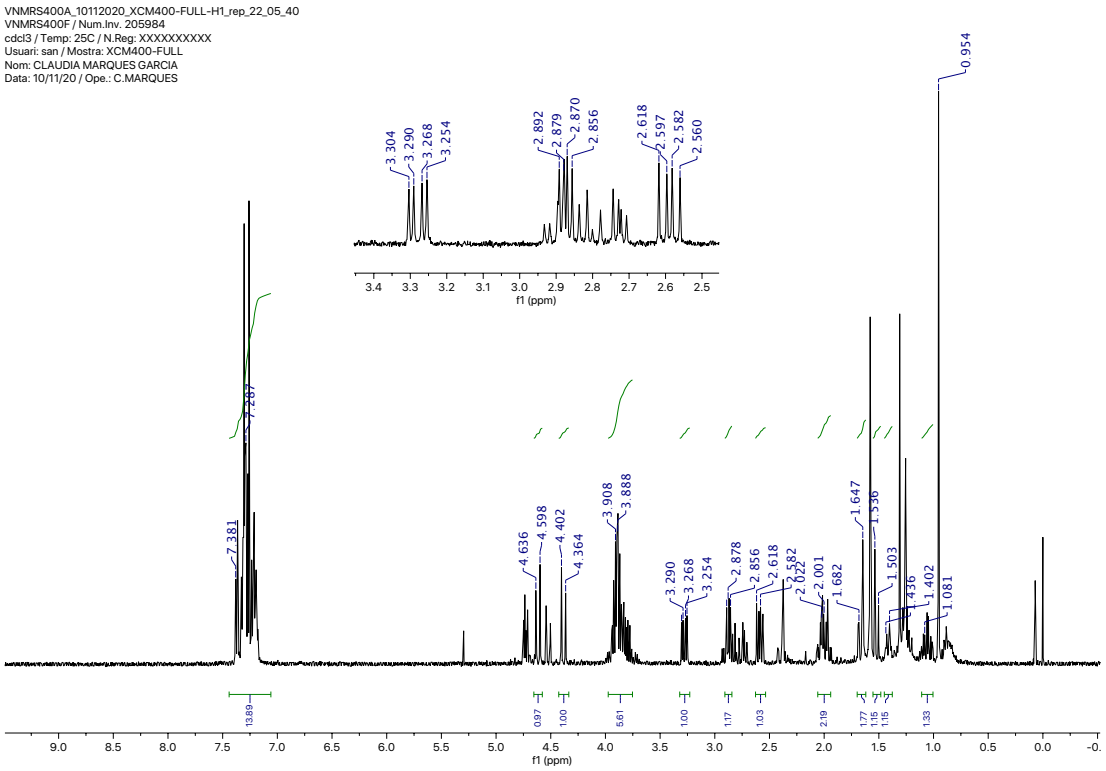


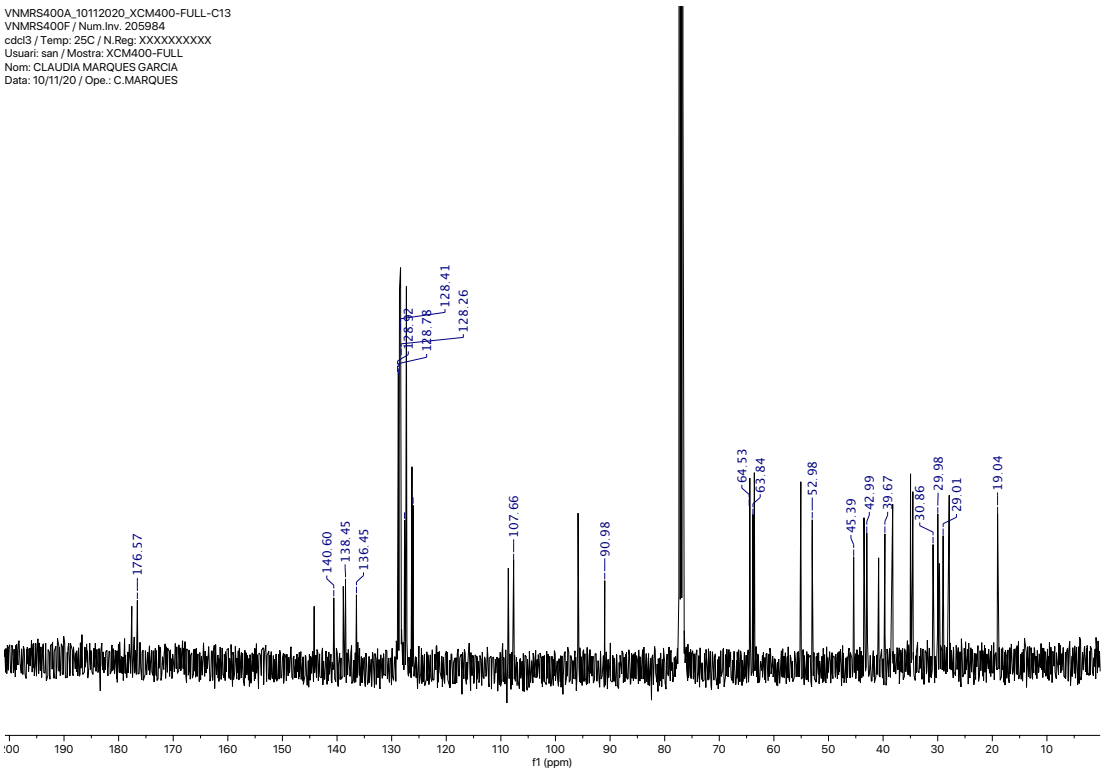
VNMRS400A_20112020_XCM400-FULL-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM400-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 20/11/20 / Ope.: C.MARQUES

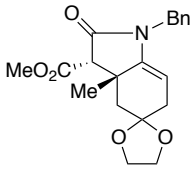




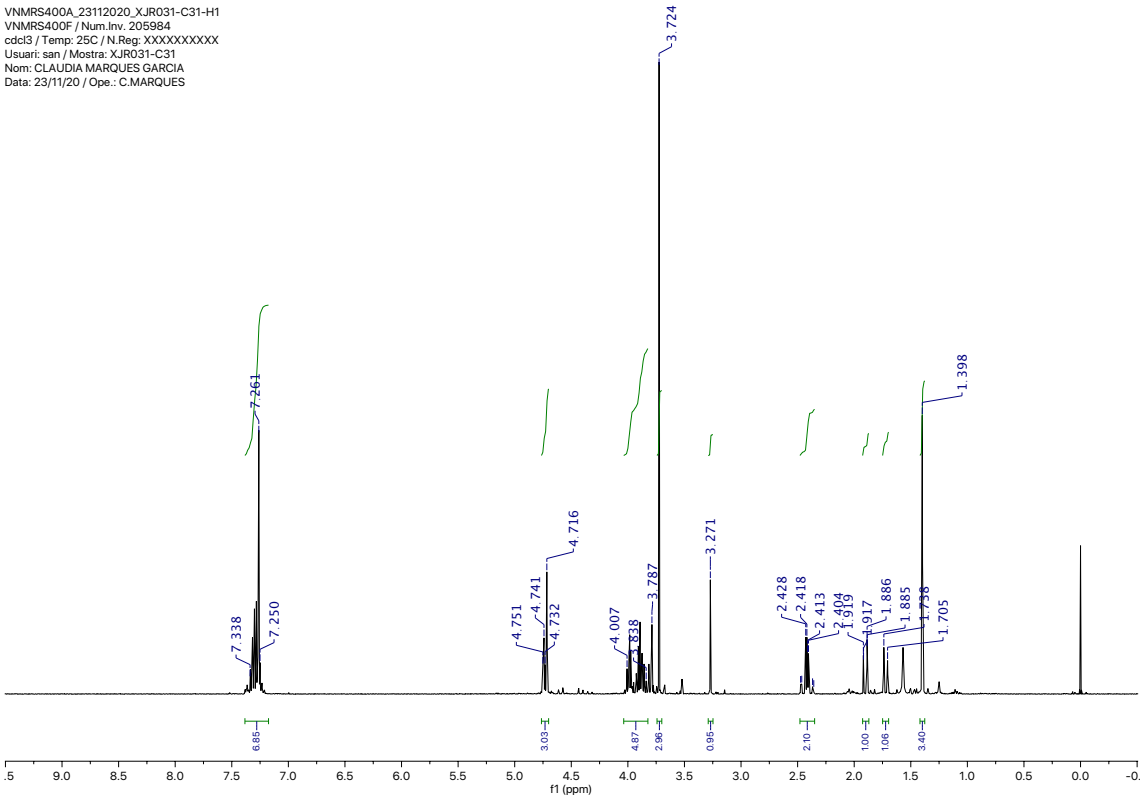
6c : **6a**

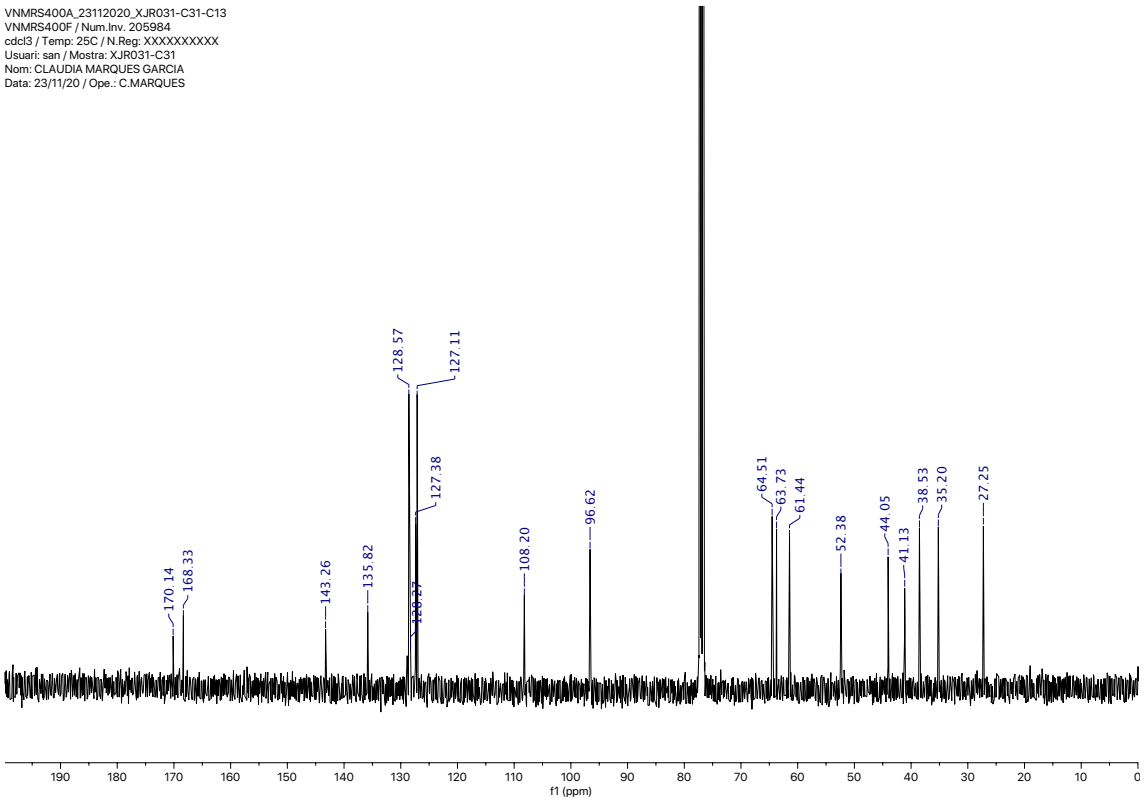

VNMRS400A_10112020_XCM400-FULL-H1_rep_22_05_40
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM400-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 10/11/20 / Ope.: C.MARQUES
f1 (ppm)

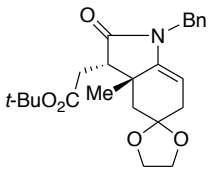

VNMRS400A_10112020_XCM400-FULL-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM400-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 10/11/20 / Ope.: C.MARQUES
f1 (ppm)


O
N
Bn
MeO2C
Me
O
O

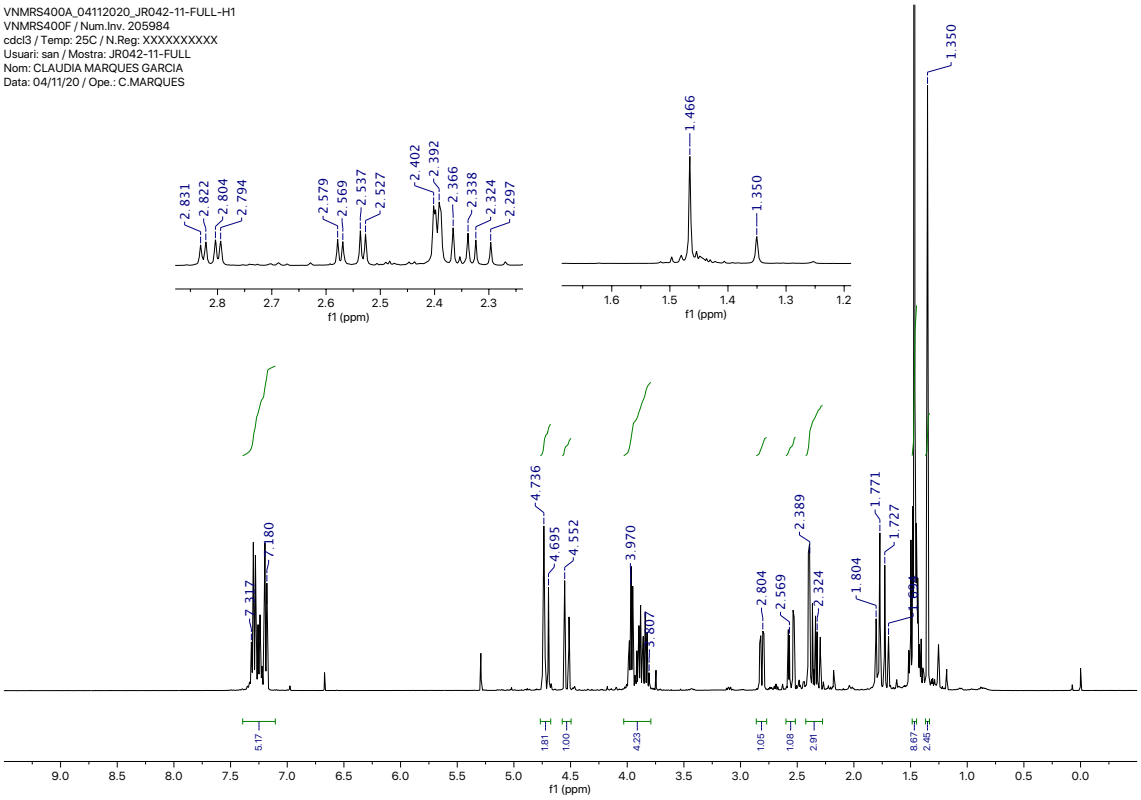
7a*

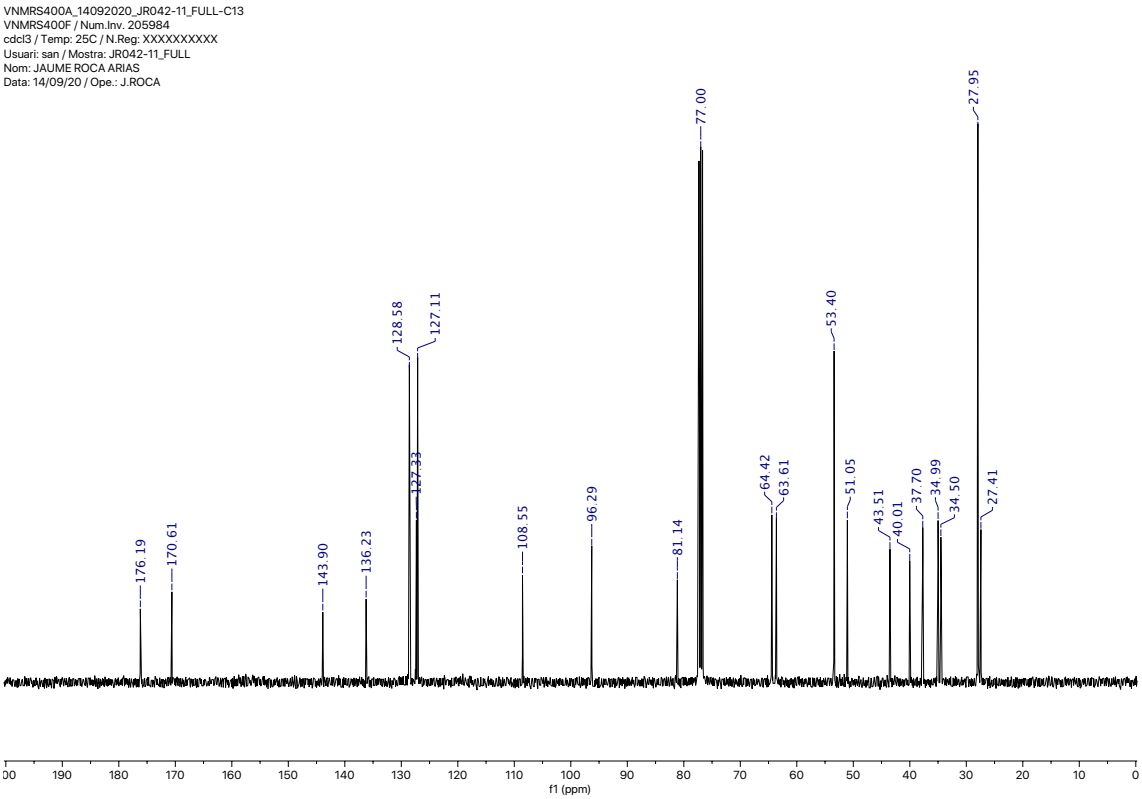

VNMRS400A_23112020_XJR031-C31-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XJR031-C31
Nom: CLAUDIA MARQUES GARCIA
Data: 23/11/20 / Ope.: C.MARQUES
3.724
7.261
1.398
4.716
3.271
7.338
7.250
4.751
4.741
4.732
4.007
3.787
2.428
2.418
2.413
2.404
1.919
1.917
1.886
1.885
1.738
1.705
6.85
3.03
4.87
2.96
0.95
2.10
1.00
1.06
3.40
f1 (ppm)

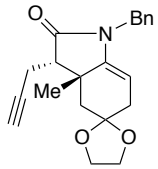

VNMRS400A_23112020_XJR031-C31-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XJR031-C31
Nom: CLAUDIA MARQUES GARCIA
Data: 23/11/20 / Ope.: C.MARQUES
128.57
127.11
127.38
64.51
63.73
61.44
96.62
38.53
35.20
27.25
44.05
41.13
52.38
108.20
135.82
143.26
170.14
168.33
f1 (ppm)


O
N
Bn
t-BuO2C
Me
O
O

8a

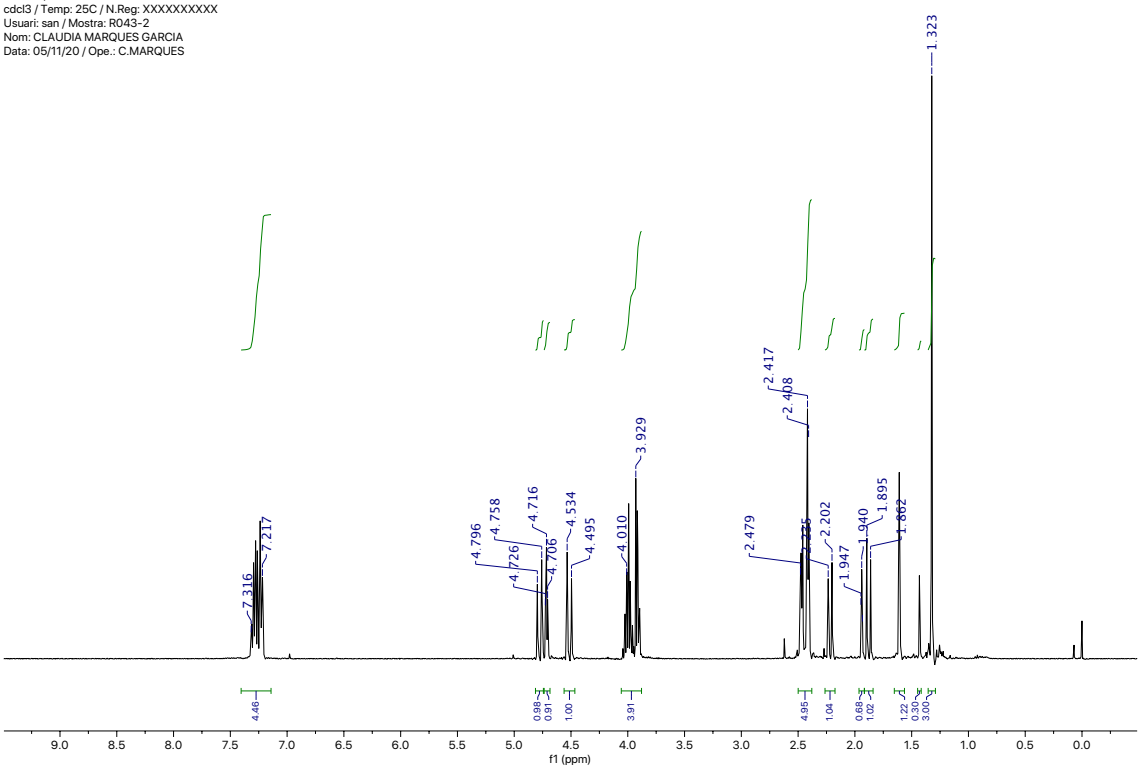

VNMRS400A_04112020_JR042-11-FULL-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR042-11-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 04/11/20 / Ope.: C.MARQUES
f1 (ppm)


VNMRS400A_14092020_JR042-11_FULL-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR042-11_FULL
Nom: JAUME ROCA ARIAS
Data: 14/09/20 / Ope.: J.ROCA
f1 (ppm)

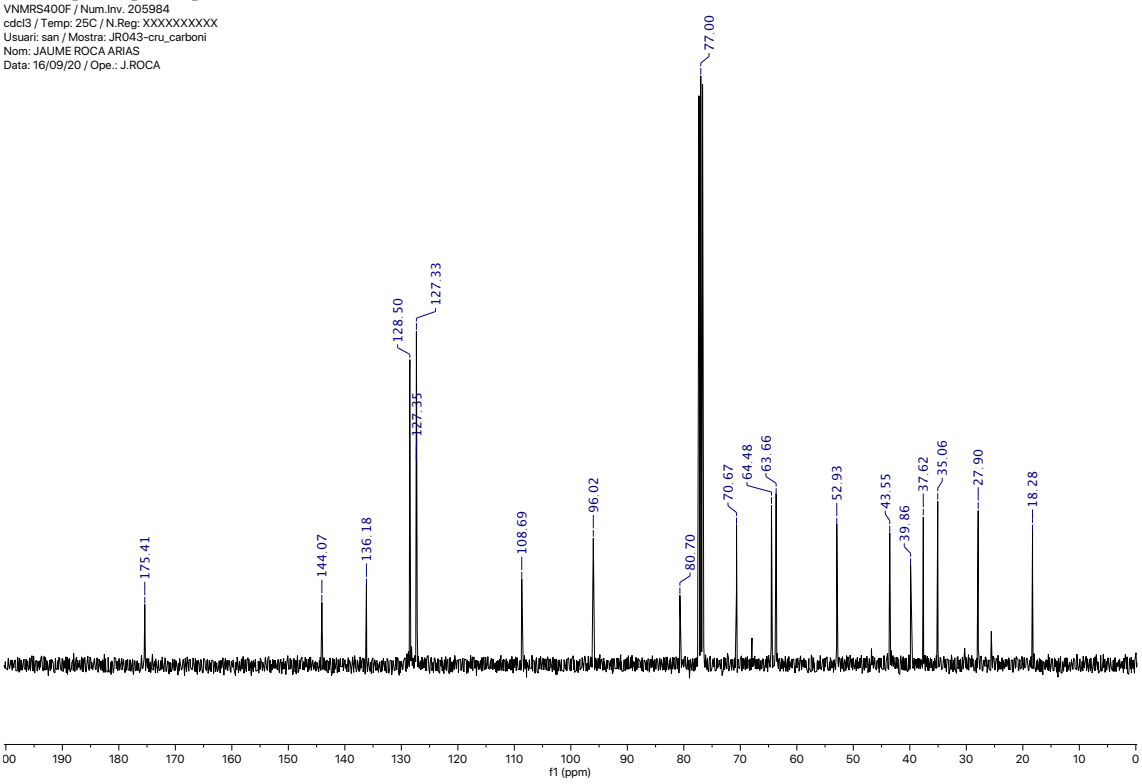


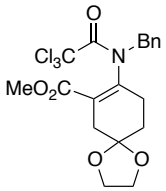
9a

M400AFF_05112020_R043-2-H1
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: R043-2
Nom: CLAUDIA MARQUES GARCIA
Data: 05/11/20 / Ope.: C.MARQUES

