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Supporting Information

Phormidolides B and C, Cytotoxic Agents from the Sea: Enantioselective Synthesis of the Macrocyclic Core

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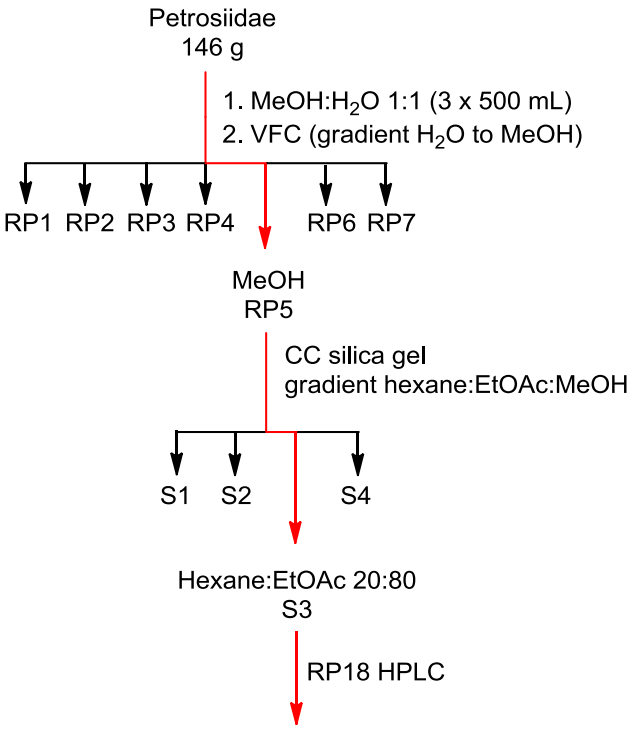
1. General Procedures

Tetrahydrofuran (THF) and *N*,*N*-dimethylformamide (DMF) were dried using a PureSolv solvent purification system. All other solvents and reagents were used as purchased without further purification, unless otherwise indicated. Flash column chromatography was performed on silica gel (60A 35-70 μm) as stationary phase. Analytical TLC was performed on pre-coated silica gel 60 F_{254} plates (0.2 mm thick, 20x20 cm) and visualized under UV light (254 and 360 nm), with anisaldehyde in conc. H_2SO_4 or with phosphomolybdic acid in ethanol. Polarimetry studies were performed on a Perkin-Elmer 241 or JascoP-2000 polarimeter equipped with a Na-lamp. IR spectra were recorded on a Thermo Nicolet FT-IR Nexus spectrometer. For the isolation ^{1}H and ^{13}C-NMR were recorded on a Varian Unity 300MHz or a Varian Unity 500MHz; for the synthesis were recorded on a Varian Mercury 400MHz or a Varian VNMRS500 500MHz. Chemical shifts are reported in ppm referenced to the appropriate residual solvent peaks ($CDCl_3$) and coupling constants are reported in Hz. Multiplicity of the carbons was assigned with gHSQC experiments. Standard abbreviations for off-resonance decoupling were employed: s = singlet, d = doublet, t = triplet, q = quadruplet, bs = broad singlet, bd = broad doublet, m = multiplet. The same abbreviations were also used for the multiplicity of signals in ^{1}H-NMR. High Resolution Mass Spectroscopy (HRMS) was performed an Agilent LC/MSD-TOF 2006 system using the ESI-MS technique.

2. Isolation of Phormidolides B and C



Scheme 1. Isolation of phormidolides B and C.

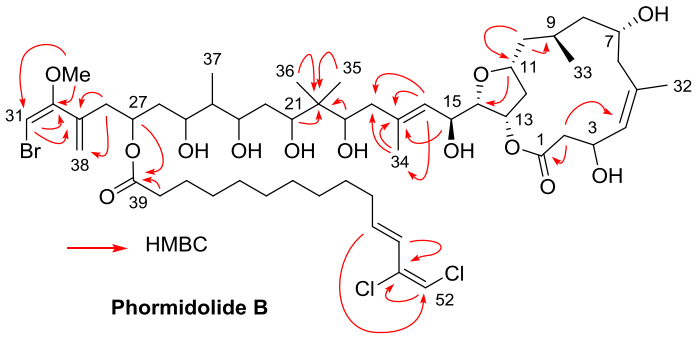
The frozen sponge (146 g, ORMA 41004) was triturated and extracted with a mixture of MeOH-CH_2Cl_2 (50:50, 3 × 500 mL) at 23 ºC (Scheme 1). The organic extract was evaporated under reduced pressure to yield a crude of 6.7 g. This material was chromatographed (VLC) on Lichroprep RP-18 with a stepped gradient from H_2O to MeOH and then to CH_2Cl_2. The fraction eluted with MeOH (340 mg) was subjected to flash Silica gel CC eluting with a gradient of hexane:EtOAc:MeOH to yield 4 fractions (S1 to S4). Fraction S3 (hexane:EtOAc 20:80) was subjected to semipreparative reversed phase HPLC (SunFire, 10 ×150 mm, 100% of CH_3CN in 30 min, UV detection, flow 3.8 mL/min) to yield **phormidolide B** (34.7 mg) and **phormidolide C** (8.3 mg).

Table 1. GI_{50} values for Phormidolides B and C.

Compound	A-549	HT-29	MDA-MB-231
Phormidolide B	1.4 µM	1.3 µM	1.0 µM
Phormidolide C	1.3 µM	0.8 µM	0.5 µM

Lung (A-549), colon (HT-29) and breast (MDA-MB-231) cancer cell lines.

3. NMR data table of phormidolides B and C. Spectra recorded in $CDCl_3$ (500MHz)



	Phormidolide B		Phormidolide C	
	δ_H, mult, J (Hz)	**δ_C, mult**	**δ_H, mult, J (Hz)**	**δ_C, mult**
1	-	171.1, s	-	171.1, s
2	2.72, dd, 13.5, 11.9 2.34, dd 13.5, 3.0	39.5, t	2.73, dd, 13.5, 12.2 2.36, dd, 13.5, 3.0	39.5, t
3	4.74, brd, 11.8	71.9, d	4.75, brd, 12.2	71.9, d
4	5.36, brs	121.6, d	5.37, brs	121.5, d
5	-	133.1, s	-	133.2, s
6	1.91, dd, 16.8, 10.9 1.76, dd, 16.8, 2.5	36.4, t	1.90, brd, 16.1, 11.2 1.78, dd, 16.1, 2.9	36.4, t
7	3.86, m	63.3, d	3.85, m	63.3, d
8	1.45, ddd, 13.2, 13.2, 3.6 1.30, ddd, 13.2, 13.2, 3.5	43.2, t	1.46, dd, 13.5, 13.5 1.31, dd, 13.5, 13.5	43.2, t
9	1.71, m	24.9, d	1.72, m	24.9, d
10	1.94, dd, 12.4, 12.4 1.15, ddd, 12.4, 12.4, 4.9	39.6, t	1.95, dd, 12.4, 12.4 1.17, ddd, 12.4, 12.4, 4.9	39.6, t
11	4.34, ddd, 12.4, 8.4, 4.9	77.6, d	4.36, ddd, 12.4, 8.3, 4.9	77.6, d
12	2.41, d, 14.4 2.09, ddd 13.4, 8.0, 4.0	34.3, t	2.43, d, 14.4 2.11, ddd 13.7, 7.8, 3.9	34.3, t
13	5.22, dd, 3.4, 3.4	75.2, d	5.24, dd, 3.9, 3.9	75.3, d

14	3.81,dd, 8.6, 3.4	83.9, d	3.84, dd, 8.3, 3.4	83.8, d
15	4.59, dd, 8.6, 8.6	66.6, d	4.64, dd, 8.3, 8.3	66.8, d
16	5.36, d, 8.6	129.0, d	5.37, d, 8.3	128.9, d
17	-	137.6, s	-	137.8, s
18	2.30, brd, 13.4 2.05, dd, 13.4, 10.9	42.0, t	2.32, d, 13.2 2.06, dd, 13.2, 10.8	41.9, t
19	3.67, dd, 10.9, 2.0	77.3, d	3.68, brd, 10.8	77.2, d
20	-	40.3, s	-	40.4, s
21	3.83, brd, 10.9	81.6, d	3.83, d, 10.7	81.6, d
22	1.63, m 1.44, m	35.1, t	1.62, m 1.43, m	35.1, t
23	4.06, brd, 10.4	77.8, d	4.07, brd, 9.8	77.8, d
24	1.47, m	41.5, d	1.44, m	41.5, d
25	3.95, ddd, 6.5, 6.5, 1.0	74.0, d	3.97, dd, 8.5, 8.5	74.0, d
26	1.80, m 1.74, m	39.3, t	1.80, m 1.74, m	39.4, t
27	4.95, dddd, 6.8, 6.8, 6.8, 6.8	70.6, d	4.96, dddd, 6.8, 6.8, 6.8, 6.8	70.6, t
28	2.57, dd, 14.4, 6.8 2.53, dd, 14.4, 6.8	39.3, t	2.58, dd, 14.2, 7.3 2.53, dd, 14.2, 5.9	39.4, t
29	-	138.2, s	-	138.2, s
30	-	158.3, s	-	158.3, s
31	5.33, s	78.9, d	5.33, s	78.6, d
32	1.69, s	23.0, q	1.70, s	23.0, q
33	0.83, d, 6.4	20.6, q	0.84, d, 6.9	20.6, q
34	1.77, s	17.0, q	1.79, s	17.2, q
35	0.73, s	21.7, q	0.74, s	21.6, q
36	0.88, s	13.8, q	0.90, s	13.8, q
37	0.91, d, 7.0	5.0, q	0.93, d, 7.5	4.9, q
38	5.41, d, 1.0 5.36, d, 1.0	122.1, d	5.42, d, 0.9 5.37, d, 0.9	122.2, t
39	-	173.7, s	-	173.8, s
40	2.27, t, 7.0	34.6, t	2.27, dd, 7.8, 7.3	34.6, t
41	1.58, m	24.8, t	1.58, m	24.9, t

42-46	1.27, m	28.5, t	1.27, m	28.6, t
47	1.40, m	29.1-29.0, t	1.27, m	29.4-28.4, t
48	2.14, ddd, 6.7, 6.7, 6.7	33.0, t	1.39, m	28.4, t
49	6.01, ddd, 15.4, 6.7, 6.7	140.4, d	1.62, m	29.5, t
50	6.06, d, 15.4	116.7, d	2.91, dd, 7.8, 6.8	33.3, t
51	-	129.7, s	-	131.3, s
52	6.36, s	116.5, d	6.53, s	105.7, d
OMe	3.58, s	55.6, q	3.59, s	55.6, q

Phormidolide B: $[\alpha]_D$ = −6.4 (c 0.4, $CHCl_3$). IR (KBr film) ν 3397 (br), 2930, 2855, 1732, 1607, 1457, 1377, 1277, 1179, 1150 cm^{-1}. MS (API-ES) 1103 (M+Na, 100), 1105 (M+Na, 45), 1102 (M+Na, 37), 1107 (M+Na, 8).

Phormidolide C: $[\alpha]_D$ = +12.3 (c 0.1, $CHCl_3$). IR (KBr film) ν 3377 (br), 2925, 2854, 1732, 1679, 1607, 1440, 1378, 1277, 1200, 1121 cm^{-1}. MS (API-ES) 1149 (M+Na, 100), 1150 (M+Na, 58), 1152 (M+Na, 40) 1148 (M+Na, 25), 1154 (M+Na, 9)

Fatty acid of phormidolide B: . ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.27–1.37 (m, 10H); 1.39–1.45 (m, 2H); 1.59–1.67 (m, 2H); 2.13–2.18 (m, 2H); 2.35 (t, *J* = 7.4 Hz, 2H); 6.02 (dt, *J* = 14.9, 6.0 Hz, 1H); 6.07 (d, *J* = 14.9 Hz, 1H); 6.37 (s) . ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 24.8 (t); 28.7 (t); 29.0 (t); 29,1 (t); 29,3 (t); 33.1 (t); 34.1 (t); 116.7 (d); 116.9 (d); 129.9 (s); 140.6 (d); 179.9 (s). MS (API-ES) 311 ($M+H_2O+H$, 100), 313 ($M+H_2O+H$, 65), 312 ($M+H_2O+H$, 15).

4. *J*-based configuration analysis

Determination of the relative stereochemistry of C9 and C7 to C11:

Protons $^{10}CH_2$ and $^{8}CH_2$ are systems with the characteristic of having two large *J*-coupling constants and one small, meaning each one has a proton in an anti-relationship[1] (*J*-coupling constants were determined based on 1D-TOCSY experiments).

As an example, assuming that $H10_L$ has H11 in an anti-relationship, and $H10_H$ has H9 in an anti-relationship, for those two cases, two dispositions are possible. With the aid of ROESY experiments, the right relative disposition is revealed for each case. Combination of the two relative dispositions C11-C10 and C10-C9 provides the relative disposition of C11-C9 (Figure 1). Same procedure is followed to obtain the relative stereochemistry of C9-C7 (Figure 2).

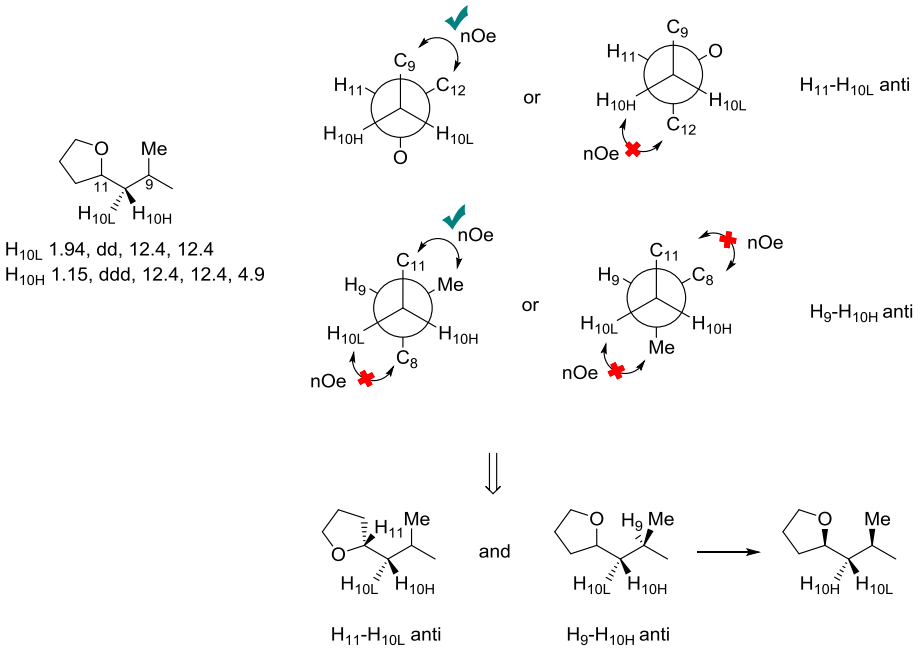


Figure 1. Determination of relative stereochemistry of C9-C11.

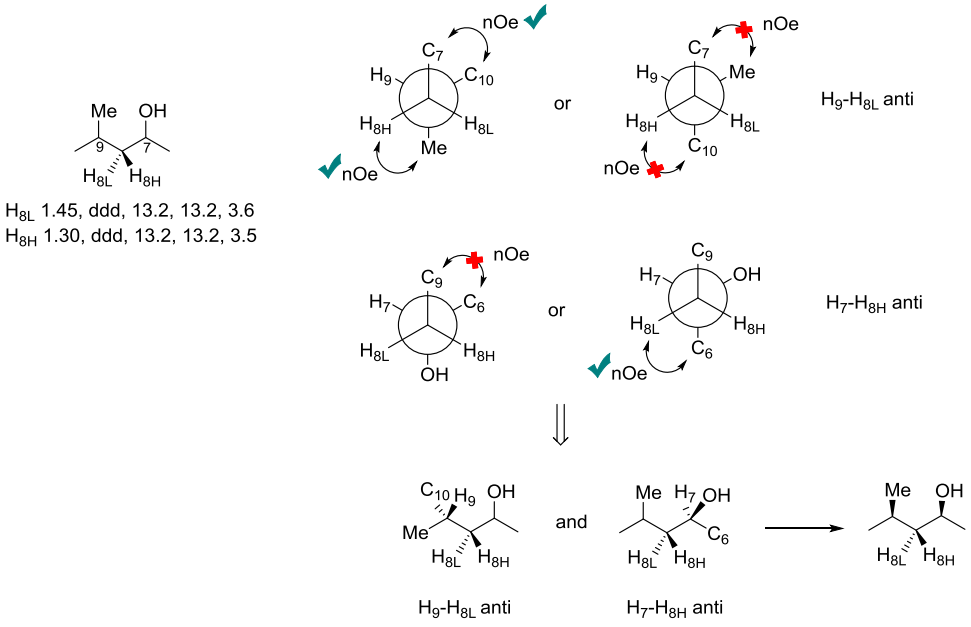


Figure 2. Determination of relative stereochemistry of C7-C9.

Determination of the relative stereochemistry of C14-C15:

Protons H14 and H15 exist on a conformation where their coupling constant value is that of $J_{(H14,H15)}$ = 8.6 Hz, indicating a dominant anti disposition for those protons.[2] A single conformer with a correct configuration can be deduced based on the lack of observation of correlation signal in the ROESY spectra, given that in the case of the H/H-anti and C/C-gauche conformation, if present, H-13 and H-16 should come within the range of NOE.[2]

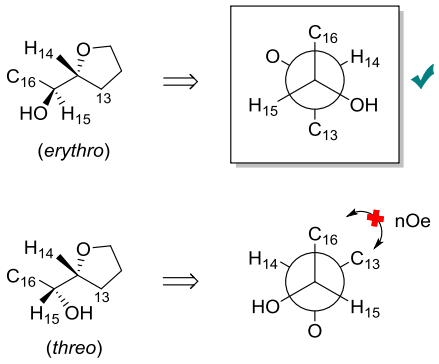


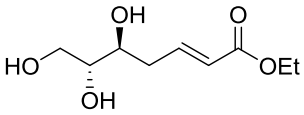
Figure 3. Determination of relative stereochemistry of C14-C15.

5. Experimental procedures and characterization

General procedure for the preparation of triol S1: [3]

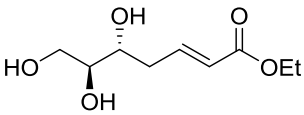
Ethyl (triphenylphosphoranylidene)acetate (1.1 eq.) was added to a solution of sugar (1 eq.) in THF. The solution was stirred at reflux temperature for 5 h and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with CH_2Cl_2-MeOH (95:5 to 90:10) yielded the corresponding triol **S1** as a colorless oil.

Ethyl (5*S*,6*R*,*E*)-5,6,7-trihydroxyhept-2-enoate (S1a).



2-Deoxy-D-ribose (3.40 g, 25.5 mmol) led to triol **S1a** (4.9 g, 94%). IR (KBr film) ν 3380, 2981, 2936, 1701, 1654, 1370, 1270, 1173 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.26 (t, *J* = 7.1 Hz, 3H); 2.33–2.52 (m, 2H); 3.55–3.61 (m, 1H); 3.66–3.72 (m, 2H); 3.74–3.82 (m, 1H); 3.93–4.13 (m, 3H, OH); 4.15 (q, *J* = 7.1, 2H); 5.91 (d, *J* = 15.7, 1H); 6.98 (dt, *J* = 15.7, 7.5 Hz, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q); 35.8 (t); 60.5 (t); 63.1 (t); 71.5 (d); 74.0 (d); 123.6 (d); 145.8 (d); 166.9 (s). HRMS (+ESI): *m/z* calcd. for $C_9H_{17}O_5$ (M+H) 205.1076, found 205.1068.

Ethyl (5*R*,6*S*,*E*)-5,6,7-trihydroxyhept-2-enoate (S1b).

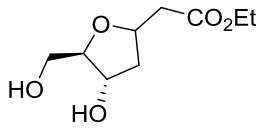


2-Deoxy-L-ribose (627 mg, 4.67 mmol) led to triol **1b** (875 mg, 92%). ^1H NMR (400 MHz, $CDCl_3$) δ 1.26 (t, *J* = 7.1 Hz, 3H); 2.33–2.52 (m, 2H); 3.55–3.61 (m, 1H); 3.66–3.72 (m, 2H); 3.74–3.82 (m, 1H); 3.93–4.13(m, 3H, OH); 4.15 (q, *J* = 7.1, 2H); 5.91 (d, *J* = 15.7, 1H); 6.98 (dt, *J* = 15.7, 7.5 Hz, 1H).

General procedure for the preparation of the tetrahydrofuran ring: [3]

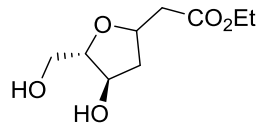
NaEtO (0.1 eq.) was added to a solution of **S1** (1 eq.) in EtOH. The reaction mixture was stirred at r.t. for 24 h. The solvent was removed under reduced pressure and the residue was filtered through a pad of silica with CH_2Cl_2-MeOH (90:10) to yield the corresponding tetrahydrofuran **4** as a mixture of diastereomers A:B (60:40).

Ethyl 2-[(2*RS*,4*S*,5*R*)-4-hydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl]acetate (4a).



Triol **S1a** (13.50 g, 64.4 mmol) led to tetrahydrofuran **4a** (10.56 g, 80%) as a mixture of diastereomers A:B (60:40). IR (KBr film) ν 3409, 2981, 2935, 1731, 1370, 1303, 1197 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.26 (t, *J* = 7.1 Hz, $3H_{A+B}$); 1.80 and 1.92 (ddd, *J* = 13.1, 6.5, 5.4 Hz and ddd, *J* = 13.1, 9.4, 6.4 Hz, $1H_{A+B}$); 2.04 and 2.44 (ddd, *J* = 13.1, 5.8, 2.6 Hz and dt, *J* = 13.1, 7.1 Hz, $1H_{B+A}$); 2.59–2.66 and 2.71–2.78 (2m, $2H_{A+B}$); 3.58–3.57 (m, $2H_{A+B}$); 3.85–3.96 (m, $1H_{A+B}$); 4.16 (q, *J* = 7.1 Hz, $2H_{A+B}$); 4.31–4.40 (m, $1H_{A+B}$); 4.45–4.57 (m, $1H_{A+B}$). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.1 (q_A+q_B); 39.9 (t_B); 40.0 (t_A); 40.6(t_B); 40.8 (t_A); 60.7 (t_A); 60.8 (t_B); 62.3 (t_A); 62.9 (t_B); 72.6 (d_A); 73.1 (d_B); 74.4 (d_A); 74.5(d_B); 85.4 (d_A); 87.1 (d_B); 171.3 (s_B); 171.6 (s_A). HRMS (+ESI): *m/z* calcd. for $C_9H_{17}O_5$ (M+H) 205.1076, found 205.1065.

Ethyl 2-[(2*RS*,4*R*,5*S*)-4-hydroxy-5-(hydroxymethyl)tetrahydrofuran-2-yl]acetate (4b).

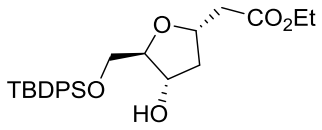


Triol **S1b** (280 mg, 2.8 mmol) led to tetrahydrofuran **4b** (517 mg, 89%) as a mixture of diastereomers A:B (60:40). IR (KBr film) ν 3340 (bs), 2980, 2935, 1730, 1304, 1094 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.26 (t, *J* = 7.1 Hz, $3H_{A+B}$); 1.80 and 1.92 (ddd, *J* = 13.1, 6.5, 5.4 Hz and ddd, *J* = 13.1, 9.4, 6.4 Hz, $1H_{A+B}$); 2.04 and 2.44 (ddd, *J* = 13.1, 5.8, 2.6 Hz and dt, *J* = 13.1, 7.1 Hz, $1H_{B+A}$); 2.59–2.66 and 2.71–2.78 (2m, $2H_{A+B}$); 3.58–3.67 (m, $2H_{A+B}$); 3.85–3.96 (m, $1H_{A+B}$); 4.16 (q, *J* = 7.1 Hz, $2H_{A+B}$); 4.31–4.40 (m, $1H_{A+B}$); 4.45–4.57 (m, $1H_{A+B}$). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q_A+q_B); 40.0 (t_B); 40.2 (t_A); 40.7 (t_B); 40.9 (t_A); 60.8 (t_A+t_B); 62.4 (t_A); 63.0 (t_B); 72.7 (d_A); 73.2 (d_B); 74.5 (d_A); 74.6 (d_B); 85.5 (d_A); 87.2 (d_B); 171.4 (s_B); 171.7 (s_A). HRMS (+ESI): *m/z* calcd. for $C_9H_{17}O_5$ (M+H) 205.1076, found 205.1065.

General procedure for TBDPS protection:

TBDPSCl (0.95 eq) was added to a solution of diol **4a** or **4b** (1 eq.), Et_3N (2 eq.) and DMAP (0.1 eq.) in CH_2Cl_2. The reaction mixture was stirred at r.t. for 48 h. After this time, the reaction mixture was washed with 1M aqueous HCl, dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-CH_2Cl_2-Et_2O (50:30:20) yielded **5a** (45%) and **5b** (28%) or **5c** (28%) and **5d** (48%) respectively as colorless oils.

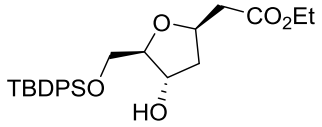
Ethyl 2-[(2*S*,4*S*,5*R*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-hydroxytetrahydrofuran-2-yl]acetate (5a).



$[\alpha]_D$ = +14.0 (c 1.0, $CHCl_3$). IR (KBr film) ν 3450, 2931, 2857, 1735, 1427, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.06 (s, 9H); 1.25 (t, *J*

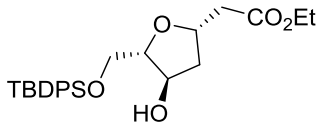
= 7.1 Hz, 3H); 1.79 (ddd, *J* = 13.2, 6.3, 4.6 Hz, 1H); 2.47 (dt, *J* = 13.2, 7.2 Hz, 1H); 2.63 (dd, *J* = 15.7, 6.0 Hz, 1H); 2.72 (dd, *J* = 15.7, 6.4 Hz, 1H); 3.62 (dd, *J* = 10.6, 6.0 Hz, 1H); 3.75 (dd, *J* = 10.6, 3.9 Hz, 1H); 3.98 (dt, *J* = 6.0, 3.9 Hz, 1H); 4.15 (q, *J* = 7.1 Hz, 2H); 4.42–4.53 (m, 2H); 7.35–7.45 (m, 6H); 7.63–7.69 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q); 19.2 (s); 26.8 (q); 39.9 (t); 40.9 (t); 60.5 (t); 64.8 (t); 74.5 (d); 75.0 (d); 85.7 (d); 127.7 (d); 129.8 (d); 133.1 (s); 135.5 (d); 171.4 (s). HRMS (+ESI): *m/z* calcd. for $C_{25}H_{34}O_5NaSi$ (M+Na) 465.2073, found 465.2083.

Ethyl 2-[(2*R*,4*S*,5*R*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-hydroxytetrahydrofuran-2-yl]acetate (5b).



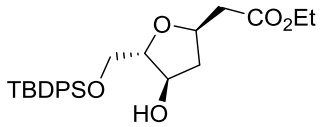
$[\alpha]_D$ = +12.1 (c 1.0, $CHCl_3$). IR (KBr film) ν 3450, 2931, 2857, 1735, 1427, 1112 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (s, 9H); 1.25 (t, *J* = 7.1 Hz, 3H); 1.84 (ddd, *J* = 13.1, 9.6, 6.2 Hz, 1H); 2.06 (ddd, *J* = 13.1, 5.7, 2.3 Hz, 1H); 2.48 (dd, *J* = 15.4, 6.0 Hz, 1H); 2.64 (dd, *J* = 15.4, 7.1 Hz, 1H); 3.58 (dd, *J* = 10.6, 6.0 Hz, 1H); 3.76 (dd, *J* = 10.6, 3.9 Hz, 1H); 3.86–3.90 (m, 1H); 4.14 (q, *J* = 7.1 Hz, 2H); 4.43-4.48 (m, 1H); 4.51–4.60 (m, 1H); 7.35–7.45 (m, 6H); 7.63–7.69 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q); 19.2 (s); 26.8 (q); 40.5 (t); 40.6 (t); 60.5 (t); 64.6 (t); 74.3 (d); 74.6 (d); 86.9 (d); 127.7 (d); 129.7 (d); 133.1 (s); 135.5 (d); 171.0 (s). HRMS (+ESI): *m/z* calcd. for $C_{25}H_{34}O_5NaSi$ (M+Na) 465.2073, found 465.2083.

Ethyl 2-[(2*S*,4*R*,5*S*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-hydroxytetrahydrofuran-2-yl]acetate (5c).



$[\alpha]_D$ = −11.2 (c 1.0, $CHCl_3$). IR (KBr film) ν 3449 (br), 2931, 2857, 1736, 1428, 1113 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.06 (s, 9H); 1.25 (t, *J* = 7.1 Hz, 3H); 1.84 (ddd, *J* = 13.1, 9.6, 6.2 Hz, 1H); 2.07 (ddd, *J* = 13.1, 5.8, 2.3 Hz, 1H); 2.48 (dd, *J* = 15.4, 5.8 Hz, 1H); 2.64 (dd, *J* = 15.4, 7.1 Hz, 1H); 3.59 (dd, *J* = 10.6, 6.0 Hz, 1H); 3.76 (dd, *J* = 10.6, 3.8 Hz, 1H); 3.86–3.90 (m, 1H); 4.14 (q, *J* = 7.1 Hz, 2H); 4.43–4.47 (m, 1H); 4.55 (ddt, *J* = 9.6, 7.1, 5.8 Hz, 1H); 7.35–7.45 (m, 6H); 7.63–7.69 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q); 19.2 (s); 26.8 (q); 40.6 (t); 40.6 (t); 60.5 (t); 64.6 (t); 74.3 (d); 74.6 (d); 86.9 (d); 127.7 (d); 129.7 (d); 133.1 (s); 133.2 (s); 135.5 (d); 135.6 (d); 171.0 (s). HRMS (+ESI): *m/z* calcd. for $C_{25}H_{34}O_5NaSi$ (M+Na) 465.2073, found 465.2067.

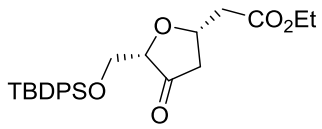
Ethyl 2-[(2*R*,4*R*,5*S*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-hydroxytetrahydrofuran-2-yl]acetate (5d).



$[\alpha]_D$ = −15.1 (c 1.0, CH_2Cl_2). IR (KBr film) ν 3449 (br), 2931, 2857, 1736, 1428, 1113 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.06 (s, 9H); 1.25 (t, *J* = 7.1, 3H); 1.70 (ddd, *J* = 13.5, 6.2, 4.7 Hz, 1H); 2.48 (dt, *J* = 13.5, 7.2 Hz, 1H); 2.55 (dd, *J* = 15.6, 6.0 Hz, 1H); 2.75 (dd, *J* = 15.6, 7.6 Hz, 1H); 3.62 (dd, *J* = 10.5, 6.1 Hz, 1H); 3.76 (dd, *J* = 10.5, 4.1 Hz, 1H); 3.97 (m, 1H); 4.15 (q, *J* = 7.1 Hz, 2H); 4.45–4.52 (m, 2H); 7.35–7.45 (m, 6H); 7.63–7.69 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.3 (q); 19.3 (s); 27.0

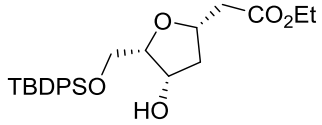
(q); 40.0 (t); 41.0 (t); 60.7 (t); 65.0 (t); 74.8 (d); 75.1 (d); 85.9 (d); 127.9 (d); 129.9 (d); 133.2 (s); 135.7 (d); 171.6 (s). HRMS (+ESI): *m/z* calcd. for $C_{25}H_{38}NO_5Si$ (M+NH_4) 460.2514, found 460.2511.

Ethyl 2-[(2*S*,5*S*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-oxotetrahydrofuran-2-yl]acetate (S2).



DMP (10.4 g, 24.5 mmol) was added to a solution of alcohol **5c** (8.35 g, 18.8 mmol) in CH_2Cl_2 (100 mL) and was stirred for 2 h. The reaction mixture was dissolved with sat. $Na_2S_2O_3$ and sat. $NaHCO_3$ and the residue was extracted with Et_2O. The organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (80:20) yielded **S2** (7.45 g, 90%) as a colorless oil. $[\alpha]_D$ = –94.4 (c 1.0, $CHCl_3$). IR (KBr film) ν 2931, 2858, 1762, 1737, 1472, 1428, 1194, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.03 (s, 9H); 1.29 (t, *J* = 7.1 Hz, 3H); 2.32 (dd, *J* = 17.7, 10.3 Hz, 1H); 2.69–2.77 (m, 2H); 2.94 (dd, *J* = 15.8, 6.7 Hz, 1H); 3.87 (dd, *J* = 11.6, 2.8 Hz, 1H); 3.90–3.94 (m, 2H); 4.21 (qd, *J* = 7.1, 1.8 Hz, 2H); 4.65 (dtd, *J* = 10.3, 6.7, 5.8 Hz, 1H); 7.36–7.45 (m, 6H); 7.65–7.74 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q); 19.2 (s); 26.7 (q); 40.9 (t); 43.5 (t); 60.8 (t); 63.0 (t); 72.2 (d); 82.2 (d); 127.7 (d); 129.7 (d); 132.7 (s); 132.9 (s); 135.6 (d); 170.3 (s); 213.7 (s). HRMS (+ESI): *m/z* calcd. for $C_{25}H_{32}O_5NaSi$ (M+Na) 463.1911, found 463.1910.

Ethyl 2-[(2*S*,4*S*,5*S*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-hydroxytetrahydrofuran-2-yl]acetate (5e).



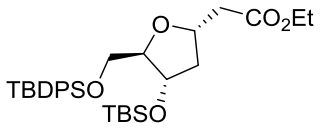
$NaBH_4$ (953 mg, 25.2 mmol) was added to a solution of ketone **S2** (5.56 g, 12.6 mmol) and $CeCl_3 \cdot 7H_2O$ (5.16 g, 13.9 mmol) in EtOH (200 mL) at –20 ºC. The reaction mixture was stirred at this temperature for 40 min. After this time, NH_4Cl was added and the solvent was removed under reduced pressure. The residue was extracted with EtOAc, and the organic layer was dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (80:20) yielded **5e** (4.8 g, 86%) as a colorless oil. $[\alpha]_D$ = –5.0 (c 1.0, $CHCl_3$). IR (KBr film) ν 3469, 2932, 2858, 1735, 1472, 1428, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.06 (s, 9H); 1.24 (t, *J* = 7.1 Hz, 3H); 1.80 (ddd, *J* = 13.4, 6.0, 3.1 Hz, 1H); 2.42 (ddd, *J* = 13.4, 7.9, 6.2 Hz, 1H); 2.65 (dd, *J* = 16.0, 6.0 Hz, 1H); 2.77 (dd, *J* = 16.0, 6.9 Hz, 1H); 3.81 (dt, *J* = 5.7, 4.2 Hz, 1H); 3.96 (dd, *J* = 10.9, 4.2 Hz, 1H); 4.00 (dd, *J* = 10.9, 5.7 Hz, 1H); 4.14 (q, *J* = 7.1 Hz, 2H); 4.34 (ddt, *J* = 7.9, 6.9, 6.0 Hz, 1H); 4.48–4.53 (m, 1H); 7.36–7.45 (m, 6H); 7.65–7.73 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.2 (q); 19.1 (s); 26.8 (q); 40.5 (t); 40.8 (t); 60.5 (t); 63.1 (t); 73.6 (d); 74.0 (d); 81.5 (d); 127.8 (d); 129.9 (d); 133.5 (s); 133.8 (s); 135.5 (d); 135.6 (d); 171.4 (s). HRMS (+ESI): *m/z* calcd. for $C_{25}H_{35}O_5Si$ (M+H) 443.2248, found 443.2251.

General procedure for TBS protection:

TBSCl (1.2 eq.) was added to a solution of alcohol **5** (1 eq.) and imidazole (1 eq.) in CH_2Cl_2 (180 mL). The reaction mixture was stirred at r.t. for 6 or 48 h. After this time, the mixture was washed with water, dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure.

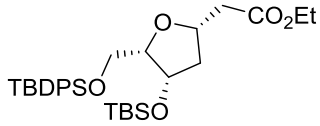
Purification by silica gel column chromatography with hexane-EtOAc (90:10) yielded the corresponding protected adduct **S3** as a colorless oil.

Ethyl 2-[(2*S*,4*S*,5*R*)-4-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetra-hydrofuran-2-yl]acetate (S3a).



Alcohol **5a** (8.8 g, 20 mmol) led to **S3a** (9.99 g, 90%). $[\alpha]_D$ = +26.0 (c 1.0, $CHCl_3$). IR (KBr film) ν 2955, 2930, 2857, 1737, 1471, 1428, 1256, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.05 (s, 3H); 0.06 (s, 3H); 0.88 (s, 9H); 1.05 (s, 9H); 1.25 (t, *J* = 7.1 Hz, 3H); 1.71 (dt, *J* = 13.0, 4.4 Hz, 1H); 2.29 (dt, *J* = 13.0, 6.7 Hz, 1H); 2.60 (dd, *J* = 15.3, 6.7 Hz, 1H); 2.78 (dd, *J* = 15.3, 7.1 Hz, 1H); 3.57 (dd, *J* = 11.0, 5.2 Hz, 1H); 3.64 (dd, *J* = 11.0, 3.8 Hz, 1H); 3.94–3.98 (m, 1H); 4.15 (q, *J* = 7.1 Hz, 2H); 4.45–4.49 (m, 1H); 4.57–4.50 (m, 1H); 7.34–7.45 (m, 6H); 7.64–7.69 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.7 (q); –4.8 (q); 14.2 (q); 17.9 (s); 19.2 (s); 25.7 (q); 26.8 (q); 40.2 (t); 41.6 (t); 60.3 (t); 64.2 (t); 73.6 (d); 75.4 (d); 86.8 (d); 127.7 (d); 129.6 (d); 129.7 (d); 133.2 (s); 133.4 (s); 135.6 (d); 171.5 (s). HRMS (+ESI): *m/z* calcd. for $C_{31}H_{48}O_5NaSi_2$ (M+Na) 579.2938, found 579.2922.

Ethyl 2-[(2*S*,4*S*,5*S*)-4-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetra-hydrofuran-2-yl]acetate (S3b).

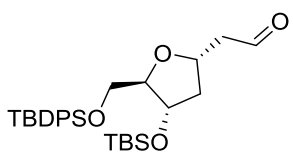


Alcohol **5e** (9.14 g, 20.6 mmol) led to **S3b** (10.7 g, 94%). $[\alpha]_D$ = +17.2 (c 1.0, $CHCl_3$). IR (KBr film) ν 2950, 2931, 2857, 1735, 1471, 1428, 1255, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ –0.01 (s, 3H); 0.04 (s, 3H); 0.83 (s, 9H); 1.07 (s, 9H); 1.24 (t, *J* = 7.1 Hz, 3H); 1.73 (ddd, *J* = 13.2, 4.7, 2.7 Hz, 1H); 2.28 (ddd, *J* = 13.2, 8.0, 5.4 Hz, 1H); 2.58 (dd, *J* = 15.5, 7.4 Hz, 1H); 2.77 (dd, *J* = 15.5, 6.6 Hz, 1H); 3.73–3.81 (m, 1H); 3.83–3.90 (m, 2H); 4.09–4.17 (m, 2H); 4.33–4.52 (m, 2H); 7.34–7.45 (m, 6H); 7.64–7.69 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.2 (q); –4.7 (q); 14.2 (q); 17.9 (s); 19.2 (s); 25.7 (q); 26.9 (q); 40.1 (t); 41.8 (t); 60.2 (t); 63.3 (t); 72.4 (d); 74.1 (d); 83.9 (d); 127.6 (d); 129.5 (d); 133.6 (s); 133.8 (s); 135.6 (d); 135.7 (d); 171.5 (s). HRMS (+ESI): *m/z* calcd. for $C_{31}H_{48}O_5NaSi_2$ (M+Na) 579.2938, found 579.2932.

General procedure for ester reduction:

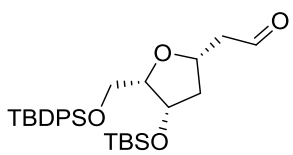
A 1 M solution of DIBALH in heptane (1 eq.) was added to a solution of ester **S3** (1 eq.) in CH_2Cl_2 at –78 ºC. The reaction mixture was stirred at this temperature for 15 min and MeOH and a saturated solution of NaK tartrate were added, the mixture was stirred at r.t. for further 2 h. After this time, water was added and the residue was extracted with CH_2Cl_2. The organic solution was dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10) yielded the corresponding aldehyde **6** as a colorless oil.

2-[(2*S*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]acetaldehyde (6a).



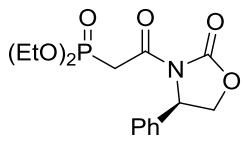
Ester **S3a** (1.65 g, 2.96 mmol) led to aldehyde **6a** (1.43 g, 94%). $[\alpha]_D$ = +28.9 (c 1.0, $CHCl_3$). IR (KBr film) ν 2955, 2930, 2857, 1726, 1471, 1428, 1256, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.06 (s, 3H); 0.07 (s, 3H); 0.89 (s, 9H); 1.07 (s, 9H); 1.69 (ddd, *J* = 13.0, 5.3, 4.0 Hz, 1H); 2.34 (ddd, *J* = 13.0, 7.4, 6.2 Hz, 1H); 2.66 (ddd, *J* = 16.7, 5.3, 2.0 Hz, 1H); 2.90 (ddd, *J* = 16.7, 7.4, 2.0 Hz, 1H); 3.61 (dd, *J* = 11.0, 5.0 Hz, 1H); 3.66 (dd, *J* = 11.0, 3.8 Hz, 1H); 3.95–3.99 (m, 1H); 4.48–4.52 (m, 1H); 4.60 (tt, *J* = 7.4, 5.3 Hz, 1H); 7.36–7.46 (m, 6H); 7.66–7.70 (m, 4H); 9.82 (t, *J* = 2.0, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.7 (q); –4.8 (q); 17.9 (s); 19.2 (s); 25.7 (q); 26.8 (q); 40.5 (t); 50.5 (t); 64.1 (t); 73.5 (d); 73.9 (d); 86.8 (d); 127.7 (d); 129.7 (d); 133.2 (s); 133.3 (s); 135.6 (d); 201.6 (s). HRMS (+ESI): *m/z* calcd. for $C_{29}H_{44}O_4NaSi_2$ (M+Na) 535.2670, found 535.2672.

2-[(2*S*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]acetaldehyde (6b).



Ester **S3b** (7.0 g, 12.6 mmol) led to aldehyde **6b** (6.43 g, 99%). $[\alpha]_D$ = +22.9 (c 1.0, $CHCl_3$). IR (KBr film) ν 2955, 2930, 2857, 1726, 1472, 1428, 1256, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ –0.02 (s, 3H); 0.02 (s, 3H); 0.81 (s, 9H); 1.07 (s, 9H); 1.66 (ddd, *J* = 13.4, 4.9, 2.5 Hz, 1H); 2.33 (ddd, *J* = 13.4, 8.2, 5.5 Hz, 1H); 2.63 (ddd, *J* = 16.8, 5.6, 1.8 Hz, 1H); 2.85 (ddd, *J* = 16.8, 7.1, 2.1 Hz, 1H); 3.74–3.82 (m, 1H); 3.83–3.90 (m, 2H); 4.33–4.37 (m, 1H); 4.38–4.47 (m, 1H); 7.36–7.46 (m, 6H); 7.66–7.70 (m, 4H); 9.78 (t, *J* = 1.9 Hz, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.2 (q); –4.7 (q); 17.9 (s); 19.2 (s); 25.7 (q); 26.9 (q); 41.0 (t); 50.7 (t); 63.4 (t); 72.4 (d); 72.6 (d); 84.1(d); 127.6 (d); 129.5 (d); 129.6 (d); 133.5 (s); 133.8 (s); 135.6 (d); 135.7 (d); 201.7 (s). HRMS (+ESI): *m/z* calcd. for $C_{29}H_{44}O_4NaSi_2$ (M+Na) 535.2670, found 535.2673.

Diethyl (*R*)-2-oxo-2-(2-oxo-4-phenyloxazolidin-3-yl)ethylphosphonate (7). [4]

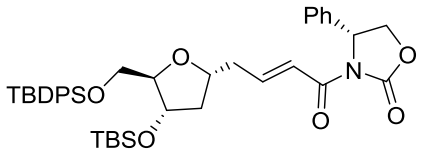


A 50% solution of T3P in EtOAc (13 mL, 21.8 mmol) was added to a solution of (*R*)-4-phenyl-2-oxazolidinone (2.45 g, 15 mmol) and diethylphosphonoacetic acid (2.65 mL, 16.5 mmol) in EtOAc (100 mL) and the mixture was stirred at reflux temperature for 48 h. After this time, water was added, and the solution was treated with a 20% solution of NaOH until pH = 6. The organic phase was washed with water, dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (50:50 to 30:70) yielded **7** (4.78 g, 94%) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 1.28 (dt, *J* = 10.3, 7.0 Hz, 6H); 3.76 (dd, *J* = 22.2, 13.9 Hz, 1H); 3.81 (dd, *J* = 22.6, 13.9 Hz, 1H); 3.71–3.86 (m, 4H); 4.11 (m, 2H); 4.28 (dd, *J* = 8.8, 3.9 Hz, 1H); 4.70 (t, *J* = 8.8 Hz, 1H); 5.46 (dd, *J* = 8.8, 3.9 Hz, 1H); 7.30–7.41 (m, 5H).

General procedure for HWE reaction:

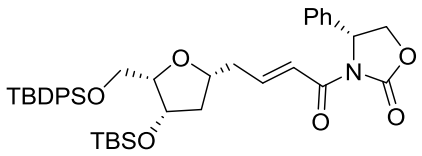
A 1 M solution of NaHMDS in THF (1.3 eq.) was added to a solution of phosphonate 7 (1.4 eq.) in THF. After 10 min, a solution of aldehyde **6** (1 eq.) in THF was added dropwise, and the mixture was stirred at r.t. for 2 h. After this time, KH_2PO_4-NaOH pH =7 buffer was added and the solvent was removed under reduced pressure. The residue was disolved in water and extracted with EtOAc. The organic layer was dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10 to 80:20) yielded the corresponding olefin **S3** as a colorless oil.

(*R*)-3-[(*E*)-4-((2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl)but-2-enoyl]-4-phenyloxazolidin-2-one (S4a).



Aldehyde **6a** (4.18g, 8.15 mmol) led to olefin **S4a** (4.46 g, 78%). $[\alpha]_D$ = −14.1 (c 1.0, $CHCl_3$). IR (KBr film) ν 2954, 2929, 2857, 1781, 1689, 1636, 1383, 1347, 1196, 1256, 1252, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.03 (s, 3H); 0.05 (s, 3H); 0.87 (s, 9H); 1.04 (s, 9H); 1.66 (ddd, *J* = 12.8, 5.9, 4.7 Hz, 1H); 2.21 (dt, *J* = 12.8, 6.4 Hz, 1H); 2.49–2.59 (m, 1H); 2.64–2.73 (m, 1H); 3.59 (dd, *J* = 11.0, 4.3 Hz, 1H); 3.63 (dd, *J* = 11.0, 3.8 Hz, 1H); 3.91 (dt, *J* = 4.3, 3.8 Hz, 1H); 4.14–4.20 (m, 1H); 4.28 (dd, *J* = 8.8, 3.9 Hz, 1H); 4.46 (ddd, *J* = 6.4, 4.7, 3.8 Hz, 1H); 4.69 (t, *J* = 8.8 Hz, 1H); 5.49 (dd, *J* = 8.8, 3.9 Hz, 1H); 7.09 (dt, *J* = 15.4, 7.3, 1H); 7.30–7.45 (m, 12H); 7.64–7.68 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.8 (q); 17.9 (s); 19.2 (s); 25.8 (q); 26.8 (q); 39.7 (t); 40.2 (t); 57.7 (d); 64.1 (t); 69.9 (t); 73.3 (d); 77.5 (d); 86.5 (d); 121.9 (d); 126.0 (d); 127.6 (d); 128.6 (d); 129.1 (d); 129.6 (d); 133.2 (s); 133.4 (s); 135.6 (d); 139.0 (s); 148.2 (d); 153.6 (s); 164.3 (s). HRMS (+ESI): *m/z* calcd. for $C_{40}H_{53}O_6NNaSi_2$ (M+Na) 722.3304, found 722.3309.

(*R*)-3-[(*E*)-4-((2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl)but-2-enoyl]-4-phenyloxazolidin-2-one (S4b).

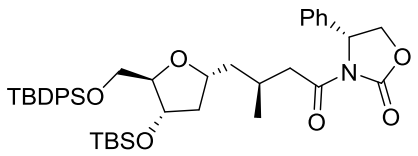


Aldehyde **6b** (5.0 g, 9.8 mmol) led to **S4b** (6.66 g, 95%). $[\alpha]_D$ = −14.8 (c 1.0, $CHCl_3$). IR (KBr film) ν 2954, 2930, 2857, 1781, 1689, 1637, 1384, 1346, 1197, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ –0.03 (s, 3H); 0.02 (s, 3H); 0.81 (s, 9H); 1.07 (s, 9H); 1.65 (ddd, *J* = 13.2, 5.1, 2.7 Hz, 1H); 2.20 (ddd, *J* = 13.2, 7.9, 5.6 Hz, 1H); 2.47–2.56 (m, 1H); 2.64–2.73 (m, 1H); 3.77 (dd, *J* = 9.8, 5.6 Hz, 1H); 3.80–3.84 (m, 1H); 3.88 (dd, *J* = 9.8, 4.4 Hz, 1H); 4.04 (dtd, *J* = 7.9, 6.6, 5.1 Hz, 1H); 4.28 (dd, *J* = 8.8, 3.9 Hz, 1H); 4.33 (ddd, *J* = 5.6, 3.9, 2.7 Hz, 1H); 4.68 (t, *J* = 8.8 Hz, 1H); 5.48 (dd, *J* = 8.7, 3.9 Hz, 1H); 7.08 (ddd, *J* = 15.4, 7.7, 6.6 Hz, 1H); 7.30–7.45 (m, 12H); 7.64–7.68 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.3 (q); –4.7 (q); 17.9 (s); 19.2 (s); 25.7 (q); 26.9 (q); 39.9 (t); 40.5 (t); 57.7 (d); 63.4 (t); 69.9 (t); 73.4 (d); 76.4 (d); 83.9 (d); 121.8 (d); 126.0 (d); 127.5 (d); 128.6 (d); 129.1 (d); 129.5 (d); 133.6 (s); 133.9 (s); 135.6 (d); 139.0 (s); 148.2 (d); 153.6 (s); 164.3 (s). HRMS (+ESI): *m/z* calcd. for $C_{40}H_{53}O_6NNaSi_2$ (M+Na) 722.3304, found 722.3300.

General procedure for 1,4-addition:

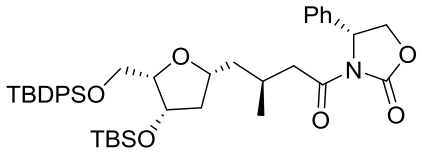
A 1.4 M solution of MeMgBr in THF (1.1 eq.) was added to a solution of CuBr·Me_2S (1.1 eq.) in THF at −40 ºC, and the mixture was stirred at −40 ºC for 1 h. The solution was cooled to −78 ºC and BF_3·Et_2O (1.1 eq.) was added, followed by a solution of oxazolidinone **S4** (1 eq.) in THF. The reaction mixture was stirred at -78 ºC for 1 h, slowly warmed to r.t. during 2 h and stirred at r.t. for further 1 hour. After this time, sat. NH_4Cl was added and the solvent was removed under reduced pressure. The residue was diluted in sat. NH_4Cl and extracted with Et_2O. The organic solution was dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10 to 80:20) yielded the corresponding methylated **8a** as a colorless oil.

(*R*)-3-[(*S*)-4-((2*R*,4*S*,5*R*)-4-(tert-Butyldimethylsilyloxy)-5-(tert-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl)-3-methylbutanoyl]-4-phenyloxazolidin-2-one (8a).



Olefin **S4a** (4.27 g, 6.1 mmol) led to methylated **8a** (3.67 g, 84%). $[\alpha]_D$ = −40.0 (c 1.0, $CHCl_3$). IR (KBr film) ν 2956, 2930, 2857, 1784, 1707, 1471, 1428, 1384, 1322, 1252, 1196, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.04 (s, 3H); 0.06 (s, 3H); 0.88 (s, 9H); 0.94 (d, *J* = 6.7 Hz, 3H); 1.05 (s, 9H); 1.46−1.69 (m, 3H); 2.09−2.25 (m, 2H); 2.85 (dd, *J* = 16.7, 7.4 Hz, 1H); 2.96 (dd, *J* = 16.7, 6.2 Hz, 1H); 3.64 (dd, *J* = 11.0, 3.9 Hz, 1H); 3.69 (dd, *J* = 11.0, 3.9 Hz, 1H); 3.81 (dt, *J* = 4.5, 3.9 Hz, 1H); 4.07−4.16 (m, 1H); 4.24 (dd, *J* = 8.8, 3.6 Hz, 1H); 4.47 (td, *J* = 6.4, 4.5 Hz, 1H); 4.62 (t, *J* = 8.8 Hz, 1H); 5.40 (dd, *J* = 8.8, 3.6 Hz, 1H); 7.27−7.45 (m, 11H); 7.66−7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −4.8 (q); −4.6 (q); 17.9 (s); 19.2 (s); 20.1 (q); 25.8 (q); 26.8 (q); 27.5 (d); 41.4 (t); 42.3 (t); 42.9 (t); 57.6 (d); 64.0 (t); 69.8 (t); 72.9 (d); 76.8 (d); 85.5 (d); 125.8 (d); 127.6 (d); 129.1 (d); 128.6 (d); 129.1 (d); 129.5 (d); 129.6 (d); 133.4 (s); 133.5 (s); 135.6 (d); 139.3 (s); 153.6 (s); 171.9 (s). HRMS (+ESI): *m/z* calcd. for $C_{41}H_{57}O_6NNaSi_2$ (M+Na) 738.3617, found 738.3614.

(*R*)-3-[(*S*)-4-((2*R*,4*S*,5*S*)-4-(tert-Butyldimethylsilyloxy)-5-(tert-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl)-3-methylbutanoyl]-4-phenyloxazolidin-2-one (8b).



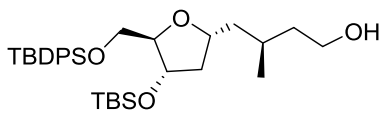
Olefin **S4b** (8.0 g, 11.4 mmol) led to methylated **8b** (7.73 g, 81%). $[\alpha]_D$ = −11.1 (c 1.0, $CHCl_3$). IR (KBr film) ν 2956, 2930, 2857, 1783, 1707, 1471, 1428, 1384, 1325, 1252, 1198, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ −0.04 (s, 3H); 0.01 (s, 3H); 0.80 (s, 9H); 0.92 (d, *J* = 6.7 Hz, 3H); 1.06 (s, 9H); 1.49 (dt, *J* = 13.6, 5.9 Hz, 1H); 1.53 (ddd, *J* = 16.6, 6.3, 3.8 Hz, 1H); 1.67 (dt, *J* = 13.6, 7.5 Hz, 1H); 2.10−2.22 (m, 2H); 2.80 (dd, *J* = 16.7, 7.6 Hz, 1H); 2.96 (dd, *J* = 16.7, 6.1 Hz, 1H); 3.71−3.79 (m, 2H); 3.81−3.94 (m, 2H); 4.23 (dd, *J* = 8.8, 3.6 Hz, 1H); 4.31 (dt, *J* = 6.0, 3.8 Hz, 1H); 4.61 (t, *J* = 8.8 Hz, 1H) 5.39 (dd, *J* = 8.8, 3.6 Hz, 1H); 7.27−7.46 (m, 11H); 7.66−7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −5.2 (q); −4.6 (q); 18.0 (s); 19.2 (s); 20.2 (q); 25.8 (q); 26.9 (q); 27.4 (d); 41.5 (t); 42.2 (t); 43.2 (t); 57.6 (d); 63.6 (t); 69.8 (t); 72.5 (d); 75.8 (d); 83.3 (d); 125.9 (d); 127.5 (d); 128.6 (d); 129.1 (d); 129.4 (d); 133.7 (s); 134.0 (s);

135.6 (d); 135.7 (d); 139.2 (s); 153.7 (s); 171.9 (s). HRMS (+ESI): *m/z* calcd. for $C_{41}H_{57}O_6NNaSi_2$ (M+Na) 738.3617, found 738.3612.

General procedure for oxazolidinone removal:

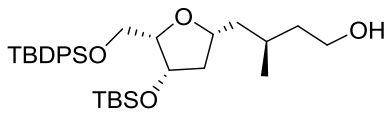
A 2 M solution of $LiBH_4$ in THF (2 eq.) was added to a solution of olefin **8** (1 eq.) in Et_2O at –10 ºC and the reaction mixture was stirred at 0 ºC for 1 h. After this time, a 1 M solution of NaOH was added and the mixture was extracted with EtOAc, dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10 to 85:15) yielded the corresponding alcohol **S5** as a colorless oil.

(*R*)-4-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydro-furan-2-yl]-3-methylbutan-1-ol (S5a).



Oxazolidinone **8a** (3.67 g, 5.12 mmol) led to alcohol **S5a** (2.25 g, 79%). $[\alpha]_D$ = +25.8 (c 1.0, $CHCl_3$). IR (KBr film) ν 3395 (br) 2955, 2929, 2857, 1472, 1428, 1252, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.04 (s, 3H); 0.05 (s, 3H); 0.87 (s, 9H); 0.95 (d, *J* = 6.6 Hz, 3H); 1.05 (s, 9H); 1.38–1.69 (m, 5H); 1.70–1.78 (m, 1H); 2.19–2.26 (m, 1H); 3.61–3.73 (m, 4H); 3.85 (dt, *J* = 4.2, 4.0 Hz, 1H); 4.16–4.23 (m, 1H); 4.49 (td, *J* = 6.3, 4.4 Hz, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.8 (q); –4.7 (q); 17.9 (s); 19.2 (s); 20.4 (q); 25.8 (q); 26.8 (q); 27.2 (d); 39.7 (t); 41.3 (t); 43.3 (t); 61.0 (t); 64.1 (t); 73.0 (d); 76.8 (d); 85.7 (d); 127.6 (d); 129.6 (d); 133.4 (s); 133.5 (s); 135.6 (d). HRMS (+ESI): *m/z* calcd. for $C_{32}H_{52}O_4NaSi_2$ (M+Na) 579.3296, found 579.3296.

(*R*)-4-[(2*R*,4*S*,5*S*)-4-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydro-furan-2-yl]-3-methylbutan-1-ol (S5b).



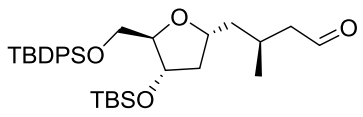
Oxazolidinone **8b** (2.3 g, 3.21 mmol) led to alcohol **S5b** (1.50 g, 84%). $[\alpha]_D$ = +12.8 (c 1.0, $CHCl_3$). IR (KBr film) ν 3408 (br), 2955, 2930, 2857, 1471, 1428, 1254, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ –0.04 (s, 3H); 0.01 (s, 3H); 0.80 (s, 9H); 0.93 (d, *J* = 6.6 Hz, 3H); 1.06 (s, 9H); 1.41 (td, *J* = 13.6, 6.8 Hz, 1H); 1.46–1.57 (m, 2H); 1.61–1.69 (m, 2H); 1.72–1.80 (m, 1H); 2.21 (ddd, *J* = 12.7, 7.2, 6.2 Hz, 1H); 3.60–3.73 (m, 2H); 3.74–3.80 (m, 2H); 3.83–3.89 (m, 1H); 3.94–4.02 (m, 1H); 4.33 (dt, *J* = 6.2, 3.9 Hz, 1H); 7.33–7.43 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.2 (q); –4.6 (q); 18.0 (s); 19.2 (s); 20.6 (q); 25.7 (q); 26.9 (q); 27.0 (d); 39.8 (t); 41.5 (t); 43.5 (t); 61.1 (t); 63.6 (t); 72.6 (d); 75.9 (d); 83.3 (d); 127.5 (d); 129.4 (d); 129.5 (d); 133.7 (s); 133.9 (s); 135.6 (d); 135.7 (d). HRMS (+ESI): *m/z* calcd. for $C_{32}H_{52}O_4NaSi_2$ (M+Na) 579.3296, found 579.3272.

General procedure for alcohol oxidation:

Dess-Martin Periodinane (DMP**,** 1,1,1-Tris(acetyloxy)-1,1-dihydro-1,2-benziodoxol-3-(1*H*)-one) (1.2 eq.) was added to a solution of alcohol **S5** (1 eq.) in CH_2Cl_2 and was stirred for 2 h. The reaction mixture was diluted with sat. $Na_2S_2O_3$ and sat. $NaHCO_3$ and the residue was extracted with CH_2Cl_2. The organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced

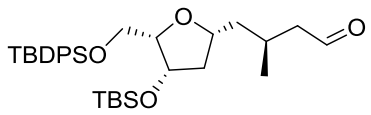
pressure. Purification by silica gel column chromatography with hexane-EtOAc (95:5) yielded the corresponding aldehyde **9** as a colorless oil.

(*S*)-4-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydro-furan-2-yl]-3-methylbutanal (9a).



Alcohol **S5a** (2.25 g, 4 mmol) led to aldehyde **9a** (2.04 g, 92%). $[\alpha]_D$ = +22.2 (c 1.0, $CHCl_3$). IR (KBr film) ν 2956, 2930, 2857, 1727, 1472, 1428, 1252, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.05 (s, 3H); 0.06 (s, 3H); 0.88 (s, 9H); 1.01 (d, *J* = 6.5 Hz, 3H); 1.06 (s, 9H); 1.50 (ddd, *J* = 14.1, 6.6, 5.1 Hz, 1H); 1.61 (ddd, *J* = 12.8, 7.3, 5.8 Hz, 1H); 1.73 (ddd, *J* = 14.1, 8.3, 6.4 Hz, 1H); 2.18–2.29 (m, 3H); 2.45–2.55 (m, 1H); 3.66 (dd, *J* = 11.1, 3.9 Hz, 1H); 3.70 (dd, *J* = 11.1, 4.1 Hz, 1H); 3.85 (dt, *J* = 4.1, 3.9 Hz, 1H); 4.11–4.19 (m, 1H); 4.47–4.53 (m, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H); 9.74 (t, *J* = 2.0 Hz, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.8 (q); –4.7 (q); 17.9 (s); 19.2 (s); 20.5 (q); 25.8 (q); 25.8 (d); 26.8 (q); 41.4 (t); 43.2 (t); 50.6 (t); 64.1 (t); 73.0 (d); 76.4 (d); 85.8 (d); 127.6 (d); 129.6 (d); 133.4 (s); 133.5 (s); 135.6 (d); 202.9 (d). HRMS (+ESI): *m/z* calcd. for $C_{32}H_{50}O_4NaSi_2$ (M+Na) 577.3140, found 577.3142.

(*S*)-4-[(2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydro-furan-2-yl]-3-methylbutanal (9b)

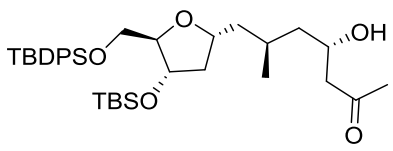


Alcohol **S5b** (5.70 g, 10.24 mmol) led to aldehyde **9b** (5.2 g, 92%). $[\alpha]_D$ = +11.9 (c 1.0, $CHCl_3$). IR (KBr film) ν 2955, 2930, 2857, 1726, 1472, 1428, 1255, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ –0.04 (s, 3H); 0.01 (s, 3H); 0.80 (s, 9H); 0.99 (d, *J* = 6.5 Hz, 3H); 1.06 (s, 9H); 1.48 (ddd, *J* = 13.9, 6.7, 5.3 Hz, 1H); 1.56 (ddd, *J* = 12.9, 6.4, 3.4 Hz, 1H); 1.73 (ddd, *J* = 13.9, 8.0, 6.3 Hz, 1H); 2.15–2.30 (m, 3H); 2.42–2.53 (m, 1H); 3.74–3.82 (m, 2H); 3.83–3.88 (m, 1H); 3.91–3.98 (m, 1H); 4.30–4.35 (m, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H); 9.73 (t, *J* = 2.1 Hz, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.2 (q); –4.6 (q); 18.0 (s); 19.2 (s); 20.6 (q); 25.7 (q); 25.8 (d); 26.9 (q); 41.6 (t); 43.4 (t); 50.6 (t); 63.7 (t); 72.5 (d); 75.4 (d); 83.5 (d); 127.5 (d); 129.5 (d); 133.7 (s); 133.9 (s); 135.6 (d); 135.7 (d); 203.1 (d). HRMS (+ESI): *m/z* calcd. for $C_{32}H_{50}O_4NaSi_2$ (M+Na) 577.3140, found 577.3147.

General procedure for stereoselective aldol addition:

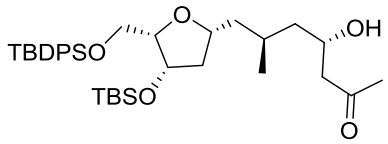
Acetone (5.5 eq.) and Et_3N (5 eq.) were added to a solution of (–)-B-chlorodiisopinocampheylborane (5 eq.) in Et_2O at 0 ºC and the solution was stirred at 0 ºC for 45 min. The solution was cooled to −78 ºC, a solution of aldehyde **9** (1 eq.) was added and the reaction mixture was stirred at −78 ºC for 1 h and at −20 ºC for 16 h. After this time, H_2O_2, KH_2PO_4-NaOH pH = 7 buffer and MeOH were added and stirring continued for further 1 h. The reaction mixture was diluted with water and extracted with Et_2O and EtOAc. The organic extracts were dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10 to 80:20) yielded the corresponding aldol **10** as a colorless oil.

(4*S*,6*R*)-7-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-4-hydroxy-6-methylheptan-2-one (10a).



Aldehyde **9a** (2.04 g, 3.7 mmol) led to aldol **10a** (1.51 g, 67%) (dr = 8:1). $[\alpha]_D$ = +36.1 (c 1.0, $CHCl_3$). IR (KBr film) ν 3454 (br), 2955, 2929, 2857, 1711, 1478, 1428, 1361, 1257, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.03 (s, 3H); 0.05 (s, 3H); 0.87 (s, 9H); 0.95 (d, *J* = 6.7 Hz, 3H); 1.05 (s, 9H); 1.36–1.41 (m, 2H); 1.50–1.64 (m, 3H); 1.71–1.81 (m, 1H); 2.12 (s, 3H); 2.19–2.26 (m, 1H); 2.46 (dd, *J* = 17.6, 9.0 Hz, 1H); 2.56 (dd, *J* = 17.6, 2.7 Hz, 1H); 3.64 (dd, *J* = 10.9, 3.9 Hz, 1H); 3.69 (dd, *J* = 10.9, 3.9 Hz, 1H); 3.83–3.87 (m, 1H); 4.10–4.22 (m, 2H); 4.43–4.49 (m, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.8 (q); –4.6 (q); 17.9 (s); 19.2 (s); 21.0 (q); 25.8 (q); 26.8 (q); 26.9 (d); 30.7 (q); 41.2 (t); 42.8 (t); 43.6 (t); 50.2 (t); 64.1 (t); 65.6 (d); 73.0 (d); 76.9 (d); 85.8 (d); 127.6 (d); 129.6 (d); 133.4 (s); 133.5 (s); 135.6 (d); 210.0 (s). HRMS (+ESI): *m/z* calcd. for $C_{35}H_{57}O_5Si_2$ (M+H) 613.3739, found 613.3743.

(4*S*,6*R*)-7-[(2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-4-hydroxy-6-methylheptan-2-one (10b).

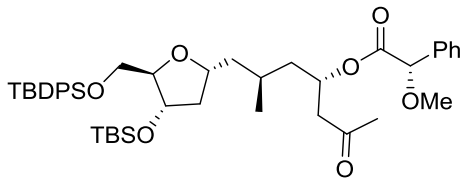


Aldehyde **9b** (1.2 g, 2.16 mmol) led to aldol **10b** (1.06 g, 80%) (dr = 6:1). $[\alpha]_D$ = 19.8 (c 1.0, $CHCl_3$). IR (KBr film) ν 3462 (br), 2955, 2930, 2857, 1711, 1472, 1428, 1361, 1255, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ –0.05 (s, 3H); 0.01 (s, 3H); 0.80 (s, 9H); 0.94 (d, *J* = 6.7 Hz, 3H); 1.06 (s, 9H); 1.38 (t, *J* = 6.8 Hz, 2H); 1.50–1.70 (m, 3H); 1.78 (h, *J* = 6.7 Hz, 1H); 2.12 (s, 3H); 2.21 (ddd, *J* = 13.2, 7.5, 6.1 Hz, 1H); 2.48 (dd, *J* = 17.5, 8.9 Hz, 1H); 2.58 (dd, *J* = 17.5, 3.0 Hz, 1H); 3.16 (bs, 1H); 3.73–3.80 (m, 2H); 3.82–3.88 (m, 1H); 3.94–4.01 (m, 1H); 4.10–4.18 (m, 1H); 4.32 (dt, *J* = 6.1, 3.6 Hz, 1H); 7.33–7.43 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.2 (q); –4.6 (q); 18.0 (s); 19.2 (s); 21.2 (q); 25.7 (q); 26.6 (d); 26.9 (q); 30.8 (q); 41.6 (t); 43.0 (t); 43.6 (t); 50.1 (t); 64.6 (t); 65.6 (d); 72.5 (d); 75.8 (d); 83.4 (d); 127.5 (d); 129.5 (d); 133.7 (s); 133.9 (s); 135.6 (d); 135.7 (d); 210.1 (s). HRMS (+ESI): *m/z* calcd. for $C_{35}H_{57}O_5Si_2$ (M+H) 613.3739, found 613.3729.

General procedure for MPA derivatization:

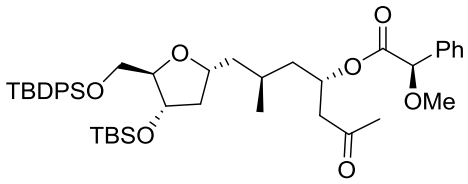
α-Methoxyphenylacetic acid (3 or 6 eq.) and DCC (3 or 6 eq.) were added to a solution of alcohol **10** (1 eq.) in THF, then DMAP (0.1 eq.) was added and the solution was stirred for 30 min. The solution was filtered, poured into Et_2O and washed with 0.2 M aqueous HCl and sat. $NaHCO_3$. The organic residue was dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10) yielded the corresponding ester **S6** as a colorless oil.

(2*S*,4*S*)-1-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-2-methyl-6-oxoheptan-4-yl (*S*)-2-methoxy-2-phenylacetate (S6a).



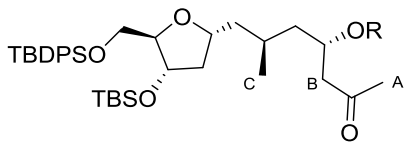
S-α-Methoxyphenylacetic acid (25 mg, 0.15 mmol) and alcohol **10a** (30 mg, 0.05 mmol) led to ester **S6a** (30 mg, 79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.03 (s, 3H); 0.05 (s, 3H); 0.87 (s, 9H); 0.94 (d, *J* = 5.6 Hz, 3H); 1.05 (s, 9H); 1.45–1.60 (m, 6H); 1.81 (s, 3H); 2.17 (dt, *J* = 12.6, 6.4 Hz, 1H); 2.45 (dd, *J* = 16.1, 4.9 Hz, 1H); 2.52 (dd, *J* = 16.1, 7.6 Hz, 1H); 3.37 (s, 3H); 3.64 (dd, *J* = 11.0, 3.9 Hz, 1H); 3.68 (dd, *J* = 11.0, 3.9 Hz, 1H); 3.81 (q, *J* = 3.9 Hz, 1H); 4.03–4.11 (m, 1H); 4.42–4.47 (m, 1H); 4.68 (s, 1H); 5.33–5.41 (m, 1H); 7.30–7.47 (m, 11H); 7.65–7.72 (m, 4H).

(2*S*,4*S*)-1-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-2-methyl-6-oxoheptan-4-yl (*R*)-2-methoxy-2-phenyl-acetate (S6b).



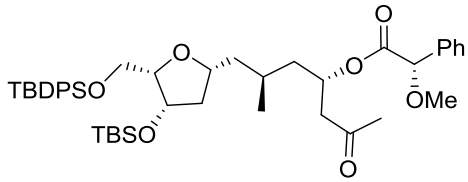
R-α-Methoxyphenylacetic acid (25 mg, 0.15 mmol) and alcohol **10a** (30 mg, 0.05 mmol) led to ester **S6b** (26 mg, 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.03 (s, 3H); 0.05 (s, 3H); 0.78 (d, *J* = 6.5 Hz, 3H); 0.87 (s, 9H); 1.04 (s, 9H); 1.54–1.98 (m, 6H); 2.05 (s, 3H); 2.06–2.10 (m, 1H); 2.56 (dd, *J* = 16.1, 5.0 Hz, 1H); 2.63 (dd, *J* = 16.1, 7.5 Hz, 1H); 3.41 (s, 3H); 3.55–3.70 (m, 2H); 3.75–3.81 (m, 1H); 3.90–4.01 (m, 1H); 4.39–4.44 (m, 1H); 4.68 (s, 1H); 5.30–5.37 (m, 1H); 7.28–7.44 (m, 11H); 7.65–7.72 (m, 4H).

Absolute configuration determination:



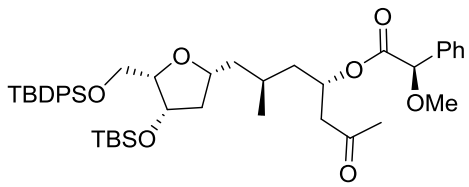
	δ H_A	δ H_B	δ H_C
R=*R*-MPA	2.05	2.59	0.78
R=*S*-MPA	1.81	2.48	0.94
Δ^{RS}	0.24	0.11	–0.16

(2*S*,4*S*)-1-[(2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-2-methyl-6-oxoheptan-4-yl (*S*)-2-methoxy-2-phenylacetate (S6c).



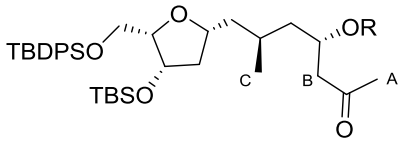
S-Methoxyphenylacetic acid (50 mg, 0.3 mmol) and alcohol **10b** (32 mg, 0.05 mmol) led to ester **S6c** (22 mg, 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.05 (s, 3H); 0.00 (s, 3H); 0.79 (s, 9H); 0.94 (d, *J* = 5.9 Hz, 3H); 1.05 (s, 9H); 1.40–1.65 (m, 6H); 1.81 (s, 3H); 2.12–2.19 (m, 1H); 2.47 (dd, *J* = 16.2, 4.8 Hz, 1H); 2.55 (dd, *J* = 16.2, 7.7 Hz, 1H); 3.38 (s, 3H); 3.72–3.78 (m, 2H); 3.81–3.88 (m, 2H); 4.30 (dt, *J* = 6.1, 3.8 Hz, 1H); 4.69 (s, 1H); 5.30–5.37 (m, 1H); 7.30–7.42 (m, 11H); 7.66–7.72 (m, 4H). HRMS (+ESI): *m/z* calcd. for $C_{44}H_{64}O_7NaSi_2$ (M+Na) 783.4083, found 783.4070.

(2*S*,4*S*)-1-[(2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-2-methyl-6-oxoheptan-4-yl (*R*)-2-methoxy-2-phenylacetate (S6d).



R-Methoxyphenylacetic acid (50 mg, 0.3 mmol) and alcohol **10b** (32 mg, 0.05 mmol) led to ester **S6d** (30 mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.06 (s, 3H); 0.00 (s, 3H); 0.79 (s, 9H); 0.80 (d, *J* = 5.9 Hz, 3H); 1.05 (s, 9H); 1.25–1.53 (m, 6H); 2.05 (s, 3H); 2.05–2.12 (m, 1H); 2.59 (dd, *J* = 16.2, 5.1 Hz, 1H); 2.65 (dd, *J* = 16.2, 7.5 Hz, 1H); 3.40 (s, 3H); 3.67–3.75 (m, 3H); 3.80–3.86 (m, 1H); 4.27 (dt, *J* = 6.2, 3.8 Hz, 1H); 4.68 (s, 1H); 5.28–5.36 (m, 1H); 7.28–7.42 (m, 11H); 7.66–7.72 (m, 4H). HRMS (+ESI): *m/z* calcd. for $C_{44}H_{64}O_7NaSi_2$ (M+Na) 783.4083, found 783.4075.

Absolute configuration determination:

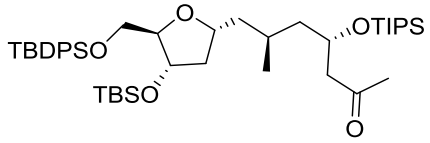


	δ H_A	δ H_B	δ H_C
R = *R*-MPA	2.11	2.68	0.86
R = *S*-MPA	1.86	2.57	0.99
Δ^{RS}	0.25	0.11	–0.13

General procedure for TIPS protection:

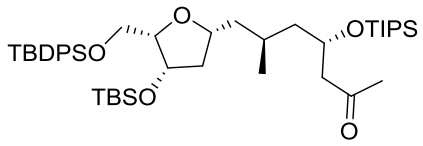
Triisopropylsilyl trifluoromethanesulfonate (1.2 eq.) was added to a solution of **10** (1 eq.), imidazole (2 eq.) and DMAP (0.1 eq.) in DMF, and the reaction mixture was stirred at 95 ºC for 6 h. The solvent was removed under reduced pressure and the residue was dissolved in water and extracted with Et_2O. The organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification by silica gel column chromatography with hexane-Et_2O (95:5) yielded the corresponding protected aldol **11** as a colorless oil.

(4*S*,6*S*)-7-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-6-methyl-4-(triisopropylsilyloxy)heptan-2-one (11a).



Alcohol **10a** (1.5 g, 2.45 mmol) led to protected **11a** (1.79 g, 93%). $[\alpha]_D$ = +27.6 (c 1.0, $CHCl_3$). IR (KBr film) ν 2956, 2930, 2864, 1719, 1476, 1427, 1251, 1112 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.03 (s, 3H); 0.05 (s, 3H); 0.87 (s, 9H); 0.93 (d, *J* = 6.3 Hz, 3H); 1.05–1.06 (3bs, 30H); 1.25–1.65 (m, 6H); 2.11 (s, 3H); 2.23 (dt, *J* = 12.6, 6.5 Hz, 1H); 2.54 (d, *J* = 5.7 Hz, 2H); 3.65 (dd, *J* = 11.0, 3.9 Hz, 1H); 3.70 (dd, *J* = 11.0, 3.9 Hz, 1H); 3.83 (dt, *J* = 4.4, 3.9 Hz, 1H); 4.11–4.19 (m, 1H); 4.36–4.43 (m, 1H); 4.47 (td, *J* = 6.2, 4.4 Hz, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.8 (q); –4.6 (q); 12.7 (d); 17.9 (s); 18.2 (q); 19.2 (s); 21.0 (q); 25.8 (q); 26.8 (q); 27.5 (d); 31.7 (q); 41.6 (t); 44.7 (t); 45.4 (t); 50.9 (t); 64.1 (t); 67.7 (d); 73.1 (d); 76.7 (d); 85.6 (d); 127.6 (d); 129.6 (d); 133.4 (s); 133.5 (s); 135.6 (d); 207.9 (s). HRMS (+ESI): *m/z* calcd. for $C_{44}H_{80}O_5NSi_3$ (M+NH_4) 786.5339, found 786.5365.

(4*S*,6*S*)-7-[(2*R*,4*S*,5*S*)-4-(*tert*-butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)tetrahydrofuran-2-yl]-6-methyl-4-(triisopropylsilyloxy)heptan-2-one (11b).

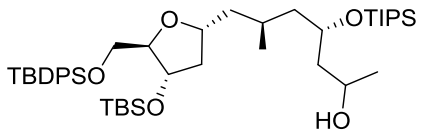


Alcohol **10b** (0.97 g, 1.58 mmol) led to protected **11b** (0.96 g, 77%). $[\alpha]_D$ = 25.6 (c 1.0, $CHCl_3$). IR (KBr film) ν 2931, 2864, 1719, 1463, 1428, 1254, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ −0.05 (s, 3H); 0.00 (s, 3H); 0.80 (s, 9H); 0.91 (d, *J* = 6.3 Hz, 3H); 1.04 and 1.05 (2bs, 30H); 1.30–1.66 (m, 7H); 2.12 (s, 3H); 2.23 (dt, *J* = 13.2, 6.7 Hz, 1H); 2.55 (d, *J* = 5.7 Hz, 1H); 3.72–3.80 (m, 2H); 3.81–3.88 (m, 1H); 3.90–3.97 (m, 1H); 4.32 (dt, *J* = 6.0, 3.7 Hz, 1H); 4.35–4.44 (m, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −5.2 (q); −4.6 (q); 12.6 (d); 17.8 (s); 18.2 (q); 19.2 (s); 20.1 (q); 25.7 (q); 26.9 (q); 27.4 (d); 31.7 (q); 41.9 (t); 44.9 (t); 45.2 (t); 50.8 (t); 63.7 (t); 67.8 (d); 72.6 (d); 75.6 (d); 83.3 (d); 127.5 (d); 129.5 (d); 133.7 (s); 134.0 (s); 135.6 (d); 135.7 (d); 208.0 (s). HRMS (+ESI): *m/z* calcd. for $C_{44}H_{80}O_5NSi_3$ (M+NH4) 786.5339, found 786.5316.

