European Commission

Joint Research Centre

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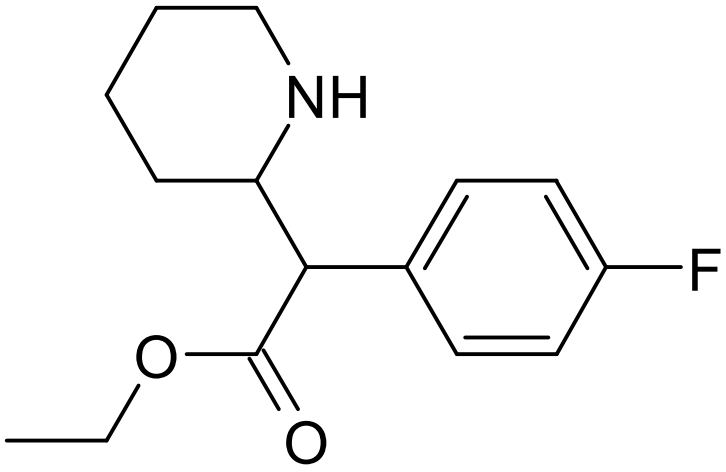
ADMINISTRATIVE ARRANGEMENT JRC-Nr 33619-CLEN2SAND-DG TAXUD-Nr TAXUD/2014/DE/315 BETWEEN DG TAXATION AND CUSTOMS UNION (DG TAXUD) AND THE JOINT RESEARCH CENTRE (JRC) for fast recognition of New Psychoactive Substances (NPS) and identification of unknown chemicals

This report was generated on 18/03/2016 based on data from the European Customs laboratories and the Joint Research Centre. This report includes sample and molecular information, spectral data and associated tables and figures. The chemical structure(s) was/were identified by Bio-chemical interactions & metabolomics (BCIM) group chemists on the basis of analytical data available. NMR assignments proposed below were performed by ACD labs tools in agreement with the chemical structure identified by analytical experts. Reported data are related to the sample in the following table:

Eurodat number	16020005	***Received on***	18 February 2016
PACKAGING	1 * glass vial white powder		
Name of customer	Service commun des laboratoires France		
Customer's identification	2016-24417		

The following structure(s) was/were identified in the sample:



Data of identified compound(s)

IUPAC Name	ethyl (4-fluorophenyl)(piperidin-2-yl)acetate		
NAME	4F-Ethylphenidate	***SYNONYMS***	4F-EPH
Formula	$C_{15}H_{20}FNO_2$	***FW***	265.3232
Monoisotopic Mass	265.147807		
InChI	InChI=1S/C15H20FNO2/c1-2-19-15(18)14(13-5-3-4-10-17-13)11-6-8-12(16)9-7-11/h6-9,13-14,17H,2-5,10H2,1H3		
InChI Key	RKXQYWFDJDYSEN-UHFFFAOYSA-N		
SMILES	Fc1ccc(cc1)C(C(=O)OCC)C2CCCCN2		

Claude Guillou, Fabiano Reniero, Hubert Chassaigne, Joana Lobo Vicente, Veronica Holland, Kamil Kolar