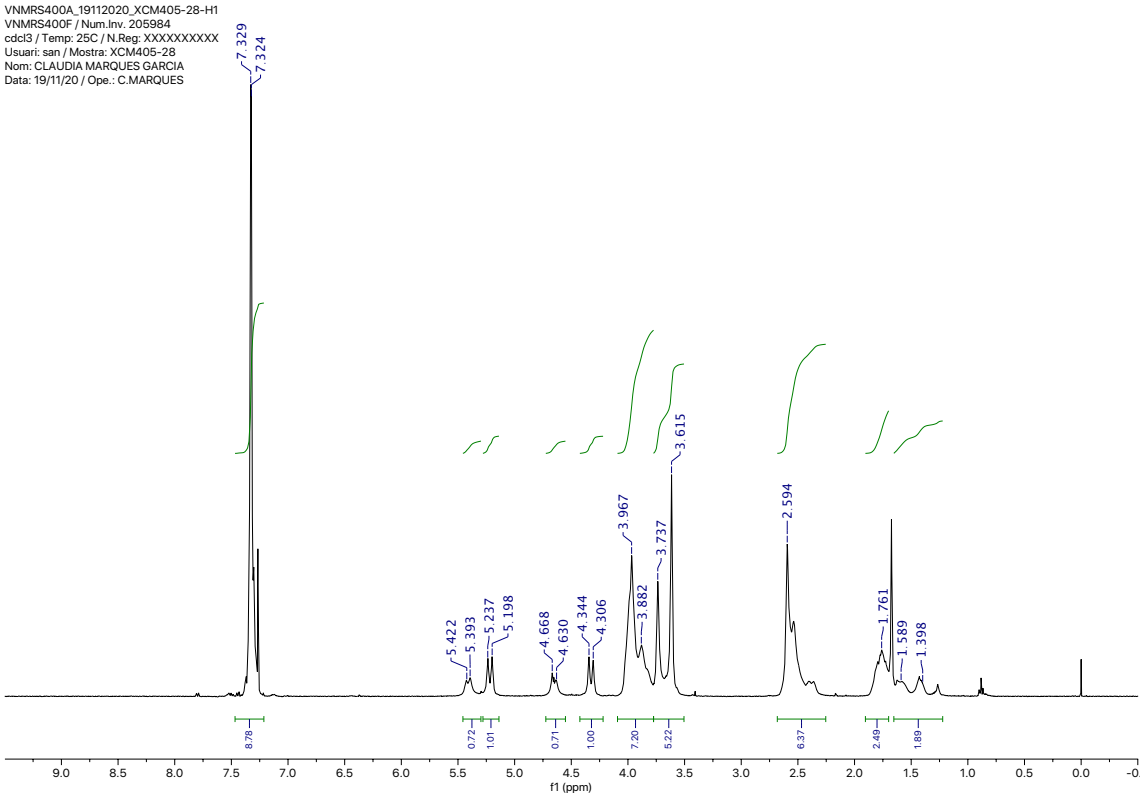


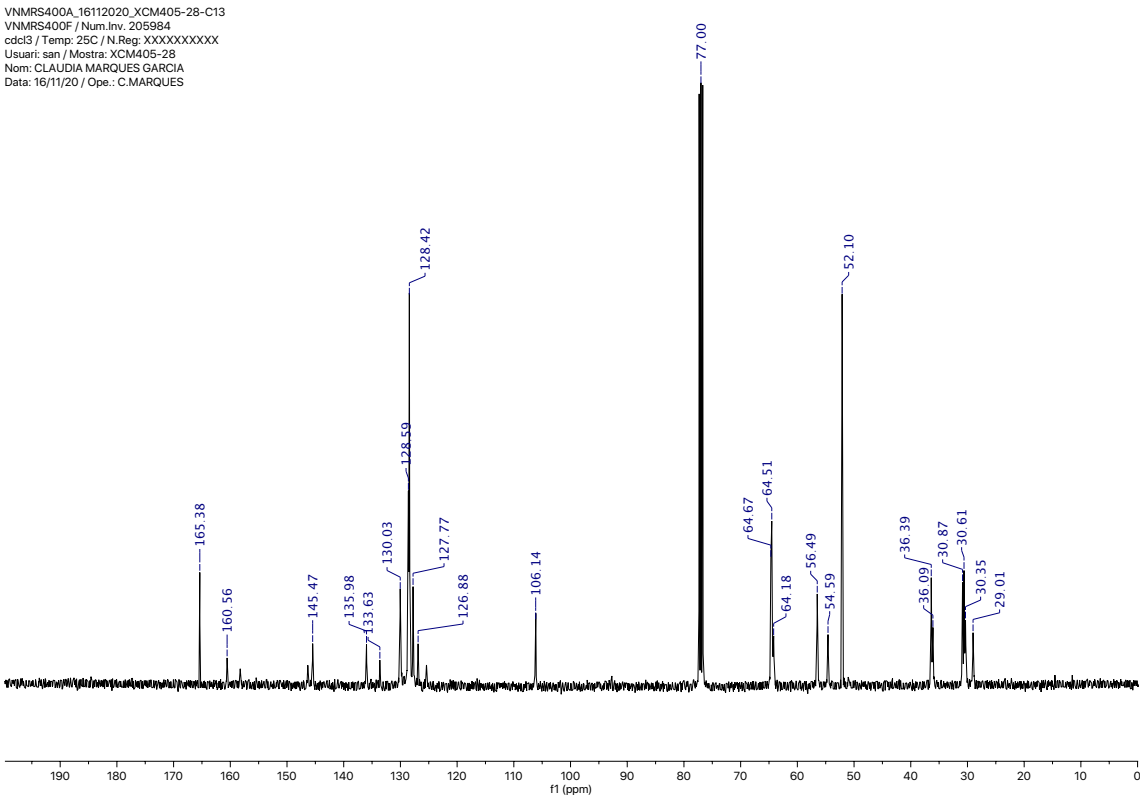
VNMRS400A_16092020_JR043-cru_carboni-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR043-cru_carboni
Nom: JAUME ROCA ARIAS
Data: 16/09/20 / Ope.: J.ROCA

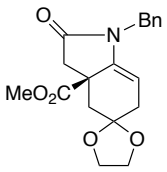



O
Cl3C
N
Bn
MeO2C
O
O

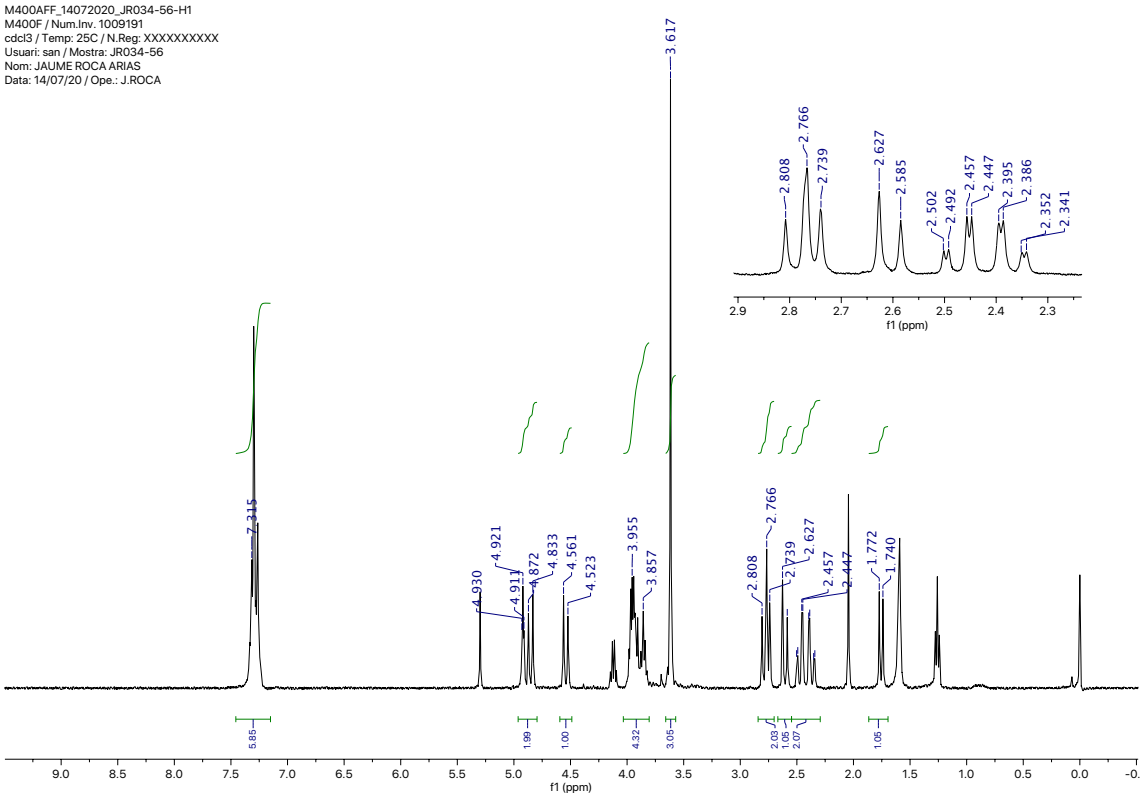
11

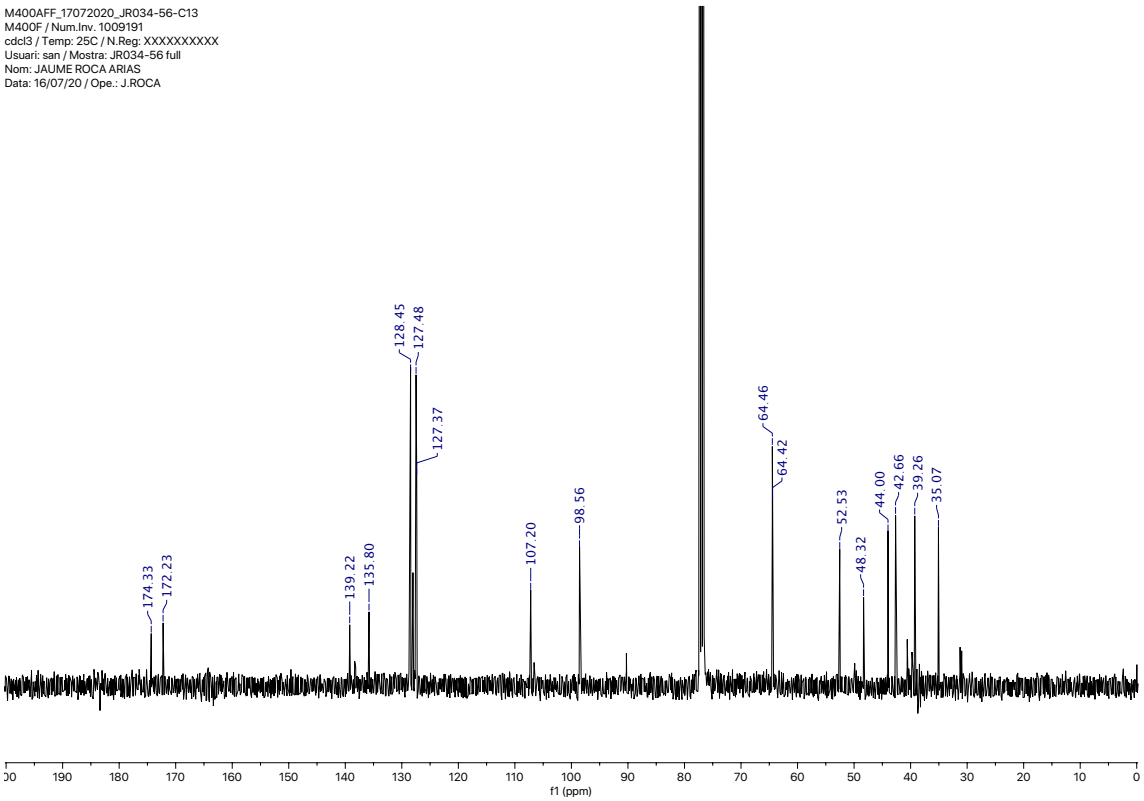

VNMRS400A_19112020_XCM405-28-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM405-28
Nom: CLAUDIA MARQUES GARCIA
Data: 19/11/20 / Ope.: C.MARQUES
7.329
7.324
5.422
5.393
5.237
5.198
4.668
4.630
4.344
4.306
3.967
3.882
3.737
3.615
2.594
1.761
1.589
1.398
8.78
0.72
1.01
0.71
1.00
7.20
5.22
6.37
2.49
1.89
f1 (ppm)

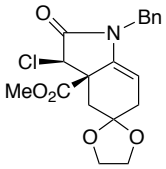

VNMRS400A_16112020_XCM405-28-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM405-28
Nom: CLAUDIA MARQUES GARCIA
Data: 16/11/20 / Ope.: C.MARQUES
77.00
165.38
160.56
145.47
135.98
133.63
130.03
128.59
128.42
127.77
126.88
106.14
64.67
64.51
64.18
56.49
54.59
52.10
36.39
36.09
30.87
30.61
30.35
29.01
f1 (ppm)


O
N
Bn
MeO2C
O
O

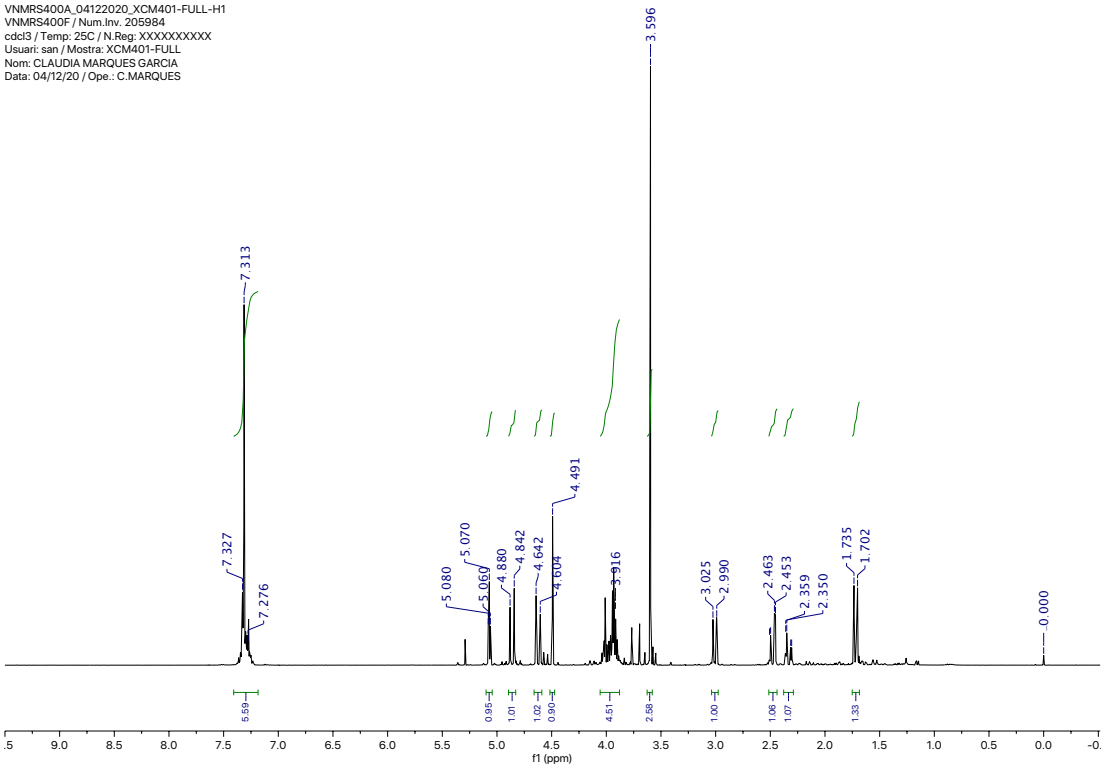
12

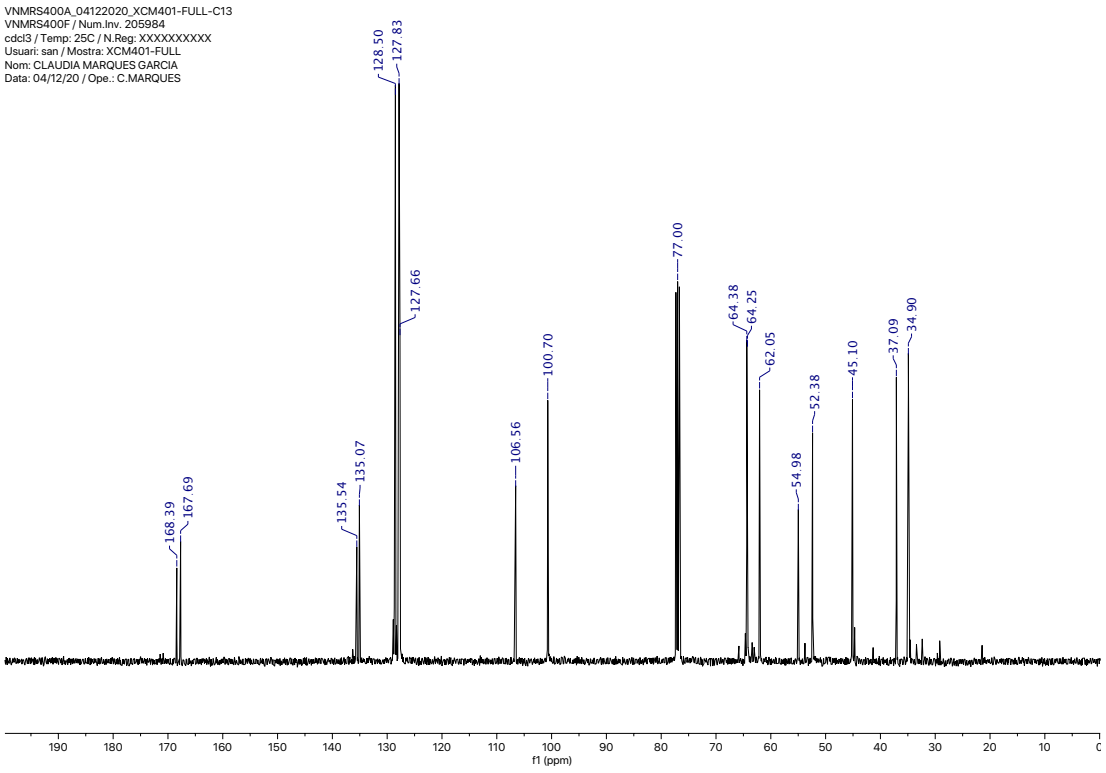

M400AFF_14072020_JR034-56-H1
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR034-56
Nom: JAUME ROCA ARIAS
Data: 14/07/20 / Ope.: J.ROCA
3.617
2.808
2.766
2.739
2.627
2.585
2.502
2.492
2.457
2.447
2.395
2.386
2.352
2.341
f1 (ppm)
7.315
4.930
4.921
4.911
4.872
4.833
4.561
4.523
3.955
3.857
2.808
2.766
2.739
2.627
2.457
2.447
1.772
1.740
5.85
1.99
1.00
4.32
3.05
2.08
1.95
2.07
1.06
f1 (ppm)

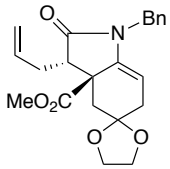

M400AFF_17072020_JR034-56-C13
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR034-56 full
Nom: JAUME ROCA ARIAS
Data: 16/07/20 / Ope.: J.ROCA
128.45
127.48
127.37
64.46
64.42
174.33
172.23
139.22
135.80
107.20
98.56
52.53
48.32
44.00
42.66
39.26
35.07
f1 (ppm)


O
N
Bn
Cl
MeO2C
O
O

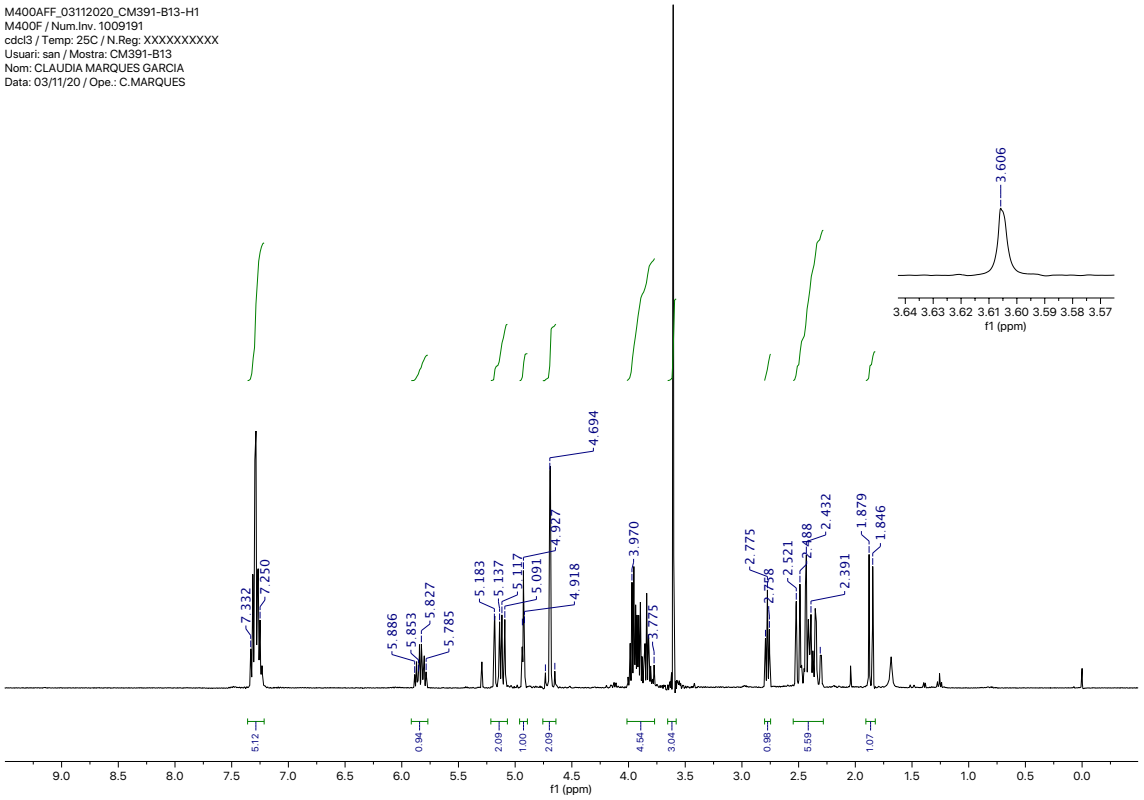
12b-Cl

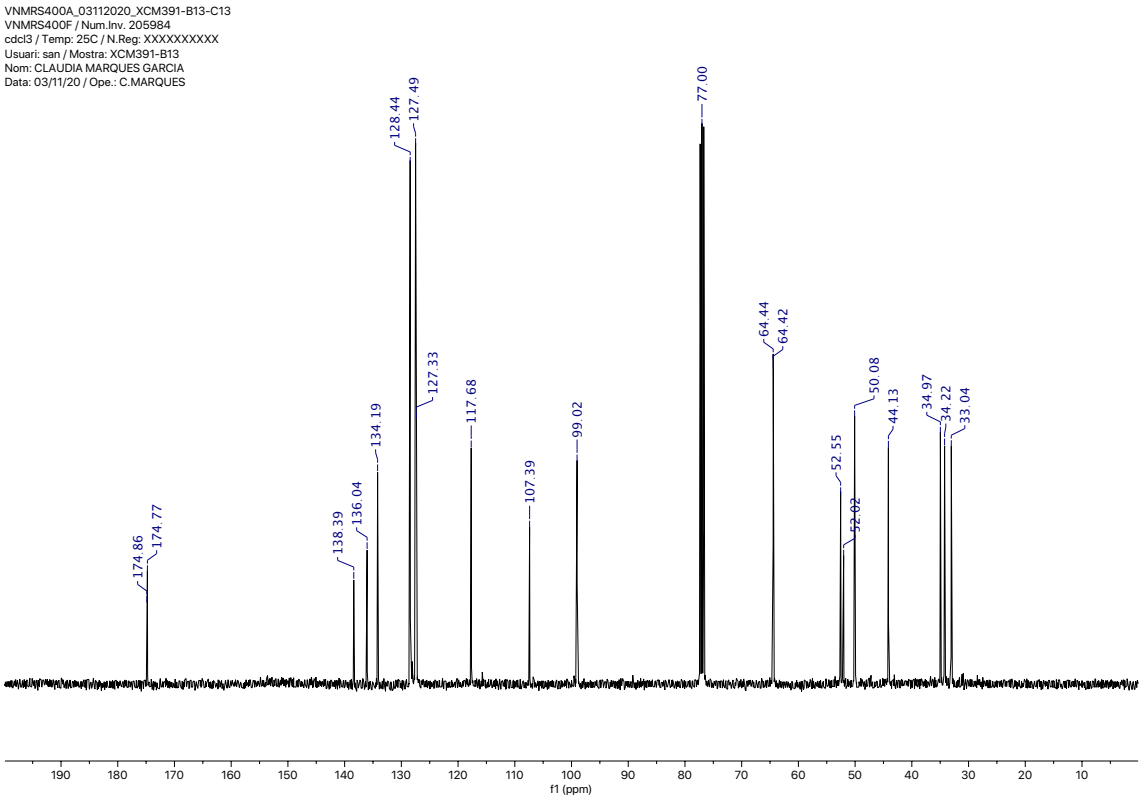

VNMRS400A_04122020_XCM401-FULL-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM401-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 04/12/20 / Ope.: C.MARQUES
3.596
7.313
7.327
7.276
5.080
5.070
5.060
4.880
4.842
4.642
4.604
4.491
3.916
3.025
2.990
2.463
2.453
2.359
2.350
1.735
1.702
0.000
5.59
0.95
1.01
1.02
0.90
4.51
2.58
1.00
1.06
1.07
1.33
f1 (ppm)

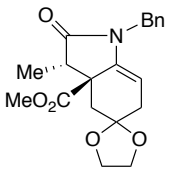

VNMRS400A_04122020_XCM401-FULL-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM401-FULL
Nom: CLAUDIA MARQUES GARCIA
Data: 04/12/20 / Ope.: C.MARQUES
128.50
127.83
127.66
77.00
64.38
64.25
62.05
106.56
100.70
168.39
167.69
135.54
135.07
54.98
52.38
45.10
37.09
34.90
f1 (ppm)