General procedre for ketone reduction:

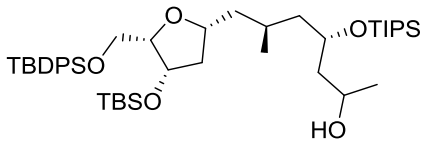
A solution of $NaBH_4$ (1.2 eq.) and ketone **11** (1 eq.) in THF-EtOH 2:1 was stirred at r.t. for 16 h. After this time, aqueous sat. NH_4Cl was added and the residue was extracted with CH_2Cl_2. The organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (95:5) yielded a 6:4 mixture of the corresponding alcohol **S7** as a colorless oil.

(2*RS*,4*S*,6*S*)-7-[(2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)-tetrahydrofuran-2-yl]-6-methyl-4-(triisopropylsilyloxy)heptan-2-ol (S7a).



Ketone **11a** led to a 6:4 mixture of alcohol **S7a** (4.79 g, 89%). IR (KBr film) ν 3506, 2955, 2931, 2865, 1463, 1428, 1252, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.03 (s, 3H); 0.05 (s, 3H); 0.87 (s, 9H); 0.91 (d, *J* = 6.1 Hz, 3H); 1.06–1.09 (3bs, 30H); 1.11 and 1.15 (2d, *J* = 6.2 Hz, 3H); 1.30–1.75 (m, 8H); 2.24 (dt, *J* = 12.8, 6.6 Hz, 1H); 3.62–3.96 (m, 2H); 3.84–3.88 (m, 1H); 3.98–4.06 (m, 1H); 4.12–4.26 (m, 2H); 4.42–4.50 (m, 1H); 7.34–7.44 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −4.8 (q); −4.6 (q); 12.4 (d); 13.1(d); 17.7 (s); 17.9 (s); 18.1 (q); 18.2 (q); 19.2 (s); 19.6 (q); 19.7 (q); 23.8 (q); 23.9 (q); 25.8 (q); 26.8 (q); 27.4 (d); 27.7 (d); 41.2 (t); 41.4 (t); 41.6 (t); 42.6 (t); 44.6 (t); 44.7 (t); 45.1 (t); 45.2 (t); 64.1 (t); 64.2 (d); 66.9 (d); 70.7 (d); 72.1 (d); 73.1 (d); 73.3 (d); 76.9 (d); 85.9 (d); 127.6 (d); 129.6 (d); 133.4 (s); 133.5 (s); 135.6 (d). HRMS (+ESI): *m/z* calcd. for $C_{44}H_{78}O_5NaSi_3$ (M+Na) 793.5049, found 793.5068.

(2*RS*,4*S*,6*S*)-7-[(2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)-tetrahydrofuran-2-yl]-6-methyl-4-(triisopropylsilyloxy)heptan-2-ol (S7b).

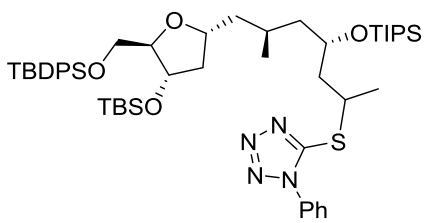


Ketone **11b** (1.81 g, 2.36 mmol) led to a 6:4 mixture of alcohol **S7b** (1.69 g, 92%). IR (KBr film) ν 3456 (br), 2955, 2931, 2863, 1463, 1428, 1255, 1113 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ –0.04 (2s, 3H); 0.01 (s, 3H); 0.81 (2s, 9H); 0.89 (d, *J* = 6.3 Hz, 3H); 1.05 and 1.09 (2s, 9H); 1.08 (bs, 21H); 1.09 and 1.15 (2d, *J* = 6.2 Hz, 3H); 1.30–1.75 (m, 8H); 2.15–2.25 (m, 1H); 3.72–3.80 (m, 2H); 3.83–3.88 (m, 1H); 3.91–4.00 (m, 1H); 4.01–4.07 and 4.11–4.25 (2m, 2H); 4.30–4.35 (m, 1H); 7.34–7.45 (m, 6H); 7.66–7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –5.2 (q); –4.6 (q); 12.3 (d); 13.1 (d); 17.7 (s); 18.0 (s); 18.1 (q); 18.2 (q); 19.2 (s); 19.7 (q); 19.8 (q); 23.8 (q); 25.7 (q); 26.9 (q); 27.4 (d); 27.5 (d); 40.9 (t); 41.7 (t); 41.9 (t); 42.0 (t); 44.3 (t); 45.2 (t); 45.3 (t); 63.5 (t); 63.6 (t); 64.3 (d); 67.0 (d); 70.9 (d); 72.4 (d); 72.5 (d); 72.6 (d); 75.4 (d); 75.5 (d); 83.3 (d); 127.5 (d); 129.4 (d); 129.5 (d); 133.8 (s); 133.9 (s); 135.6 (d); 135.7 (d). HRMS (+ESI): *m/z* calcd. for $C_{44}H_{79}O_5Si_3$ (M+H) 771.5230, found 771.5217.

General procedure for thioester formation by Mitsunobu reaction:

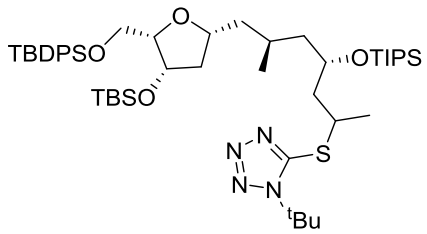
Diisopropyl azodicarboxylate (1.5 eq.) was added to a solution of alcohol **S7a** (1 eq.), 1-R-1*H*-tetrazole-5-thiol (1.5 eq.) and PPh_3 (1.5 eq.) in THF. The reaction mixture was stirred at r.t. for 5 h. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography with hexane-EtOAc (98:2 to 95:5) to yield the corresponding thioester **S8** as a colorless oil.

5-[(2*RS*,4*S*,6*S*)-7-((2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)-tetrahydrofuran-2-yl)-6-methyl-4-(triisopropylsilyloxy)heptan-2-yl-thio]-1-phenyl-1*H*-tetrazole (S8a).



Alcohol **S7a** (4.9 g, 6.35 mmol) and 1-phenyl-1*H*-tetrazole-5-thiol (1.69 g, 9.5 mmol) led to thioester **S8a** (4.19 g, 71%). IR (KBr film) ν 2930, 2864, 1598, 1500, 1462, 1428, 1388, 1251, 1113 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.03 (2s, 3H); 0.04 and 0.05 (2s, 3H); 0.86 and 0.87 (2s, 9H); 0.92 (d, *J* = 6.3 Hz, 3H); 1.01 (s, 13H); 1.04, 1.05 and 1.06 (3s, 17H); 1.35–1.50 (m, 2H); 1.56 and 1.58 (2d, *J* = 6.7 Hz, 3H); 1.54–1.68 (m, 5H); 1.85–2.02 (m, 1H); 2.20–2.27 (m, 1H); 3.61–3.71 (m, 2H); 3.80–3.85 (m, 1H); 4.04–4.22 (m, 3H); 4.43–4.50 (m, 1H); 7.34–7.44 (m, 6H); 7.52–7.56 (m, 5H); 7.65–7.71 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ –4.8 (q); –4.6 (q); 12.9 (d); 17.8 (s); 17.9 (s); 18.2 (q); 18.3 (q); 19.2 (s); 19.8 (q); 21.8 (q); 23.1 (q); 25.8 (q); 26.8 (q); 27.4 (d); 41.3 (t); 41.6 (t); 42.0 (d); 43.2 (t); 44.7 (t); 44.9 (t); 45.1 (t); 64.1 (t); 68.6 (d); 68.7 (d); 73.1 (d); 76.9 (d); 85.6 (d); 85.7 (d); 124.0 (d); 127.6 (d); 129.6 (d); 129.9 (d); 130.0 (d); 133.4 (s); 133.5 (s); 133.8 (s); 135.6 (d); 153.8 (s). HRMS (+ESI): *m/z* calcd. for $C_{51}H_{82}O_4N_4NaSSi_3$ (M+Na) 953.5257, found 953.5268.

5-[(2*RS*,4*S*,6*S*)-7-((2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)-tetrahydrofuran-2-yl)-6-methyl-4-(triisopropylsilyloxy)heptan-2-yl-thio]-1-*tert*-butyl-1*H*-tetrazole (S8b).

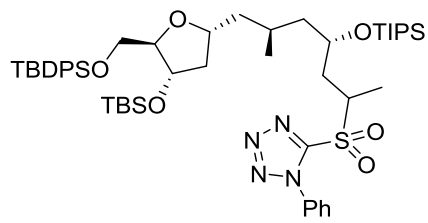


Alcohol **S7b** (490 mg, 0.63 mmol), and 1-(*tert*-butyl)-1*H*-tetrazole-5-thiol[5] (140 mg, 0.89 mmol) led to **S8b** (529 mg, 92%). IR (KBr film) ν 2931, 2864, 1463, 1390, 1253, 1112 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ −0.05 and −0.04 (2s, 3H); −0.00 and 0.00 (2s, 3H); 0.79 and 0.80 (2s, 9H); 0.91 (2d, *J* = 6.2 Hz, 3H); 1.03 (s, 10H); 1.05, 1.06 and 1.07 (3s 20H); 1.36−1.48 (m, 3H); 1.53 and 1.54 (2d, *J* = 6.6 Hz, 3H); 1.57−1.67 (m, 4H); 1.68 and 1.70 (2s, 9H); 1.84−2.02 (m, 1H); 2.19−2.26 (m, 1H); 3.72−3.80 (m, 2H); 3.82−3.86 (m, 1H); 3.87−3.96 (m, 1H); 4.07−4.26 (m, 2H); 4.30−4.36 (m, 1H); 7.32−7.42 (m, 6H); 7.67−7.72 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −5.2 (q); −4.7(q); −4.6 (q); 12.9 (d); 17.9 (s); 18.2 (q); 18.3 (q); 19.2 (s); 19.9 (q); 21.8 (q); 23.2 (q); 25.7 (q); 26.9 (q); 27.4 (d); 28.7 (q); 41.7 (t); 41.8 (t); 41.9 (d); 42.3 (d); 43.1 (t); 43.2 (t); 44.9 (t); 45.0 (t); 45.1 (t); 60.8 (s); 63.7 (t); 68.8 (d); 72.6 (d); 75.6 (d); 75.9 (d); 83.2 (d); 83.3 (d); 127.5 (d); 129.4 (d); 132.2 (s); 133.7 (s); 135.6 (d); 135.7 (d); 152.1 (s). HRMS (+ESI): *m/z* calcd. for $C_{49}H_{87}N_4O_4SSi_3$ (M+H) 911.5750, found 911.5740.

General procedure for oxidation to the sulfone:

A solution of 70% m-CPBA (2.2 eq) and thioester **S8** (1 eq.) in CH_2Cl_2 was stirred for 16 h. The reaction mixture was diluted with sat. $Na_2S_2O_3$ and sat. $NaHCO_3$ and the residue was extracted with CH_2Cl_2. The organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (95:5) yielded the corresponding sulfone **2** as a colorless oil.

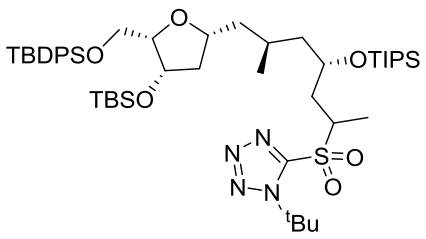
5-[(2*RS*,4*S*,6*S*)-7-((2*R*,4*S*,5*R*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)-tetrahydrofuran-2-yl)-6-methyl-4-(triisopropylsilyloxy)heptan-2-yl-sulfonyl]-1-phenyl-1*H*-tetrazole (2a).



Thioester **S8a** (4.19 g, 4.5 mmol) led to sulfone **2a** (3.85 g, 89%). IR (KBr film) ν 2930, 2865, 1498, 1463, 1338, 1252, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.03, 0.04, 0.05 and 0.06 (4s, 6H); 0.88 and 0.89 (2s, 9H); 0.89 and 0.92 (2d, *J* = 6.0 Hz, 3H); 1.06, 1.07 and 1.08 (3s, 30H); 1.38−1.70 and 1.83−1.91 (2m, 7H); 1.49 and 1.56 (2d, *J* = 6.8 Hz, 3H); 2.12−2.28 and 2.42−2.50 (2m, 2H); 3.62−3.71 (m, 2H); 3.77−3.85 (m, 1H); 4.07−4.21 and 4.24−4.31 (2m, 3H); 4.40−4.51 (m, 1H); 7.34−7.43 (m, 6H); 7.55−7.62 (m, 3H); 7.64−7.71 (m, 6H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −4.8 (q); −4.7 (q); 12.9 (d); 13.5 (q); 15.9 (q); 17.7 (s); 17.9 (s); 18.2 (q); 18.3 (q); 19.0 (s); 19.2 (q); 19.7 (q); 25.8 (q); 26.8 (q); 27.3 (d); 27.4 (d); 34.1 (t); 35.7 (t); 41.4 (t); 41.6 (t); 44.6 (t); 45.0 (t); 45.1 (t); 58.6 (d); 58.8 (d); 64.1 (t); 64.3 (t); 67.5 (d); 69.3 (d); 73.1 (d); 73.3 (d); 76.5 (d); 76.9 (d); 85.8 (d); 85.9

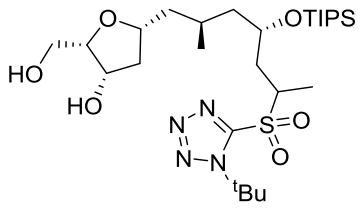
(d); 125.3 (d); 125.4 (d); 127.6 (d); 129.5 (d); 129.6 (d); 131.3 (d); 133.2 (s); 133.4 (s); 133.5 (s); 135.6 (d); 152.6 (s); 152.7 (s). HRMS (+ESI): *m/z* calcd. for $C_{51}H_{83}O_6N_4SSi_3$ (M+H) 963.5336, found 963.5351.

5-[(2*RS*,4*S*,6*S*)-7-((2*R*,4*S*,5*S*)-4-(*tert*-Butyldimethylsilyloxy)-5-(*tert*-butyldiphenylsilyloxymethyl)-tetrahydrofuran-2-yl)-6-methyl-4-(triisopropylsilyloxy)heptan-2-yl-sulfonyl]-1-*tert*-butyl-1*H*-tetrazole (2b).



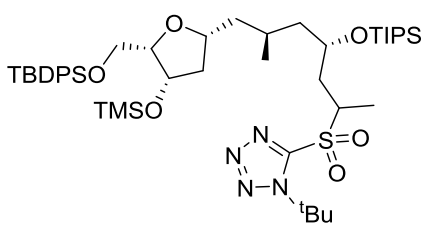
Thiotetrazole **S8b** (2.56 g, 2.80 mmol) led to sulfone **2b** (2.10 g, 80%). IR (KBr film) ν 2941, 2865, 1463, 1332, 1158, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ −0.04 (s, 3H); 0.00 and 0.01 (2s, 3H); 0.80 (2s, 9H); 0.91 (d, *J* = 6.2 Hz, 3H); 1.05 (2s, 9H); 1.06 and 1.07 (2s, 21H); 1.37–1.70 (m, 7H); 1.50 and 1.56 (2d, *J* = 6.9 Hz, 3H); 1.84 (s, 9H); 2.12–2.26 and 2.42–2.49 (2m, 2H); 3.71–3.78 (m, 2H); 3.82–3.96 (m, 2H); 4.07–4.16 and 4.24–4.43 (2m, 3H); 7.32–7.42 (m, 6H); 7.67–7.71 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ −5.2 (q); −4.6 (q); 12.9 (d); 13.9 (q); 16.3 (q); 18.0 (s); 18.2 (q); 18.3 (q); 19.1 (q); 19.2 (s); 19.6 (q); 25.7 (q); 26.9 (q); 27.4 (d); 27.5 (d); 29.6 (q); 29.7 (q); 34.2 (t); 35.9 (t); 41.7 (t); 41.9 (t); 45.0 (t); 45.2 (t); 45.3 (t); 58.8 (d); 59.3 (d); 63.5 (t); 65.3 (s); 65.4 (s); 67.6 (d); 69.5 (d); 72.5 (d); 75.6 (d); 75.9 (d); 83.2 (d); 83.3 (d); 127.5 (d); 129.5 (d); 133.7 (s); 133.8 (s); 134.0 (s); 135.6 (d); 135.7 (d); 153.2 (s); 153.3 (s). HRMS (+ESI): *m/z* calcd. for $C_{49}H_{90}N_5O_6SSi_3$ (M+NH_4) 960.5914, found 960.5907.

(2*S*,3*S*,5*R*)-5-[(2*S*,4*S*,6*RS*)-6-(1-(*tert*-Butyl)-1*H*-tetrazol-5-yl-sulfonyl)-2-methyl-4-(triisopropyl-silyloxy)heptyl]-2-(hydroxymethyl)tetrahydrofuran-3-ol (S9).



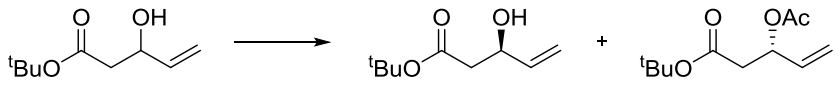
PPTS (666 mg, 2.6 mmol) was added to a solution of sulfone **2b** (250 mg, 0.26 mmol) in MeOH (10 mL) and the reaction was stirred at 65 ºC for 5 h. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography with hexane-EtOAc (80:20 to 50:50) to yield **S9** (114 mg, 95%) as a colorless oil. IR (KBr film) ν 3419 (br), 2943, 2867, 1464, 1377, 1332, 1159 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.92 and 0.94 (2d, *J* = 6.3 Hz, 3H); 1.08 (bs, 21H); 1.41–1.78 and 1.94–2.03 (m, 6H); 1.55 and 1.61 (2d, *J* = 6.8 Hz, 3H); 1.86 (2s, 9H); 2.20–2.27 and 2.36–2.50 (2m, 3H); 3.73–3.80 (m, 1H); 3.86–4.01 (m, 3H); 4.16–4.30 (m, 1H); 4.33–4.41 (m, 1H); 4.43–4.51 (m, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 12.7 (d); 12.9 (d); 14.4 (q); 16.0 (q); 18.2 (q); 19.8 (q); 20.1 (q); 26.9 (d); 27.4 (d); 29.7 (q); 33.7 (t); 36.4 (t); 42.7 (t); 42.9 (t); 43.1 (t); 44.0 (t); 44.5 (t); 45.0 (t); 59.2 (d); 59.3 (d); 61.7 (t); 61.8 (t); 65.5 (s); 65.6 (s); 68.1 (d); 69.6 (d); 73.9 (d); 74.1 (d); 75.2 (d); 75.3 (d); 80.6 (d); 80.7 (d); 153.1 (s); 153.2 (s). HRMS (+ESI): *m/z* calcd. for $C_{27}H_{55}N_4O_6SSi$ (M+H) 591.3606, found 591.3608.

1-(*tert*-Butyl)-5-[(2*RS*,4*S*,6*S*)-7-((2*R*,4*S*,5*S*)-5-(*tert*-butyldiphenylsilyloxymethyl)-4-(trimethylsilyl-oxy)tetrahydrofuran-2-yl)-6-methyl-4-(triisopropylsilyloxy)heptan-2-yl-sulfonyl]-1*H*-tetrazole (2c).



TBDPSCl (71 µL, 0.27 mmol) was added to a solution of diol **S9** (150 mg, 0.25 mmol) and imidazole (68 mg, 1 mmol) in CH_2Cl_2 (10 mL) and the reaction was stirred for 1 h. After this time, TMSCl (48 µL, 0.37 mmol) was added and the reaction was stirred for further 15 min. Finally, the resulting mixture was washed with water, dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (97:3) yielded **2c** (165 mg, 73%) as a colorless oil. IR (KBr film) ν 2943, 2866, 1463, 1333, 1113 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 0.05 and 0.06 (2s, 9H); 0.91 (d, *J* = 5.9 Hz, 3H); 1.04 (s, 9H); 1.07 (2bs, 21H); 1.38–1.68 (m, 7H); 1.50 and 1.56 (2d, *J* = 6.8 Hz, 3H); 1.84 (s, 9H); 1.89–1.94, 2.13–2.30 and 2.41–2.49 (3m, 2H); 3.70–3.77 (m, 2H); 3.83–3.93 (m, 2H); 4.07–4.16 and 4.23–4.31 (2m, 1H); 4.32–4.44 (m, 2H); 7.32–7.43 (m, 6H); 7.66–7.74 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 0.0 (q); 12.9 (d); 14.0 (q); 16.2 (q); 18.2 (q); 18.3 (q); 19.2 (s); 19.2 (q); 19.7 (q); 26.9 (q); 27.4 (d); 27.5 (d); 29.6 (q); 29.7 (q); 34.2 (t); 35.9 (t); 41.8 (t); 41.9 (t); 44.7 (t); 45.0 (t); 45.1 (t); 58.9 (d); 59.2 (d); 62.8 (t); 65.3 (s); 65.4 (s); 67.7 (d); 69.4 (d); 72.1 (d); 75.5 (d); 75.9 (d); 82.8 (d); 83.0 (d); 127.5 (d); 129.5 (d); 133.7 (s); 133.8 (s); 134.0 (s); 135.6 (d); 135.7 (d); 153.2 (s); 153.3 (s). HRMS (+ESI): *m/z* calcd. For $C_{46}H_{84}N_5O_6Ssi_3$ (M+NH_4) 918.5445, found 918.5443.

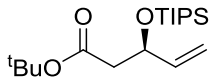
Kinetic resolution of **12**: [6]



12 ***R*-12** ***S*-Ac-12**

Lipase PS-30 (4.32 g) was added to a solution of **(±)-12** (2.15 g, 12.5 mmol) in vinyl acetate (10 mL) and pentane (25 mL), and the suspension was stirred for 48 h at 37 ºC. The residue was filtered through Celite® 545, and the solvent was removed under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (90:10) yielded ***R*-12** (1.05 g, 49%) and ***S*-Ac-12** (1.28 g, 48%) as colorless oils. ***R*-12**: ^1H NMR (400 MHz, $CDCl_3$) δ 1.46 (s, 9H); 2.43 (dd, *J* = 16.2, 8.3 Hz, 1H); 2.51 (dd, *J* = 16.2, 4.0 Hz, 1H); 3.11 (bs, OH); 4.45–4.52 (m, 1H); 5.14 (dt, *J* = 10.5, 1.4 Hz, 1H); 5.30 (dt, *J* = 17.2, 1.4 Hz, 1H); 5.87 (ddd, *J* = 17.2, 10.5, 5.5 Hz, 1H). ***S*-Ac-12**: ^1H NMR (400 MHz, $CDCl_3$) δ 1.44 (s, 9H); 2.05 (s, 3H); 2.52 (dd, *J* = 15.3, 5.8 Hz, 1H); 2.60 (dd, *J* = 15.3, 8.0 Hz, 1H); 5.20 (dd, *J* = 10.5, 1.0 Hz, 1H); 5.30 (dd, *J* = 17.2, 1.0 Hz, 1H); 5.57–5.63 (m, 1H); 5.83 (ddd, *J* = 17.2, 10.5, 6.2 Hz, 1H).

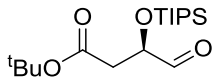
***tert*-Butyl (*R*)-3-(triisopropylsilyloxy)pent-4-enoate (S10).**



Triisopropylsilyl trifluoromethanesulfonate (1.98 mL, 7.34 mmol) was added to a solution of aldol **(*R*)-12** (1.05 g, 6.1 mmol), imidazole (830 mg, 12.2 mmol) and

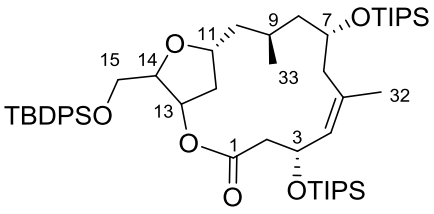
DMAP (20 mg) in THF (60 mL), and the reaction mixture was stirred for 16 h at reflux temperature. The solvent was removed under reduced pressure and the residue was dissolved in water and extracted with CH_2Cl_2. The organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification by silica gel column chromatography with hexane-EtOAc (97:3) yielded **S10** (1.78 g, 89%) as a colorless oil. $[\alpha]_D$ = −3.8 (c 1.0, $CHCl_3$). IR (KBr film) ν 2944, 2867, 1732, 1464, 1367, 1256, 1161 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.04–1.07 (m, 21H); 1.43 (s, 9H); 2.37 (dd, *J* = 14.4, 7.5 Hz, 1H); 2.56 (dd, *J* = 14.4, 5.8 Hz, 1H); 4.59–4.65 (m, 1H); 5.06 (ddd, *J* = 10.4, 1.7, 1.1 Hz, 1H); 5.20 (ddd, *J* = 17.2, 1.7, 1.1 Hz, 1H); 5.87 (ddd, *J* = 17.2, 10.4, 6.7 Hz, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 12.3 (d); 18.0 (q); 18.1 (q); 28.1 (q); 45.2 (t); 71.3 (d); 80.4 (s); 114.5 (t); 140.6 (d); 170.1 (s). HRMS (+ESI): *m/z* calcd. for $C_{18}H_{37}O_3Si$ (M+H) 329.2507, found 329.2506.

***tert*-Butyl (*R*)-4-oxo-3-(triisopropylsilyloxy)butanoate (3).**



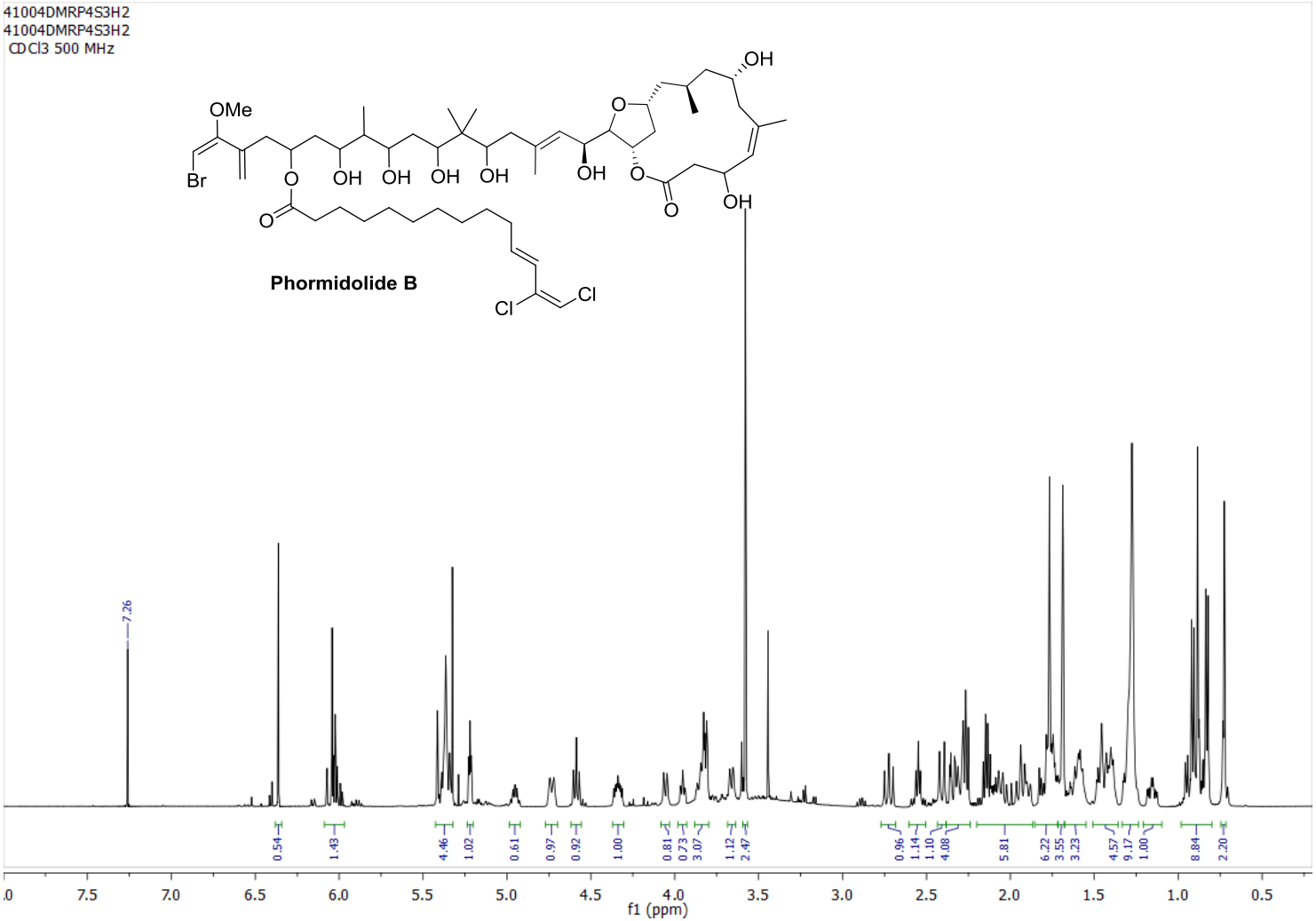
Ozone gas was bubbled into a solution of olefin **S10** (1.45 g, 4.41 mmol) in a 4:1 mixture of CH_2Cl_2-MeOH (100 mL) at −78ºC until the blue color persisted. Argon was passed through the solution for 10 min at −78ºC to remove any excess ozone. Then, PPh_3 (1.5 g, 5.73 mmol) was added and the solution was stirred at r.t. for 16 h. The reation mixture was concentrated under reduced pressure and filtered through silica to yield **3** (1.35 g, 93%) as a colorless oil. $[\alpha]_D$ = +11.5 (c 1.0, $CHCl_3$). IR (KBr film) ν 2944, 2868, 1736, 1464, 1367, 1256, 1157 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 1.04–1.07 (m, 21H); 1.44 (s, 9H); 2.66 (ddd, *J* = 15.7, 5.8, 0.8 Hz, 1H); 2.78 (dd, *J* = 15.7, 4.0 Hz, 1H); 4.32 (ddd, *J* = 5.8, 4.0, 0.8 Hz, 1H); 9.80 (t, *J* = 0.8, 1H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 12.1 (d); 17.8 (q); 28.0 (q); 41.22 (t); 74.2 (d); 81.4 (s); 169.0 (s); 204.2 (d). HRMS (+ESI): *m/z* calcd. for $C_{17}H_{35}O_4Si$ (M+H) 331.2299, found 331.2297.

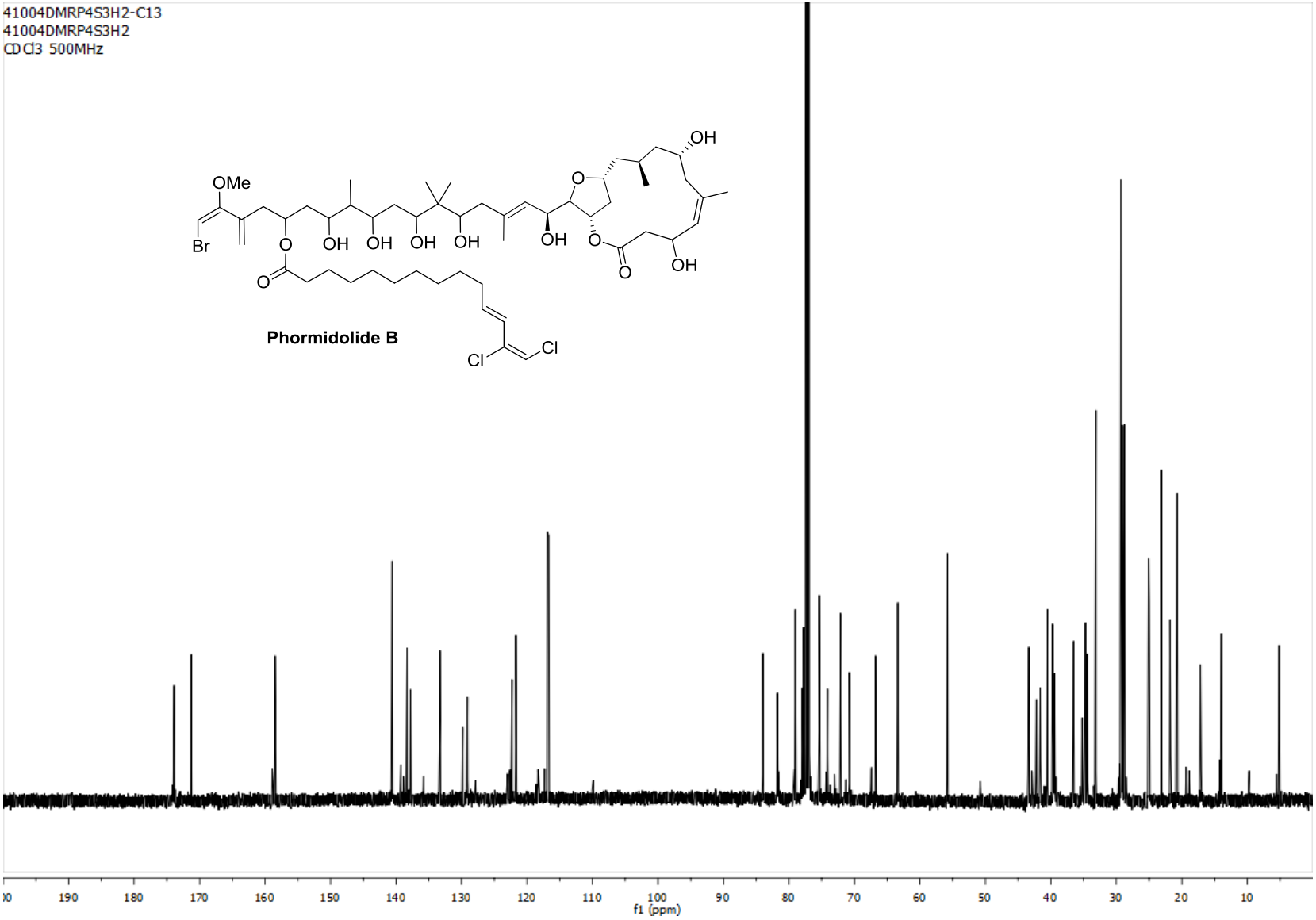
6. NMR data table of macrocycles 1a-c. Spectra recorded in $CDCl_3$

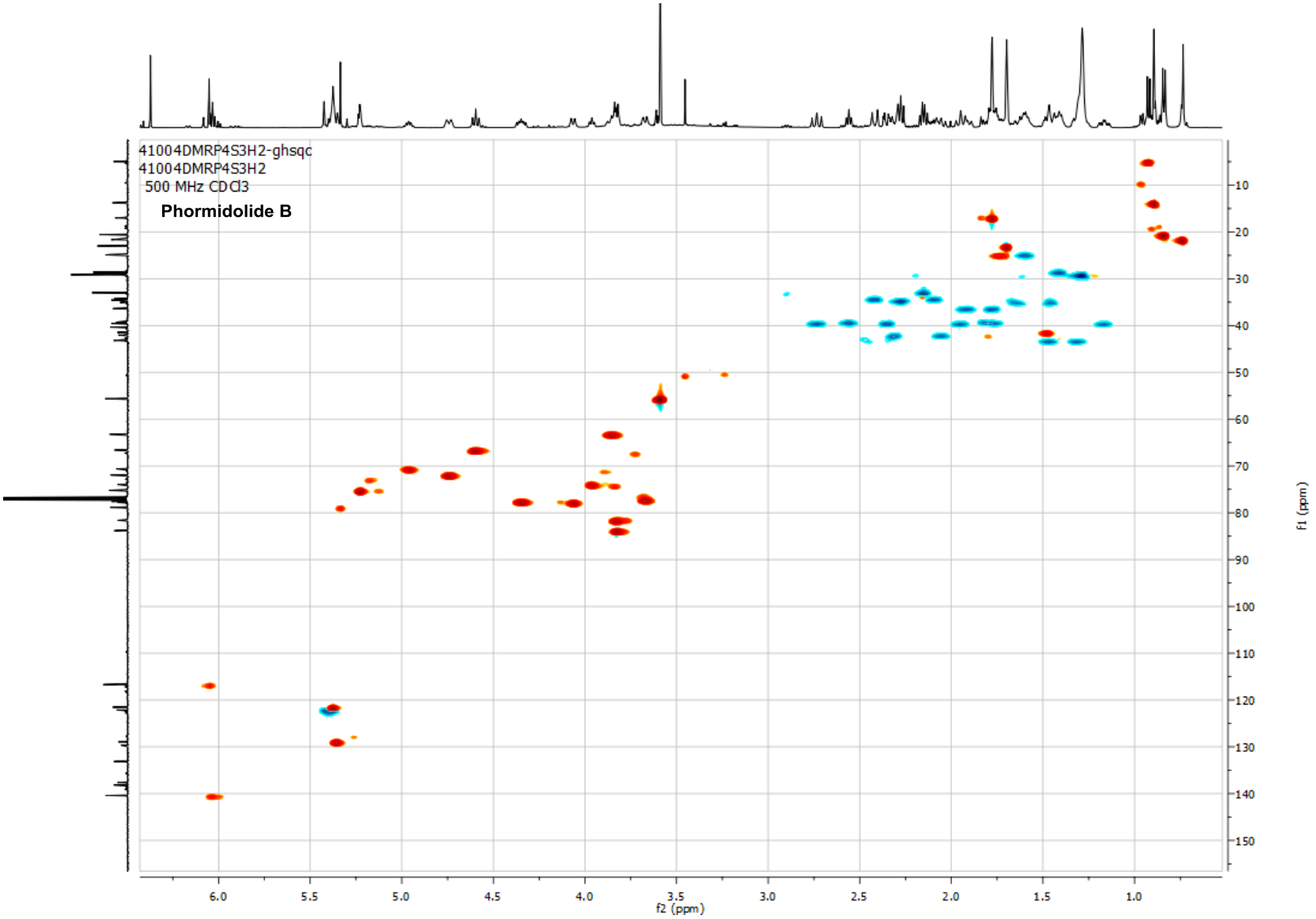


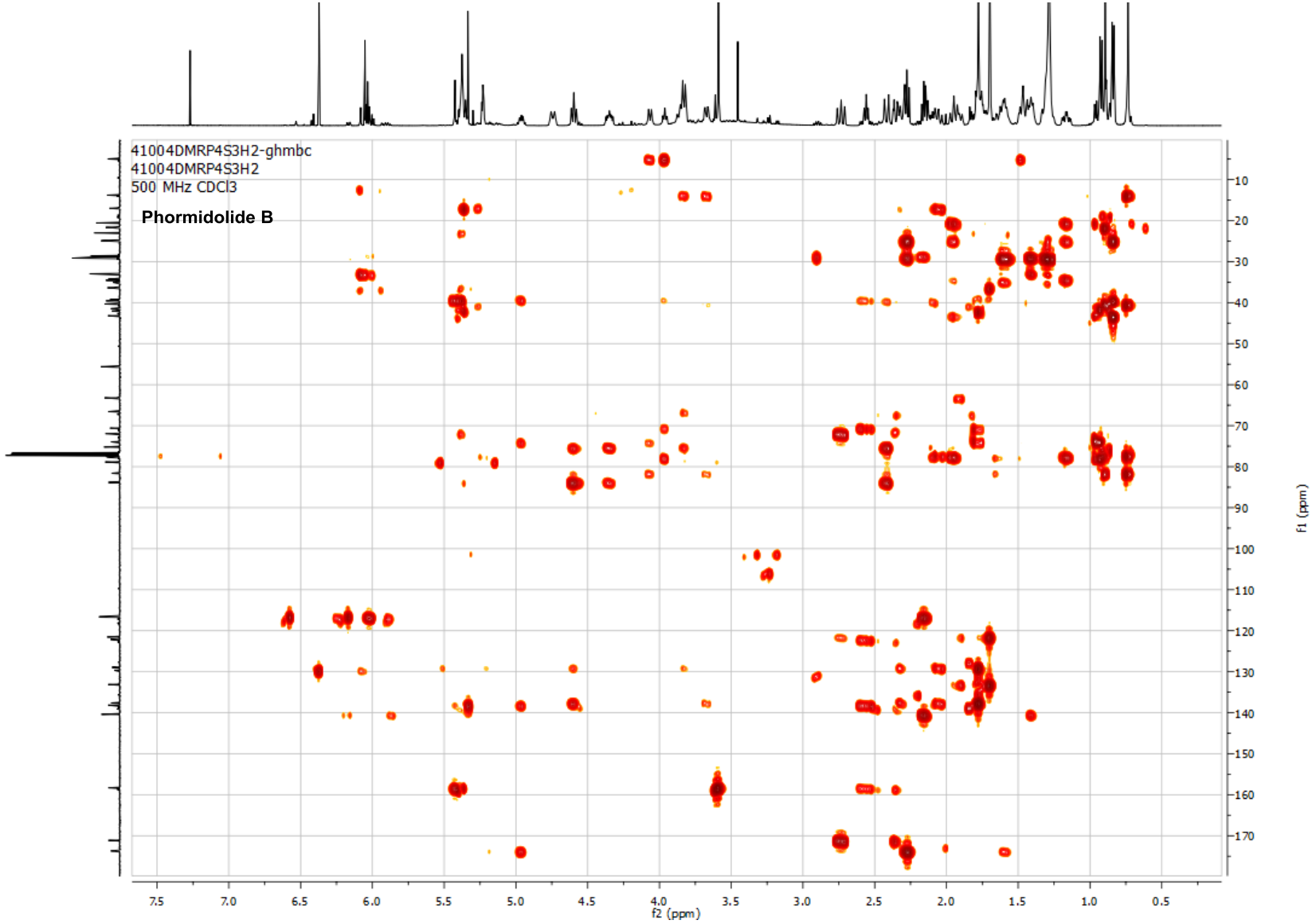
	1a		1b		1c	
	δ_H, mult, J (Hz)	**δ_C, mult**	**δ_H, mult, J (Hz)**	**δ_C, mult**	**δ_H, mult, J (Hz)**	**δ_C, mult**
1	-	170.9, s	-	170.6, s	-	170.8, s
2	2.65, d, 5.3	46.6, t	2.42, dd, 15.1, 8.4 2.30, dd, 15.1, 4.0	44.3, t	2.62, dd, 15.1, 5.3 2.47 dd, 15.1, 6.2	46.1, t
3	4.97, m	67.8, d	4.86, td, 8.4, 4.0	66.8, d	4.75 dt, 7.8, 5.8	67.0, d
4	5.41, d, 7.7	131.6, d	5.21, d, 8.4	131.3, d	5.33, m	131.5, d
5	-	134.8, s	-	133.4, s	-	135.0, s
6	2.48, dd, 14.0, 6.9 2.29 dd, 14.0, 4.4	40.0, t	2.36, dd, 13.6, 6.7 2.03, dd, 13.6, 6.0	40.2, t	2.42, dd, 14.3, 5.8 2.31 dd, 14.3, 5.0	40.3, t
7	4.11, m	71.0, d	4.04, m	70.1, d	3.95, m	71.1, d
8	1.69, dt, 13.5, 6.6 1.42, m	47.2, t	1.34, m	45.9, t	1.60, m 1.33, m	47.1, t
9	1.56, m	27.2, d	1.63, m	28.8, d	1.34 m	27.0, d
10	2.05, m 1.42, m	42.0, t	1.57, m 1.44, m	43.3, t	1.66, m 1.38, m	43.6, t
11	4.46, m	78.5, d	3.78, m	77.5, d	4.22, m	77.0, d
12	2.22, ddd, 13.8, 7.8, 5.8 2.01, d, 13.8	34.3, t	2.48, dt, 13.7, 7.0 1.36, m	40.8, t	2.17, m 1.99, dd, 14.2, 2.1	35.1 t
13	5.27, d, 5.6	77.7, d	5.18, m	74.3, d	5.33, m	74.6, d
14	4.10, m	84.4, d	3.84, m	81.4, d	4.04, td, 6.6, 3.4	82.2, d
15	3.66, dd, 10.9, 4.0 3.80, dd, 10.9, 3.3	64.9 t	3.86, m	62.8 t	3.82, m	62.3 t
32	1.78, s	24.6, q	1.72, d, 1.2	25.5, q	1.74, d, 1.4	25.3, q
33	0.94, d, 6.3	20.6, q	1.00, d, 6.6	20.5, q	0.88 d, 5.2	20.5, q

7. NMR Spectra of compounds

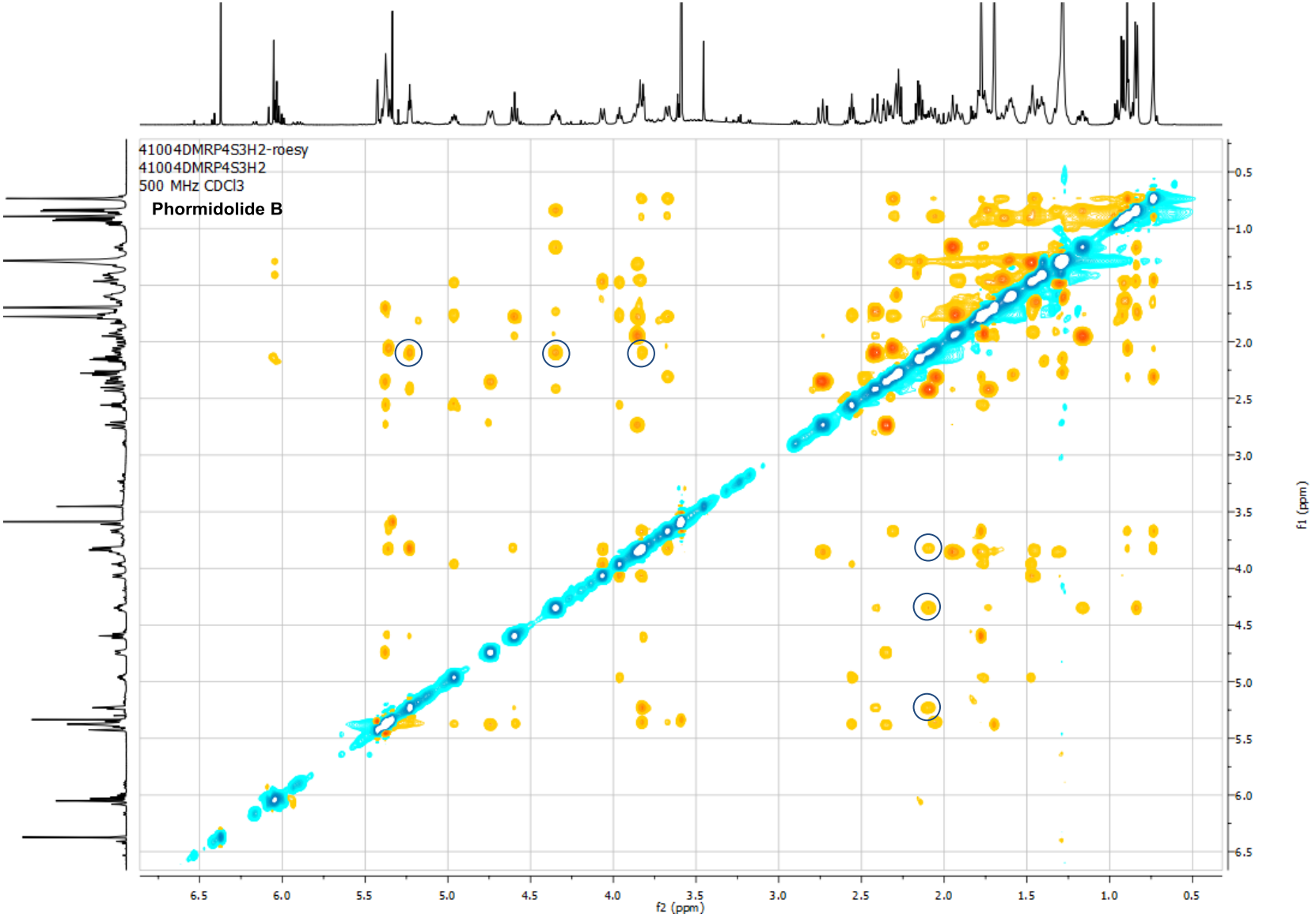


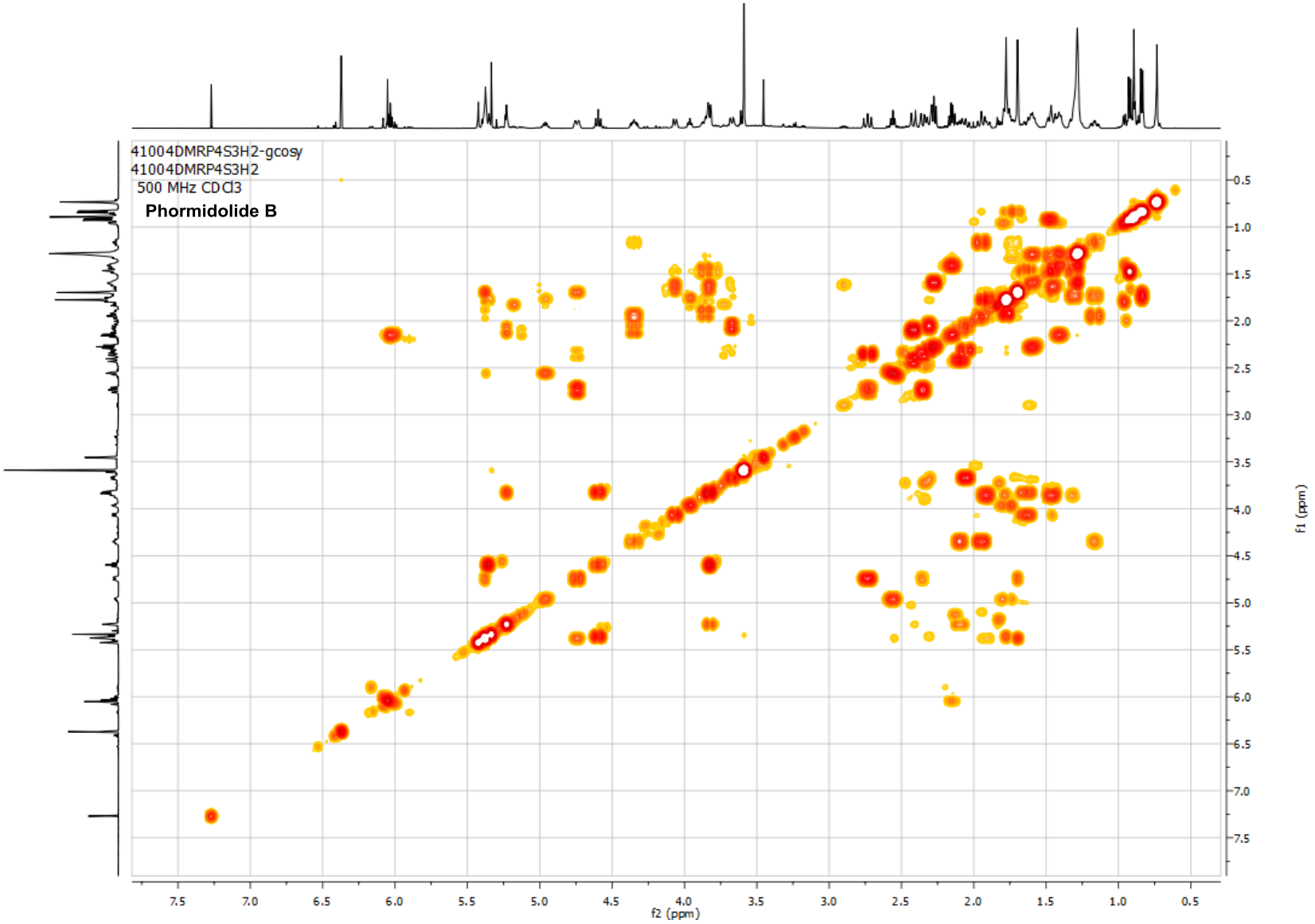

41004DMRP4S3H2-C13
41004DMRP4S3H2
CDCl3 500MHz
OMe
Br
O
OH
OH
OH
OH
OH
O
O
O
OH
OH
O
Cl
Cl
Phormidolide B
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

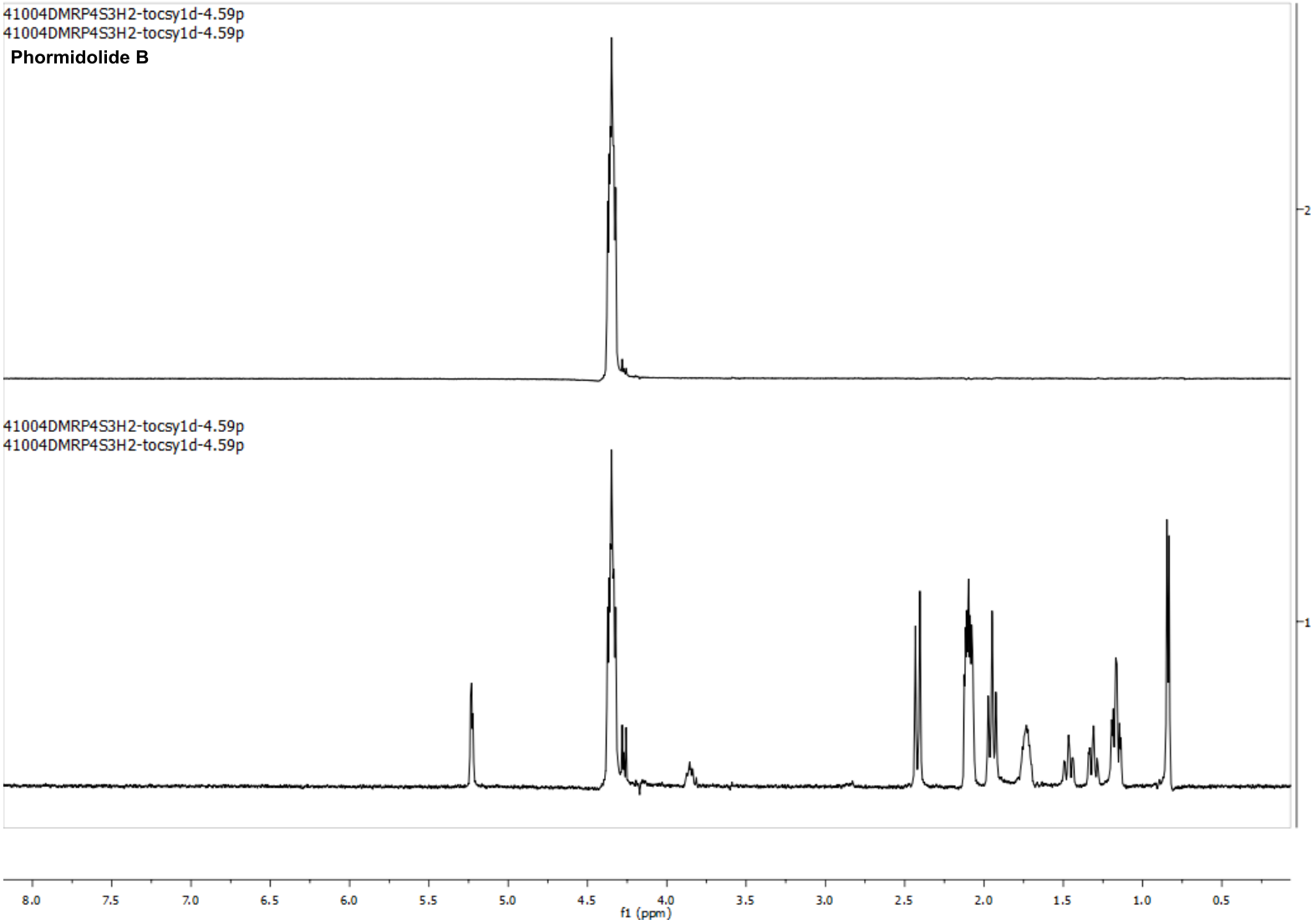

41004DMRP4S3H2-ghsqc
41004DMRP4S3H2
500 MHz CDCl3
Phormidolide B
f2 (ppm)
f1 (ppm)

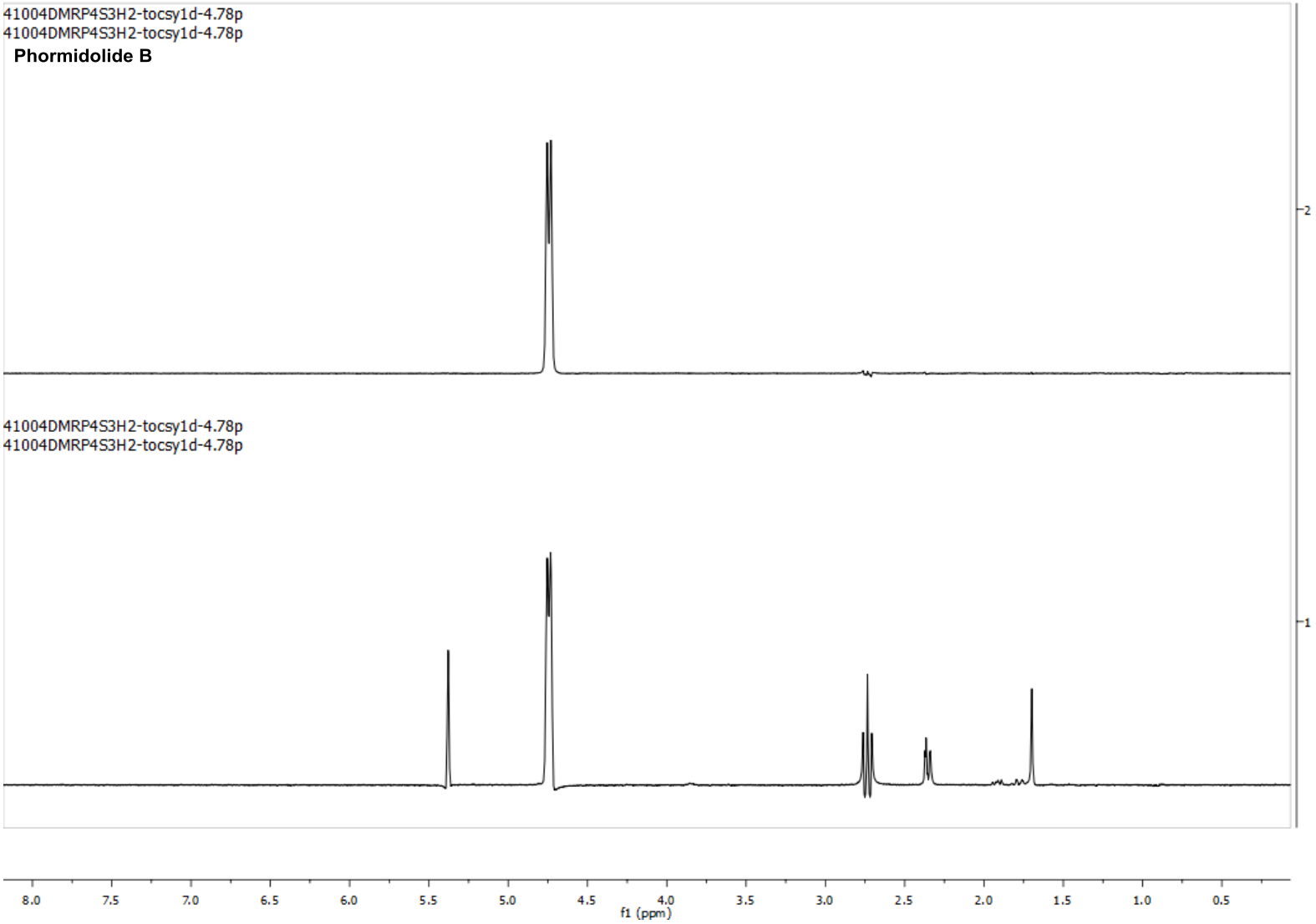

41004DMRP4S3H2-ghmbc
41004DMRP4S3H2
500 MHz CDCl3
Phormidolide B
f2 (ppm)
f1 (ppm)

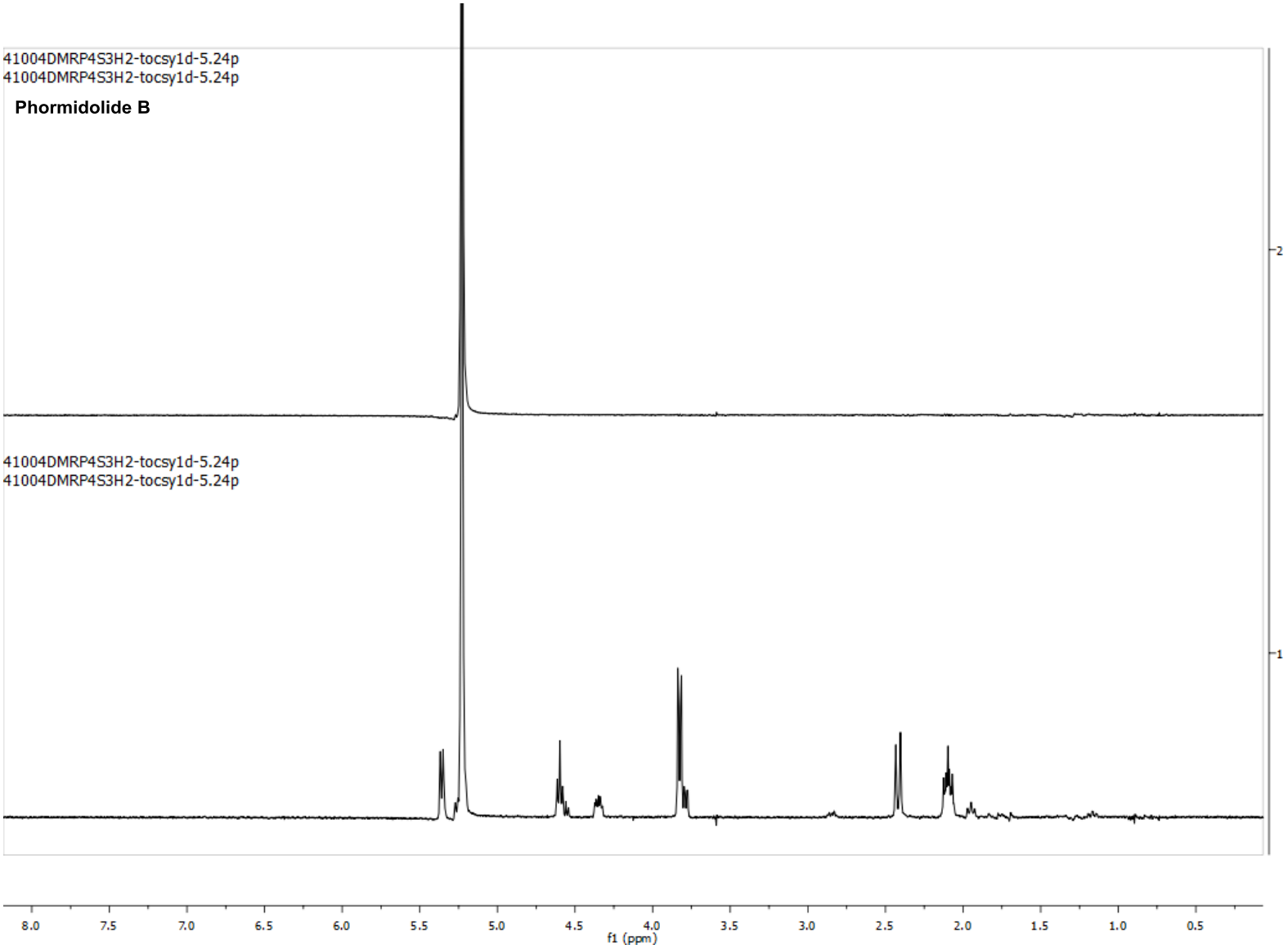
⬭ Signals used to assign relative stereochemistry of the THF ring

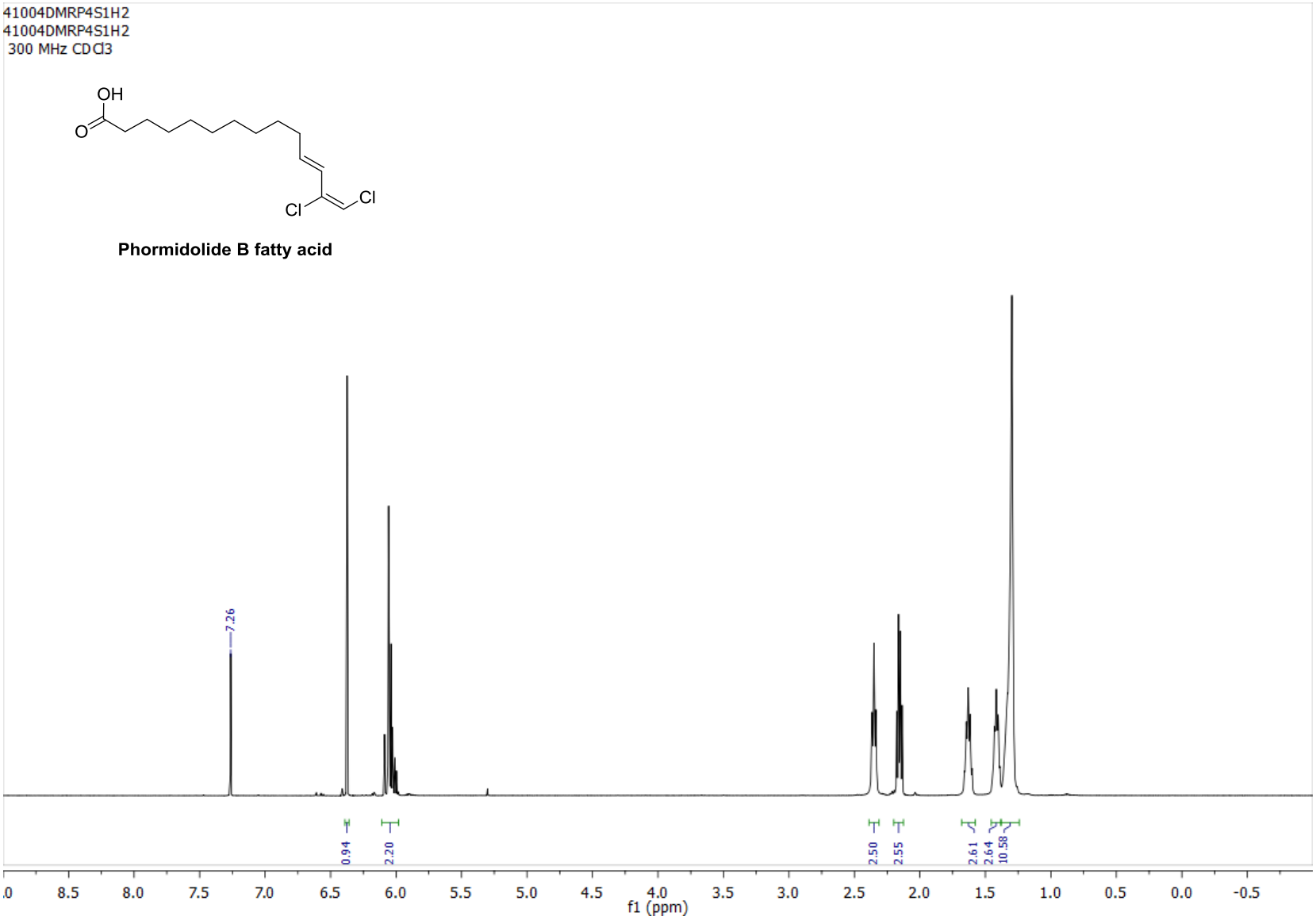



41004DMRP4S3H2-gcosy
41004DMRP4S3H2
500 MHz CDCl3
Phormidolide B
f2 (ppm)
f1 (ppm)

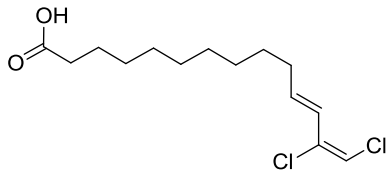

41004DMRP4S3H2-tocsy1d-4.59p
41004DMRP4S3H2-tocsy1d-4.59p
Phormidolide B
41004DMRP4S3H2-tocsy1d-4.59p
41004DMRP4S3H2-tocsy1d-4.59p
2
1
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


41004DMRP4S3H2-tocsy1d-4.78p
41004DMRP4S3H2-tocsy1d-4.78p
Phormidolide B
41004DMRP4S3H2-tocsy1d-4.78p
41004DMRP4S3H2-tocsy1d-4.78p
2
1
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
f1 (ppm)

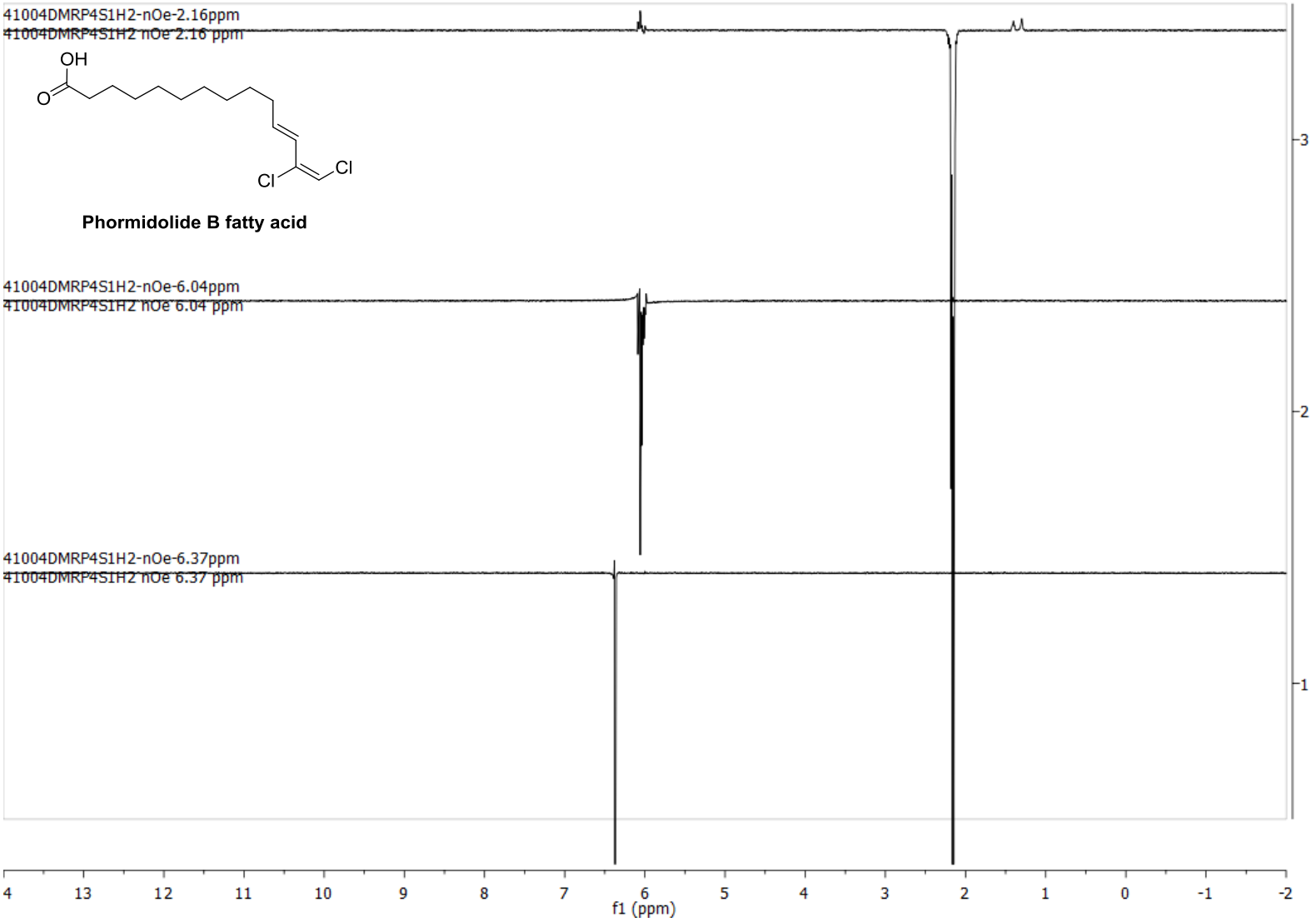

41004DMRP4S3H2-tocsy1d-5.24p
41004DMRP4S3H2-tocsy1d-5.24p
Phormidolide B
41004DMRP4S3H2-tocsy1d-5.24p
41004DMRP4S3H2-tocsy1d-5.24p
2
1
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

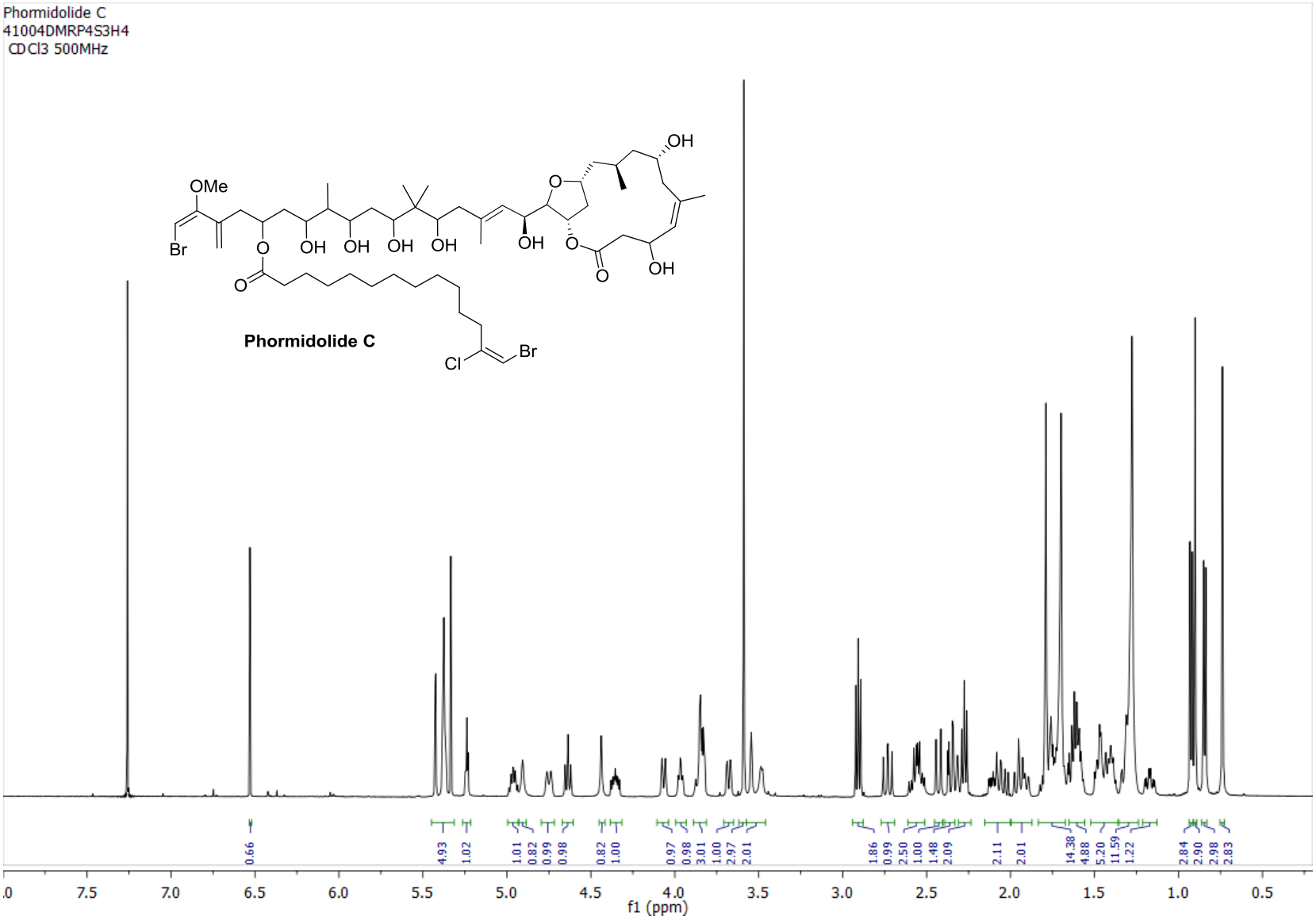

41004DMRP4S1H2
41004DMRP4S1H2
300 MHz CDCl3
OH
O
Cl
Cl
Phormidolide B fatty acid
7.26
0.94
2.20
2.50
2.55
2.61
2.64
10.58
f1 (ppm)

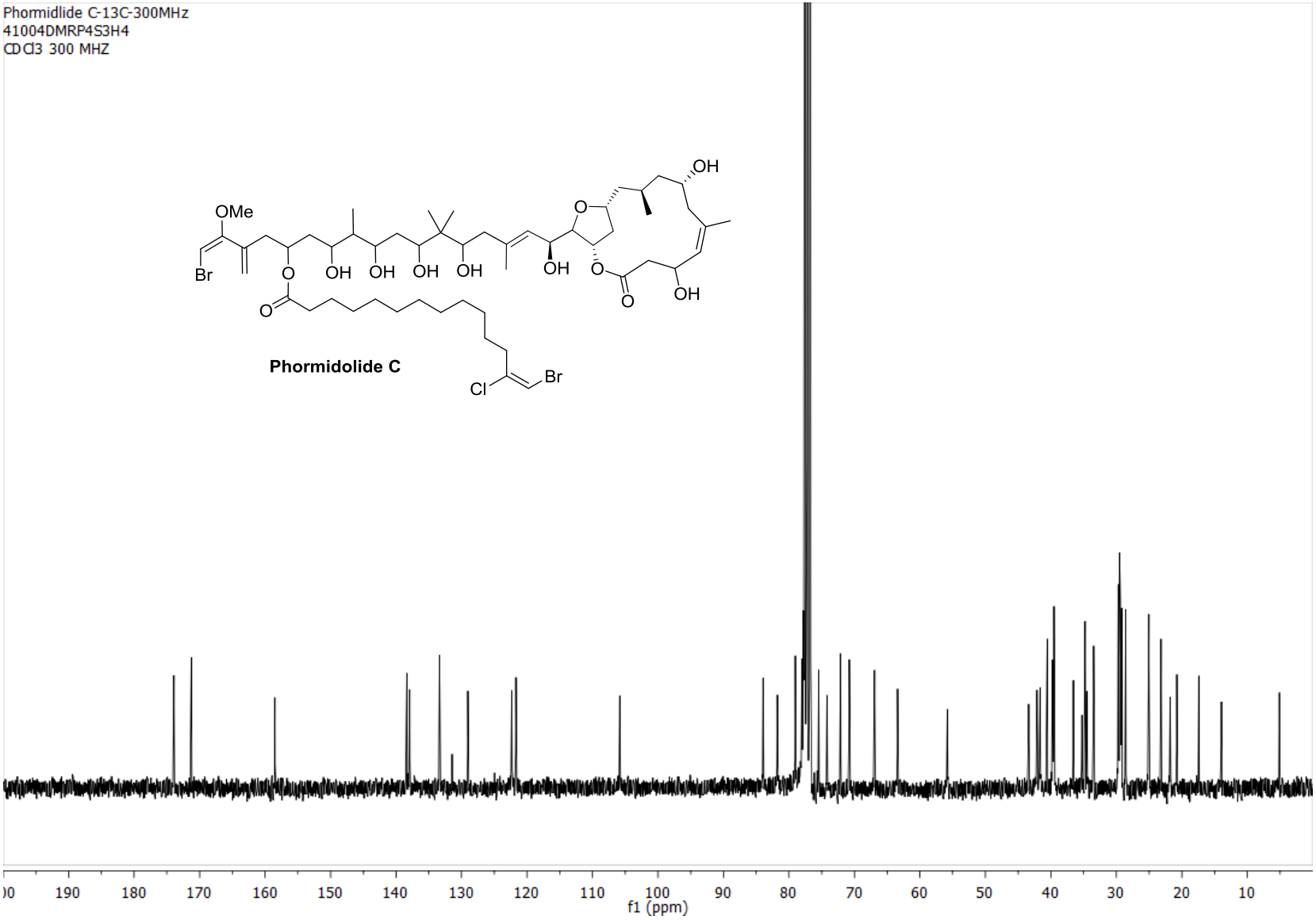
41004DMRP4S1H2-13C-300MHz
41004DMRP4S1H2
300 MHz CDCl3