European Commission, Joint Research Centre

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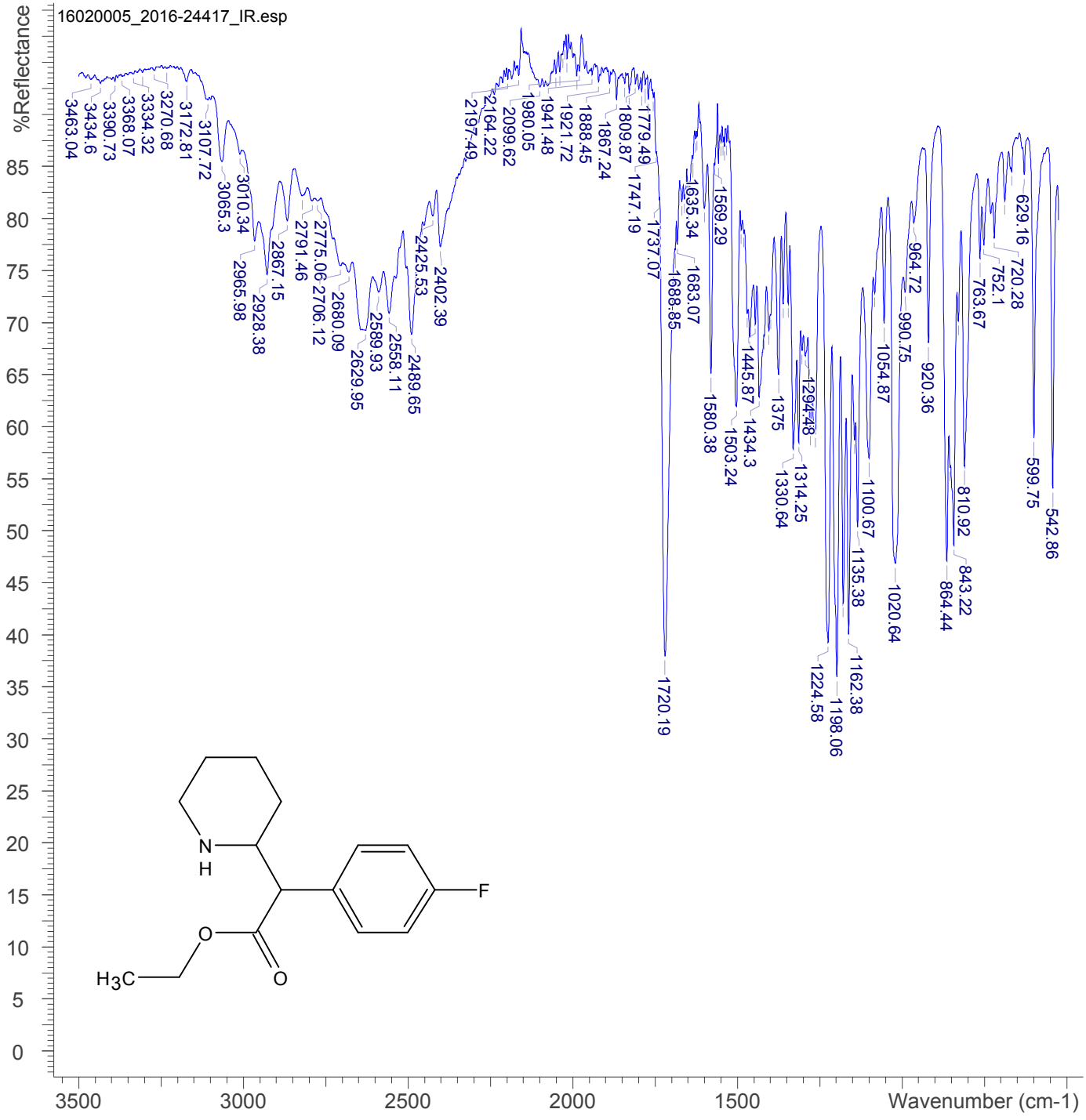
Processing and interpretation based on data provided by:

Service commun des laboratoires France

Title	24417		
File Name	\\139.191.6.82\ihcp$\I01\BIO_CHEMICAL_INTERACTION_METABONOMICS\CLEN2SAND\!PROPOSED-STRUCTURE\CLEN_NAS\16020005_2016-24417\2016-24417\2016-24417\IR.SPA		
Date Stamp	29 Jan 2016 11:33:53	***Date***	29 Jan 2016 12:34:06
Technique	Infrared	***Spectral Region***	IR
X Axis	Wavenumber (cm-1)	***Y Axis***	%Reflectance
Spectrum Range	525.0251 - 3500.1670	***Points Count***	6172
Data Spacing	0.4821		



Processing and interpretation based on data provided by:

Service commun des laboratoires France

Count	166	***Data Type***	Condensed
Date	9:42:28 AM	***Date Stamp***	25 Jan 16 09:30 am
File Name	\\139.191.6.82\ihcp$\I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure_SAND-DB\Results\16020005_2016-24417\16020005_2016-24417_EI+_3.76min		
Ion Mode	EI+	***Mass Spec Model***	GCMS Pyro
Plot Type	Stick	***Retention Time***	3.760
Sample	ds CH2Cl2/MeOH : 2016-24417		
Scan	419	***Scan Mode***	Centroid
Spectrum Assigned	69.2% [33-281/0-100]	***Spectrum Type***	MS
TIC	253.78	***Total Signal***	3335484

16020005_2016-24417_EI+3.760min.esp

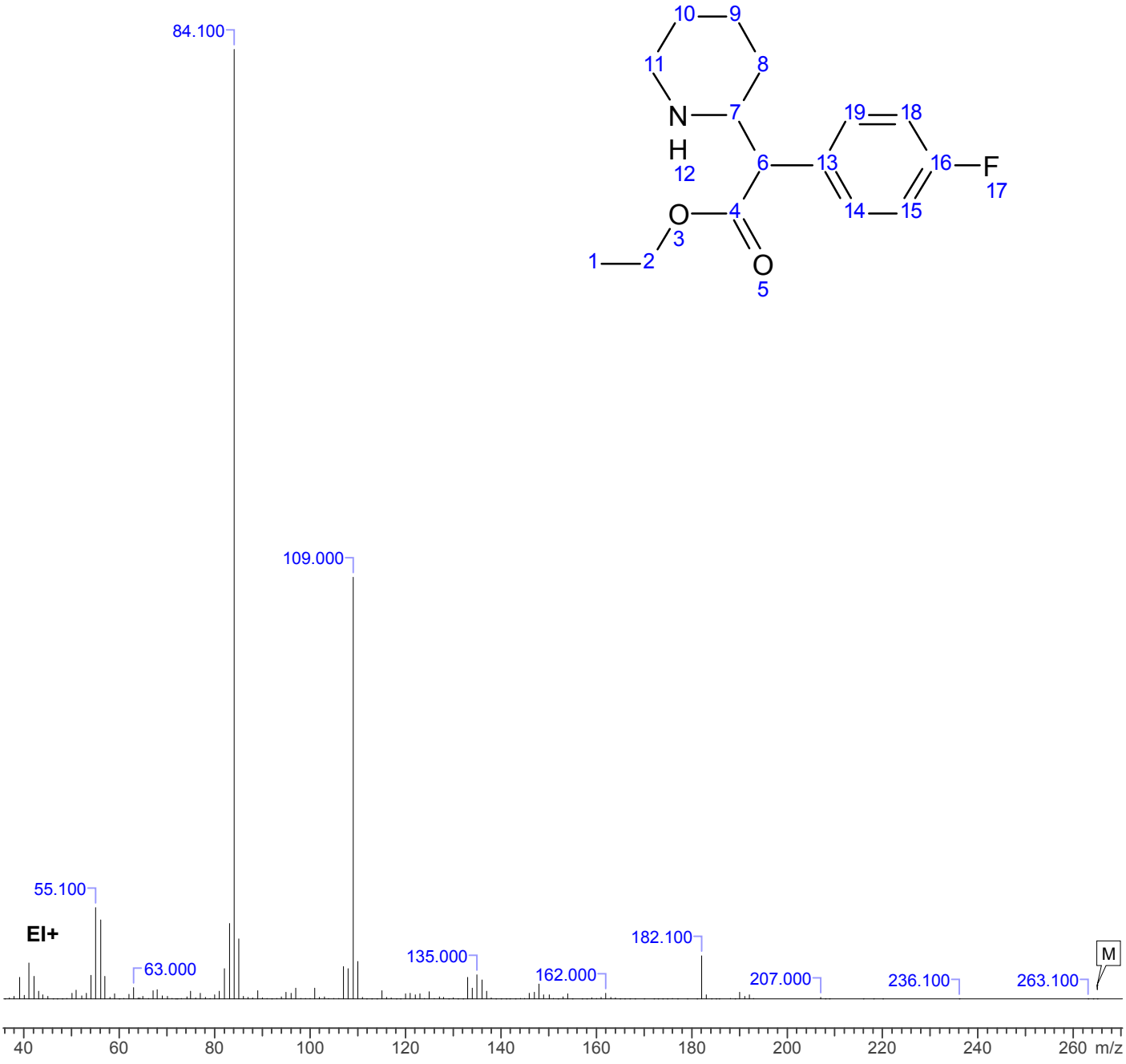
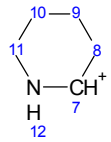
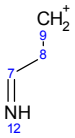
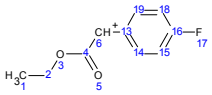
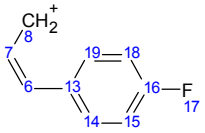
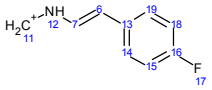
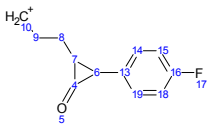
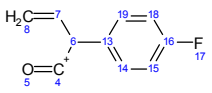
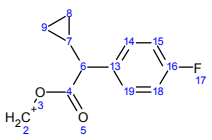
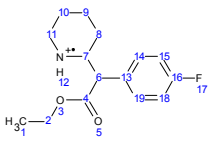