O
N
Bn
MeO2C
O
O

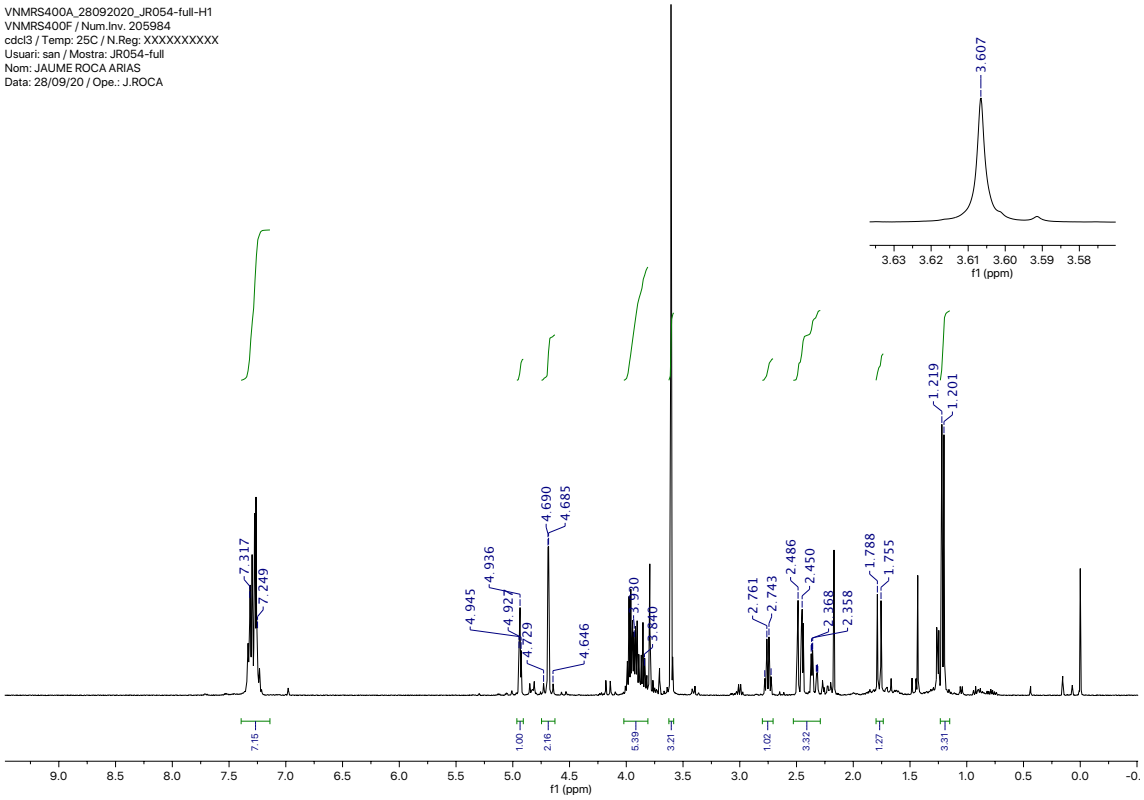
13a

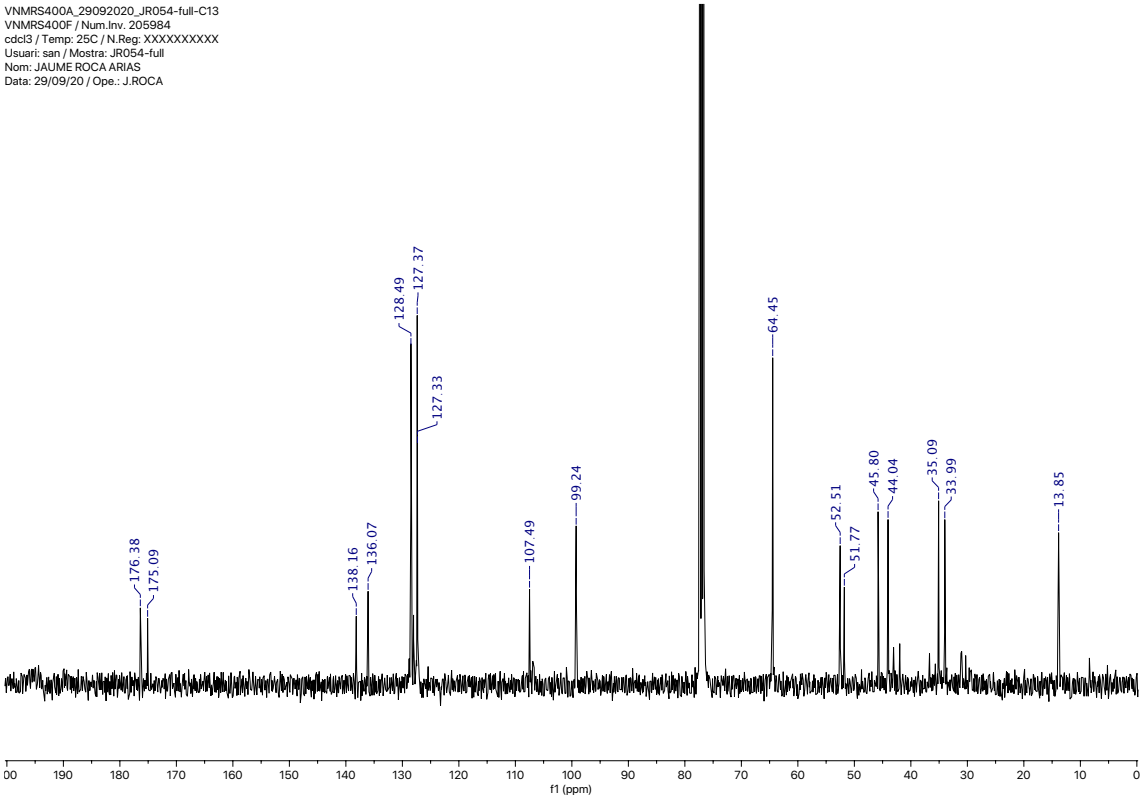

M400AFF_03112020_CM391-B13-H1
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: CM391-B13
Nom: CLAUDIA MARQUES GARCIA
Data: 03/11/20 / Ope.: C.MARQUES
3.606
3.64 3.63 3.62 3.61 3.60 3.59 3.58 3.57
f1 (ppm)
4.694
4.927
4.918
5.183
5.137
5.117
5.091
3.970
3.775
7.332
7.250
5.886
5.853
5.827
5.785
2.775
2.758
2.521
2.488
2.432
2.391
1.879
1.846
5.12
0.94
2.09
1.00
2.09
4.54
3.04
0.98
5.59
1.07
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

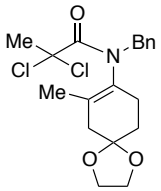

VNMRS400A_03112020_XCM391-B13-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM391-B13
Nom: CLAUDIA MARQUES GARCIA
Data: 03/11/20 / Ope.: C.MARQUES
77.00
128.44
127.49
127.33
64.44
64.42
50.08
44.13
34.97
34.22
33.04
117.68
134.19
99.02
107.39
52.55
52.02
138.39
136.04
174.86
174.77
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


O
N
Bn
Me
MeO2C
O
O

14a

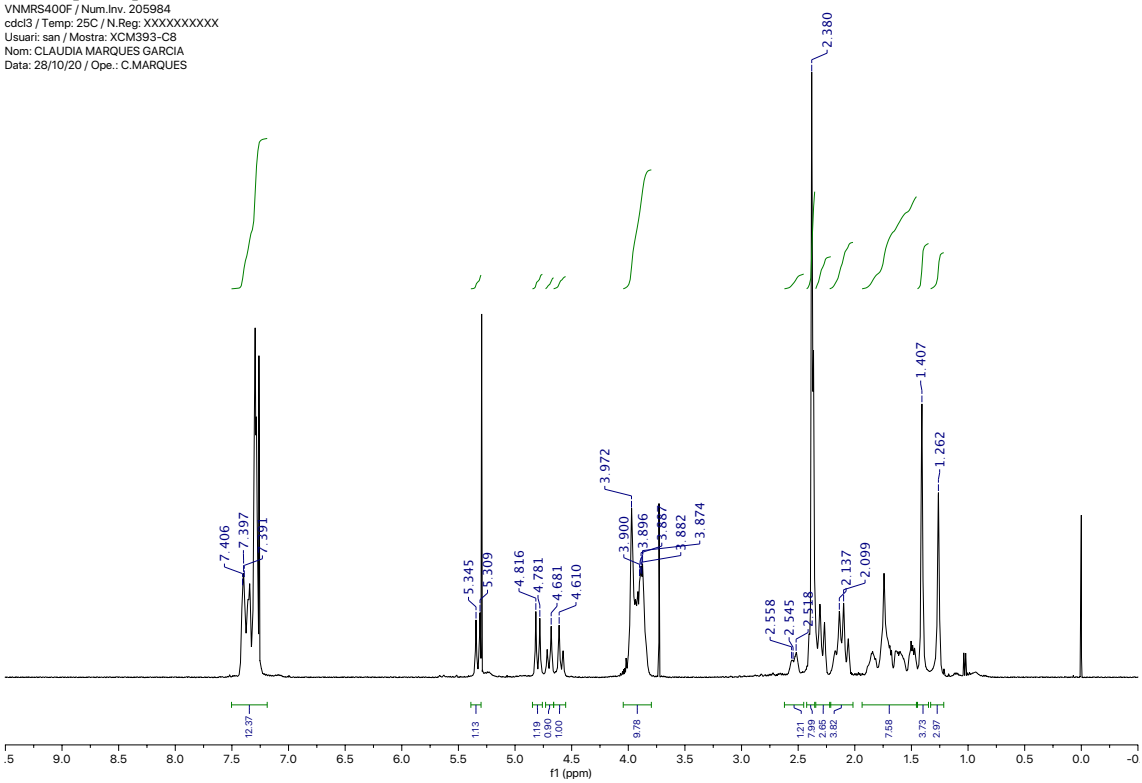

VNMRS400A_28092020_JR054-full-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR054-full
Nom: JAUME ROCA ARIAS
Data: 28/09/20 / Ope.: J.ROCA
3.607
7.317
7.249
4.945
4.936
4.927
4.729
4.690
4.685
4.646
3.930
3.840
2.761
2.743
2.486
2.450
2.368
2.358
1.788
1.755
1.219
1.201
f1 (ppm)


VNMRS400A_29092020_JR054-full-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR054-full
Nom: JAUME ROCA ARIAS
Data: 29/09/20 / Ope.: J.ROCA
176.38
175.09
138.16
136.07
128.49
127.37
127.33
107.49
99.24
64.45
52.51
51.77
45.80
44.04
35.09
33.99
13.85
f1 (ppm)

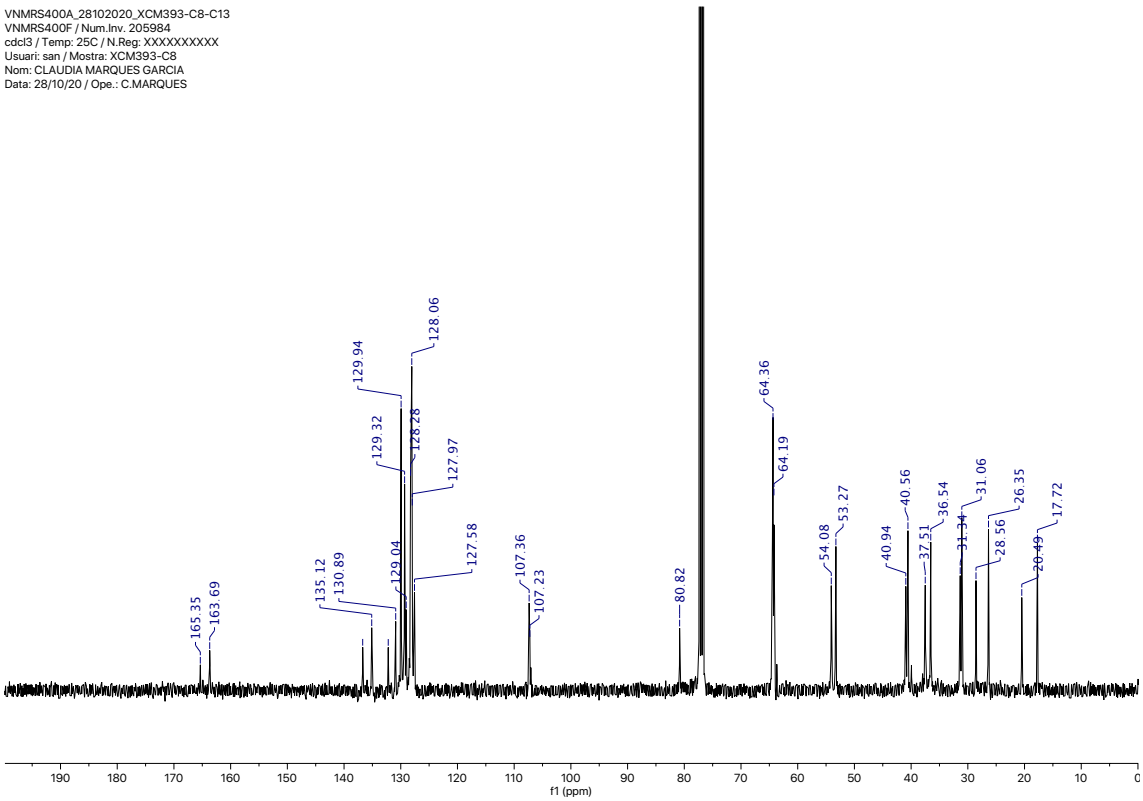


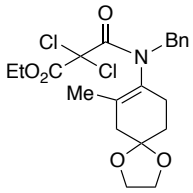
15

VNMRS400A_28102020_XCM393-C8-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM393-C8
Nom: CLAUDIA MARQUES GARCIA
Data: 28/10/20 / Ope.: C.MARQUES



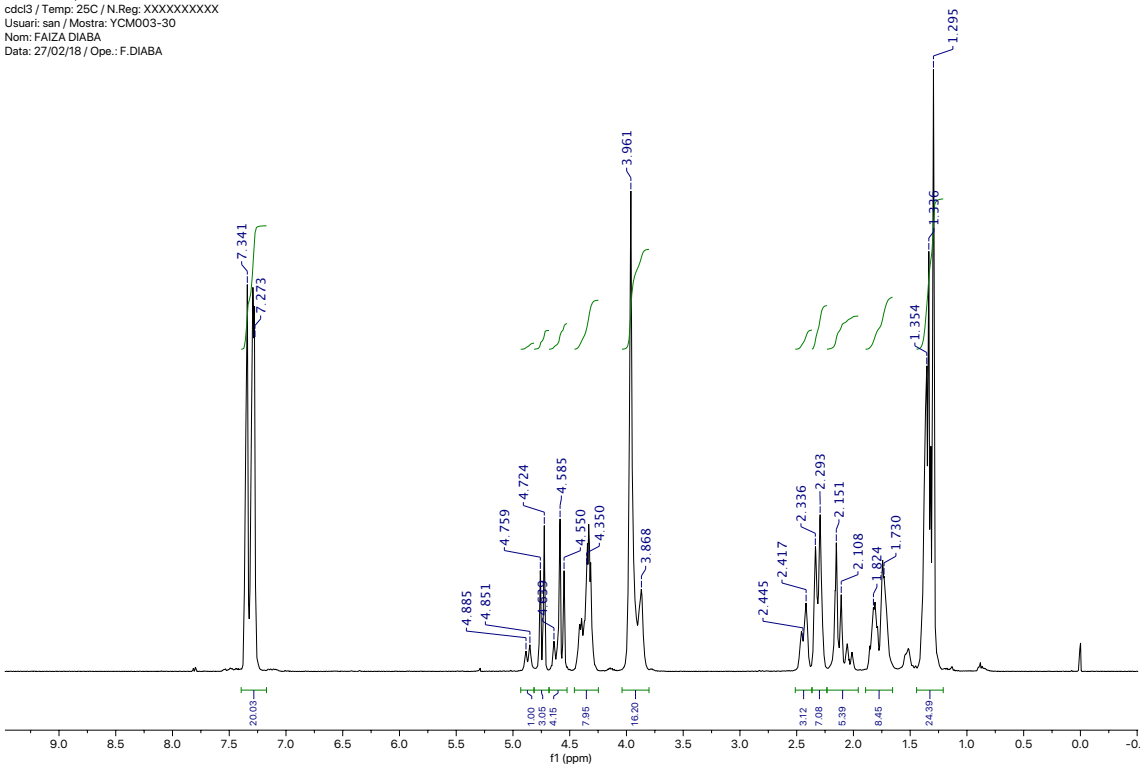
VNMRS400A_28102020_XCM393-C8-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM393-C8
Nom: CLAUDIA MARQUES GARCIA
Data: 28/10/20 / Ope.: C.MARQUES



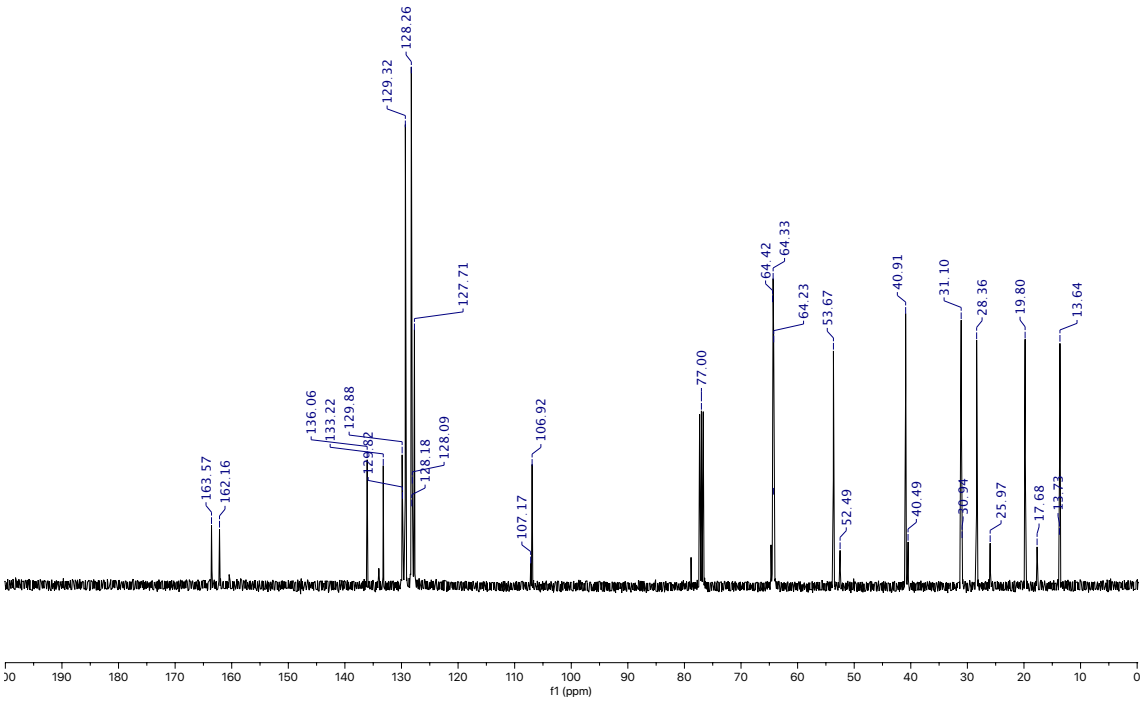


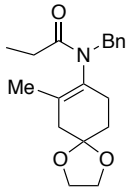
16

VNMRS400A_27022018_YCM003-30-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: YCM003-30
Nom: FAIZA DIABA
Data: 27/02/18 / Ope.: F.DIABA



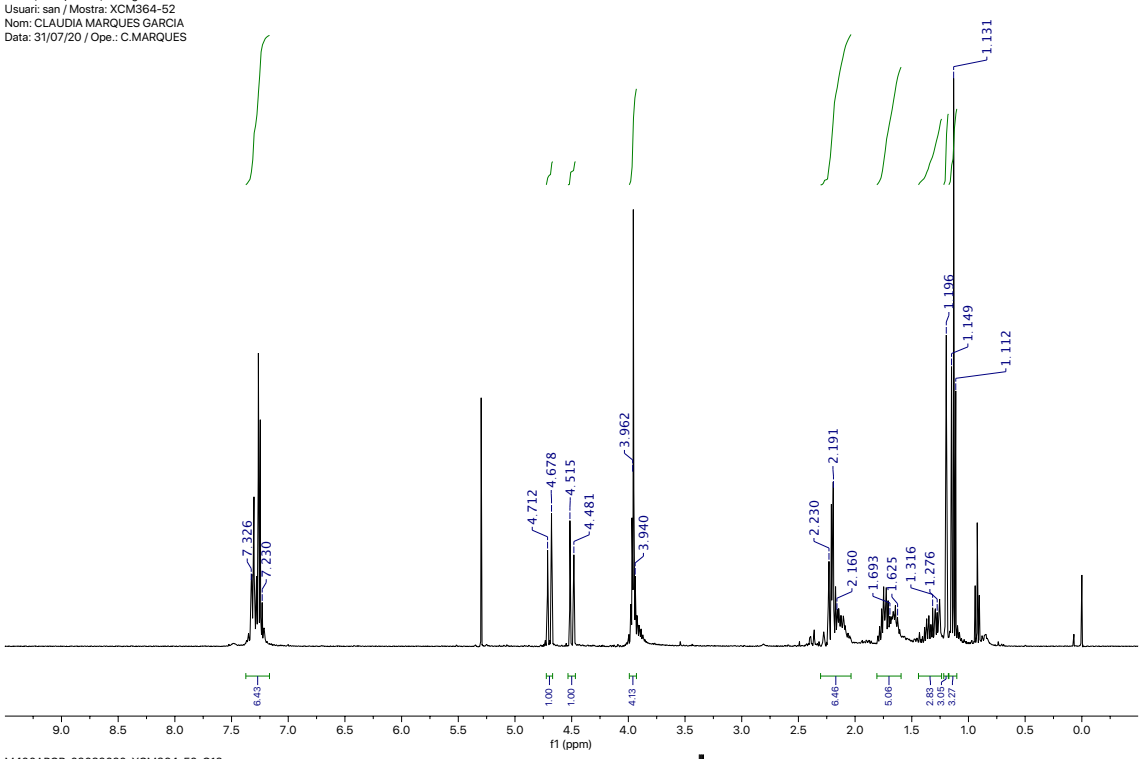
VNMRS400A_27022018_YCM003-30-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: YCM003-30
Nom: FAIZA DIABA
Data: 27/02/18 / Ope.: F.DIABA



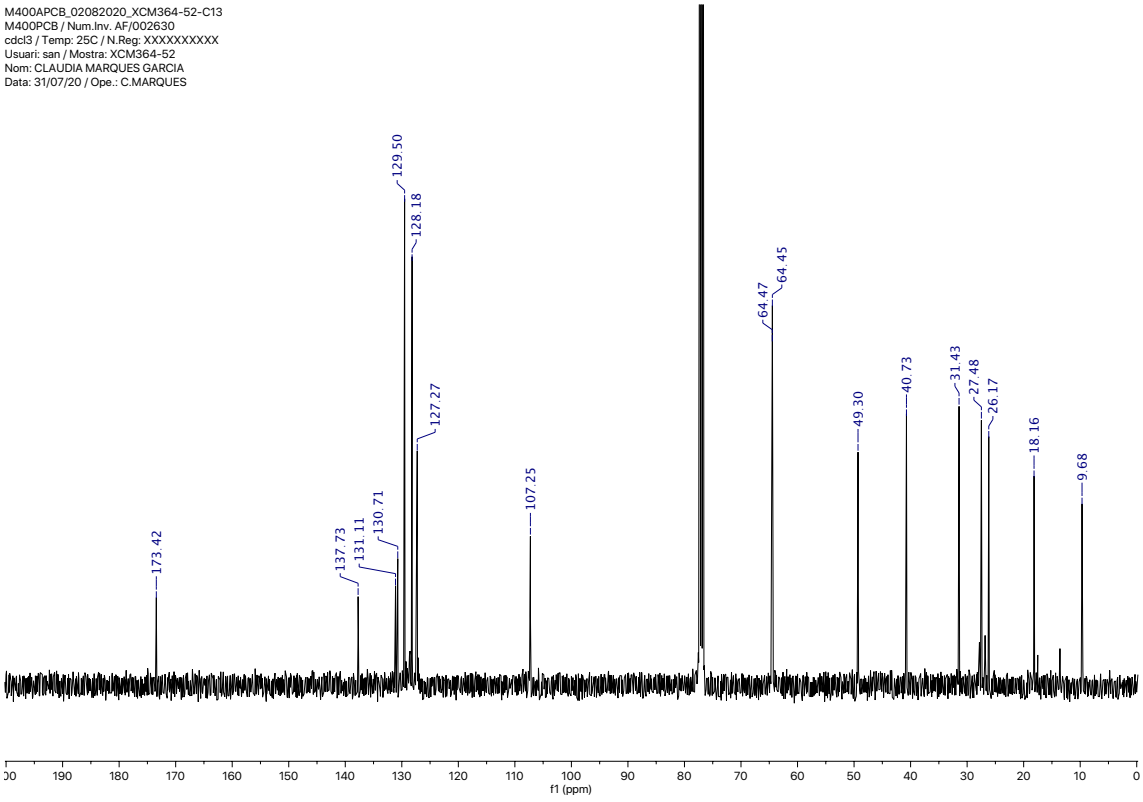


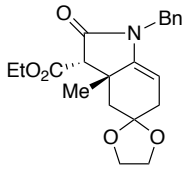
17

M400APCB_02082020_XCM364-52-H1
M400PCB / Num.Inv. AF/002630
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM364-52
Nom: CLAUDIA MARQUES GARCIA
Data: 31/07/20 / Ope.: C.MARQUES



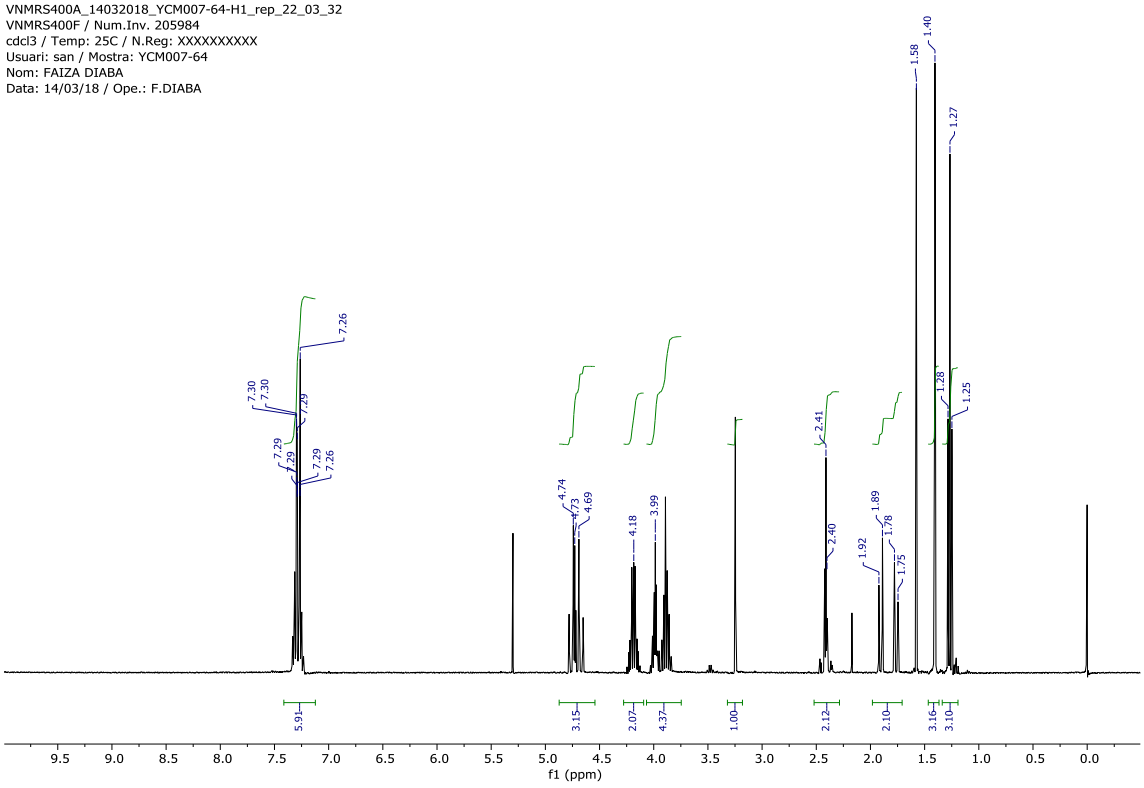
M400APCB_02082020_XCM364-52-C13
M400PCB / Num.Inv. AF/002630
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM364-52
Nom: CLAUDIA MARQUES GARCIA
Data: 31/07/20 / Ope.: C.MARQUES