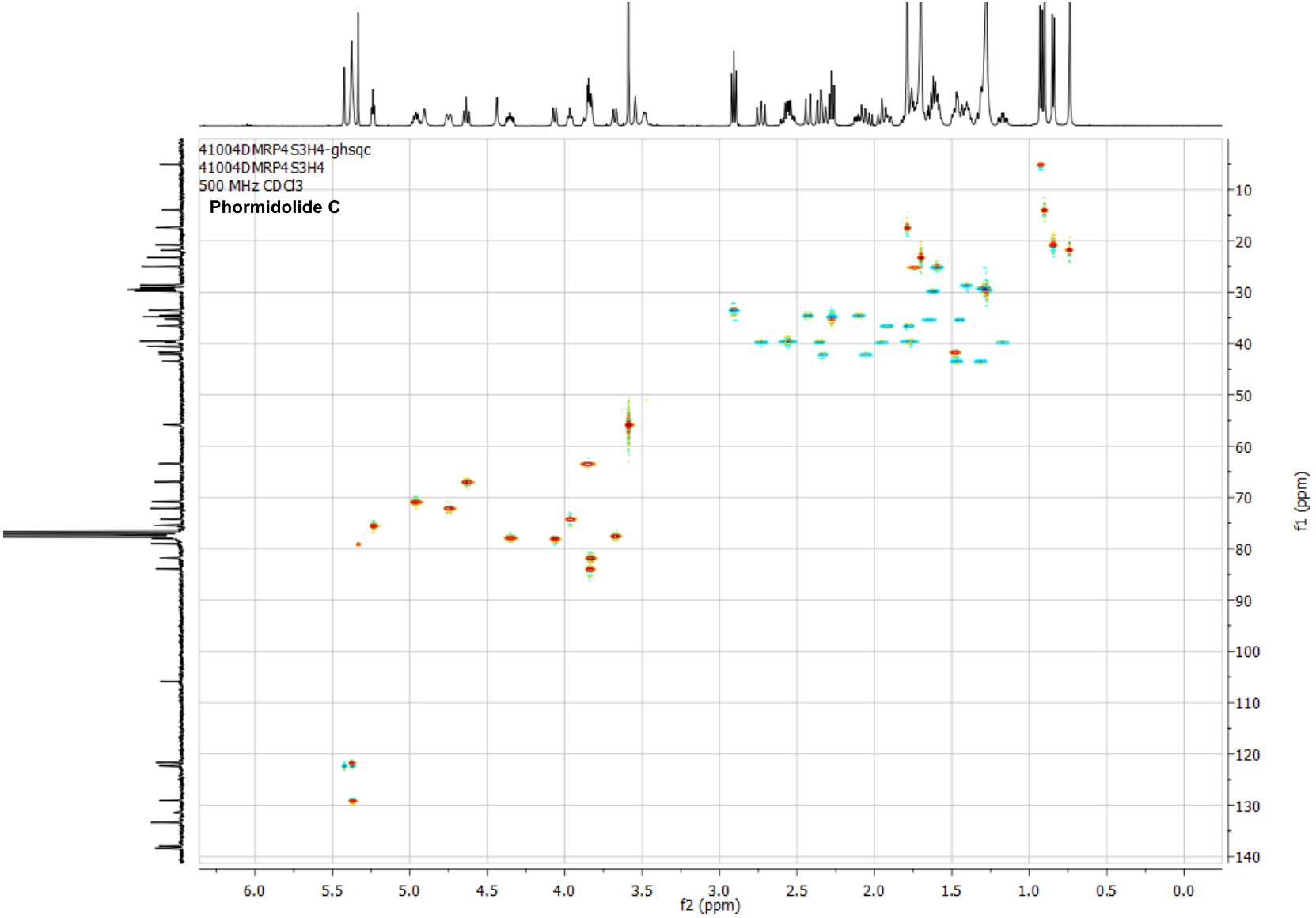


Phormidolide B fatty acid


41004DMRP4S1H2-nOe-2.16ppm
41004DMRP4S1H2 nOe 2.16 ppm
OH
O
Cl
Cl
Phormidolide B fatty acid
41004DMRP4S1H2-nOe-6.04ppm
41004DMRP4S1H2 nOe 6.04 ppm
41004DMRP4S1H2-nOe-6.37ppm
41004DMRP4S1H2 nOe 6.37 ppm
3
2
1
4
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)


Phormidolide C
41004DMRP4S3H4
CDCl3 500MHz
OMe
Br
O
OH
OH
OH
OH
OH
O
O
O
OH
OH
Cl
Br
Phormidolide C
0.66
4.93
1.02
1.01
0.82
0.99
0.98
0.82
1.00
0.97
0.98
3.01
1.00
2.97
2.01
1.86
0.99
2.50
1.00
1.48
2.09
2.11
2.01
14.38
4.88
5.20
11.59
1.22
2.84
2.90
2.98
2.83
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
f1 (ppm)


Phormidlide C-13C-300MHz
41004DMRP4S3H4
CDCl3 300 MHZ
OMe
Br
O
OH
OH
OH
OH
OH
O
O
O
OH
OH
O
Cl
Br
Phormidolide C
f1 (ppm)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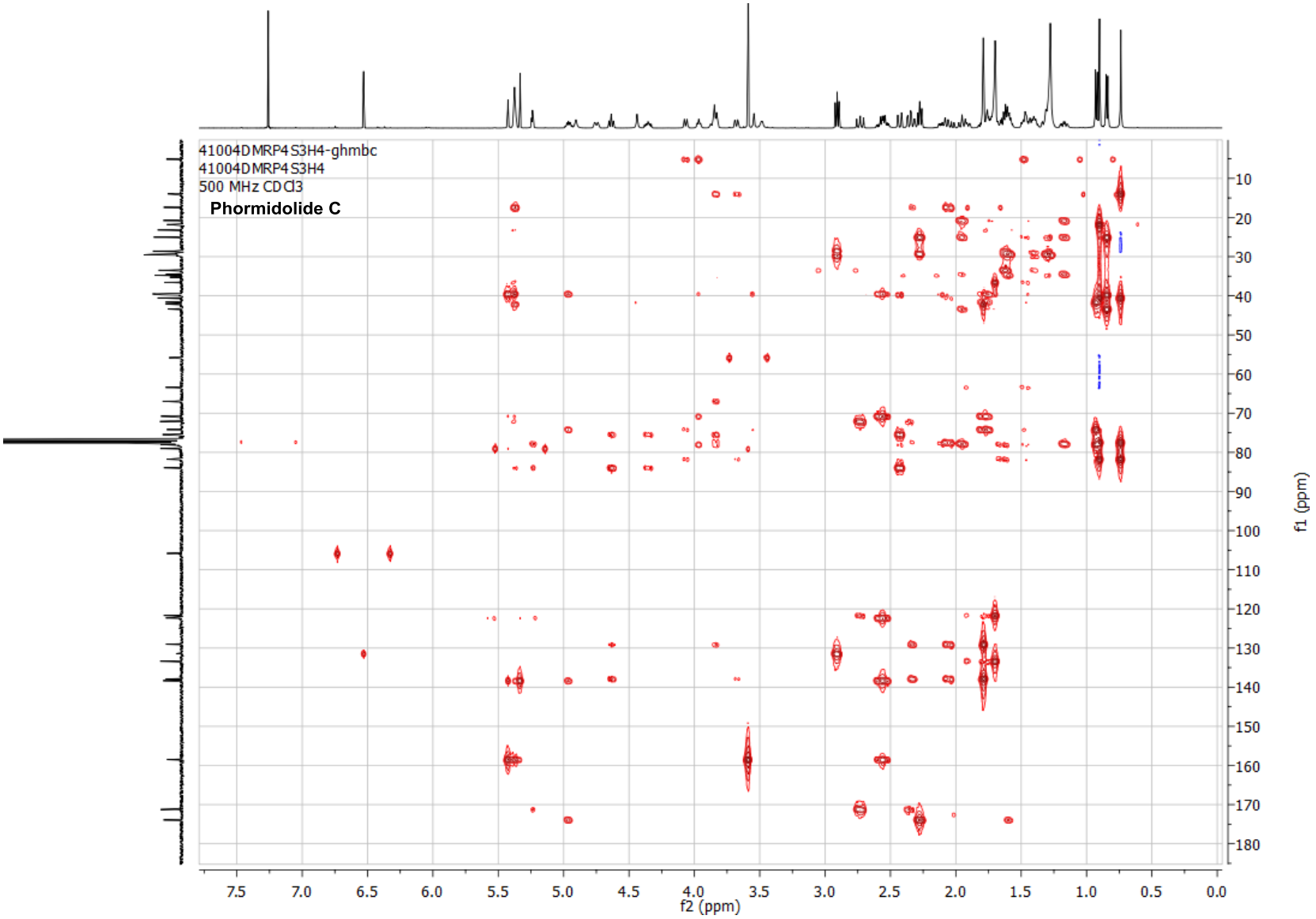
41004DMRP4S3H4
500 MHz CDCl3
Phormidolide C
f2 (ppm)
f1 (ppm)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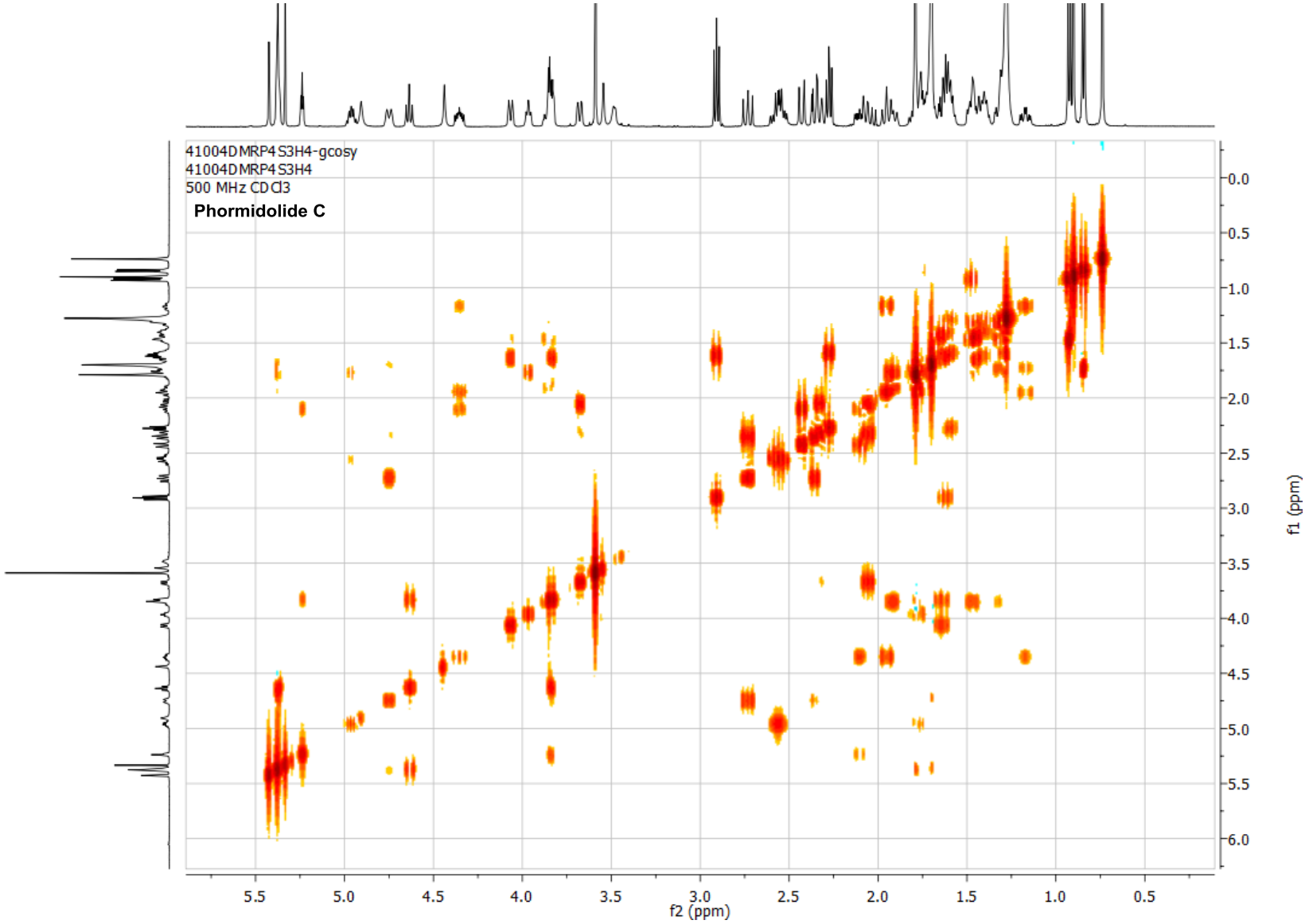
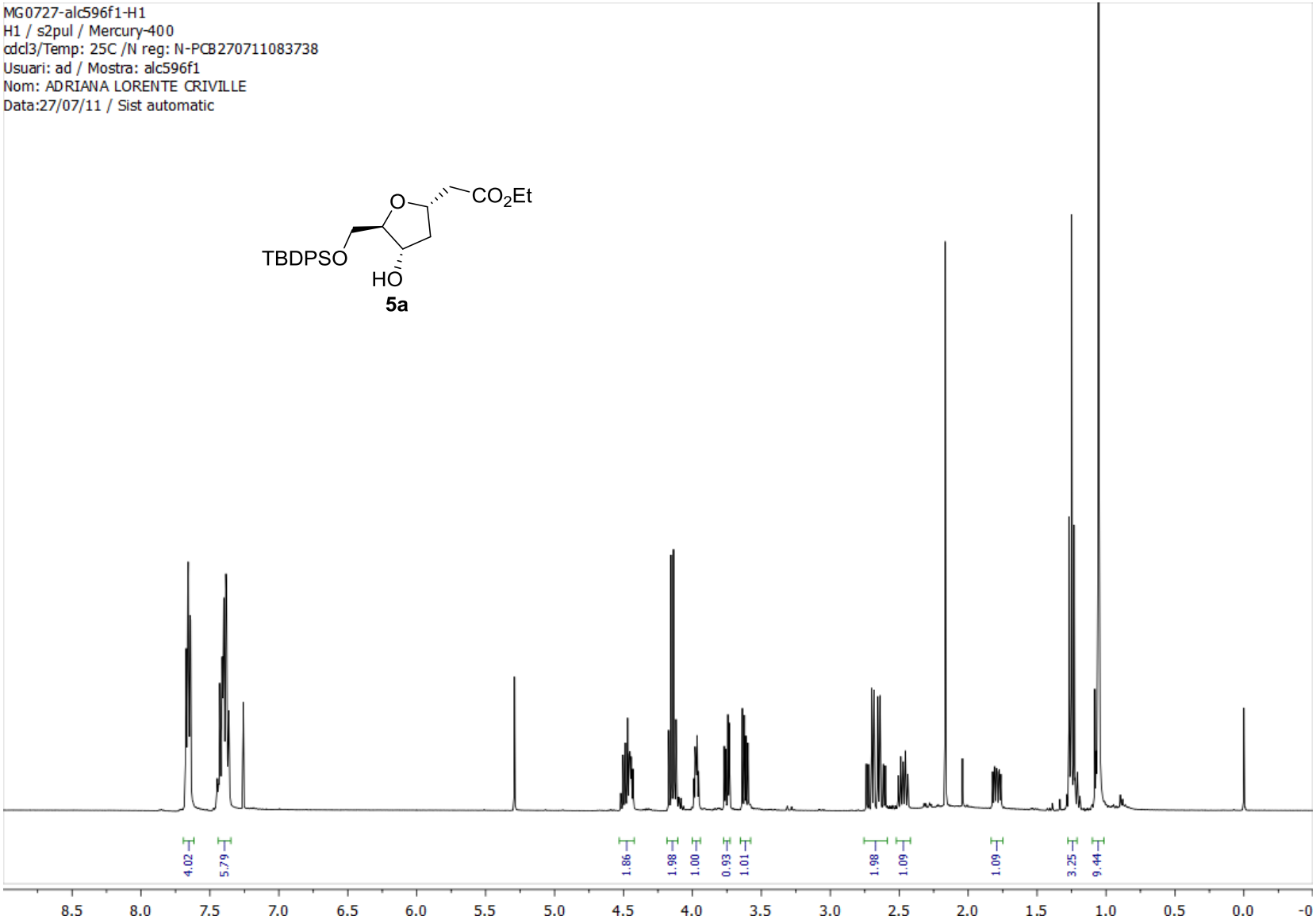
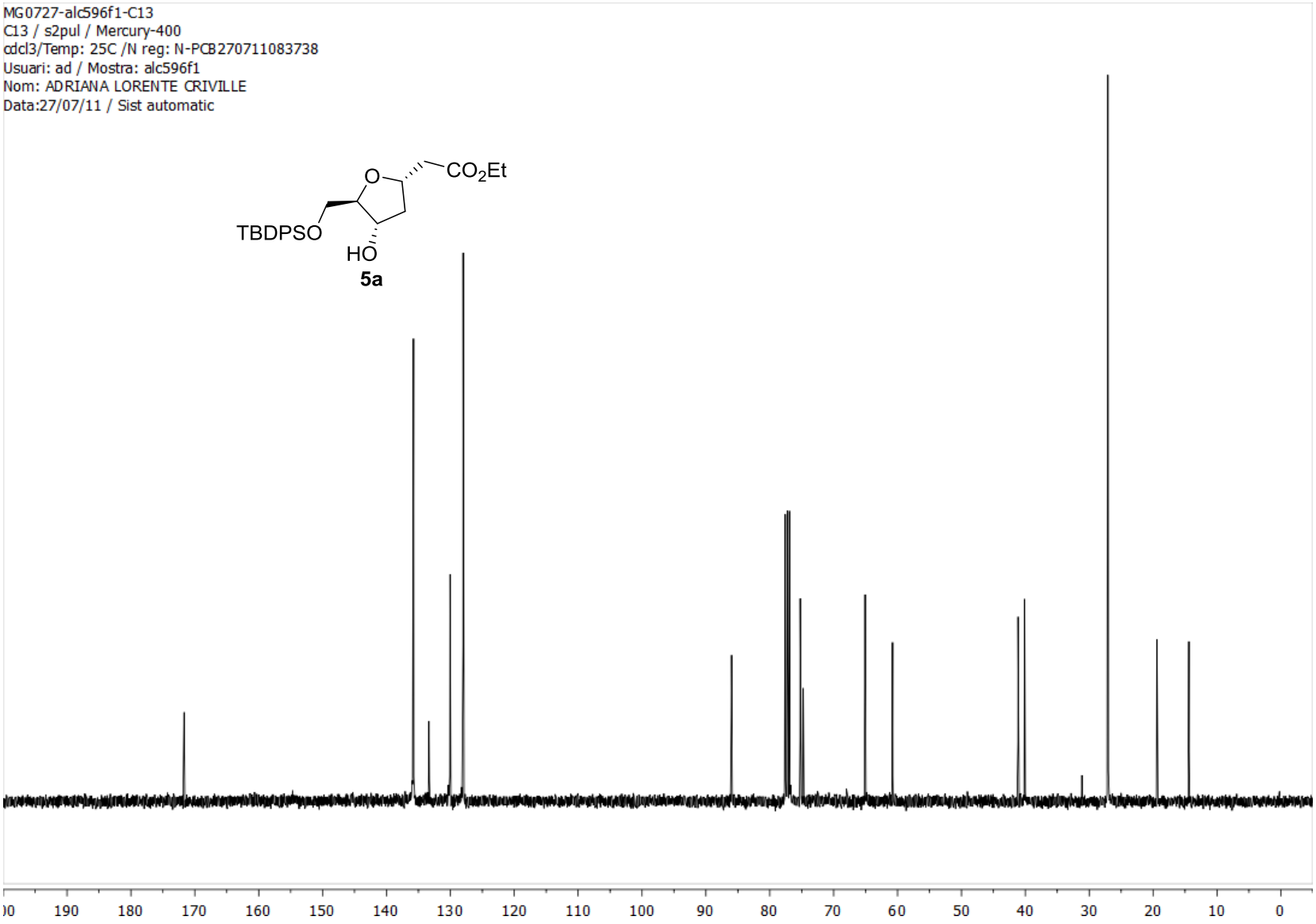
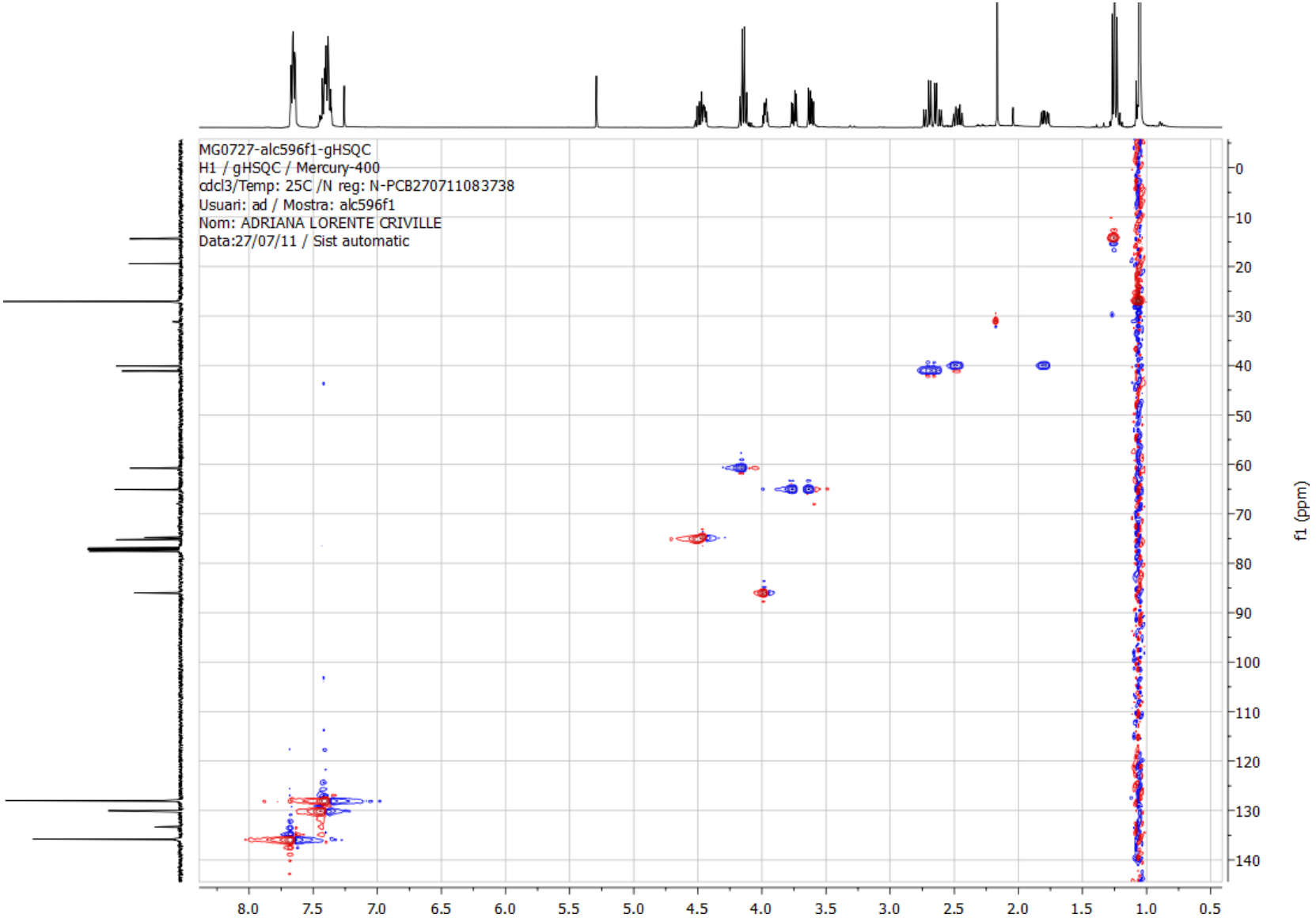
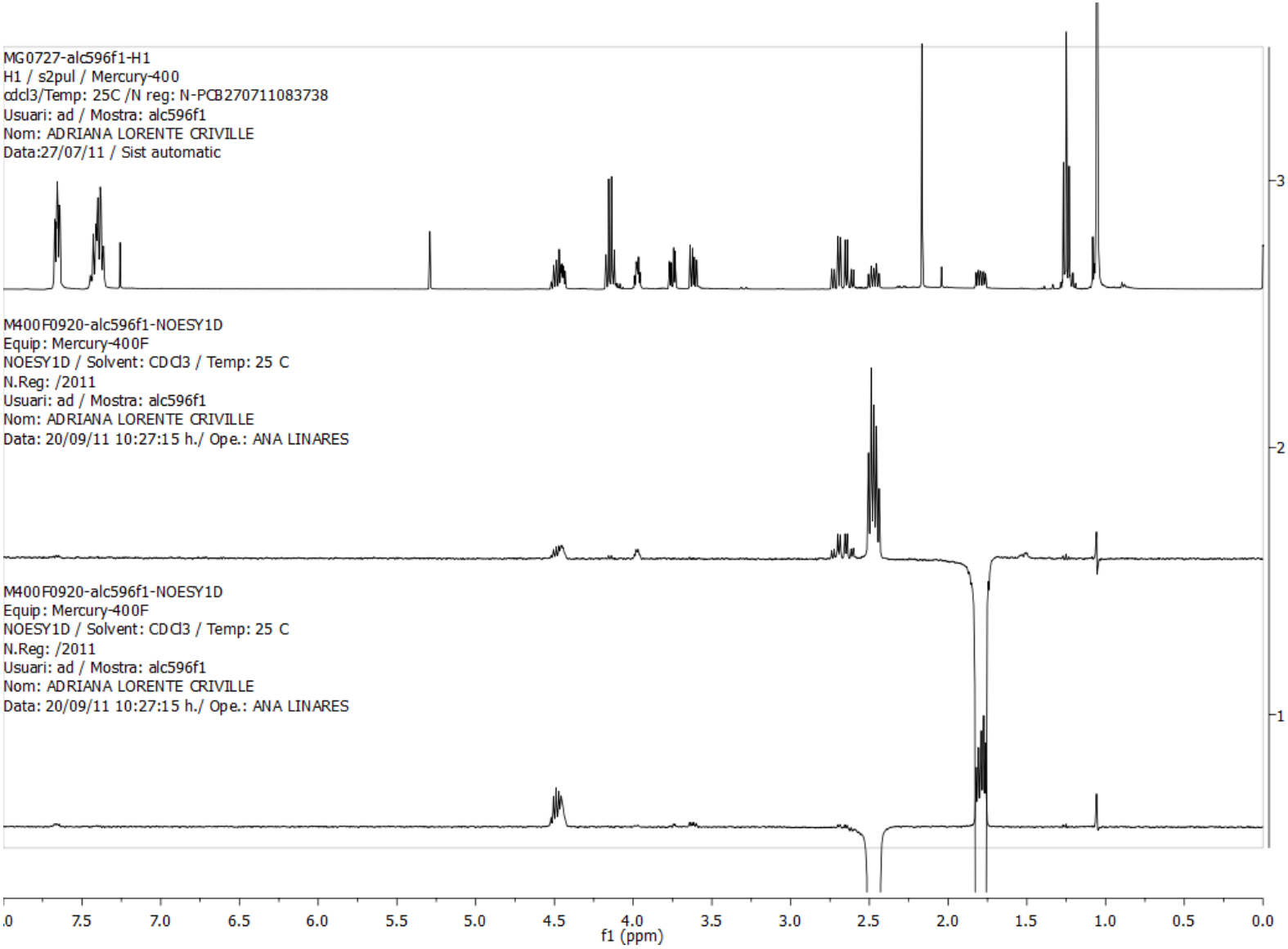
41004DMRP4S3H4
500 MHz CDCl3
Phormidolide C
f2 (ppm)
f1 (ppm)

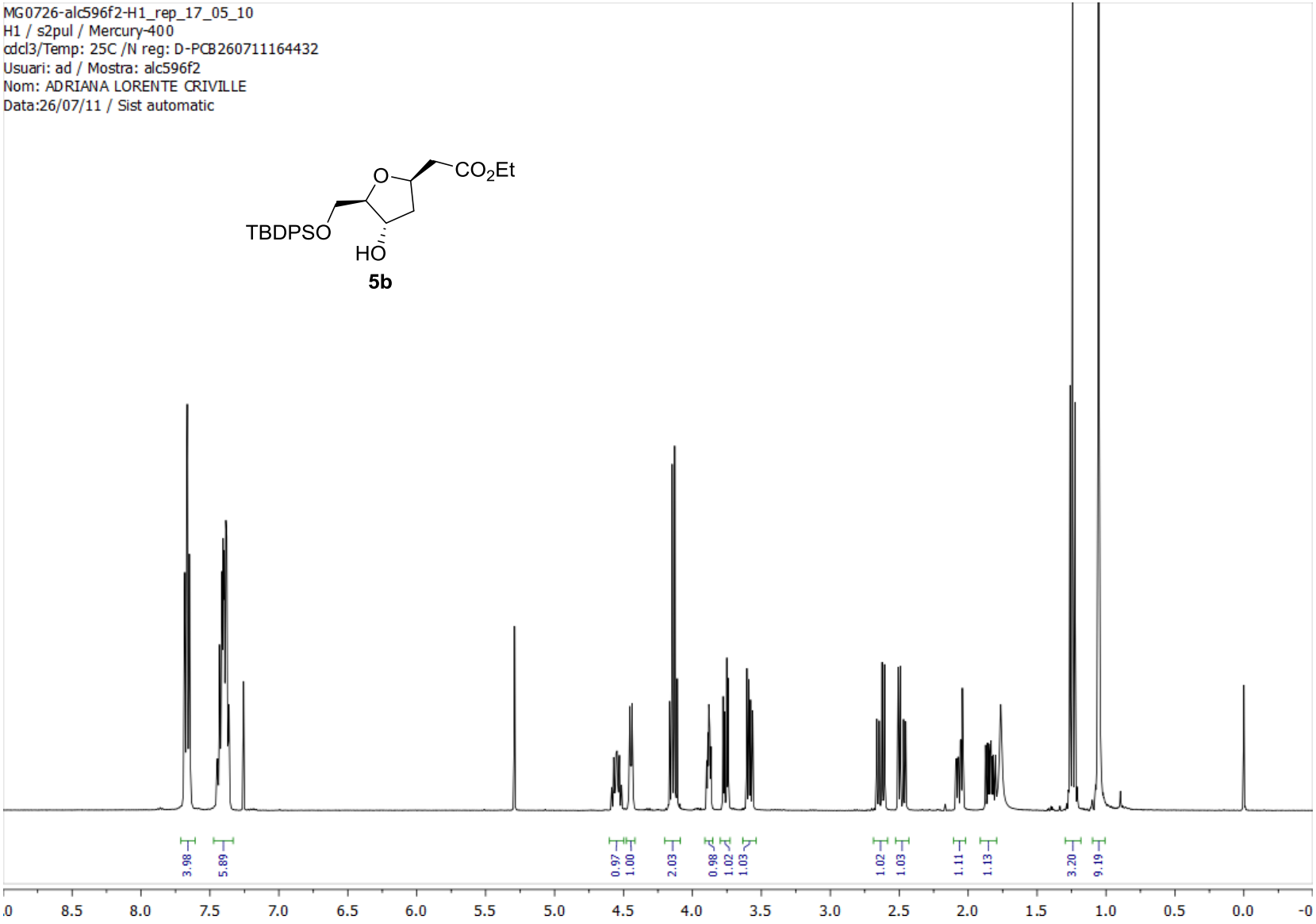

41004DMRP4S3H4-gcosy
41004DMRP4S3H4
500 MHz CDCl3
Phormidolide C
f2 (ppm)
f1 (ppm)

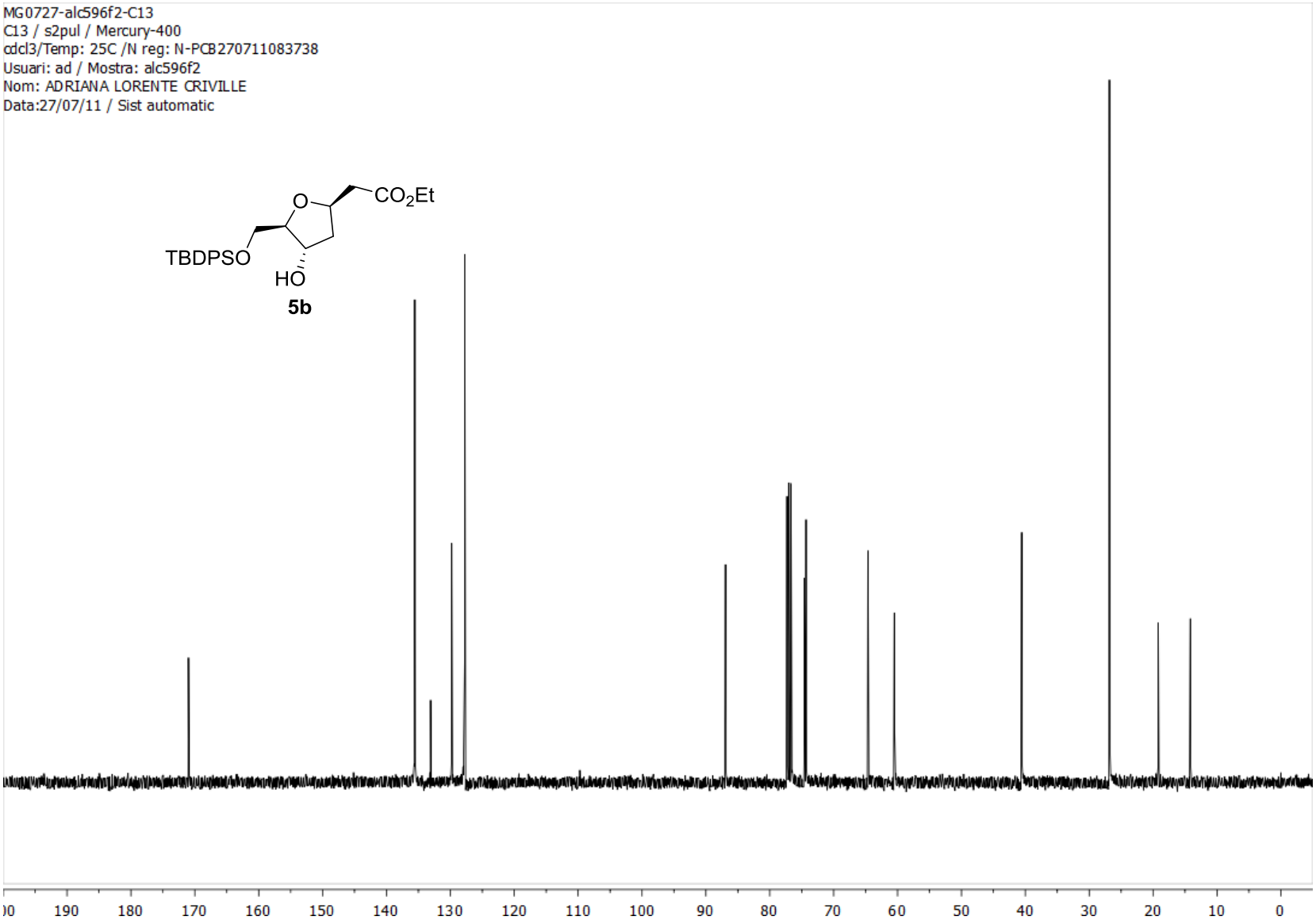

MG0727-alc596f1-H1
H1 / s2pul / Mercury-400
cdcl3/Temp: 25C /N reg: N-PCB270711083738
Usuari: ad / Mostra: alc596f1
Nom: ADRIANA LORENTE CRIVILLE
Data:27/07/11 / Sist automatic
O
CO2Et
TBDPSO
HO
5a
4.02
5.79
1.86
1.98
1.00
0.93
1.01
1.98
1.09
1.09
3.25
9.44
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0

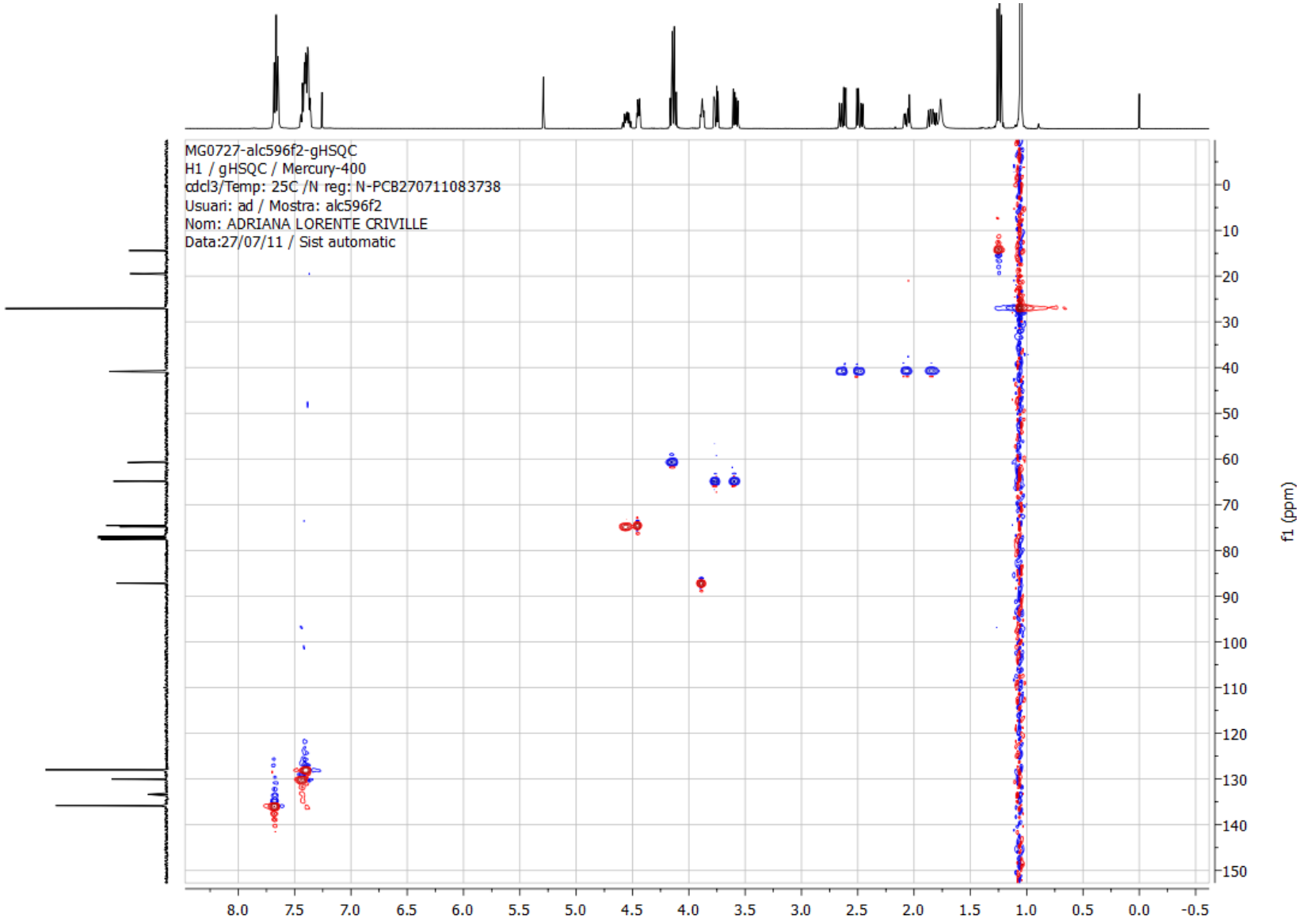

MG0727-alc596f1-C13
C13 / s2pul / Mercury-400
cdcl3/Temp: 25C /N reg: N-PCB270711083738
Usuari: ad / Mostra: alc596f1
Nom: ADRIANA LORENTE CRIVILLE
Data:27/07/11 / Sist automatic
O
CO2Et
TBDPSO
HO
5a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

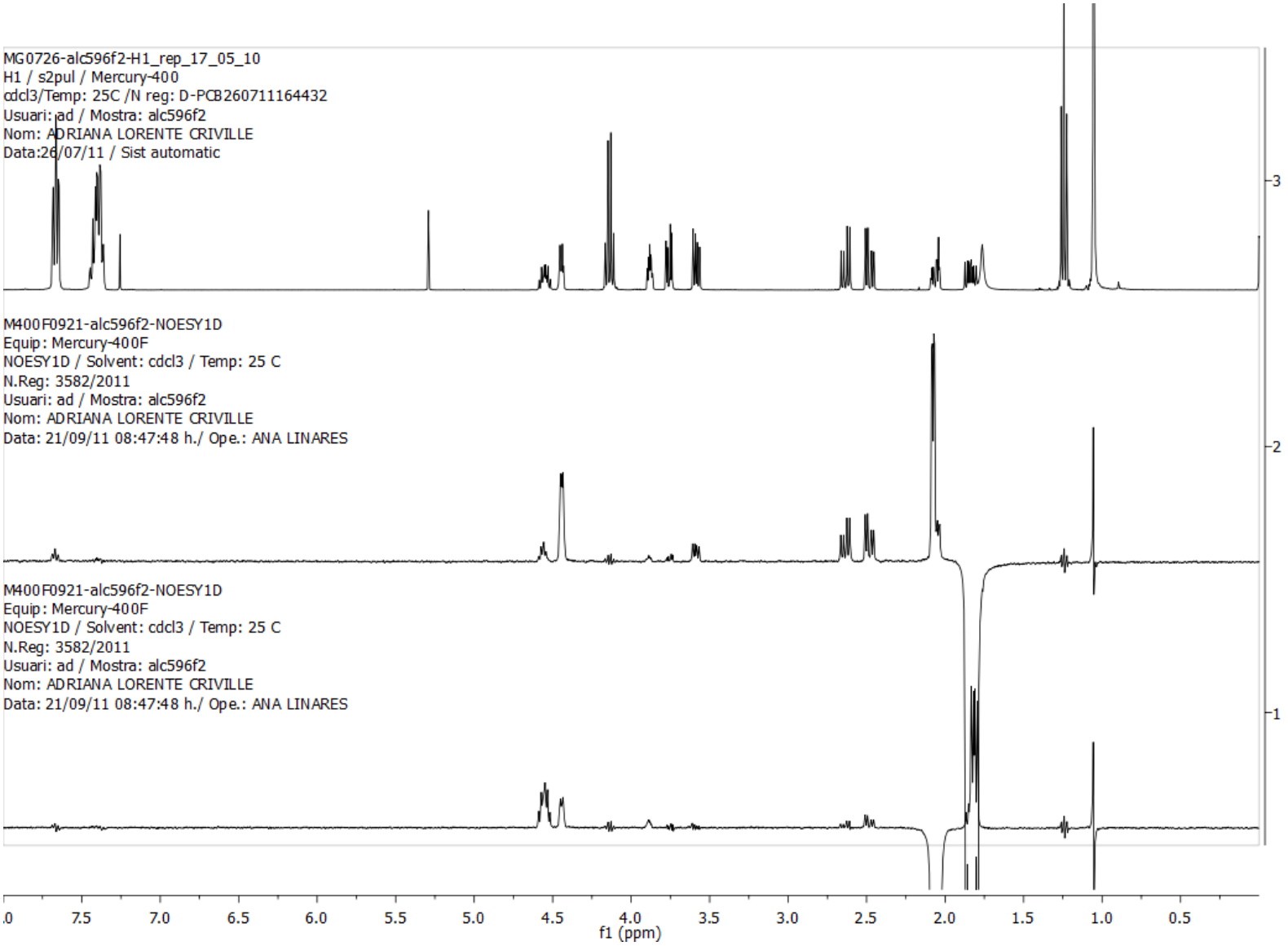

MG0727-alc596f1-gHSQC
H1 / gHSQC / Mercury-400
cdcl3/Temp: 25C /N reg: N-PCB270711083738
Usuari: ad / Mostra: alc596f1
Nom: ADRIANA LORENTE CRIVILLE
Data:27/07/11 / Sist automatic
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

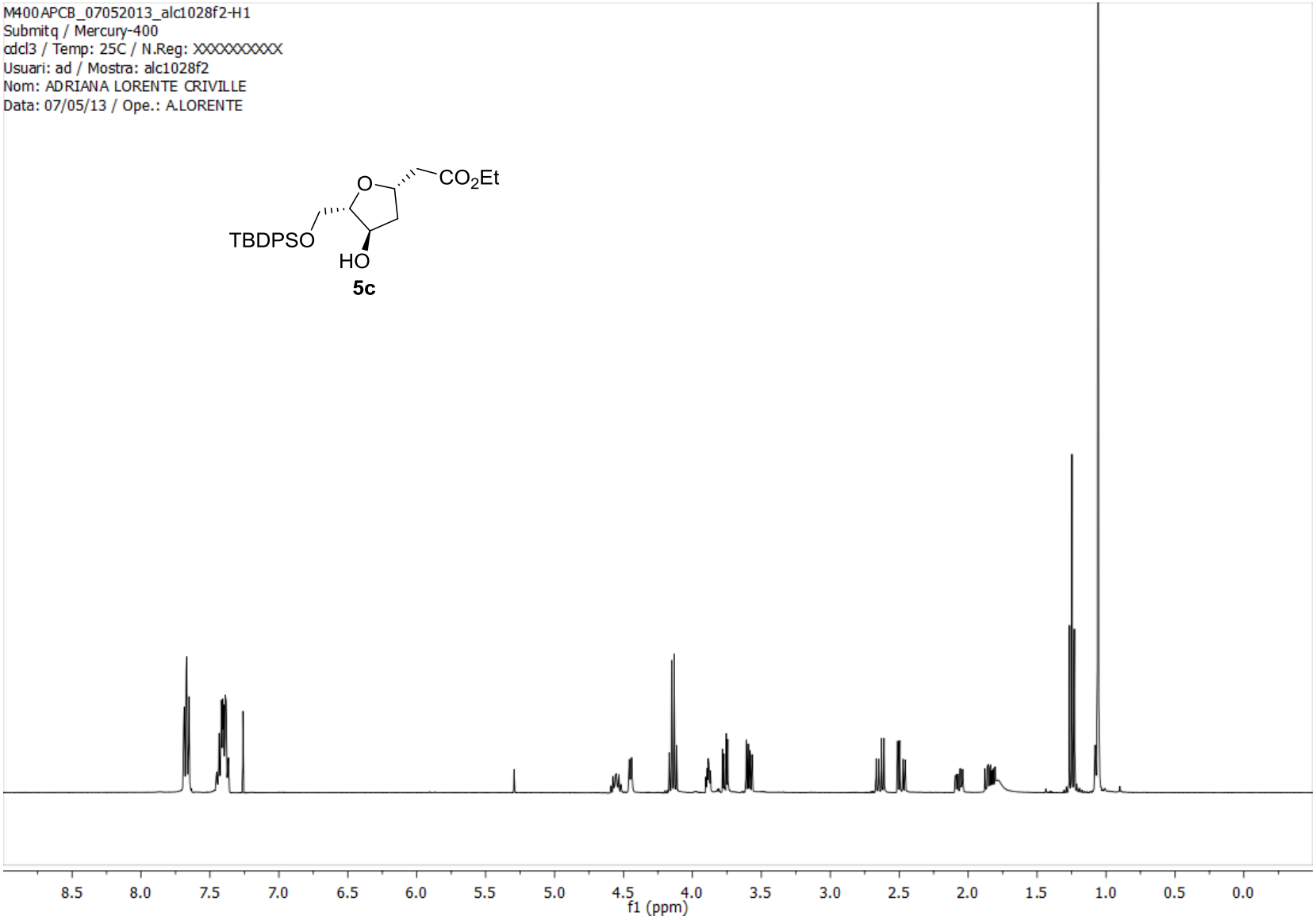

MG0727-alc596f1-H1
H1 / s2pul / Mercury-400
cdcl3/Temp: 25C /N reg: N-PCB270711083738
Usuari: ad / Mostra: alc596f1
Nom: ADRIANA LORENTE CRIVILLE
Data:27/07/11 / Sist automatic
M400F0920-alc596f1-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: CDCl3 / Temp: 25 C
N.Reg: /2011
Usuari: ad / Mostra: alc596f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 20/09/11 10:27:15 h./ Ope.: ANA LINARES
M400F0920-alc596f1-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: CDCl3 / Temp: 25 C
N.Reg: /2011
Usuari: ad / Mostra: alc596f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 20/09/11 10:27:15 h./ Ope.: ANA LINARES
3
2
1
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)

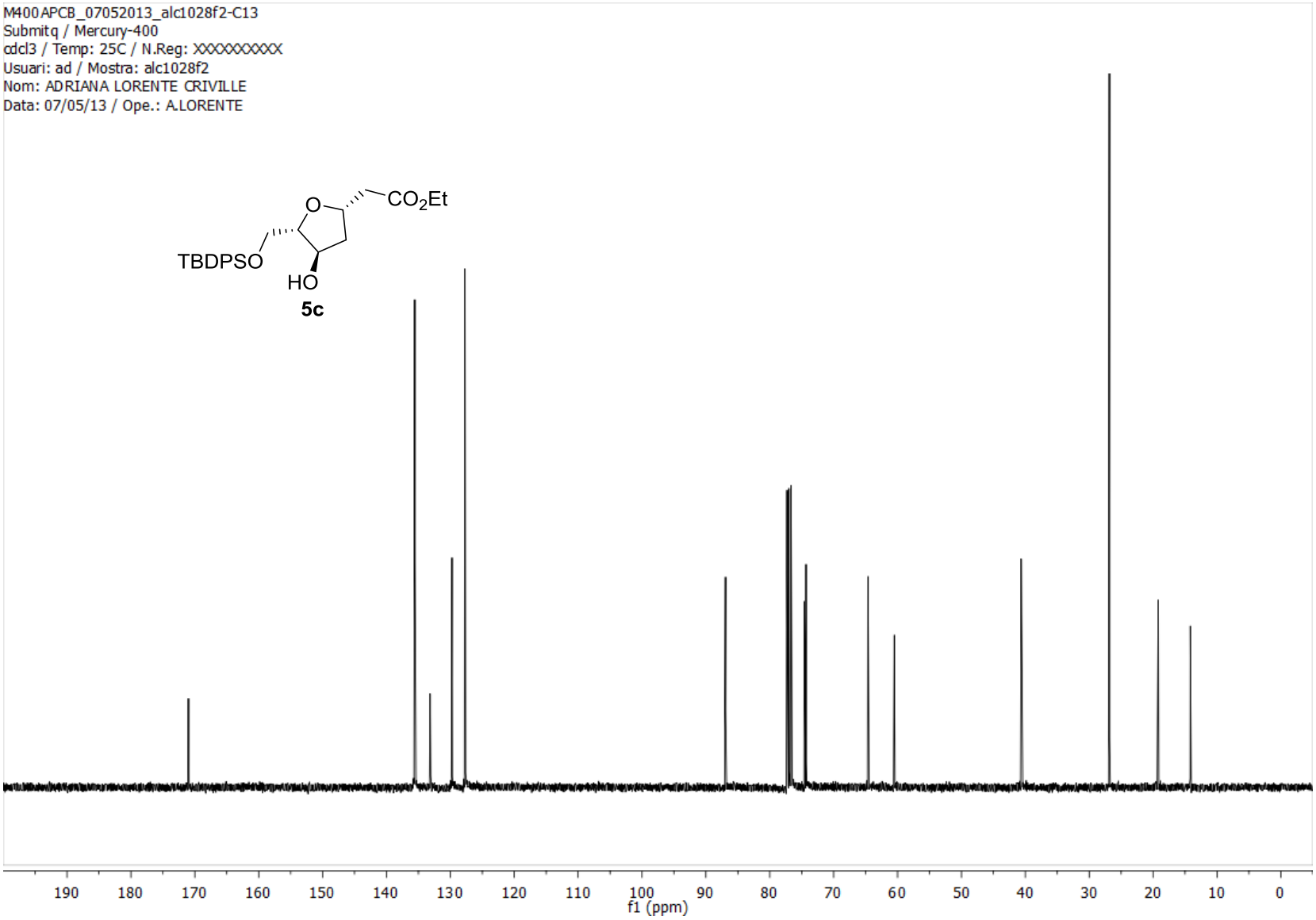

MG0726-alc596f2-H1_rep_17_05_10
H1 / s2pul / Mercury-400
cdcl3/Temp: 25C /N reg: D-PCB260711164432
Usuari: ad / Mostra: alc596f2
Nom: ADRIANA LORENTE CRIVILLE
Data:26/07/11 / Sist automatic
O
CO2Et
TBDPSO
HO
5b
3.98
5.89
0.97
1.00
2.03
0.98
1.02
1.03
1.02
1.03
1.11
1.13
3.20
9.19
8.5
8.0
7.5
7.0
6.5
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

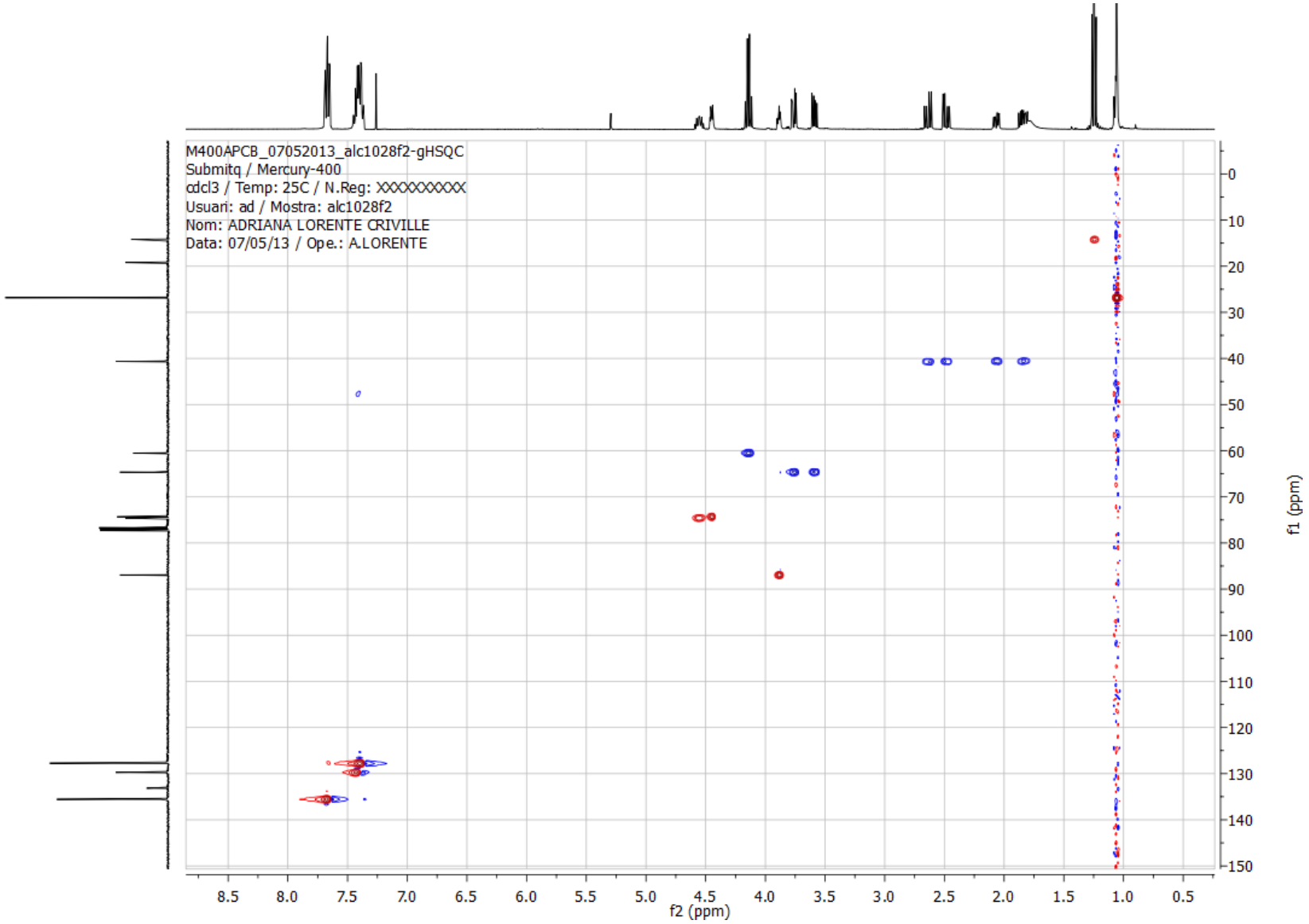

MG0727-alc596f2-C13
C13 / s2pul / Mercury-400
cdcl3/Temp: 25C /N reg: N-PCB270711083738
Usuari: ad / Mostra: alc596f2
Nom: ADRIANA LORENTE CRIVILLE
Data:27/07/11 / Sist automatic
O
CO2Et
TBDPSO
HO
5b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


MG0727-alc596f2-gHSQC
H1 / gHSQC / Mercury-400
cdcl3/Temp: 25C /N reg: N-PCB270711083738
Usuari: ad / Mostra: alc596f2
Nom: ADRIANA LORENTE CRIVILLE
Data:27/07/11 / Sist automatic
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
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
-0.5

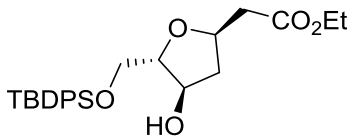

MG0726-alc596f2-H1_rep_17_05_10
H1 / s2pul / Mercury-400
cdcl3/Temp: 25C /N reg: D-PCB260711164432
Usuari: ad / Mostra: alc596f2
Nom: ADRIANA LORENTE CRIVILLE
Data:26/07/11 / Sist automatic
M400F0921-alc596f2-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 3582/2011
Usuari: ad / Mostra: alc596f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 21/09/11 08:47:48 h./ Ope.: ANA LINARES
M400F0921-alc596f2-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 3582/2011
Usuari: ad / Mostra: alc596f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 21/09/11 08:47:48 h./ Ope.: ANA LINARES
3
2
1
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
f1 (ppm)


M400APCB_07052013_alc1028f2-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1028f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 07/05/13 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
HO
5c
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)


M400APCB_07052013_alc1028f2-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1028f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 07/05/13 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
HO
5c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

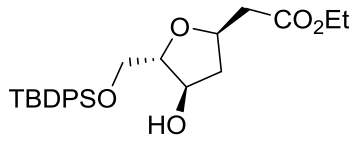

M400APCB_07052013_alc1028f2-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1028f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 07/05/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

M400APCB_13012014_ALC_1118-1-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC_1118-1
Nom: JANIRE LAMARIANO MERKETEGI
Data: 13/01/14 / Ope.: J.LAMARIANO

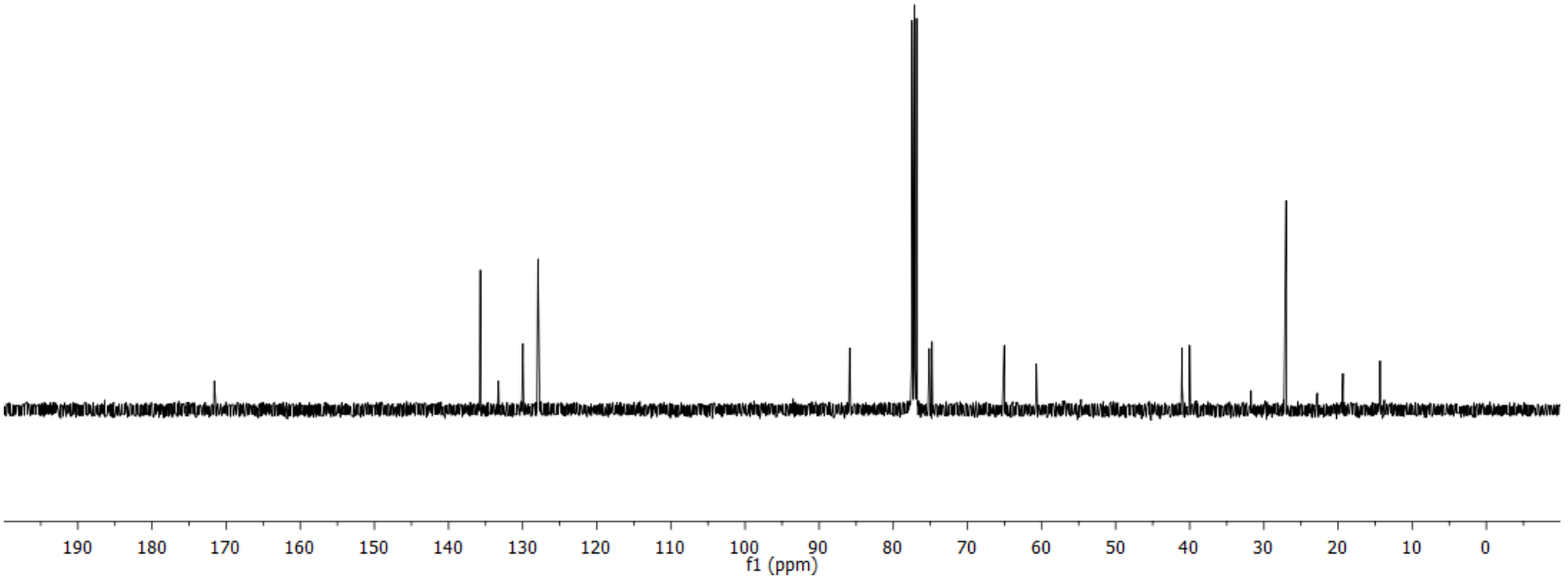


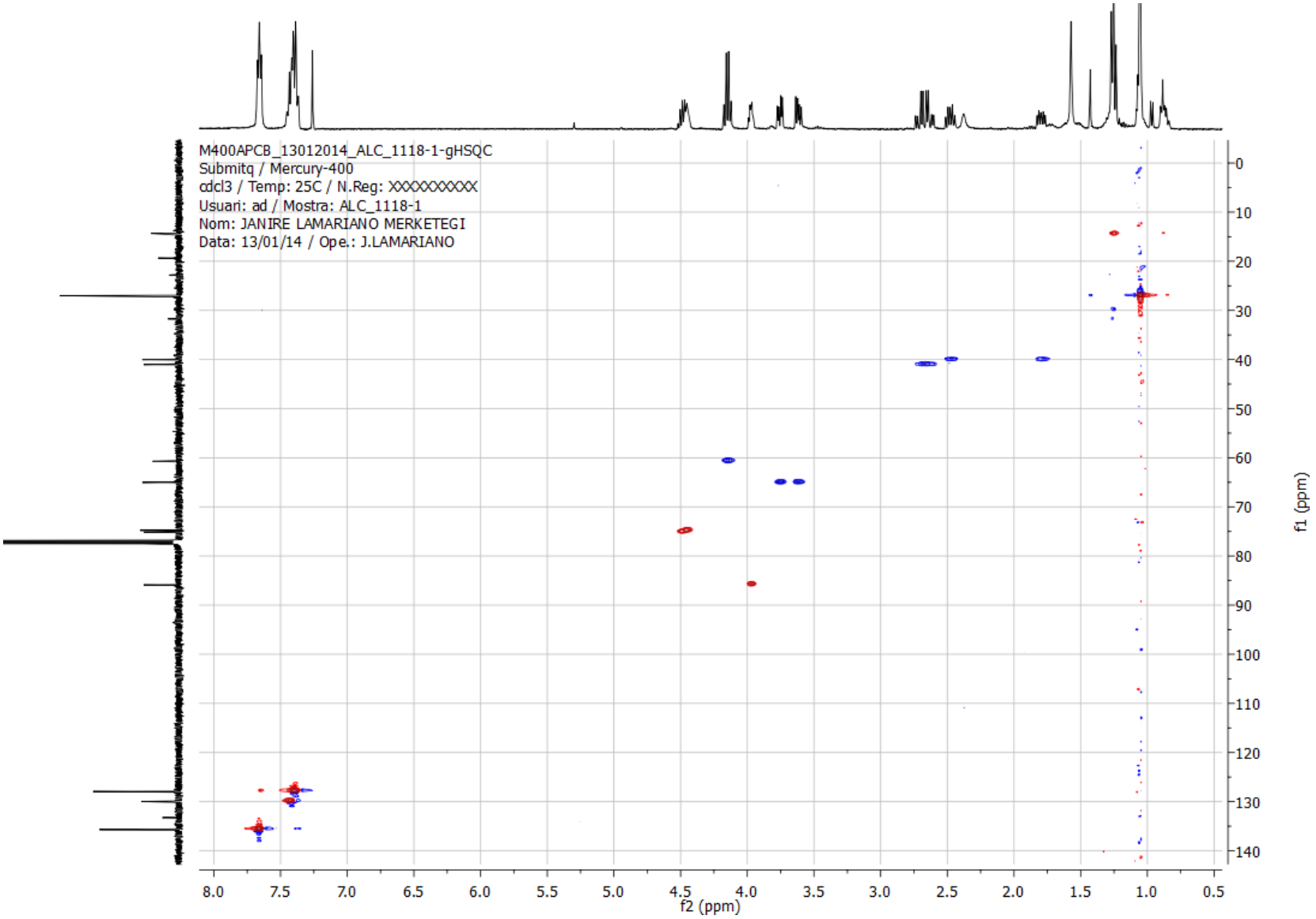
5d

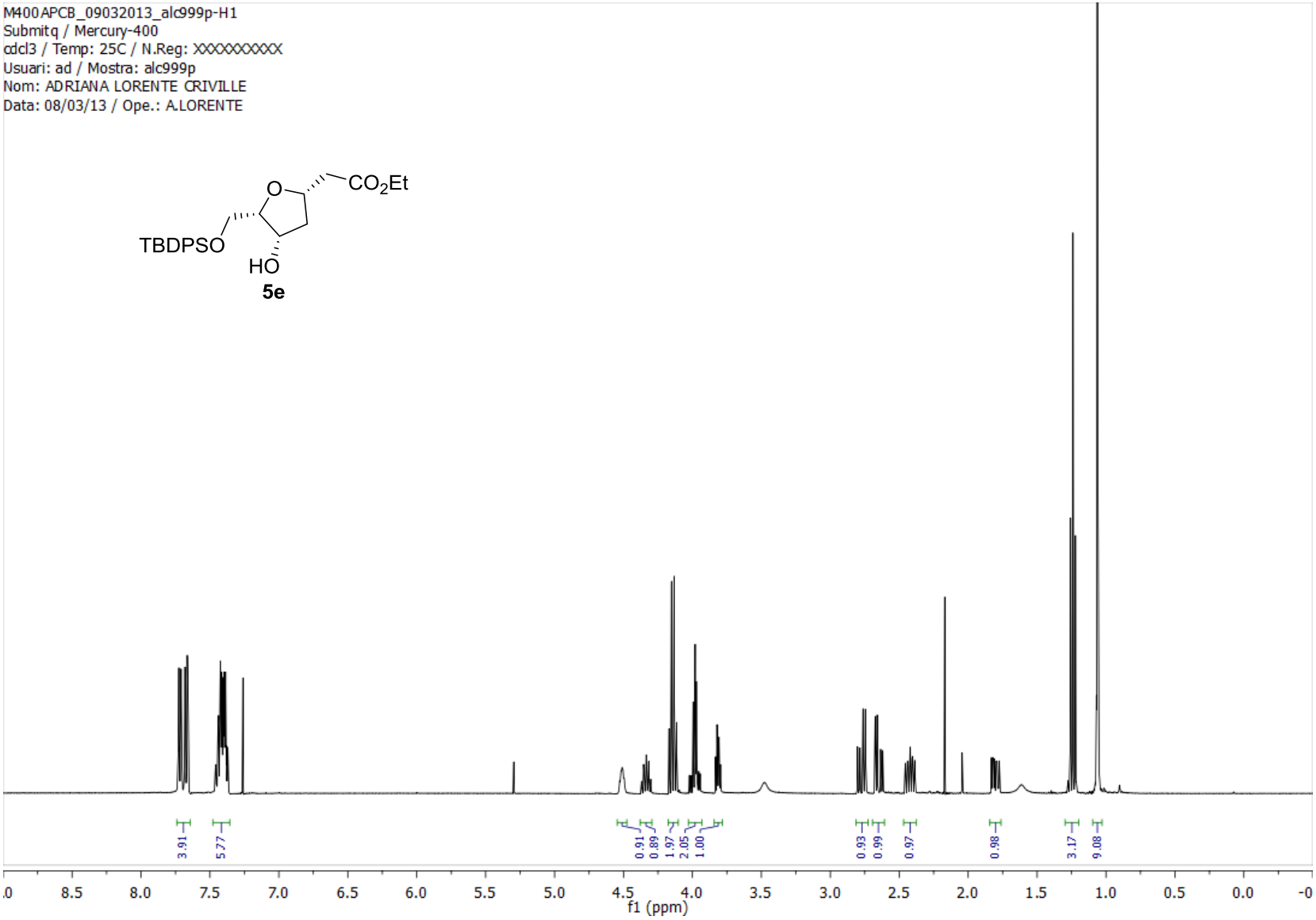
M400APCB_13012014_ALC_1118-1-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC_1118-1
Nom: JANIRE LAMARIANO MERKETEGI
Data: 13/01/14 / Ope.: J.LAMARIANO

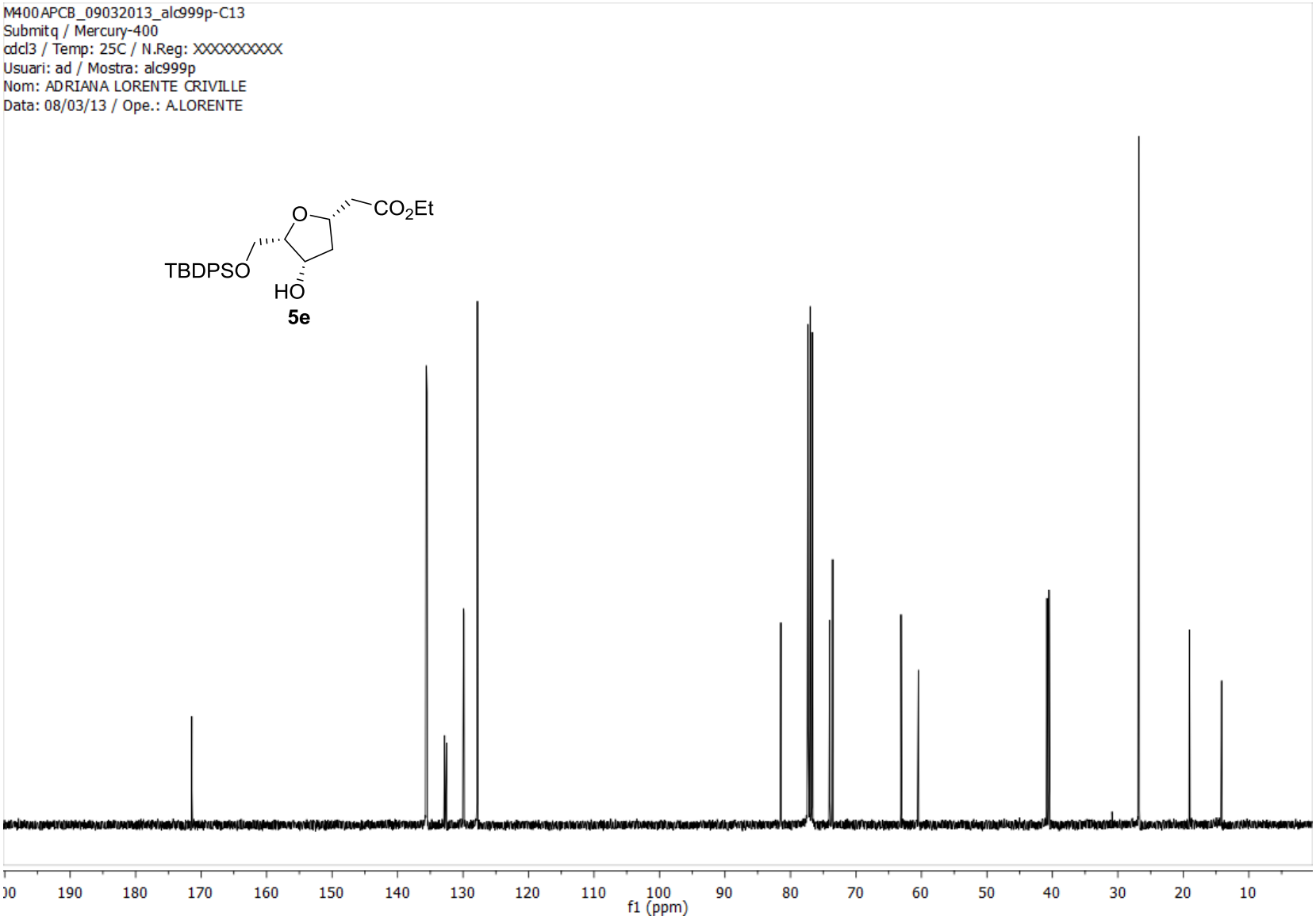


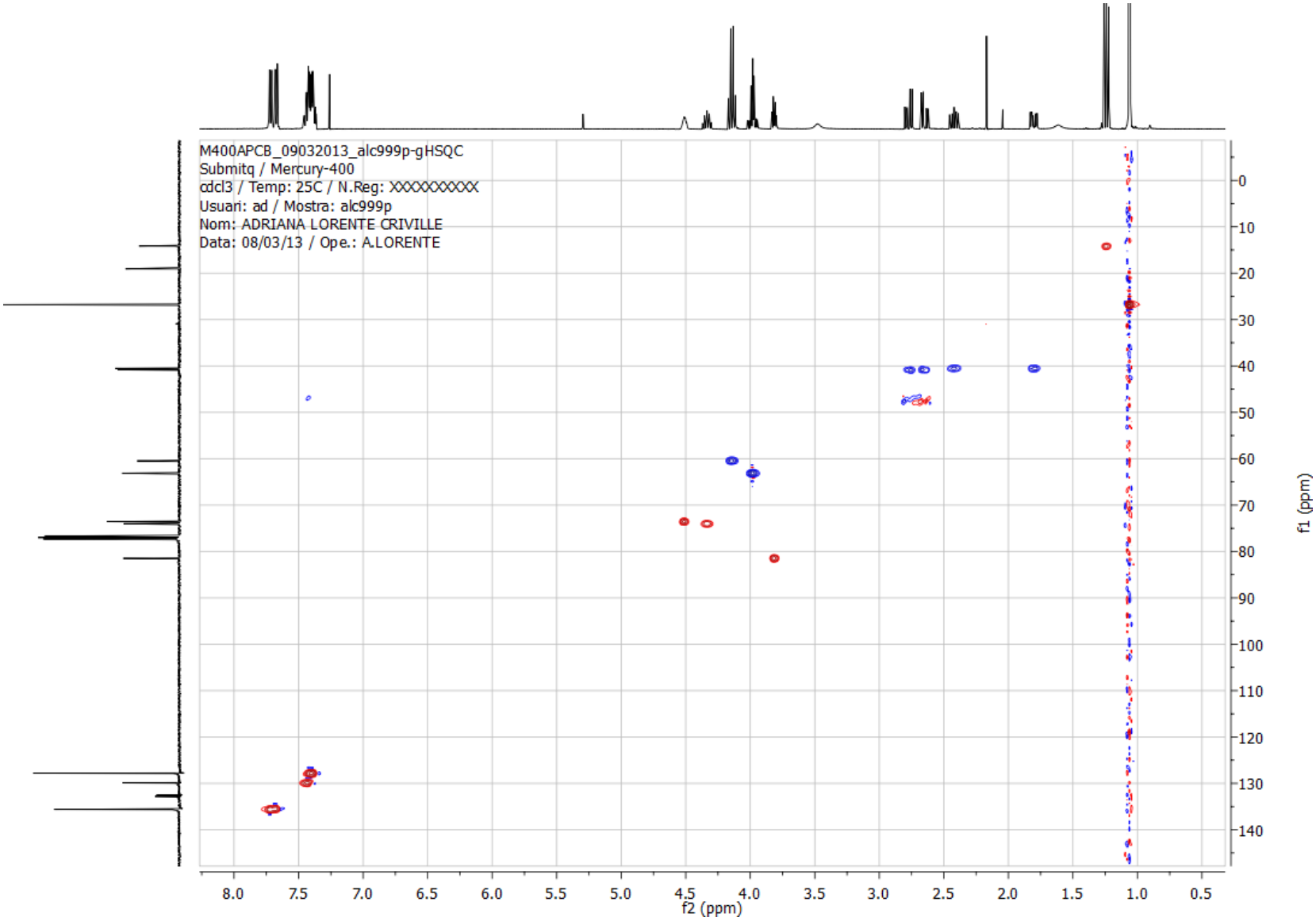
5d

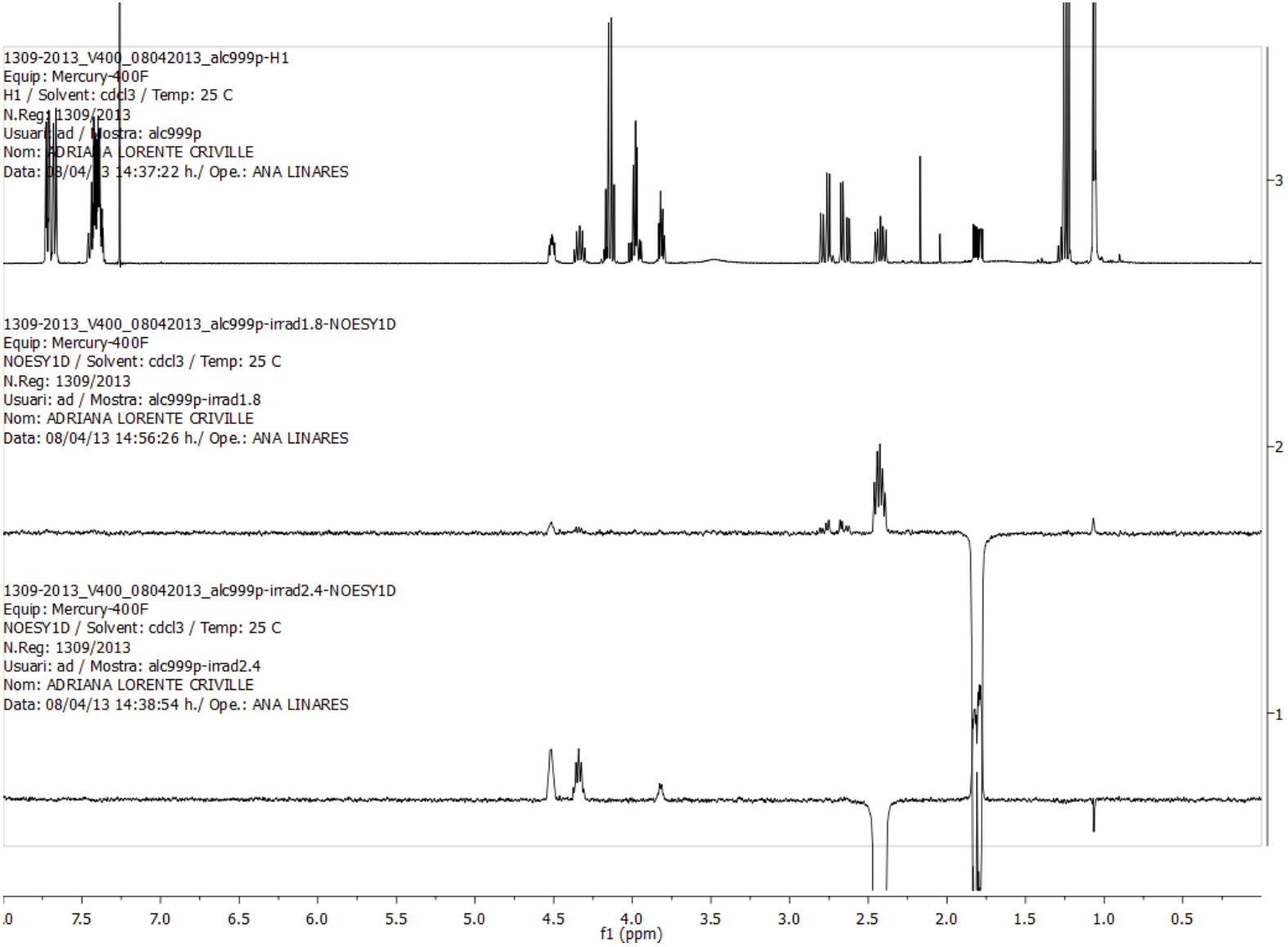


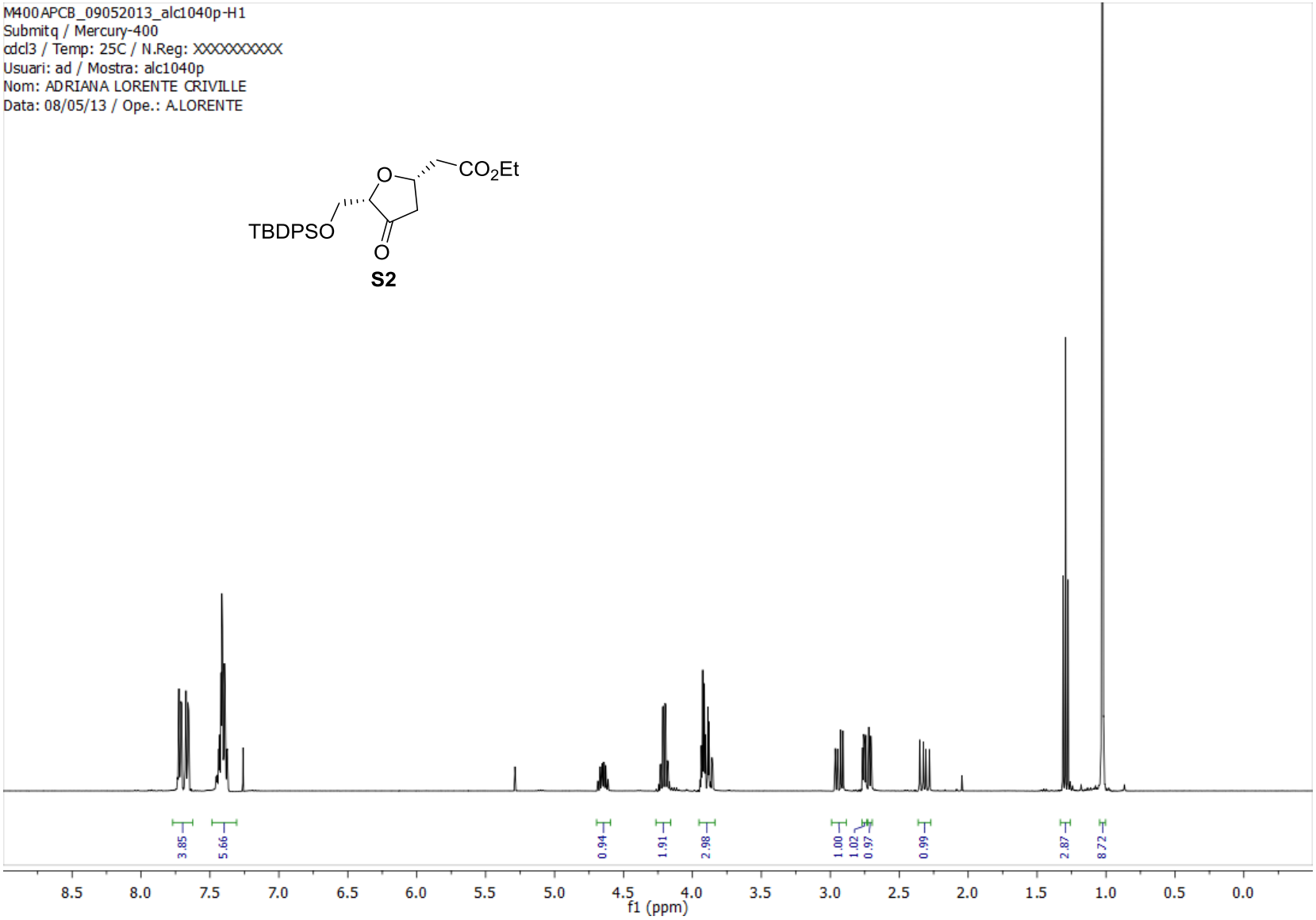

M400APCB_13012014_ALC_1118-1-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC_1118-1
Nom: JANIRE LAMARIANO MERKETEGI
Data: 13/01/14 / Ope.: J.LAMARIANO
f2 (ppm)
f1 (ppm)