Table of fragments

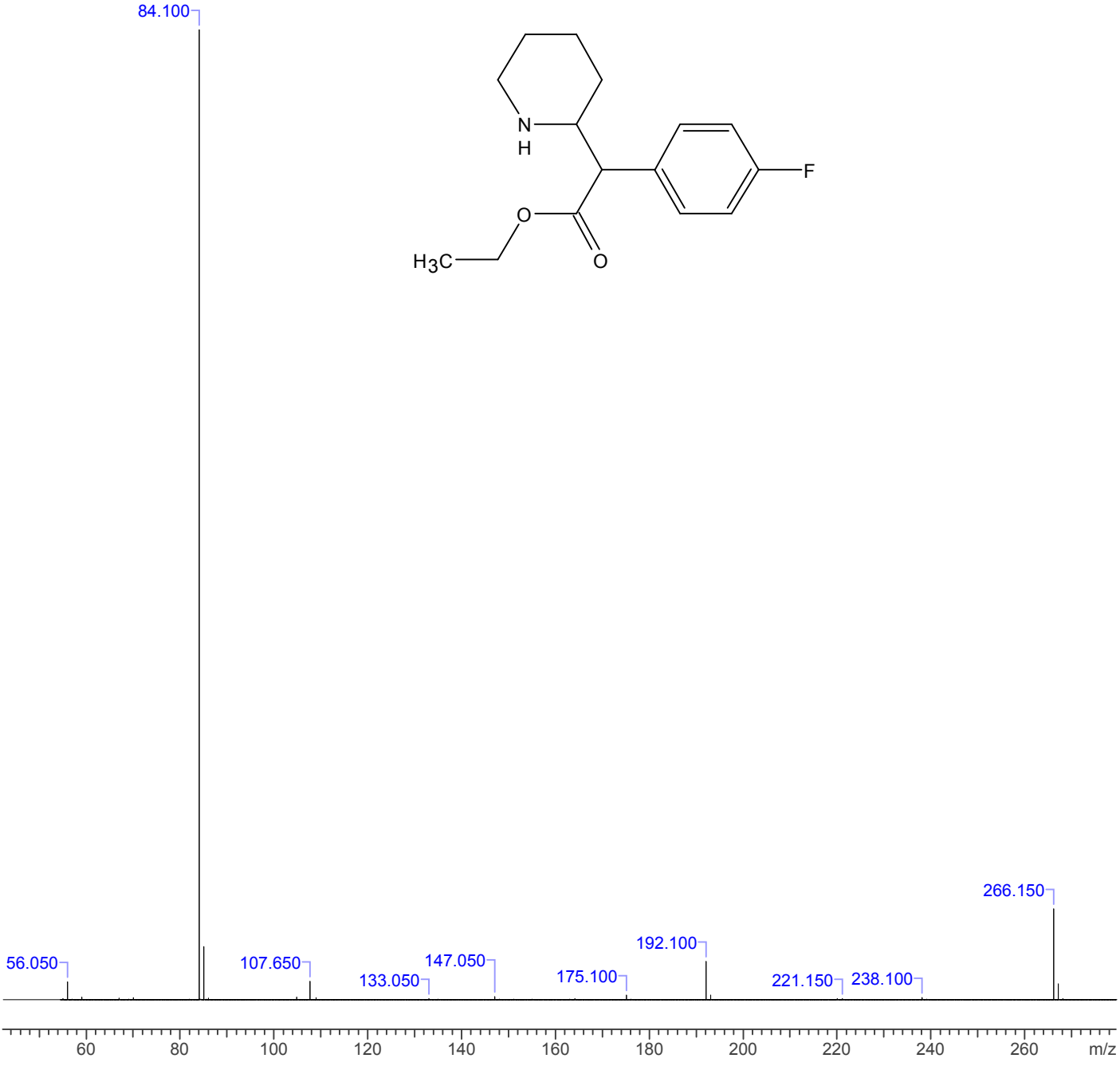
No.	Fragment	Structure	Formula	Label	*m/z* Calc.	TIC Calc. (%)	Difference (Da)	*m/z* Exp.	RI Exp. (%)	TIC Exp. (%)
1	7-12		C5H10N	M - C10H10FO2	84.081	41.842	0.019	84.100	100.000	41.842
2	6,13-19(+H)		C7H6F	M - C8H14NO2	109.045	18.925	-0.045	109.000	44.424	18.925
3	7-9,12(-H)		C3H5N	M - C12H15FO2	55.042	3.938	0.058	55.100	9.628	3.938
4	1-6,13-19(+H)		C10H11FO2	M - C5H9N	182.074	2.014	0.026	182.100	4.550	2.002
5	6-8,13-19		C9H8F	M - C6H12NO2	135.060	1.121	-0.060	135.000	2.574	1.121
6	6-7,11-19(-H2)		C9H7FN	M - C6H13O2	148.056	0.702	-0.056	148.000	1.606	0.702
7	4-10,13-19(-H)		C12H11FO	M - C3H9NO	190.079	0.324	0.021	190.100	0.717	0.324
8	4-8,13-19(-H)		C10H7FO	M - C5H13NO	162.048	0.262	-0.048	162.000	0.593	0.262
9	2-9,13-19		C12H12FO2	M - C3H8N	207.082	0.081	-0.082	207.000	0.178	0.081
10	M		C15H20FNO2	M	265.147	0.006	-0.147	265.000	0.013	0.005