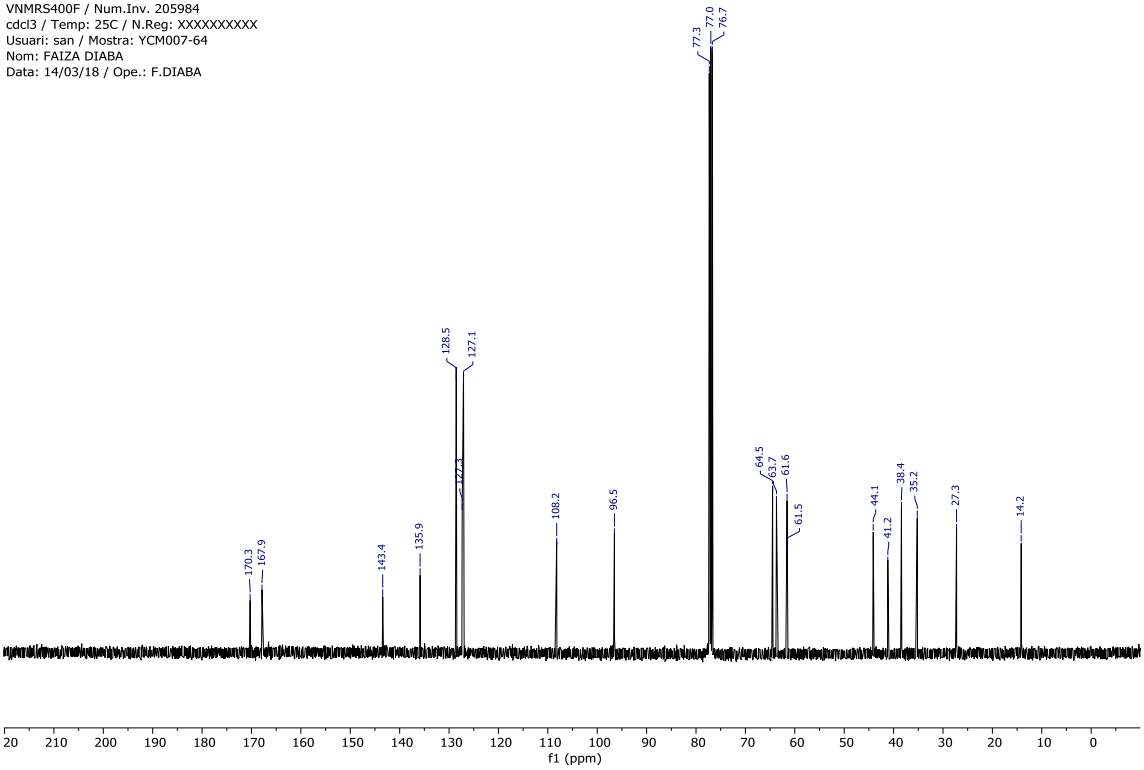


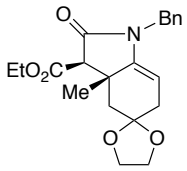
7a

VNMRS400A_14032018_YCM007-64-H1_rep_22_03_32
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: YCM007-64
Nom: FAIZA DIABA
Data: 14/03/18 / Ope.: F.DIABA

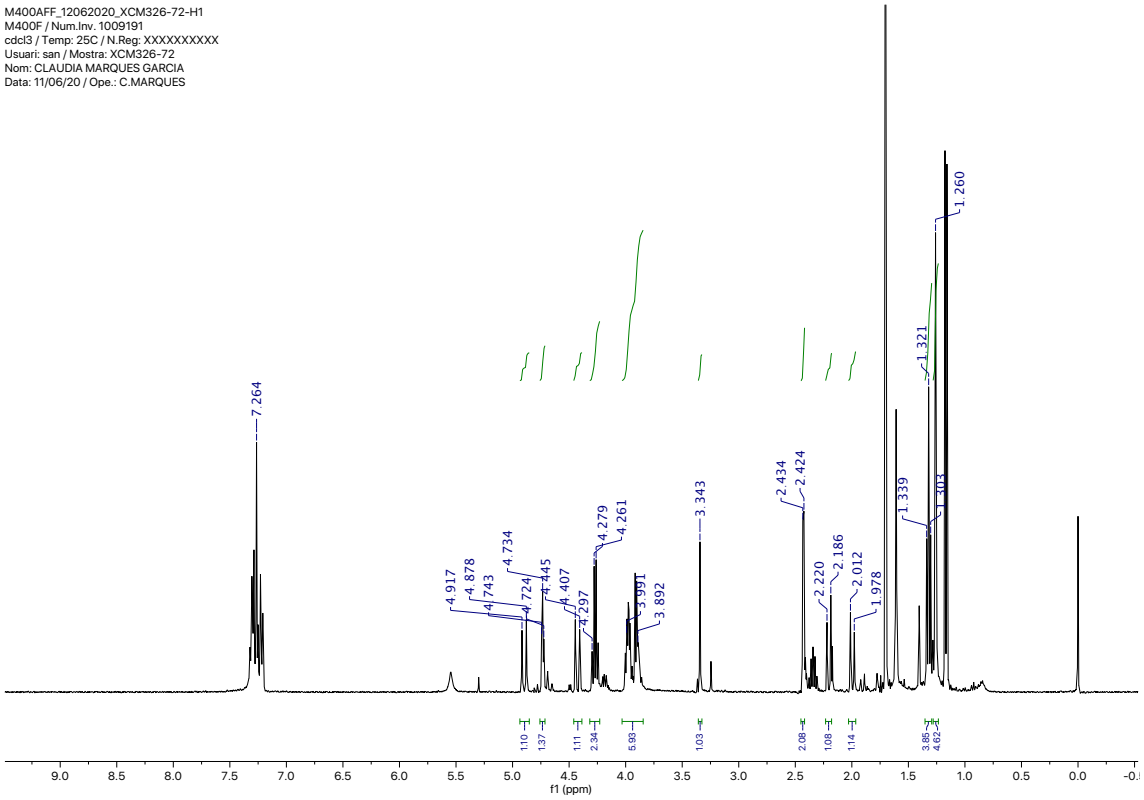


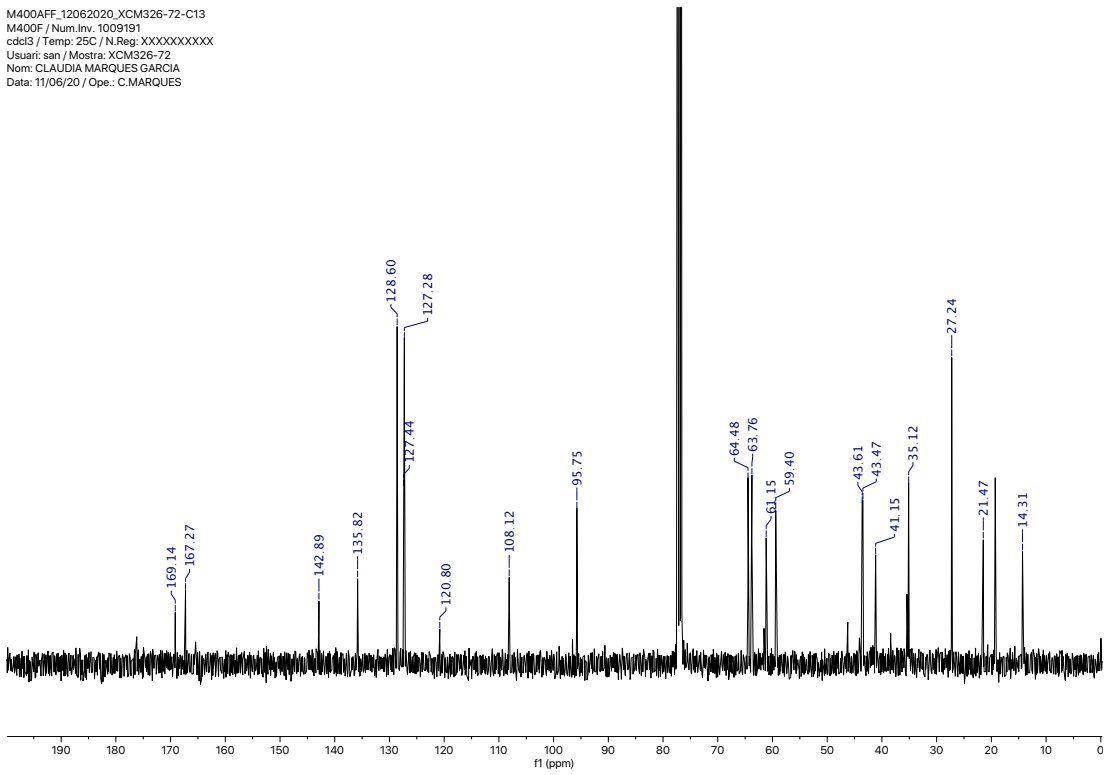
VNMRS400A_14032018_YCM007-64-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: YCM007-64
Nom: FAIZA DIABA
Data: 14/03/18 / Ope.: F.DIABA

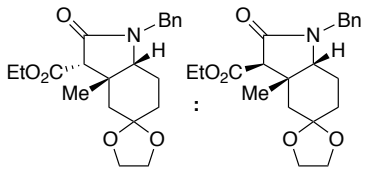



O
N
Bn
EtO2C
Me
O
O

7b

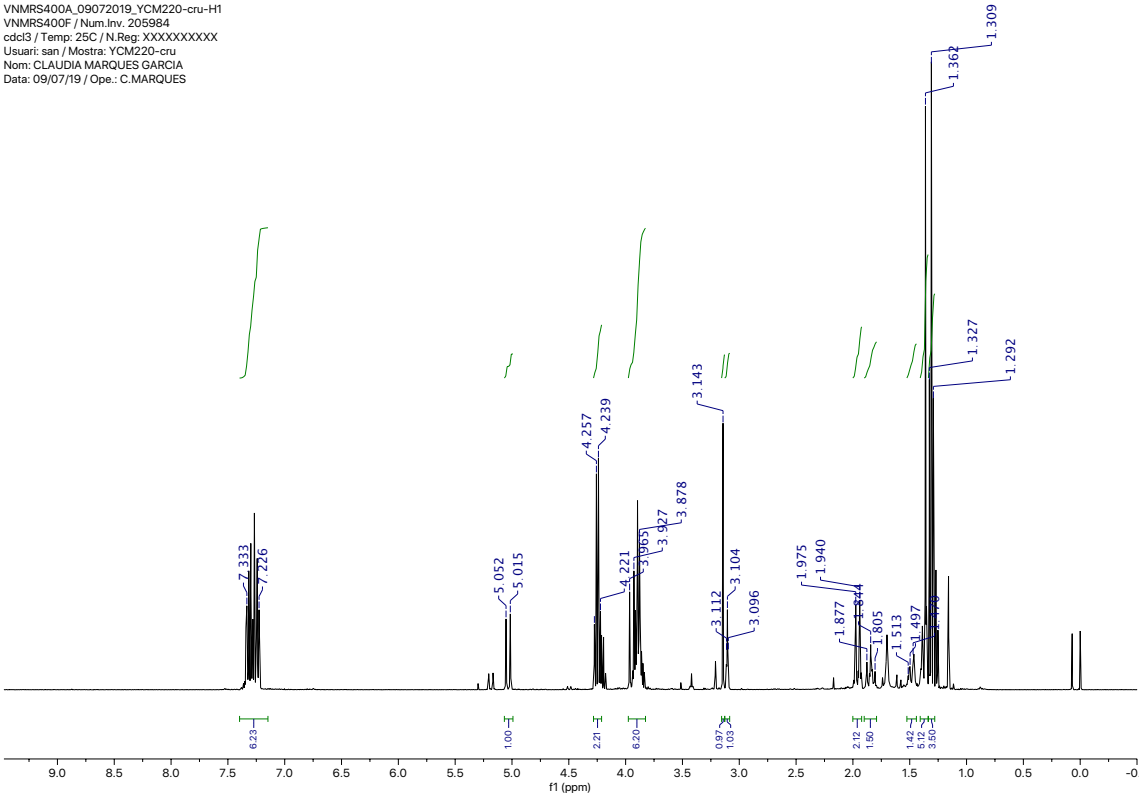

M400AFF_12062020_XCM326-72-H1
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM326-72
Nom: CLAUDIA MARQUES GARCIA
Data: 11/06/20 / Ope.: C.MARQUES
7.264
4.917
4.878
4.743
4.734
4.724
4.445
4.407
4.297
4.279
4.261
3.991
3.892
3.343
2.434
2.424
2.220
2.186
2.012
1.978
1.339
1.321
1.260
f1 (ppm)


M400AFF_12062020_XCM326-72-C13
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM326-72
Nom: CLAUDIA MARQUES GARCIA
Data: 11/06/20 / Ope.: C.MARQUES
169.14
167.27
142.89
135.82
128.60
127.28
127.44
120.80
108.12
95.75
64.48
63.76
61.15
59.40
43.61
43.47
41.15
35.12
27.24
21.47
14.31
f1 (ppm)

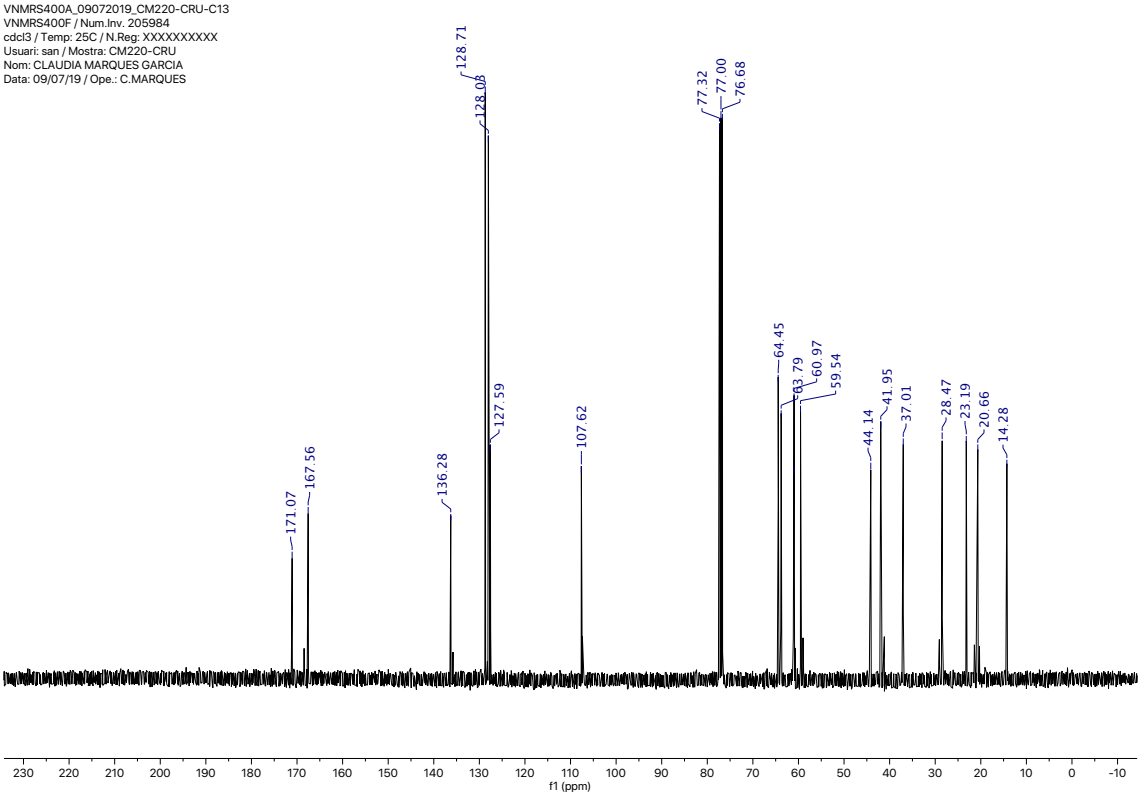


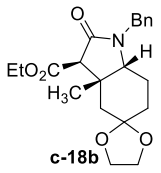
c-18a : **c-18b**

VNMRS400A_09072019_YCM220-cru-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: YCM220-cru
Nom: CLAUDIA MARQUES GARCIA
Data: 09/07/19 / Ope.: C.MARQUES

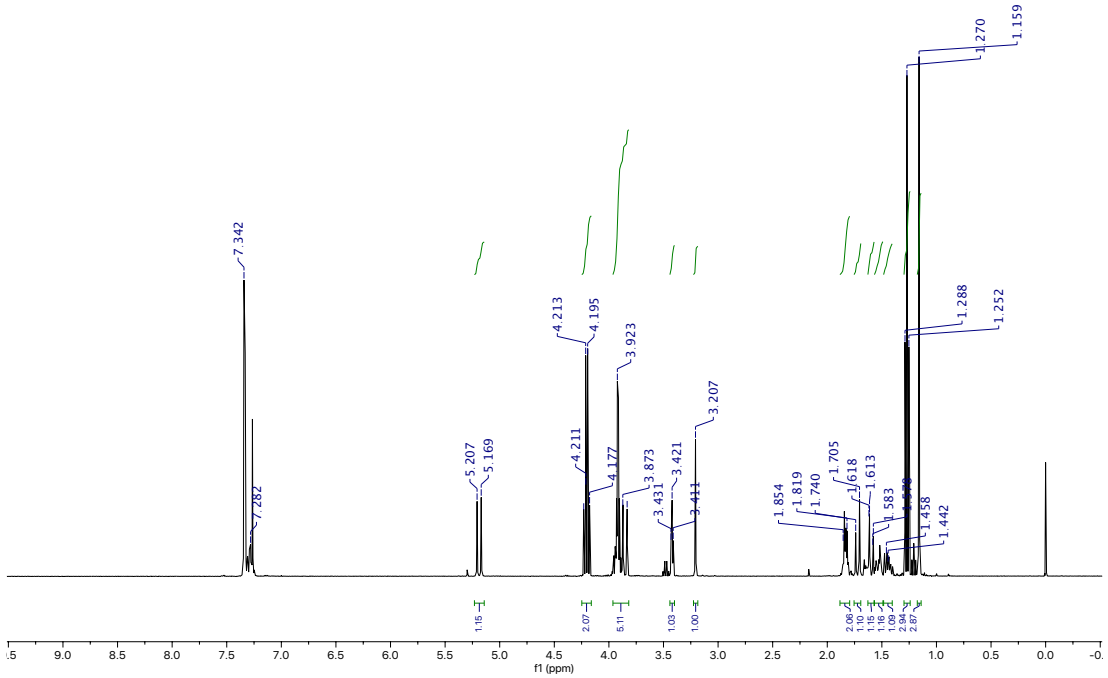


VNMRS400A_09072019_CM220-CRU-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: CM220-CRU
Nom: CLAUDIA MARQUES GARCIA
Data: 09/07/19 / Ope.: C.MARQUES

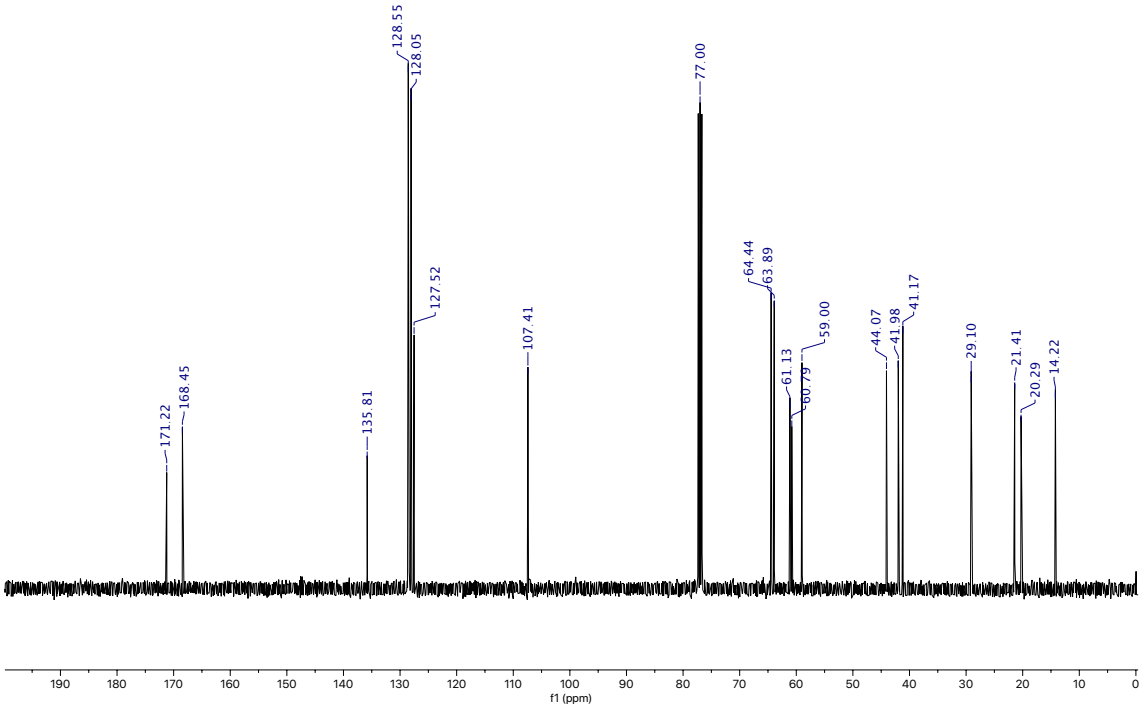


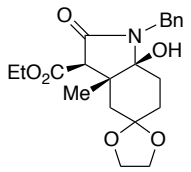


VNMRS400A_10052019_XCM110-176-CRIS-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM110-176-CRIS
Nom: CLAUDIA MARQUES GARCIA
Data: 10/05/19 / Ope.: C.MARQUES

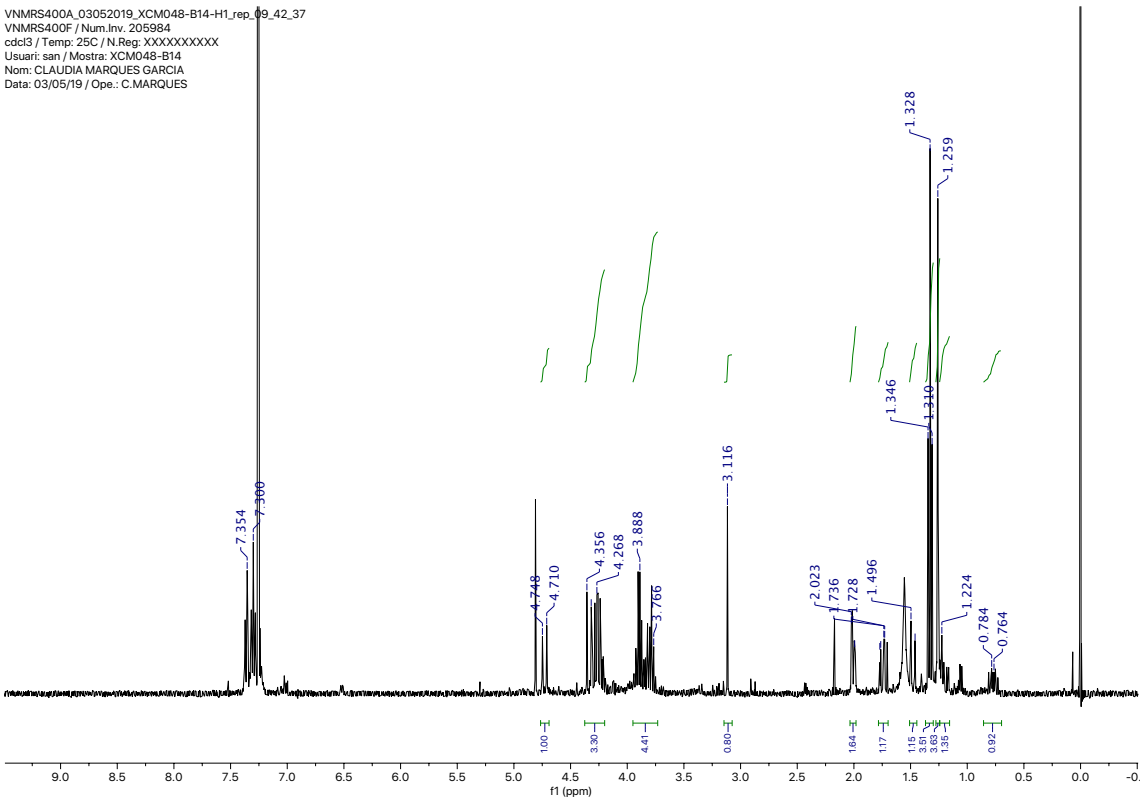


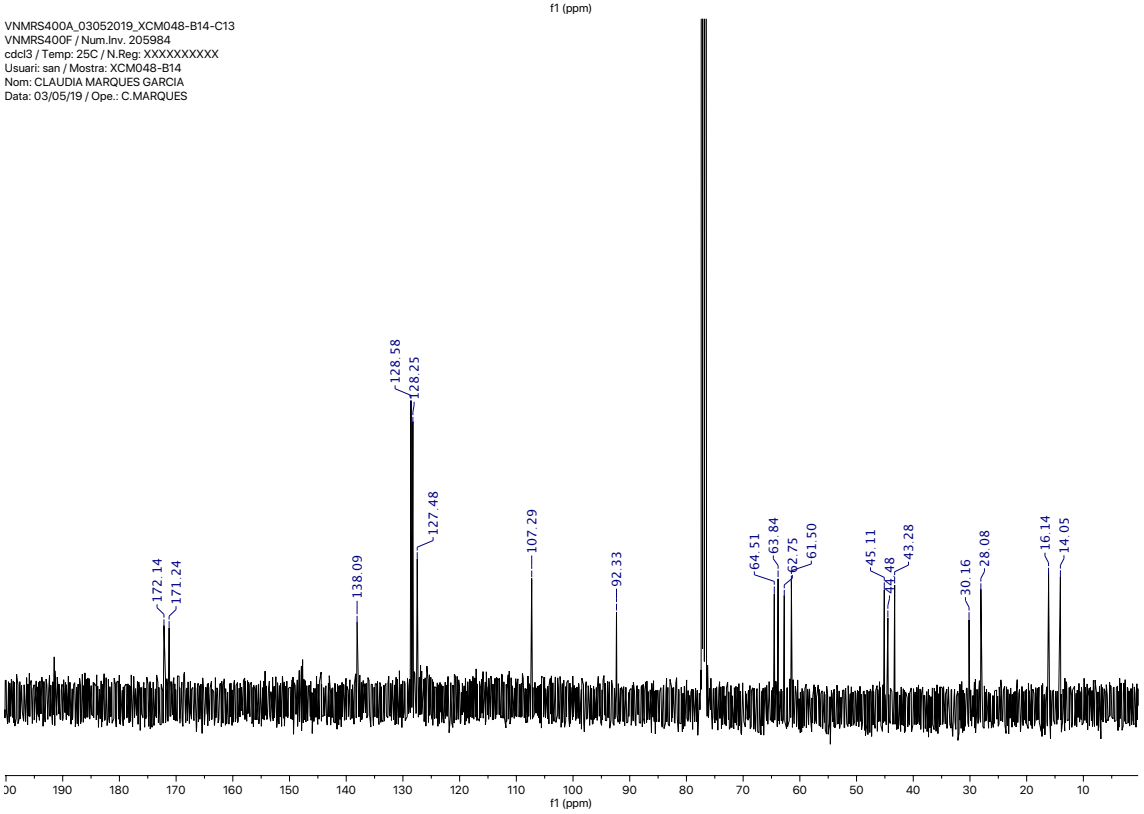
VNMRS400A_10052019_XCM110-176-CRIS-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM110-176-CRIS
Nom: CLAUDIA MARQUES GARCIA
Data: 10/05/19 / Ope.: C.MARQUES