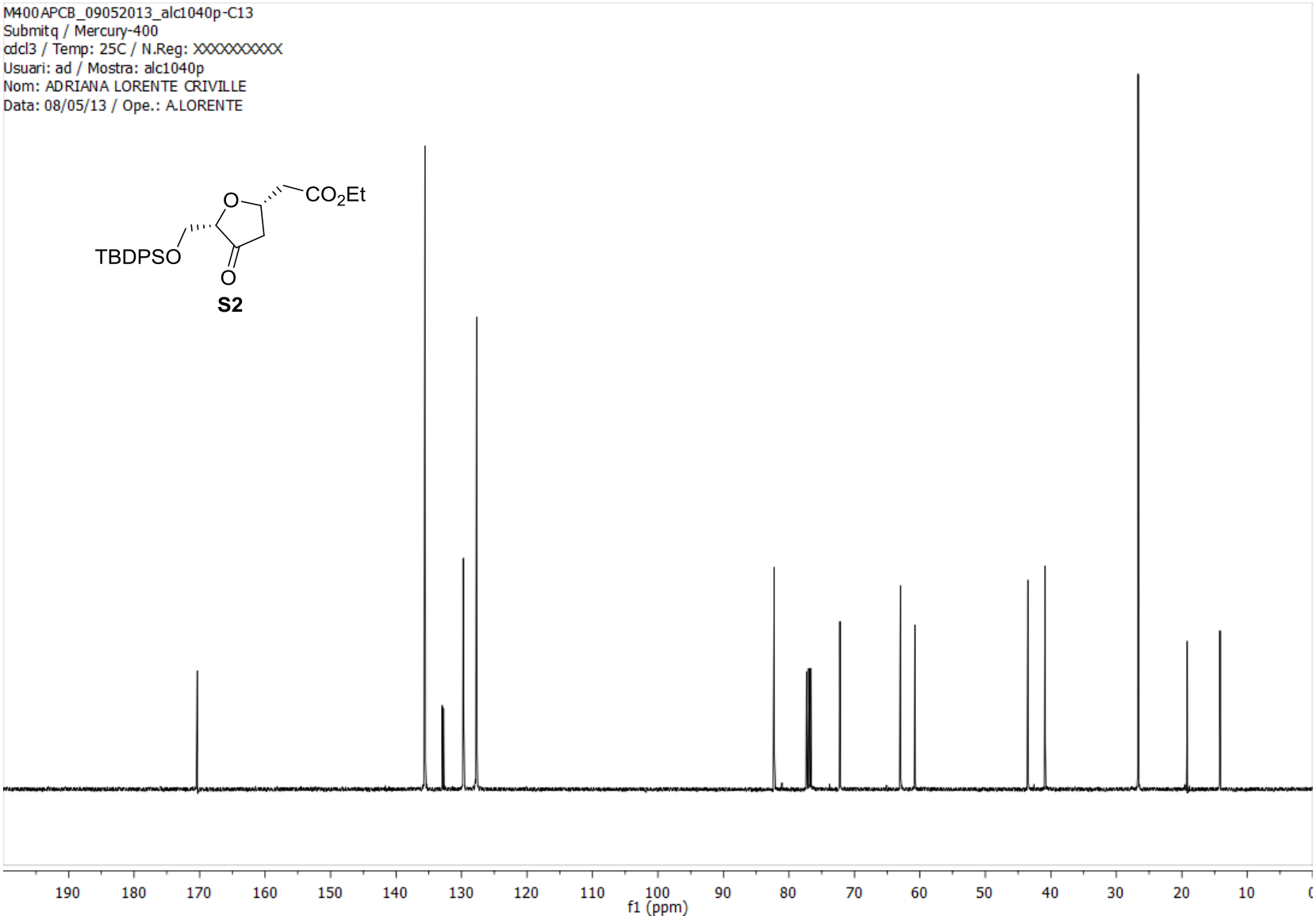

M400APCB_09032013_alc999p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc999p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/03/13 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
HO
5e
3.91
5.77
0.91
0.89
1.97
2.05
1.00
0.93
0.99
0.97
0.98
3.17
9.08
f1 (ppm)

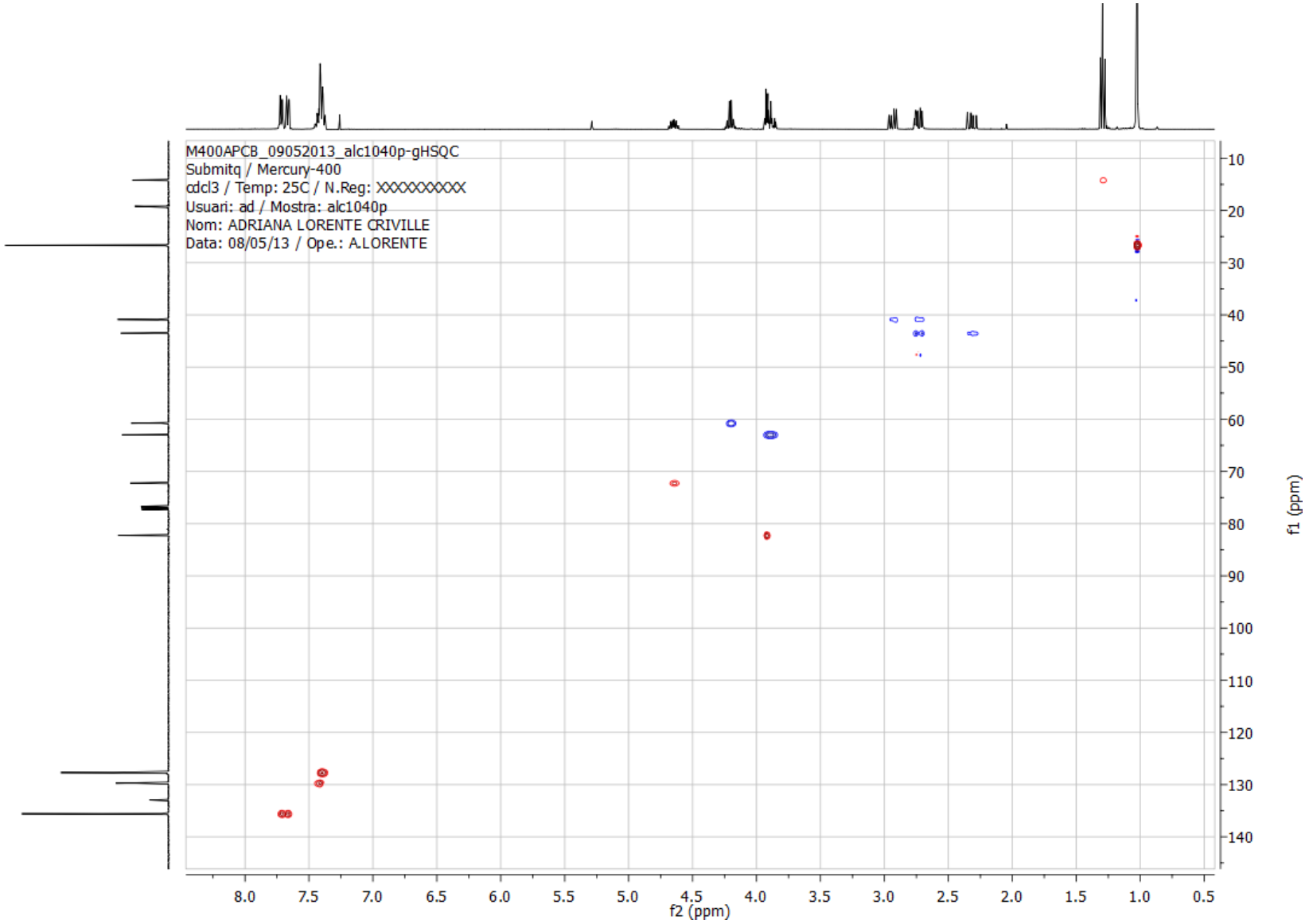

M400APCB_09032013_alc999p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: ad / Mostra: alc999p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/03/13 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
HO
5e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

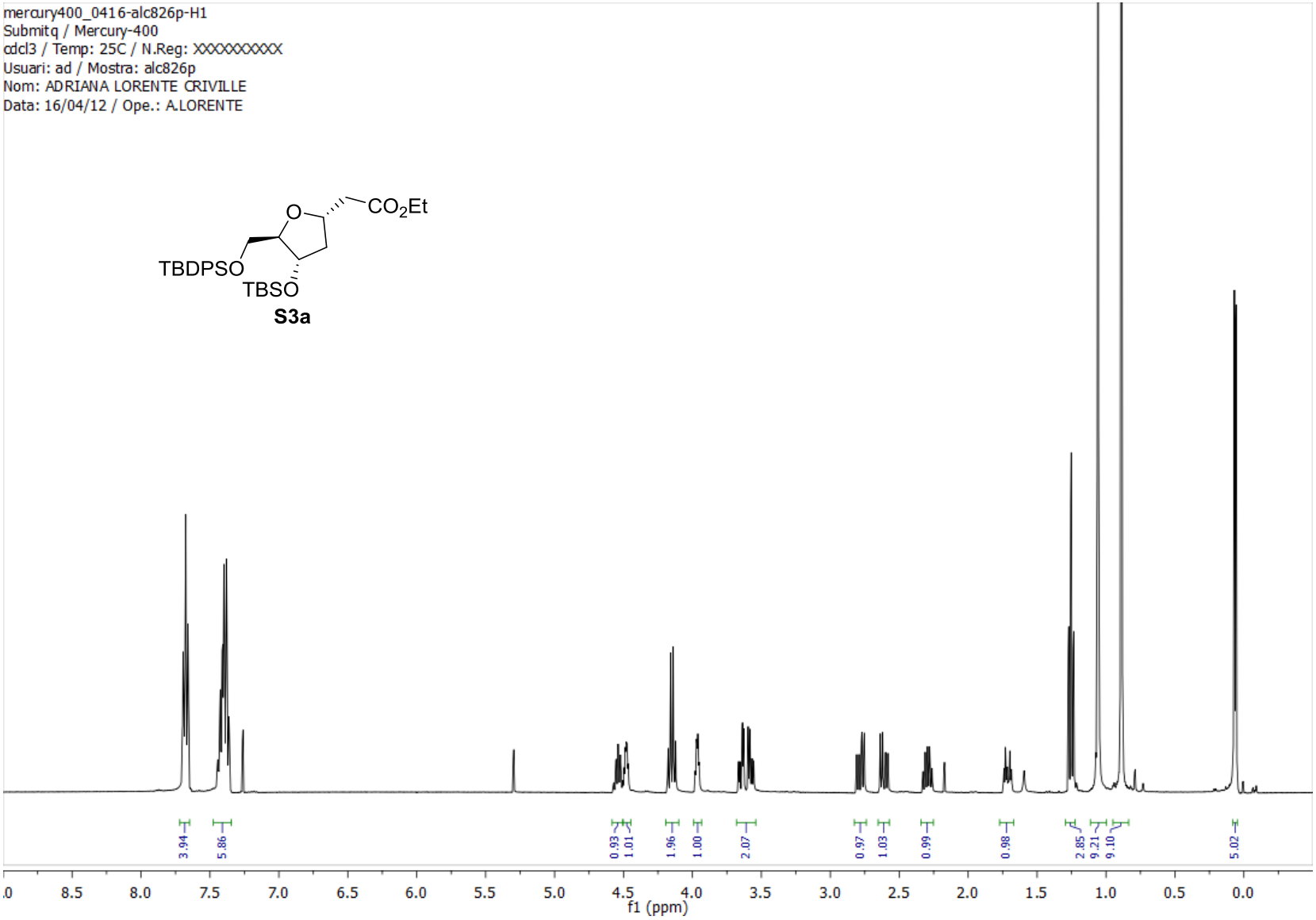

M400APCB_09032013_alc999p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc999p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/03/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

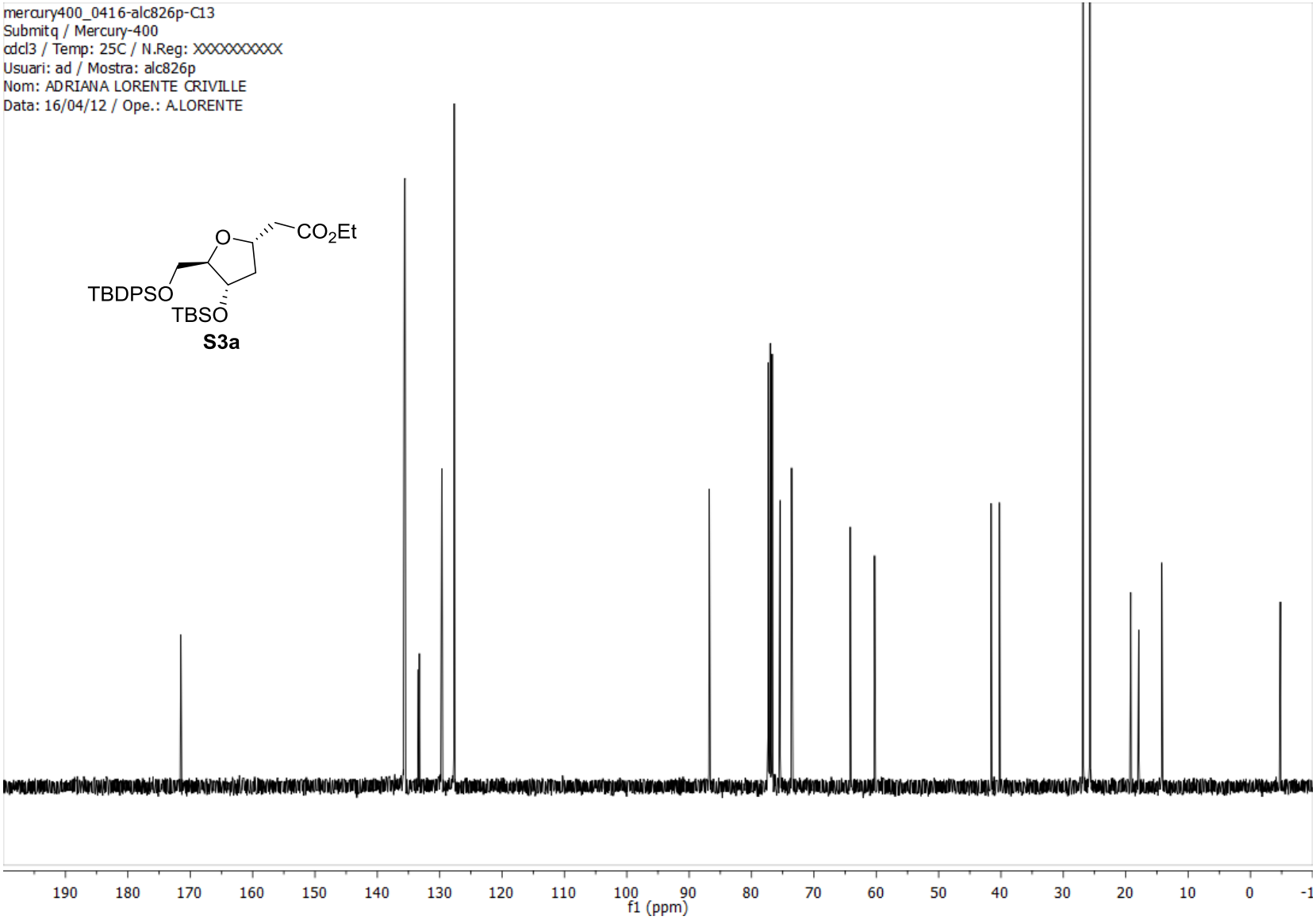

1309-2013_V400_08042013_alc999p-H1
Equip: Mercury-400F
H1 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 1309/2013
Usuari: ad / Mostra: alc999p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/04/13 14:37:22 h./ Ope.: ANA LINARES
1309-2013_V400_08042013_alc999p-irrad1.8-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 1309/2013
Usuari: ad / Mostra: alc999p-irrad1.8
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/04/13 14:56:26 h./ Ope.: ANA LINARES
1309-2013_V400_08042013_alc999p-irrad2.4-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 1309/2013
Usuari: ad / Mostra: alc999p-irrad2.4
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/04/13 14:38:54 h./ Ope.: ANA LINARES
3
2
1
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
f1 (ppm)

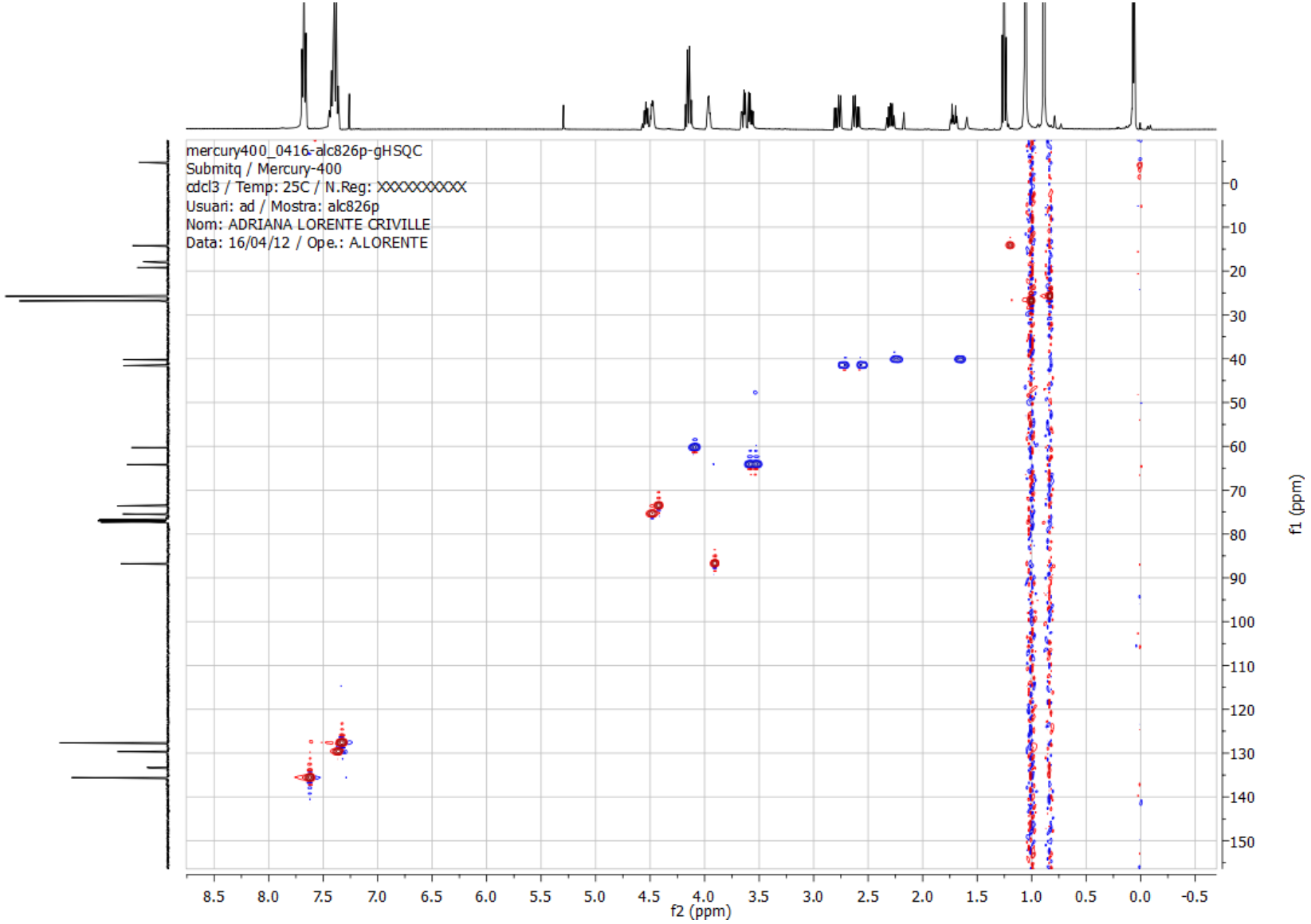

M400APCB_09052013_alc1040p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1040p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/05/13 / Ope.: A.LORENTE
CO2Et
O
TBDPSO
O
S2
3.85
5.66
0.94
1.91
2.98
1.00
1.02
0.97
0.99
2.87
8.72
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

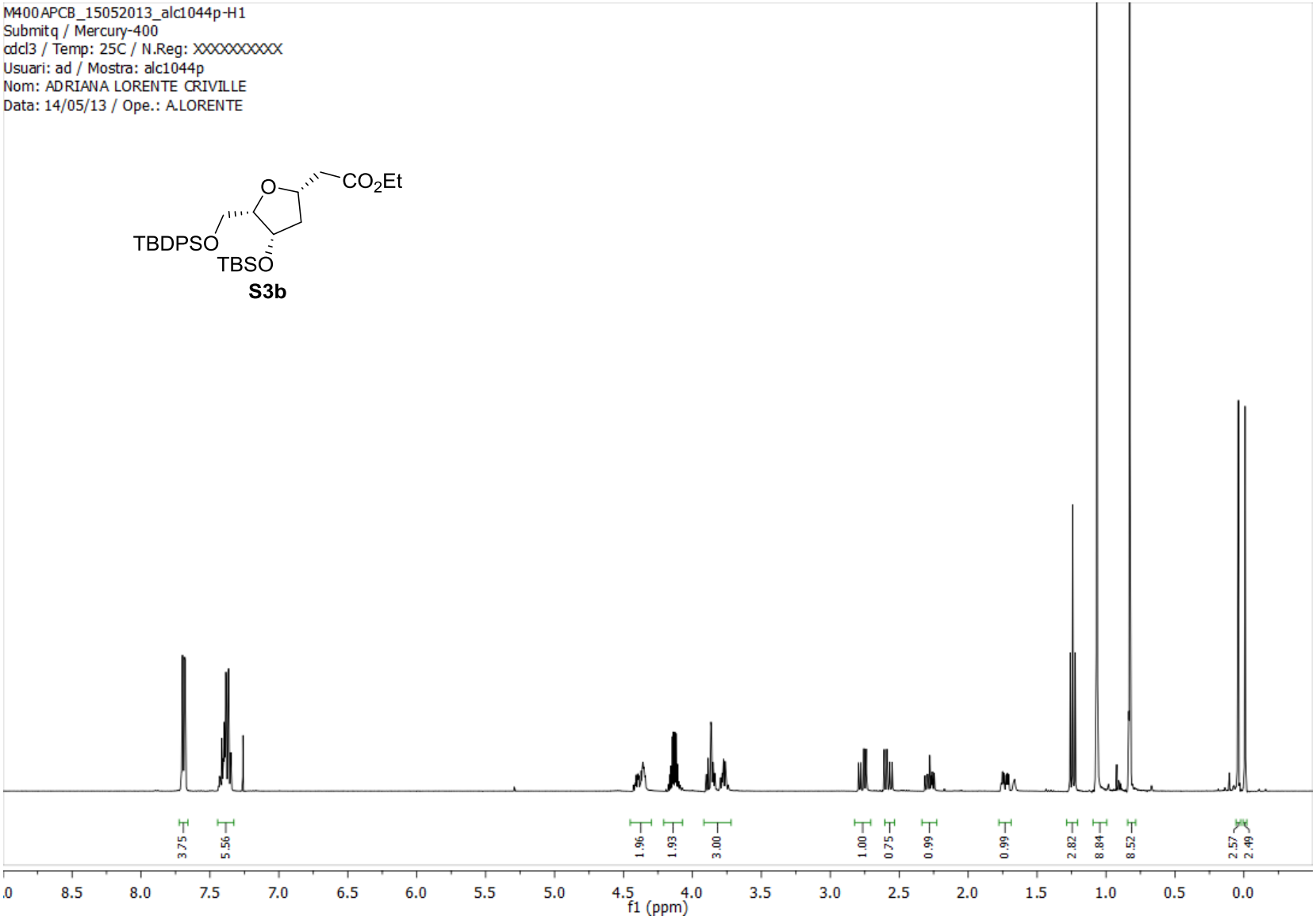

M400APCB_09052013_alc1040p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1040p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/05/13 / Ope.: A.LORENTE
CO2Et
O
TBDPSO
O
S2
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

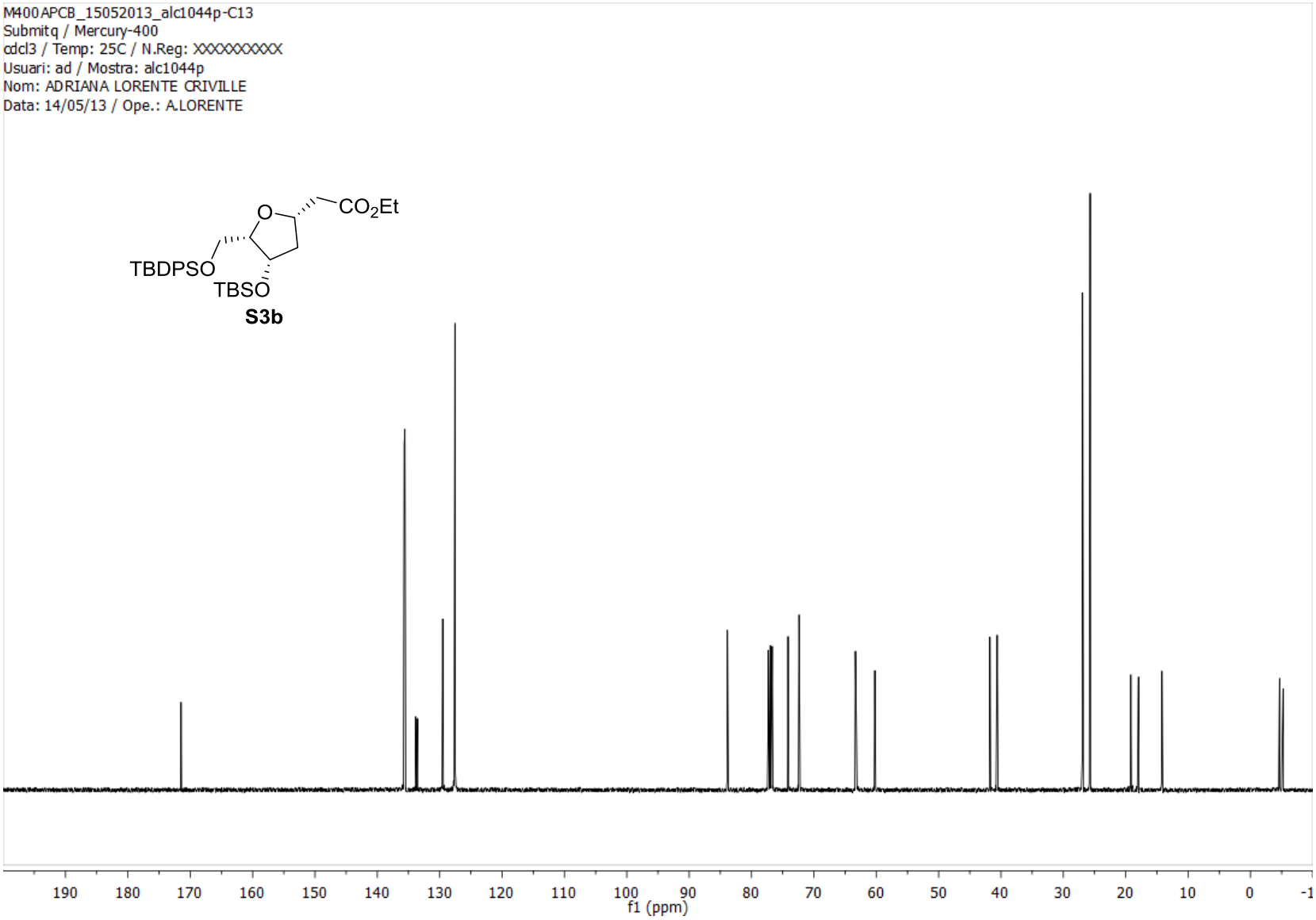

M400APCB_09052013_alc1040p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1040p
Nom: ADRIANA LORENTE CRIVILLE
Data: 08/05/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

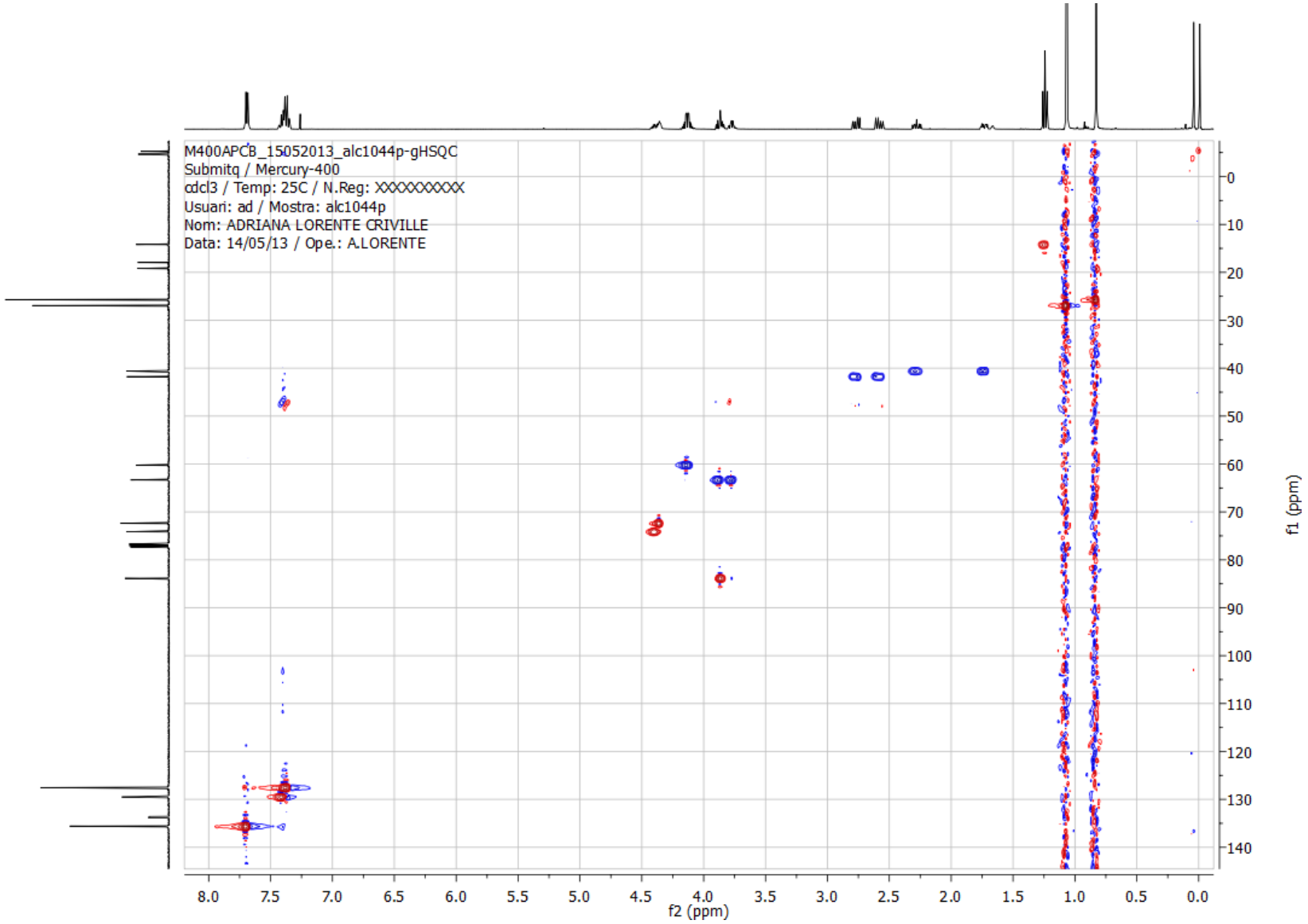

mercury400_0416-alc826p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc826p
Nom: ADRIANA LORENTE CRIVILLE
Data: 16/04/12 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
TBSO
S3a
3.94
5.86
0.93
1.01
1.96
1.00
2.07
0.97
1.03
0.99
0.98
2.85
9.21
9.10
5.02
8.5
8.0
7.5
7.0
6.5
6.0
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)

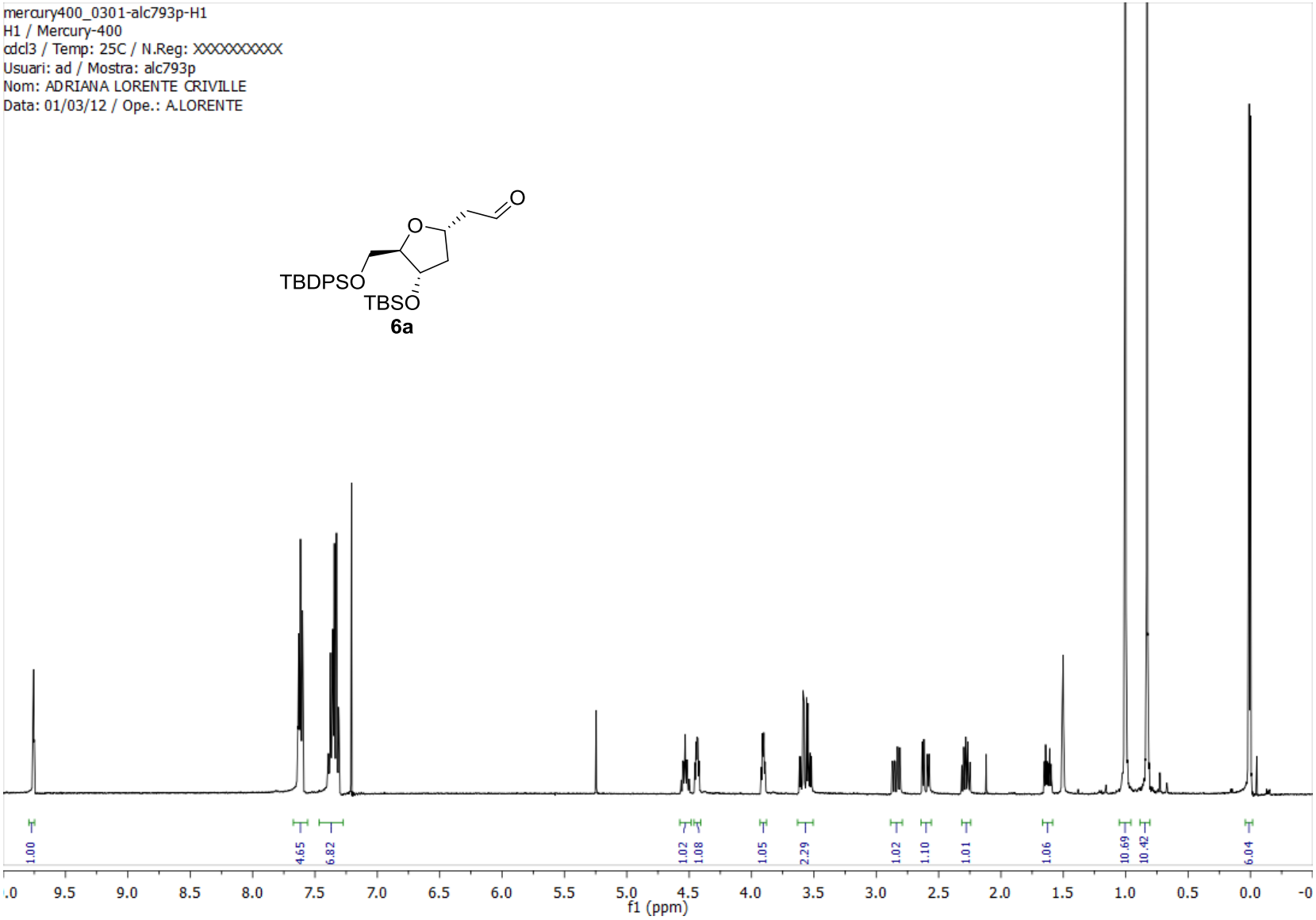

mercury400_0416-alc826p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc826p
Nom: ADRIANA LORENTE CRIVILLE
Data: 16/04/12 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
TBSO
S3a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

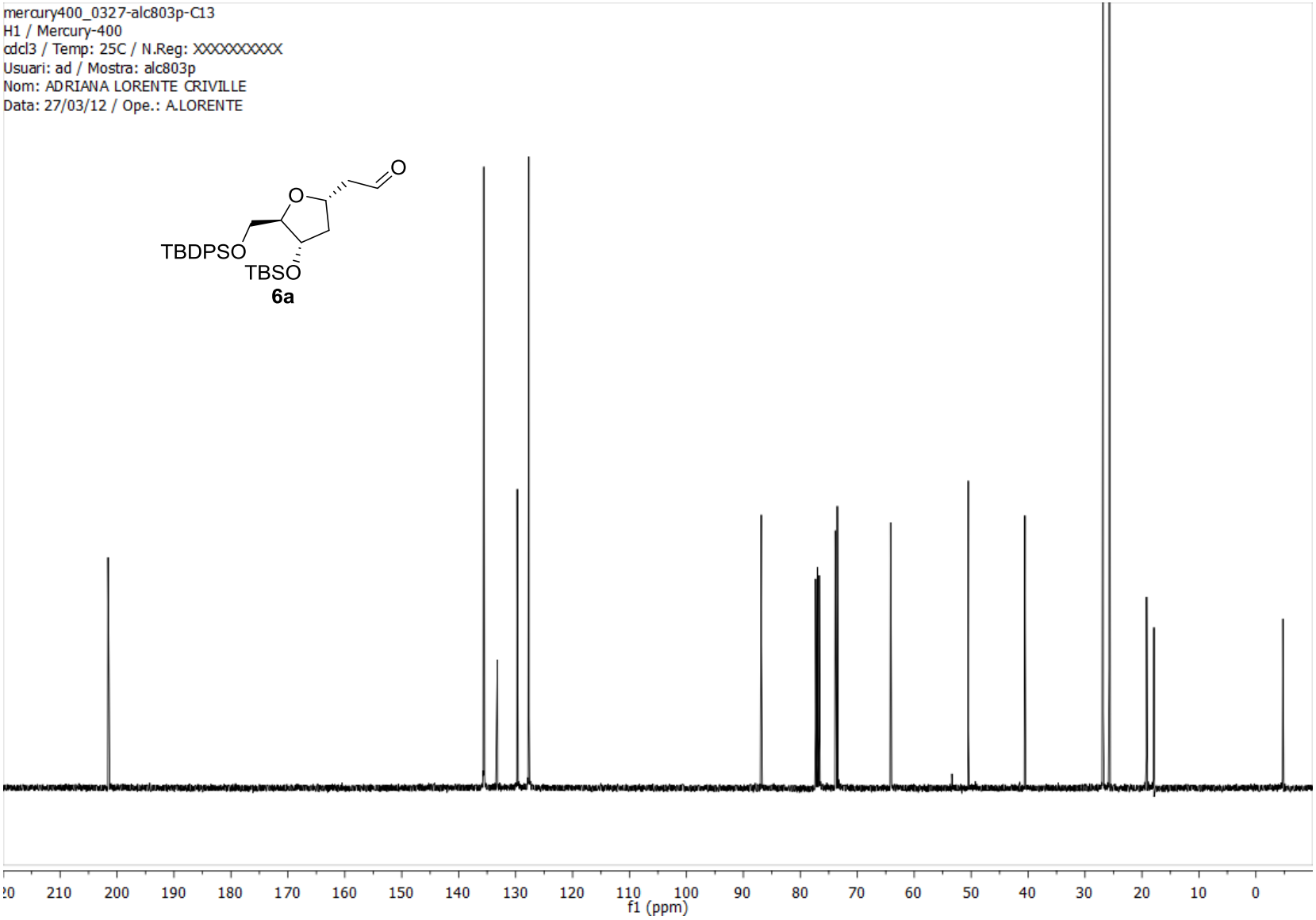

mercury400_0416-alc826p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc826p
Nom: ADRIANA LORENTE CRIVILLE
Data: 16/04/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

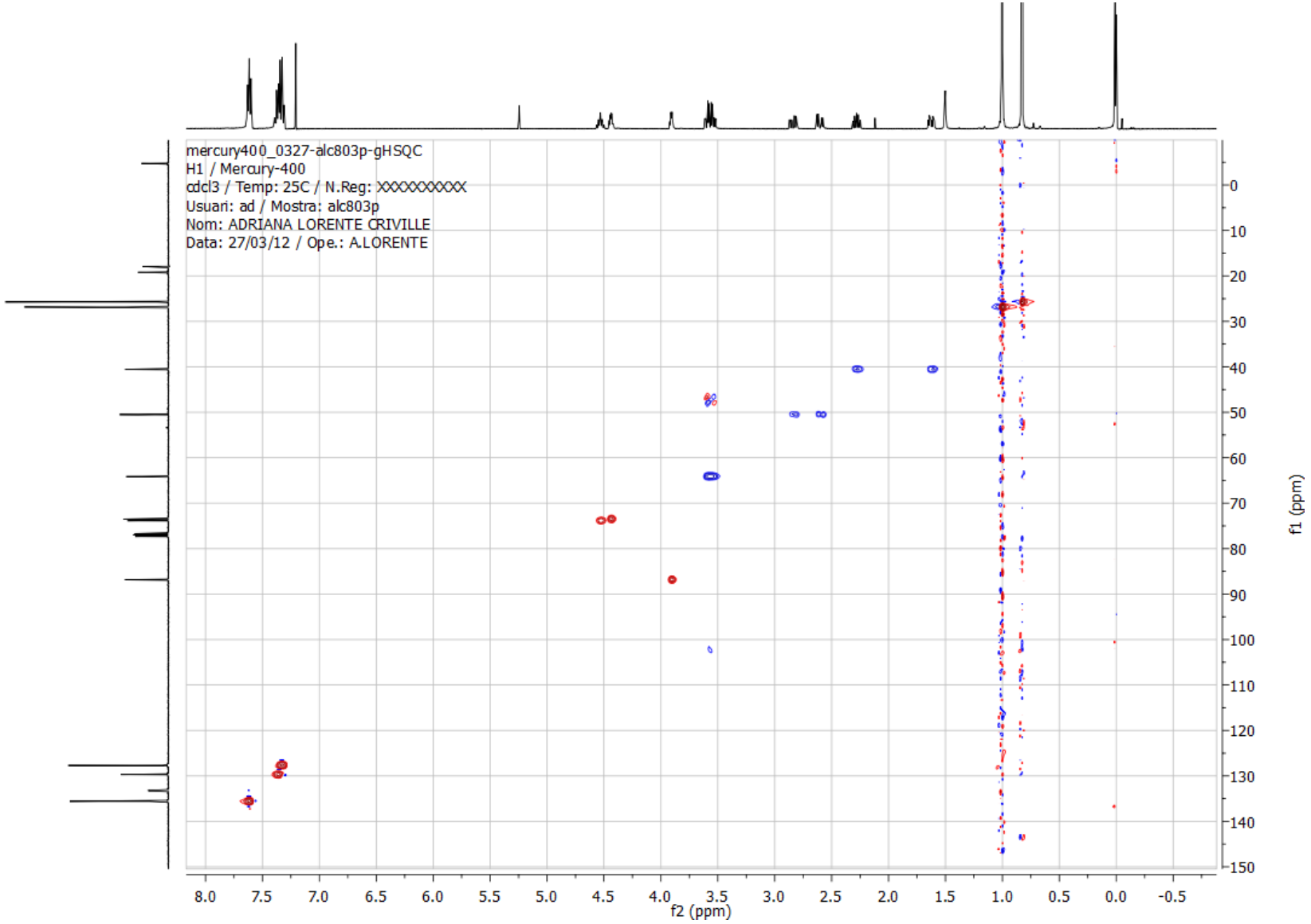

M400APCB_15052013_alc1044p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1044p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/13 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
TBSO
S3b
3.75
5.56
1.96
1.93
3.00
1.00
0.75
0.99
0.99
2.82
8.84
8.52
2.57
2.49
f1 (ppm)

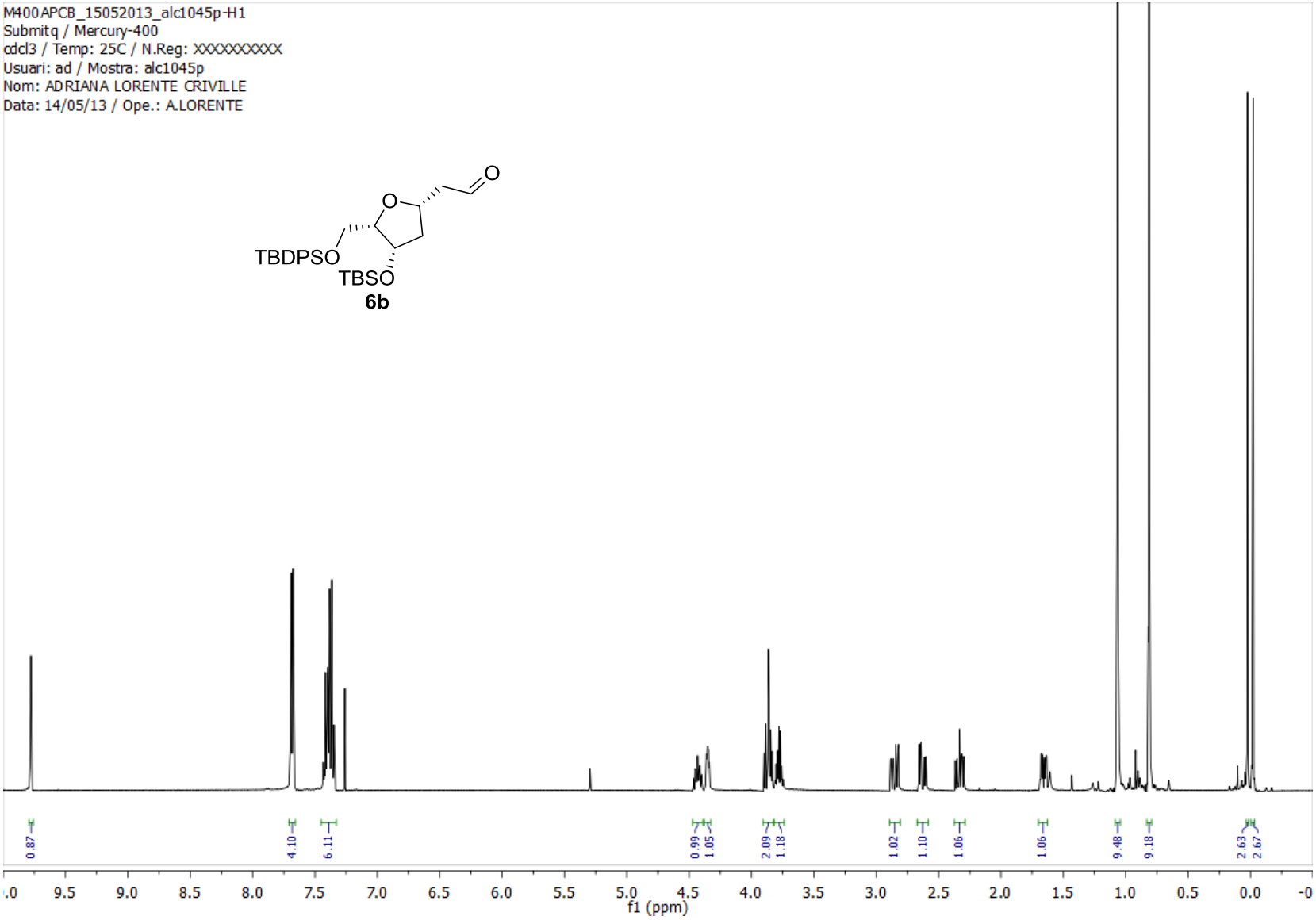

M400APCB_15052013_alc1044p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1044p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/13 / Ope.: A.LORENTE
O
CO2Et
TBDPSO
TBSO
S3b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

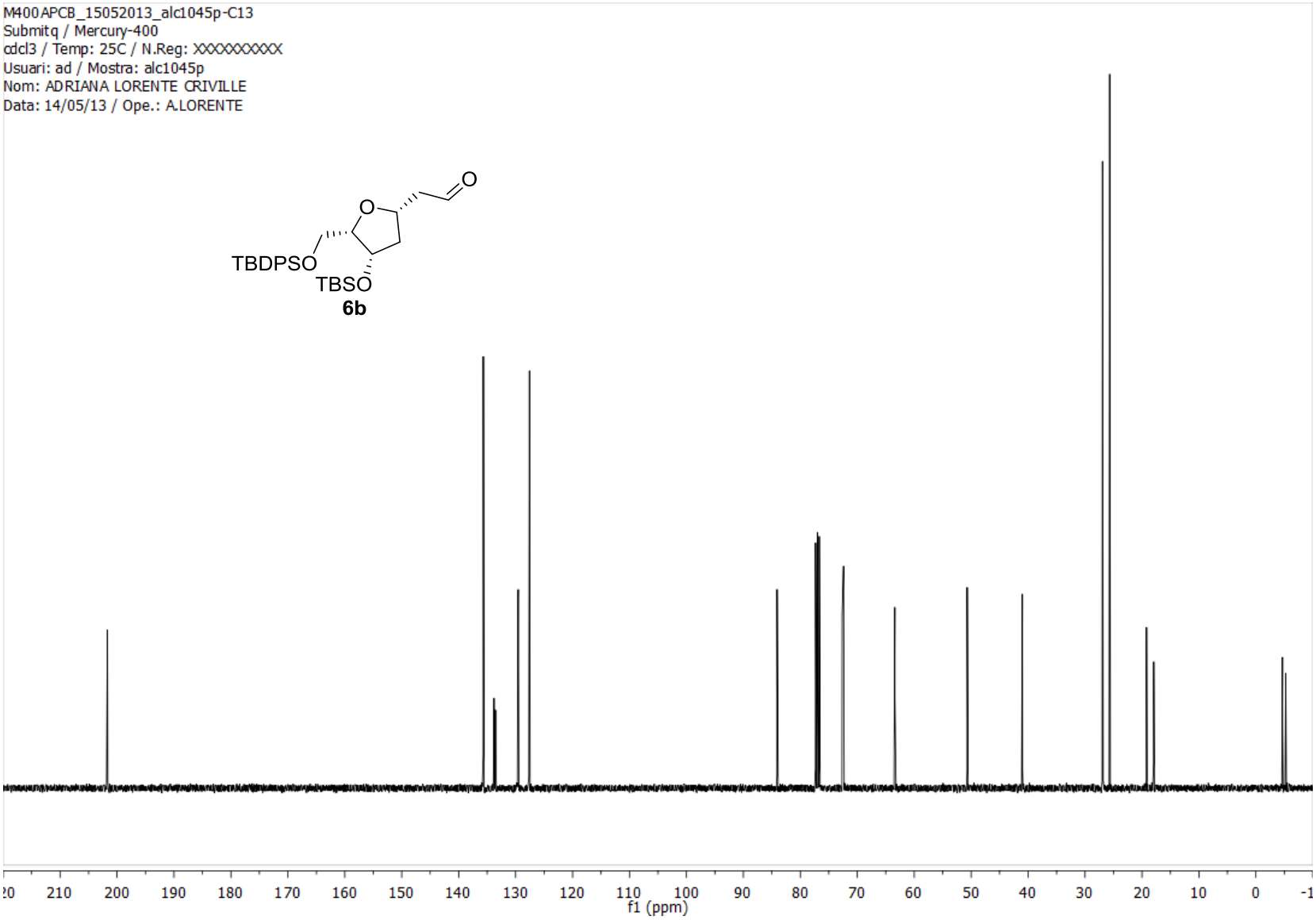

M400APCB_15052013_alc1044p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1044p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

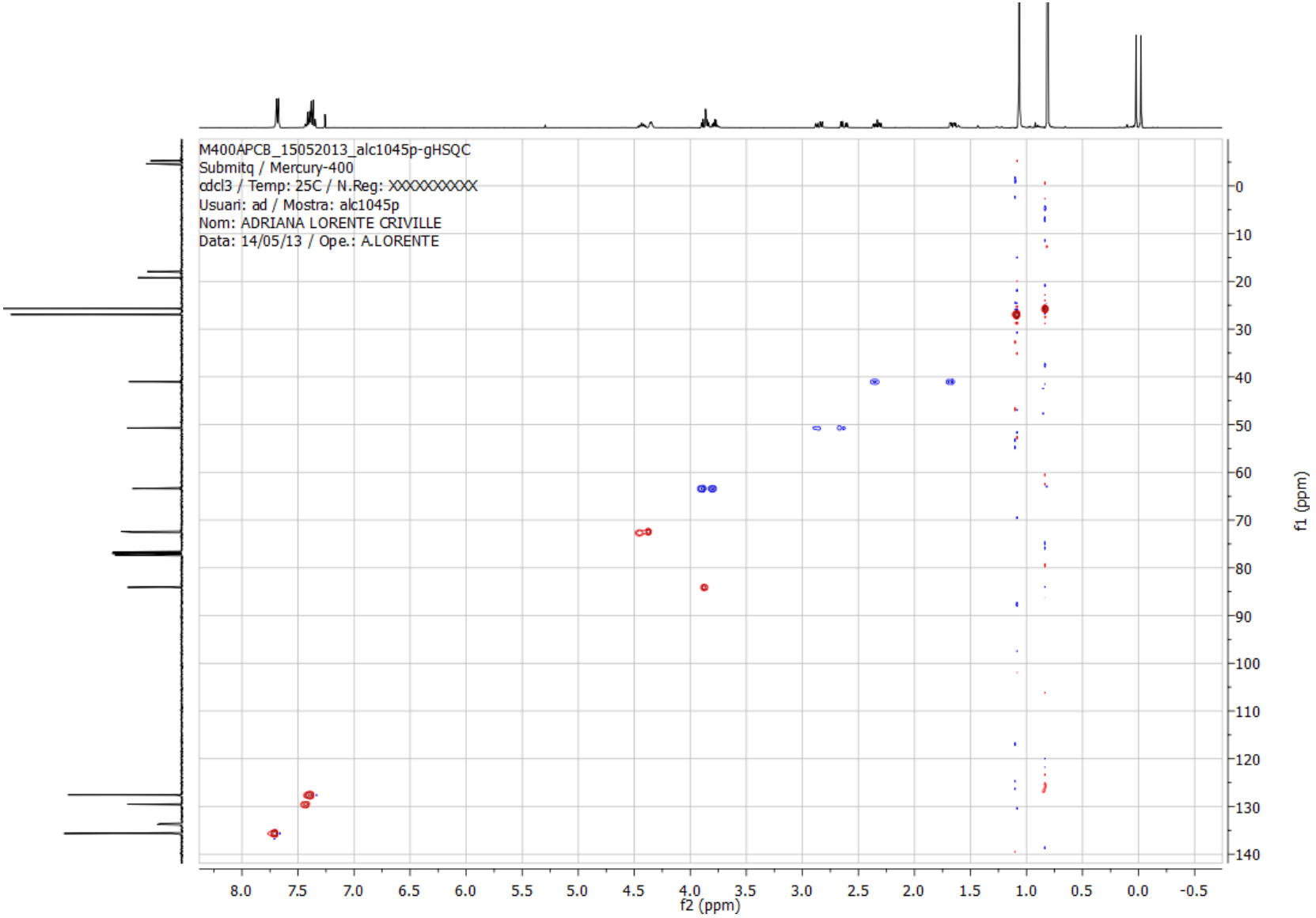

mercury400_0301-alc793p-H1
H1 / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc793p
Nom: ADRIANA LORENTE CRIVILLE
Data: 01/03/12 / Ope.: A.LORENTE
O
O
TBDPSO
TBSO
6a
1.00
4.65
6.82
1.02
1.08
1.05
2.29
1.02
1.10
1.01
1.06
10.69
10.42
6.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

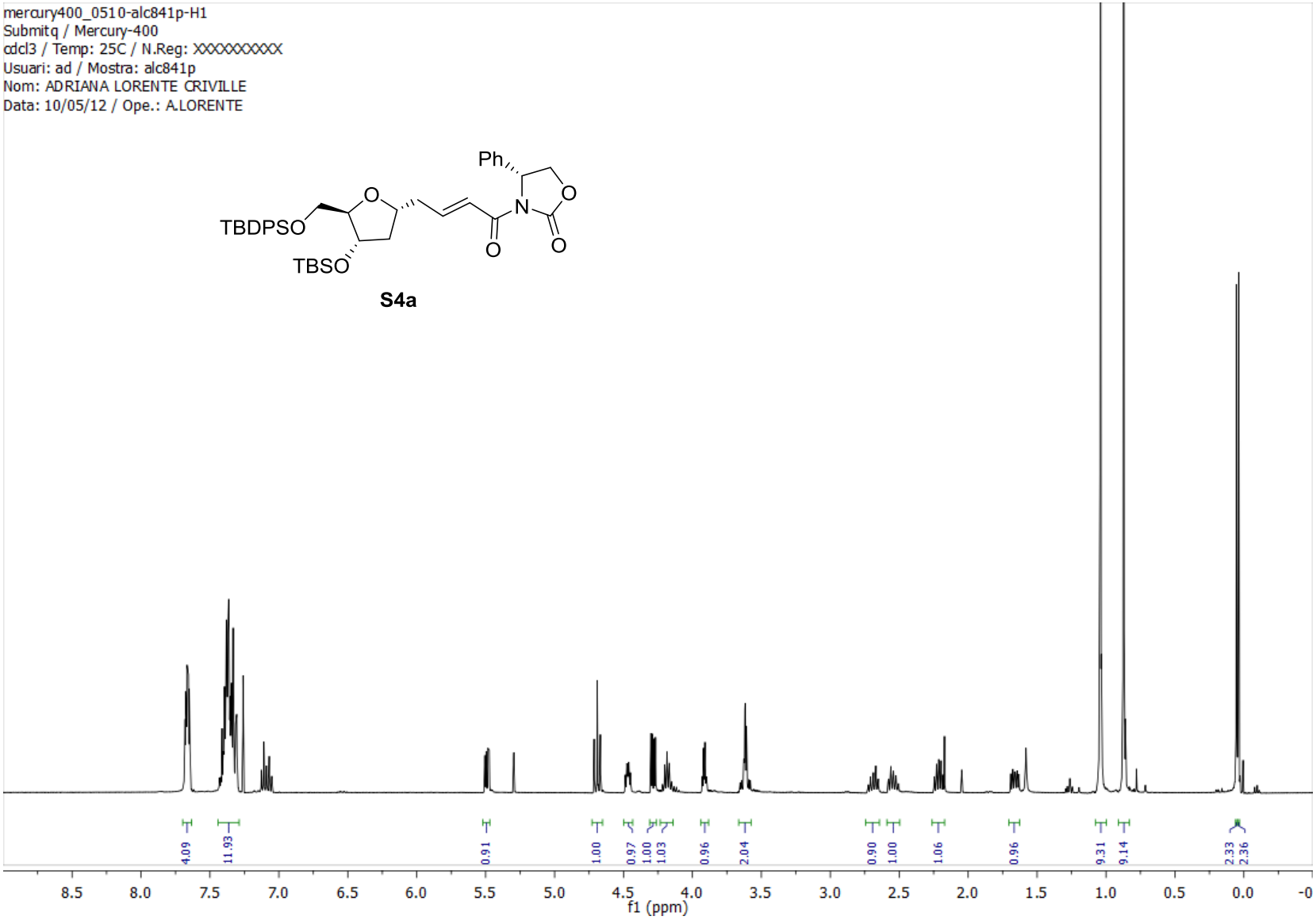

mercury400_0327-alc803p-C13
H1 / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: ad / Mostra: alc803p
Nom: ADRIANA LORENTE CRIVILLE
Data: 27/03/12 / Ope.: A.LORENTE
O
O
TBDPSO
TBSO
6a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


mercury400_0327-alc803p-gHSQC
H1 / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc803p
Nom: ADRIANA LORENTE CRIVILLE
Data: 27/03/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

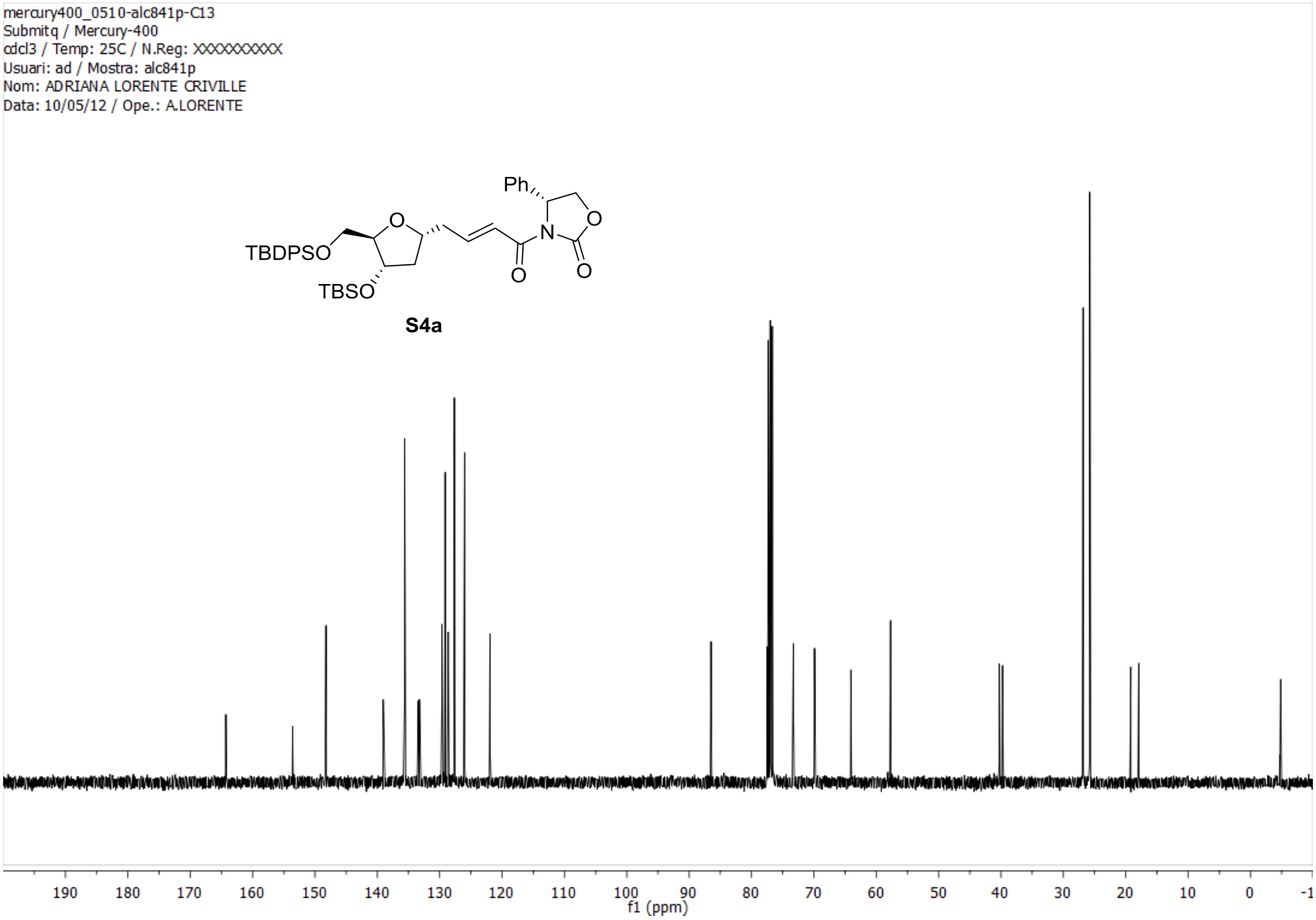

M400APCB_15052013_alc1045p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1045p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/13 / Ope.: A.LORENTE
O
O
TBDPSO
TBSO
6b
0.87
4.10
6.11
0.99
1.05
2.09
1.18
1.02
1.10
1.06
1.06
9.48
9.18
2.63
2.67
f1 (ppm)

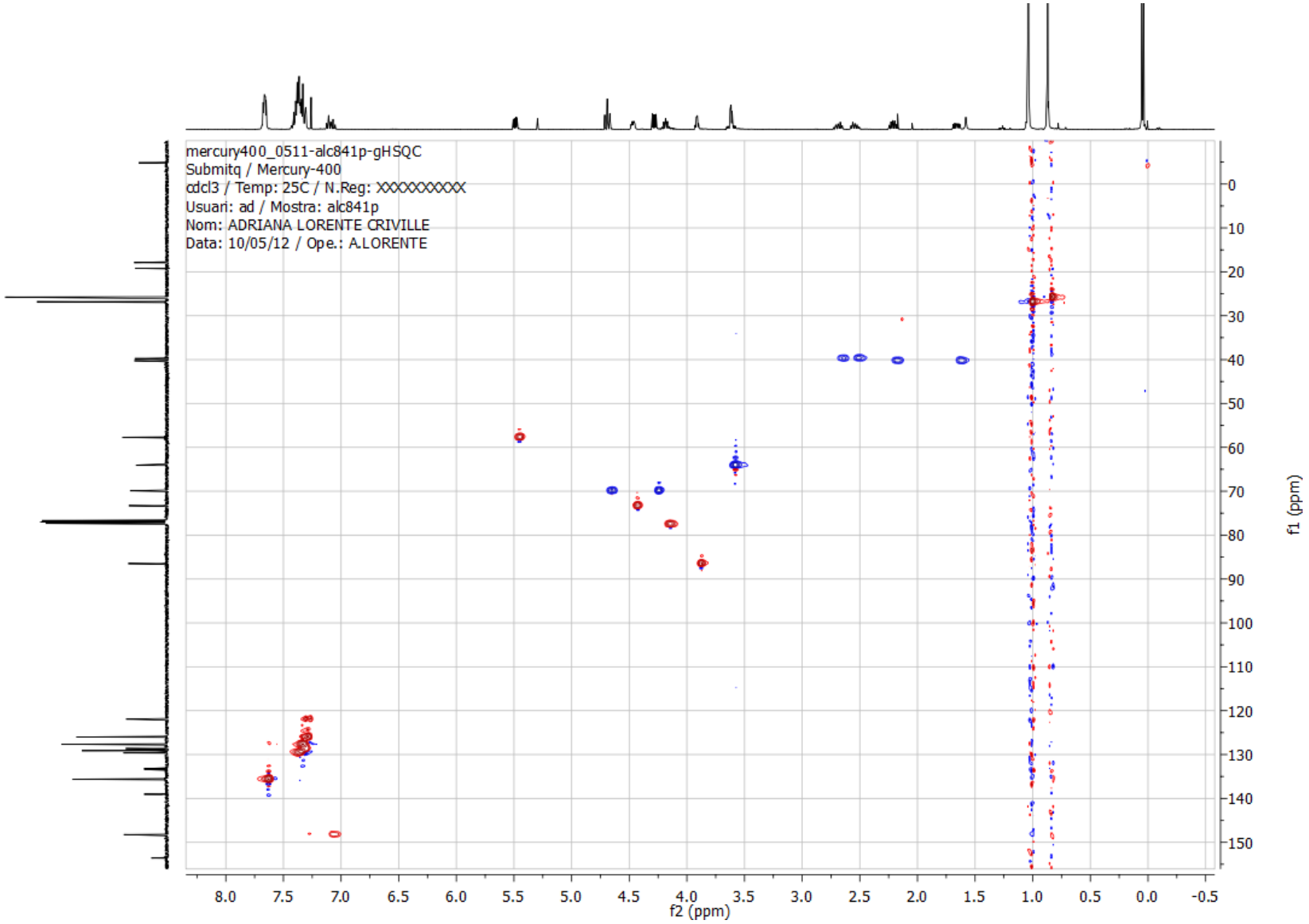

M400APCB_15052013_alc1045p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1045p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/13 / Ope.: A.LORENTE
O
O
TBDPSO
TBSO
6b
f1 (ppm)

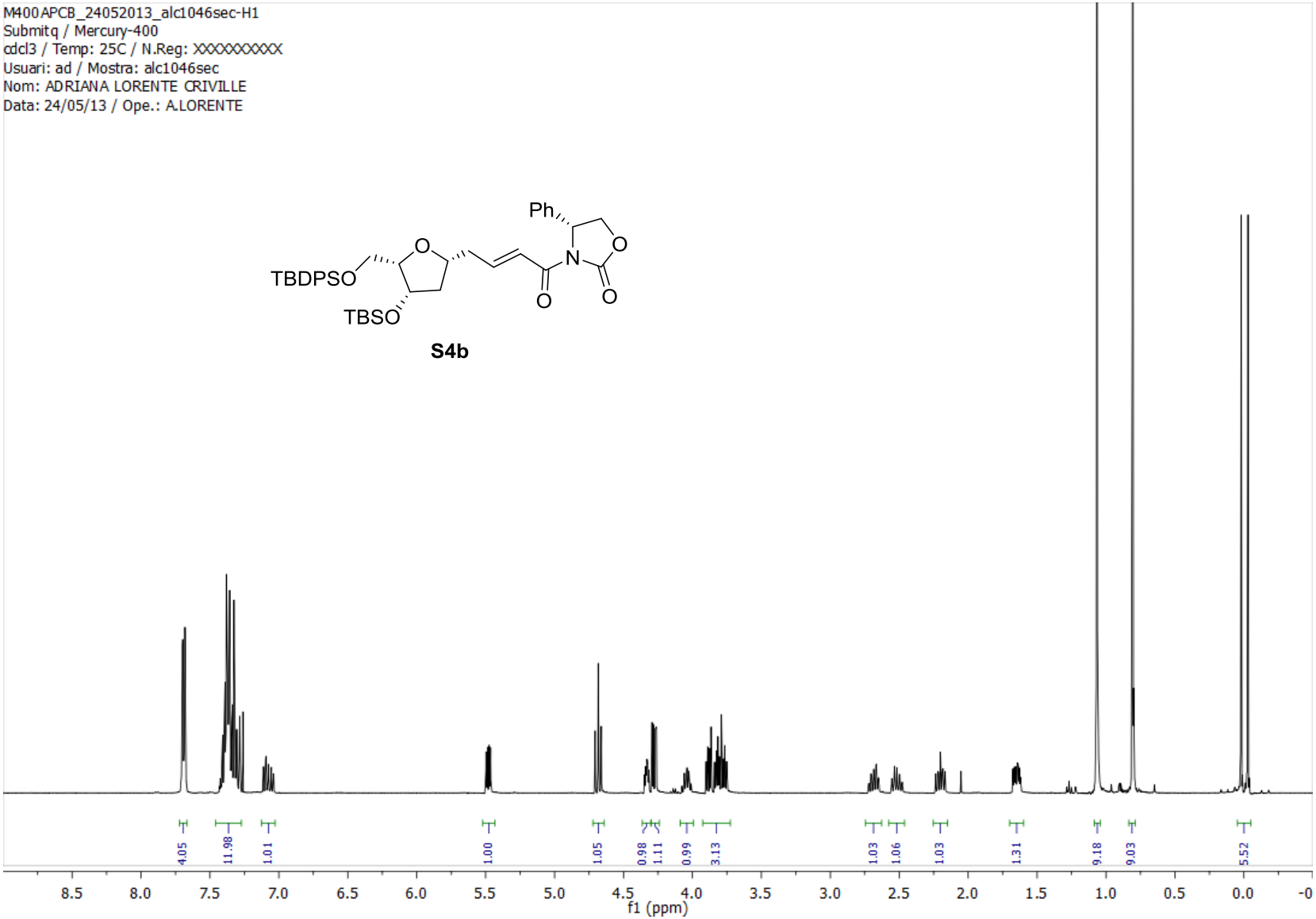

M400APCB_15052013_alc1045p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1045p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

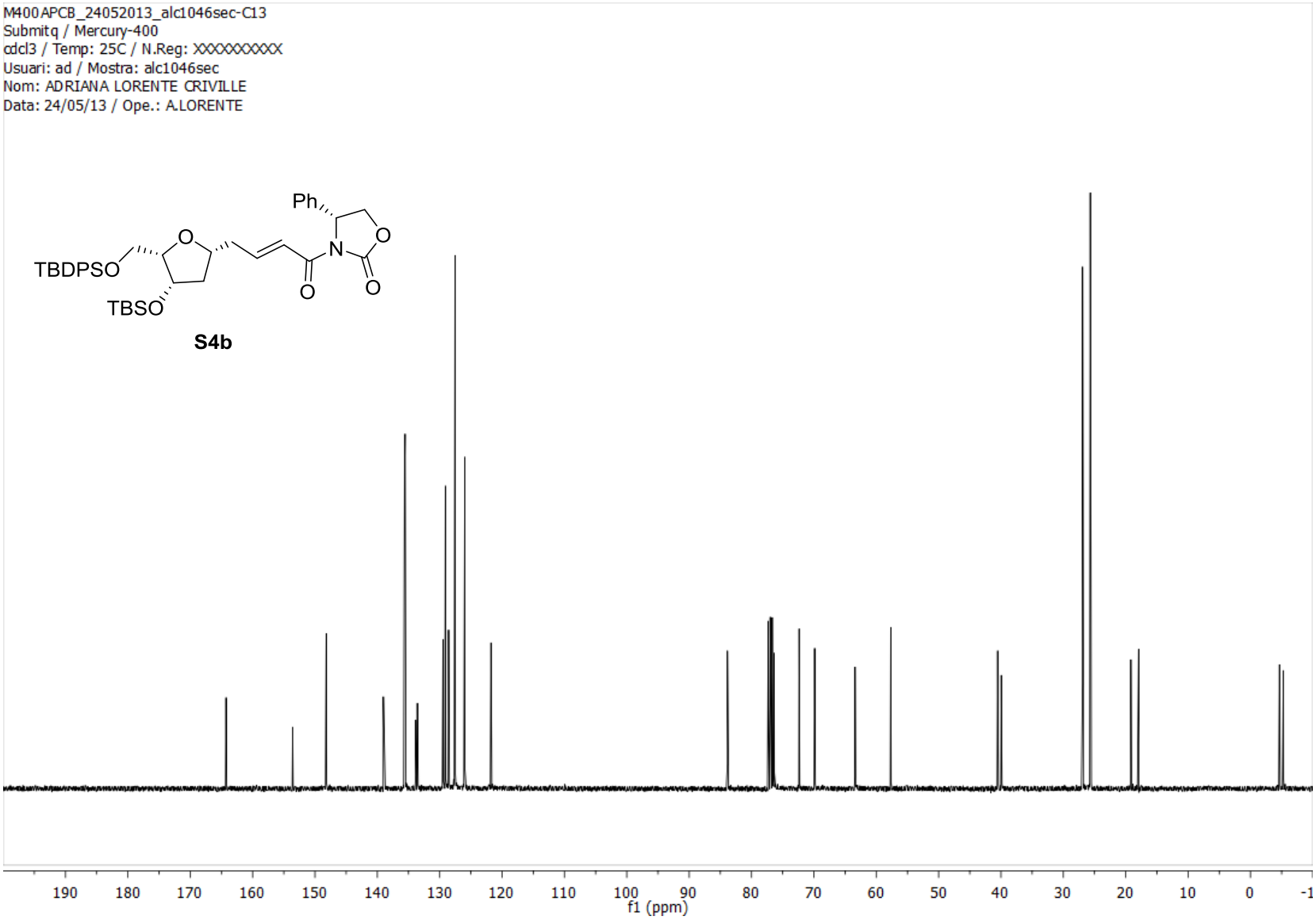

mercury400_0510-alc841p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc841p
Nom: ADRIANA LORENTE CRIVILLE
Data: 10/05/12 / Ope.: A.LORENTE
Ph
O
N
O
O
O
TBDPSO
TBSO
4.09
11.93
0.91
1.00
0.97
1.00
1.03
0.96
2.04
0.90
1.00
1.06
0.96
9.31
9.14
2.33
2.36
8.5
8.0
7.5
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
-0
f1 (ppm)