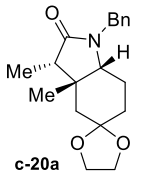



O
N
Bn
OH
EtO2C
Me
O
O

19

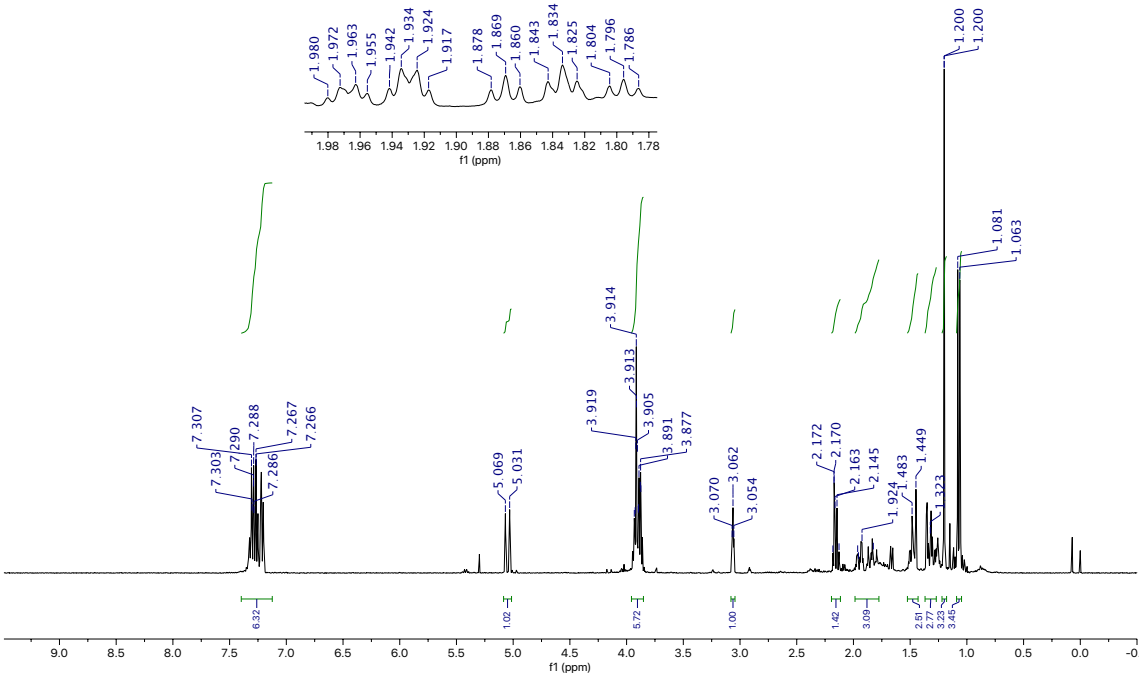

VNMRS400A_03052019_XCM048-B14-H1_rep_09_42_37
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM048-B14
Nom: CLAUDIA MARQUES GARCIA
Data: 03/05/19 / Ope.: C.MARQUES
7.354
7.360
4.748
4.710
4.356
4.268
3.888
3.766
3.116
2.023
1.736
1.728
1.496
1.346
1.328
1.310
1.259
1.224
0.784
0.764
1.00
3.30
4.41
0.80
1.64
1.17
1.15
3.91
3.63
1.35
0.92
f1 (ppm)


VNMRS400A_03052019_XCM048-B14-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM048-B14
Nom: CLAUDIA MARQUES GARCIA
Data: 03/05/19 / Ope.: C.MARQUES
172.14
171.24
138.09
128.58
128.25
127.48
107.29
92.33
64.51
63.84
62.75
61.50
45.11
44.48
43.28
30.16
28.08
16.14
14.05
f1 (ppm)

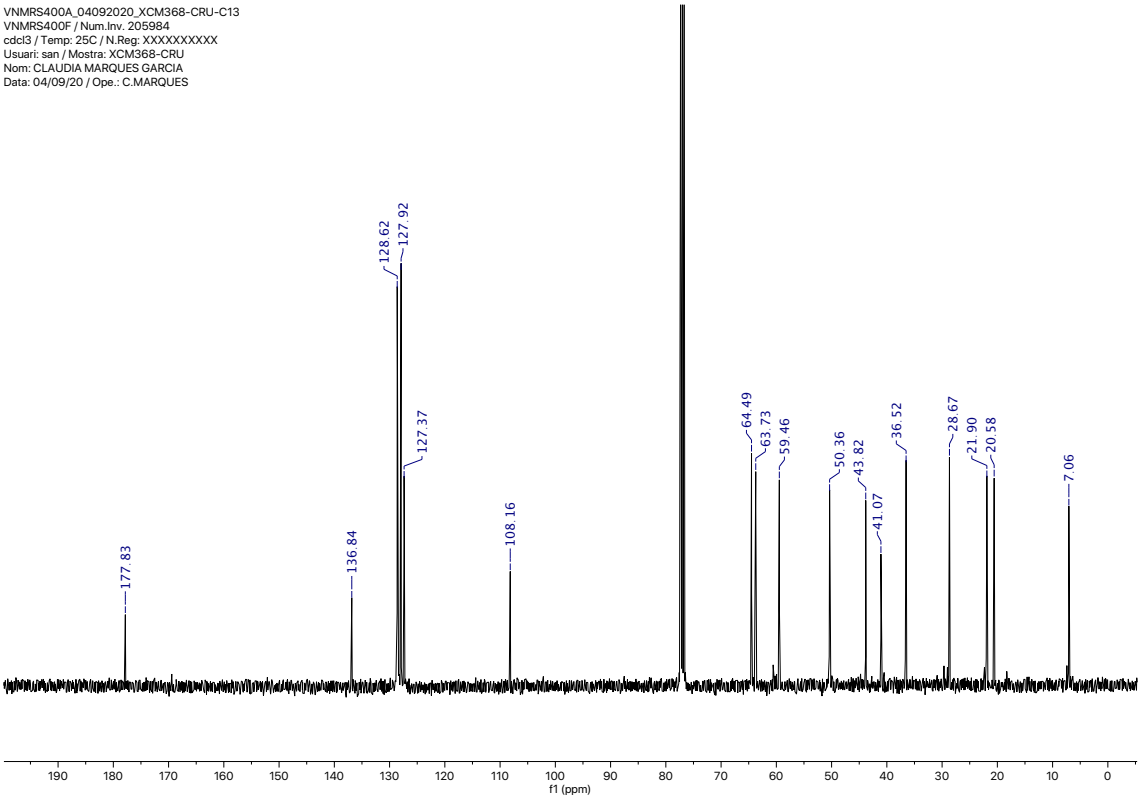


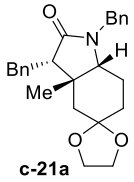
c-20a

VNMRS400A_04092020_XCM368-CRU-H1_rep_17_11_36
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM368-CRU
Nom: CLAUDIA MARQUES GARCIA
Data: 04/09/20 / Ope.: C.MARQUES



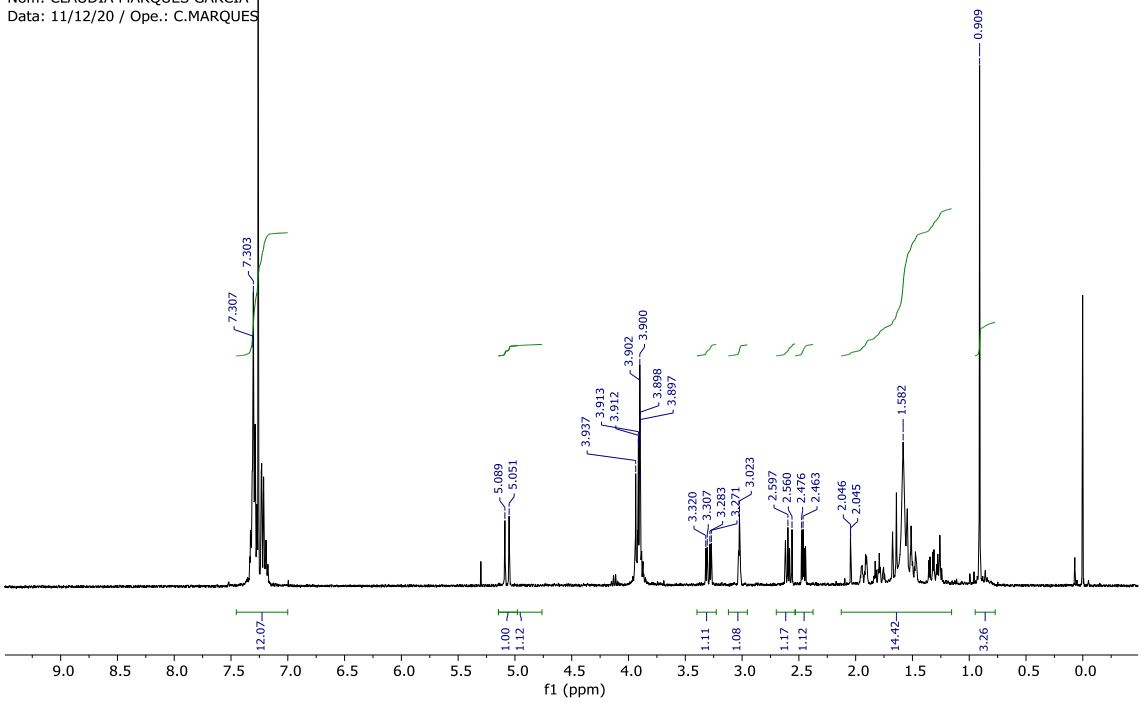
VNMRS400A_04092020_XCM368-CRU-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM368-CRU
Nom: CLAUDIA MARQUES GARCIA
Data: 04/09/20 / Ope.: C.MARQUES



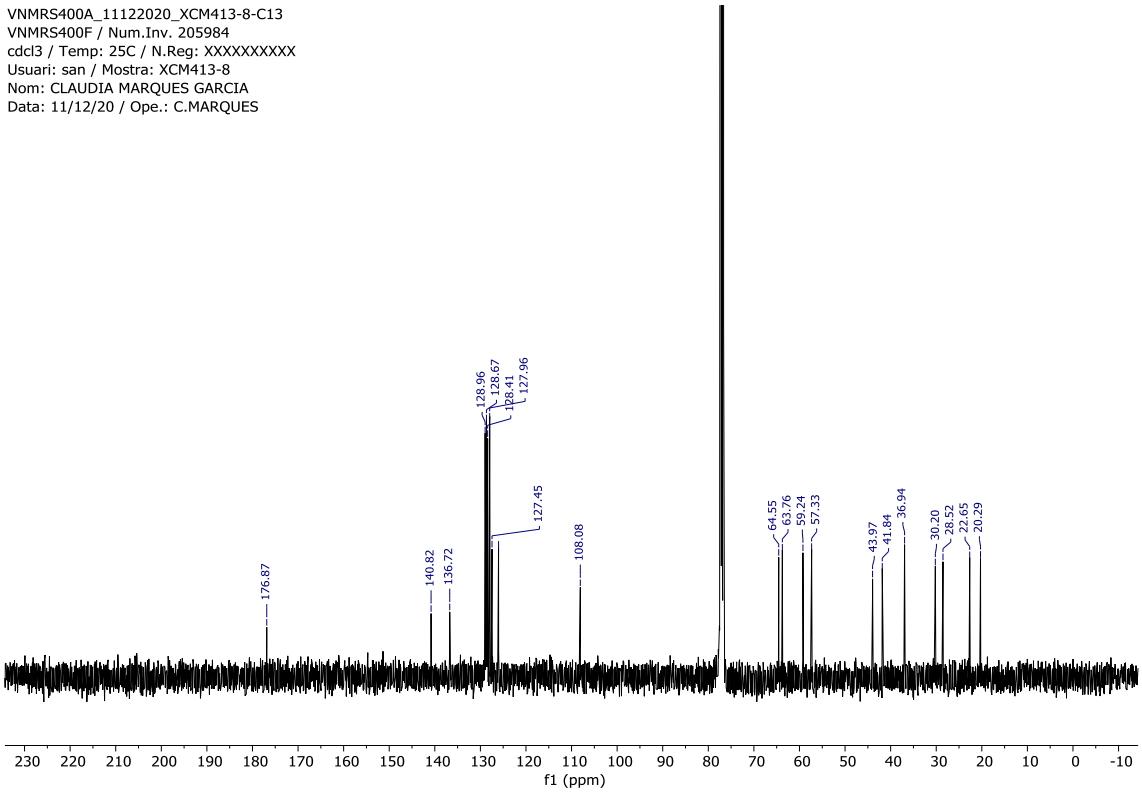


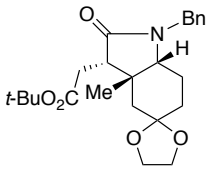
c-21a

VNMRS400A_11122020_XCM413-8-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM413-8
Nom: CLAUDIA MARQUES GARCIA
Data: 11/12/20 / Ope.: C.MARQUES

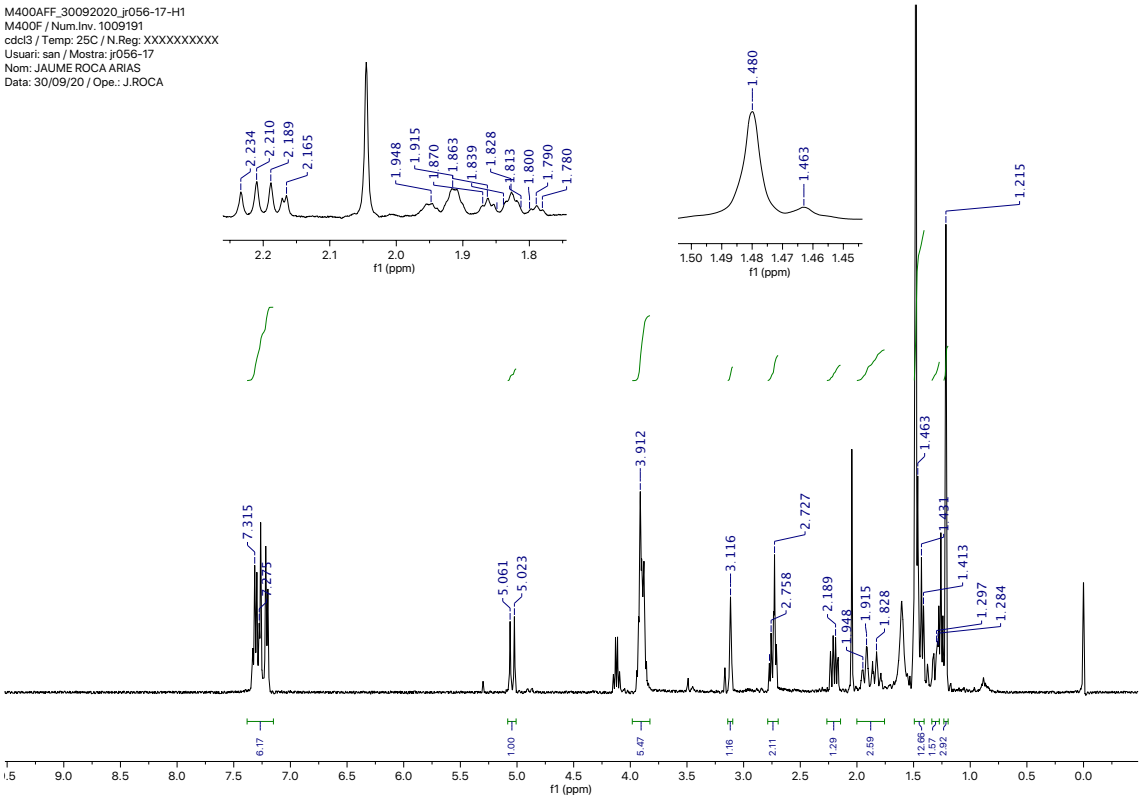


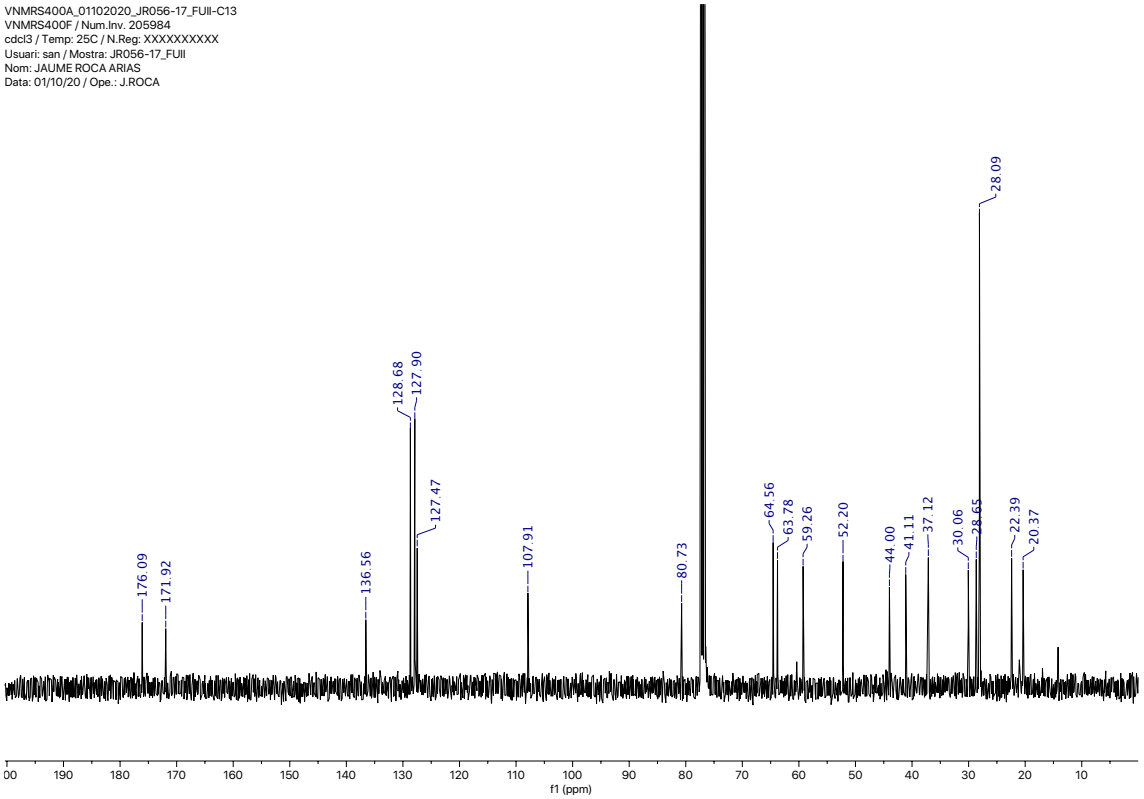
VNMRS400A_11122020_XCM413-8-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM413-8
Nom: CLAUDIA MARQUES GARCIA
Data: 11/12/20 / Ope.: C.MARQUES

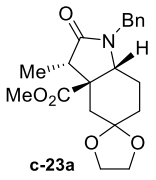



O
N
Bn
H
t-BuO2C
Me
O
O

c-22a

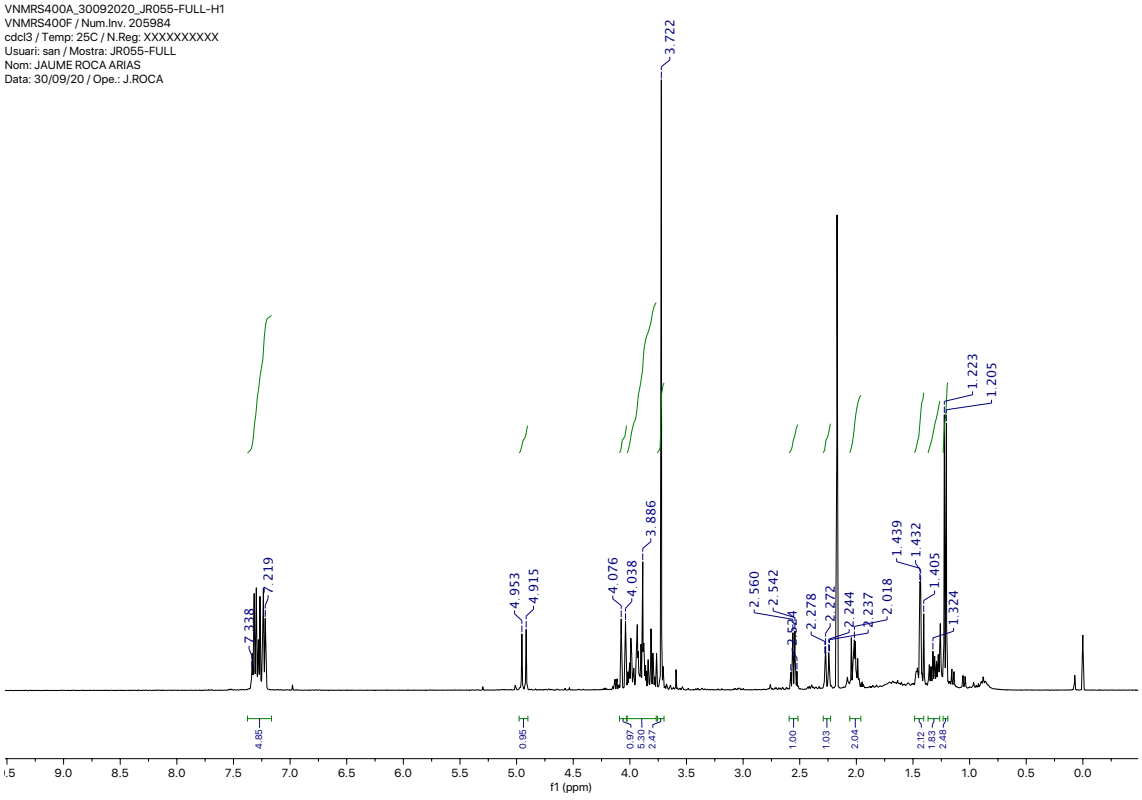

M400AFF_30092020_jr056-17-H1
M400F / Num.Inv. 1009191
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: jr056-17
Nom: JAUME ROCA ARIAS
Data: 30/09/20 / Ope.: J.ROCA
f1 (ppm)


VNMRS400A_01102020_JR056-17_FUII-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR056-17_FUII
Nom: JAUME ROCA ARIAS
Data: 01/10/20 / Ope.: J.ROCA
f1 (ppm)

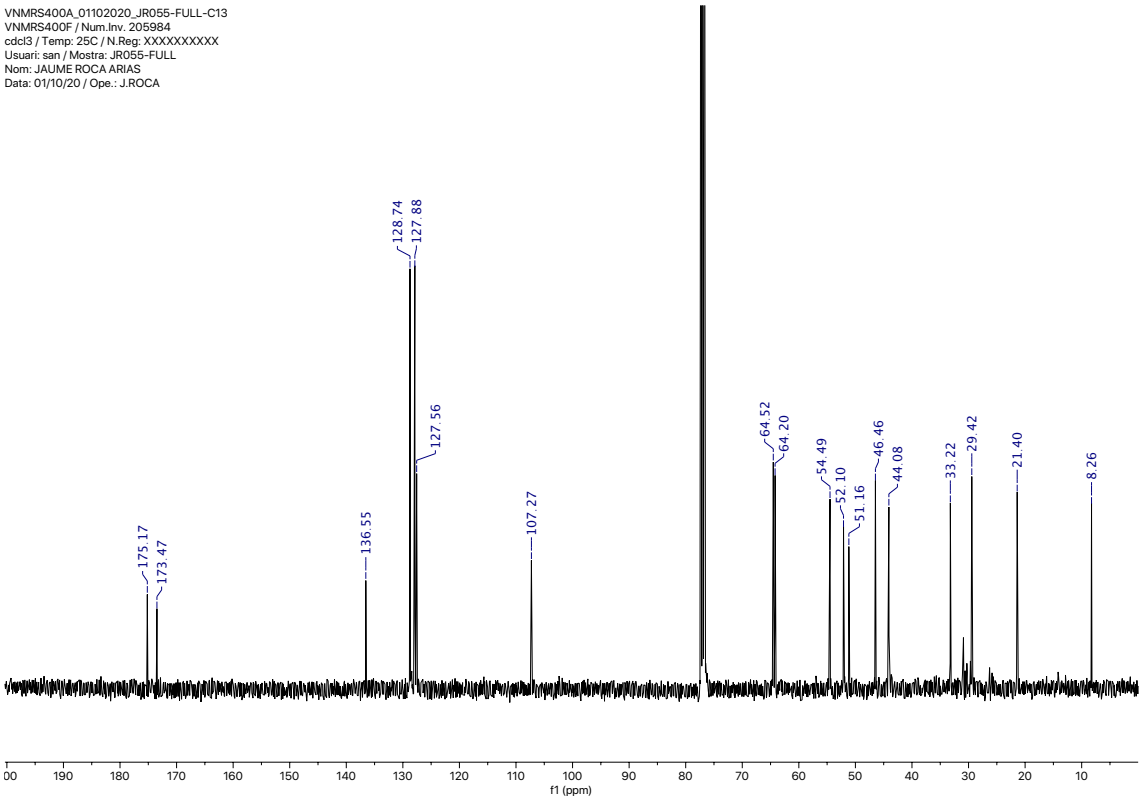


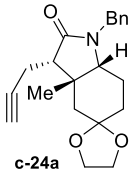
c-23a

VNMRS400A_30092020_JR055-FULL-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR055-FULL
Nom: JAUME ROCA ARIAS
Data: 30/09/20 / Ope.: J.ROCA