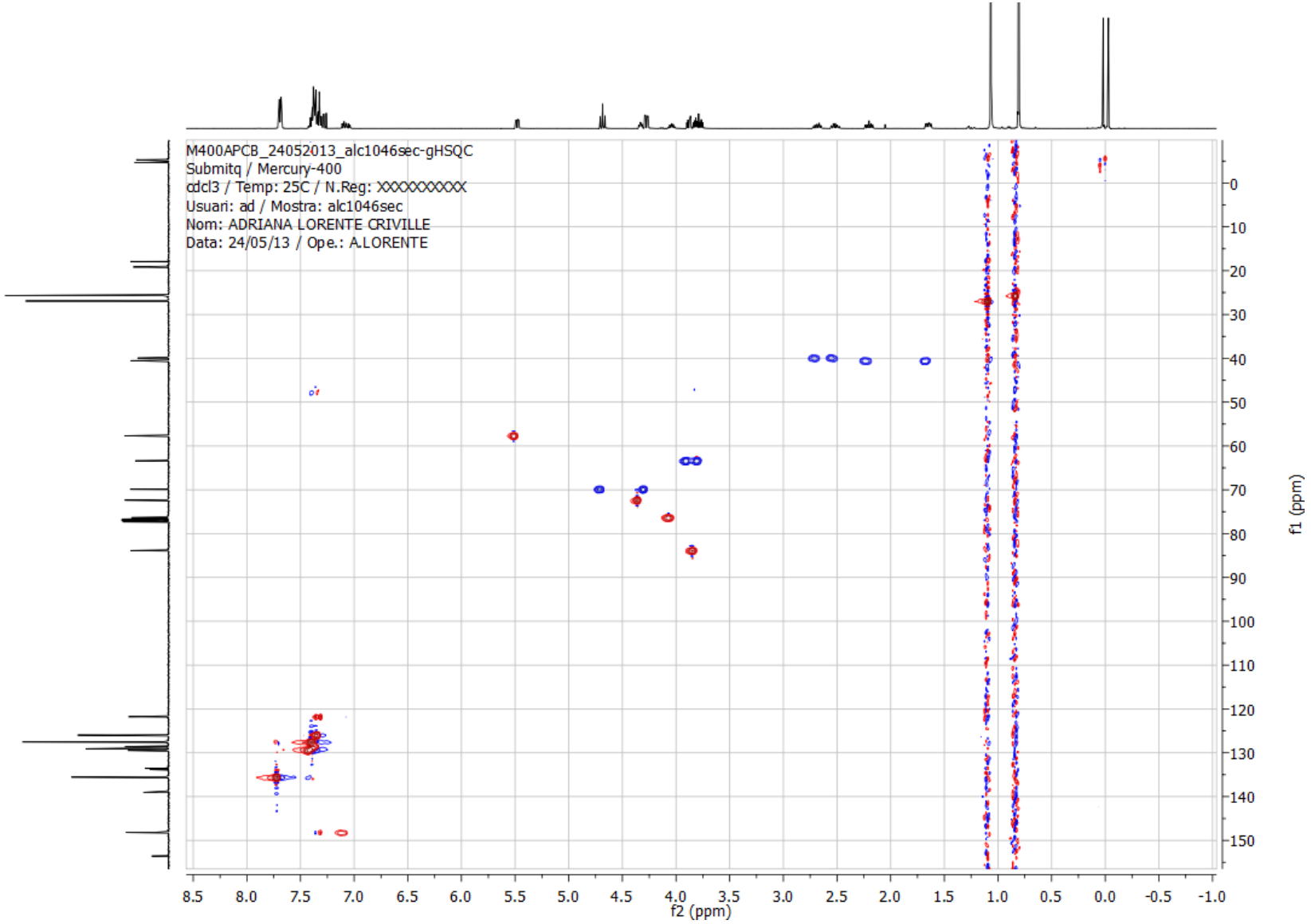
S4a

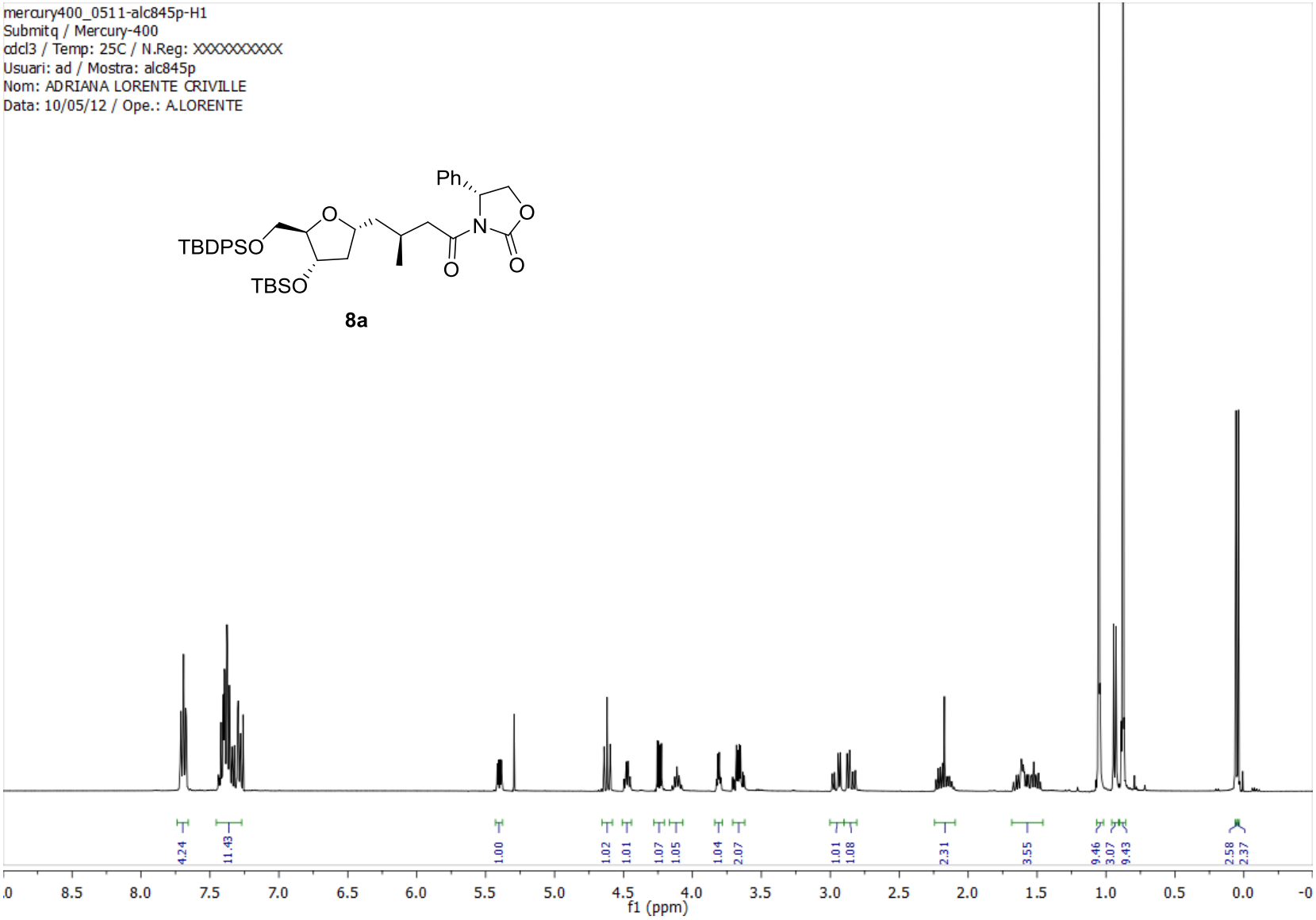

mercury400_0510-alc841p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc841p
Nom: ADRIANA LORENTE CRIVILLE
Data: 10/05/12 / Ope.: A.LORENTE
Ph
O
N
O
TBDPSO
TBSO
O
O
S4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)


mercury400_0511-alc841p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc841p
Nom: ADRIANA LORENTE CRIVILLE
Data: 10/05/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

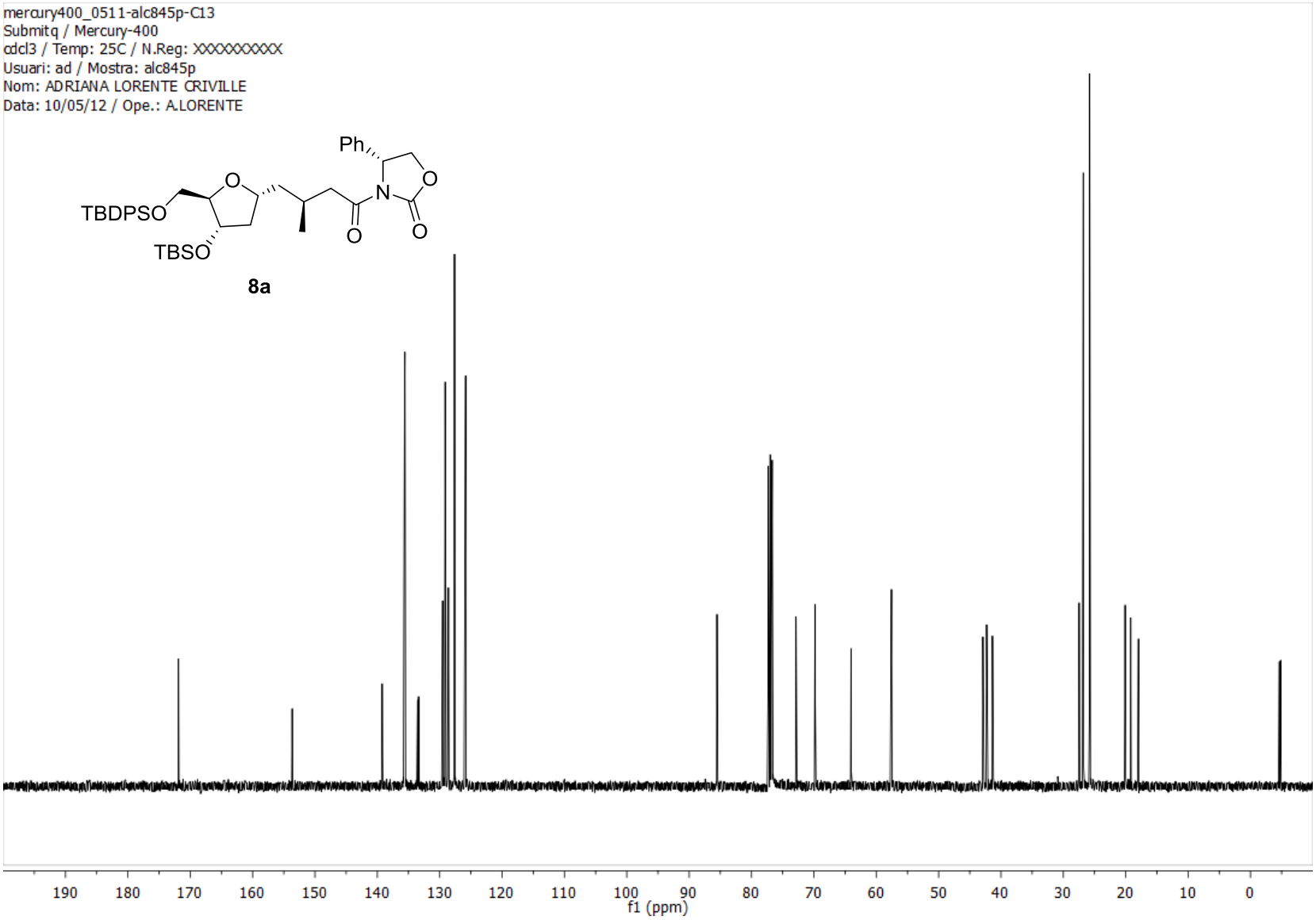

M400APCB_24052013_alc1046sec-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1046sec
Nom: ADRIANA LORENTE CRIVILLE
Data: 24/05/13 / Ope.: A.LORENTE
Ph
O
N
O
O
O
TBDPSO
TBSO
S4b
4.05
11.98
1.01
1.00
1.05
0.98
1.11
0.99
3.13
1.03
1.06
1.03
1.31
9.18
9.03
5.52
8.5
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
-0
f1 (ppm)

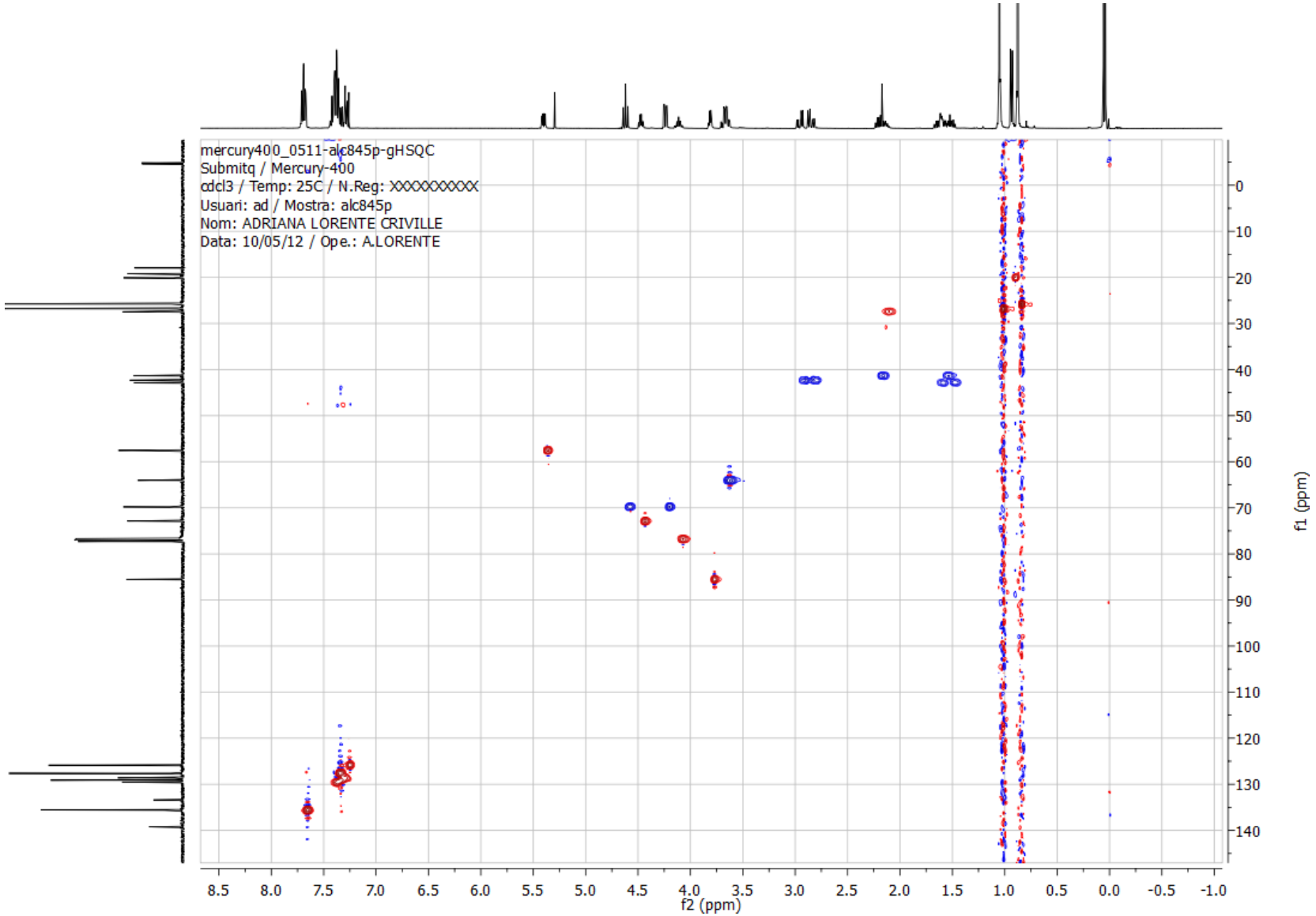

M400APCB_24052013_alc1046sec-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1046sec
Nom: ADRIANA LORENTE CRIVILLE
Data: 24/05/13 / Ope.: A.LORENTE
Ph
TBDPSO
TBSO
O
N
O
O
O
S4b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

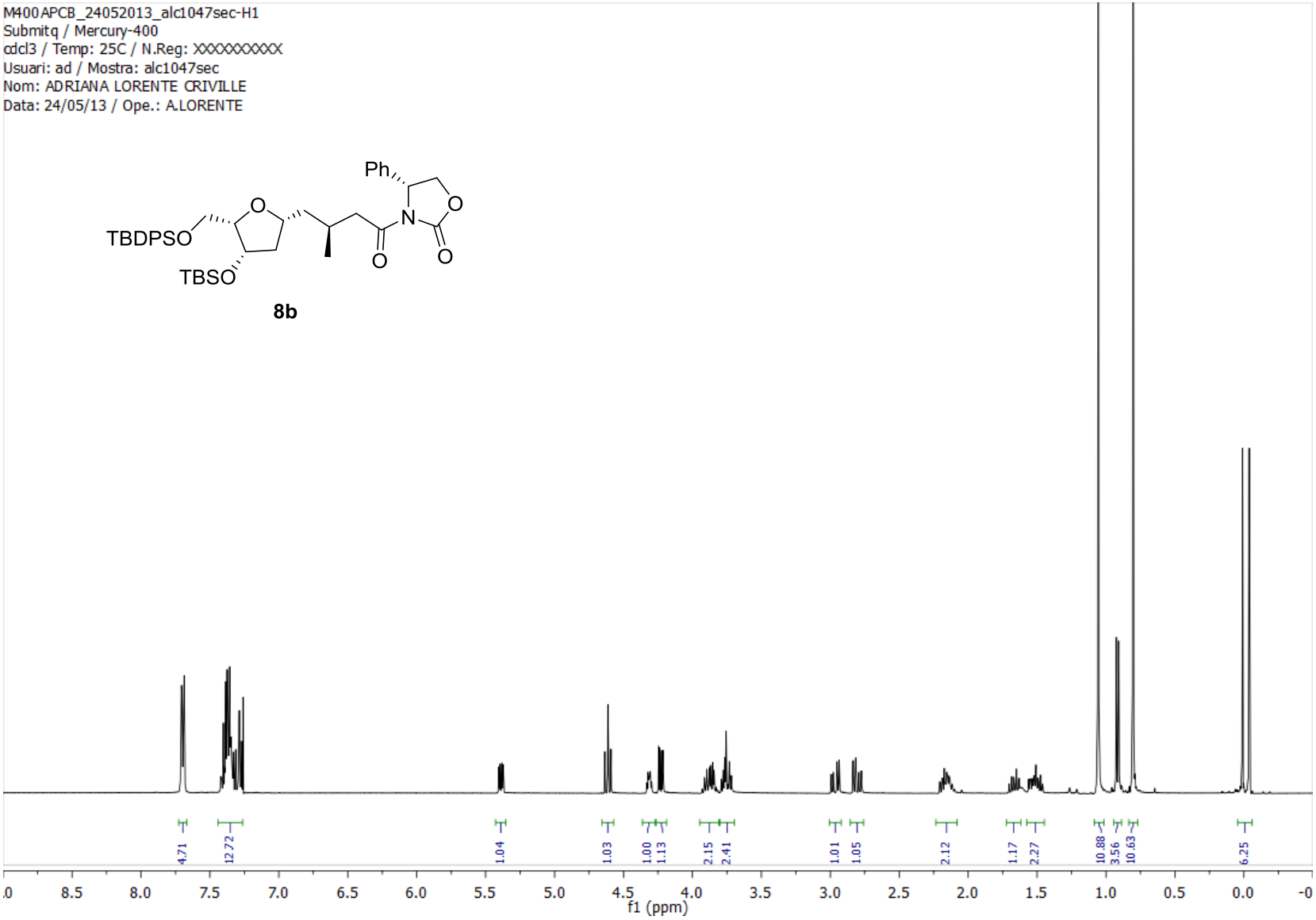

M400APCB_24052013_alc1046sec-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1046sec
Nom: ADRIANA LORENTE CRIVILLE
Data: 24/05/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)


mercury400_0511-alc845p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc845p
Nom: ADRIANA LORENTE CRIVILLE
Data: 10/05/12 / Ope.: A.LORENTE
Ph
TBDPSO
TBSO
O
N
O
O
O
4.24
11.43
1.00
1.02
1.01
1.07
1.05
1.04
2.07
1.01
1.08
2.31
3.55
9.46
3.07
9.43
2.58
2.37
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)

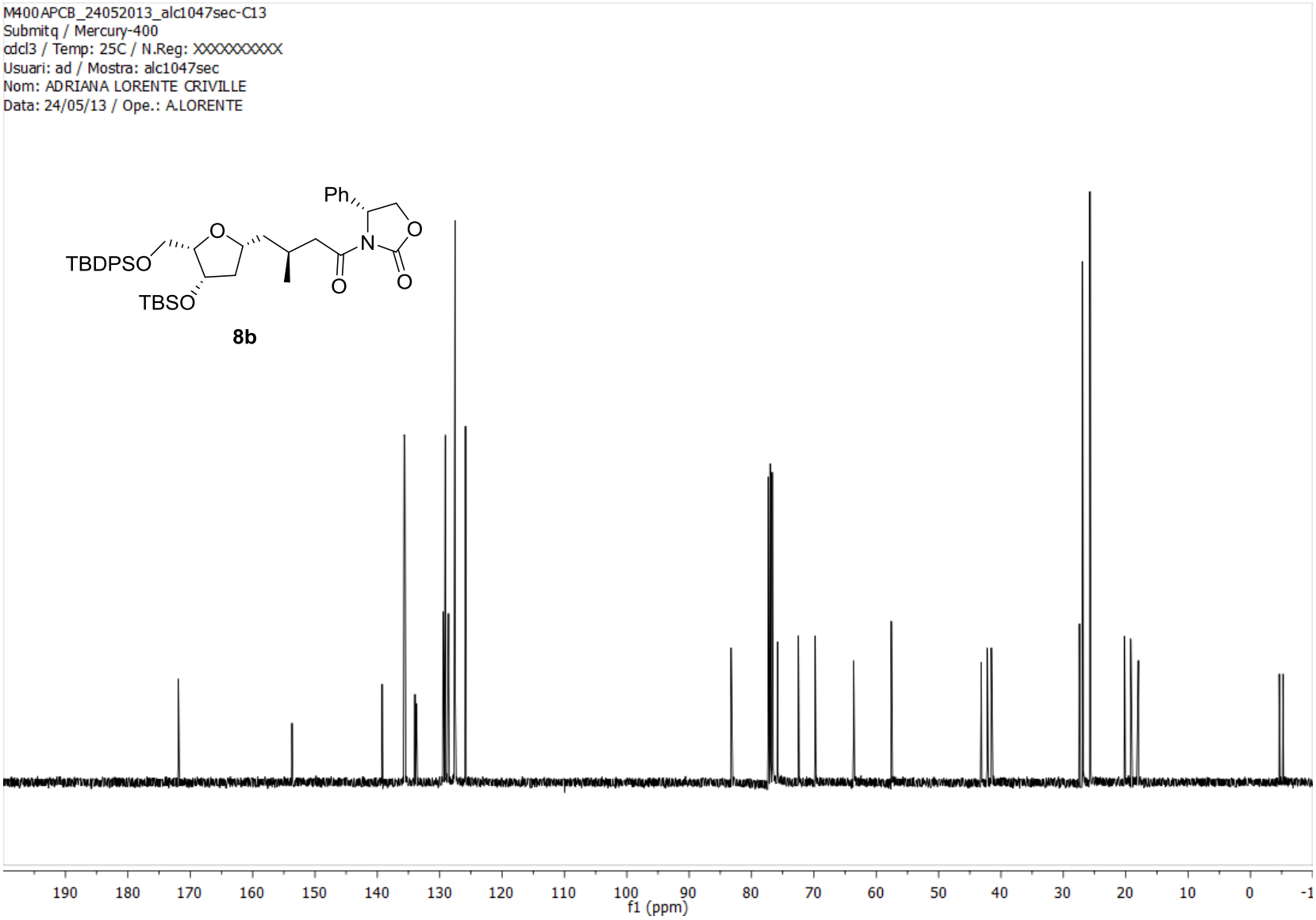
8a

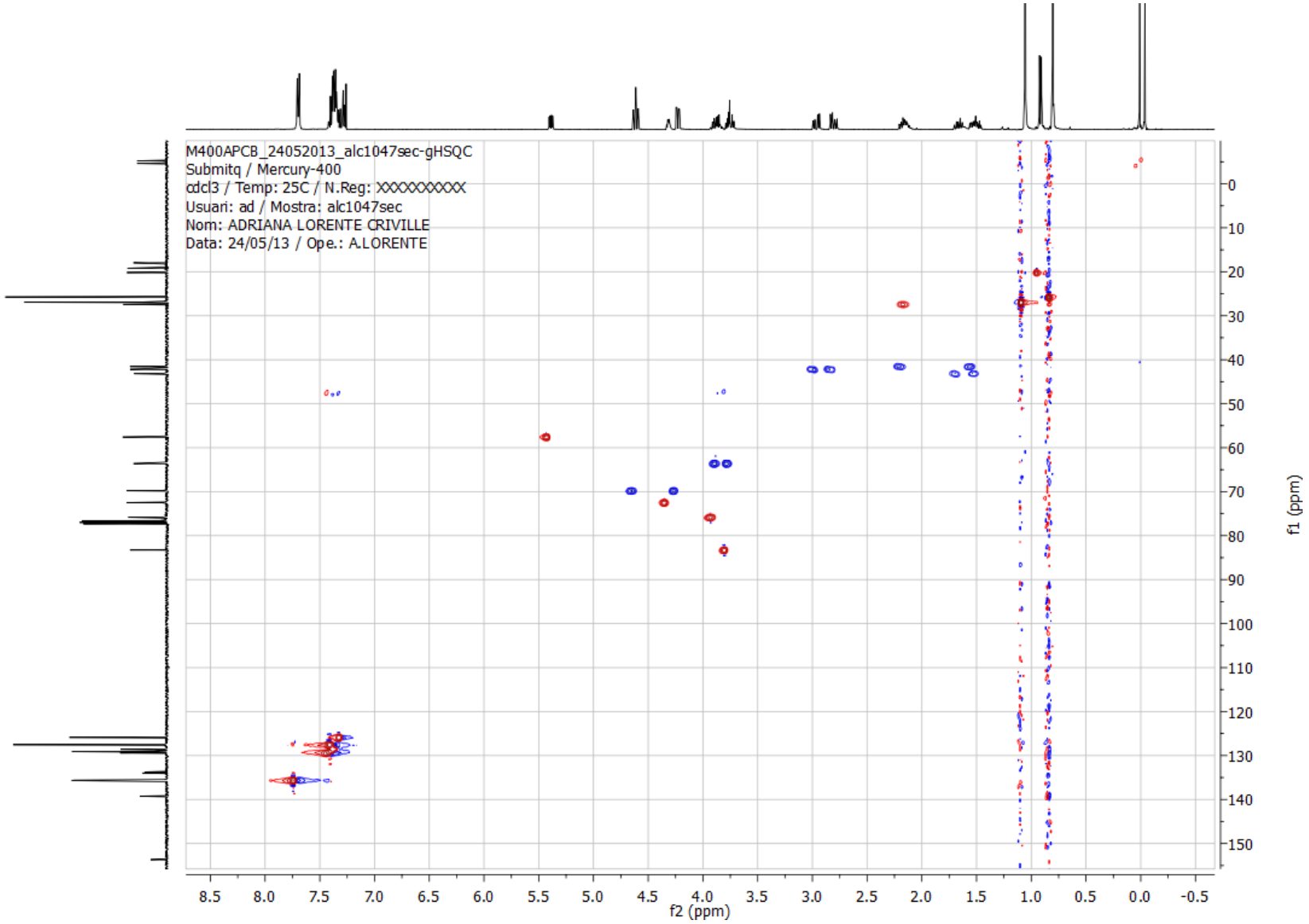

mercury400_0511-alc845p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc845p
Nom: ADRIANA LORENTE CRIVILLE
Data: 10/05/12 / Ope.: A.LORENTE
Ph
O
O
N
O
O
TBDPSO
TBSO
8a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

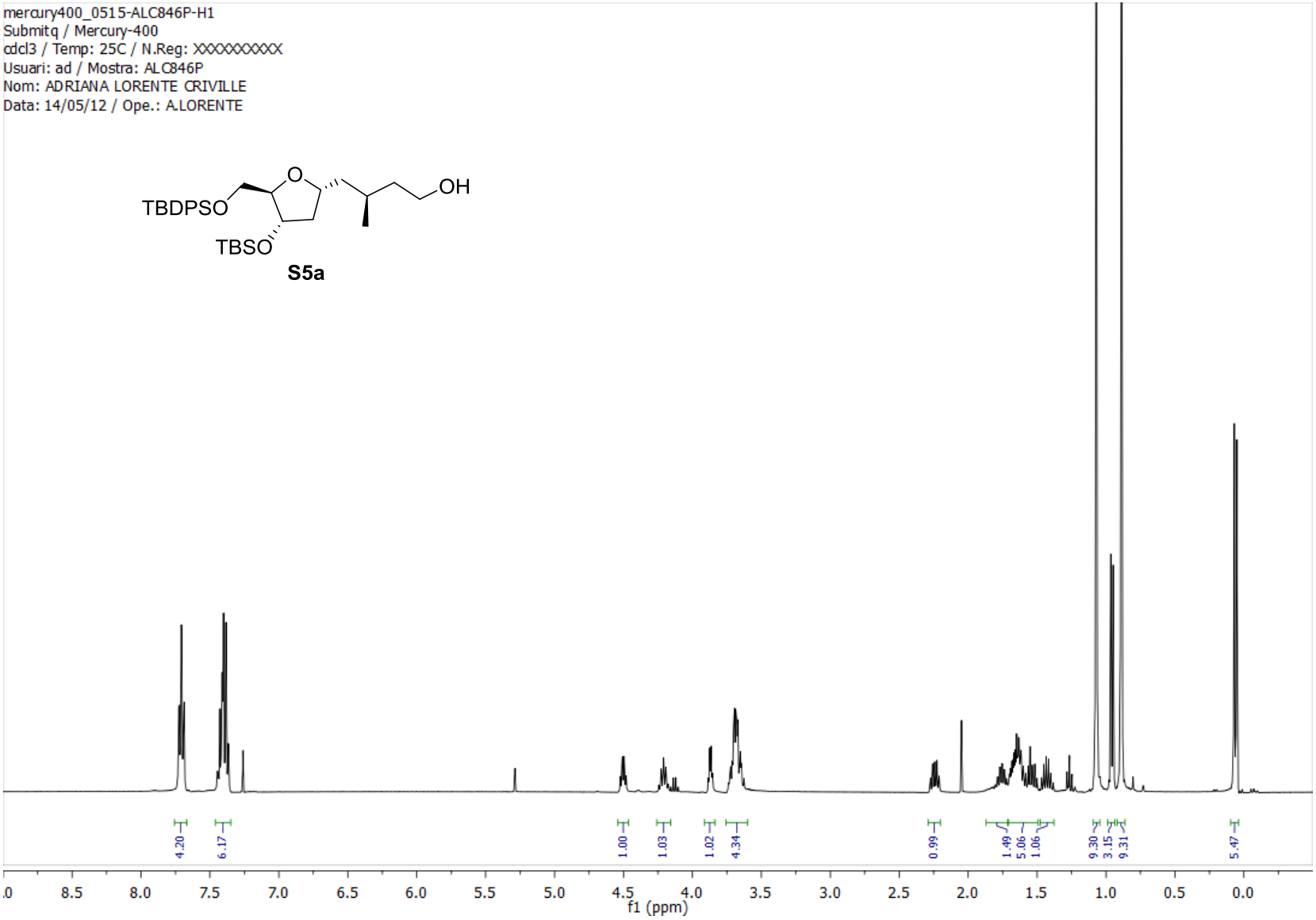

mercury400_0511-alc845p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc845p
Nom: ADRIANA LORENTE CRIVILLE
Data: 10/05/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

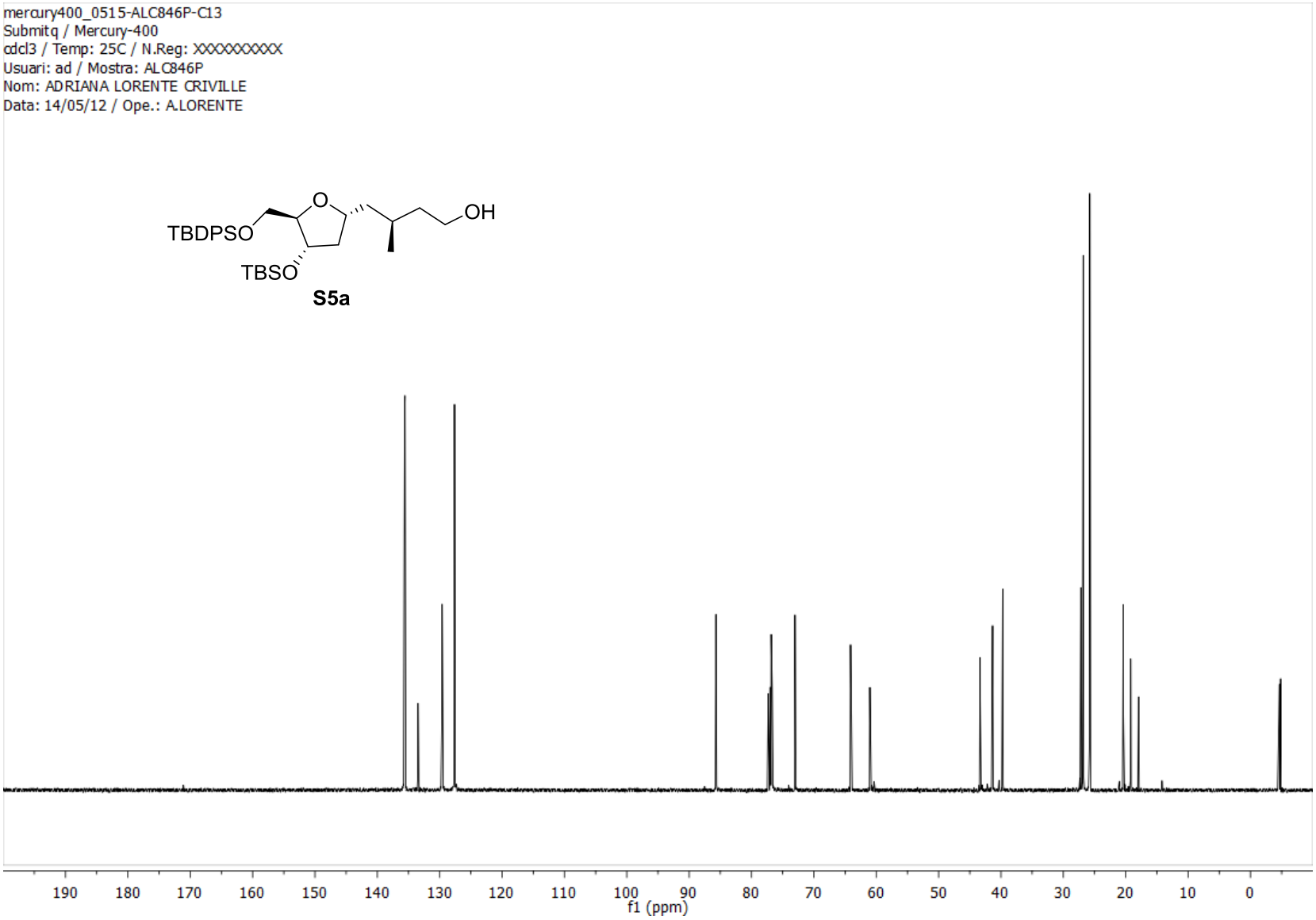

M400APCB_24052013_alc1047sec-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1047sec
Nom: ADRIANA LORENTE CRIVILLE
Data: 24/05/13 / Ope.: A.LORENTE
Ph
O
O
N
O
O
TBDPSO
TBSO
4.71
12.72
1.04
1.03
1.00
1.13
2.15
2.41
1.01
1.05
2.12
1.17
2.27
10.88
3.56
10.63
6.25
8.5
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)

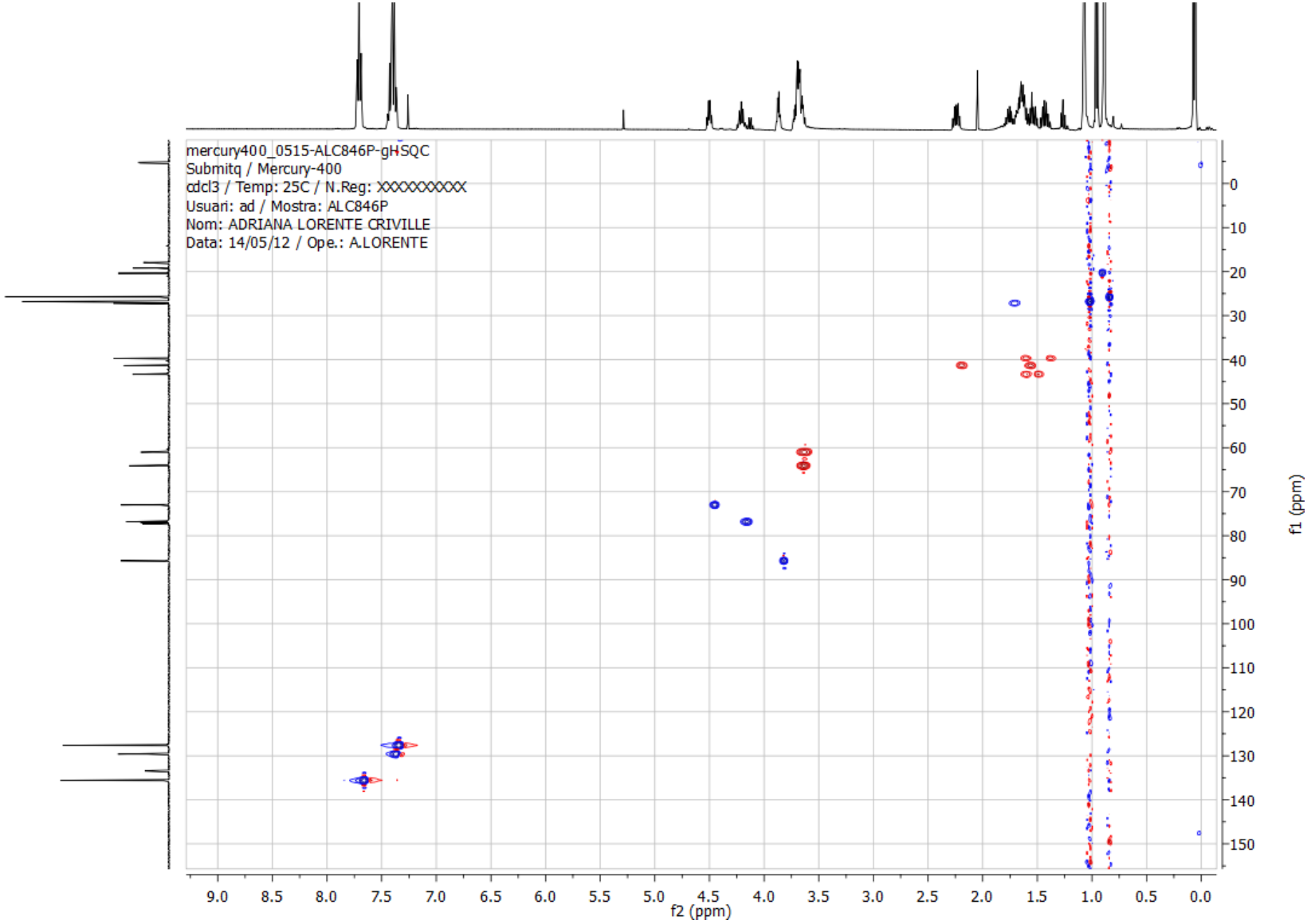
8b

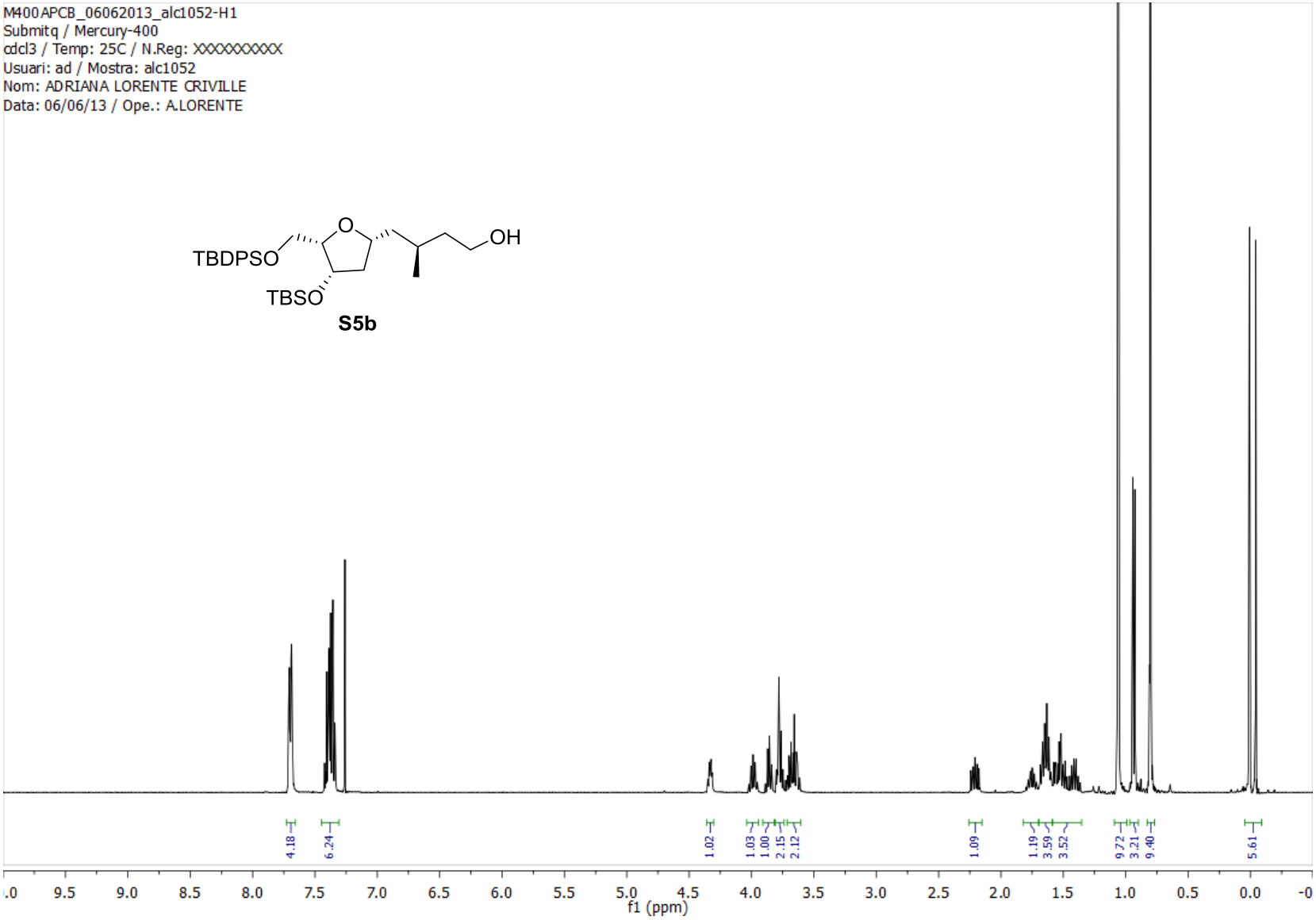

M400APCB_24052013_alc1047sec-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: ad / Mostra: alc1047sec
Nom: ADRIANA LORENTE CRIVILLE
Data: 24/05/13 / Ope.: A.LORENTE
Ph
O
N
O
O
O
TBDPSO
TBSO
8b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

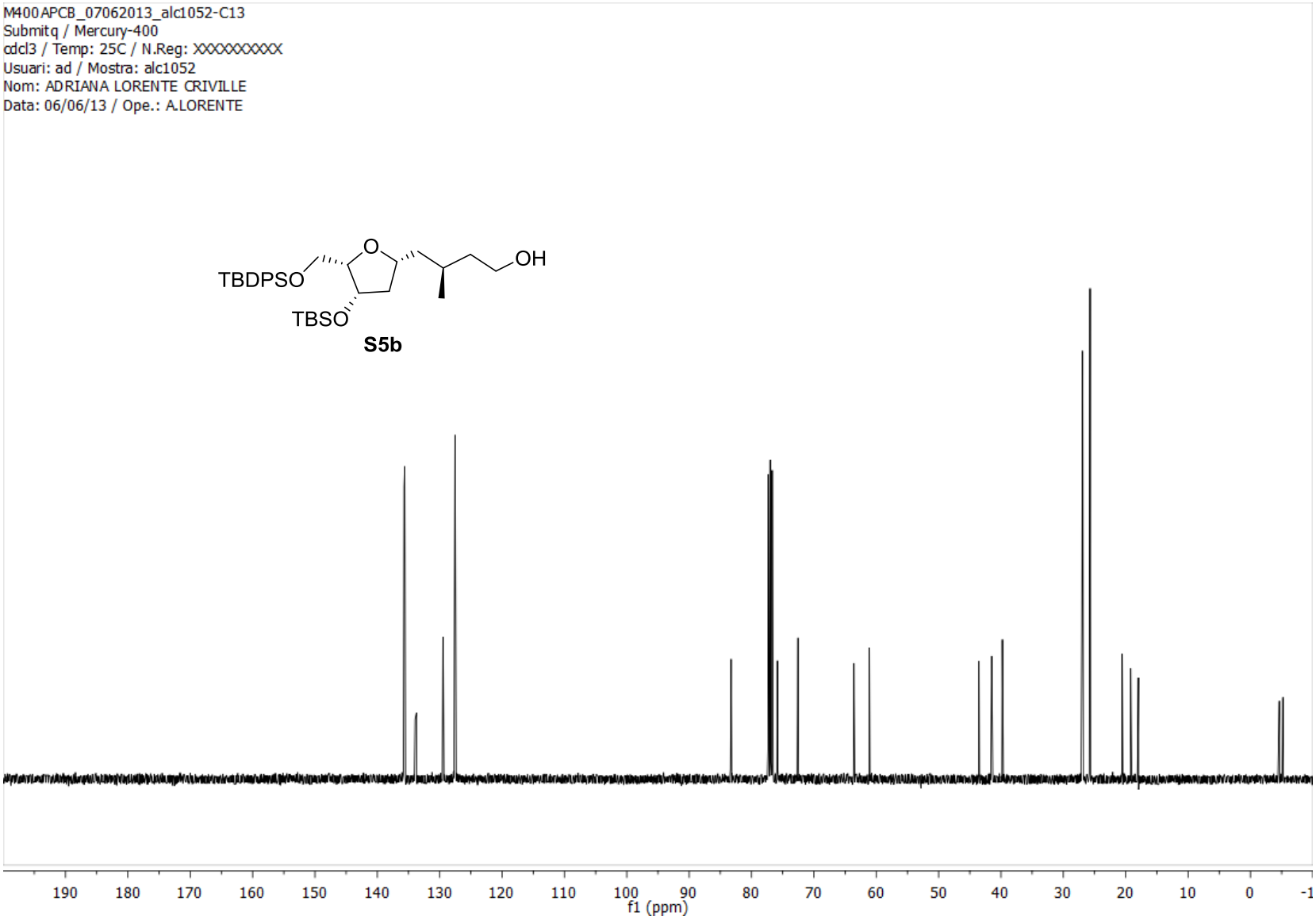

M400APCB_24052013_alc1047sec-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1047sec
Nom: ADRIANA LORENTE CRIVILLE
Data: 24/05/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

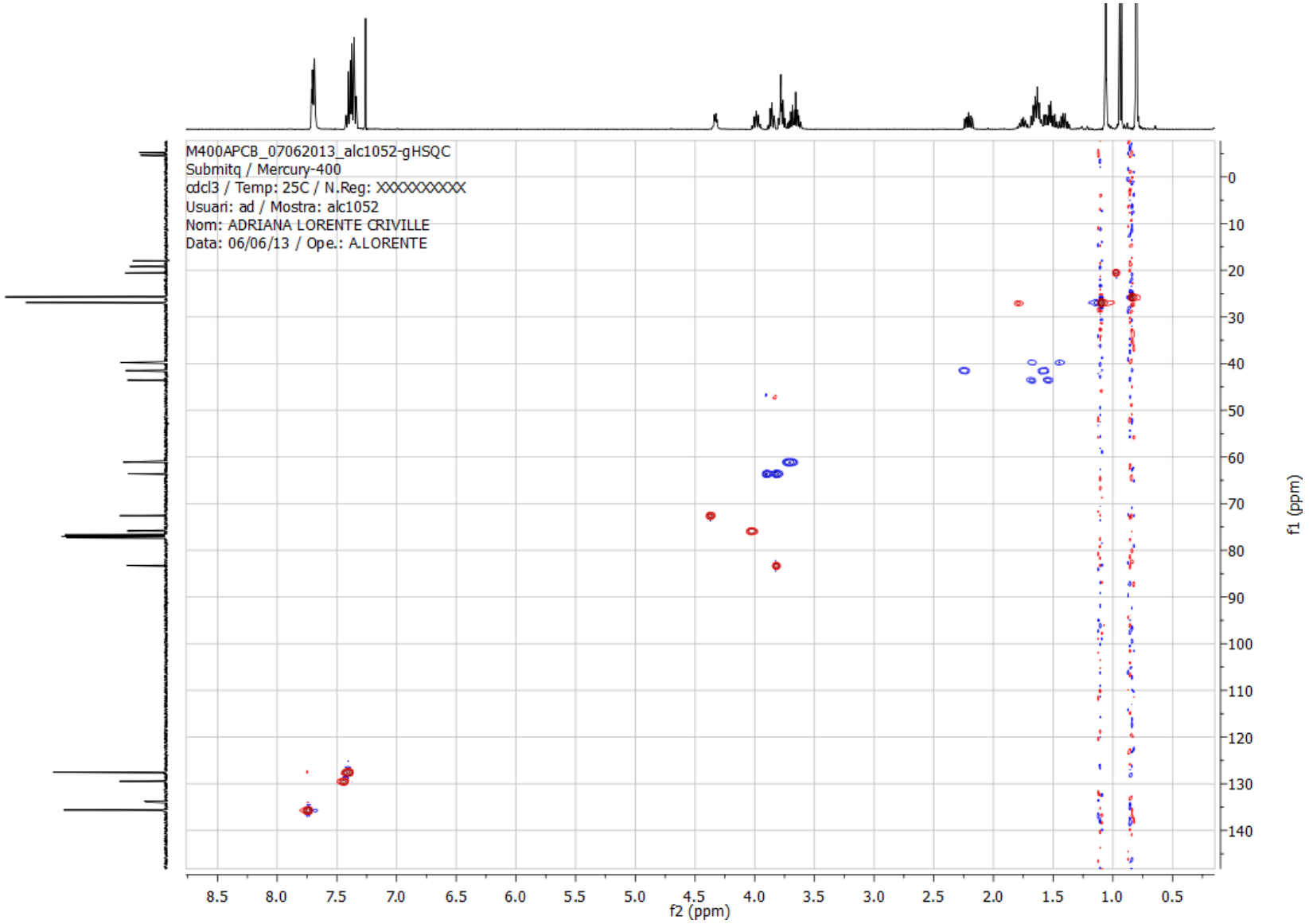

mercury400_0515-ALC846P-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC846P
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/12 / Ope.: A.LORENTE
TBDPSO
O
OH
TBSO
S5a
4.20
6.17
1.00
1.03
1.02
4.34
0.99
1.49
5.06
1.06
9.30
3.15
9.31
5.47
f1 (ppm)

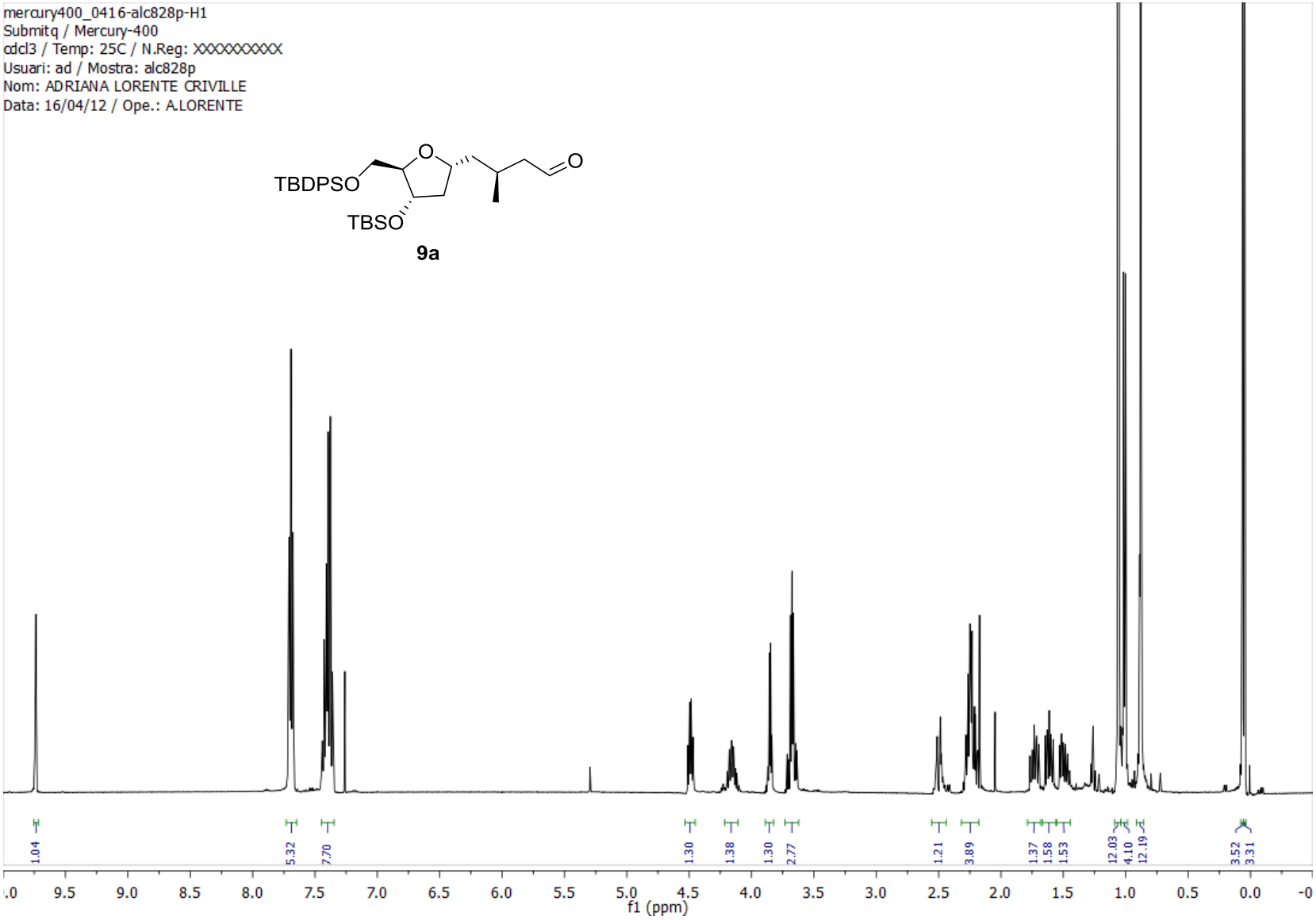

mercury400_0515-ALC846P-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC846P
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/12 / Ope.: A.LORENTE
TBDPSO
O
OH
TBSO
S5a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

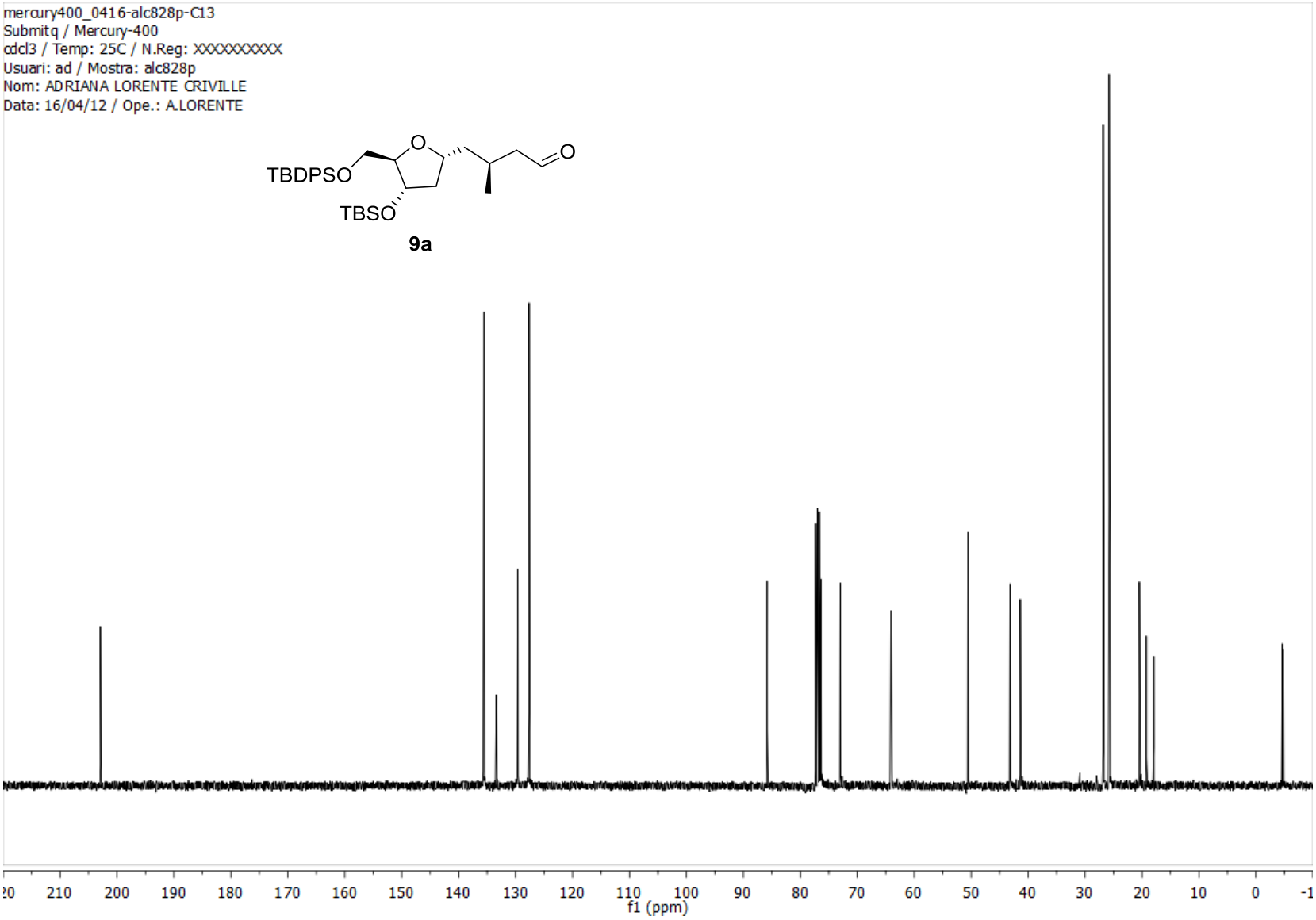

mercury400_0515-ALC846P-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC846P
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/05/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

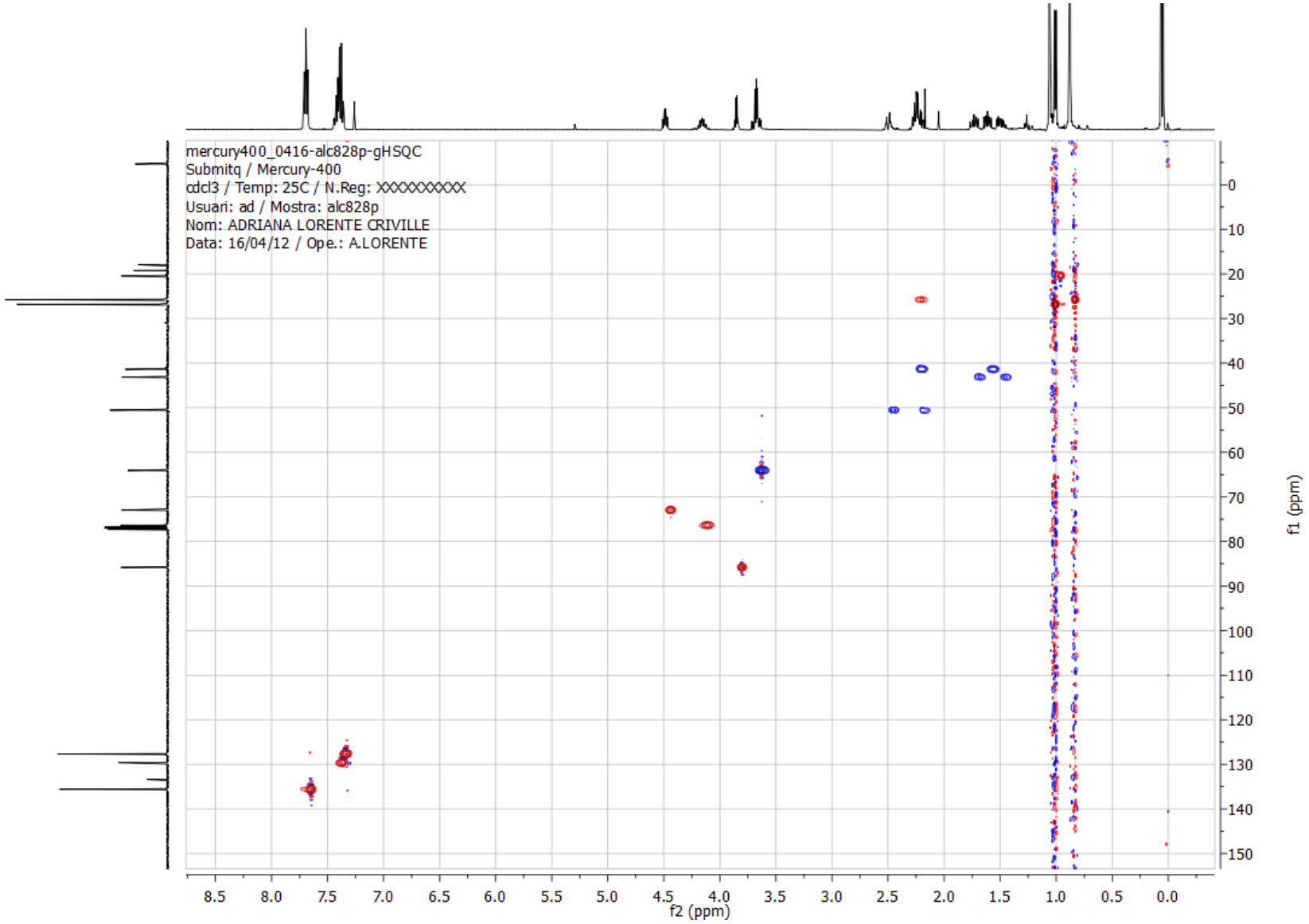

M400APCB_06062013_alc1052-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1052
Nom: ADRIANA LORENTE CRIVILLE
Data: 06/06/13 / Ope.: A.LORENTE
TBDPSO
O
OH
TBSO
S5b
4.18
6.24
1.02
1.03
1.00
2.15
2.12
1.09
1.19
3.59
3.52
9.72
3.21
9.40
5.61
f1 (ppm)

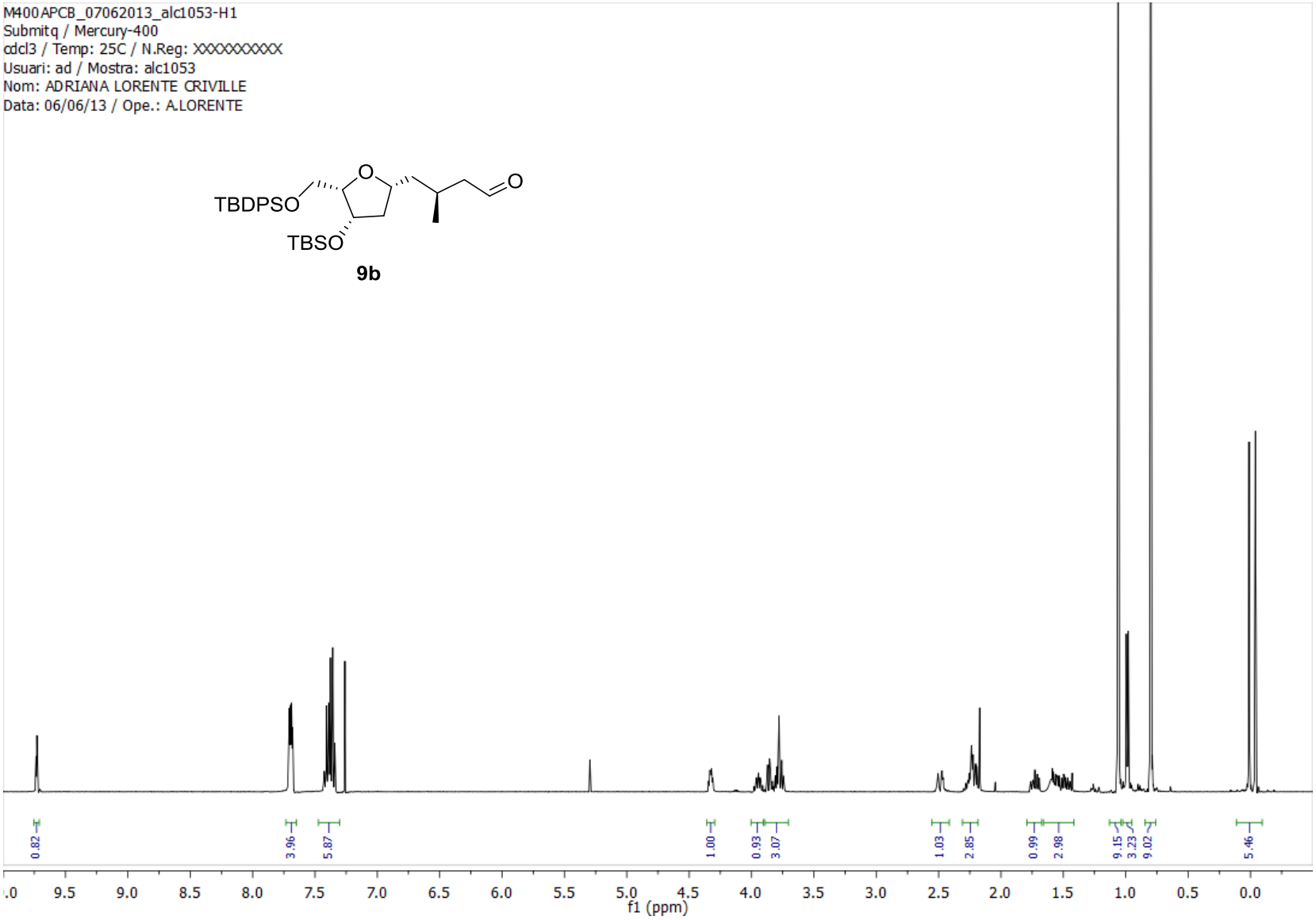

M400APCB_07062013_alc1052-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1052
Nom: ADRIANA LORENTE CRIVILLE
Data: 06/06/13 / Ope.: A.LORENTE
TBDPSO
O
OH
TBSO
S5b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

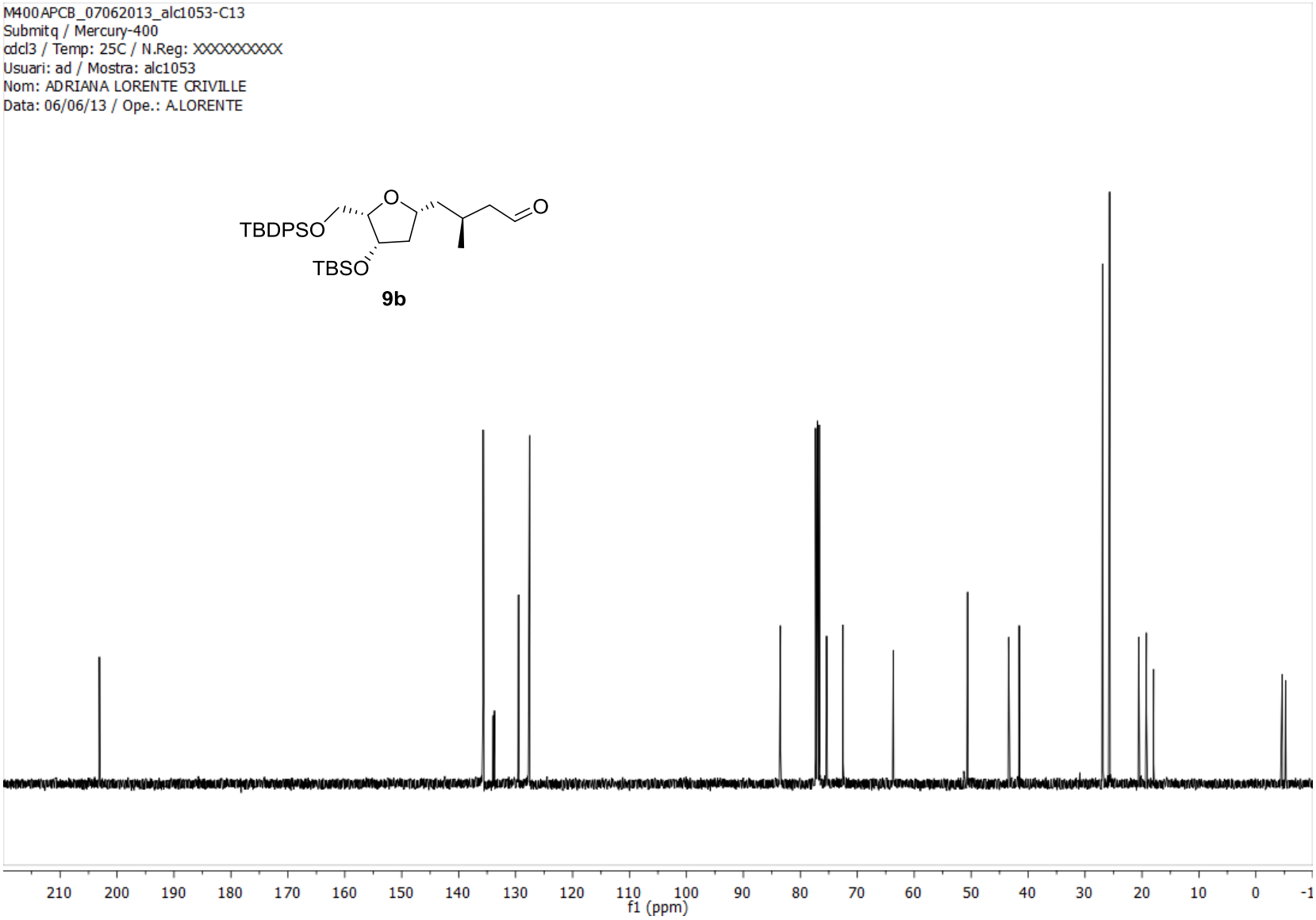

M400APCB_07062013_alc1052-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1052
Nom: ADRIANA LORENTE CRIVILLE
Data: 06/06/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

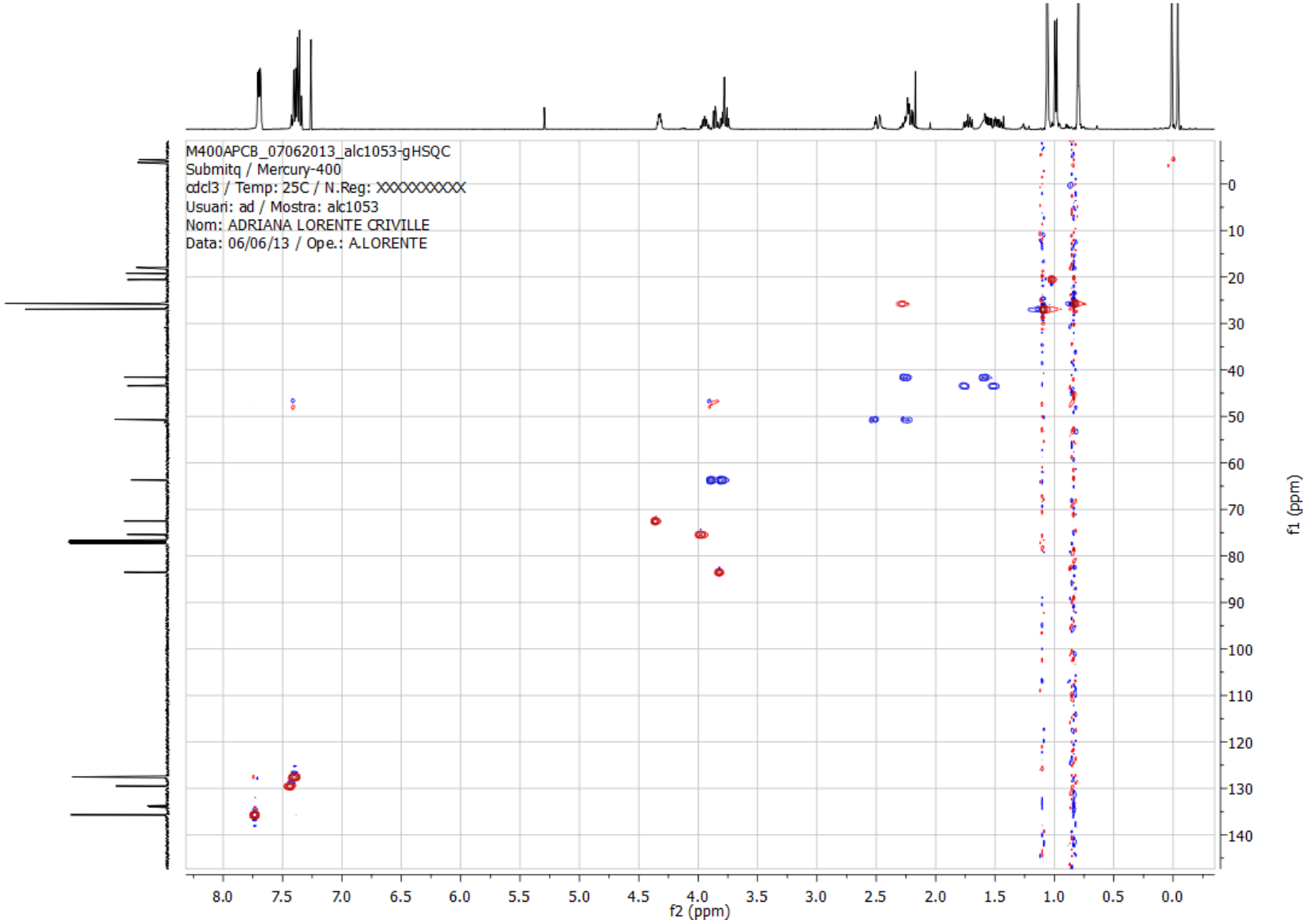

mercury400_0416-alc828p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc828p
Nom: ADRIANA LORENTE CRIVILLE
Data: 16/04/12 / Ope.: A.LORENTE
TBDPSO
TBSO
O
O
9a
1.04
5.32
7.70
1.30
1.38
1.30
2.77
1.21
3.89
1.37
1.58
1.53
12.03
4.10
12.19
3.52
3.31
9.5
9.0
8.5
8.0
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)

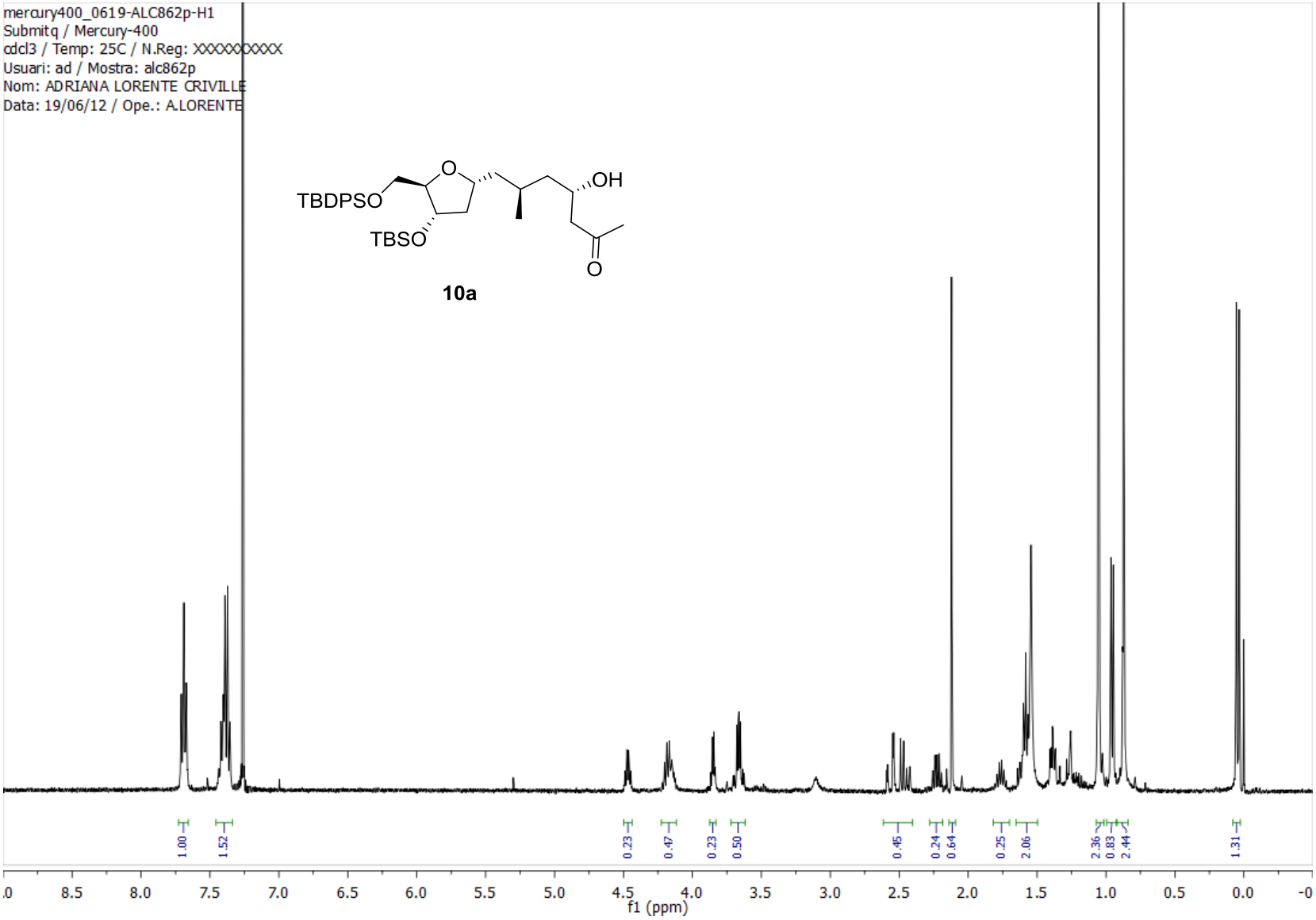

mercury400_0416-alc828p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc828p
Nom: ADRIANA LORENTE CRIVILLE
Data: 16/04/12 / Ope.: A.LORENTE
TBDPSO
TBSO
O
O
9a
f1 (ppm)

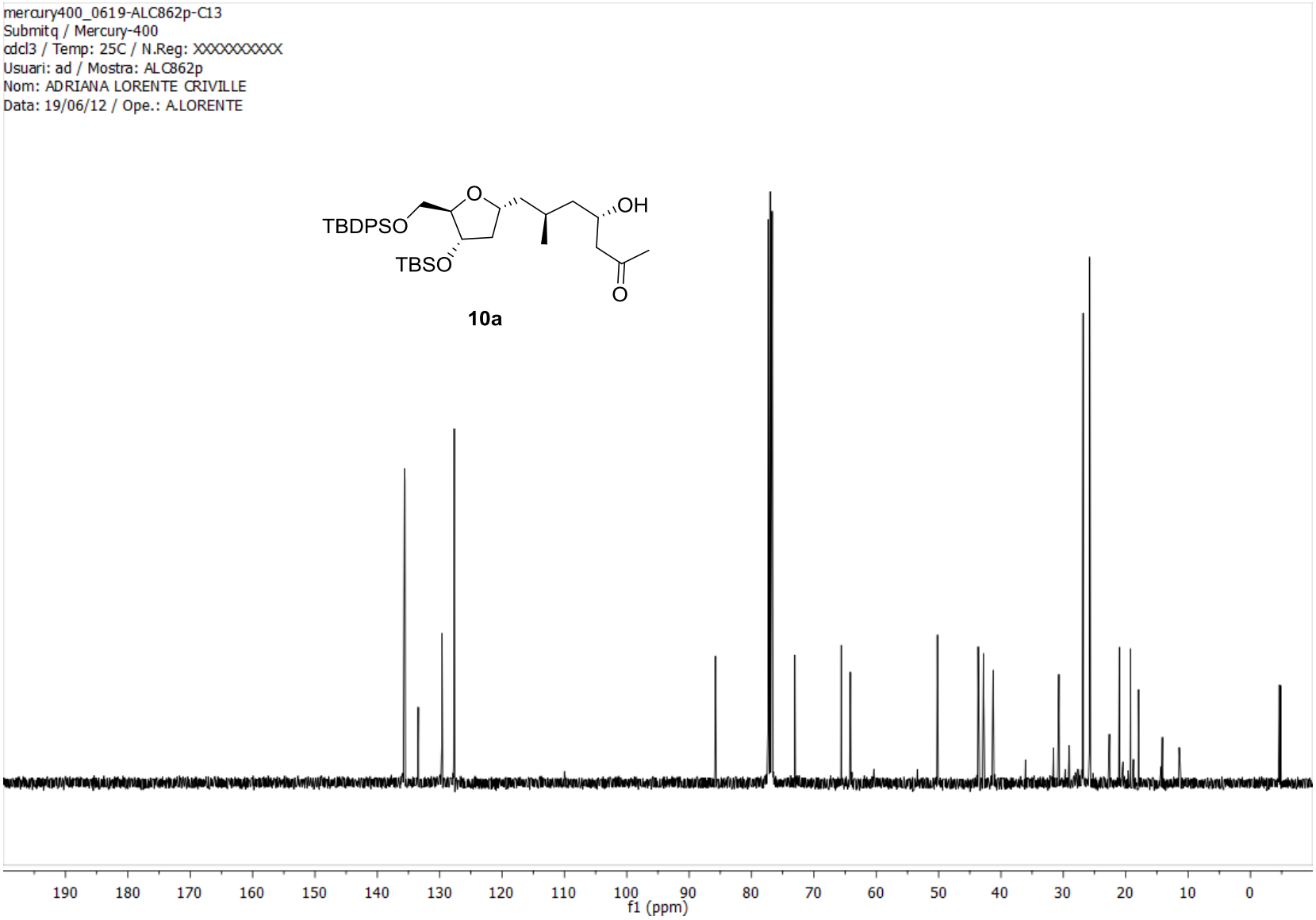

mercury400_0416-alc828p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc828p
Nom: ADRIANA LORENTE CRIVILLE
Data: 16/04/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

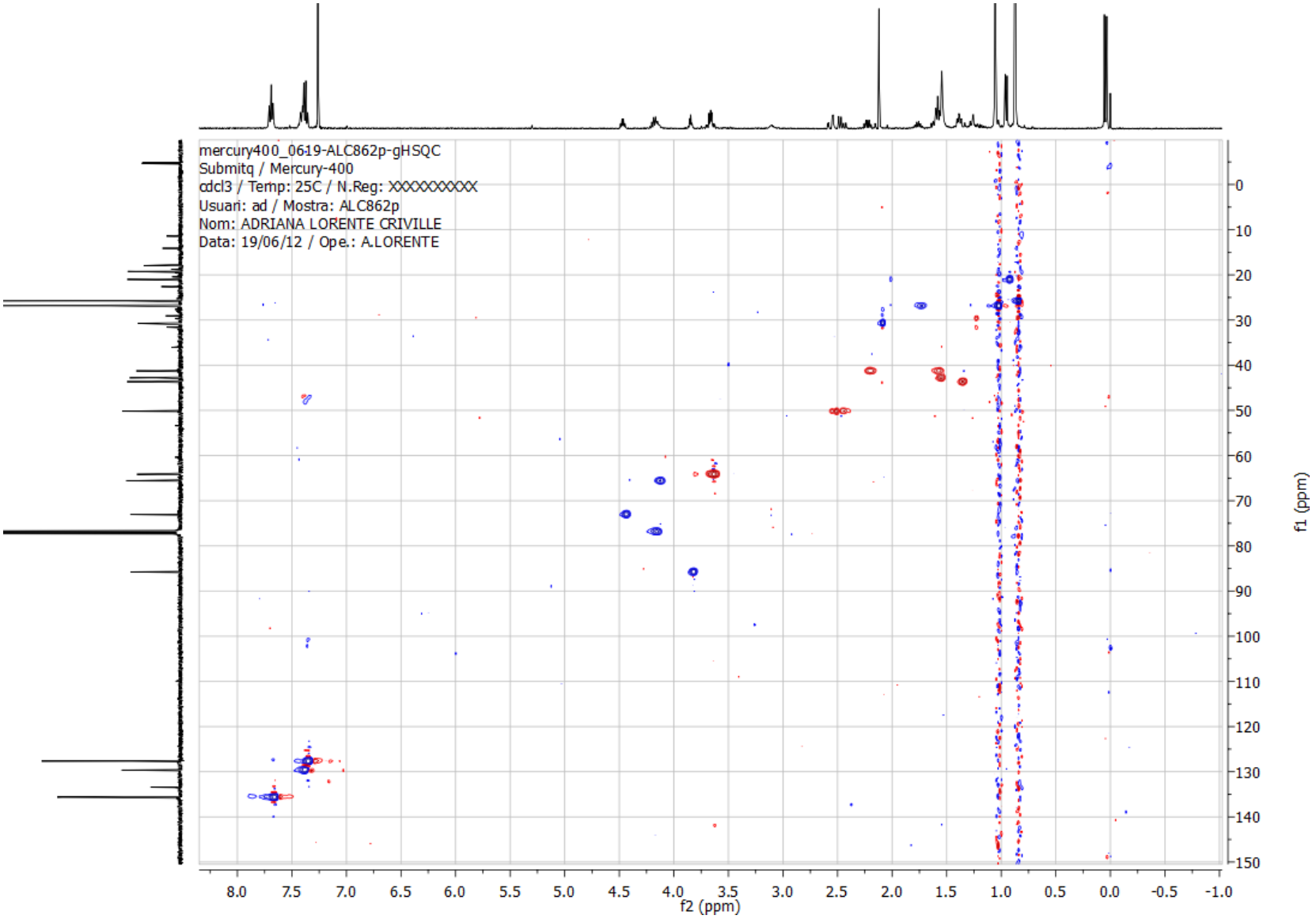

M400APCB_07062013_alc1053-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1053
Nom: ADRIANA LORENTE CRIVILLE
Data: 06/06/13 / Ope.: A.LORENTE
TBDPSO
TBSO
O
O
9b
0.82
3.96
5.87
1.00
0.93
3.07
1.03
2.85
0.99
2.98
9.15
3.23
9.02
5.46
f1 (ppm)

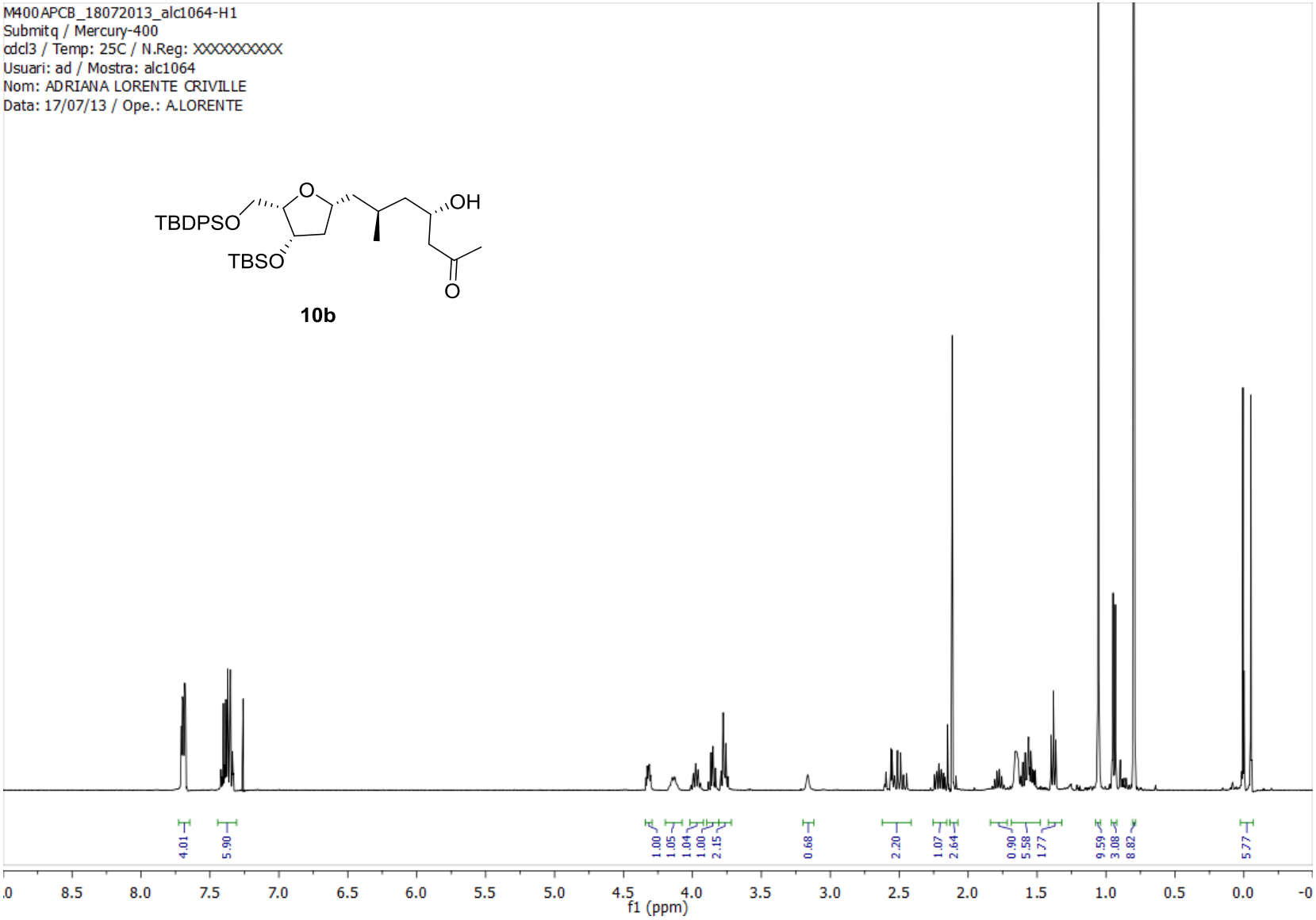

M400APCB_07062013_alc1053-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1053
Nom: ADRIANA LORENTE CRIVILLE
Data: 06/06/13 / Ope.: A.LORENTE
TBDPSO
TBSO
O
O
9b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

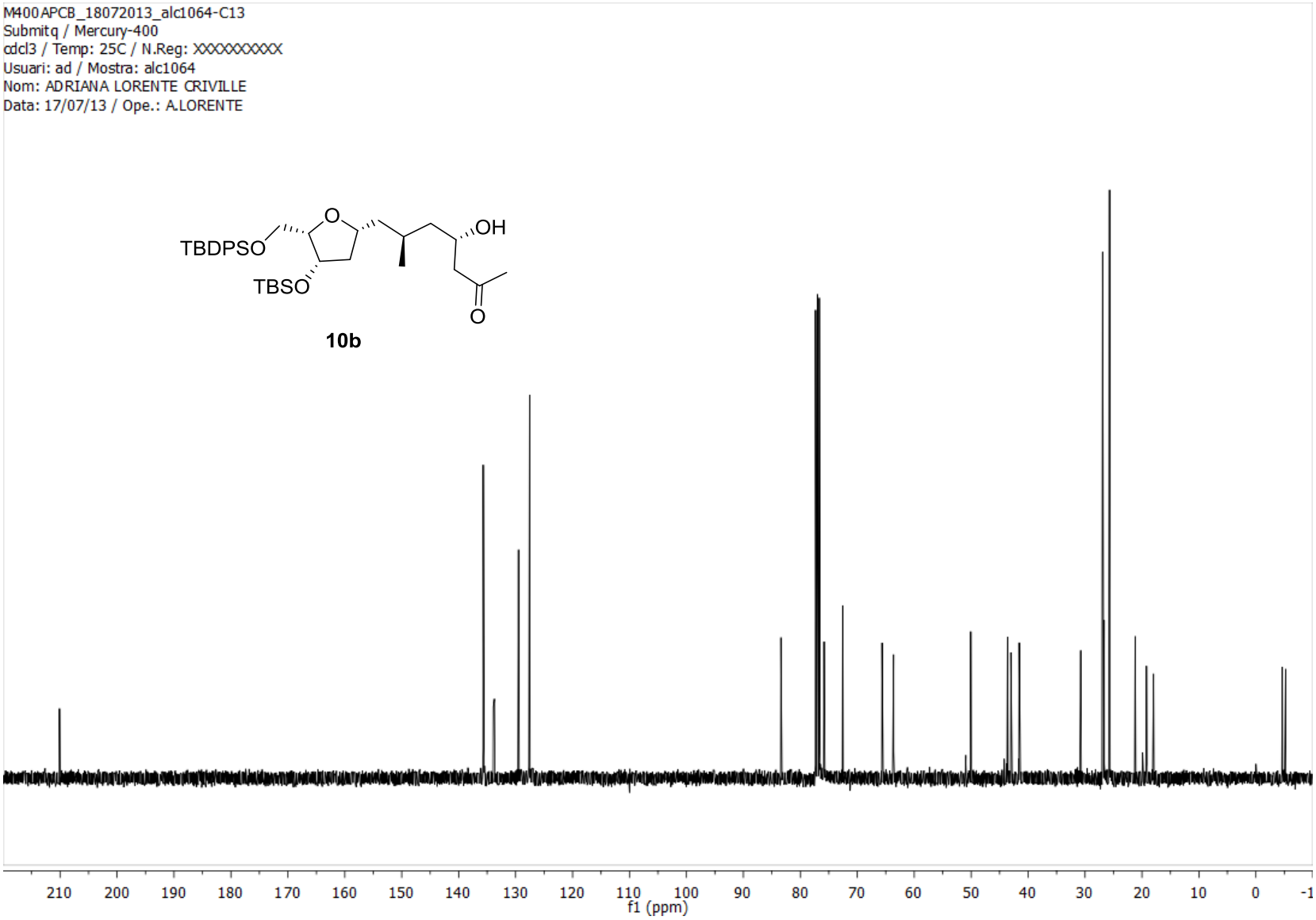

M400APCB_07062013_alc1053-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1053
Nom: ADRIANA LORENTE CRIVILLE
Data: 06/06/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

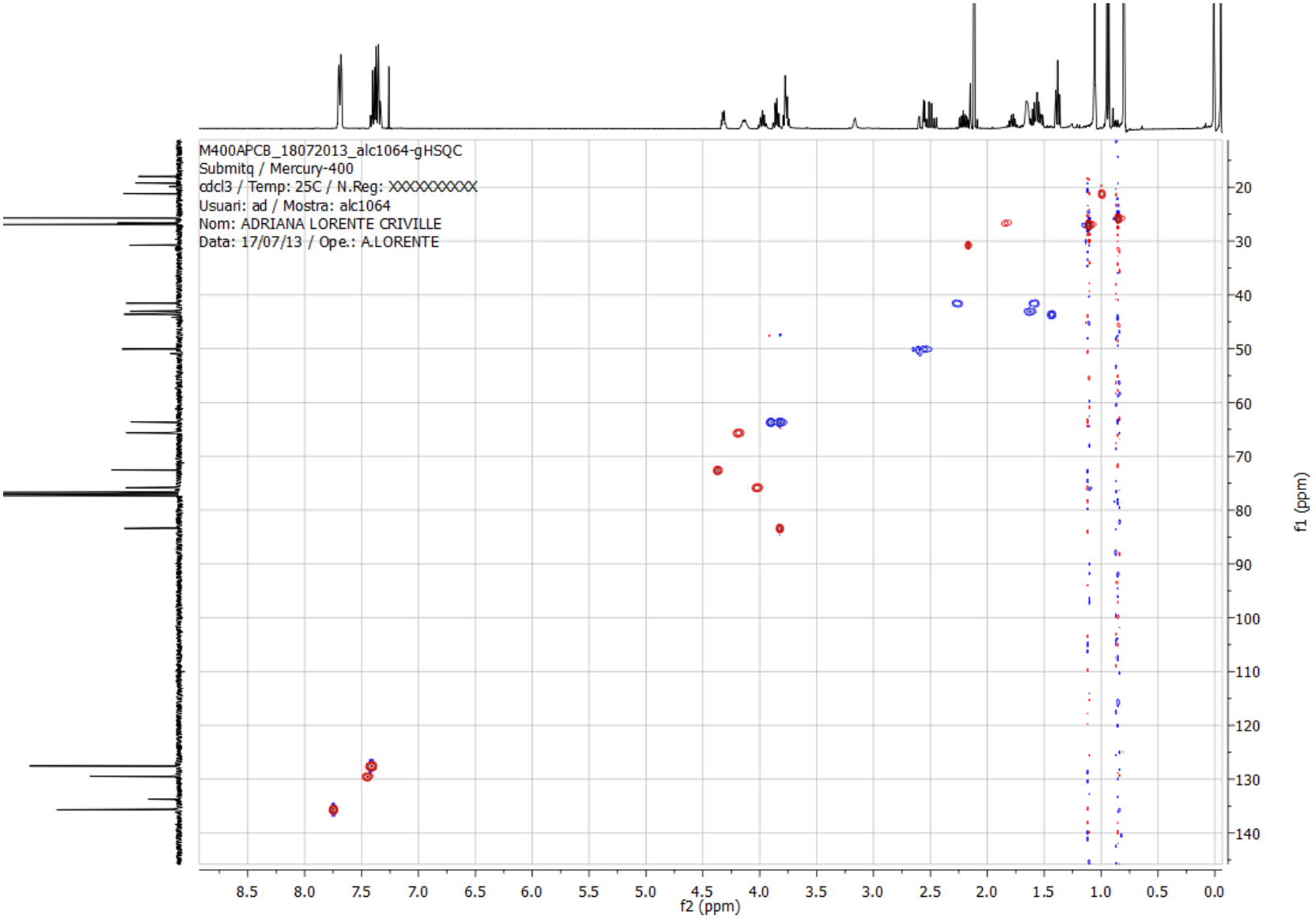

mercury400_0619-ALC862p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc862p
Nom: ADRIANA LORENTE CRIVILLE
Data: 19/06/12 / Ope.: A.LORENTE
TBDPSO
TBSO
O
OH
O
10a
1.00
1.52
0.23
0.47
0.23
0.50
0.45
0.24
0.64
0.25
2.06
2.36
0.83
2.44
1.31
8.5
8.0
7.5
7.0
6.5
6.0
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)

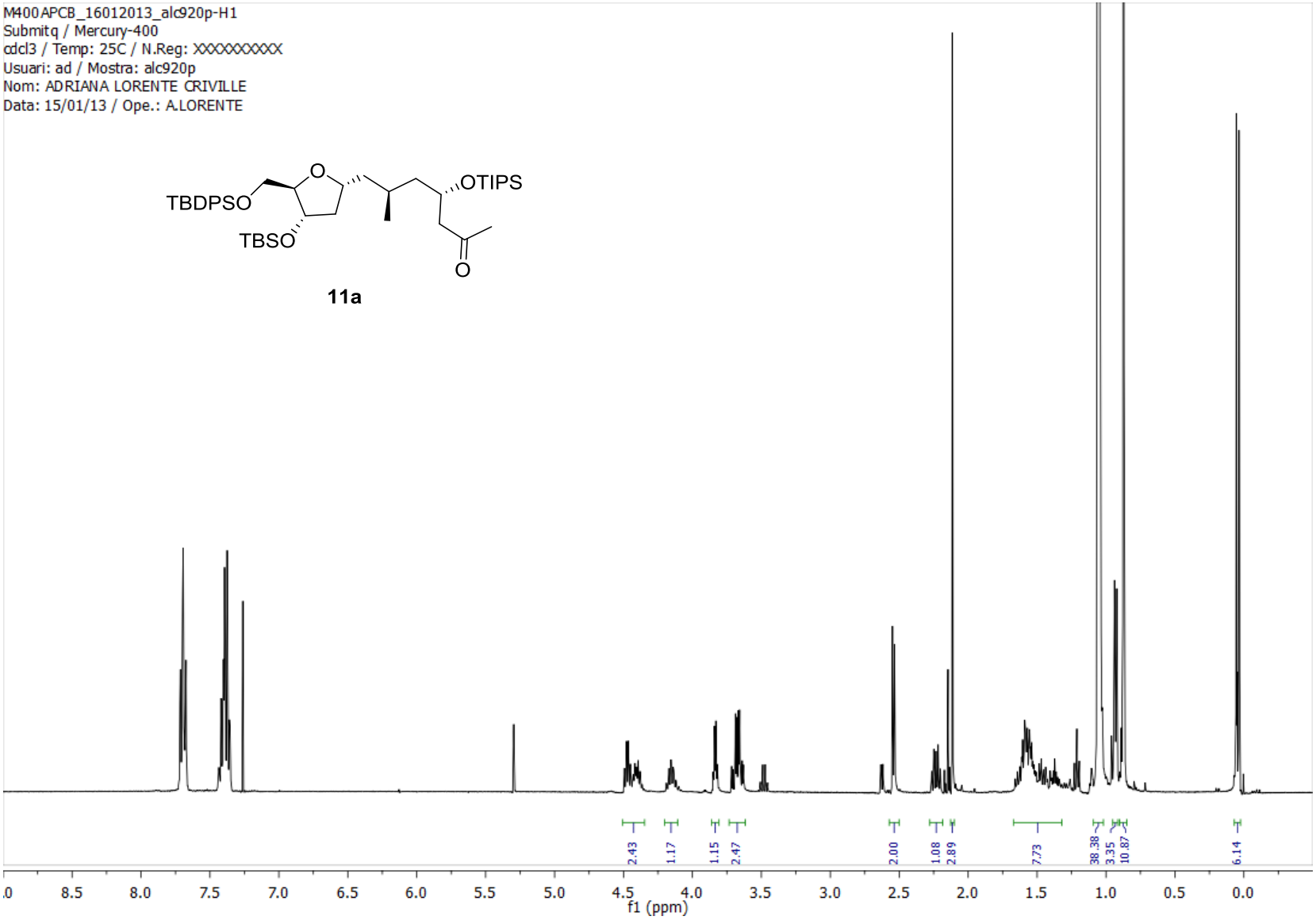

mercury400_0619-ALC862p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC862p
Nom: ADRIANA LORENTE CRIVILLE
Data: 19/06/12 / Ope.: A.LORENTE
TBDPSO
TBSO
O
OH
O
10a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

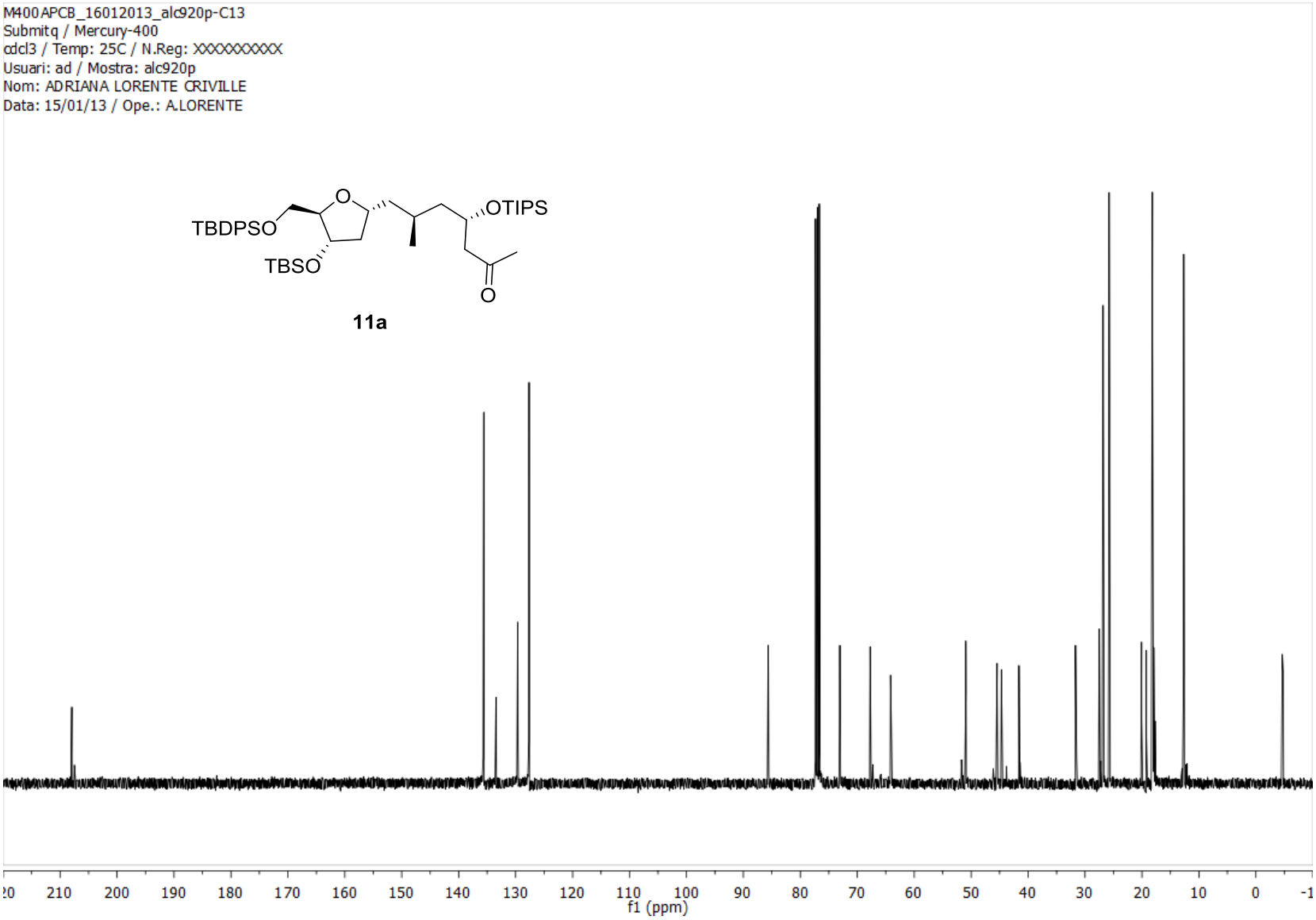

mercury400_0619-ALC862p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC862p
Nom: ADRIANA LORENTE CRIVILLE
Data: 19/06/12 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

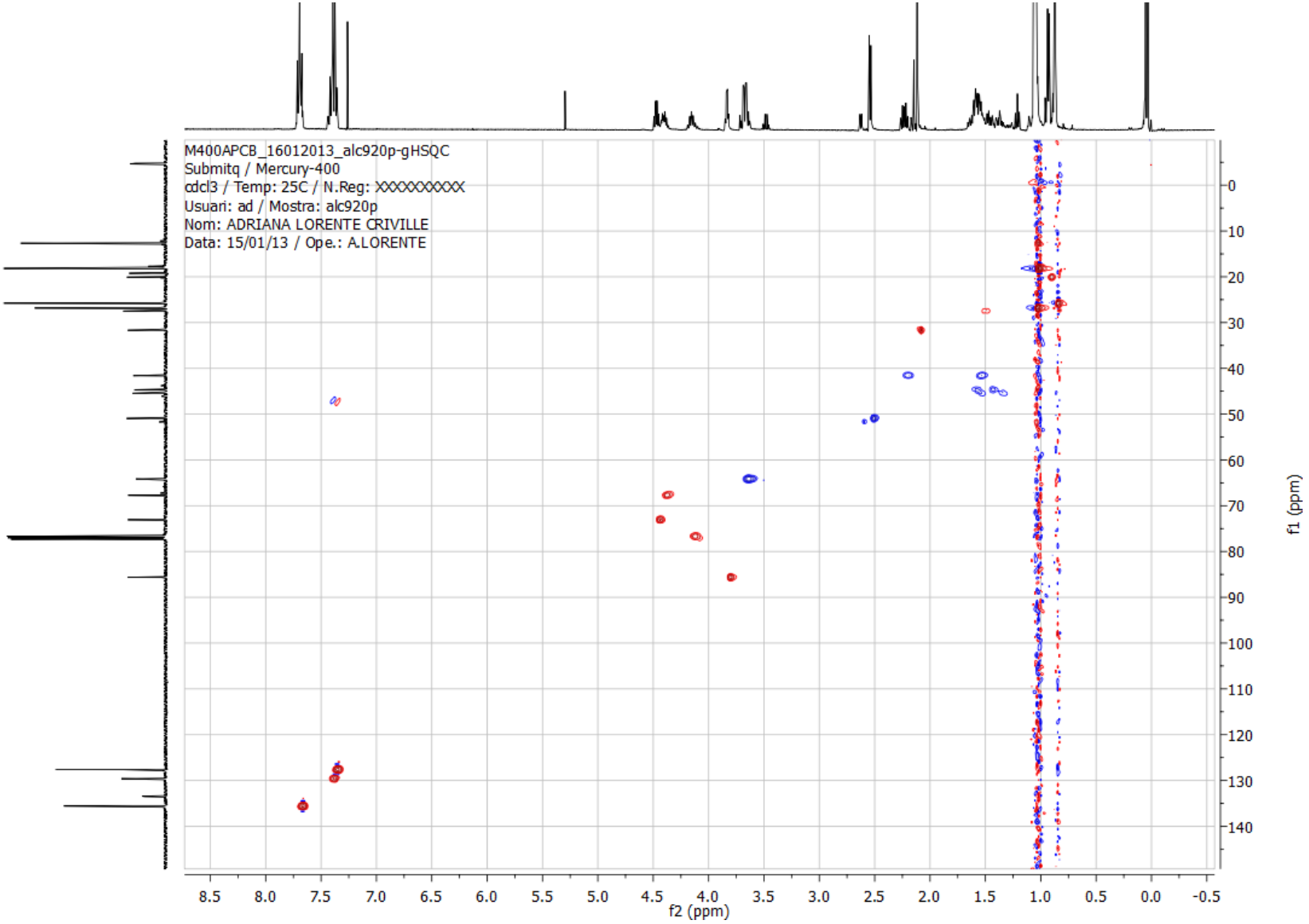

M400APCB_18072013_alc1064-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1064
Nom: ADRIANA LORENTE CRIVILLE
Data: 17/07/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OH
O
O
10b
4.01
5.90
1.00
1.05
1.04
1.00
2.15
0.68
2.20
1.07
2.64
0.90
5.58
1.77
9.59
3.08
8.82
5.77
f1 (ppm)

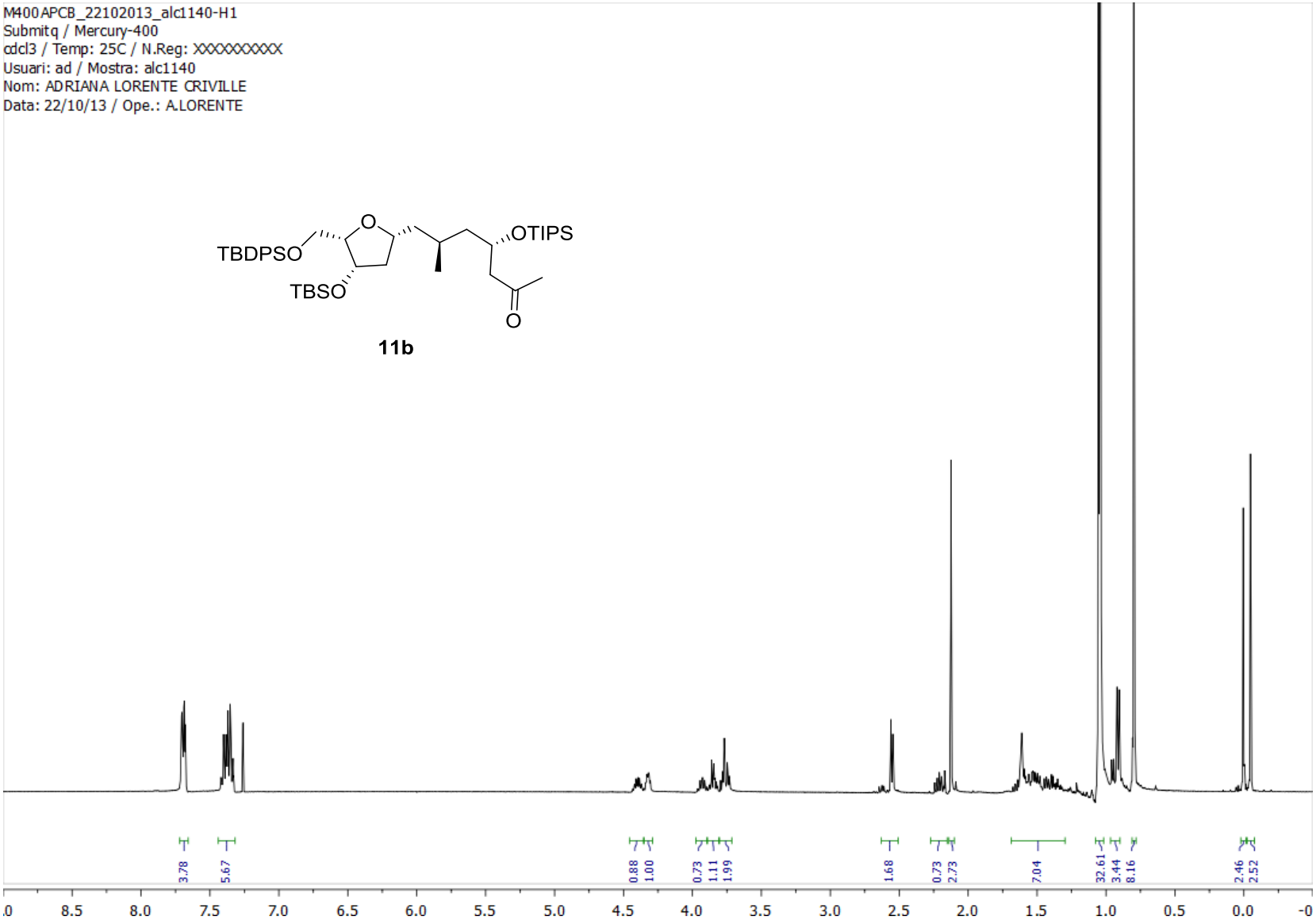

M400APCB_18072013_alc1064-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1064
Nom: ADRIANA LORENTE CRIVILLE
Data: 17/07/13 / Ope.: A.LORENTE
TBDPSO
TBSO
O
OH
O
10b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

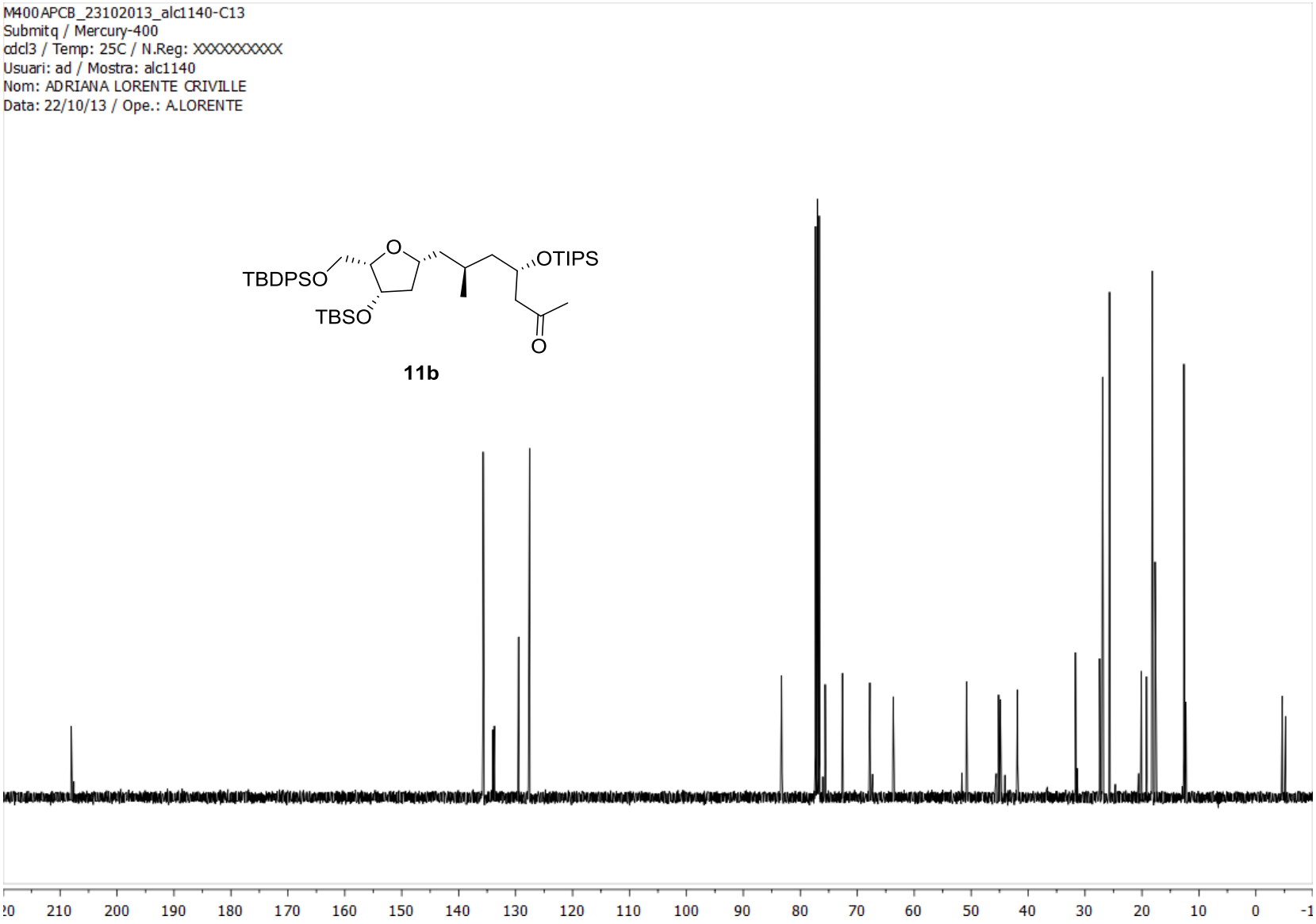

M400APCB_18072013_alc1064-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1064
Nom: ADRIANA LORENTE CRIVILLE
Data: 17/07/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

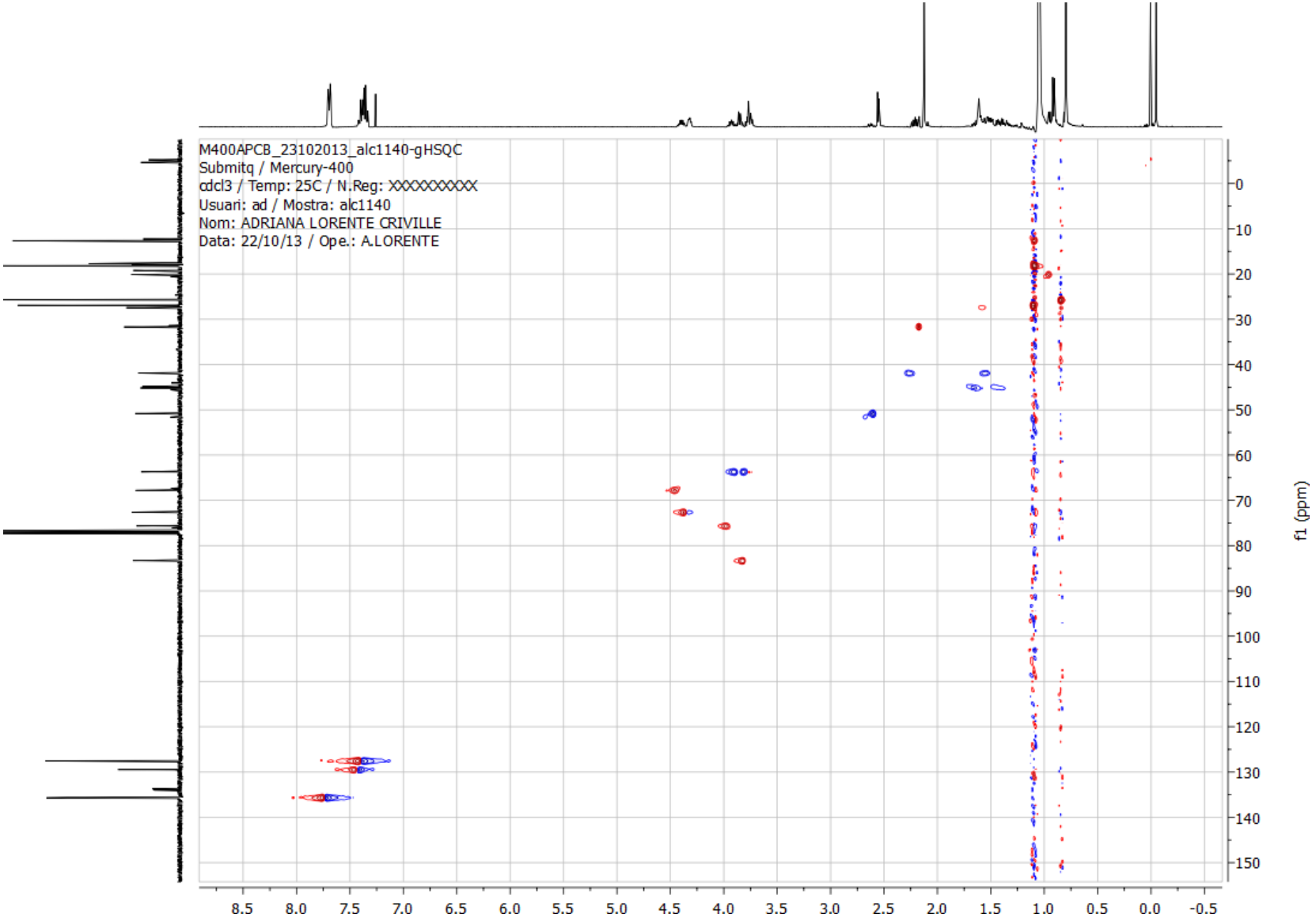

M400APCB_16012013_alc920p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc920p
Nom: ADRIANA LORENTE CRIVILLE
Data: 15/01/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
O
O
11a
2.43
1.17
1.15
2.47
2.00
1.08
2.89
7.73
38.38
3.35
10.87
6.14
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

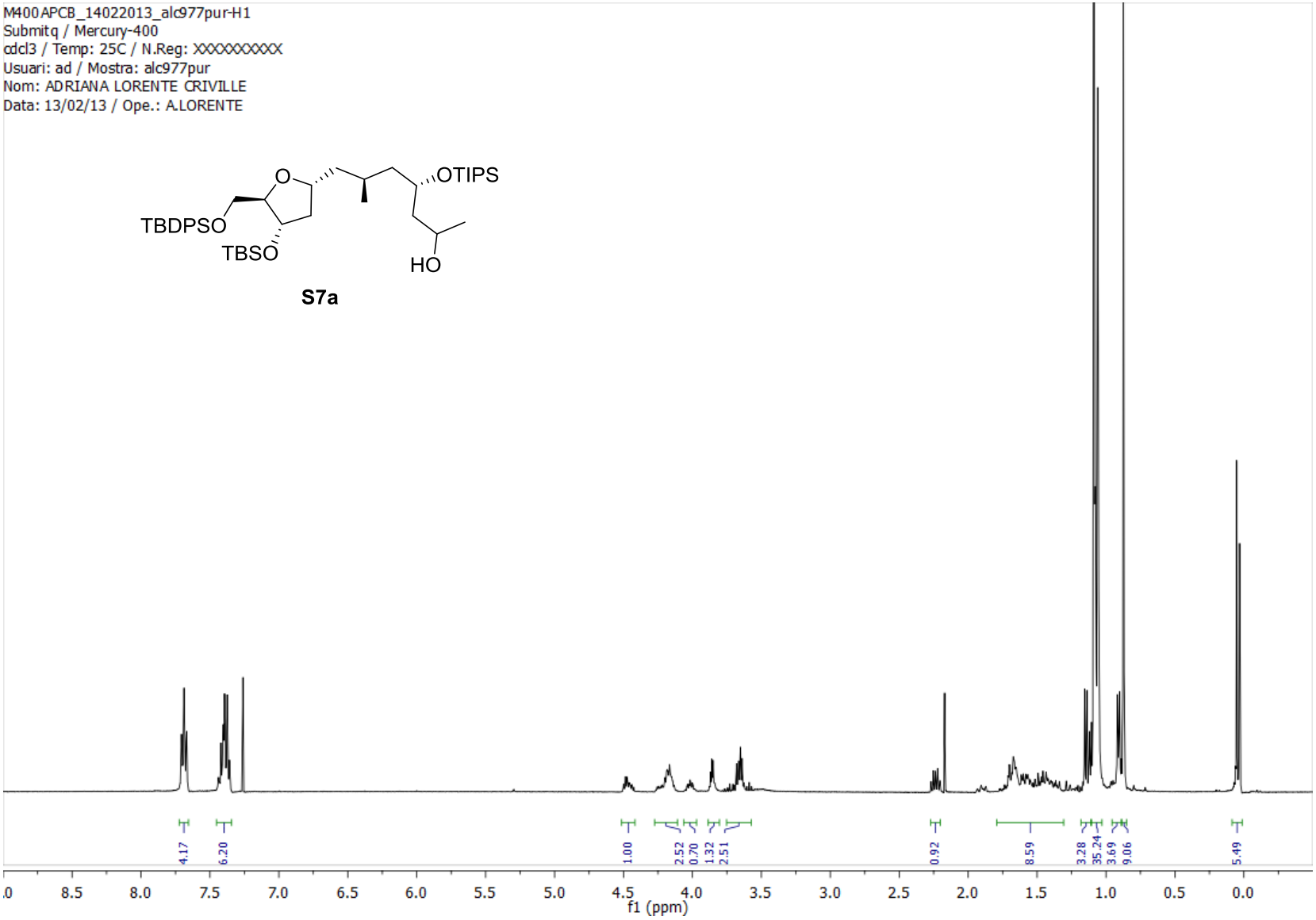

M400APCB_16012013_alc920p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc920p
Nom: ADRIANA LORENTE CRIVILLE
Data: 15/01/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
O
O
11a
f1 (ppm)

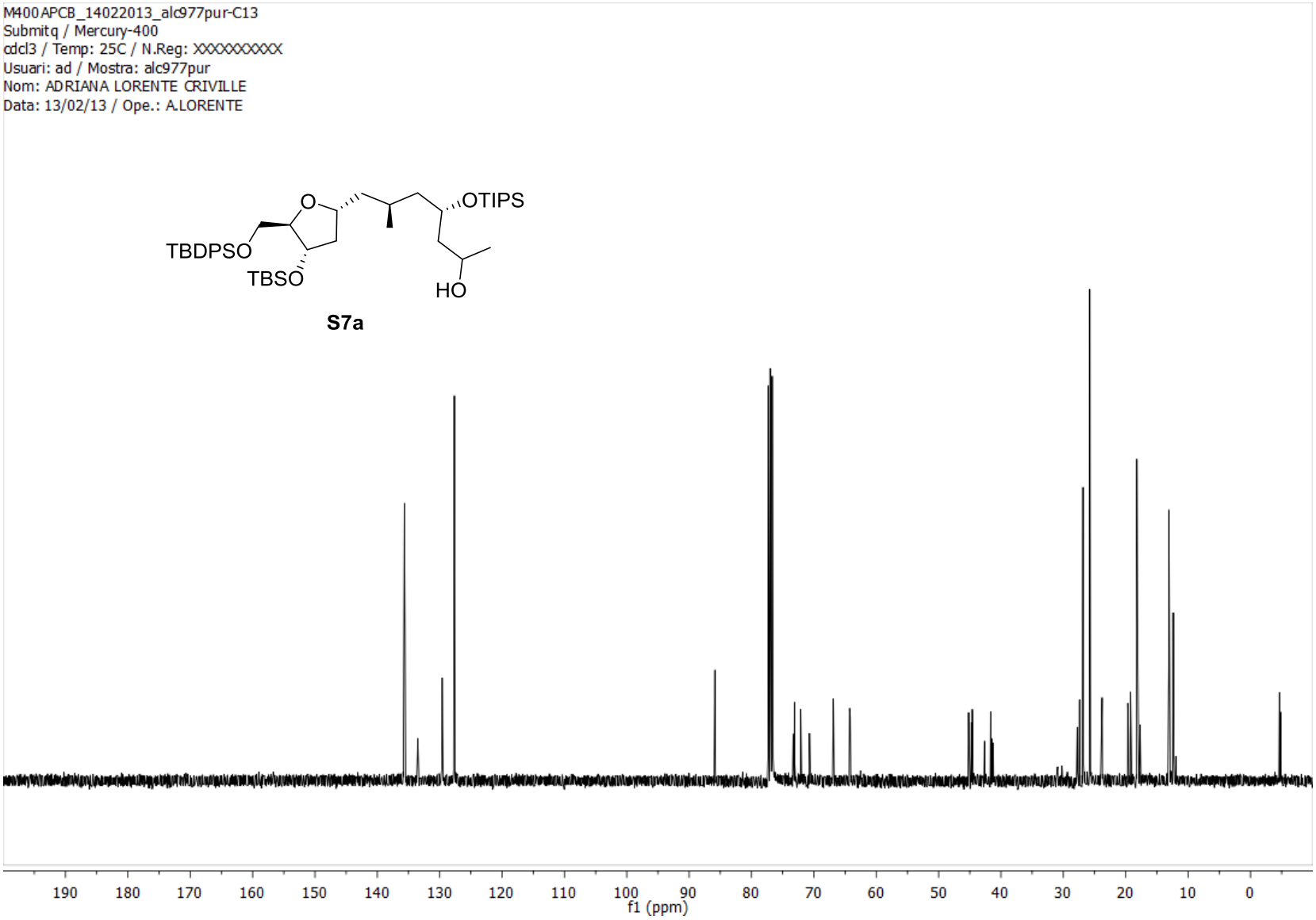

M400APCB_16012013_alc920p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc920p
Nom: ADRIANA LORENTE CRIVILLE
Data: 15/01/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

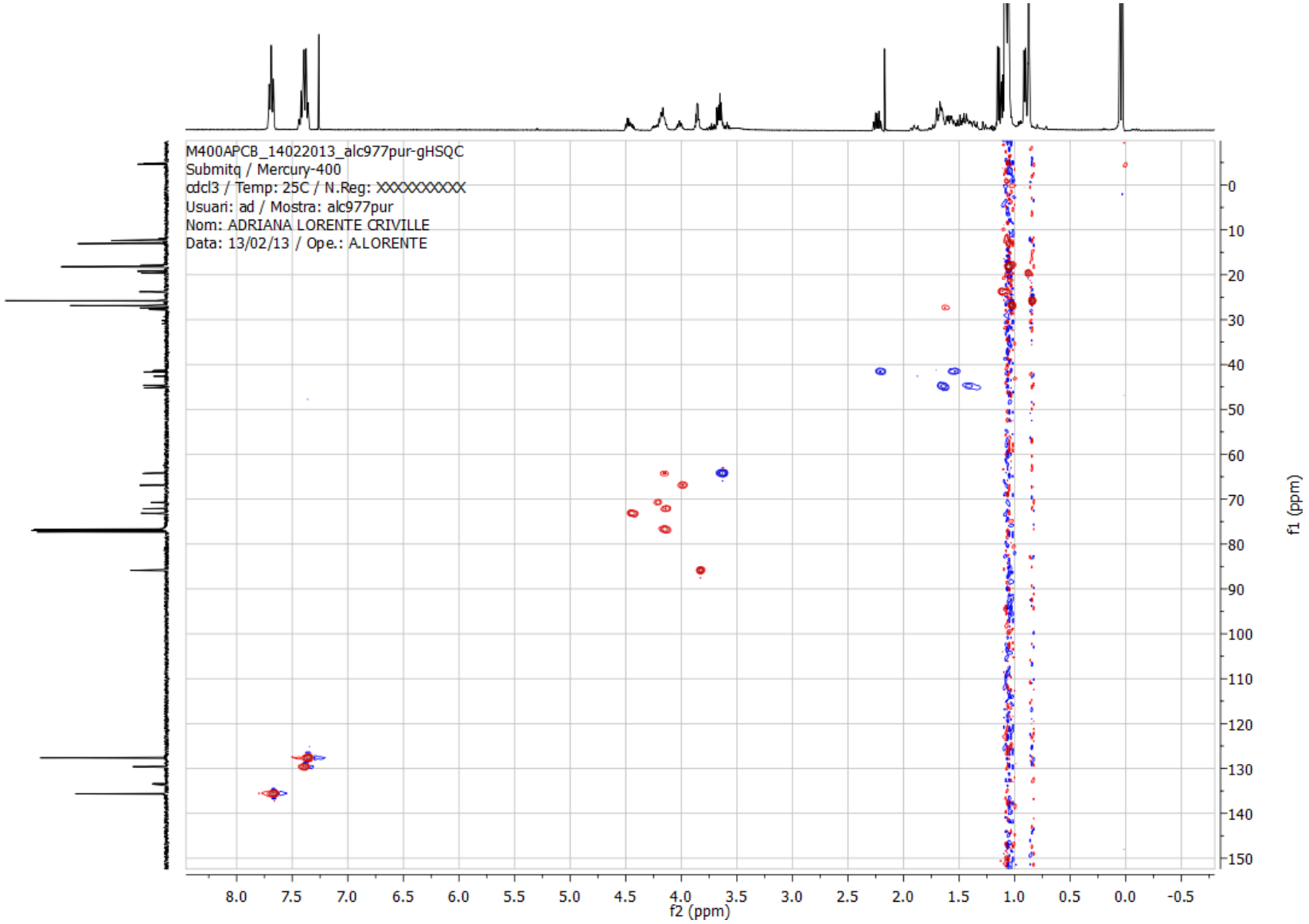

M400APCB_22102013_alc1140-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1140
Nom: ADRIANA LORENTE CRIVILLE
Data: 22/10/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
O
O
11b
3.78
5.67
0.88
1.00
0.73
1.11
1.99
1.68
0.73
2.73
7.04
32.61
3.44
8.16
2.46
2.52
8.5
8.0
7.5
7.0
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

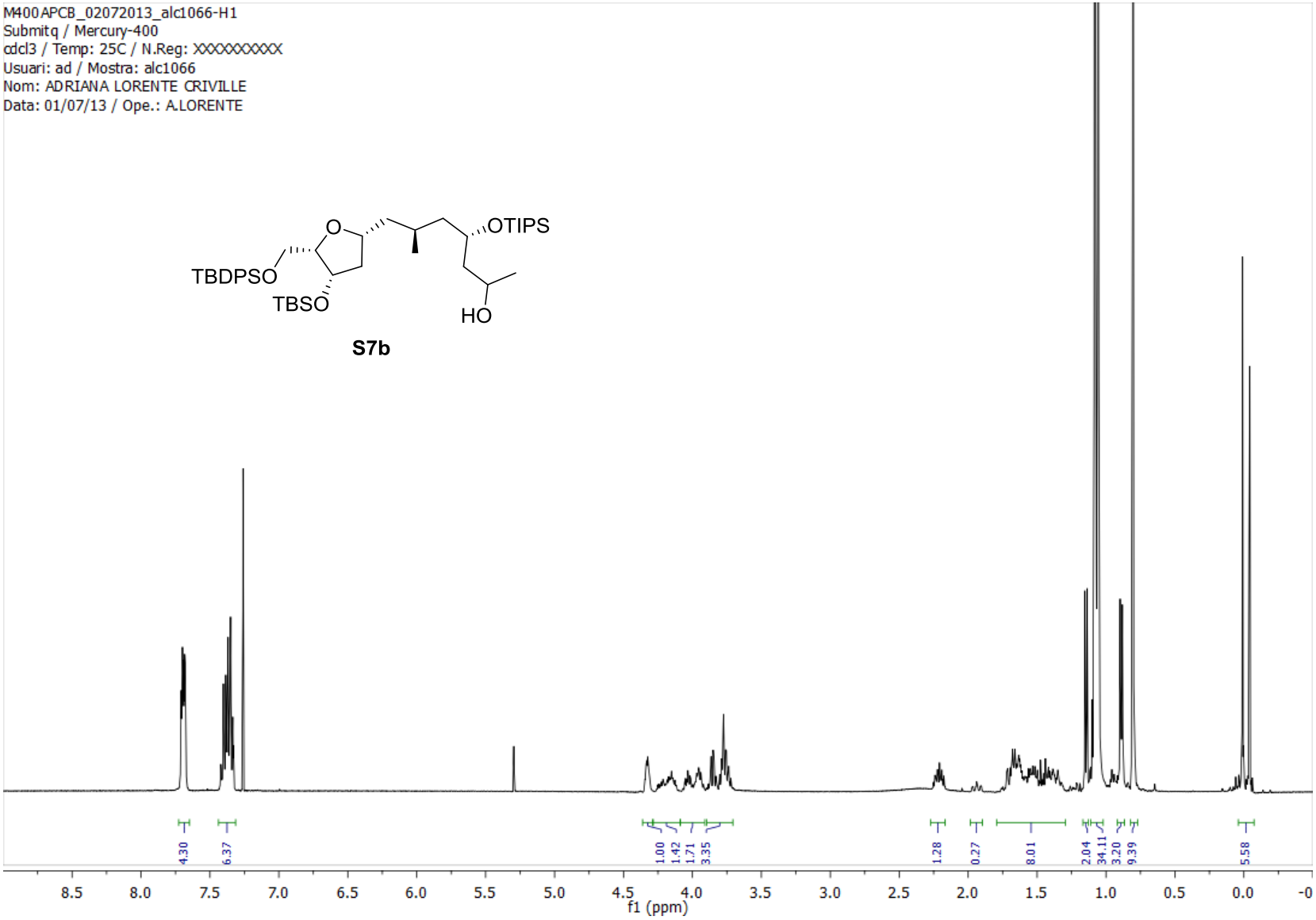

M400APCB_23102013_alc1140-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1140
Nom: ADRIANA LORENTE CRIVILLE
Data: 22/10/13 / Ope.: A.LORENTE
TBDPSO
TBSO
O
OTIPS
O
11b
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

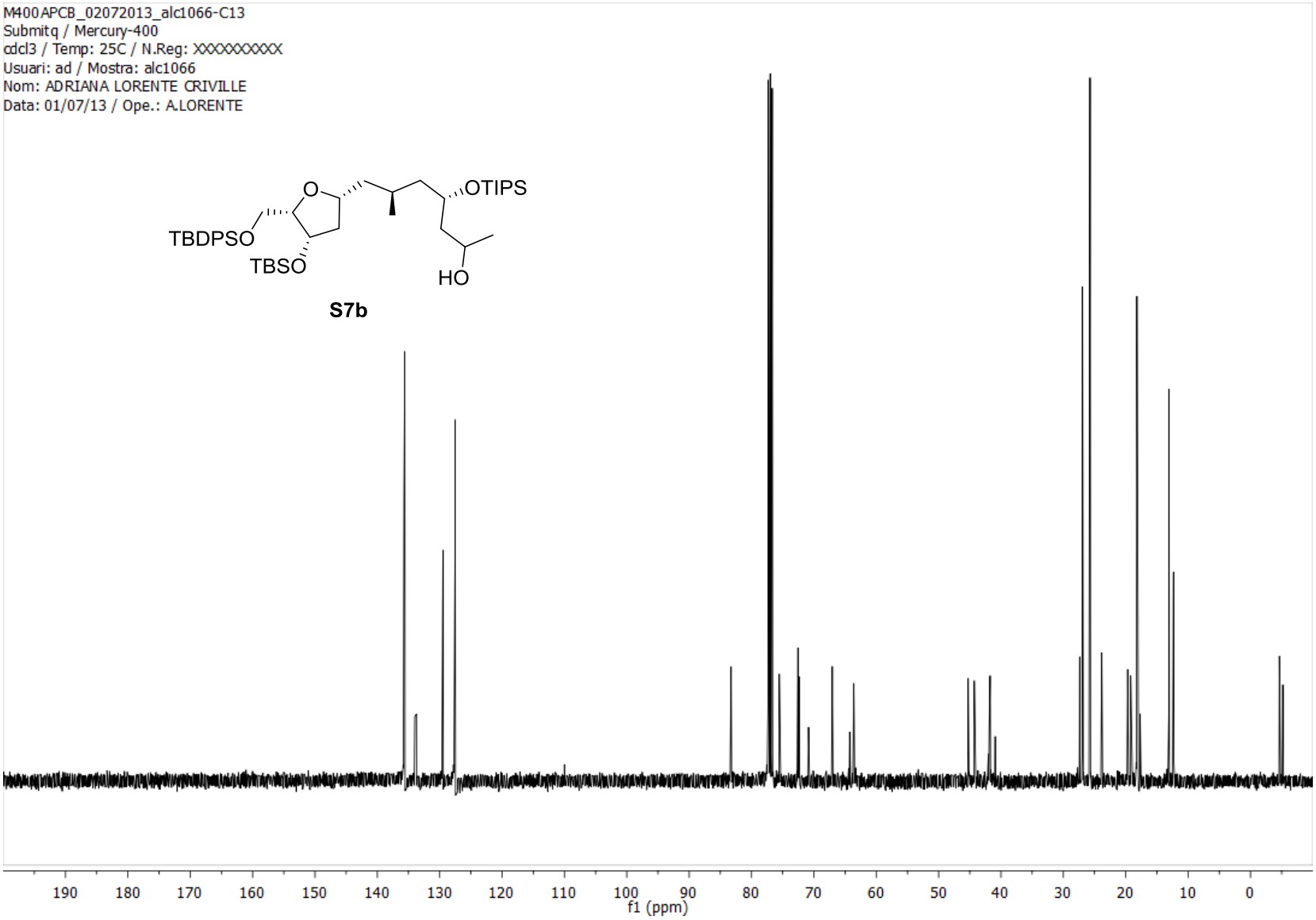

M400APCB_23102013_alc1140-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1140
Nom: ADRIANA LORENTE CRIVILLE
Data: 22/10/13 / Ope.: A.LORENTE
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

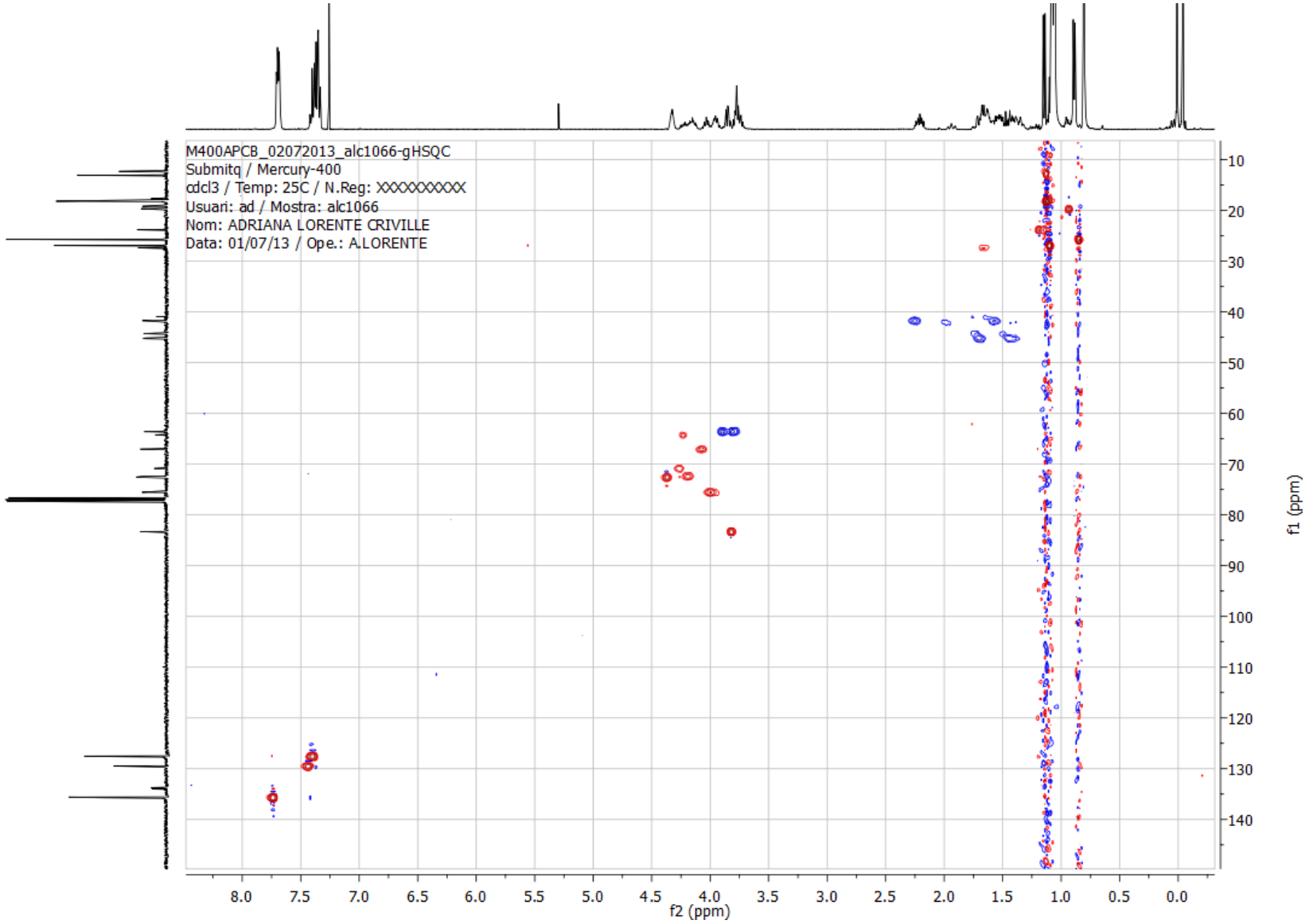

M400APCB_14022013_alc977pur-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc977pur
Nom: ADRIANA LORENTE CRIVILLE
Data: 13/02/13 / Ope.: A.LORENTE
O
OTIPS
TBDPSO
TBSO
HO
S7a
4.17
6.20
1.00
2.52
0.70
1.32
2.51
0.92
8.59
3.28
35.24
3.69
9.06
5.49
8.5
8.0
7.5
7.0
6.5
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)

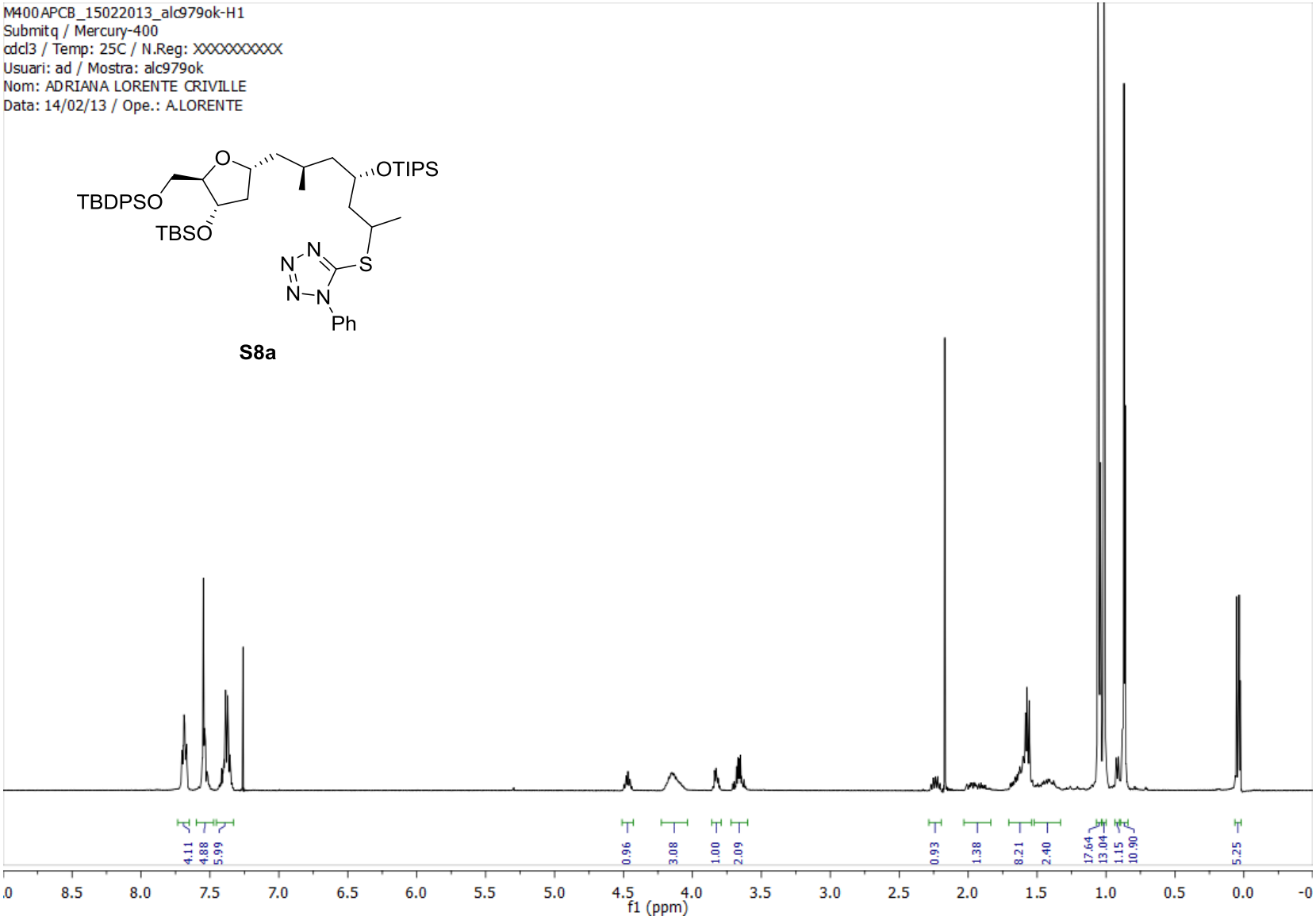

M400APCB_14022013_alc977pur-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc977pur
Nom: ADRIANA LORENTE CRIVILLE
Data: 13/02/13 / Ope.: A.LORENTE
O
OTIPS
TBDPSO
TBSO
HO
S7a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

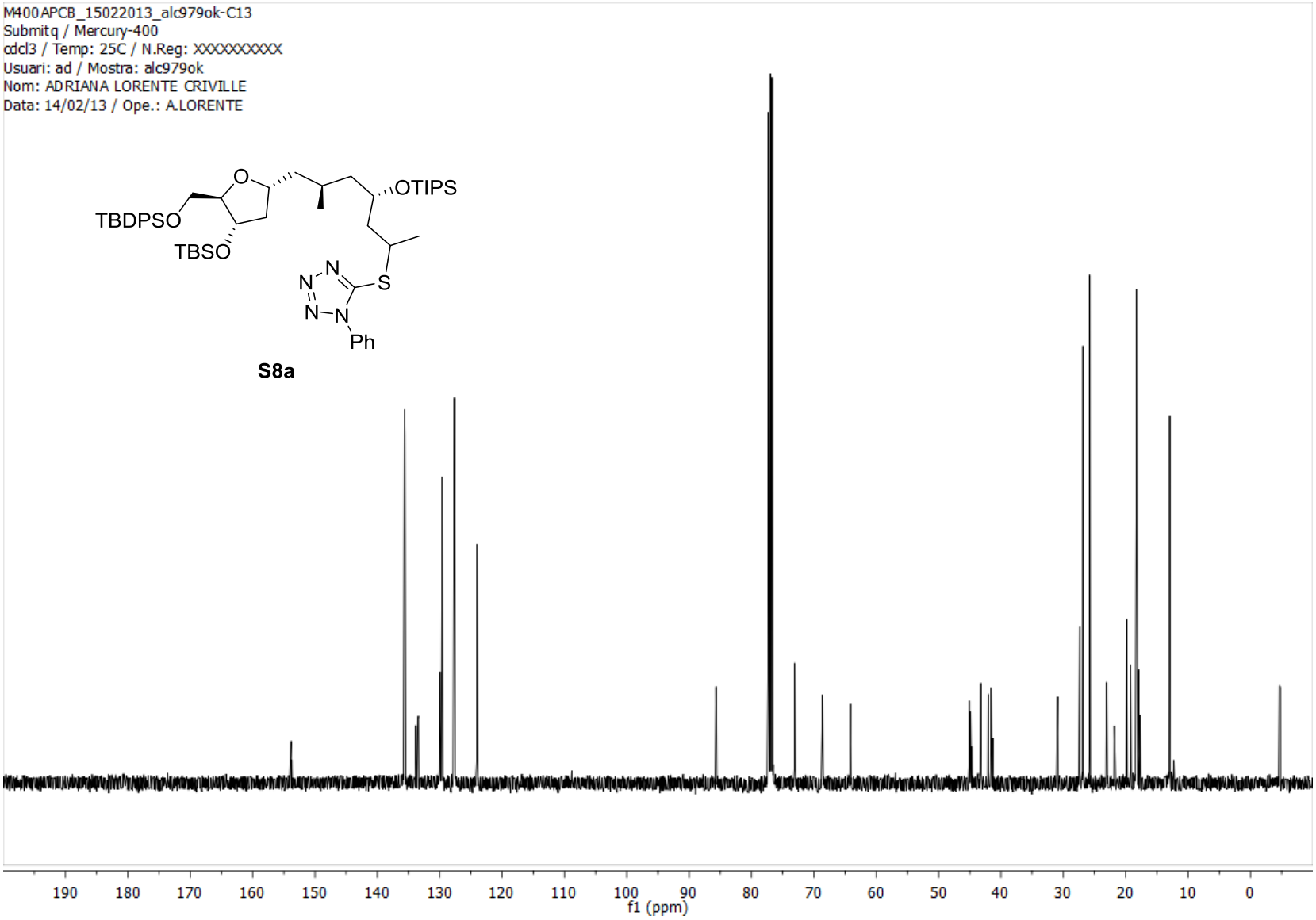

M400APCB_14022013_alc977pur-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc977pur
Nom: ADRIANA LORENTE CRIVILLE
Data: 13/02/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

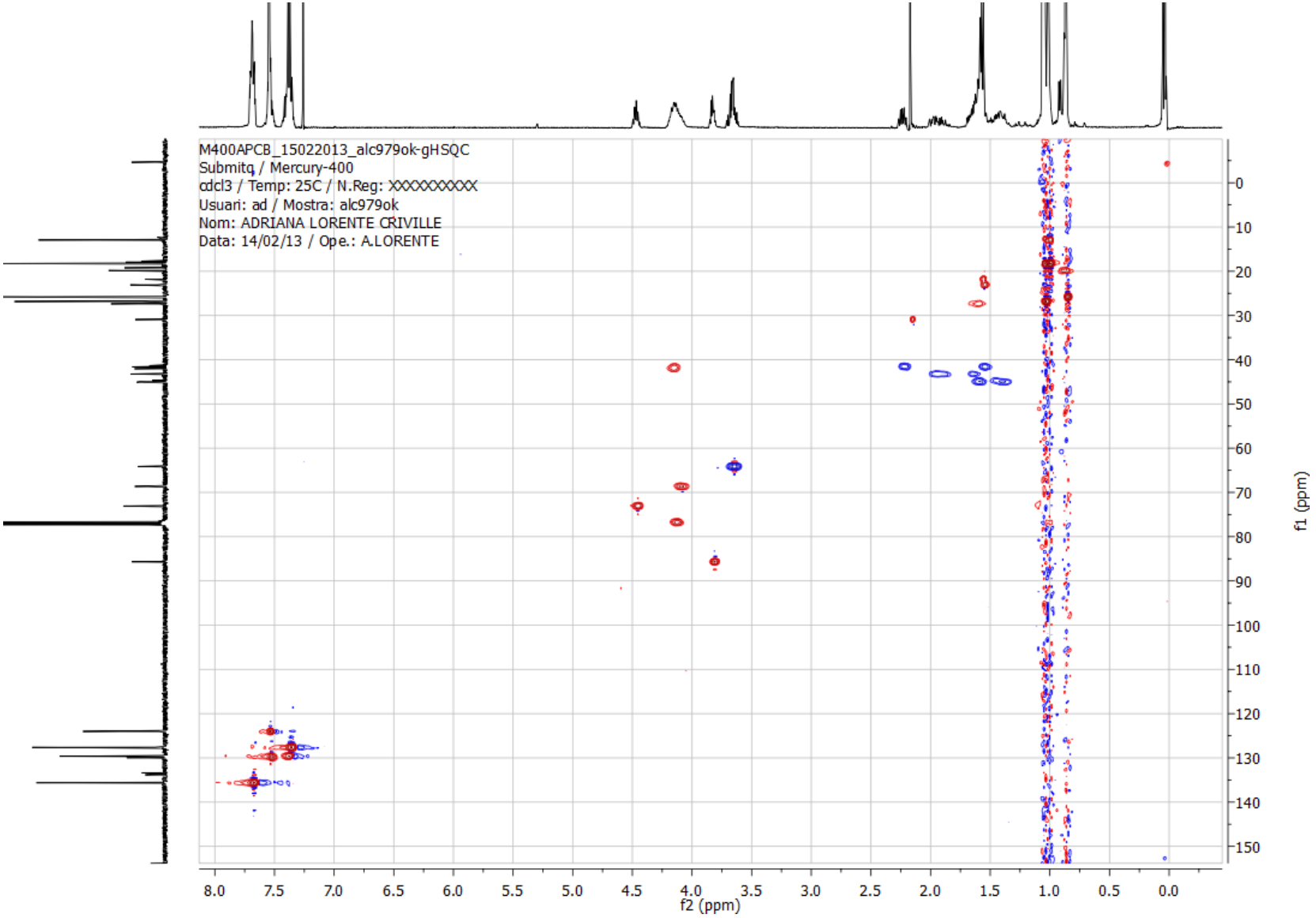

M400APCB_02072013_alc1066-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1066
Nom: ADRIANA LORENTE CRIVILLE
Data: 01/07/13 / Ope.: A.LORENTE
O
OTIPS
TBDPSO
TBSO
HO
S7b
4.30
6.37
1.00
1.42
1.71
3.35
1.28
0.27
8.01
2.04
34.11
3.20
9.39
5.58
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

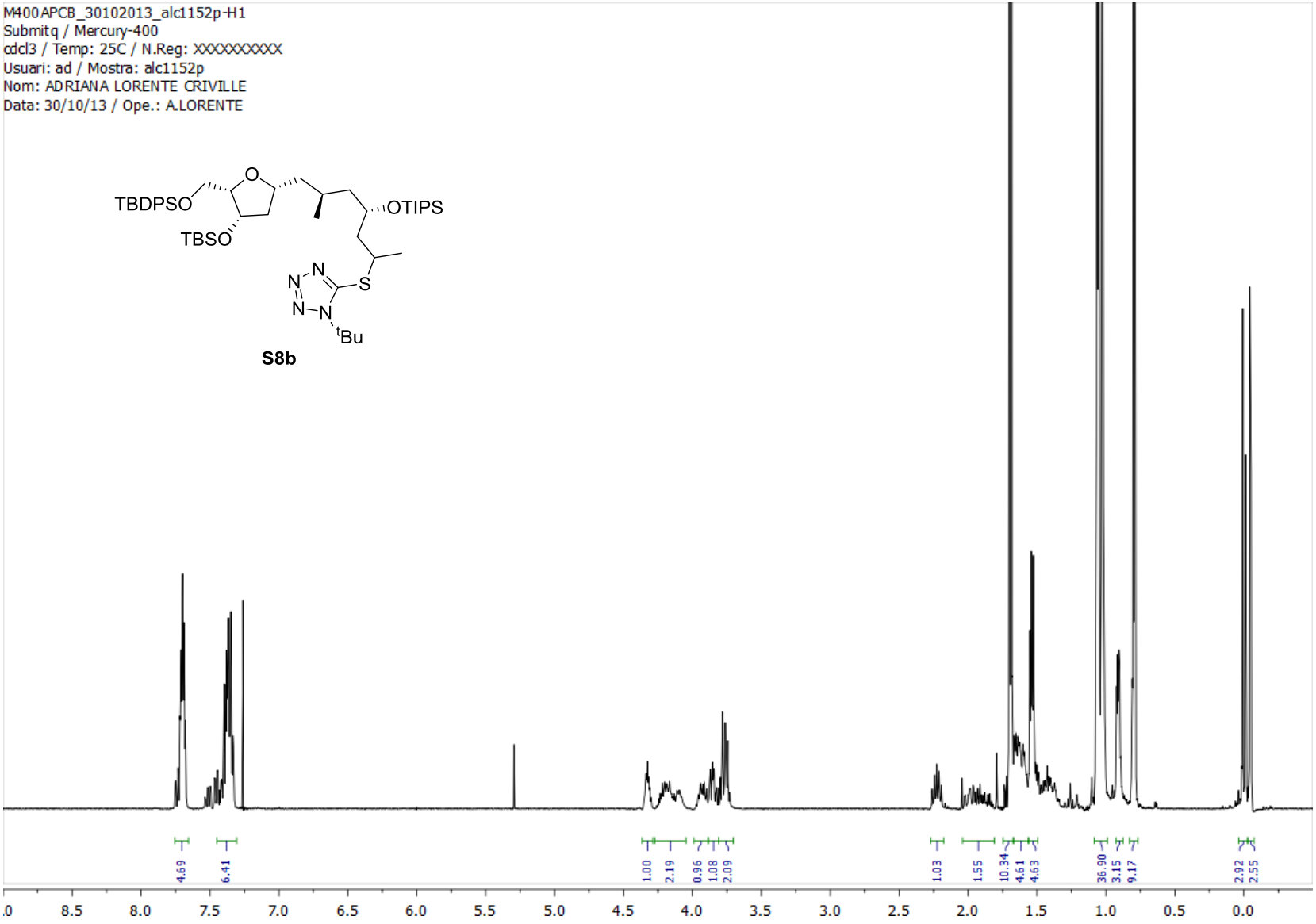

M400APCB_02072013_alc1066-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1066
Nom: ADRIANA LORENTE CRIVILLE
Data: 01/07/13 / Ope.: A.LORENTE
O
OTIPS
TBDPSO
TBSO
HO
S7b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


M400APCB_02072013_alc1066-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1066
Nom: ADRIANA LORENTE CRIVILLE
Data: 01/07/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

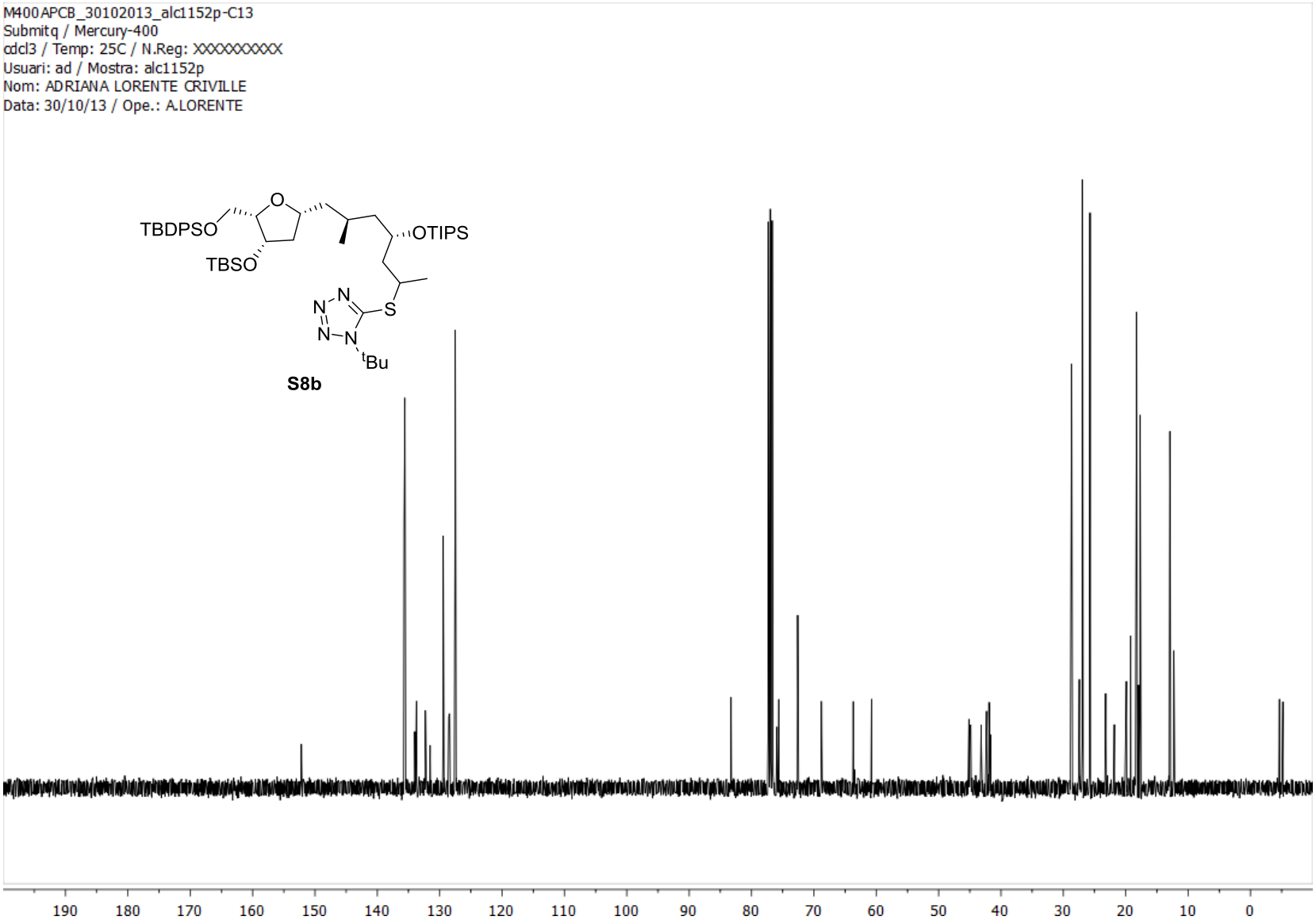

M400APCB_15022013_alc979ok-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc979ok
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
S
Ph
S8a
4.11
4.88
5.99
0.96
3.08
1.00
2.09
0.93
1.38
8.21
2.40
17.64
13.04
1.15
10.90
5.25
f1 (ppm)