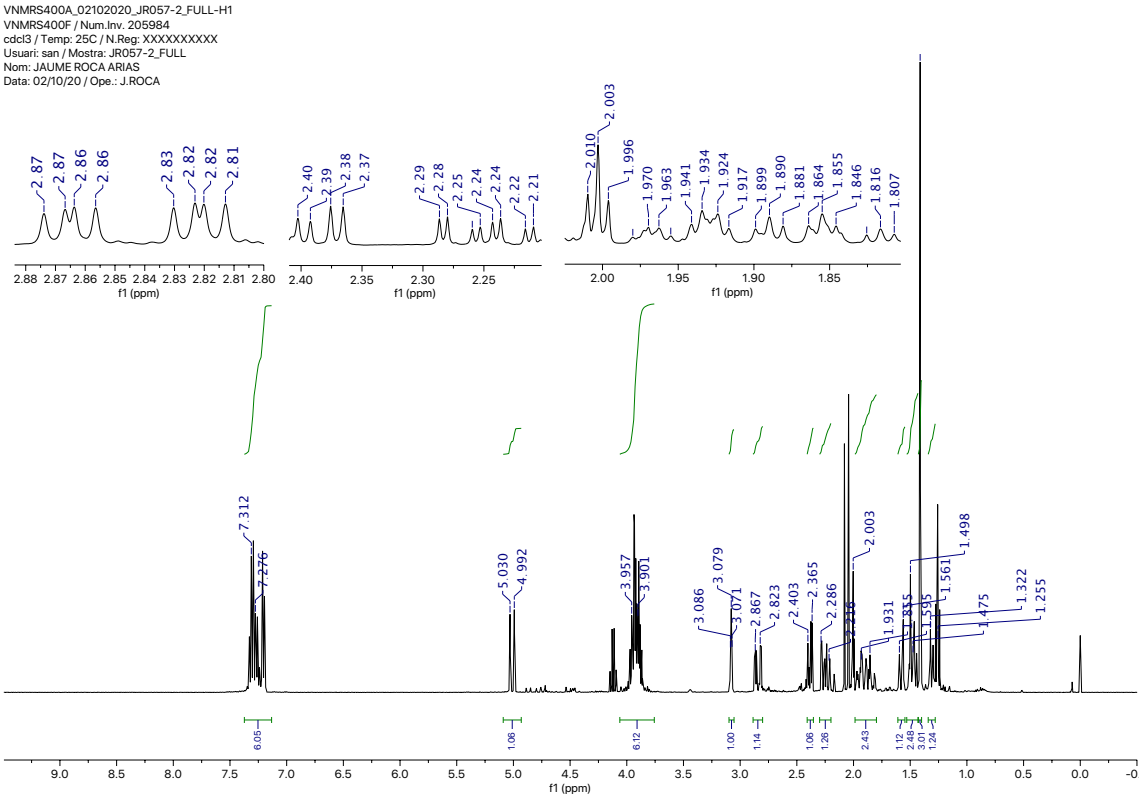


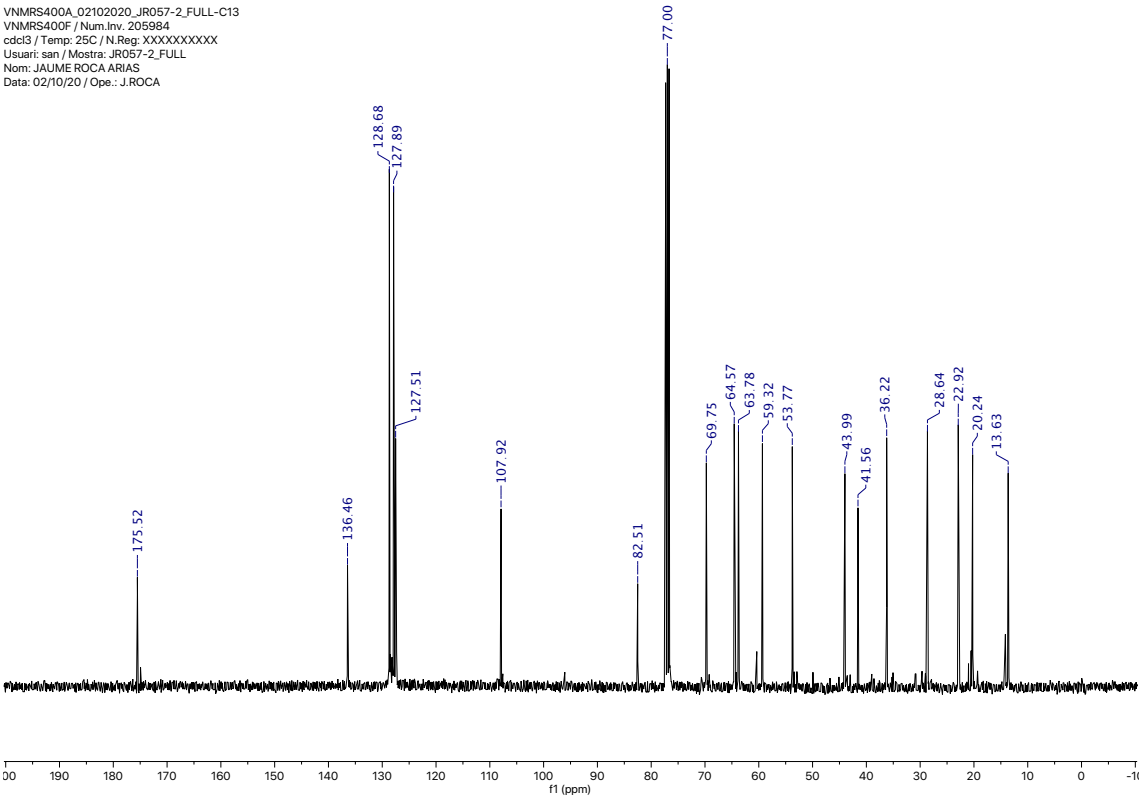
VNMRS400A_01102020_JR055-FULL-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR055-FULL
Nom: JAUME ROCA ARIAS
Data: 01/10/20 / Ope.: J.ROCA

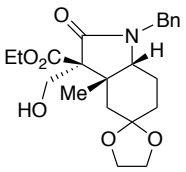



O
Bn
N
H
Me
O
O

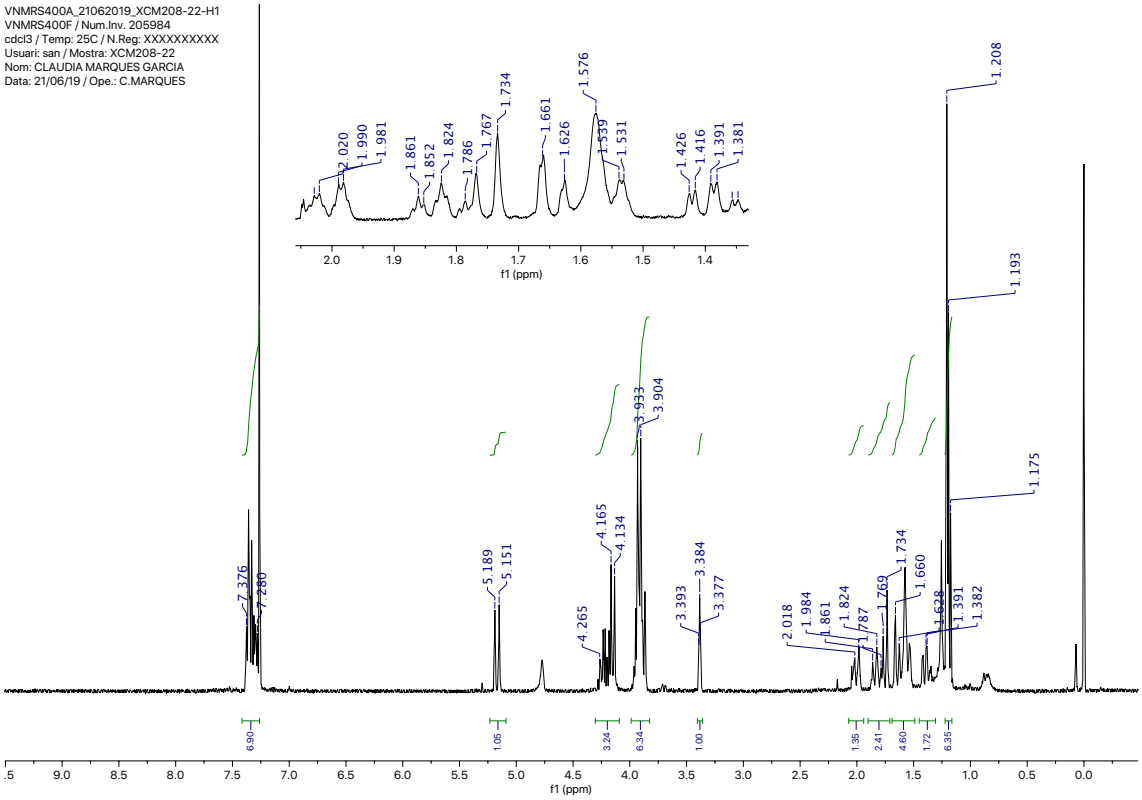
c-24a

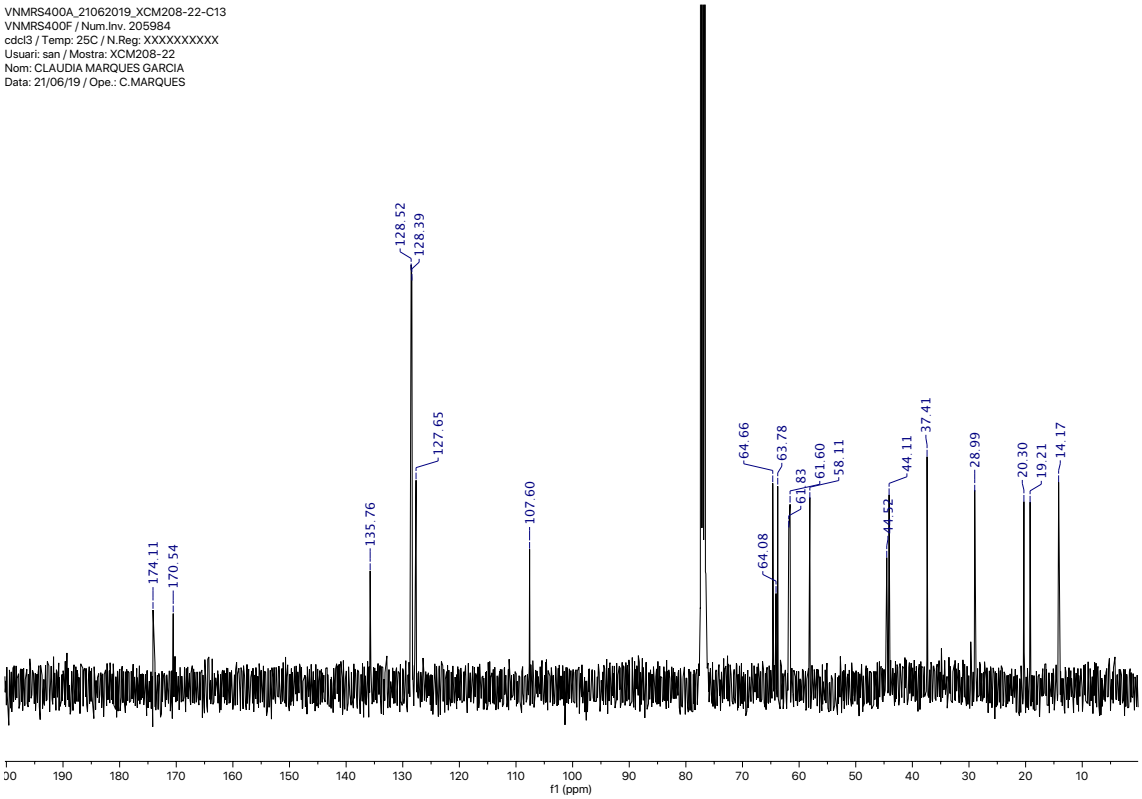

VNMRS400A_02102020_JR057-2_FULL-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR057-2_FULL
Nom: JAUME ROCA ARIAS
Data: 02/10/20 / Ope.: J.ROCA
f1 (ppm)

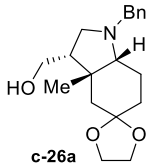

VNMRS400A_02102020_JR057-2_FULL-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR057-2_FULL
Nom: JAUME ROCA ARIAS
Data: 02/10/20 / Ope.: J.ROCA
f1 (ppm)


O
N
Bn
H
EtO2C
Me
HO
O
O

25

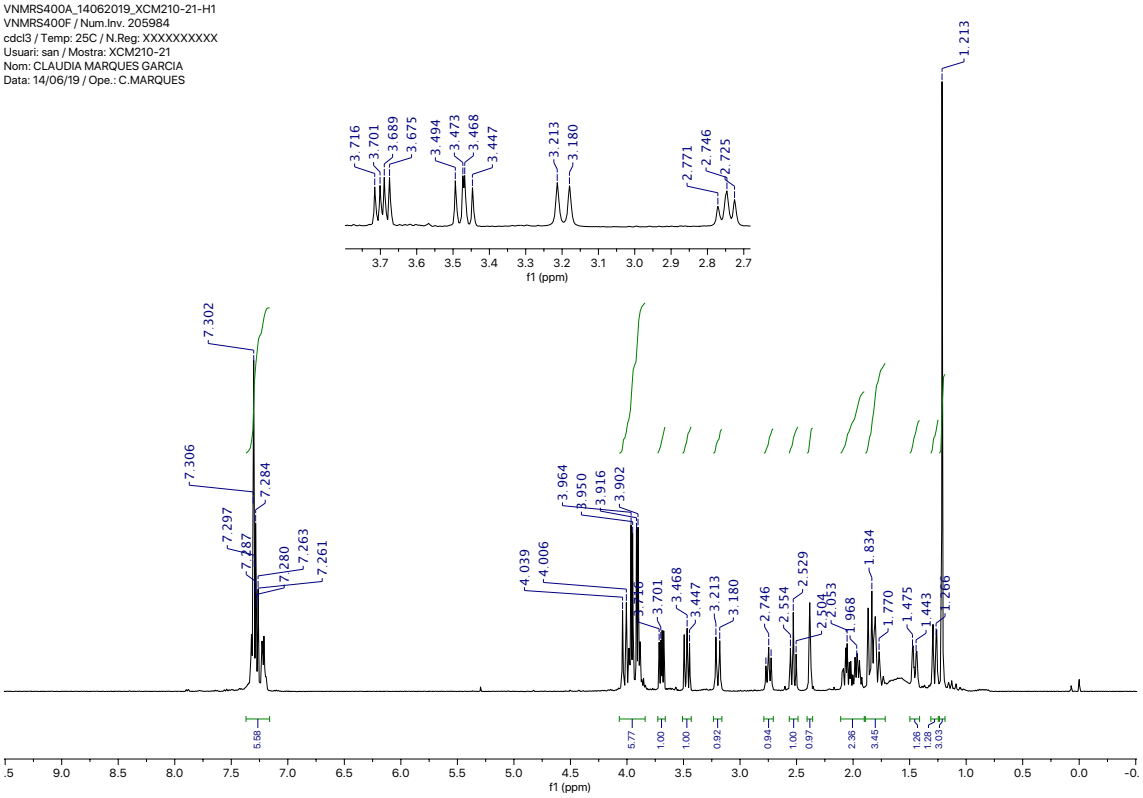

VNMRS400A_21062019_XCM208-22-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM208-22
Nom: CLAUDIA MARQUES GARCIA
Data: 21/06/19 / Ope.: C.MARQUES
f1 (ppm)


VNMRS400A_21062019_XCM208-22-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM208-22
Nom: CLAUDIA MARQUES GARCIA
Data: 21/06/19 / Ope.: C.MARQUES
f1 (ppm)

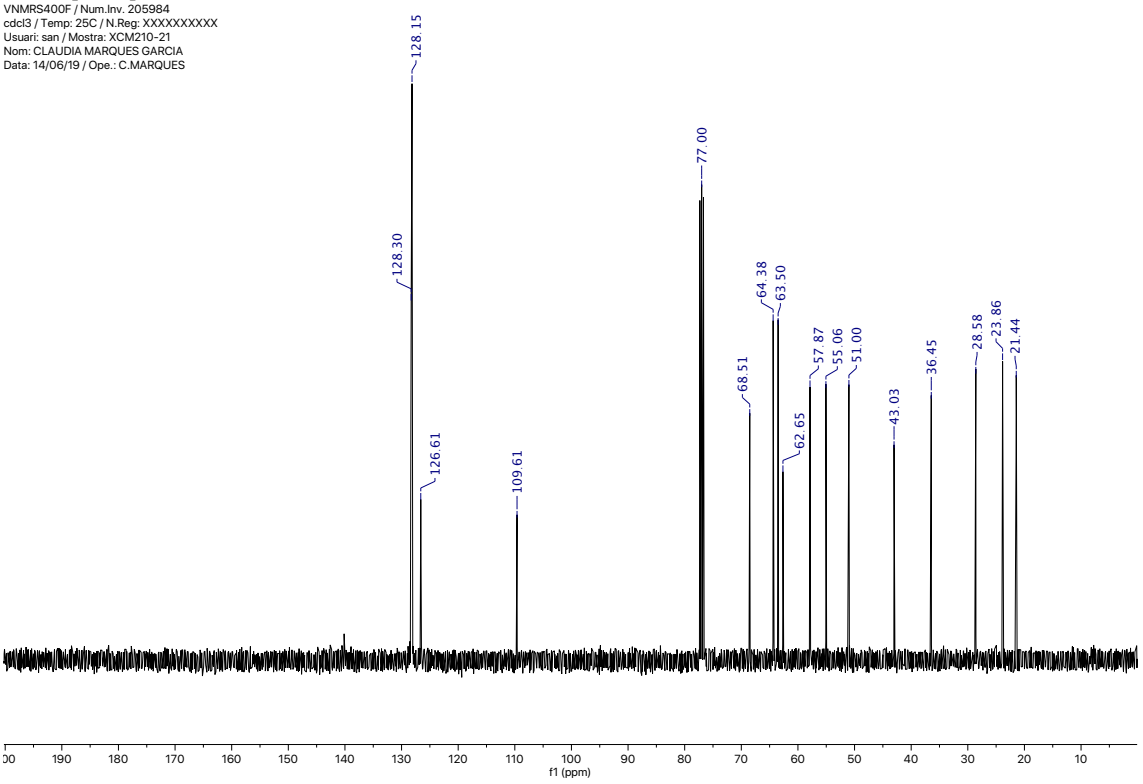


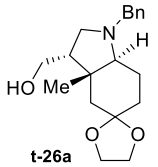
c-26a

VNMRS400A_14062019_XCM210-21-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM210-21
Nom: CLAUDIA MARQUES GARCIA
Data: 14/06/19 / Ope.: C.MARQUES



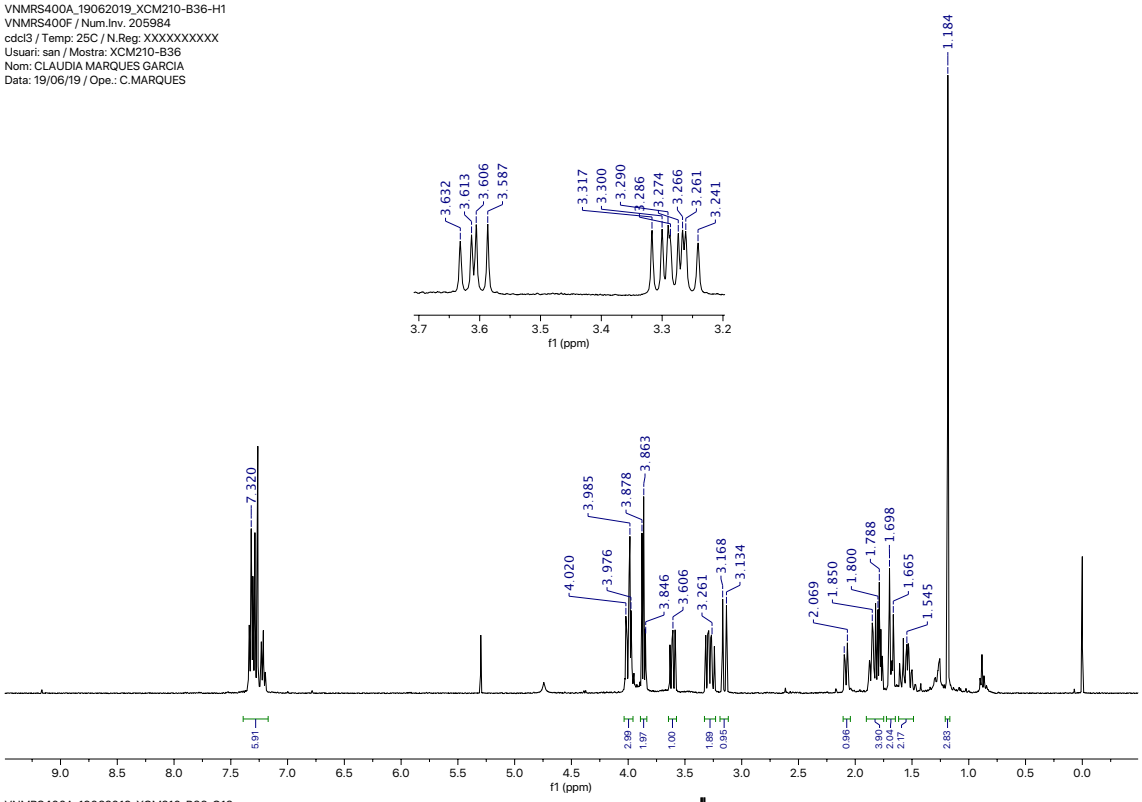
VNMRS400A_14062019_XCM210-21-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM210-21
Nom: CLAUDIA MARQUES GARCIA
Data: 14/06/19 / Ope.: C.MARQUES



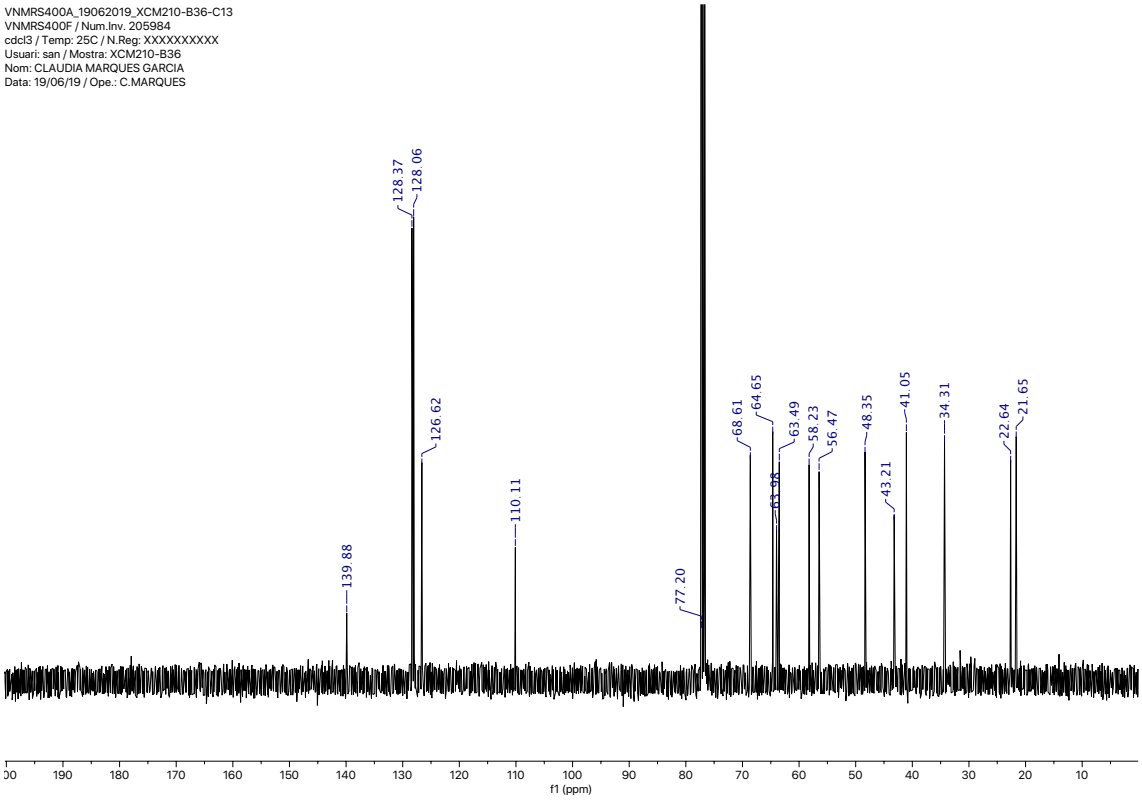


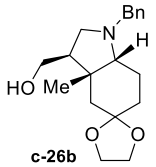
t-26a

VNMRS400A_19062019_XCM210-B36-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM210-B36
Nom: CLAUDIA MARQUES GARCIA
Data: 19/06/19 / Ope.: C.MARQUES



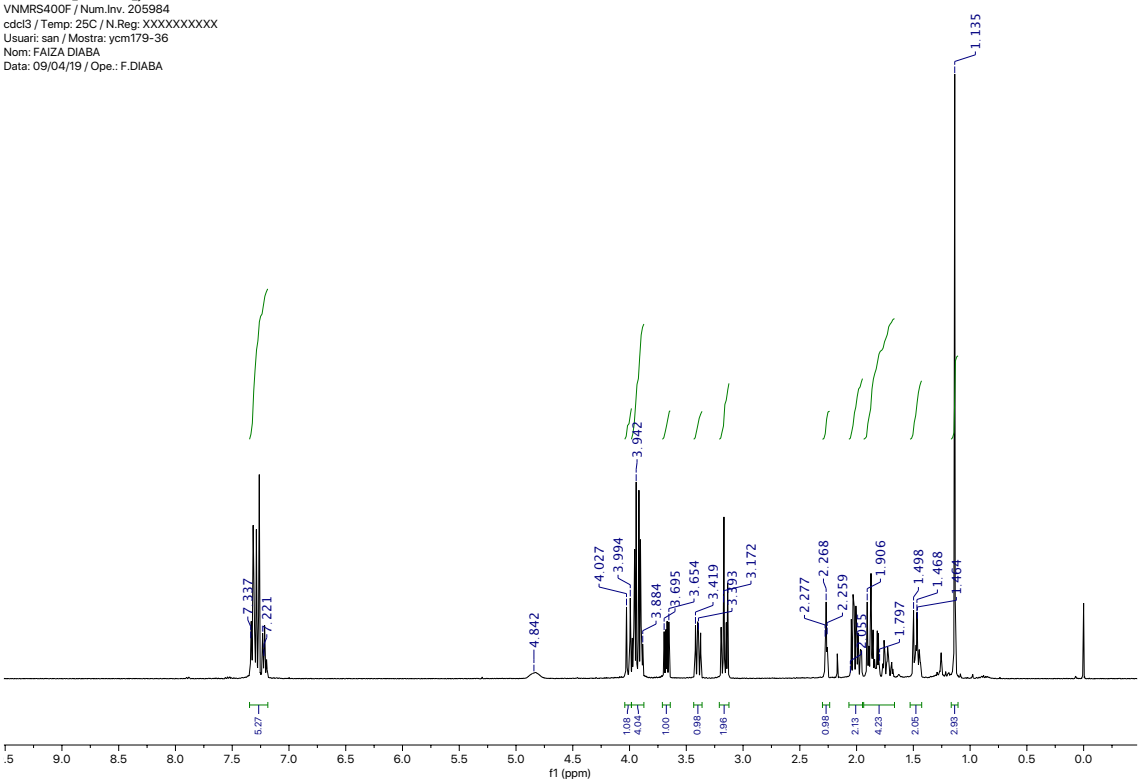
VNMRS400A_19062019_XCM210-B36-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: XCM210-B36
Nom: CLAUDIA MARQUES GARCIA
Data: 19/06/19 / Ope.: C.MARQUES