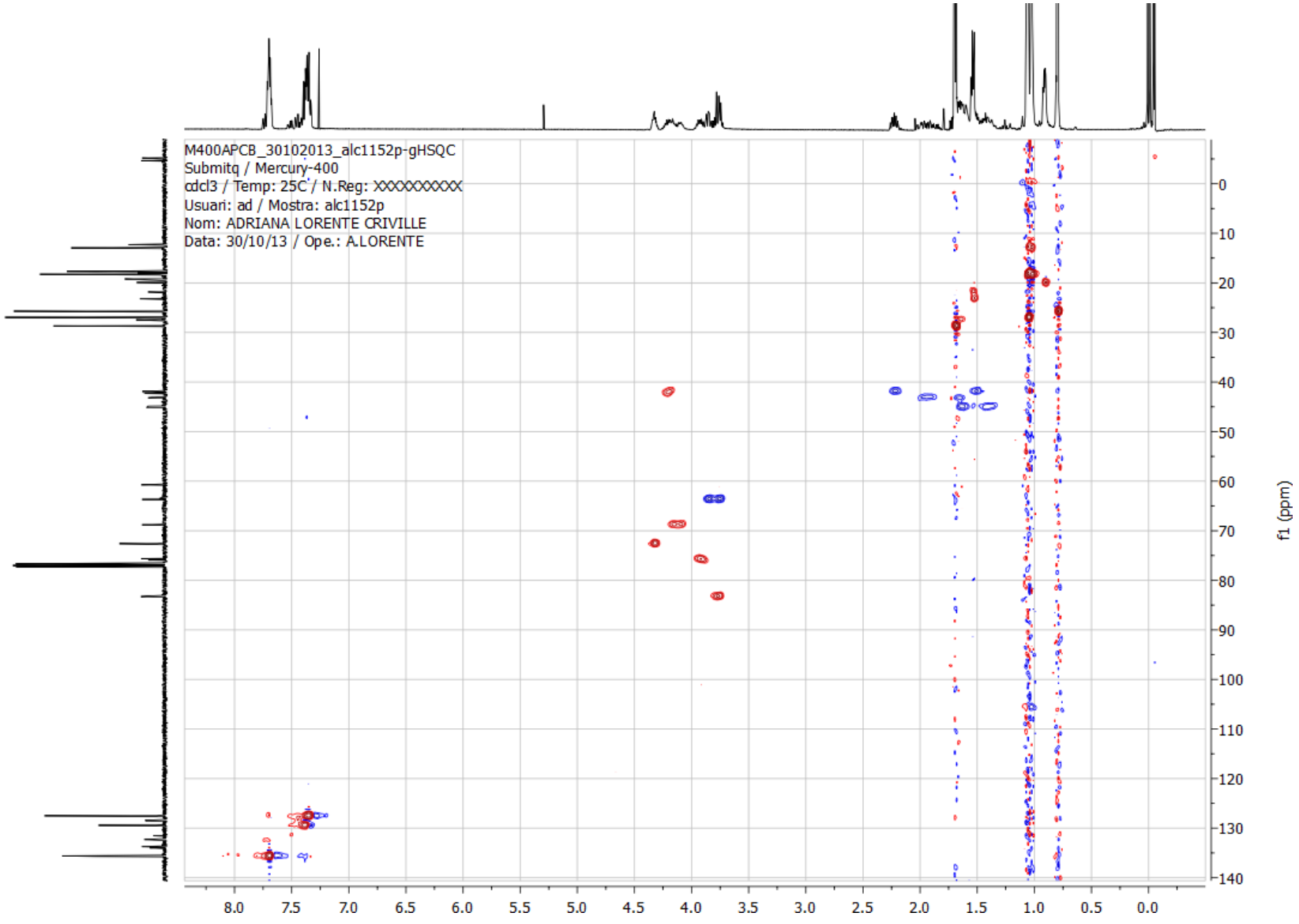

M400APCB_15022013_alc979ok-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc979ok
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
S
N
N
N
N
Ph
S8a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

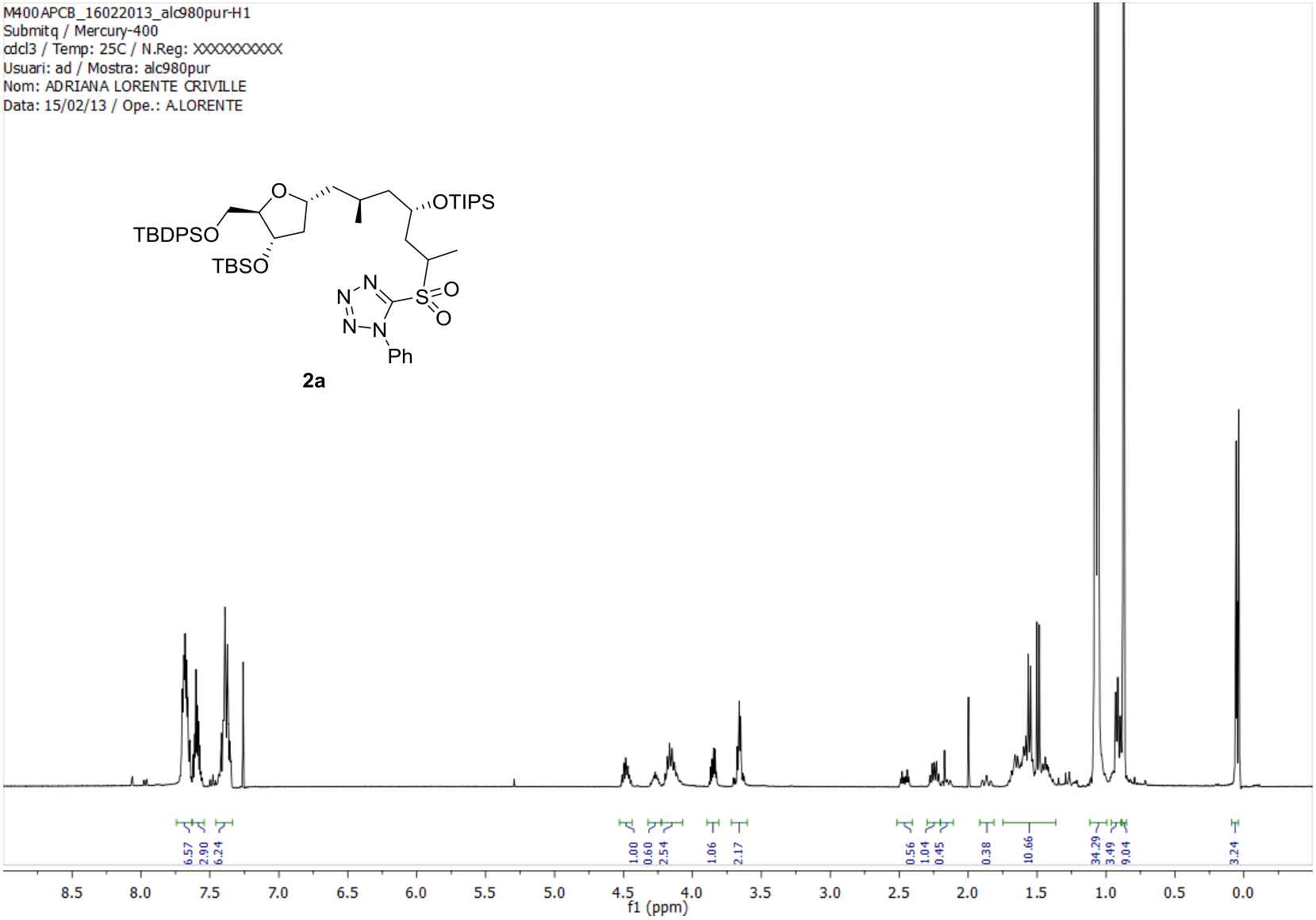

M400APCB_15022013_alc979ok-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc979ok
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

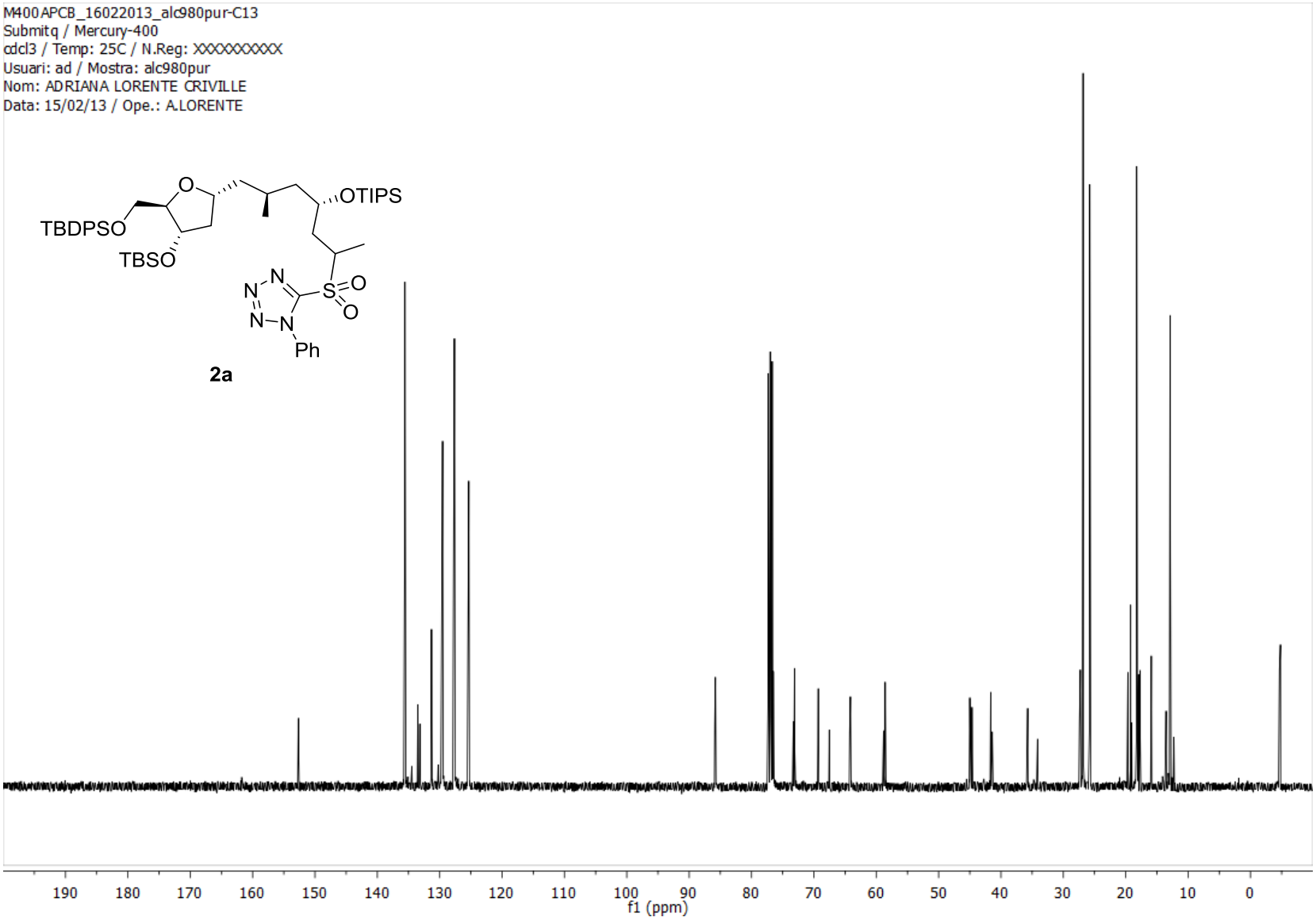

M400APCB_30102013_alc1152p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1152p
Nom: ADRIANA LORENTE CRIVILLE
Data: 30/10/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
S
tBu
4.69
6.41
1.00
2.19
0.96
1.08
2.09
1.03
1.55
10.34
4.61
4.63
36.90
3.15
9.17
2.92
2.55
8.5
8.0
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

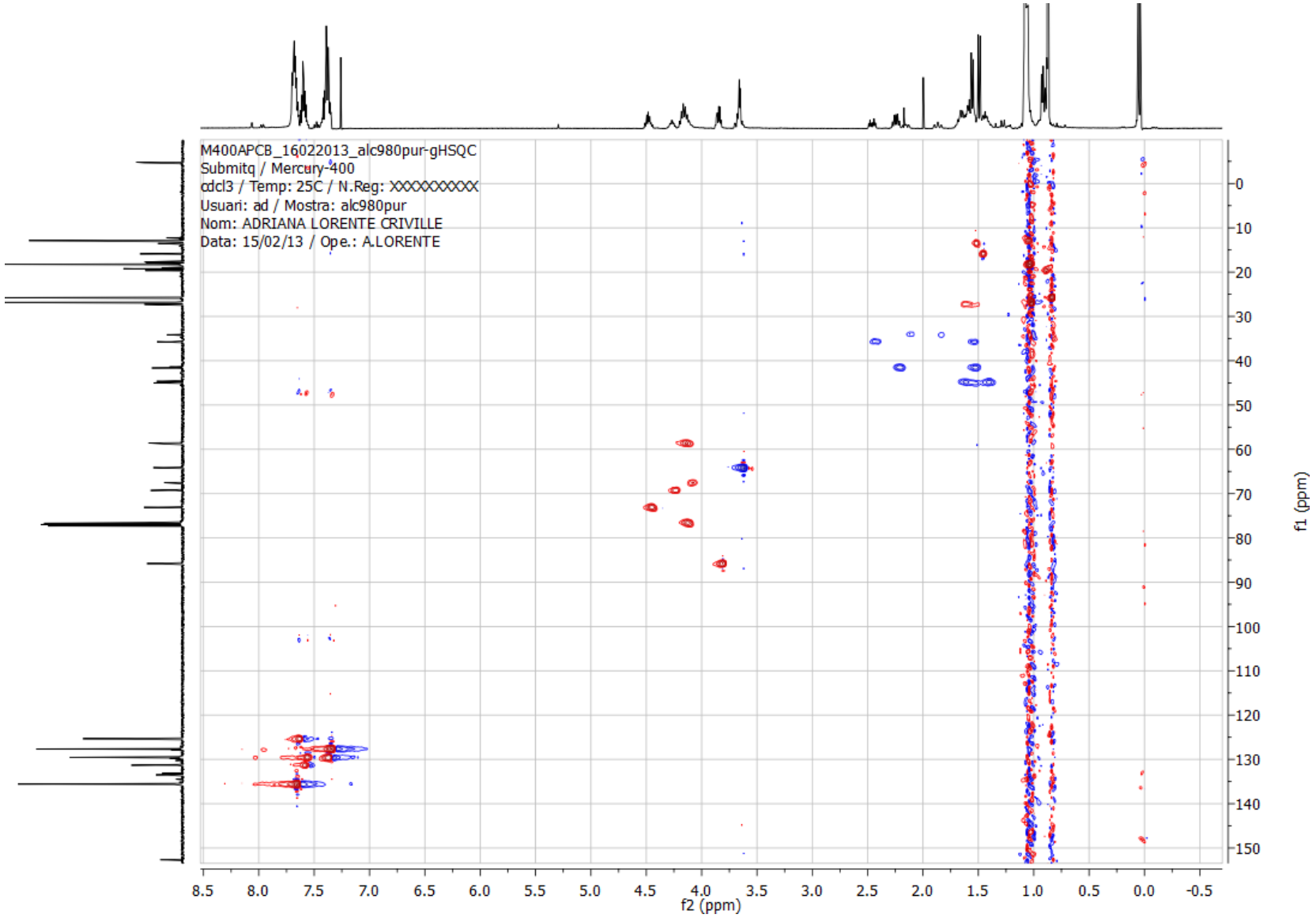
S8b

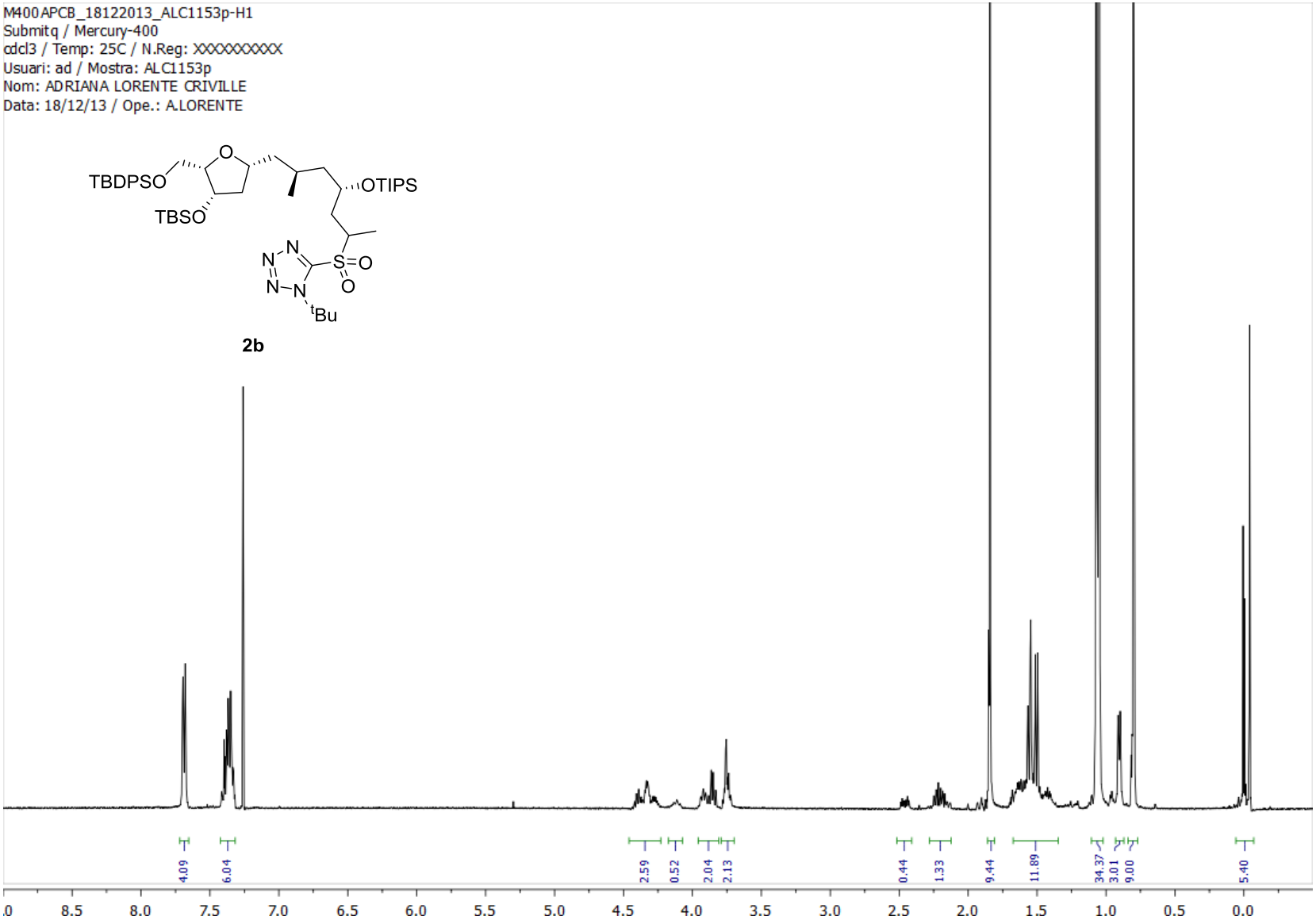

M400APCB_30102013_alc1152p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1152p
Nom: ADRIANA LORENTE CRIVILLE
Data: 30/10/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
S8b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

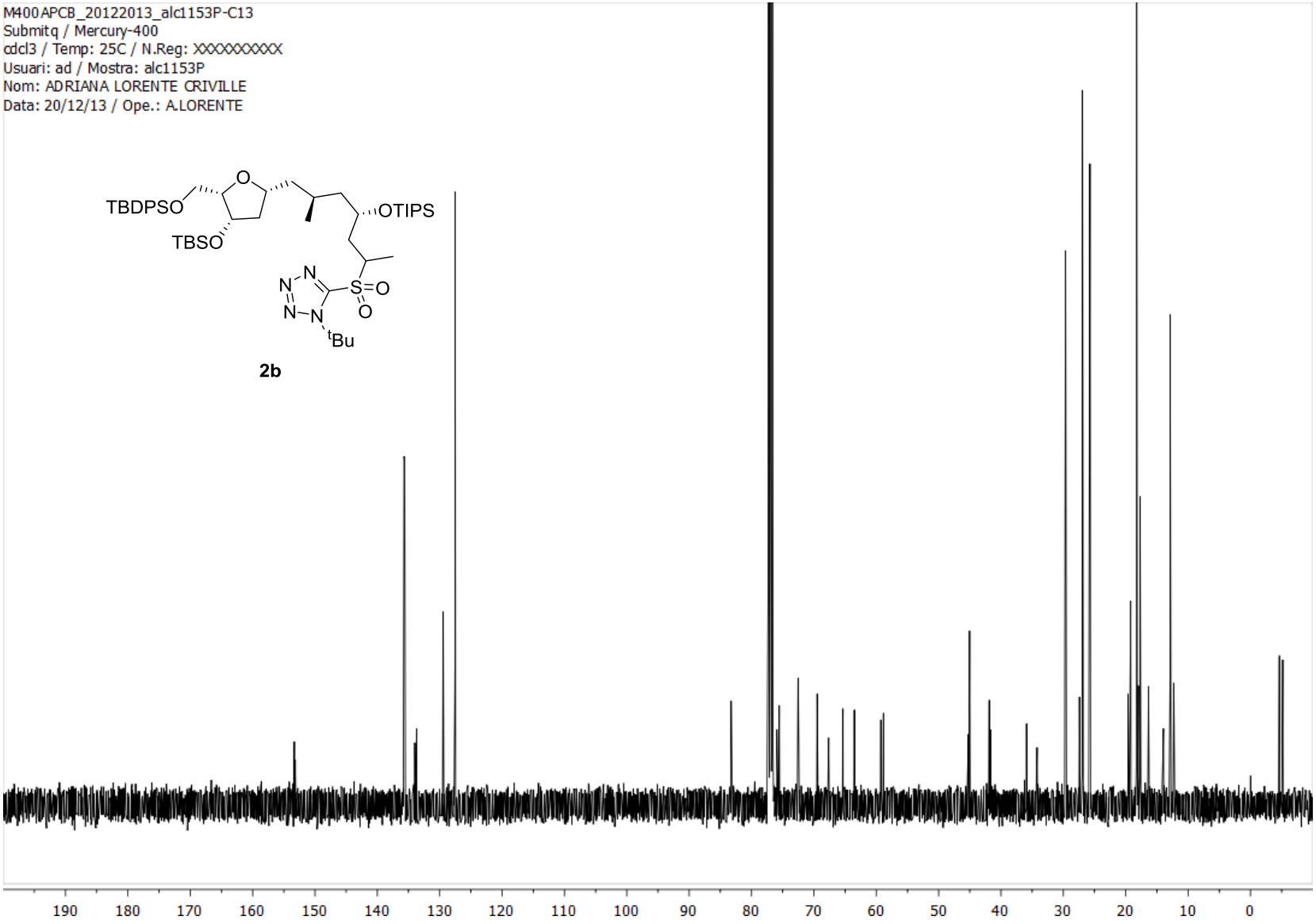

M400APCB_30102013_alc1152p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1152p
Nom: ADRIANA LORENTE CRIVILLE
Data: 30/10/13 / Ope.: A.LORENTE
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

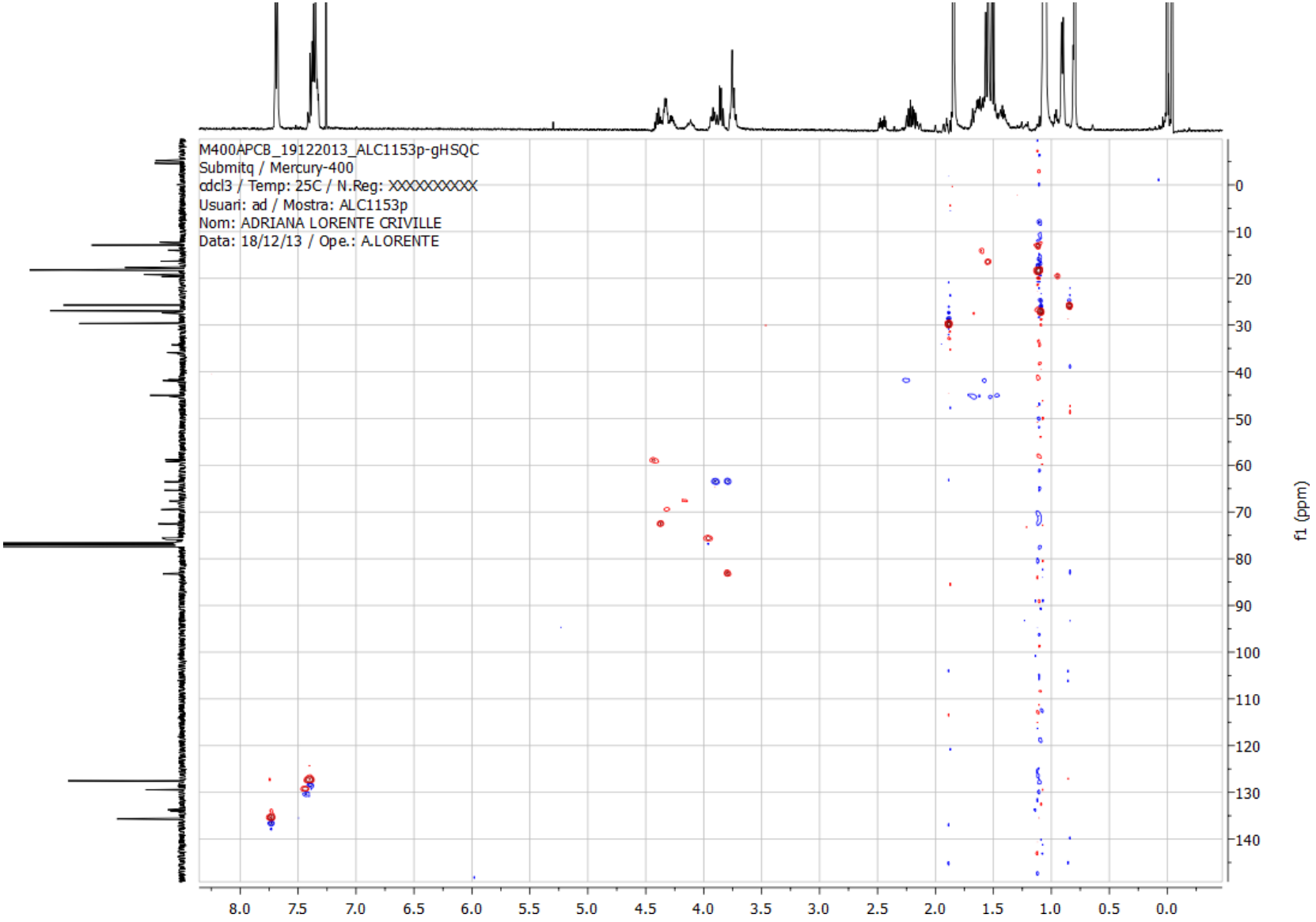

M400APCB_16022013_alc980pur-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc980pur
Nom: ADRIANA LORENTE CRIVILLE
Data: 15/02/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
Ph
2a
6.57
2.90
6.24
1.00
0.60
2.54
1.06
2.17
0.56
1.04
0.45
0.38
10.66
34.29
3.49
9.04
3.24
8.5
8.0
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)

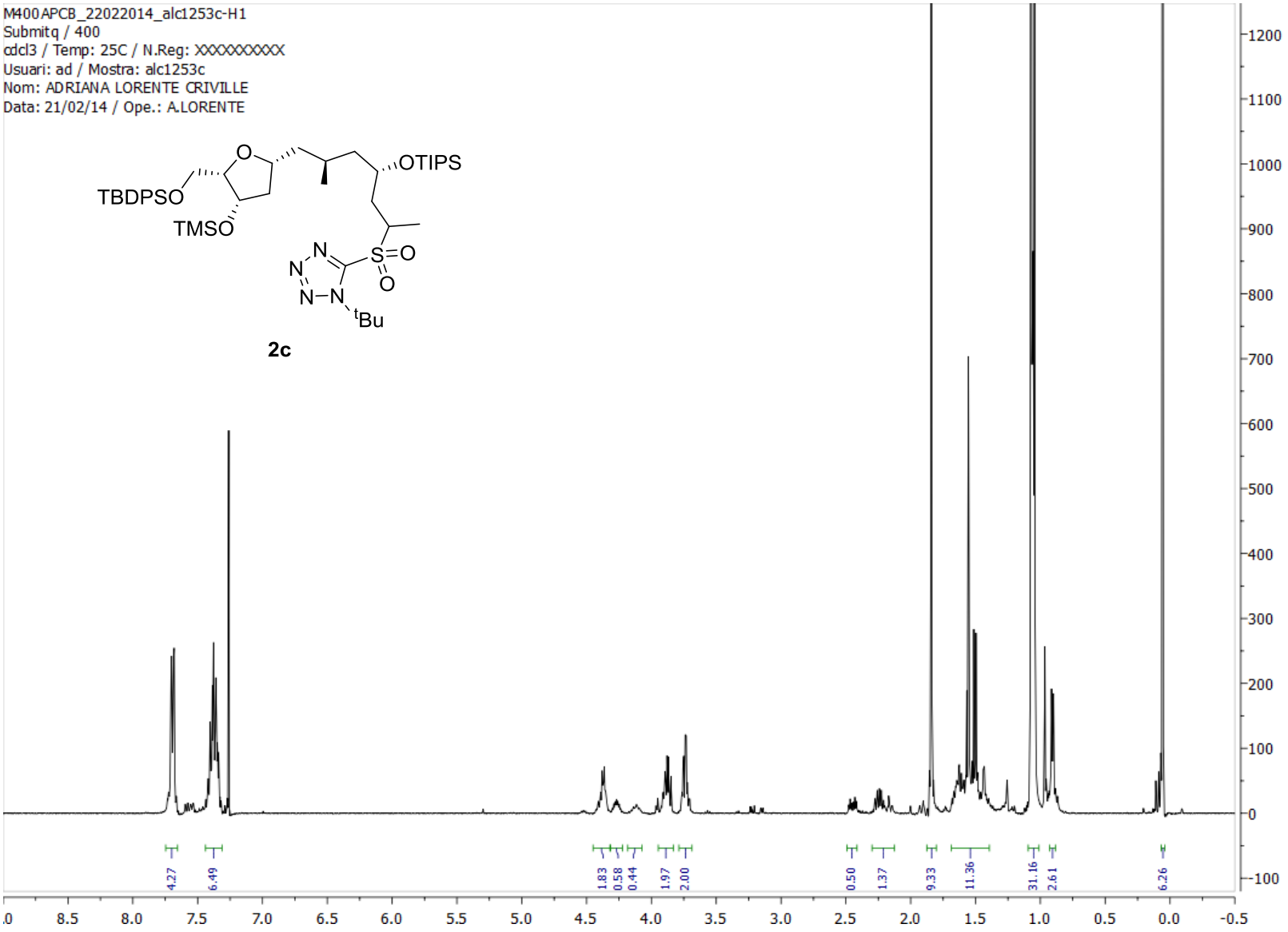

M400APCB_16022013_alc980pur-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc980pur
Nom: ADRIANA LORENTE CRIVILLE
Data: 15/02/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
N
N
N
N
S
O
O
Ph
2a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

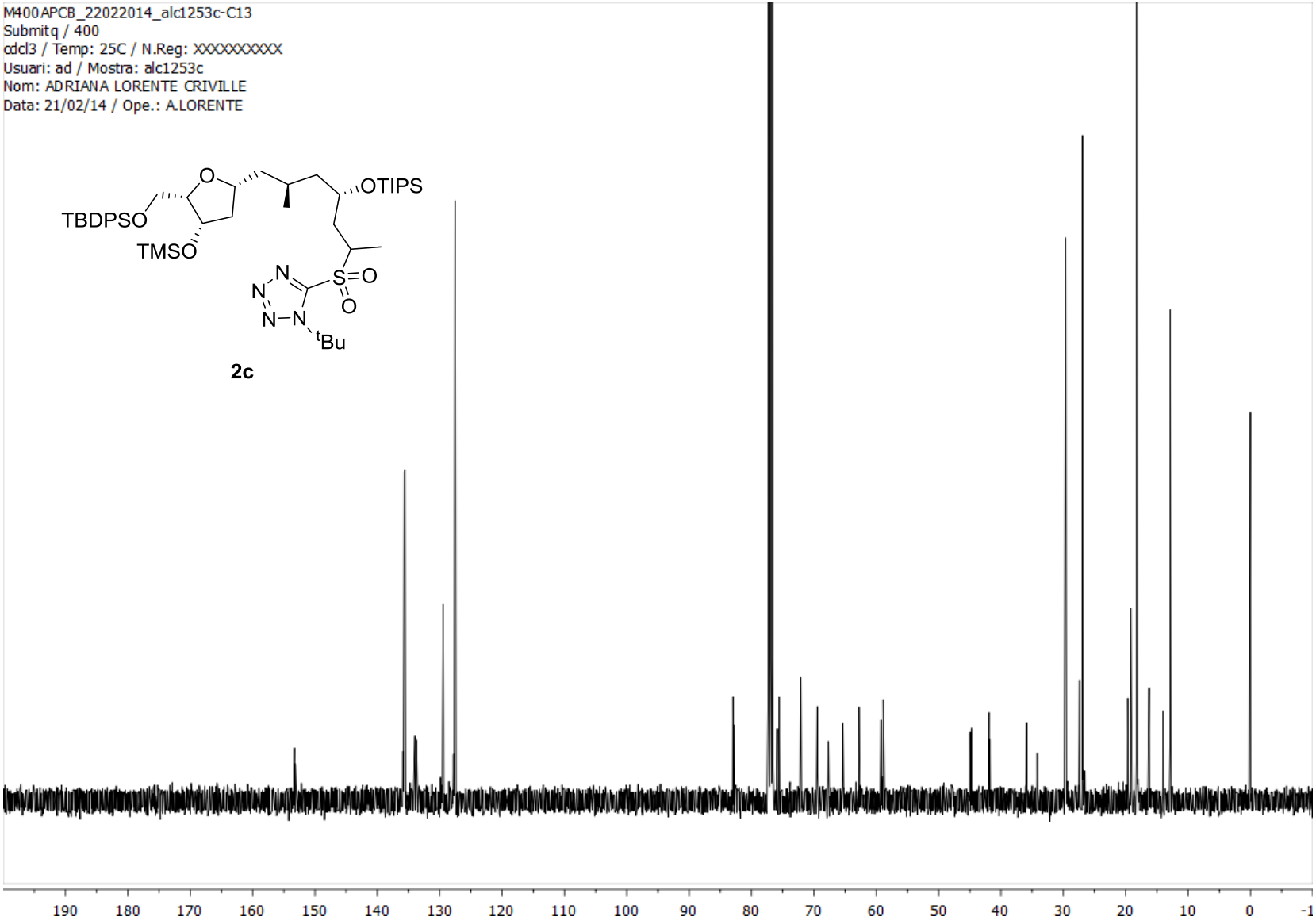

M400APCB_16022013_alc980pur-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc980pur
Nom: ADRIANA LORENTE CRIVILLE
Data: 15/02/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

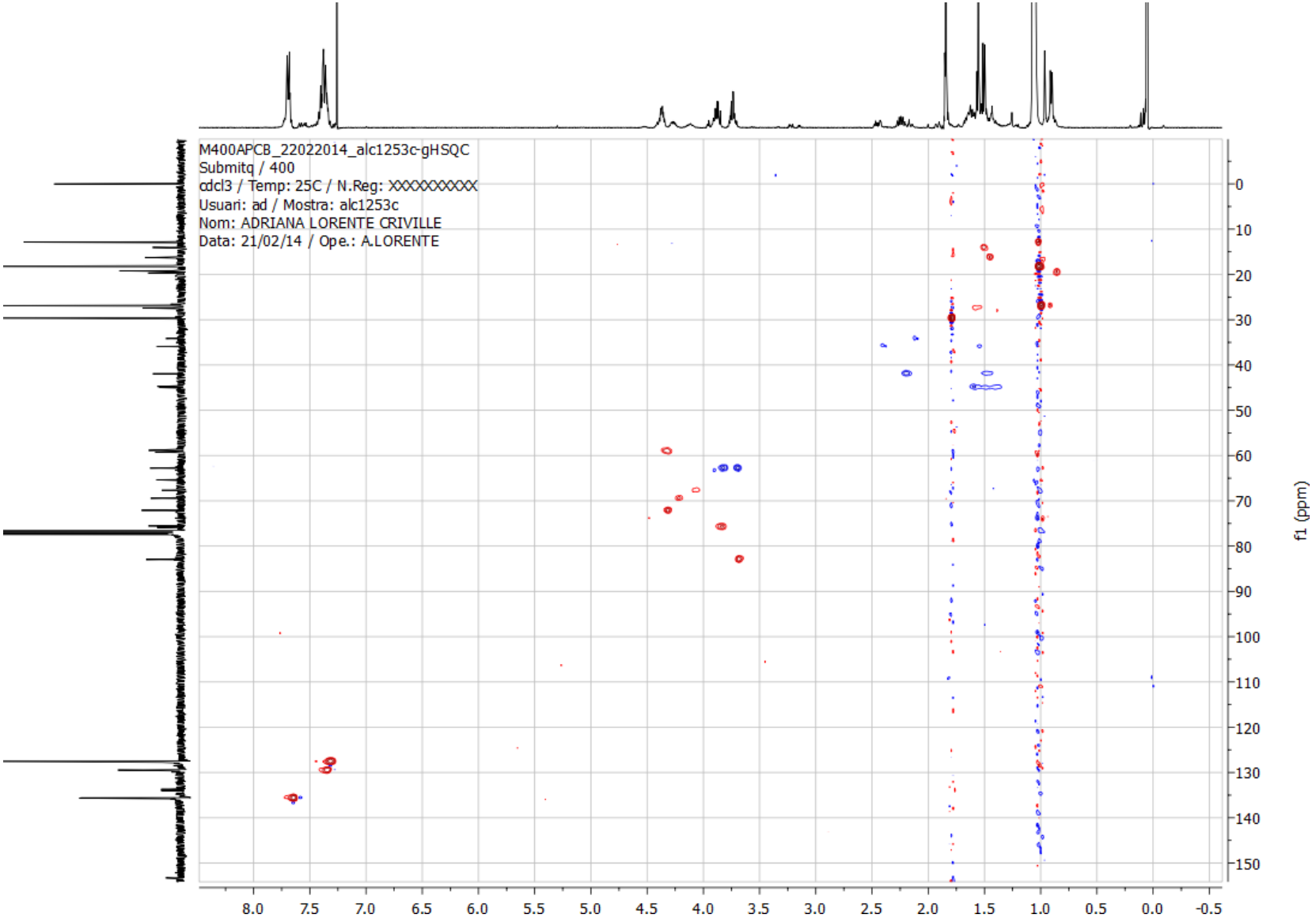

M400APCB_18122013_ALC1153p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC1153p
Nom: ADRIANA LORENTE CRIVILLE
Data: 18/12/13 / Ope.: ALORENTE
TBDPSO
TBSO
OTIPS
N
N
N
N
S
O
O
tBu
2b
4.09
6.04
2.59
0.52
2.04
2.13
0.44
1.33
9.44
11.89
34.37
3.01
9.00
5.40
8.5
8.0
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

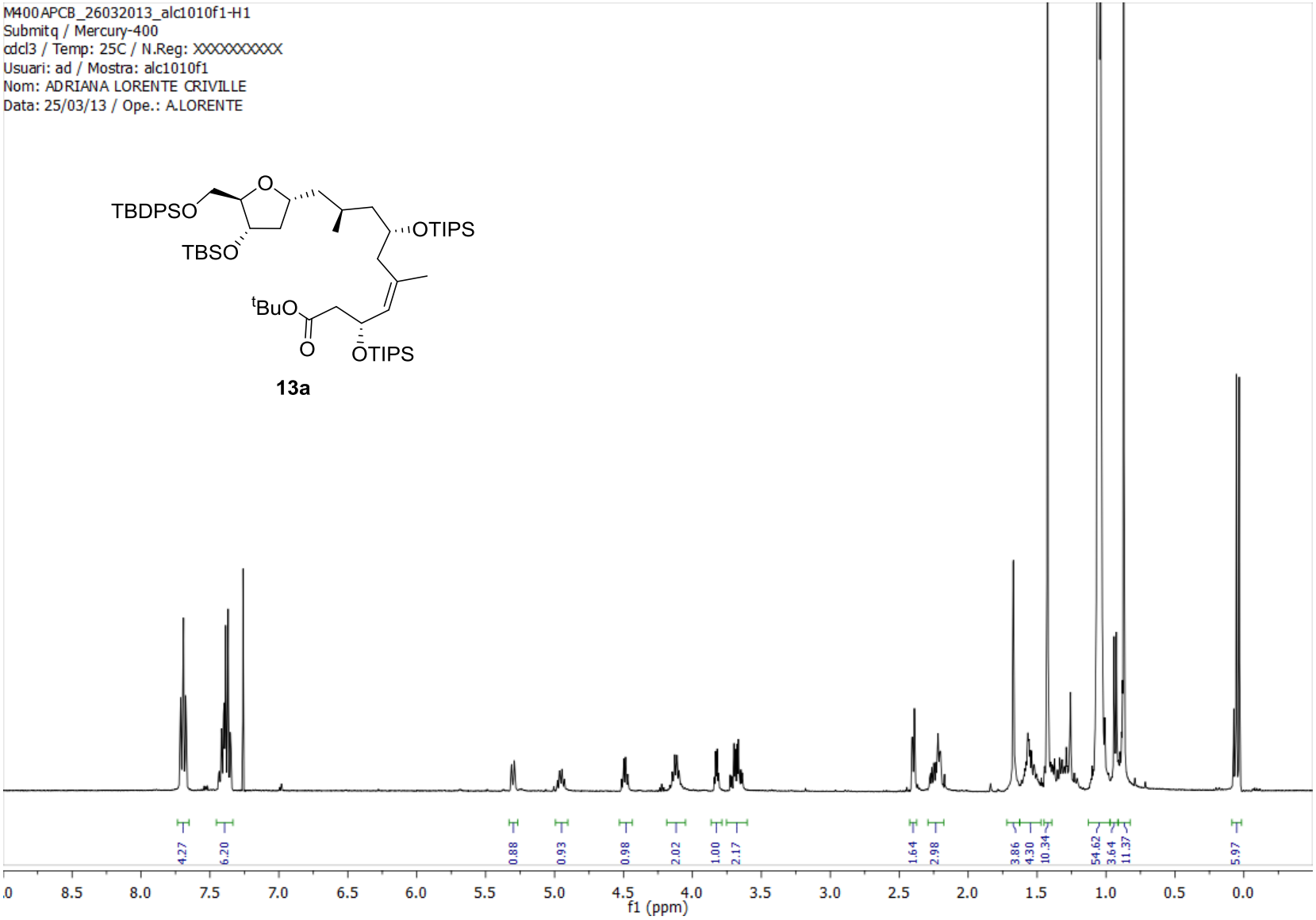

M400APCB_20122013_alc1153P-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1153P
Nom: ADRIANA LORENTE CRIVILLE
Data: 20/12/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
N
N
N
N
S=O
O
tBu
2b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

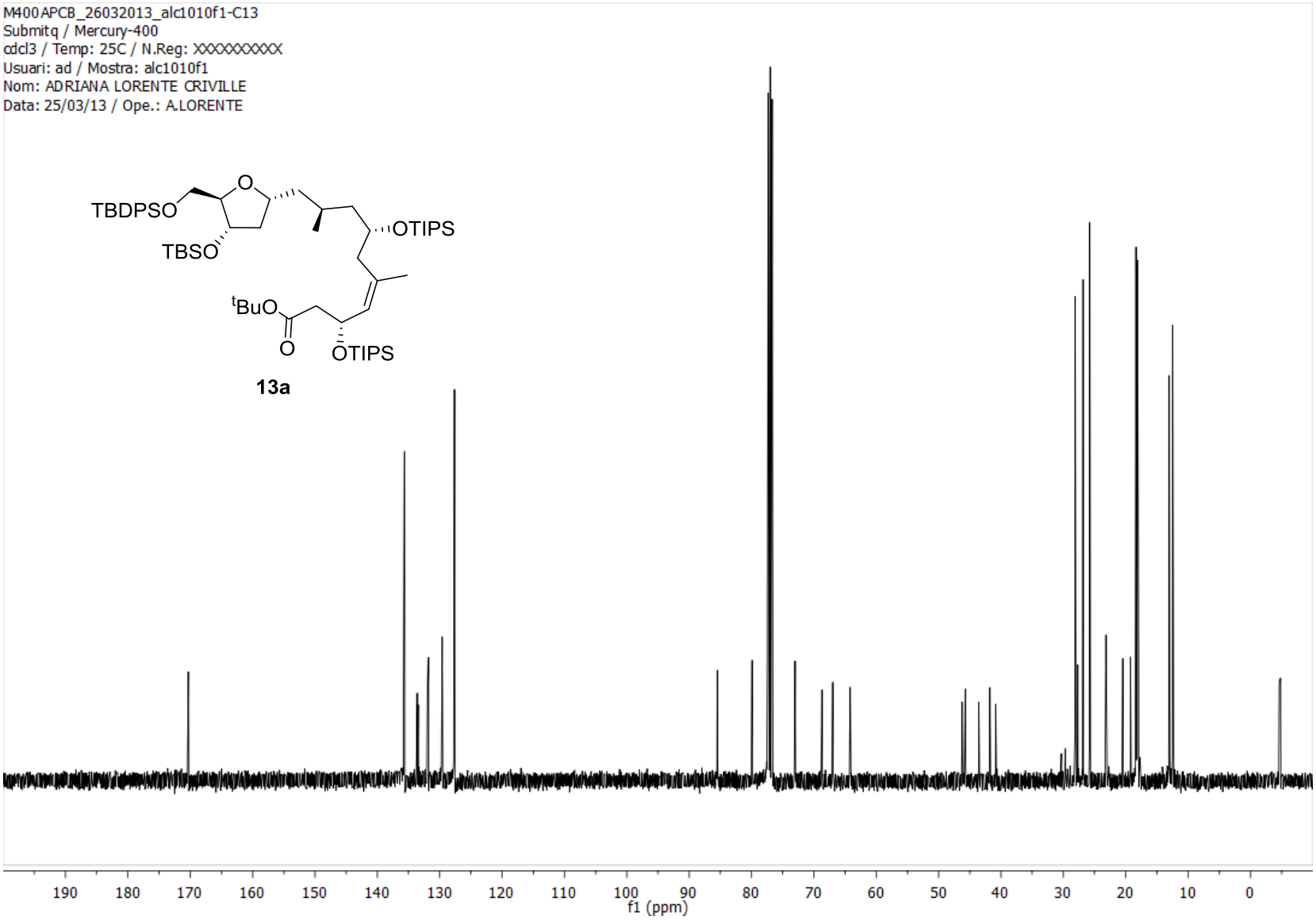

M400APCB_19122013_ALC1153p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC1153p
Nom: ADRIANA LORENTE CRIVILLE
Data: 18/12/13 / Ope.: A.LORENTE
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

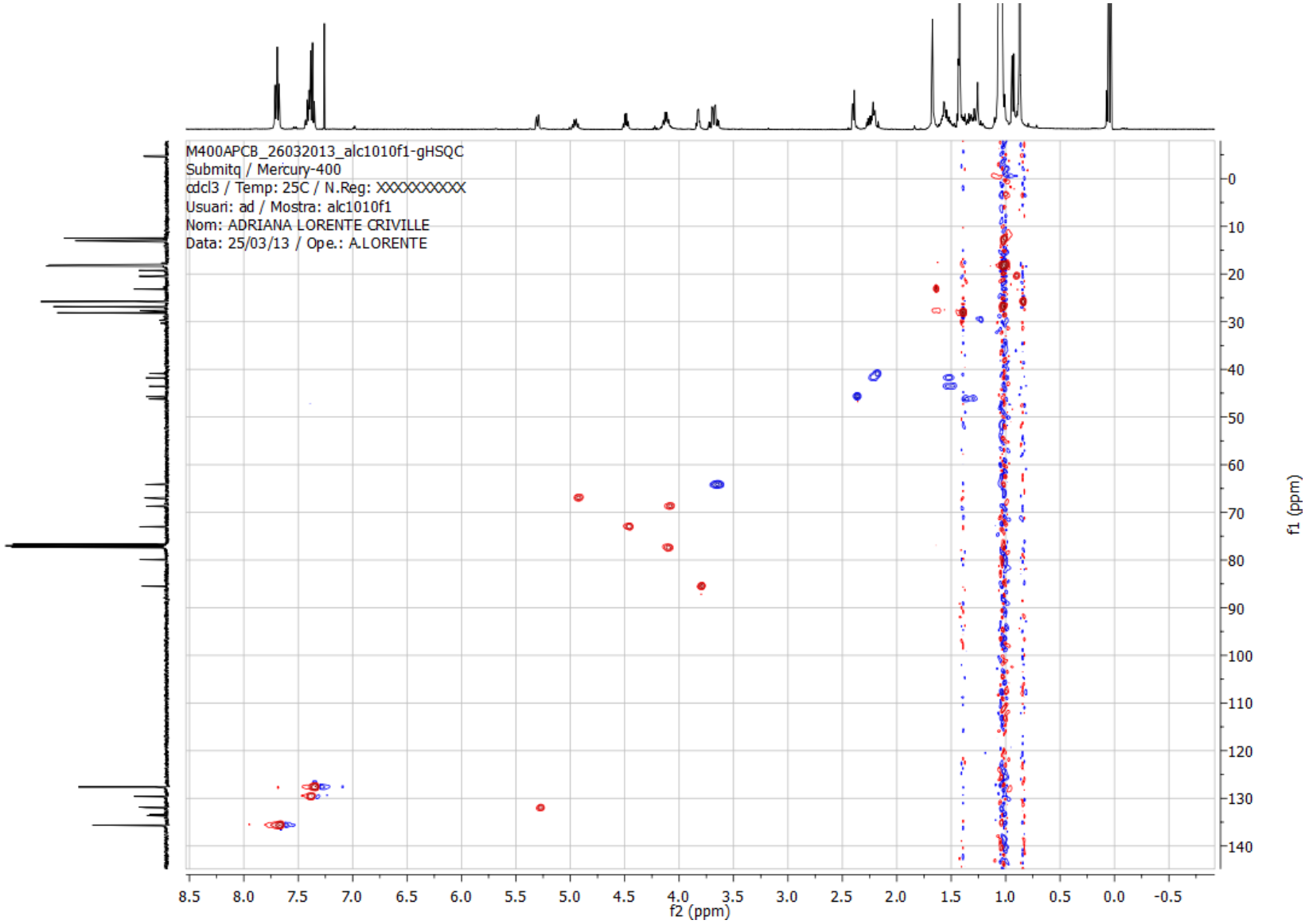

M400APCB_22022014_alc1253c-H1
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1253c
Nom: ADRIANA LORENTE CRIVILLE
Data: 21/02/14 / Ope.: A.LORENTE
TBDPSO
TMSO
OTIPS
N
N
N
S
O
O
tBu
2c
4.27
6.49
1.83
0.58
0.44
1.97
2.00
0.50
1.37
9.33
11.36
31.16
2.61
6.26

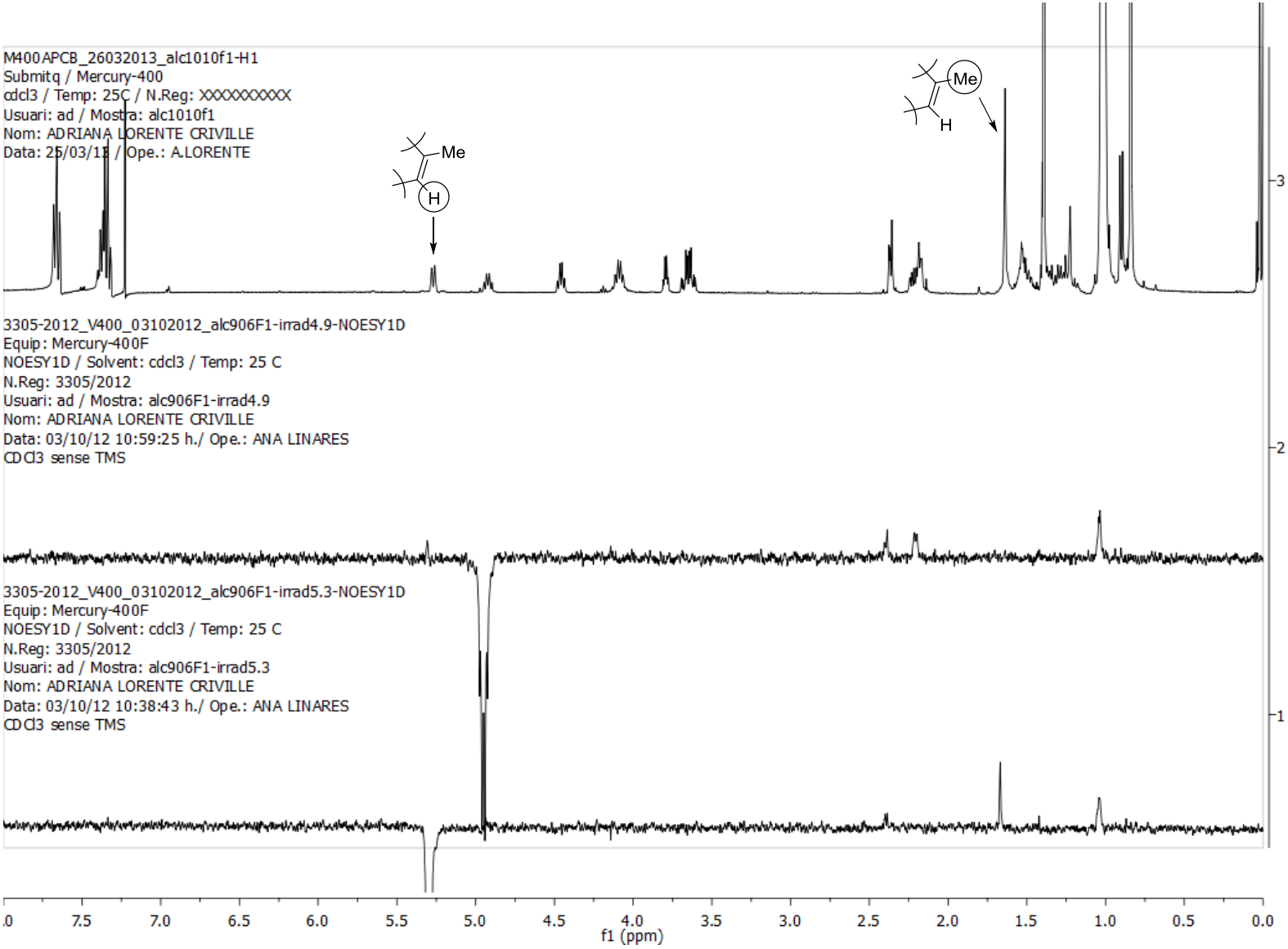

M400APCB_22022014_alc1253c-C13
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1253c
Nom: ADRIANA LORENTE CRIVILLE
Data: 21/02/14 / Ope.: A.LORENTE
TBDPSO
TMSO
OTIPS
tBu
2c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1

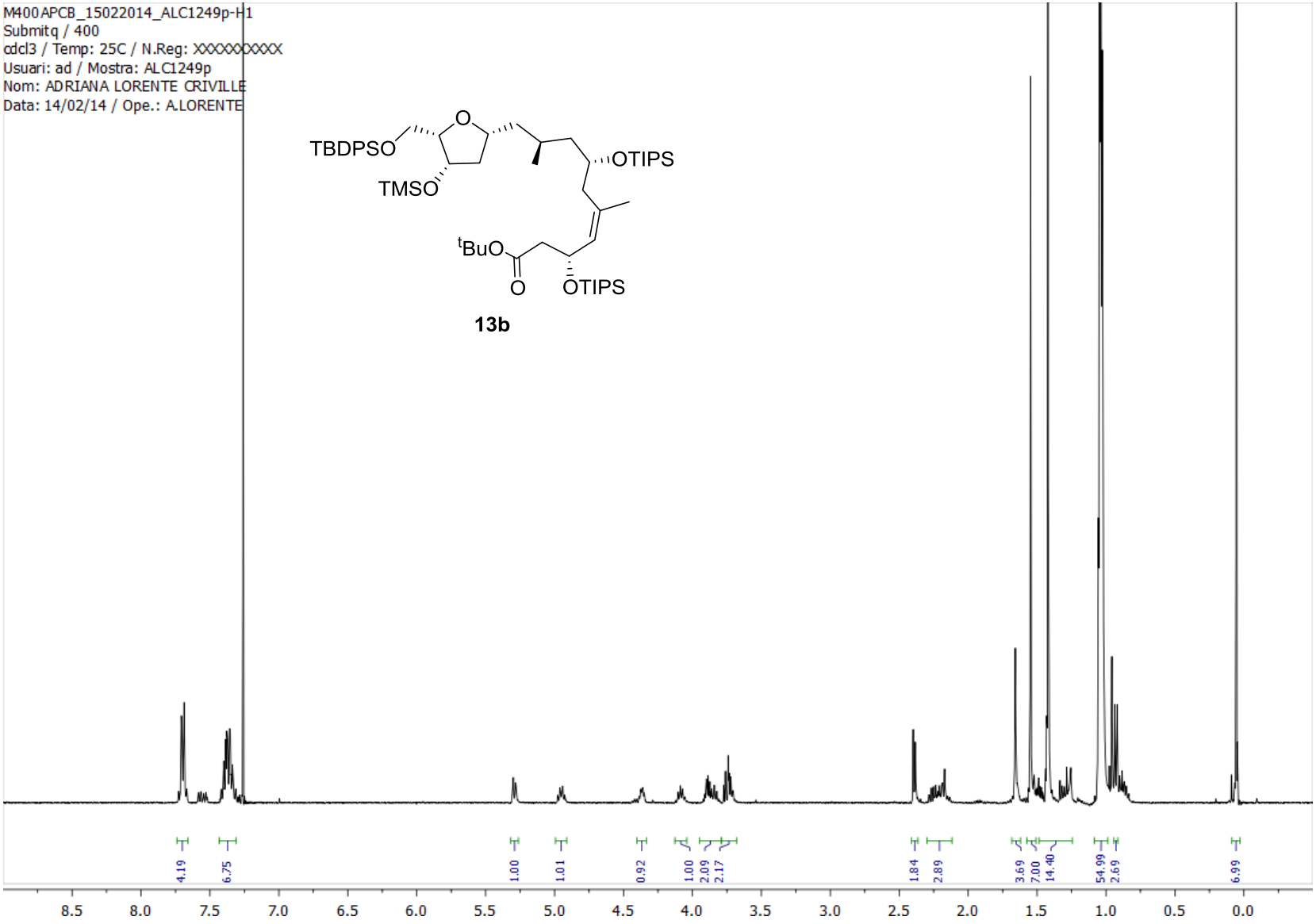

M400APCB_22022014_alc1253c-gHSQC
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1253c
Nom: ADRIANA LORENTE CRIVILLE
Data: 21/02/14 / Ope.: A.LORENTE
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

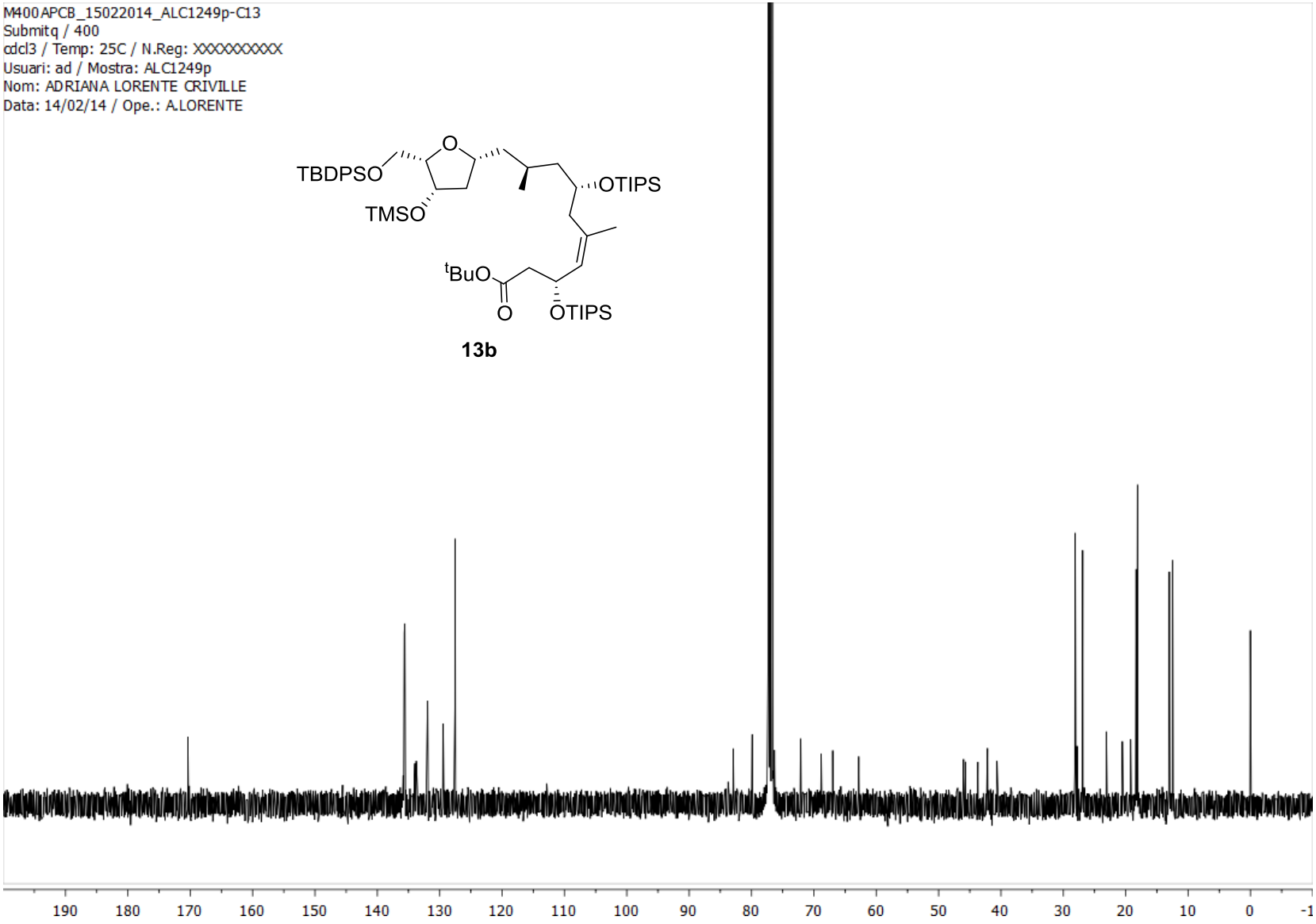

M400APCB_26032013_alc1010f1-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1010f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
tBuO
O
OTIPS
13a
4.27
6.20
0.88
0.93
0.98
2.02
1.00
2.17
1.64
2.98
3.86
4.30
10.34
54.62
3.64
11.37
5.97
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)

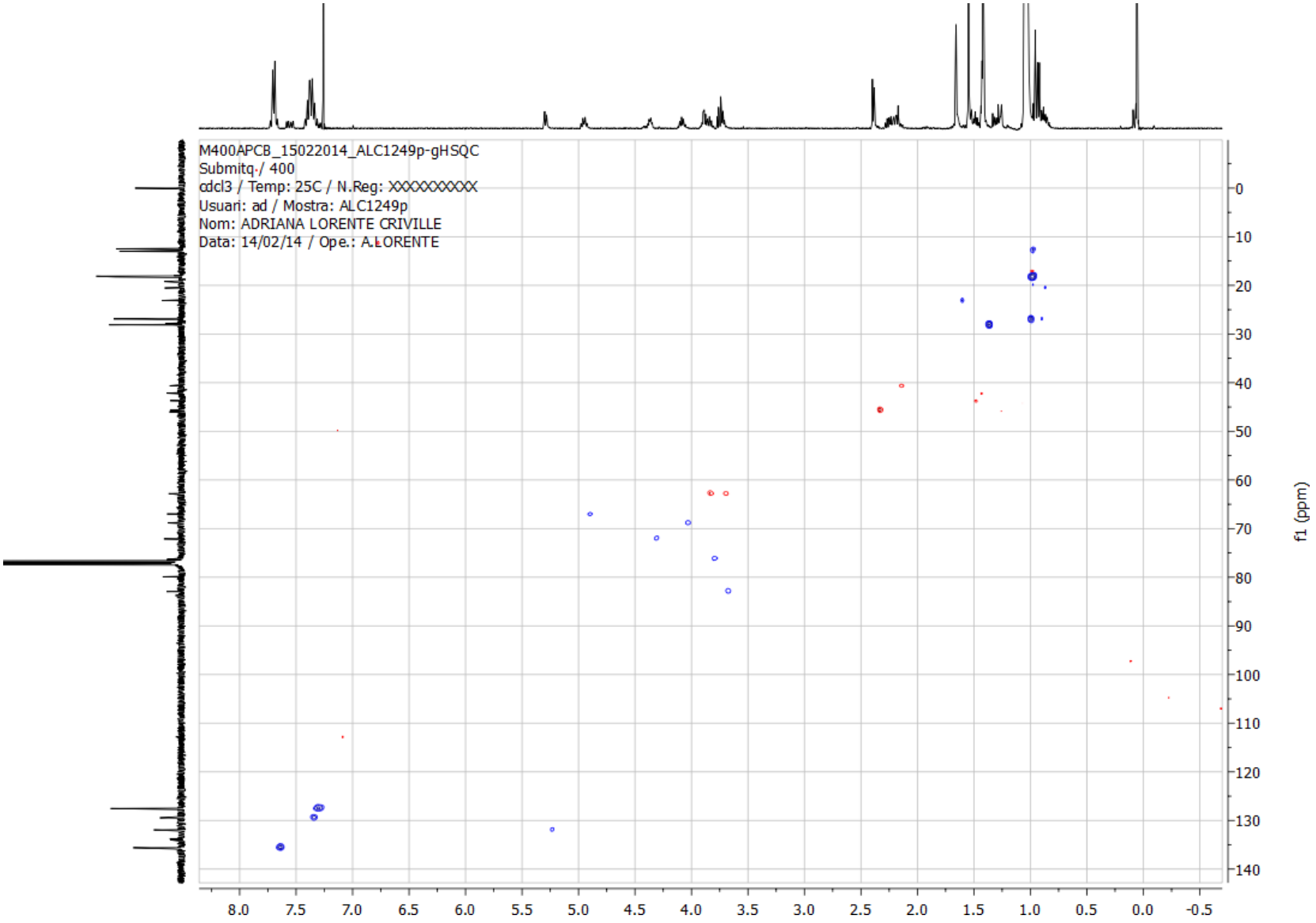

M400APCB_26032013_alc1010f1-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1010f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
TBDPSO
TBSO
OTIPS
tBuO
O
OTIPS
13a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

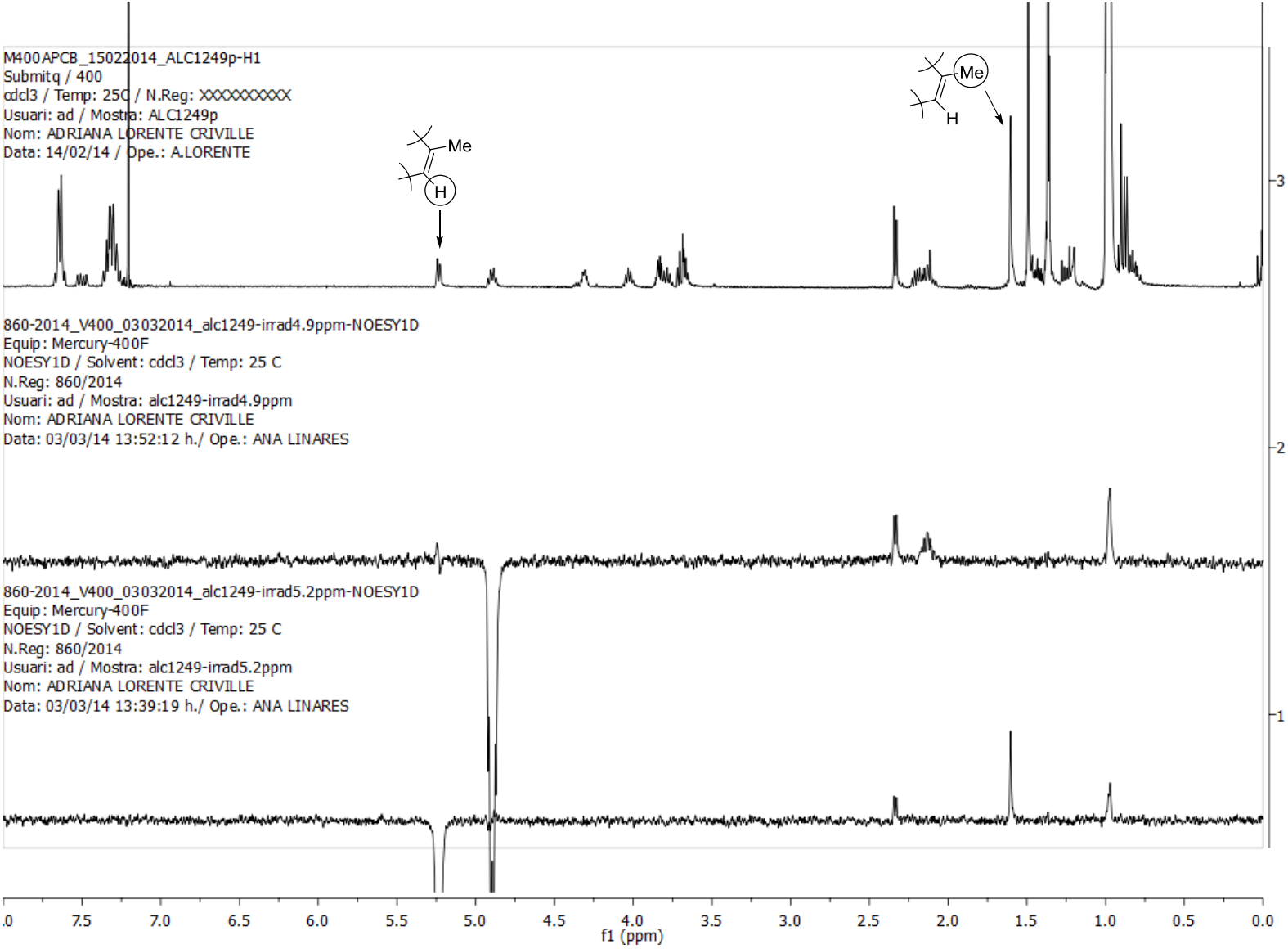

M400APCB_26032013_alc1010f1-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1010f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

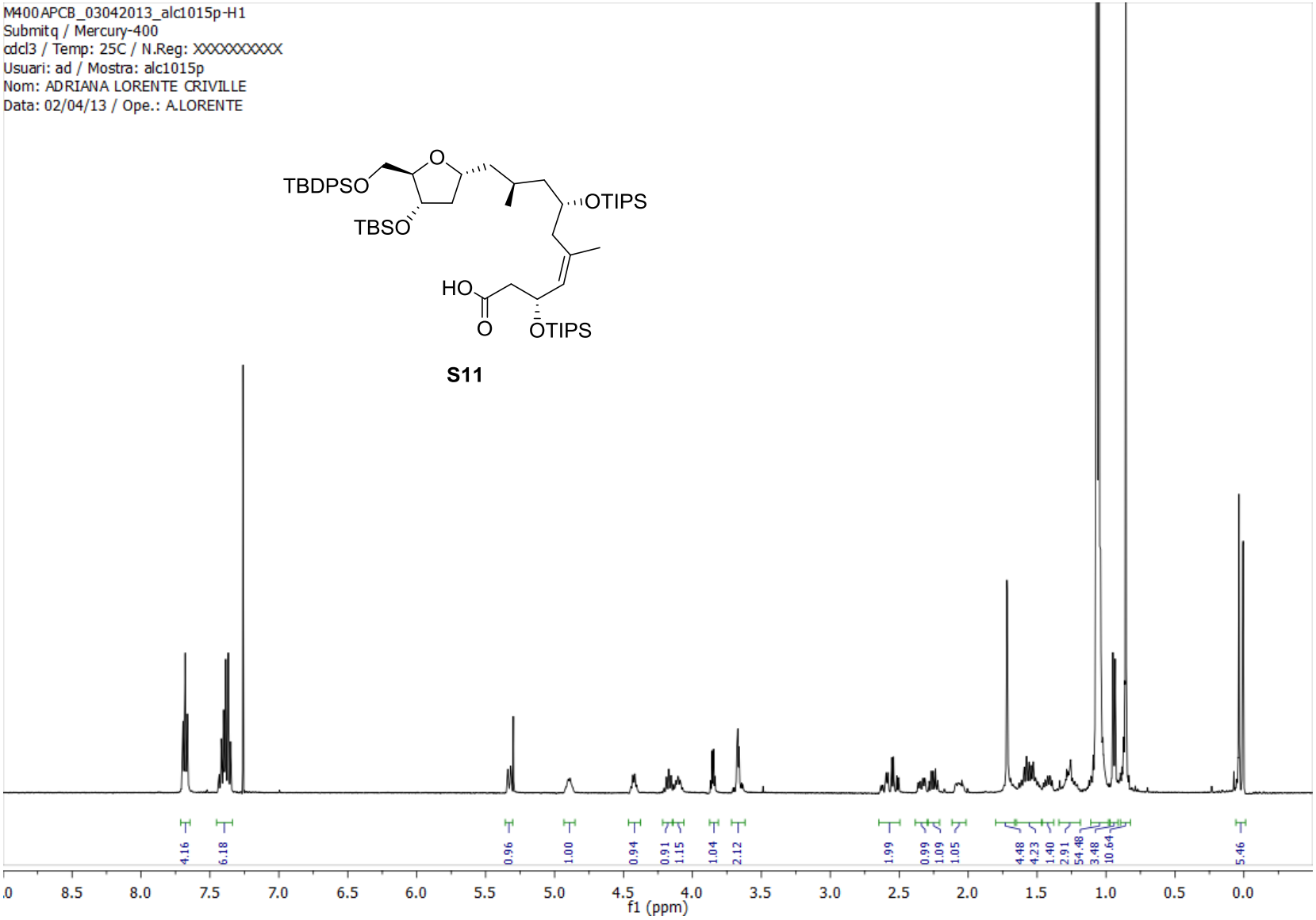

M400APCB_26032013_alc1010f1-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1010f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
Me
H
Me
H
3305-2012_V400_03102012_alc906F1-irrad4.9-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 3305/2012
Usuari: ad / Mostra: alc906F1-irrad4.9
Nom: ADRIANA LORENTE CRIVILLE
Data: 03/10/12 10:59:25 h./ Ope.: ANA LINARES
CDCl3 sense TMS
3305-2012_V400_03102012_alc906F1-irrad5.3-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 3305/2012
Usuari: ad / Mostra: alc906F1-irrad5.3
Nom: ADRIANA LORENTE CRIVILLE
Data: 03/10/12 10:38:43 h./ Ope.: ANA LINARES
CDCl3 sense TMS
3
2
1
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)


M400APCB_15022014_ALC1249p-H1
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: ad / Mostra: ALC1249p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
TBDPSO
TMSO
O
OTIPS
tBuO
O
OTIPS
13b
4.19
6.75
1.00
1.01
0.92
1.00
2.09
2.17
1.84
2.89
3.69
7.00
14.40
54.99
2.69
6.99
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

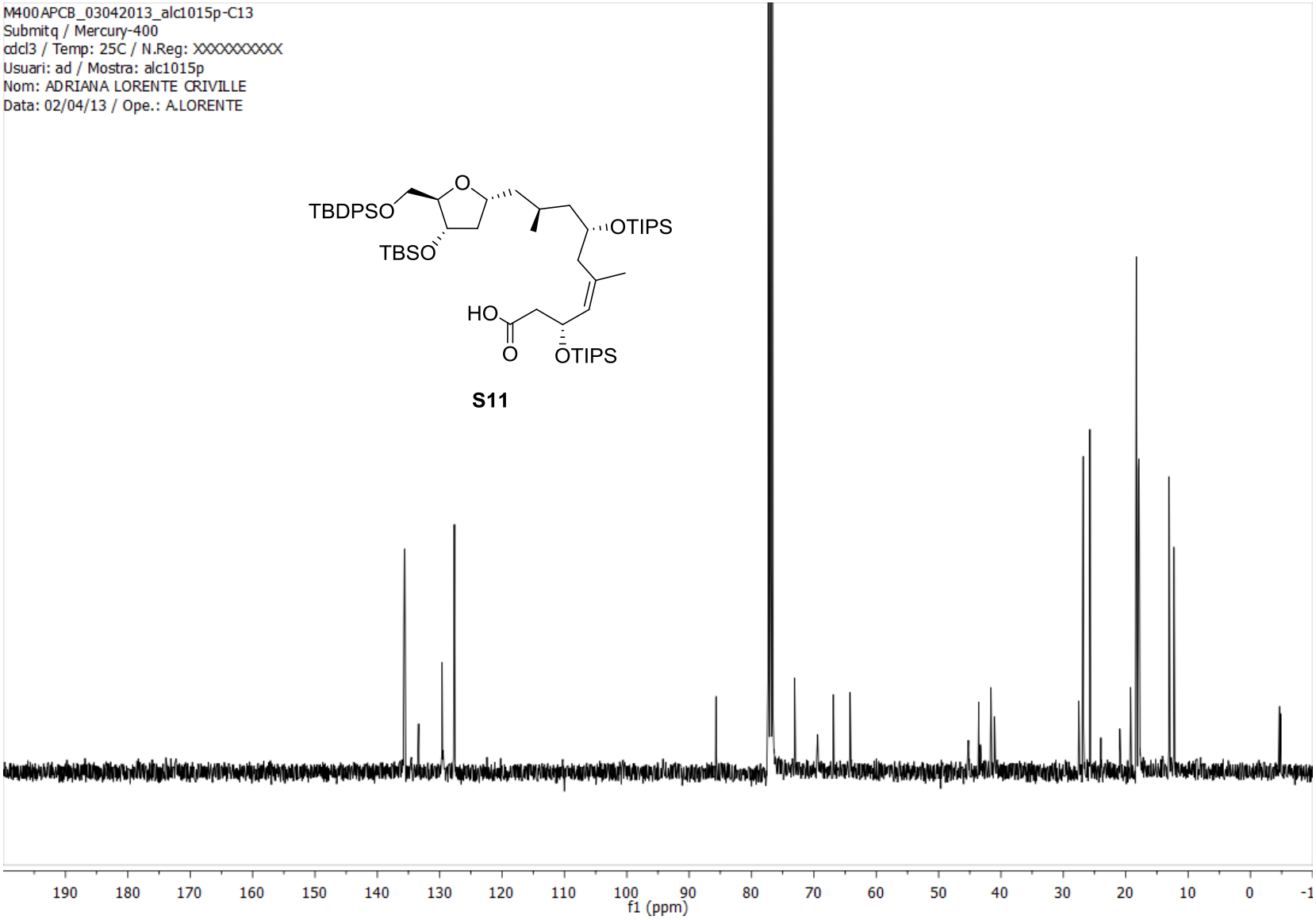

M400APCB_15022014_ALC1249p-C13
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC1249p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
TBDPSO
TMSO
OTIPS
tBuO
O
OTIPS
13b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1

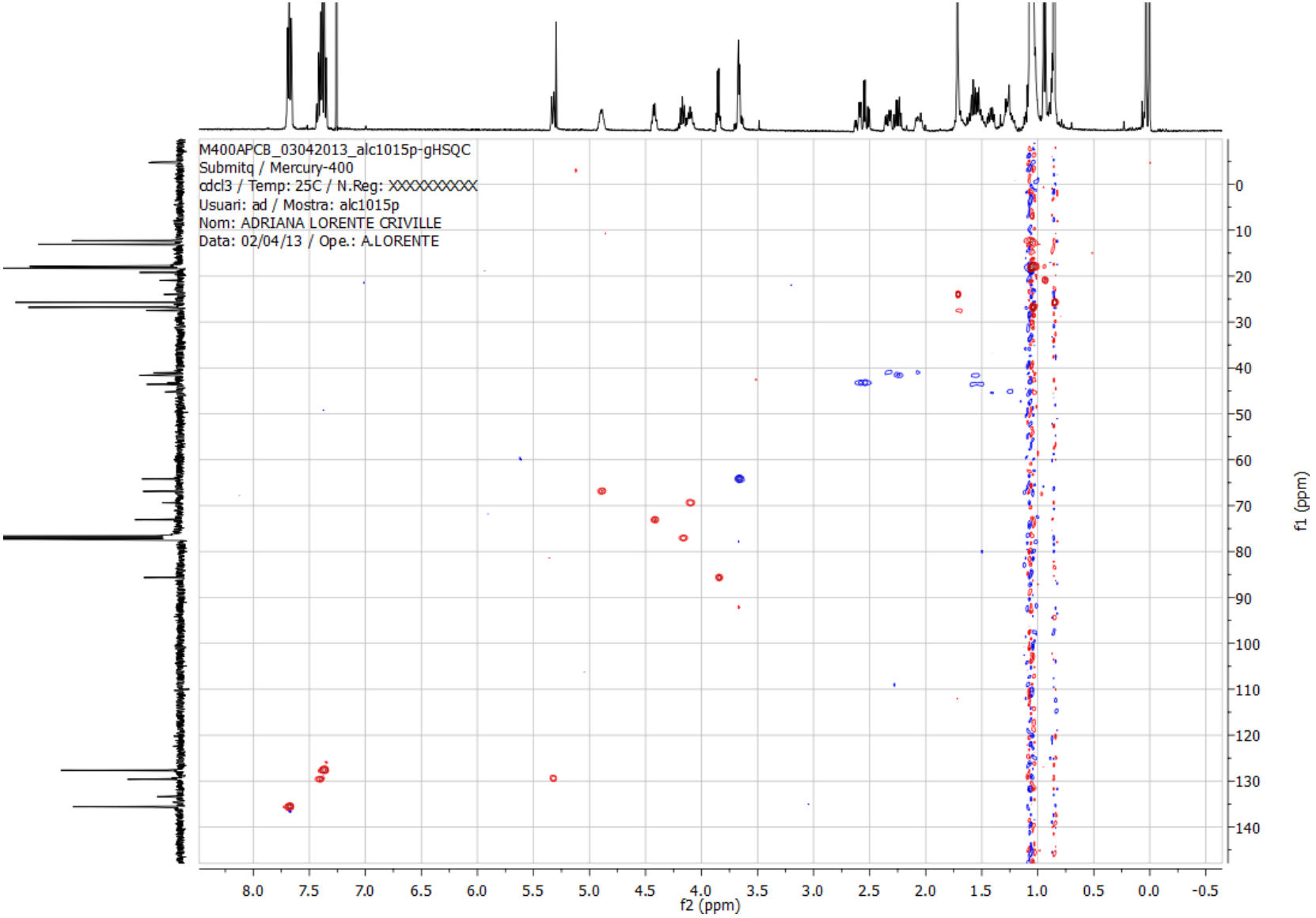

M400APCB_15022014_ALC1249p-gHSQC
Submitq./ 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC1249p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

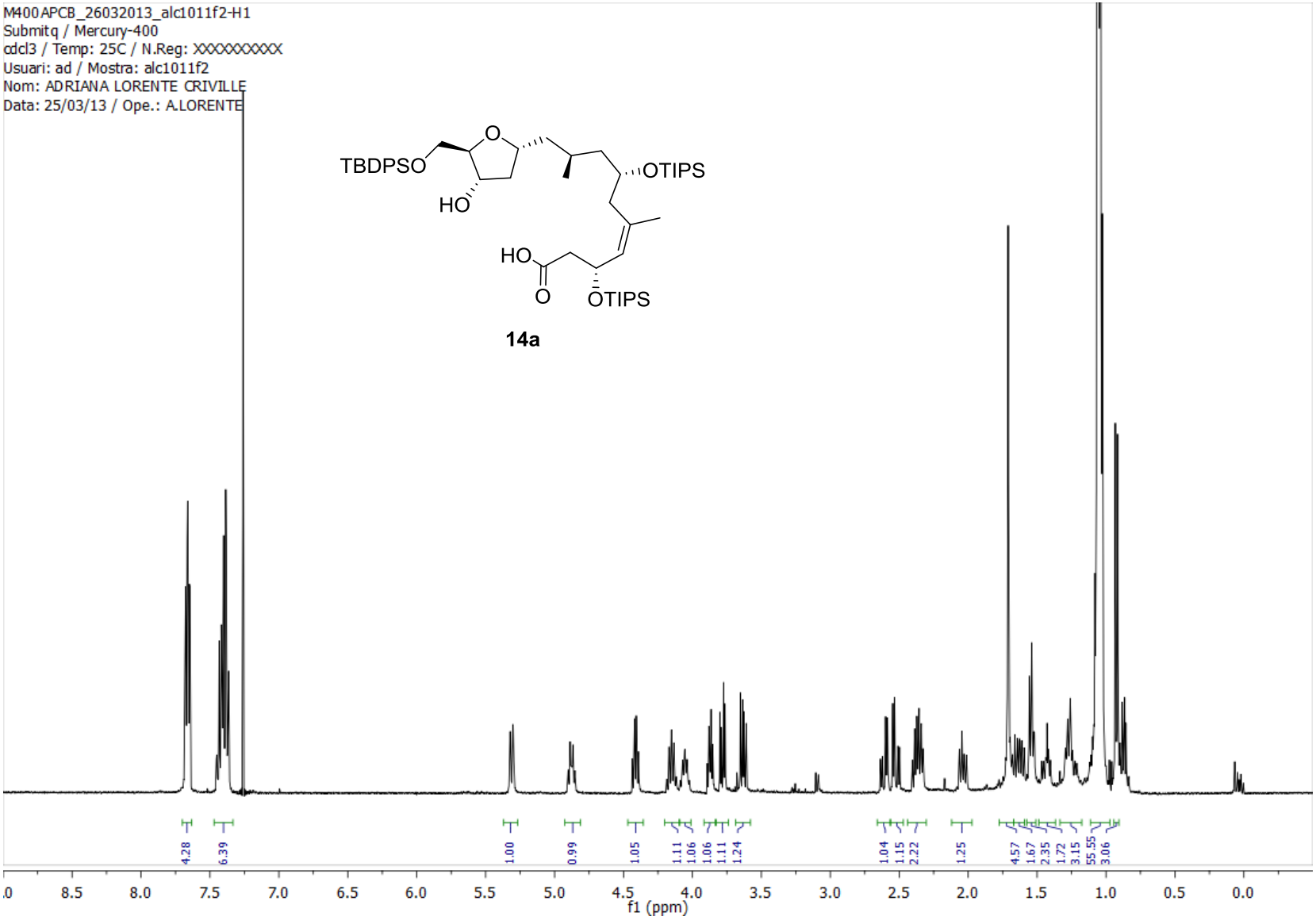

M400APCB_15022014_ALC1249p-H1
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: ALC1249p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
Me
H
Me
H
860-2014_V400_03032014_alc1249-irrad4.9ppm-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 860/2014
Usuari: ad / Mostra: alc1249-irrad4.9ppm
Nom: ADRIANA LORENTE CRIVILLE
Data: 03/03/14 13:52:12 h./ Ope.: ANA LINARES
860-2014_V400_03032014_alc1249-irrad5.2ppm-NOESY1D
Equip: Mercury-400F
NOESY1D / Solvent: cdcl3 / Temp: 25 C
N.Reg: 860/2014
Usuari: ad / Mostra: alc1249-irrad5.2ppm
Nom: ADRIANA LORENTE CRIVILLE
Data: 03/03/14 13:39:19 h./ Ope.: ANA LINARES
3
2
1
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)

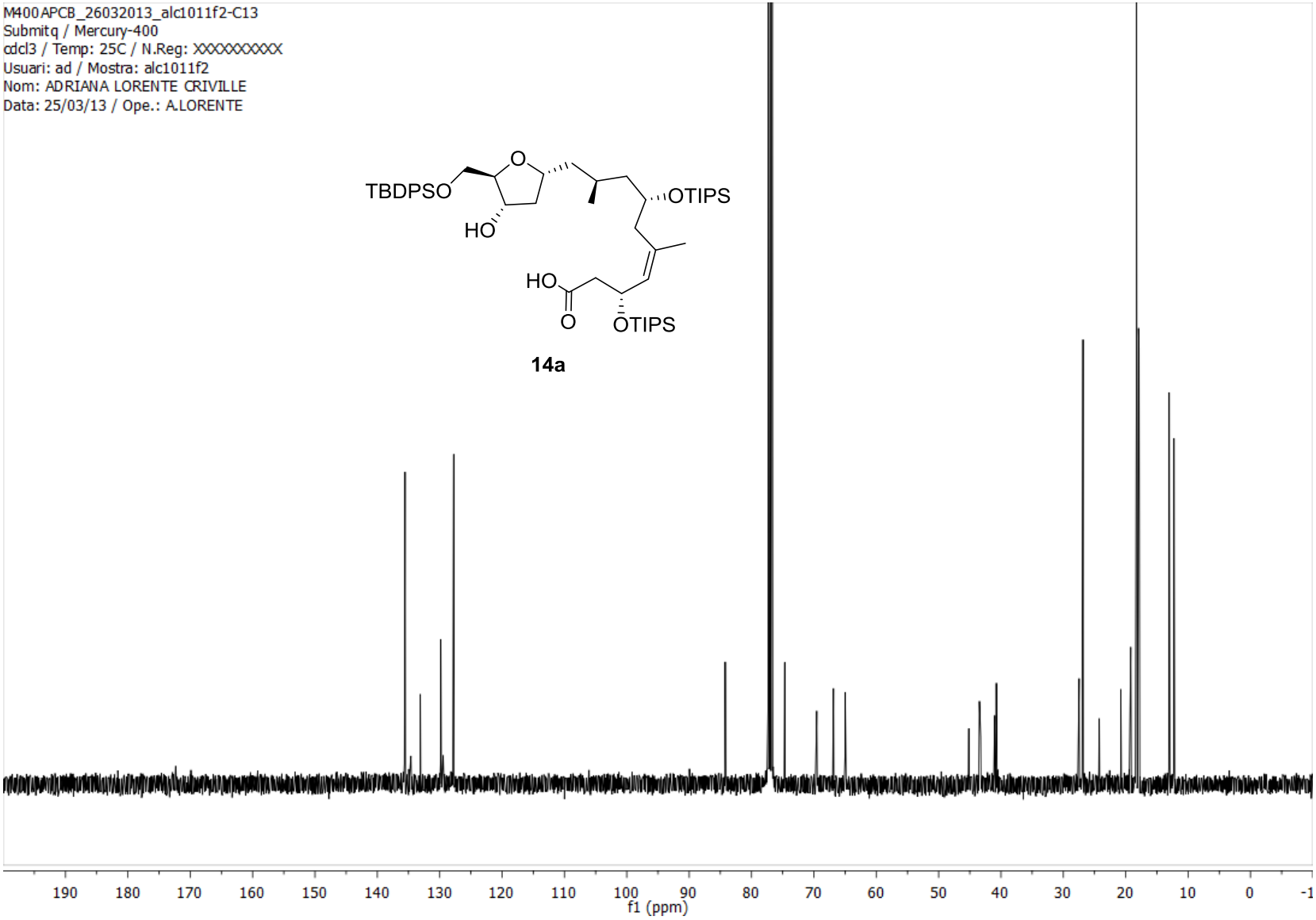

M400APCB_03042013_alc1015p-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1015p
Nom: ADRIANA LORENTE CRIVILLE
Data: 02/04/13 / Ope.: A.LORENTE
TBDPSO
O
TBSO
OTIPS
HO
O
OTIPS
4.16
6.18
0.96
1.00
0.94
0.91
1.15
1.04
2.12
1.99
0.99
1.09
1.05
4.48
4.23
1.40
2.91
54.48
3.48
10.64
5.46
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)

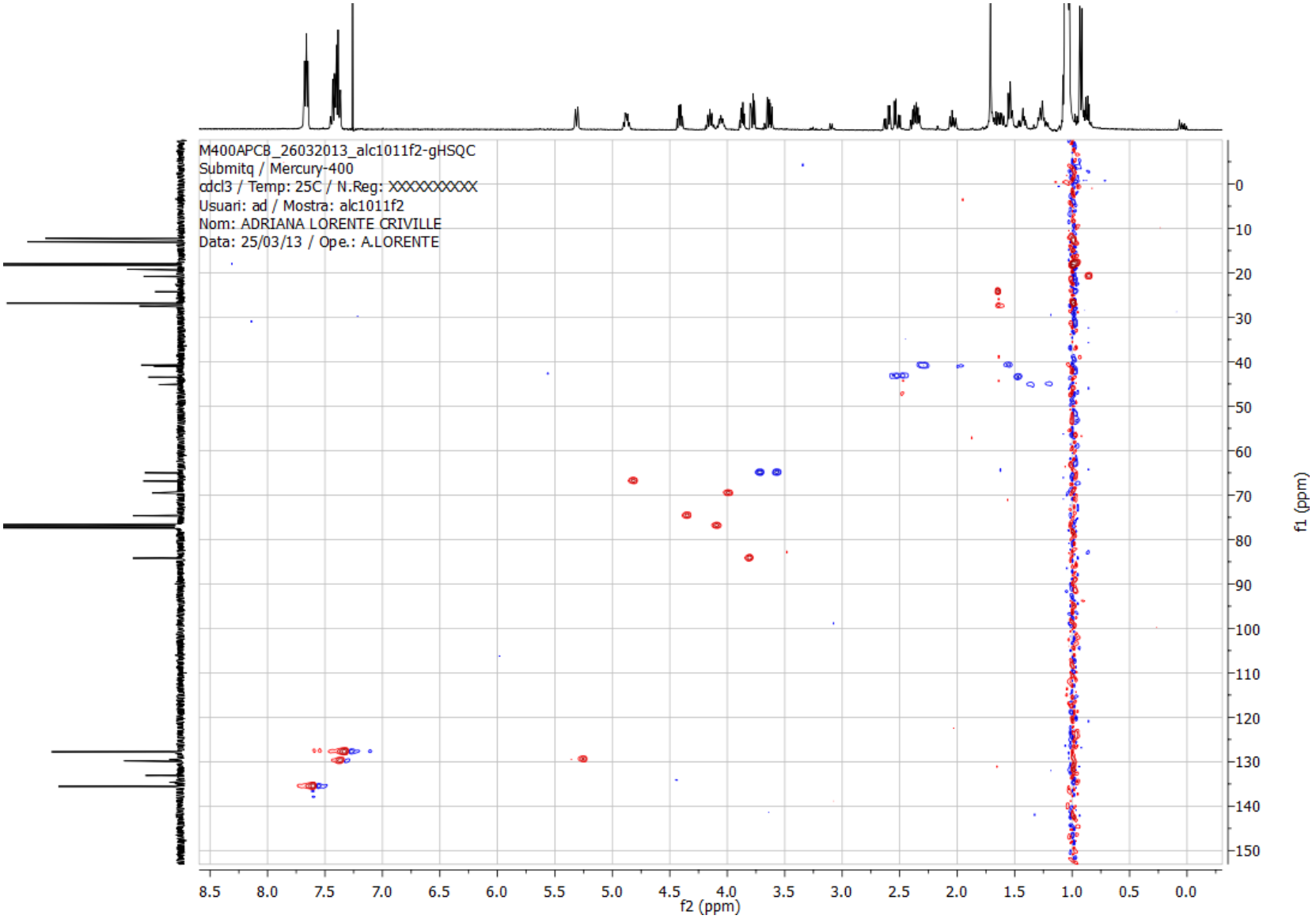
S11

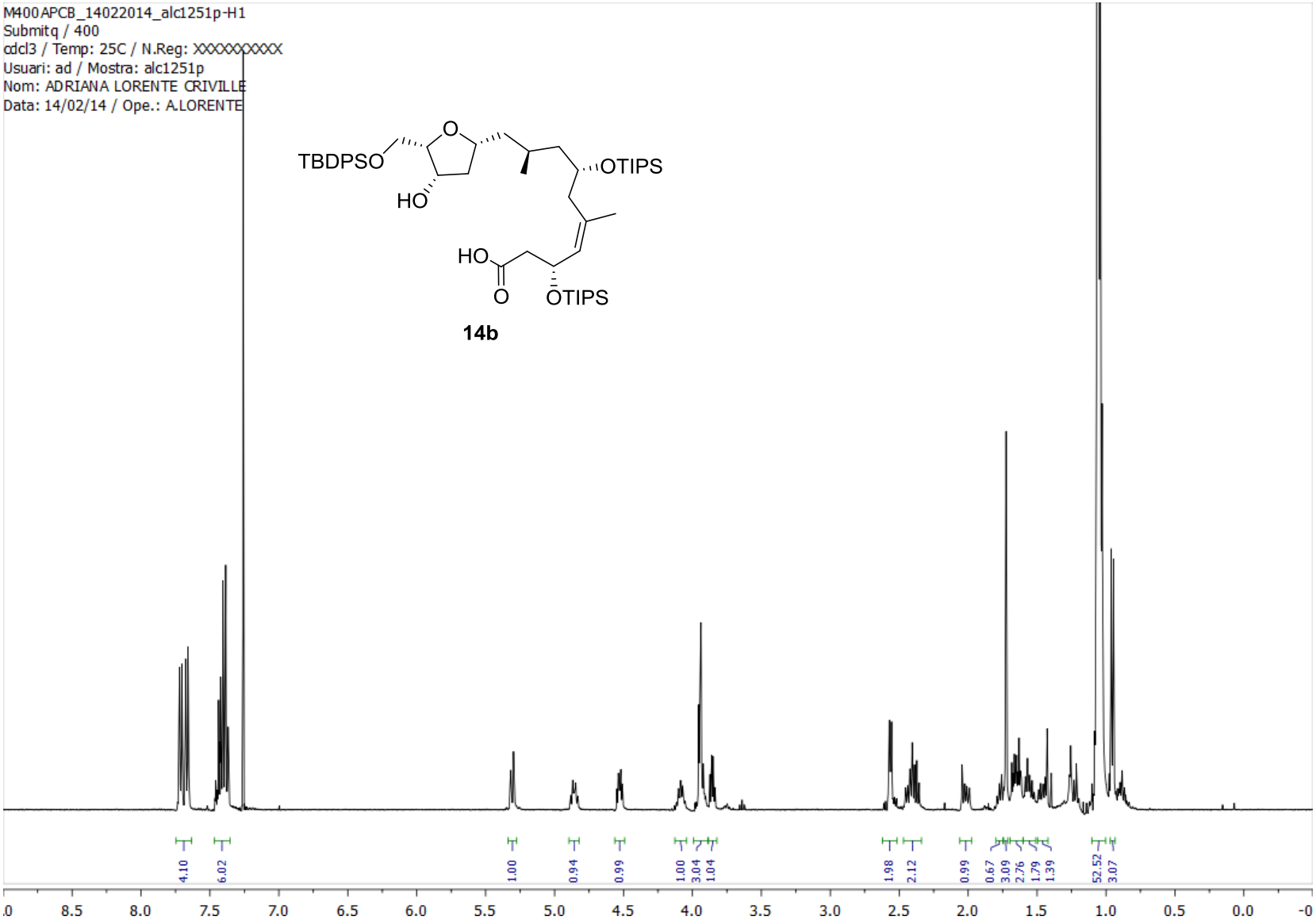

M400APCB_03042013_alc1015p-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1015p
Nom: ADRIANA LORENTE CRIVILLE
Data: 02/04/13 / Ope.: A.LORENTE
TBDPSO
TBSO
O
OTIPS
HO
O
OTIPS
S11
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

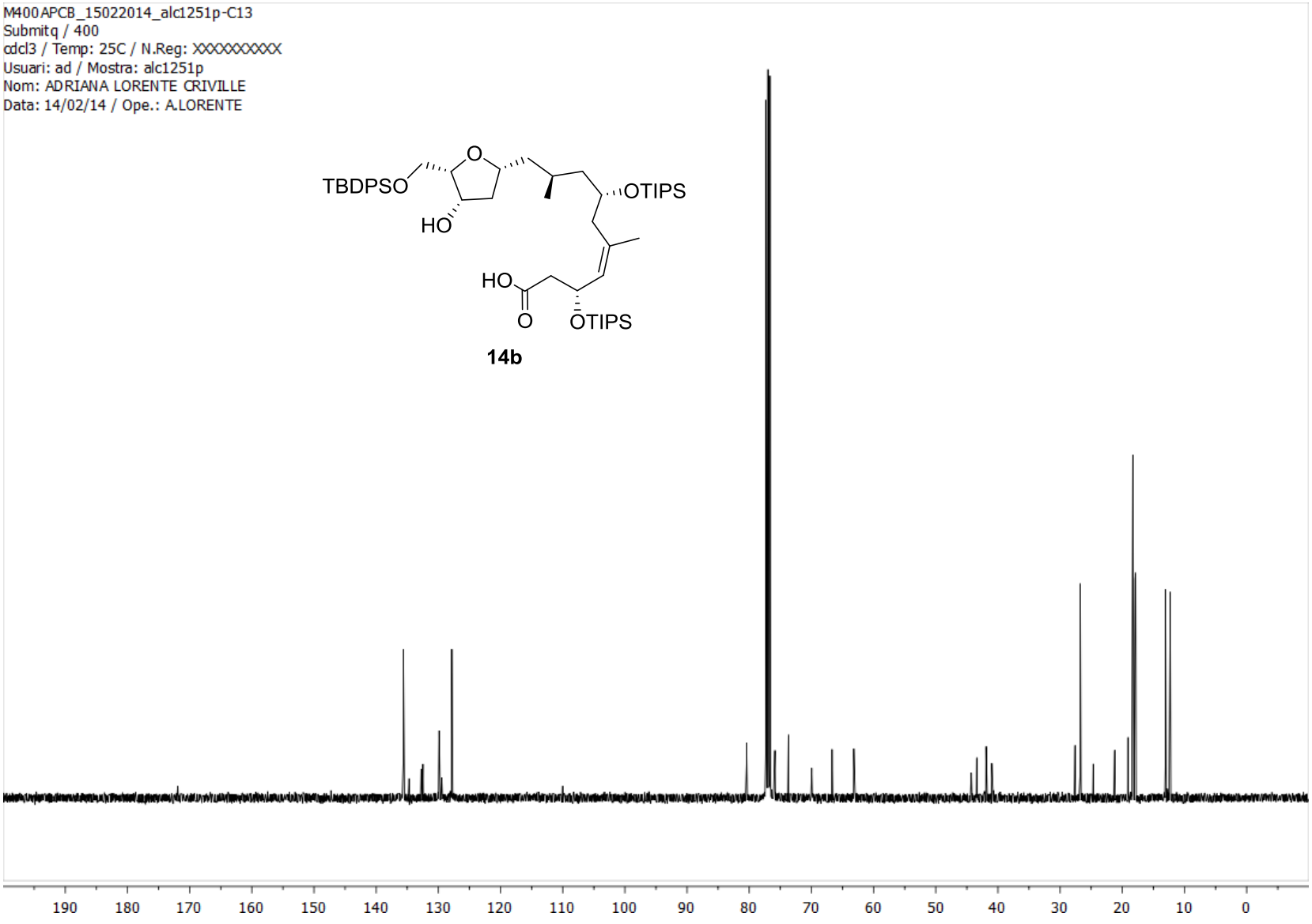

M400APCB_03042013_alc1015p-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1015p
Nom: ADRIANA LORENTE CRIVILLE
Data: 02/04/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

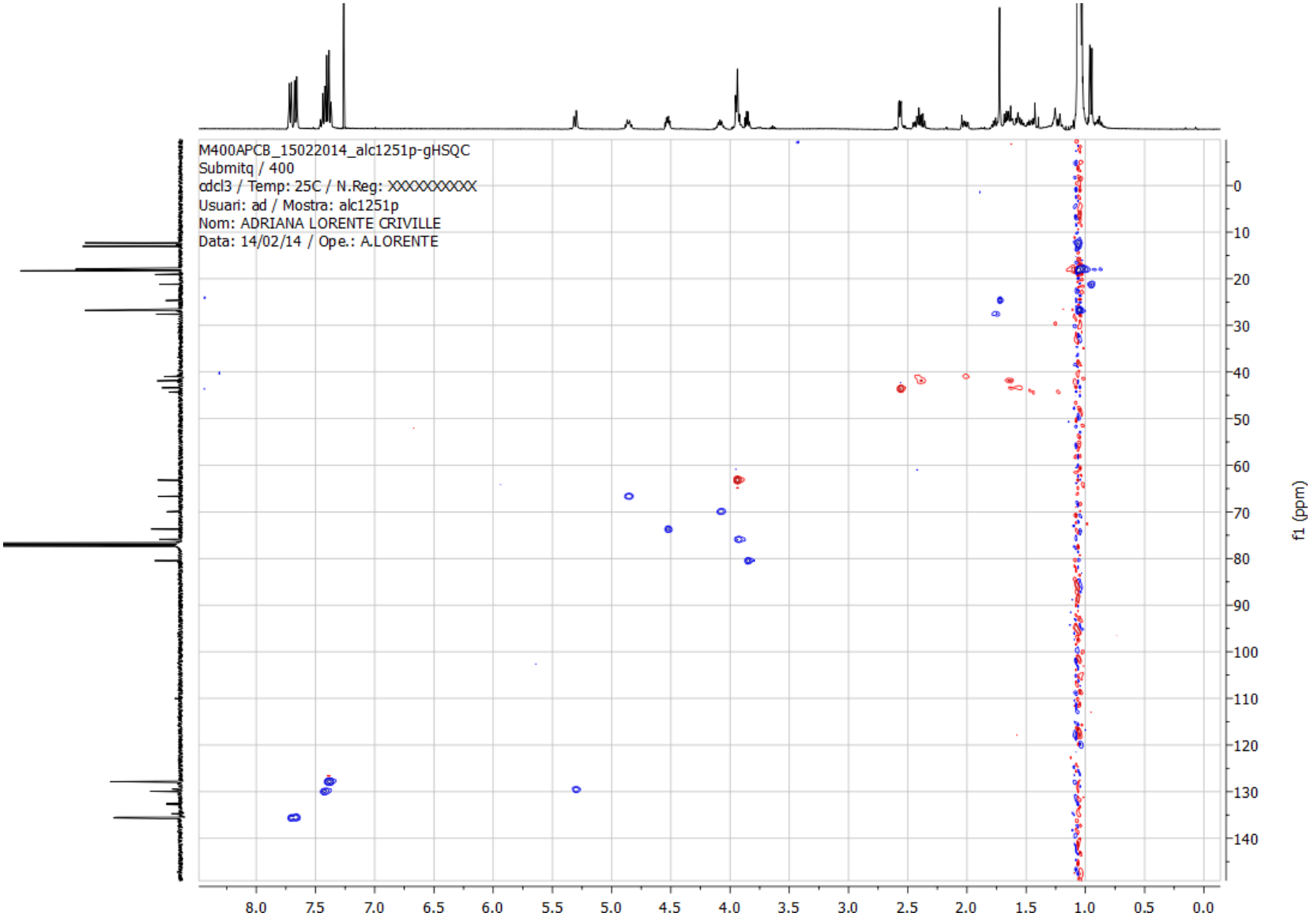

M400APCB_26032013_alc1011f2-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1011f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
TBDPSO
O
HO
OTIPS
HO
O
OTIPS
14a
4.28
6.39
1.00
0.99
1.05
1.11
1.06
1.06
1.11
1.24
1.04
1.15
2.22
1.25
4.57
1.67
2.35
1.72
3.15
55.55
3.06
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)


M400APCB_26032013_alc1011f2-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1011f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
TBDPSO
O
HO
OTIPS
HO
O
OTIPS
14a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

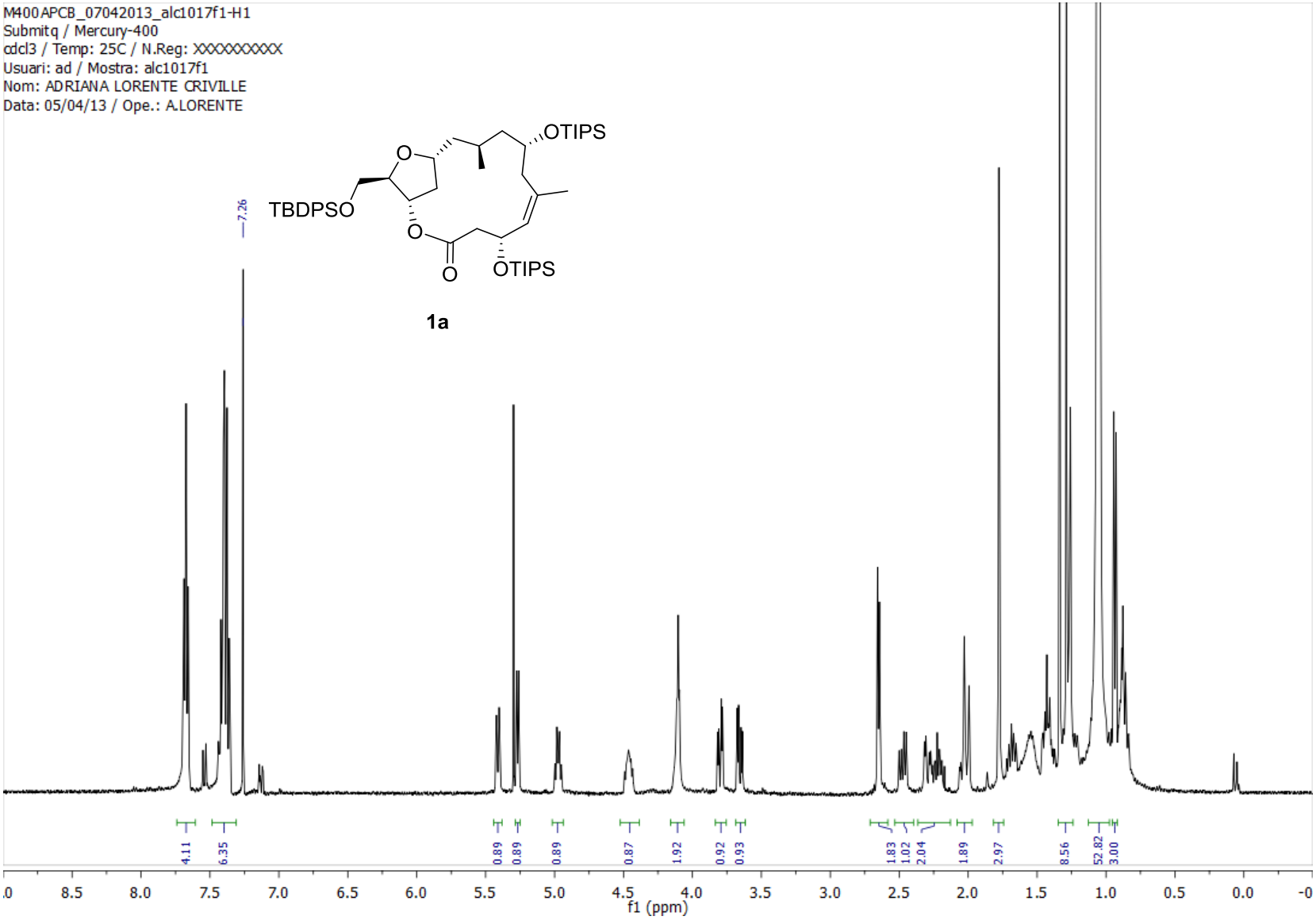

M400APCB_26032013_alc1011f2-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1011f2
Nom: ADRIANA LORENTE CRIVILLE
Data: 25/03/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

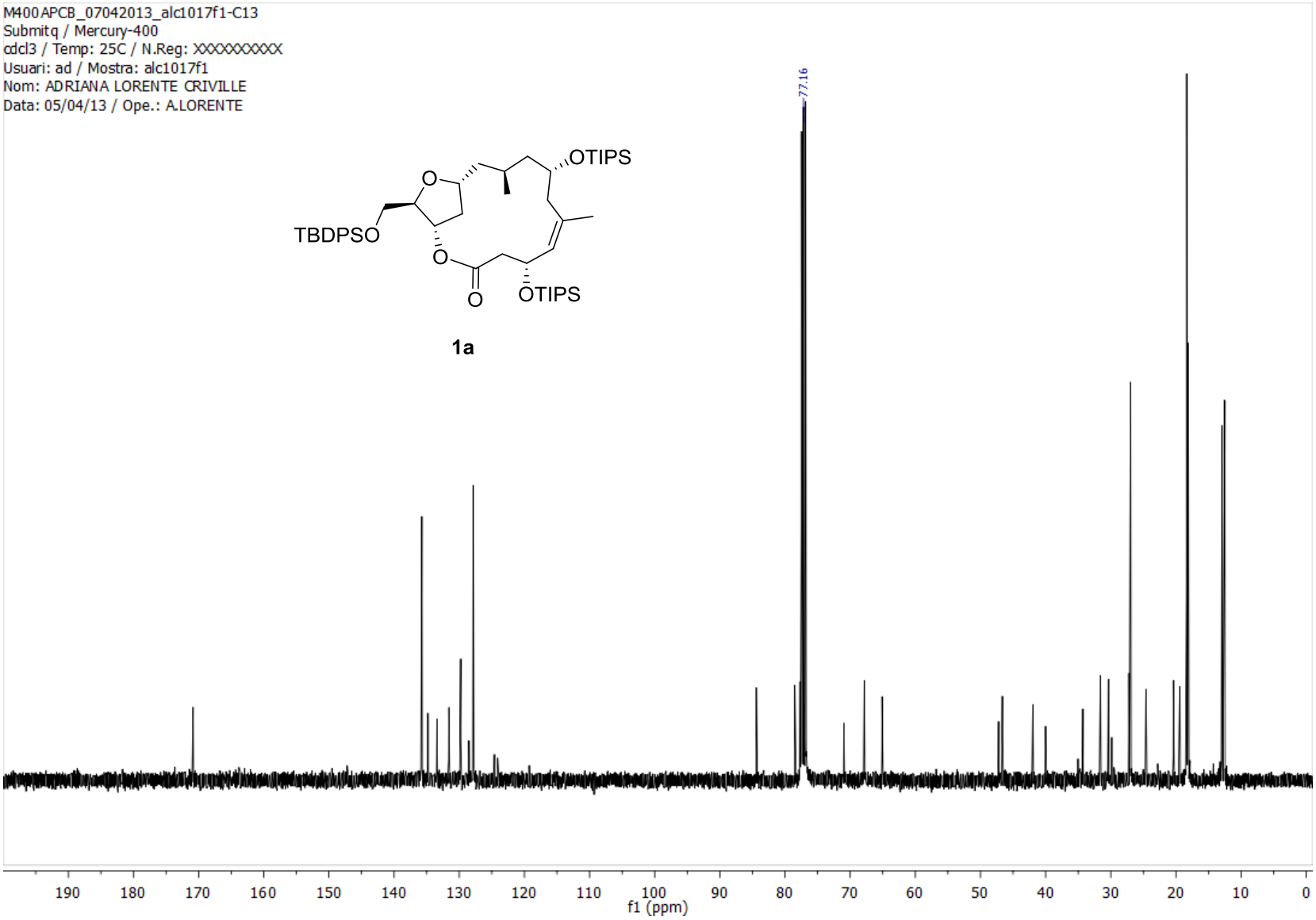

M400APCB_14022014_alc1251p-H1
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXX
Usuari: ad / Mostra: alc1251p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
TBDPSO
O
HO
OTIPS
HO
O
OTIPS
14b
4.10
6.02
1.00
0.94
0.99
1.00
3.04
1.04
1.98
2.12
0.99
0.67
3.09
2.76
1.79
1.39
52.52
3.07
8.5
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

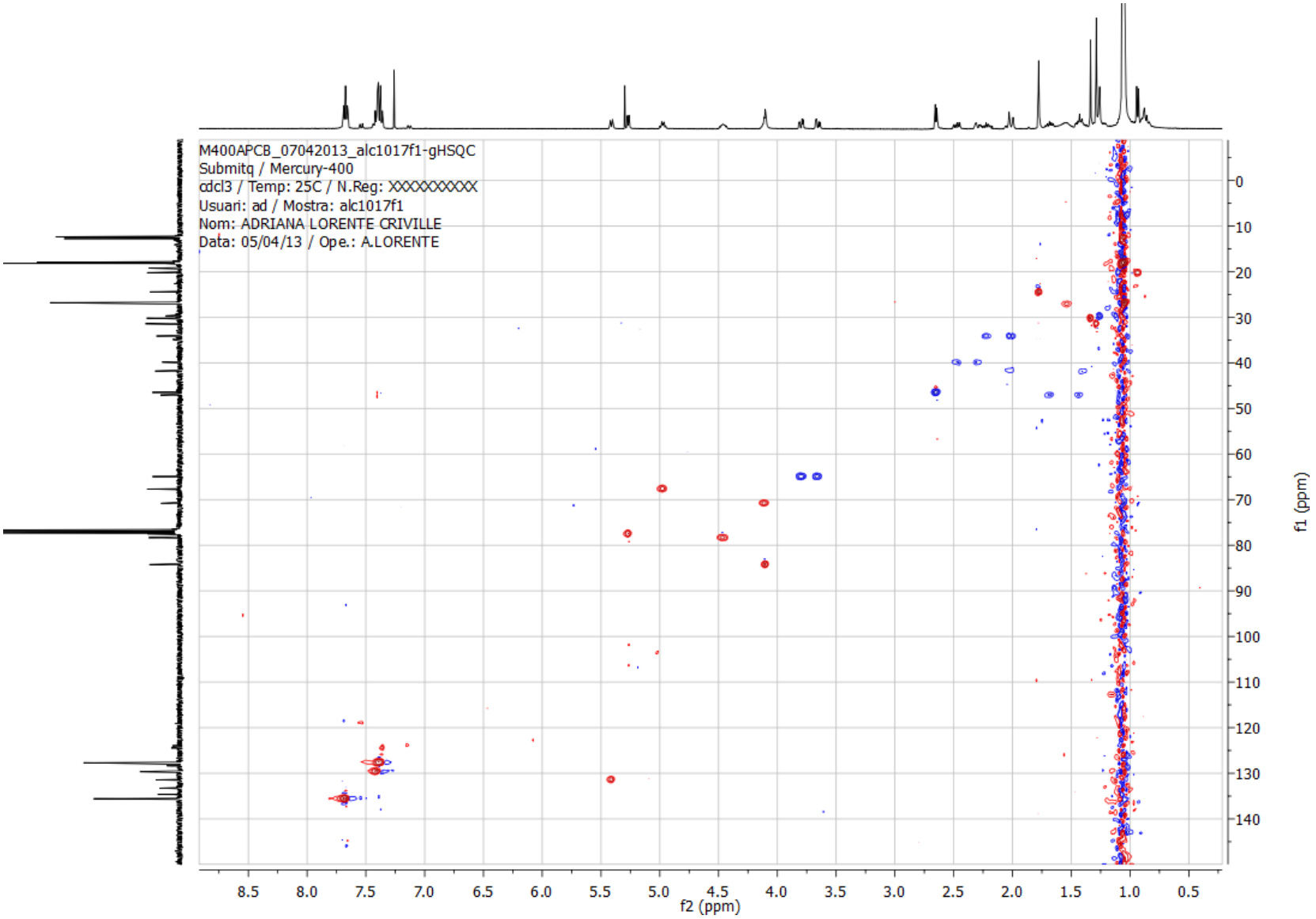

M400APCB_15022014_alc1251p-C13
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1251p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
TBDPSO
O
HO
OTIPS
HO
O
OTIPS
14b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

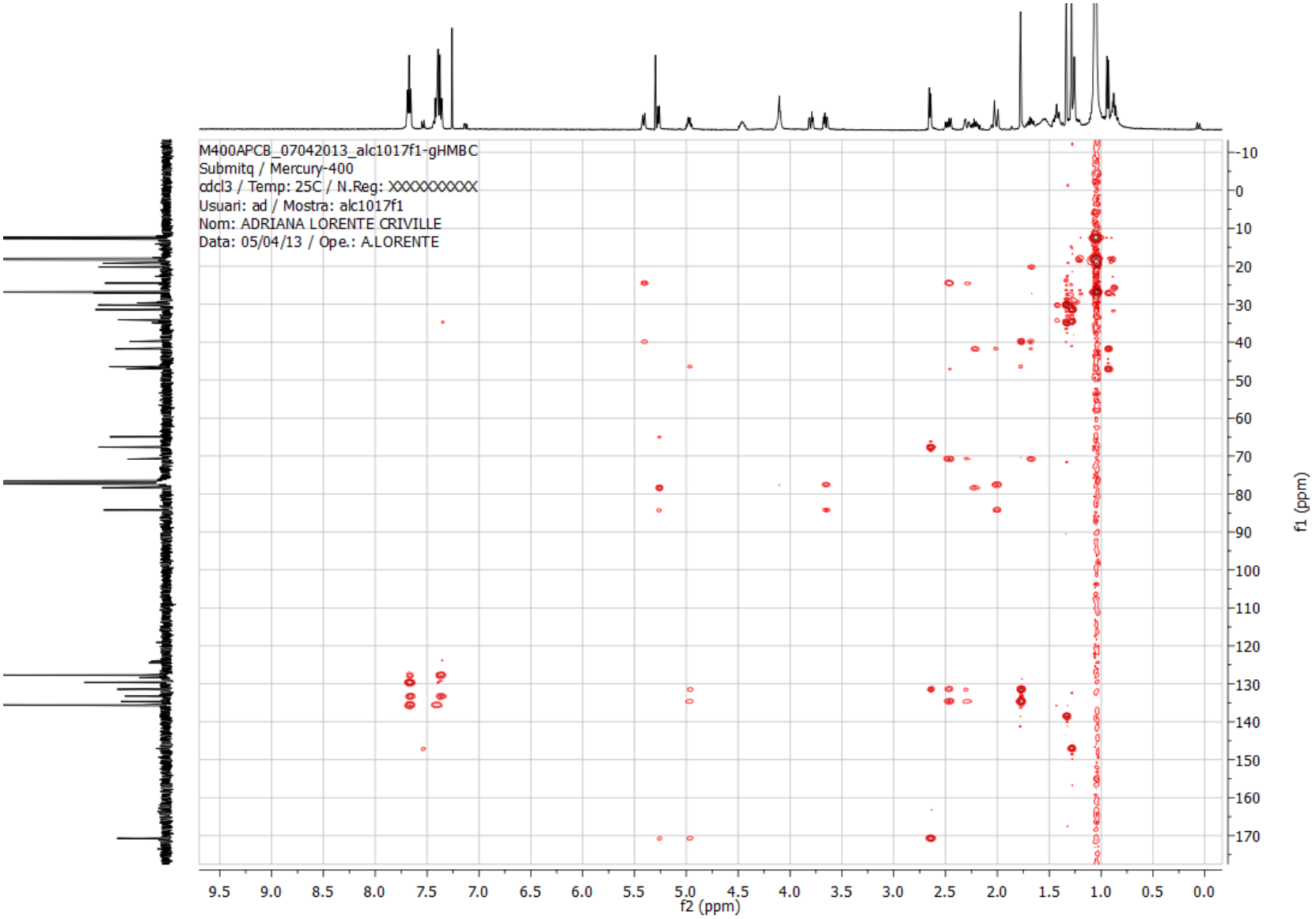

M400APCB_15022014_alc1251p-gHSQC
Submitq / 400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1251p
Nom: ADRIANA LORENTE CRIVILLE
Data: 14/02/14 / Ope.: A.LORENTE
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

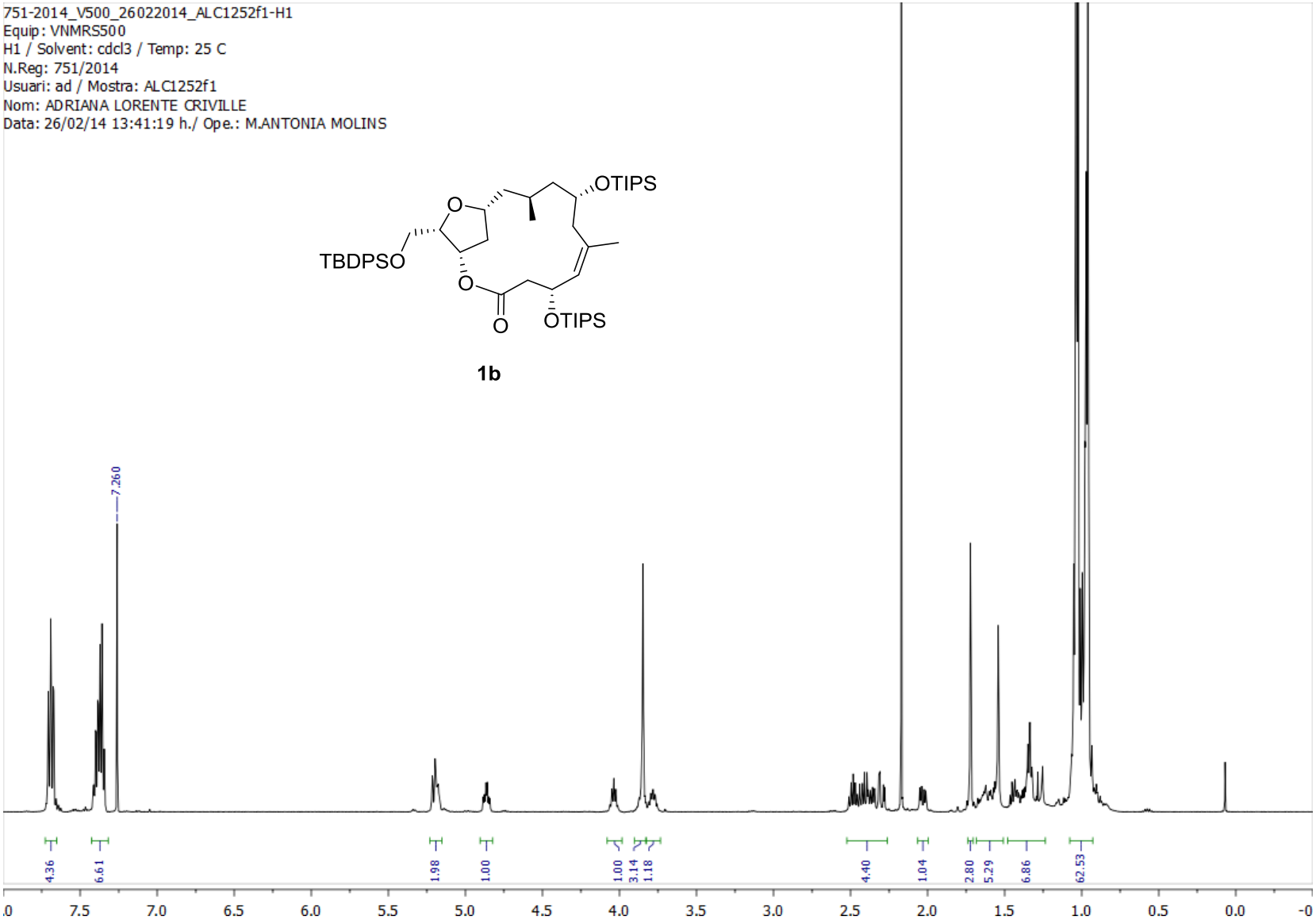
SI 150


M400APCB_07042013_alc1017f1-H1
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1017f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 05/04/13 / Ope.: A.LORENTE
OTIPS
TBDPSO
O
O
O
OTIPS
7.26
1a
4.11
6.35
0.89
0.89
0.89
0.87
1.92
0.92
0.93
1.83
1.02
2.04
1.89
2.97
8.56
52.82
3.00
f1 (ppm)

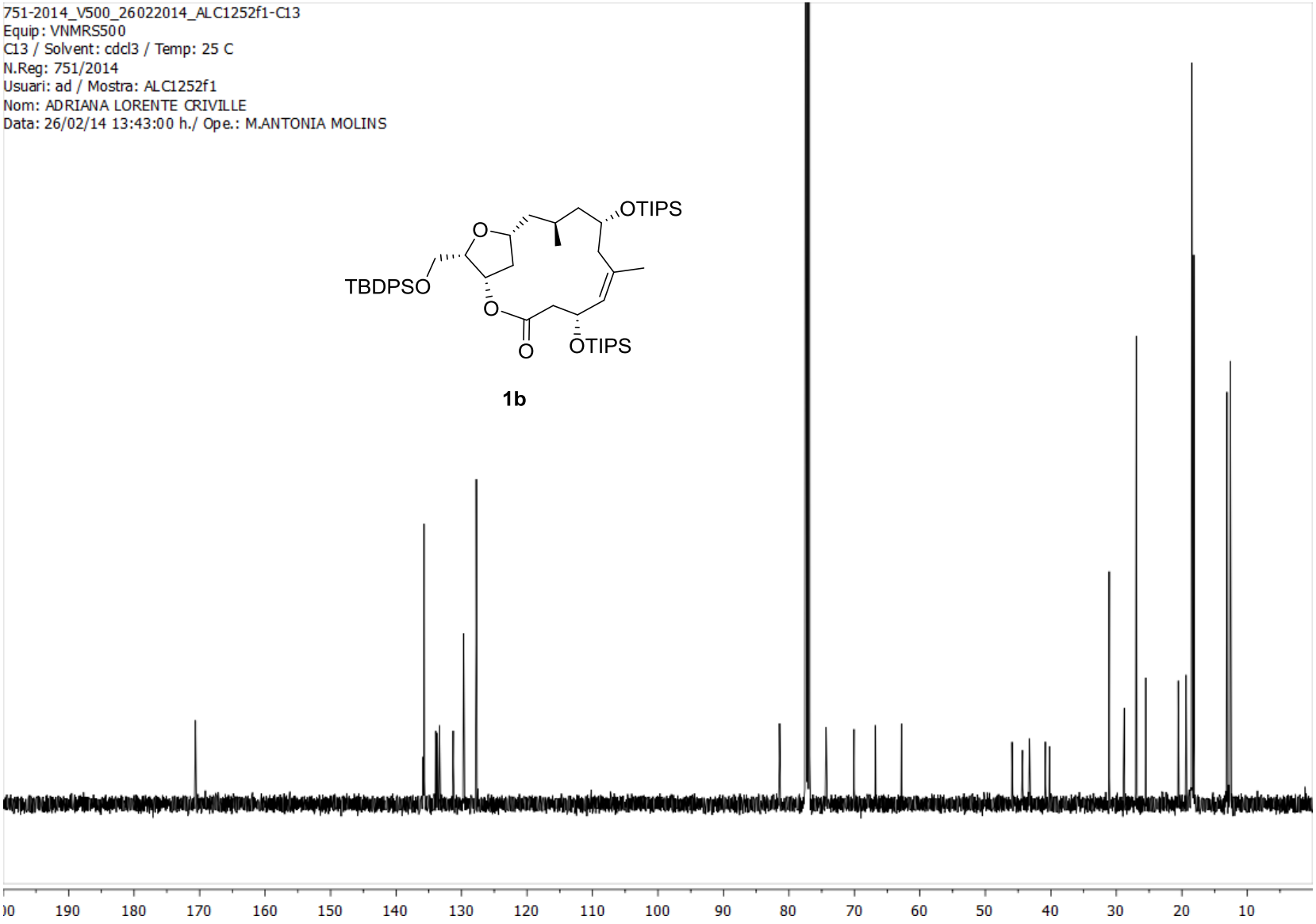

M400APCB_07042013_alc1017f1-C13
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1017f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 05/04/13 / Ope.: A.LORENTE
77.16
OTIPS
TBDPSO
O
O
O
OTIPS
1a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

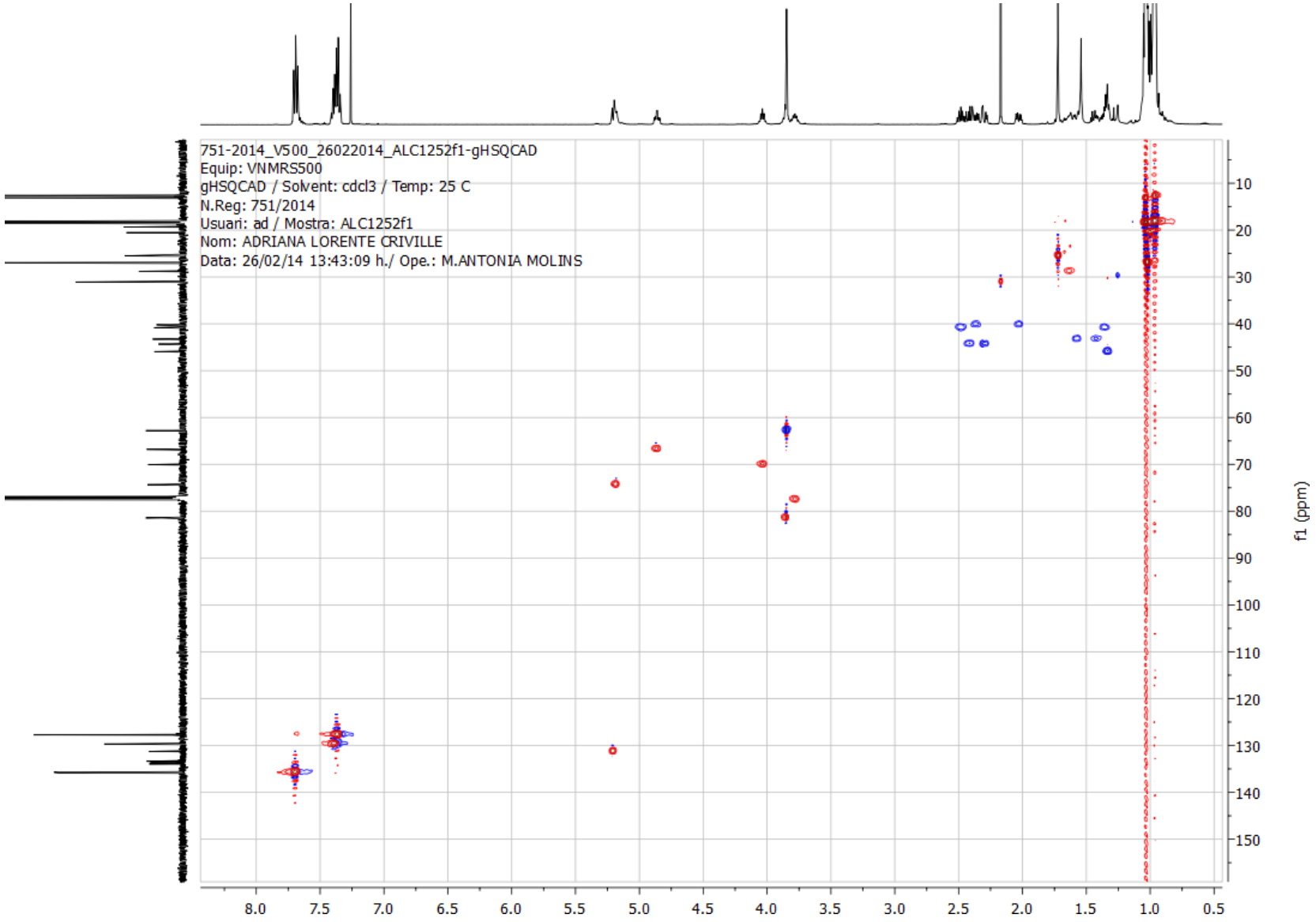

M400APCB_07042013_alc1017f1-gHSQC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1017f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 05/04/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

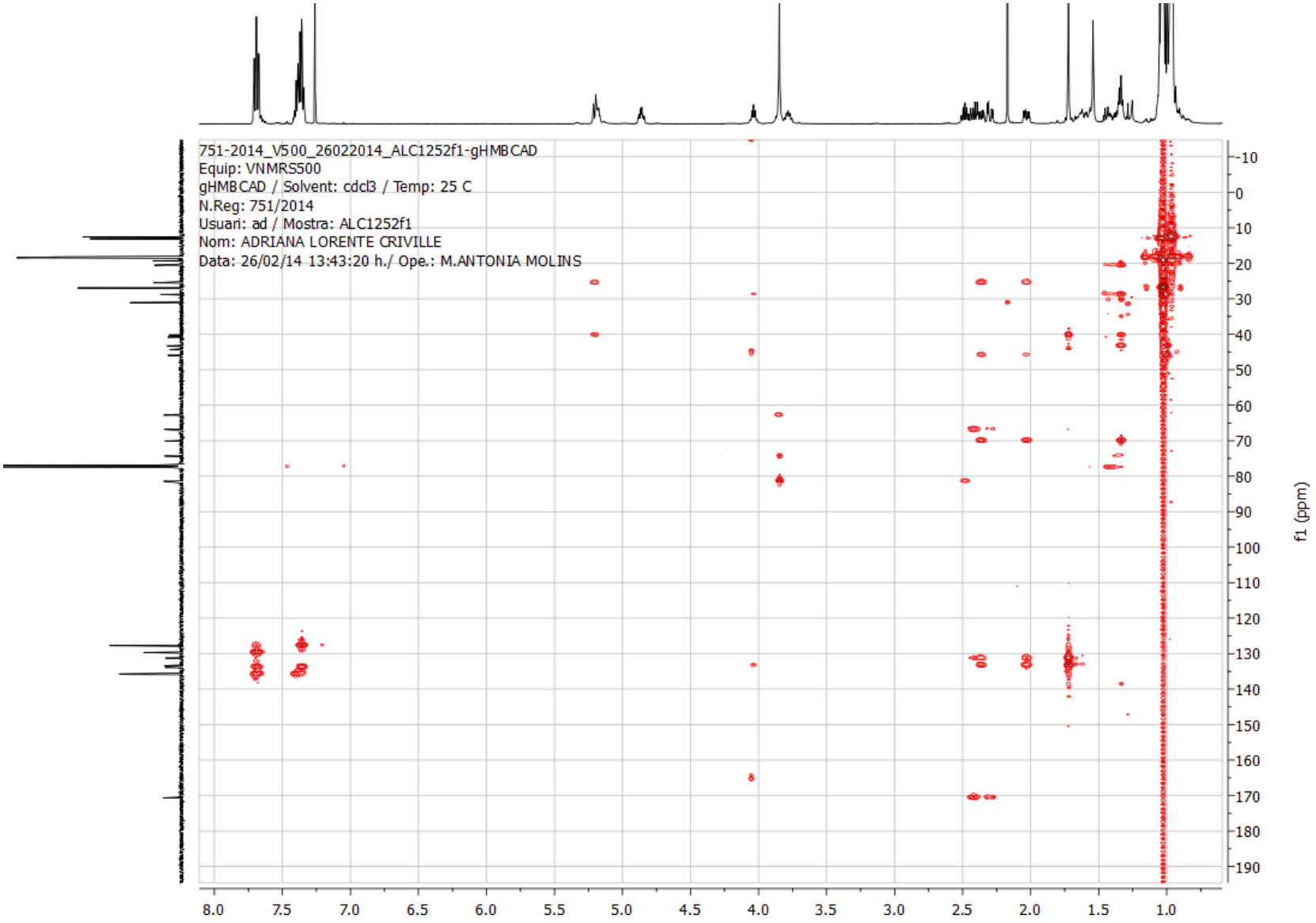

M400APCB_07042013_alc1017f1-gHMBC
Submitq / Mercury-400
cdcl3 / Temp: 25C / N.Reg: XXXXXXXXXX
Usuari: ad / Mostra: alc1017f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 05/04/13 / Ope.: A.LORENTE
f2 (ppm)
f1 (ppm)

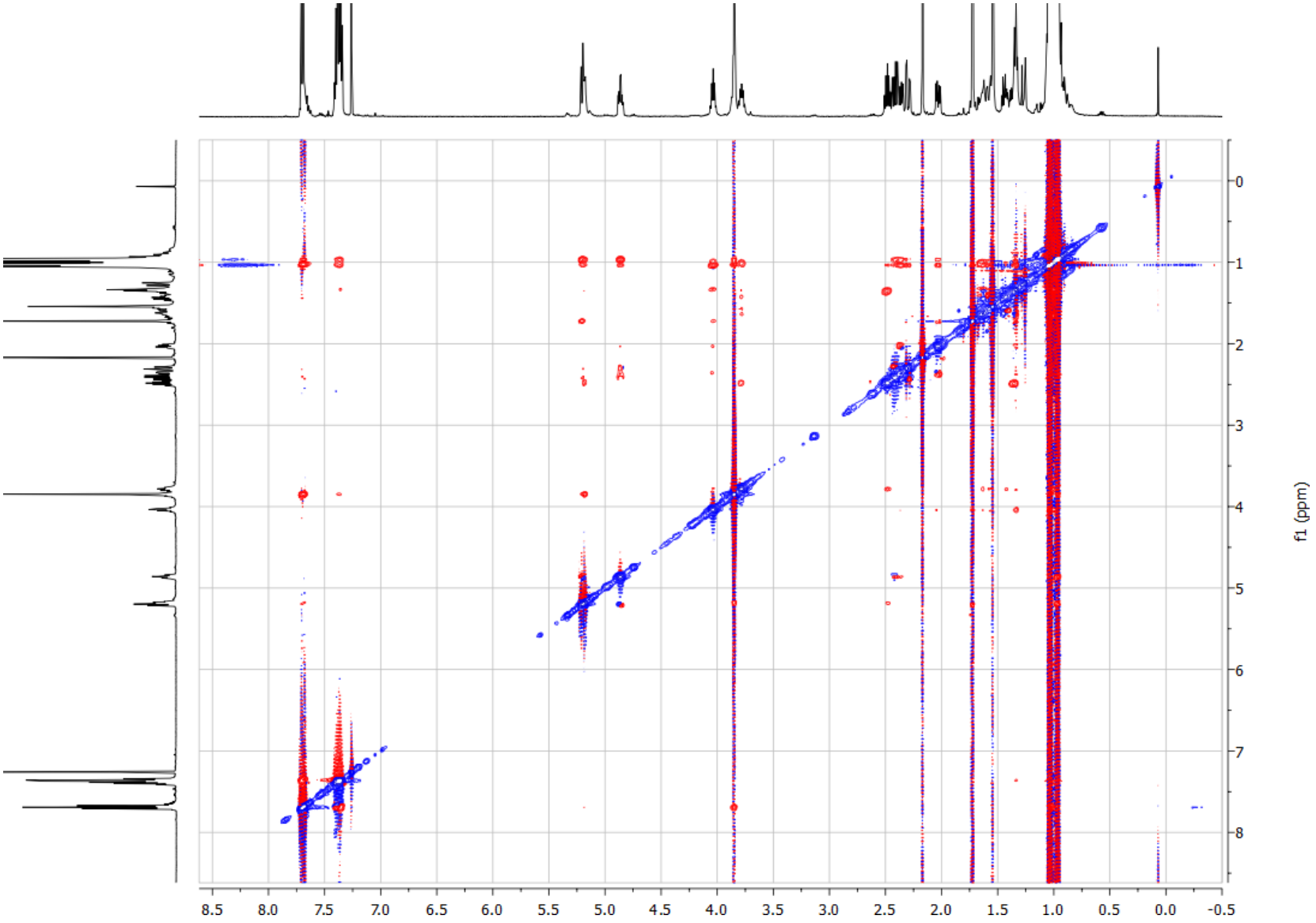

751-2014_V500_26022014_ALC1252f1-H1
Equip: VNMRS500
H1 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 751/2014
Usuari: ad / Mostra: ALC1252f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 26/02/14 13:41:19 h./ Ope.: M.ANTONIA MOLINS
OTIPS
TBDPSO
O
O
O
OTIPS
7.260
4.36
6.61
1.98
1.00
1.00
3.14
1.18
4.40
1.04
2.80
5.29
6.86
62.53
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

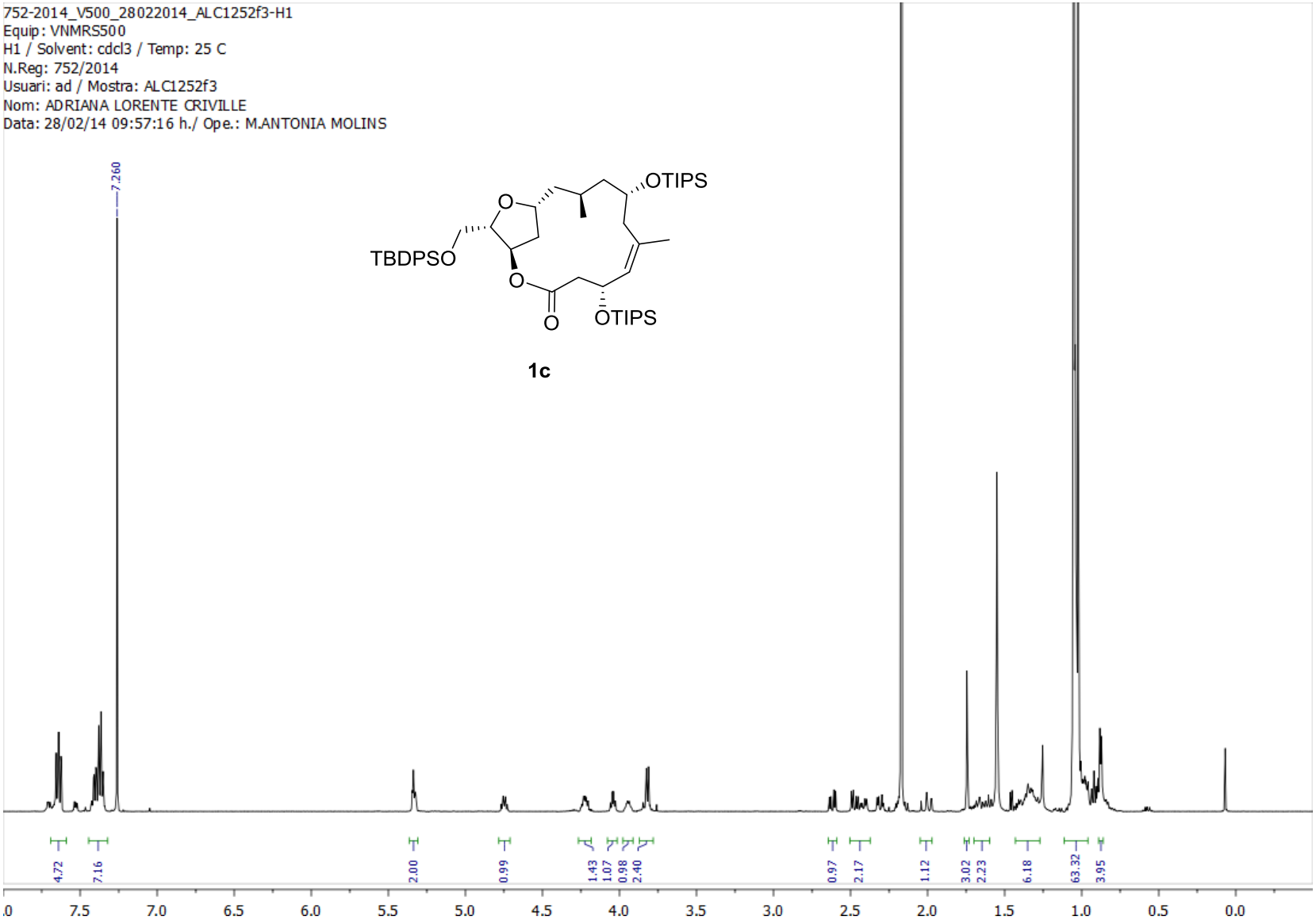
1b


751-2014_V500_26022014_ALC1252f1-C13
Equip: VNMRS500
C13 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 751/2014
Usuari: ad / Mostra: ALC1252f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 26/02/14 13:43:00 h./ Ope.: M.ANTONIA MOLINS
OTIPS
TBDPSO
O
O
O
OTIPS
1b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

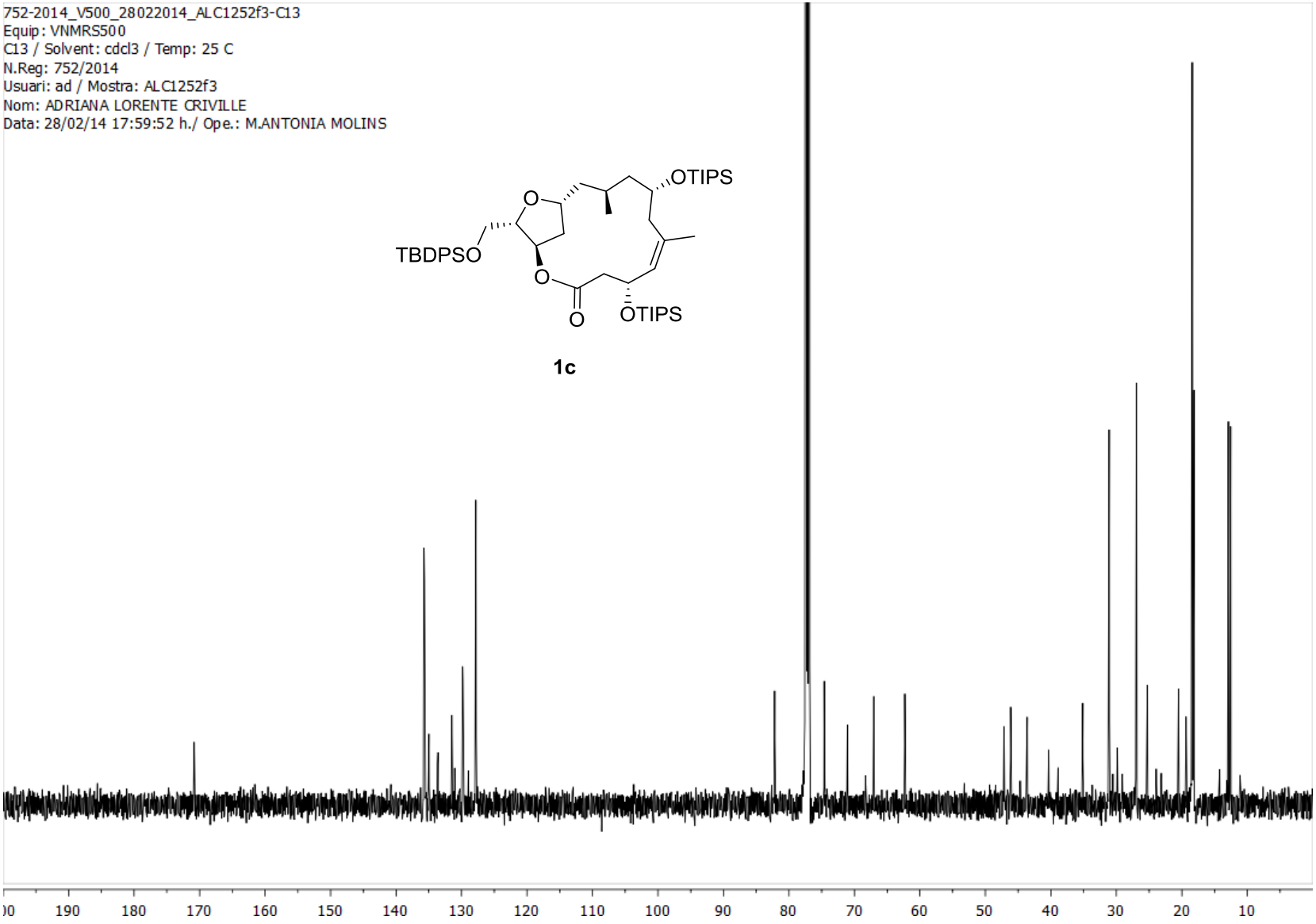

751-2014_V500_26022014_ALC1252f1-gHSQCAD
Equip: VNMRS500
gHSQCAD / Solvent: cdcl3 / Temp: 25 C
N.Reg: 751/2014
Usuari: ad / Mostra: ALC1252f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 26/02/14 13:43:09 h./ Ope.: M.ANTONIA MOLINS
f1 (ppm)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150


751-2014_V500_26022014_ALC1252f1-gHMBCAD
Equip: VNMRS500
gHMBCAD / Solvent: cdcl3 / Temp: 25 C
N.Reg: 751/2014
Usuari: ad / Mostra: ALC1252f1
Nom: ADRIANA LORENTE CRIVILLE
Data: 26/02/14 13:43:20 h./ Ope.: M.ANTONIA MOLINS
f1 (ppm)
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
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

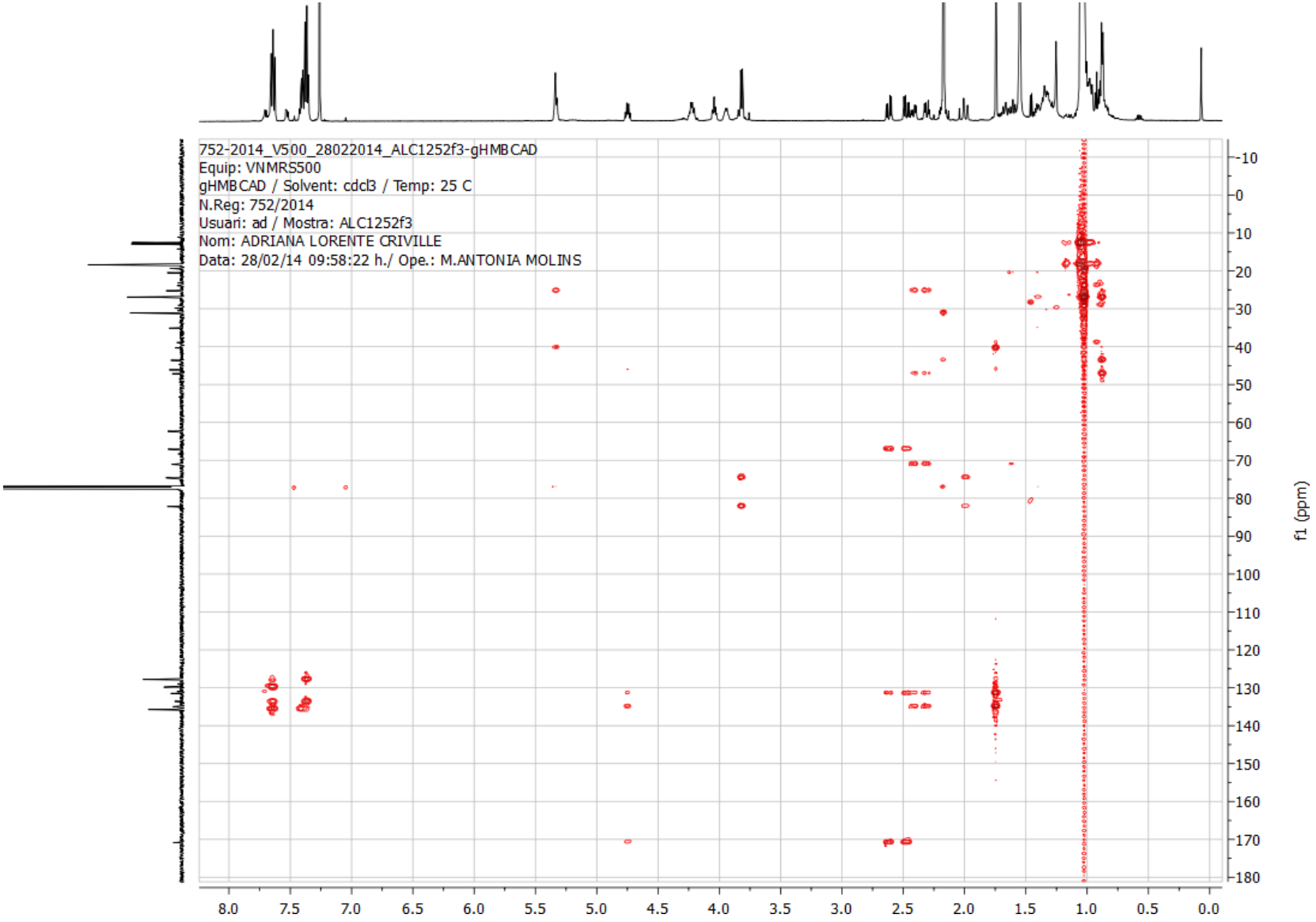

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
1
2
3
4
5
6
7
8
f1 (ppm)

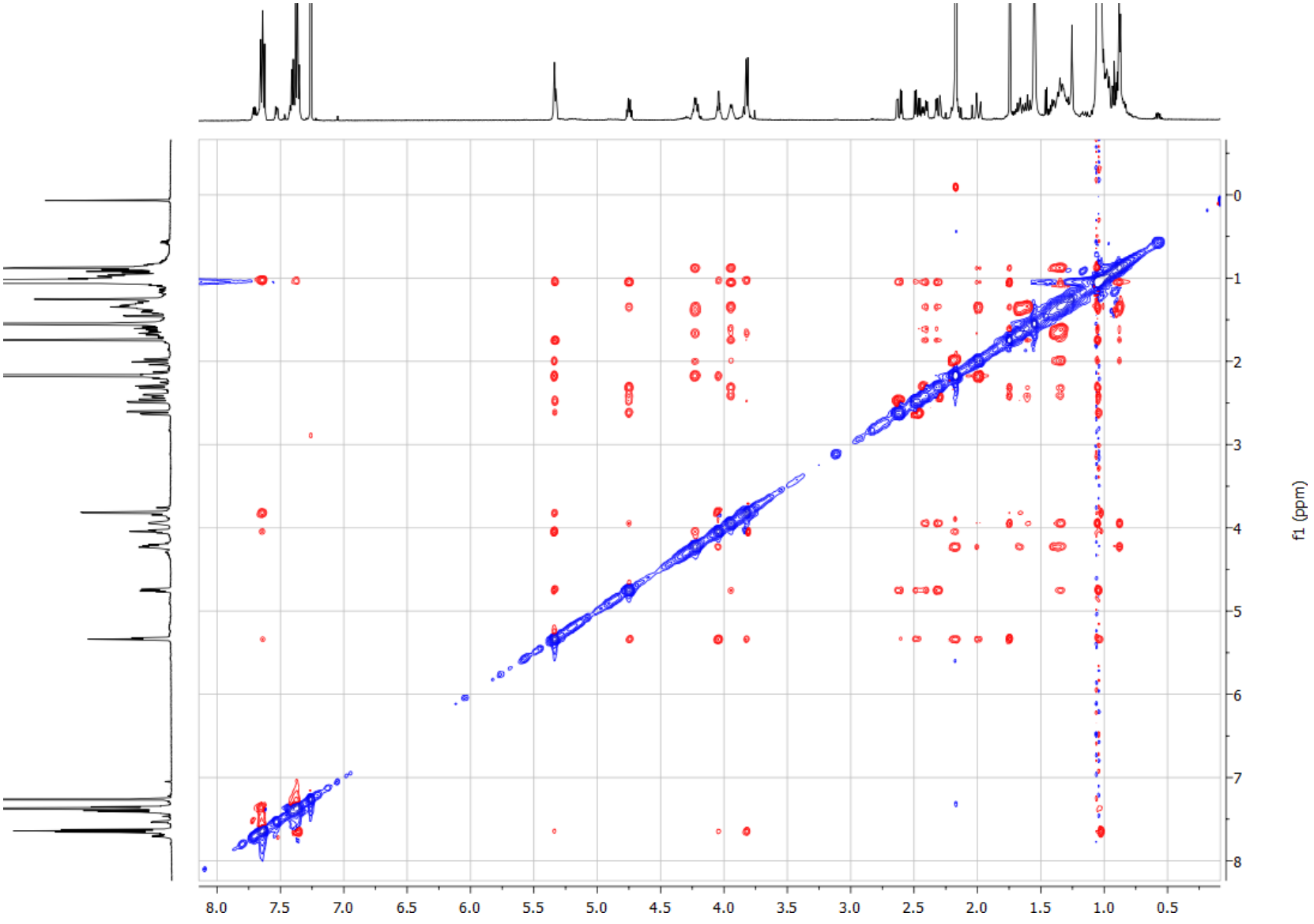

752-2014_V500_28022014_ALC1252f3-H1
Equip: VNMRS500
H1 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 752/2014
Usuari: ad / Mostra: ALC1252f3
Nom: ADRIANA LORENTE CRIVILLE
Data: 28/02/14 09:57:16 h./ Ope.: M.ANTONIA MOLINS
7.260
OTIPS
TBDPSO
O
O
O
OTIPS
4.72
7.16
2.00
0.99
1.43
1.07
0.98
2.40
0.97
2.17
1.12
3.02
2.23
6.18
63.32
3.95
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

1c


752-2014_V500_28022014_ALC1252f3-C13
Equip: VNMRS500
C13 / Solvent: cdcl3 / Temp: 25 C
N.Reg: 752/2014
Usuari: ad / Mostra: ALC1252f3
Nom: ADRIANA LORENTE CRIVILLE
Data: 28/02/14 17:59:52 h./ Ope.: M.ANTONIA MOLINS
OTIPS
TBDPSO
O
O
O
OTIPS
1c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


752-2014_V500_28022014_ALC1252f3-gHSQCAD
Equip: VNMRS500
gHSQCAD / Solvent: cdcl3 / Temp: 25 C
N.Reg: 752/2014
Usuari: ad / Mostra: ALC1252f3
Nom: ADRIANA LORENTE CRIVILLE
Data: 28/02/14 09:57:32 h./ Ope.: M.ANTONIA MOLINS
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
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


752-2014_V500_28022014_ALC1252f3-gHMBCAD
Equip: VNMRS500
gHMBCAD / Solvent: cdcl3 / Temp: 25 C
N.Reg: 752/2014
Usuari: ad / Mostra: ALC1252f3
Nom: ADRIANA LORENTE CRIVILLE
Data: 28/02/14 09:58:22 h./ Ope.: M.ANTONIA MOLINS
f1 (ppm)
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
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


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
1
2
3
4
5
6
7
8
f1 (ppm)

8. References

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