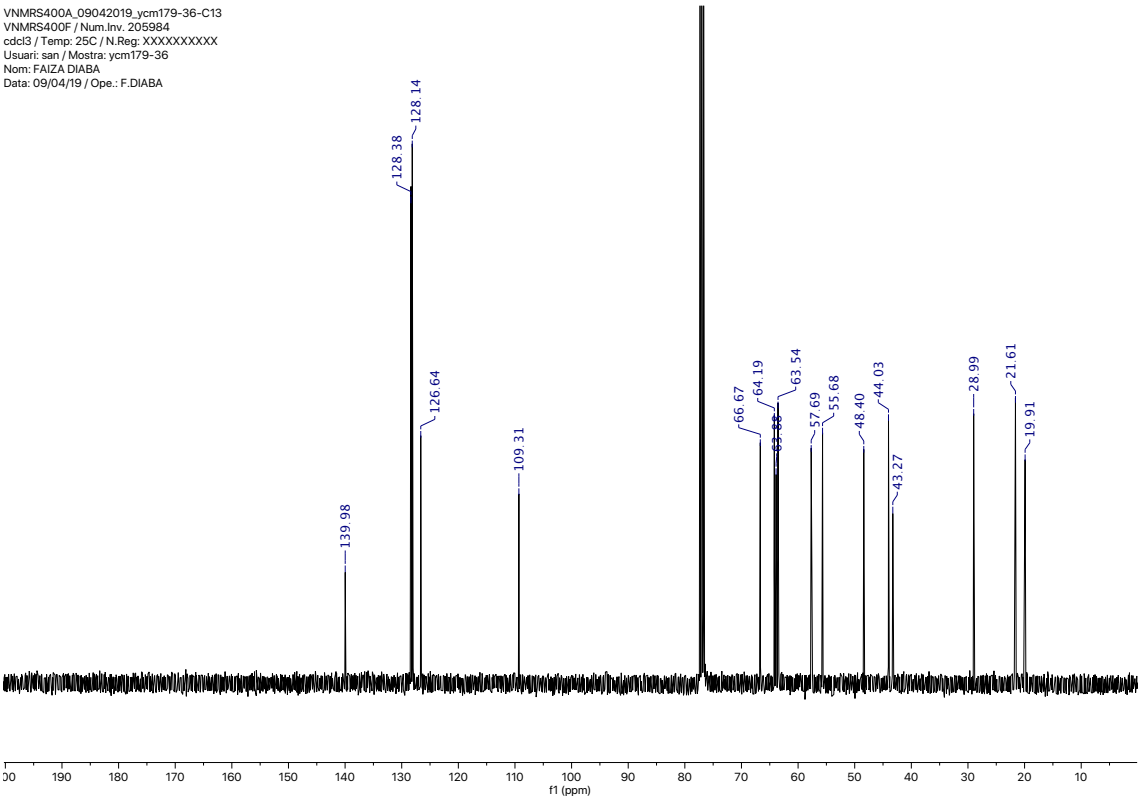


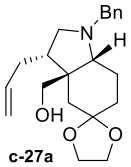
c-26b

VNMRS400A_09042019_ycm179-36-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: ycm179-36
Nom: FAIZA DIABA
Data: 09/04/19 / Ope.: F.DIABA

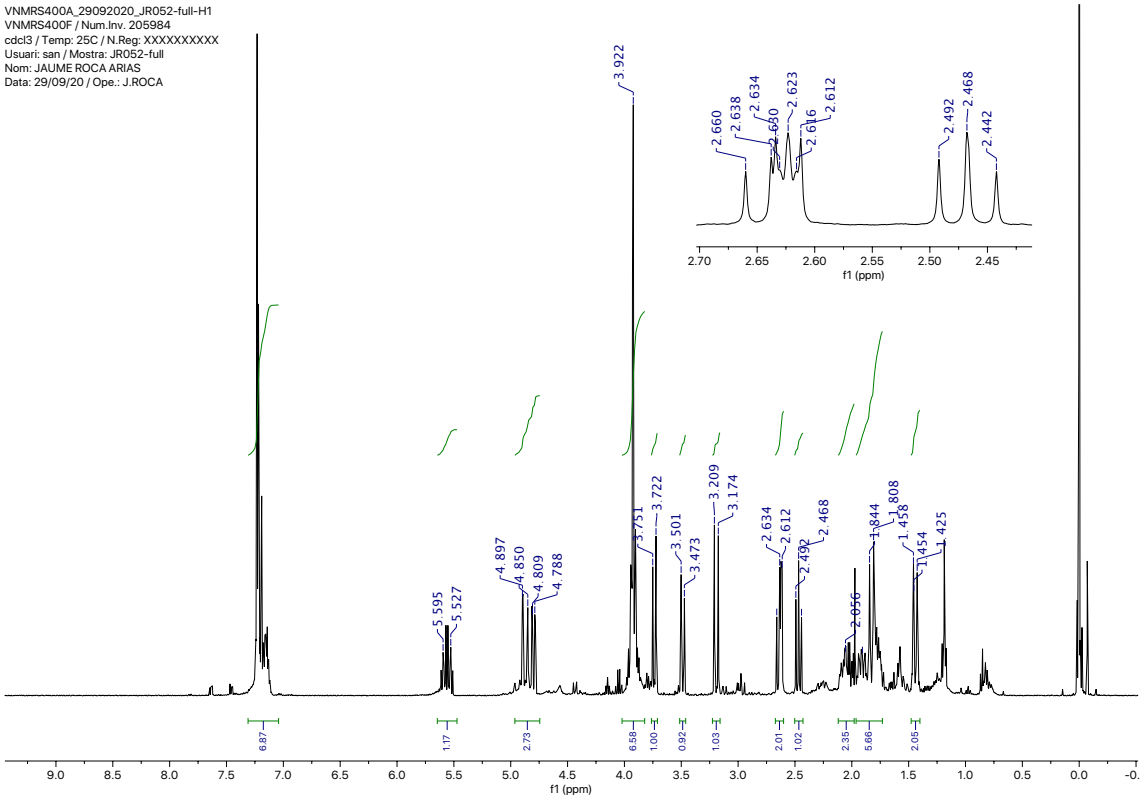


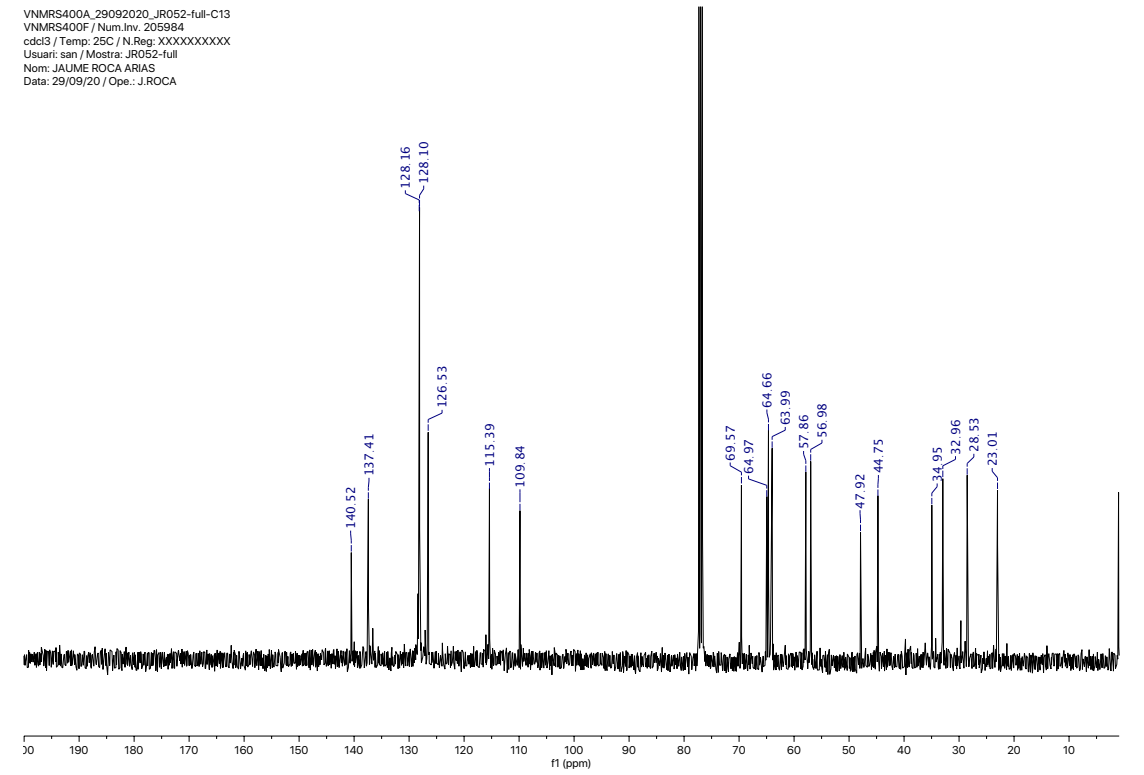
VNMRS400A_09042019_ycm179-36-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: ycm179-36
Nom: FAIZA DIABA
Data: 09/04/19 / Ope.: F.DIABA




Bn
N
H
OH
O
O

c-27a


VNMRS400A_29092020_JR052-full-H1
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR052-full
Nom: JAUME ROCA ARIAS
Data: 29/09/20 / Ope.: J.ROCA
3.922
2.660
2.638
2.634
2.630
2.623
2.616
2.612
2.492
2.468
2.442
f1 (ppm)
5.595
5.527
4.897
4.850
4.809
4.788
3.751
3.722
3.501
3.473
3.209
3.174
2.634
2.612
2.492
2.468
2.056
1.844
1.808
1.458
1.454
1.425
6.87
1.17
2.73
6.58
1.00
0.92
1.03
2.01
1.02
2.35
5.66
2.05
f1 (ppm)


VNMRS400A_29092020_JR052-full-C13
VNMRS400F / Num.Inv. 205984
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: san / Mostra: JR052-full
Nom: JAUME ROCA ARIAS
Data: 29/09/20 / Ope.: J.ROCA
128.16
128.10
126.53
140.52
137.41
115.39
109.84
69.57
64.97
64.66
63.99
57.86
56.98
47.92
44.75
34.95
32.96
28.53
23.01
f1 (ppm)

6. X-ray Crystallography Data

Accesion Codes

CCDC 2047318 (compound **7a**) and 2047315 (compound **c-18b**) 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 or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge CrystallographicData Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033

Compound 7a

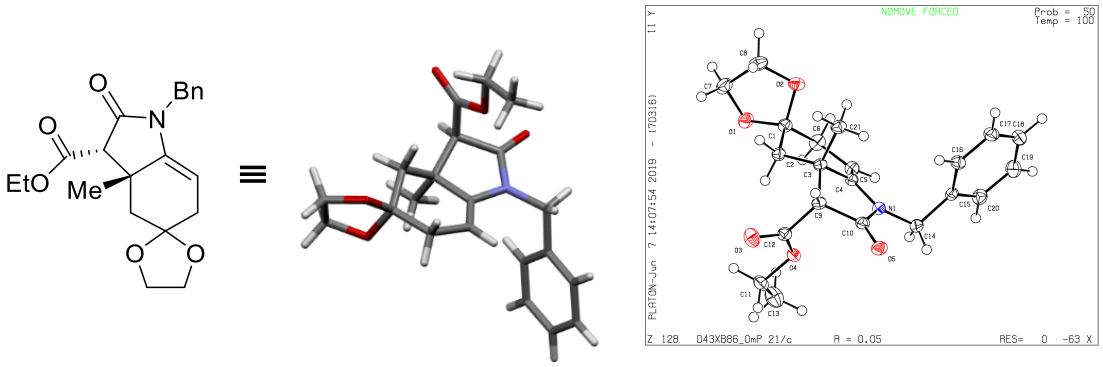


Table 1. Crystal data and structure refinement for D43XB86_0m_a.

Identification code	D43XB86_0m_a	
Empirical formula	C21 H25 N O5	
Formula weight	371.42	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 8.443(3) Å	α= 90°.
	b = 7.818(3) Å	β= 96.255(12)°.
	c = 29.113(11) Å	γ = 90°.
Volume	1910.3(12) $Å^3$	
Z	4	
Density (calculated)	1.291 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	792	
Crystal size	0.608 x 0.314 x 0.264 mm^3	
Theta range for data collection	2.427 to 35.572°.	
Index ranges	-13<=h<=9, -12<=k<=12, -47<=l<=46	
Reflections collected	35945	
Independent reflections	8666 [R(int) = 0.0317]	

Completeness to theta = 25.242°	99.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7473 and 0.6610
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8666 / 0 / 246
Goodness-of-fit on F^2	1.049
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.1164
R indices (all data)	R1 = 0.0654, wR2 = 0.1356
Extinction coefficient	n/a
Largest diff. peak and hole	0.452 and -0.270 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for D43XB86_0m_a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	7832(1)	-80(1)	4633(1)	25(1)
O(2)	5863(1)	1840(1)	4688(1)	23(1)
O(3)	11847(1)	5062(1)	4392(1)	31(1)
O(4)	10755(1)	4020(1)	3708(1)	21(1)
O(5)	8952(1)	7878(1)	3468(1)	25(1)
N(1)	7371(1)	5466(1)	3442(1)	19(1)
C(1)	7151(1)	1461(1)	4429(1)	19(1)
C(2)	8414(1)	2885(1)	4469(1)	19(1)
C(3)	7822(1)	4518(1)	4213(1)	17(1)
C(4)	7128(1)	4053(1)	3728(1)	17(1)
C(5)	6472(1)	2554(1)	3600(1)	21(1)
C(6)	6500(1)	1063(1)	3929(1)	23(1)
C(7)	7653(1)	8(2)	5118(1)	29(1)
C(8)	6423(1)	1434(1)	5160(1)	28(1)
C(9)	9144(1)	5829(1)	4114(1)	19(1)
C(10)	8507(1)	6561(1)	3641(1)	20(1)
C(11)	12200(1)	3024(1)	3672(1)	25(1)
C(12)	10745(1)	4970(1)	4090(1)	20(1)
C(13)	11847(2)	1829(2)	3270(1)	37(1)
C(14)	6698(1)	5601(1)	2961(1)	22(1)
C(15)	4915(1)	5904(1)	2905(1)	19(1)
C(16)	4186(1)	6888(1)	3224(1)	22(1)

C(17)	2551(1)	7196(1)	3159(1)	24(1)
C(18)	1631(1)	6546(1)	2773(1)	27(1)
C(19)	2343(1)	5573(2)	2454(1)	29(1)
C(20)	3978(1)	5247(1)	2521(1)	24(1)
C(21)	6572(1)	5508(1)	4462(1)	22(1)

Table 3. Bond lengths [Å] and angles [°] for D43XB86_0m_a.

O(1)-C(1)	1.4352(12)
O(1)-C(7)	1.4380(14)
O(2)-C(1)	1.4195(12)
O(2)-C(8)	1.4396(14)
O(3)-C(12)	1.2097(12)
O(4)-C(12)	1.3380(12)
O(4)-C(11)	1.4603(12)
O(5)-C(10)	1.2237(12)
N(1)-C(10)	1.3668(13)
N(1)-C(4)	1.4122(12)
N(1)-C(14)	1.4554(13)
C(1)-C(6)	1.5299(14)
C(1)-C(2)	1.5375(13)
C(2)-C(3)	1.5345(13)
C(2)-H(2A)	0.9900
C(2)-H(2AB)	0.9900
C(3)-C(4)	1.5141(13)
C(3)-C(21)	1.5497(13)
C(3)-C(9)	1.5650(13)
C(4)-C(5)	1.3315(13)
C(5)-C(6)	1.5072(14)
C(5)-H(5)	0.9500
C(6)-H(6A)	0.9900
C(6)-H(6AB)	0.9900
C(7)-C(8)	1.5376(17)
C(7)-H(7A)	0.9900
C(7)-H(7AB)	0.9900
C(8)-H(8A)	0.9900
C(8)-H(8AB)	0.9900
C(9)-C(12)	1.5178(14)
C(9)-C(10)	1.5341(14)
C(9)-H(9)	1.0000
C(11)-C(13)	1.5014(17)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(13)-H(13A)	0.9800

C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(15)	1.5150(14)
C(14)-H(14A)	0.9900
C(14)-H(14B)	0.9900
C(15)-C(20)	1.3942(14)
C(15)-C(16)	1.3988(13)
C(16)-C(17)	1.3938(14)
C(16)-H(16)	0.9500
C(17)-C(18)	1.3911(16)
C(17)-H(17)	0.9500
C(18)-C(19)	1.3886(17)
C(18)-H(18)	0.9500
C(19)-C(20)	1.3965(15)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
C(21)-H(21A)	0.9800
C(21)-H(21B)	0.9800
C(21)-H(21C)	0.9800

C(1)-O(1)-C(7)	106.64(8)
C(1)-O(2)-C(8)	105.65(8)
C(12)-O(4)-C(11)	115.91(8)
C(10)-N(1)-C(4)	112.33(8)
C(10)-N(1)-C(14)	123.05(8)
C(4)-N(1)-C(14)	123.85(8)
O(2)-C(1)-O(1)	104.53(7)
O(2)-C(1)-C(6)	109.28(8)
O(1)-C(1)-C(6)	108.00(8)
O(2)-C(1)-C(2)	111.96(8)
O(1)-C(1)-C(2)	109.45(8)
C(6)-C(1)-C(2)	113.20(8)
C(3)-C(2)-C(1)	112.27(8)
C(3)-C(2)-H(2A)	109.2
C(1)-C(2)-H(2A)	109.2
C(3)-C(2)-H(2AB)	109.2
C(1)-C(2)-H(2AB)	109.2
H(2A)-C(2)-H(2AB)	107.9

C(4)-C(3)-C(2)	109.00(7)
C(4)-C(3)-C(21)	109.97(7)
C(2)-C(3)-C(21)	113.06(7)
C(4)-C(3)-C(9)	101.35(7)
C(2)-C(3)-C(9)	115.67(7)
C(21)-C(3)-C(9)	107.12(7)
C(5)-C(4)-N(1)	127.36(8)
C(5)-C(4)-C(3)	125.36(8)
N(1)-C(4)-C(3)	107.23(7)
C(4)-C(5)-C(6)	121.76(8)
C(4)-C(5)-H(5)	119.1
C(6)-C(5)-H(5)	119.1
C(5)-C(6)-C(1)	115.21(8)
C(5)-C(6)-H(6A)	108.5
C(1)-C(6)-H(6A)	108.5
C(5)-C(6)-H(6AB)	108.5
C(1)-C(6)-H(6AB)	108.5
H(6A)-C(6)-H(6AB)	107.5
O(1)-C(7)-C(8)	104.85(8)
O(1)-C(7)-H(7A)	110.8
C(8)-C(7)-H(7A)	110.8
O(1)-C(7)-H(7AB)	110.8
C(8)-C(7)-H(7AB)	110.8
H(7A)-C(7)-H(7AB)	108.9
O(2)-C(8)-C(7)	103.80(8)
O(2)-C(8)-H(8A)	111.0
C(7)-C(8)-H(8A)	111.0
O(2)-C(8)-H(8AB)	111.0
C(7)-C(8)-H(8AB)	111.0
H(8A)-C(8)-H(8AB)	109.0
C(12)-C(9)-C(10)	110.56(7)
C(12)-C(9)-C(3)	112.05(8)
C(10)-C(9)-C(3)	102.84(7)
C(12)-C(9)-H(9)	110.4
C(10)-C(9)-H(9)	110.4
C(3)-C(9)-H(9)	110.4
O(5)-C(10)-N(1)	125.83(9)
O(5)-C(10)-C(9)	126.17(9)

N(1)-C(10)-C(9)	107.99(8)
O(4)-C(11)-C(13)	106.97(9)
O(4)-C(11)-H(11A)	110.3
C(13)-C(11)-H(11A)	110.3
O(4)-C(11)-H(11B)	110.3
C(13)-C(11)-H(11B)	110.3
H(11A)-C(11)-H(11B)	108.6
O(3)-C(12)-O(4)	124.20(9)
O(3)-C(12)-C(9)	123.97(9)
O(4)-C(12)-C(9)	111.71(7)
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
N(1)-C(14)-C(15)	113.23(7)
N(1)-C(14)-H(14A)	108.9
C(15)-C(14)-H(14A)	108.9
N(1)-C(14)-H(14B)	108.9
C(15)-C(14)-H(14B)	108.9
H(14A)-C(14)-H(14B)	107.7
C(20)-C(15)-C(16)	118.86(9)
C(20)-C(15)-C(14)	119.91(8)
C(16)-C(15)-C(14)	121.20(8)
C(17)-C(16)-C(15)	120.42(9)
C(17)-C(16)-H(16)	119.8
C(15)-C(16)-H(16)	119.8
C(18)-C(17)-C(16)	120.24(9)
C(18)-C(17)-H(17)	119.9
C(16)-C(17)-H(17)	119.9
C(19)-C(18)-C(17)	119.78(10)
C(19)-C(18)-H(18)	120.1
C(17)-C(18)-H(18)	120.1
C(18)-C(19)-C(20)	119.98(10)
C(18)-C(19)-H(19)	120.0
C(20)-C(19)-H(19)	120.0
C(15)-C(20)-C(19)	120.72(9)

C(15)-C(20)-H(20)	119.6
C(19)-C(20)-H(20)	119.6
C(3)-C(21)-H(21A)	109.5
C(3)-C(21)-H(21B)	109.5
H(21A)-C(21)-H(21B)	109.5
C(3)-C(21)-H(21C)	109.5
H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for D43XB86_0m_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	29(1)	17(1)	28(1)	5(1)	5(1)	0(1)
O(2)	22(1)	22(1)	28(1)	0(1)	9(1)	-3(1)
O(3)	24(1)	36(1)	31(1)	-5(1)	-6(1)	-3(1)
O(4)	18(1)	23(1)	21(1)	0(1)	2(1)	2(1)
O(5)	27(1)	20(1)	30(1)	6(1)	10(1)	1(1)
N(1)	19(1)	21(1)	18(1)	3(1)	3(1)	2(1)
C(1)	20(1)	16(1)	23(1)	2(1)	4(1)	-2(1)
C(2)	19(1)	17(1)	20(1)	2(1)	2(1)	-2(1)
C(3)	18(1)	15(1)	18(1)	0(1)	4(1)	-2(1)
C(4)	17(1)	18(1)	18(1)	1(1)	4(1)	1(1)
C(5)	22(1)	21(1)	19(1)	-3(1)	1(1)	-1(1)
C(6)	28(1)	18(1)	24(1)	-3(1)	1(1)	-4(1)
C(7)	32(1)	29(1)	26(1)	8(1)	2(1)	-6(1)
C(8)	34(1)	25(1)	26(1)	-1(1)	9(1)	-10(1)
C(9)	20(1)	16(1)	20(1)	0(1)	4(1)	-3(1)
C(10)	19(1)	18(1)	22(1)	2(1)	7(1)	2(1)
C(11)	19(1)	25(1)	32(1)	1(1)	4(1)	4(1)
C(12)	19(1)	19(1)	21(1)	1(1)	2(1)	-4(1)
C(13)	36(1)	30(1)	45(1)	-9(1)	3(1)	9(1)
C(14)	22(1)	29(1)	17(1)	3(1)	5(1)	6(1)
C(15)	21(1)	18(1)	18(1)	3(1)	4(1)	2(1)
C(16)	22(1)	21(1)	22(1)	-2(1)	5(1)	1(1)
C(17)	22(1)	23(1)	27(1)	2(1)	7(1)	3(1)
C(18)	21(1)	30(1)	30(1)	6(1)	3(1)	2(1)
C(19)	26(1)	35(1)	26(1)	-1(1)	-3(1)	-1(1)
C(20)	28(1)	26(1)	20(1)	-1(1)	2(1)	3(1)
C(21)	25(1)	20(1)	22(1)	-1(1)	8(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for D43XB86_0m_a.

	x	y	z	U(eq)
H(2A)	8710	3156	4799	22
H(2AB)	9382	2469	4340	22
H(5)	5974	2420	3294	25
H(6A)	7154	140	3813	28
H(6AB)	5401	620	3927	28
H(7A)	8680	292	5299	35
H(7AB)	7264	-1095	5229	35
H(8A)	5540	1027	5329	33
H(8AB)	6925	2443	5322	33
H(9)	9237	6753	4353	22
H(11A)	12491	2369	3960	30
H(11B)	13097	3790	3620	30
H(13A)	11513	2491	2991	56
H(13B)	10992	1043	3333	56
H(13C)	12807	1172	3225	56
H(14A)	6927	4533	2798	27
H(14B)	7226	6553	2813	27
H(16)	4810	7349	3486	26
H(17)	2064	7853	3380	29
H(18)	518	6767	2729	32
H(19)	1719	5130	2189	35
H(20)	4458	4569	2304	29
H(21A)	6318	6591	4301	33
H(21B)	7006	5742	4781	33
H(21C)	5603	4818	4460	33

Compound c-18b

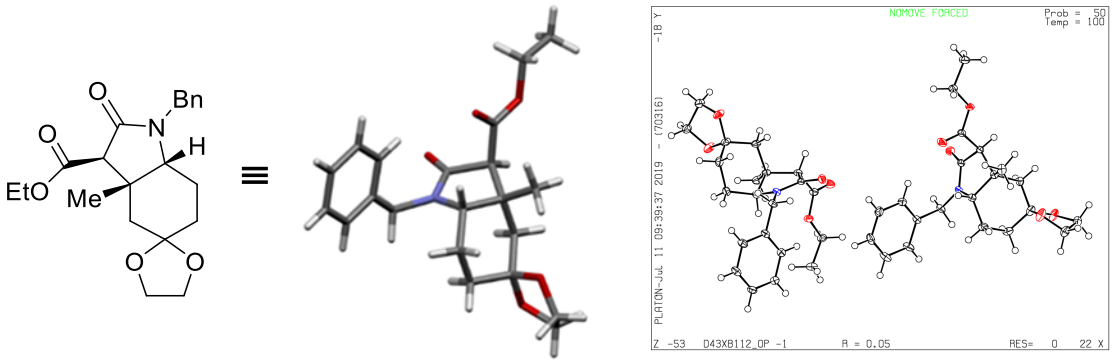


Table 1. Crystal data and structure refinement for D43XB112_0m_a.

Identification code	D43XB112_0m_a	
Empirical formula	C21 H27 N O5	
Formula weight	373.43	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.6698(8) Å	α= 107.614(4)°.
	b = 13.6733(12) Å	β= 91.299(4)°.
	c = 15.2154(12) Å	γ = 93.979(4)°.
Volume	1910.8(3) $Å^3$	
Z	4	
Density (calculated)	1.298 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	800	
Crystal size	0.680 x 0.365 x 0.257 mm^3	
Theta range for data collection	2.404 to 31.071°.	
Index ranges	-14<=h<=14, -19<=k<=19, -22<=l<=22	
Reflections collected	77889	
Independent reflections	12102 [R(int) = 0.0442]	
Completeness to theta = 25.242°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7462 and 0.6572	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12102 / 0 / 491	
Goodness-of-fit on F^2	1.051	
Final R indices [I>2sigma(I)]	R1 = 0.0461, wR2 = 0.1124	

R indices (all data)	R1 = 0.0582, wR2 = 0.1196
Extinction coefficient	n/a
Largest diff. peak and hole	0.482 and -0.248 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for D43XB112_0m_a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1A)	10134(1)	3458(1)	1337(1)	19(1)
O(2A)	8840(1)	-850(1)	1956(1)	26(1)
O(3A)	9830(1)	-1122(1)	553(1)	24(1)
O(4A)	9214(1)	3693(1)	3641(1)	26(1)
O(5A)	11536(1)	3648(1)	3745(1)	18(1)
N(1A)	8384(1)	2279(1)	1456(1)	13(1)
C(1A)	9640(1)	2812(1)	1674(1)	14(1)
C(2A)	10348(1)	2475(1)	2434(1)	15(1)
C(3A)	9482(1)	1462(1)	2399(1)	15(1)
C(4A)	10123(1)	585(1)	1661(1)	16(1)
C(5A)	9154(1)	-380(1)	1258(1)	18(1)
C(6A)	8818(1)	-1940(1)	1530(1)	23(1)
C(7A)	9932(1)	-2021(1)	835(1)	21(1)
C(8A)	7839(1)	-119(1)	836(1)	21(1)
C(9A)	7109(1)	671(1)	1575(1)	19(1)
C(10A)	8031(1)	1647(1)	2056(1)	14(1)
C(11A)	7386(1)	2467(1)	810(1)	16(1)
C(12A)	6088(1)	2915(1)	1254(1)	15(1)
C(13A)	4798(1)	2580(1)	799(1)	22(1)
C(14A)	3605(1)	3033(1)	1169(1)	26(1)
C(15A)	3696(1)	3816(1)	1996(1)	24(1)
C(16A)	4978(1)	4144(1)	2467(1)	23(1)
C(17A)	6172(1)	3691(1)	2097(1)	19(1)
C(18A)	10276(1)	3336(1)	3335(1)	16(1)
C(19A)	11592(1)	4501(1)	4606(1)	18(1)
C(20A)	13094(1)	4779(1)	4921(1)	23(1)
C(21A)	9437(1)	1259(1)	3333(1)	21(1)
O(1B)	4620(1)	6526(1)	3710(1)	18(1)
O(2B)	3848(1)	10775(1)	3018(1)	28(1)
O(3B)	4982(1)	11120(1)	4433(1)	26(1)
O(4B)	5783(1)	6438(1)	1207(1)	26(1)
O(5B)	3510(1)	6241(1)	1450(1)	17(1)
N(1B)	3020(1)	7643(1)	3564(1)	13(1)

C(1B)	4190(1)	7148(1)	3358(1)	13(1)
C(2B)	4904(1)	7510(1)	2611(1)	14(1)
C(3B)	4163(1)	8487(1)	2621(1)	15(1)
C(4B)	4971(1)	9399(1)	3356(1)	17(1)
C(5B)	4157(1)	10338(1)	3736(1)	20(1)
C(6B)	4058(1)	11864(1)	3404(1)	23(1)
C(7B)	5239(1)	11987(1)	4108(1)	21(1)
C(8B)	2842(1)	10041(1)	4154(1)	22(1)
C(9B)	1950(1)	9212(1)	3418(1)	19(1)
C(10B)	2707(1)	8259(1)	2956(1)	14(1)
C(11B)	2046(1)	7437(1)	4214(1)	16(1)
C(12B)	627(1)	7007(1)	3773(1)	16(1)
C(13B)	480(1)	6162(1)	2980(1)	20(1)
C(14B)	-833(1)	5759(1)	2590(1)	23(1)
C(15B)	-2010(1)	6196(1)	3000(1)	22(1)
C(16B)	-1875(1)	7038(1)	3788(1)	22(1)
C(17B)	-559(1)	7449(1)	4171(1)	19(1)
C(18B)	4806(1)	6669(1)	1686(1)	16(1)
C(19B)	3339(1)	5471(1)	537(1)	18(1)
C(20B)	1815(1)	5145(1)	349(1)	24(1)
C(21B)	4081(1)	8667(1)	1677(1)	20(1)

Table 3. Bond lengths [Å] and angles [°] for D43XB112_0m_a.

O(1A)-C(1A)	1.2233(13)
O(2A)-C(5A)	1.4268(13)
O(2A)-C(6A)	1.4326(14)
O(3A)-C(7A)	1.4288(14)
O(3A)-C(5A)	1.4382(13)
O(4A)-C(18A)	1.2098(13)
O(5A)-C(18A)	1.3367(12)
O(5A)-C(19A)	1.4631(13)
N(1A)-C(1A)	1.3521(13)
N(1A)-C(11A)	1.4520(13)
N(1A)-C(10A)	1.4661(13)
C(1A)-C(2A)	1.5324(14)
C(2A)-C(18A)	1.5194(14)
C(2A)-C(3A)	1.5538(15)
C(2A)-H(2A)	1.0000
C(3A)-C(21A)	1.5280(14)
C(3A)-C(10A)	1.5493(14)
C(3A)-C(4A)	1.5507(14)
C(4A)-C(5A)	1.5194(16)
C(4A)-H(4AA)	0.9900
C(4A)-H(4AB)	0.9900
C(5A)-C(8A)	1.5236(17)
C(6A)-C(7A)	1.5113(16)
C(6A)-H(6AA)	0.9900
C(6A)-H(6AB)	0.9900
C(7A)-H(7AA)	0.9900
C(7A)-H(7AB)	0.9900
C(8A)-C(9A)	1.5284(16)
C(8A)-H(8AA)	0.9900
C(8A)-H(8AB)	0.9900
C(9A)-C(10A)	1.5242(15)
C(9A)-H(9AA)	0.9900
C(9A)-H(9AB)	0.9900
C(10A)-H(10A)	1.0000
C(11A)-C(12A)	1.5175(14)
C(11A)-H(11A)	0.9900

C(11A)-H(11B)	0.9900
C(12A)-C(13A)	1.3909(15)
C(12A)-C(17A)	1.3927(15)
C(13A)-C(14A)	1.3958(16)
C(13A)-H(13A)	0.9500
C(14A)-C(15A)	1.3804(19)
C(14A)-H(14A)	0.9500
C(15A)-C(16A)	1.3923(17)
C(15A)-H(15A)	0.9500
C(16A)-C(17A)	1.3969(15)
C(16A)-H(16A)	0.9500
C(17A)-H(17A)	0.9500
C(19A)-C(20A)	1.5053(15)
C(19A)-H(19A)	0.9900
C(19A)-H(19B)	0.9900
C(20A)-H(20A)	0.9800
C(20A)-H(20B)	0.9800
C(20A)-H(20C)	0.9800
C(21A)-H(21A)	0.9800
C(21A)-H(21B)	0.9800
C(21A)-H(21C)	0.9800
O(1B)-C(1B)	1.2243(12)
O(2B)-C(6B)	1.4231(14)
O(2B)-C(5B)	1.4291(13)
O(3B)-C(7B)	1.4259(14)
O(3B)-C(5B)	1.4353(13)
O(4B)-C(18B)	1.2075(13)
O(5B)-C(18B)	1.3393(13)
O(5B)-C(19B)	1.4653(13)
N(1B)-C(1B)	1.3523(12)
N(1B)-C(11B)	1.4562(13)
N(1B)-C(10B)	1.4675(13)
C(1B)-C(2B)	1.5305(14)
C(2B)-C(18B)	1.5199(14)
C(2B)-C(3B)	1.5557(15)
C(2B)-H(2B)	1.0000
C(3B)-C(21B)	1.5308(14)
C(3B)-C(4B)	1.5477(14)

C(3B)-C(10B)	1.5509(14)
C(4B)-C(5B)	1.5215(16)
C(4B)-H(4BA)	0.9900
C(4B)-H(4BB)	0.9900
C(5B)-C(8B)	1.5184(18)
C(6B)-C(7B)	1.5119(16)
C(6B)-H(6BA)	0.9900
C(6B)-H(6BB)	0.9900
C(7B)-H(7BA)	0.9900
C(7B)-H(7BB)	0.9900
C(8B)-C(9B)	1.5279(16)
C(8B)-H(8BA)	0.9900
C(8B)-H(8BB)	0.9900
C(9B)-C(10B)	1.5262(15)
C(9B)-H(9BA)	0.9900
C(9B)-H(9BB)	0.9900
C(10B)-H(10B)	1.0000
C(11B)-C(12B)	1.5121(14)
C(11B)-H(11C)	0.9900
C(11B)-H(11D)	0.9900
C(12B)-C(13B)	1.3922(16)
C(12B)-C(17B)	1.3969(15)
C(13B)-C(14B)	1.3923(16)
C(13B)-H(13B)	0.9500
C(14B)-C(15B)	1.3930(17)
C(14B)-H(14B)	0.9500
C(15B)-C(16B)	1.3853(18)
C(15B)-H(15B)	0.9500
C(16B)-C(17B)	1.3942(16)
C(16B)-H(16B)	0.9500
C(17B)-H(17B)	0.9500
C(19B)-C(20B)	1.5056(16)
C(19B)-H(19C)	0.9900
C(19B)-H(19D)	0.9900
C(20B)-H(20D)	0.9800
C(20B)-H(20E)	0.9800
C(20B)-H(20F)	0.9800
C(21B)-H(21D)	0.9800

C(21B)-H(21E)	0.9800
C(21B)-H(21F)	0.9800

C(5A)-O(2A)-C(6A)	106.81(8)
C(7A)-O(3A)-C(5A)	108.22(8)
C(18A)-O(5A)-C(19A)	115.91(8)
C(1A)-N(1A)-C(11A)	122.64(9)
C(1A)-N(1A)-C(10A)	113.44(8)
C(11A)-N(1A)-C(10A)	122.95(8)
O(1A)-C(1A)-N(1A)	126.71(10)
O(1A)-C(1A)-C(2A)	125.35(9)
N(1A)-C(1A)-C(2A)	107.94(9)
C(18A)-C(2A)-C(1A)	107.45(8)
C(18A)-C(2A)-C(3A)	114.06(9)
C(1A)-C(2A)-C(3A)	103.18(8)
C(18A)-C(2A)-H(2A)	110.6
C(1A)-C(2A)-H(2A)	110.6
C(3A)-C(2A)-H(2A)	110.6
C(21A)-C(3A)-C(10A)	112.00(9)
C(21A)-C(3A)-C(4A)	111.83(8)
C(10A)-C(3A)-C(4A)	110.57(8)
C(21A)-C(3A)-C(2A)	113.38(9)
C(10A)-C(3A)-C(2A)	102.15(8)
C(4A)-C(3A)-C(2A)	106.39(8)
C(5A)-C(4A)-C(3A)	114.70(9)
C(5A)-C(4A)-H(4AA)	108.6
C(3A)-C(4A)-H(4AA)	108.6
C(5A)-C(4A)-H(4AB)	108.6
C(3A)-C(4A)-H(4AB)	108.6
H(4AA)-C(4A)-H(4AB)	107.6
O(2A)-C(5A)-O(3A)	106.52(8)
O(2A)-C(5A)-C(4A)	109.80(9)
O(3A)-C(5A)-C(4A)	110.03(9)
O(2A)-C(5A)-C(8A)	111.26(9)
O(3A)-C(5A)-C(8A)	109.01(9)
C(4A)-C(5A)-C(8A)	110.15(9)
O(2A)-C(6A)-C(7A)	102.15(9)
O(2A)-C(6A)-H(6AA)	111.3

C(7A)-C(6A)-H(6AA)	111.3
O(2A)-C(6A)-H(6AB)	111.3
C(7A)-C(6A)-H(6AB)	111.3
H(6AA)-C(6A)-H(6AB)	109.2
O(3A)-C(7A)-C(6A)	103.54(9)
O(3A)-C(7A)-H(7AA)	111.1
C(6A)-C(7A)-H(7AA)	111.1
O(3A)-C(7A)-H(7AB)	111.1
C(6A)-C(7A)-H(7AB)	111.1
H(7AA)-C(7A)-H(7AB)	109.0
C(5A)-C(8A)-C(9A)	109.74(9)
C(5A)-C(8A)-H(8AA)	109.7
C(9A)-C(8A)-H(8AA)	109.7
C(5A)-C(8A)-H(8AB)	109.7
C(9A)-C(8A)-H(8AB)	109.7
H(8AA)-C(8A)-H(8AB)	108.2
C(10A)-C(9A)-C(8A)	113.49(9)
C(10A)-C(9A)-H(9AA)	108.9
C(8A)-C(9A)-H(9AA)	108.9
C(10A)-C(9A)-H(9AB)	108.9
C(8A)-C(9A)-H(9AB)	108.9
H(9AA)-C(9A)-H(9AB)	107.7
N(1A)-C(10A)-C(9A)	113.60(9)
N(1A)-C(10A)-C(3A)	102.09(8)
C(9A)-C(10A)-C(3A)	114.88(9)
N(1A)-C(10A)-H(10A)	108.6
C(9A)-C(10A)-H(10A)	108.6
C(3A)-C(10A)-H(10A)	108.6
N(1A)-C(11A)-C(12A)	113.67(8)
N(1A)-C(11A)-H(11A)	108.8
C(12A)-C(11A)-H(11A)	108.8
N(1A)-C(11A)-H(11B)	108.8
C(12A)-C(11A)-H(11B)	108.8
H(11A)-C(11A)-H(11B)	107.7
C(13A)-C(12A)-C(17A)	119.14(10)
C(13A)-C(12A)-C(11A)	119.95(10)
C(17A)-C(12A)-C(11A)	120.86(9)
C(12A)-C(13A)-C(14A)	120.62(11)

C(12A)-C(13A)-H(13A)	119.7
C(14A)-C(13A)-H(13A)	119.7
C(15A)-C(14A)-C(13A)	120.06(11)
C(15A)-C(14A)-H(14A)	120.0
C(13A)-C(14A)-H(14A)	120.0
C(14A)-C(15A)-C(16A)	119.87(10)
C(14A)-C(15A)-H(15A)	120.1
C(16A)-C(15A)-H(15A)	120.1
C(15A)-C(16A)-C(17A)	120.09(11)
C(15A)-C(16A)-H(16A)	120.0
C(17A)-C(16A)-H(16A)	120.0
C(12A)-C(17A)-C(16A)	120.21(10)
C(12A)-C(17A)-H(17A)	119.9
C(16A)-C(17A)-H(17A)	119.9
O(4A)-C(18A)-O(5A)	124.37(10)
O(4A)-C(18A)-C(2A)	124.26(9)
O(5A)-C(18A)-C(2A)	111.37(8)
O(5A)-C(19A)-C(20A)	107.67(9)
O(5A)-C(19A)-H(19A)	110.2
C(20A)-C(19A)-H(19A)	110.2
O(5A)-C(19A)-H(19B)	110.2
C(20A)-C(19A)-H(19B)	110.2
H(19A)-C(19A)-H(19B)	108.5
C(19A)-C(20A)-H(20A)	109.5
C(19A)-C(20A)-H(20B)	109.5
H(20A)-C(20A)-H(20B)	109.5
C(19A)-C(20A)-H(20C)	109.5
H(20A)-C(20A)-H(20C)	109.5
H(20B)-C(20A)-H(20C)	109.5
C(3A)-C(21A)-H(21A)	109.5
C(3A)-C(21A)-H(21B)	109.5
H(21A)-C(21A)-H(21B)	109.5
C(3A)-C(21A)-H(21C)	109.5
H(21A)-C(21A)-H(21C)	109.5
H(21B)-C(21A)-H(21C)	109.5
C(6B)-O(2B)-C(5B)	107.30(9)
C(7B)-O(3B)-C(5B)	108.21(8)
C(18B)-O(5B)-C(19B)	114.74(8)

C(1B)-N(1B)-C(11B)	122.67(9)
C(1B)-N(1B)-C(10B)	113.48(8)
C(11B)-N(1B)-C(10B)	122.99(8)
O(1B)-C(1B)-N(1B)	126.46(10)
O(1B)-C(1B)-C(2B)	125.39(9)
N(1B)-C(1B)-C(2B)	108.14(8)
C(18B)-C(2B)-C(1B)	111.70(8)
C(18B)-C(2B)-C(3B)	114.68(8)
C(1B)-C(2B)-C(3B)	103.29(8)
C(18B)-C(2B)-H(2B)	109.0
C(1B)-C(2B)-H(2B)	109.0
C(3B)-C(2B)-H(2B)	109.0
C(21B)-C(3B)-C(4B)	111.82(9)
C(21B)-C(3B)-C(10B)	112.08(8)
C(4B)-C(3B)-C(10B)	110.49(8)
C(21B)-C(3B)-C(2B)	113.27(9)
C(4B)-C(3B)-C(2B)	106.24(8)
C(10B)-C(3B)-C(2B)	102.43(8)
C(5B)-C(4B)-C(3B)	114.84(9)
C(5B)-C(4B)-H(4BA)	108.6
C(3B)-C(4B)-H(4BA)	108.6
C(5B)-C(4B)-H(4BB)	108.6
C(3B)-C(4B)-H(4BB)	108.6
H(4BA)-C(4B)-H(4BB)	107.5
O(2B)-C(5B)-O(3B)	106.42(9)
O(2B)-C(5B)-C(8B)	111.32(10)
O(3B)-C(5B)-C(8B)	109.14(9)
O(2B)-C(5B)-C(4B)	109.71(9)
O(3B)-C(5B)-C(4B)	110.21(9)
C(8B)-C(5B)-C(4B)	109.98(9)
O(2B)-C(6B)-C(7B)	102.32(9)
O(2B)-C(6B)-H(6BA)	111.3
C(7B)-C(6B)-H(6BA)	111.3
O(2B)-C(6B)-H(6BB)	111.3
C(7B)-C(6B)-H(6BB)	111.3
H(6BA)-C(6B)-H(6BB)	109.2
O(3B)-C(7B)-C(6B)	103.26(9)
O(3B)-C(7B)-H(7BA)	111.1

C(6B)-C(7B)-H(7BA)	111.1
O(3B)-C(7B)-H(7BB)	111.1
C(6B)-C(7B)-H(7BB)	111.1
H(7BA)-C(7B)-H(7BB)	109.1
C(5B)-C(8B)-C(9B)	109.72(9)
C(5B)-C(8B)-H(8BA)	109.7
C(9B)-C(8B)-H(8BA)	109.7
C(5B)-C(8B)-H(8BB)	109.7
C(9B)-C(8B)-H(8BB)	109.7
H(8BA)-C(8B)-H(8BB)	108.2
C(10B)-C(9B)-C(8B)	113.63(9)
C(10B)-C(9B)-H(9BA)	108.8
C(8B)-C(9B)-H(9BA)	108.8
C(10B)-C(9B)-H(9BB)	108.8
C(8B)-C(9B)-H(9BB)	108.8
H(9BA)-C(9B)-H(9BB)	107.7
N(1B)-C(10B)-C(9B)	114.03(9)
N(1B)-C(10B)-C(3B)	102.04(8)
C(9B)-C(10B)-C(3B)	114.76(9)
N(1B)-C(10B)-H(10B)	108.6
C(9B)-C(10B)-H(10B)	108.6
C(3B)-C(10B)-H(10B)	108.6
N(1B)-C(11B)-C(12B)	113.11(8)
N(1B)-C(11B)-H(11C)	109.0
C(12B)-C(11B)-H(11C)	109.0
N(1B)-C(11B)-H(11D)	109.0
C(12B)-C(11B)-H(11D)	109.0
H(11C)-C(11B)-H(11D)	107.8
C(13B)-C(12B)-C(17B)	119.16(10)
C(13B)-C(12B)-C(11B)	120.85(9)
C(17B)-C(12B)-C(11B)	119.98(10)
C(12B)-C(13B)-C(14B)	120.41(10)
C(12B)-C(13B)-H(13B)	119.8
C(14B)-C(13B)-H(13B)	119.8
C(13B)-C(14B)-C(15B)	119.95(11)
C(13B)-C(14B)-H(14B)	120.0
C(15B)-C(14B)-H(14B)	120.0
C(16B)-C(15B)-C(14B)	120.12(10)

C(16B)-C(15B)-H(15B)	119.9
C(14B)-C(15B)-H(15B)	119.9
C(15B)-C(16B)-C(17B)	119.83(10)
C(15B)-C(16B)-H(16B)	120.1
C(17B)-C(16B)-H(16B)	120.1
C(16B)-C(17B)-C(12B)	120.51(11)
C(16B)-C(17B)-H(17B)	119.7
C(12B)-C(17B)-H(17B)	119.7
O(4B)-C(18B)-O(5B)	123.99(10)
O(4B)-C(18B)-C(2B)	123.53(10)
O(5B)-C(18B)-C(2B)	112.45(9)
O(5B)-C(19B)-C(20B)	107.70(9)
O(5B)-C(19B)-H(19C)	110.2
C(20B)-C(19B)-H(19C)	110.2
O(5B)-C(19B)-H(19D)	110.2
C(20B)-C(19B)-H(19D)	110.2
H(19C)-C(19B)-H(19D)	108.5
C(19B)-C(20B)-H(20D)	109.5
C(19B)-C(20B)-H(20E)	109.5
H(20D)-C(20B)-H(20E)	109.5
C(19B)-C(20B)-H(20F)	109.5
H(20D)-C(20B)-H(20F)	109.5
H(20E)-C(20B)-H(20F)	109.5
C(3B)-C(21B)-H(21D)	109.5
C(3B)-C(21B)-H(21E)	109.5
H(21D)-C(21B)-H(21E)	109.5
C(3B)-C(21B)-H(21F)	109.5
H(21D)-C(21B)-H(21F)	109.5
H(21E)-C(21B)-H(21F)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for D43XB112_0m_a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1A)	17(1)	18(1)	23(1)	7(1)	3(1)	-1(1)
O(2A)	42(1)	17(1)	20(1)	8(1)	13(1)	4(1)
O(3A)	38(1)	15(1)	19(1)	4(1)	11(1)	5(1)
O(4A)	15(1)	29(1)	24(1)	-6(1)	-2(1)	6(1)
O(5A)	13(1)	19(1)	16(1)	-1(1)	-3(1)	1(1)
N(1A)	10(1)	17(1)	14(1)	6(1)	0(1)	1(1)
C(1A)	12(1)	14(1)	13(1)	1(1)	1(1)	2(1)
C(2A)	12(1)	16(1)	14(1)	1(1)	-1(1)	3(1)
C(3A)	15(1)	16(1)	12(1)	4(1)	1(1)	3(1)
C(4A)	18(1)	16(1)	13(1)	4(1)	2(1)	4(1)
C(5A)	26(1)	15(1)	14(1)	4(1)	7(1)	2(1)
C(6A)	30(1)	16(1)	25(1)	7(1)	7(1)	2(1)
C(7A)	24(1)	19(1)	19(1)	5(1)	1(1)	5(1)
C(8A)	21(1)	18(1)	19(1)	2(1)	0(1)	-5(1)
C(9A)	15(1)	21(1)	21(1)	5(1)	2(1)	-3(1)
C(10A)	13(1)	17(1)	13(1)	5(1)	2(1)	1(1)
C(11A)	13(1)	24(1)	14(1)	7(1)	0(1)	4(1)
C(12A)	12(1)	20(1)	16(1)	8(1)	1(1)	2(1)
C(13A)	14(1)	34(1)	17(1)	6(1)	-2(1)	1(1)
C(14A)	12(1)	42(1)	26(1)	15(1)	-1(1)	3(1)
C(15A)	16(1)	28(1)	32(1)	16(1)	8(1)	8(1)
C(16A)	21(1)	18(1)	29(1)	5(1)	7(1)	4(1)
C(17A)	14(1)	17(1)	24(1)	5(1)	1(1)	-1(1)
C(18A)	14(1)	17(1)	15(1)	1(1)	-3(1)	1(1)
C(19A)	17(1)	18(1)	15(1)	0(1)	-2(1)	0(1)
C(20A)	17(1)	26(1)	21(1)	1(1)	-3(1)	-3(1)
C(21A)	29(1)	24(1)	12(1)	6(1)	2(1)	8(1)
O(1B)	17(1)	18(1)	19(1)	7(1)	0(1)	4(1)
O(2B)	47(1)	16(1)	20(1)	7(1)	-16(1)	-4(1)
O(3B)	42(1)	16(1)	19(1)	4(1)	-14(1)	-4(1)
O(4B)	15(1)	35(1)	20(1)	-2(1)	4(1)	-1(1)
O(5B)	13(1)	19(1)	16(1)	-1(1)	1(1)	-2(1)
N(1B)	10(1)	17(1)	14(1)	6(1)	0(1)	1(1)

C(1B)	11(1)	14(1)	13(1)	2(1)	-1(1)	0(1)
C(2B)	11(1)	17(1)	14(1)	2(1)	0(1)	-1(1)
C(3B)	16(1)	16(1)	12(1)	4(1)	-2(1)	-2(1)
C(4B)	19(1)	17(1)	14(1)	5(1)	-4(1)	-4(1)
C(5B)	29(1)	15(1)	15(1)	4(1)	-10(1)	-2(1)
C(6B)	28(1)	16(1)	26(1)	7(1)	-8(1)	0(1)
C(7B)	22(1)	20(1)	20(1)	6(1)	-2(1)	-2(1)
C(8B)	26(1)	18(1)	19(1)	2(1)	-3(1)	7(1)
C(9B)	18(1)	19(1)	20(1)	5(1)	-3(1)	6(1)
C(10B)	13(1)	16(1)	13(1)	5(1)	-3(1)	1(1)
C(11B)	11(1)	25(1)	14(1)	7(1)	1(1)	1(1)
C(12B)	11(1)	21(1)	16(1)	9(1)	0(1)	1(1)
C(13B)	16(1)	19(1)	24(1)	6(1)	0(1)	2(1)
C(14B)	20(1)	17(1)	29(1)	4(1)	-5(1)	-1(1)
C(15B)	14(1)	22(1)	33(1)	12(1)	-6(1)	-3(1)
C(16B)	12(1)	29(1)	26(1)	12(1)	1(1)	3(1)
C(17B)	14(1)	28(1)	16(1)	6(1)	2(1)	2(1)
C(18B)	14(1)	18(1)	15(1)	4(1)	1(1)	-1(1)
C(19B)	19(1)	18(1)	14(1)	0(1)	-1(1)	-1(1)
C(20B)	20(1)	25(1)	23(1)	5(1)	-4(1)	-6(1)
C(21B)	26(1)	22(1)	13(1)	6(1)	-3(1)	-6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for D43XB112_0m_a.

	x	y	z	U(eq)
H(2A)	11334	2340	2292	18
H(4AA)	10426	848	1152	19
H(4AB)	10958	398	1941	19
H(6AA)	7902	-2228	1222	28
H(6AB)	9048	-2297	1985	28
H(7AA)	10860	-2023	1123	25
H(7AB)	9753	-2654	305	25
H(8AA)	7211	-750	578	25
H(8AB)	8079	164	327	25
H(9AA)	6284	861	1285	23
H(9AB)	6782	351	2043	23
H(10A)	7540	2069	2595	17
H(11A)	7116	1811	322	20
H(11B)	7837	2948	509	20
H(13A)	4728	2040	230	26
H(14A)	2729	2801	852	31
H(15A)	2886	4130	2244	28
H(16A)	5041	4677	3040	27
H(17A)	7044	3913	2422	23
H(19A)	11069	4292	5080	21
H(19B)	11174	5100	4502	21
H(20A)	13498	4180	5017	35
H(20B)	13163	5346	5501	35
H(20C)	13598	4993	4450	35
H(21A)	8865	619	3268	32
H(21B)	9038	1832	3782	32
H(21C)	10381	1197	3546	32
H(2B)	5904	7714	2804	17
H(4BA)	5275	9158	3875	20
H(4BB)	5815	9608	3080	20
H(6BA)	3217	12158	3702	28
H(6BB)	4320	12194	2929	28

H(7BA)	6147	11973	3819	25
H(7BB)	5220	12640	4616	25
H(8BA)	2314	10654	4398	26
H(8BB)	3086	9778	4671	26
H(9BA)	1120	9001	3707	23
H(9BB)	1627	9509	2939	23
H(10B)	2120	7815	2413	17
H(11C)	1950	8084	4716	20
H(11D)	2428	6941	4494	20
H(13B)	1280	5859	2703	24
H(14B)	-927	5186	2045	27
H(15B)	-2906	5916	2737	27
H(16B)	-2678	7334	4068	26
H(17B)	-468	8034	4705	23
H(19C)	3863	4870	525	22
H(19D)	3696	5769	61	22
H(20D)	1304	5748	379	36
H(20E)	1480	4831	812	36
H(20F)	1667	4643	-267	36
H(21D)	5021	8780	1480	31
H(21E)	3571	9274	1721	31
H(21F)	3598	8064	1225	31