Processing and interpretation based on data provided by:

Service commun des laboratoires France

Count	19022	***Data Type***	Full	***Date***	7:46:52 AM
Date Stamp	04 Feb 2016 3:43 pm			***Estimated Molecular Ion***	1920.8
File Name	C:\Windows\system32\LCQTOF-mrm_1_24417mrm_Compressed (Centroid)				
Mass Spec Model	impact H	***Operator***	alex	***Retention Time***	6.085
Sample	24417mrm	***Scan Mode***	Compressed (Centroid)		
Spectrum Assigned	47.6% [1-1921/0-100]			***Spectrum Type***	MS
TIC	132.39	***Total Signal***	12921917		

16020005_2016-24417_LCQTOF-mrm_1_24417mrm.esp

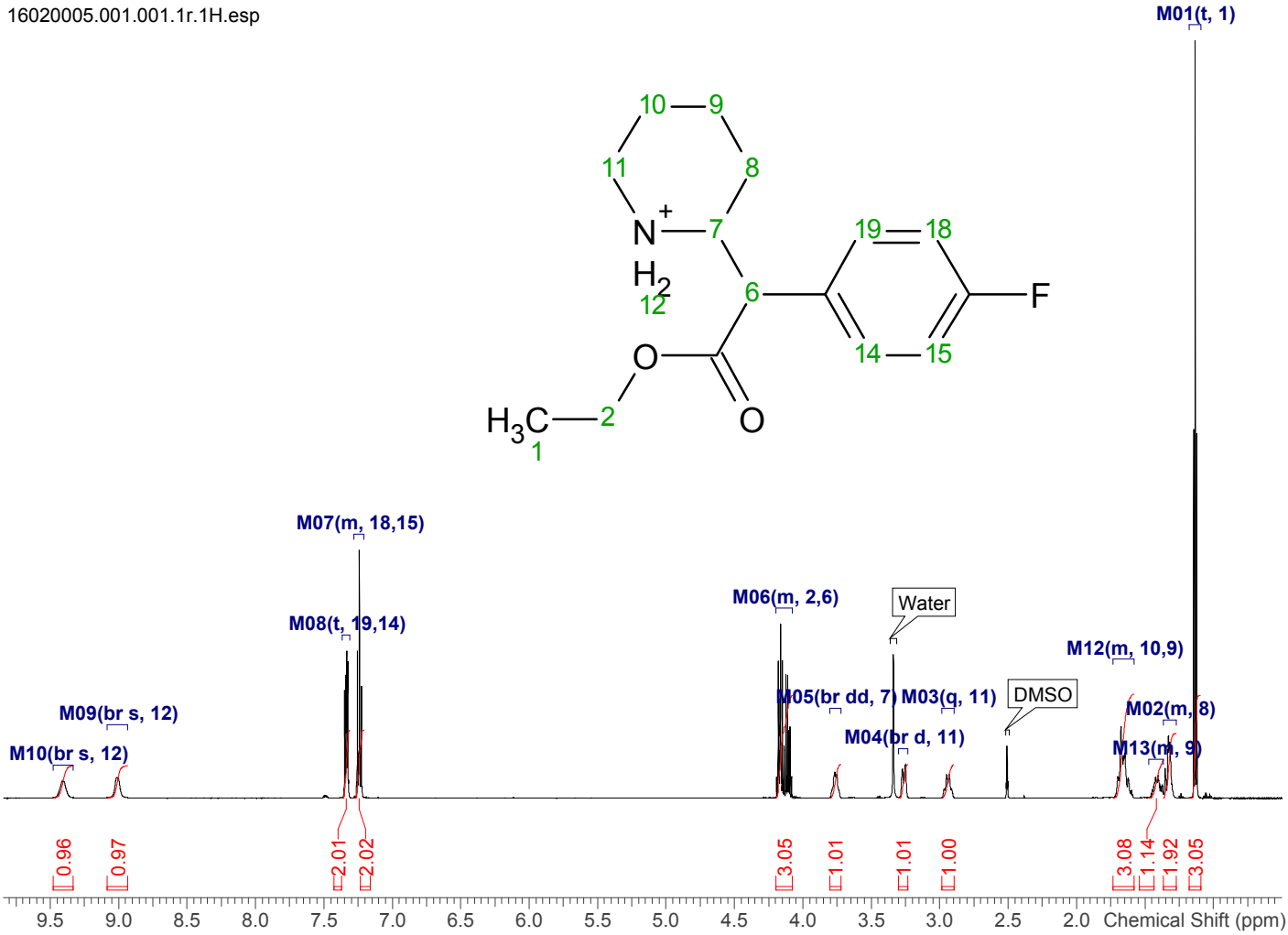


Data from BCIM group at JRC, IHCP Ispra

NMR spectra

Acquisition Time (sec)	2.7263	***Comment***	2016-24417 SCL Paris		
D	0.0002	***D1***	1	***DE***	10
DS	2	***Date***	19 Feb 2016 11:52:41		
Date Stamp	19 Feb 2016 11:52:41				
File Name	\\139.191.6.82\ihcp$\I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_600\16020005_2016-24417\1\PDATA\1\1r				
Frequency (MHz)	600.1300	***GB***	0	***INSTRUM***	<spect>
LB	0	***NS***	256	***Nucleus***	1H
Number of Transients	256	***Origin***	spect	***Original Points Count***	32768
Owner	nmrsu	***PC***	1		
PROBHD	<5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012 >				
PULPROG	<zg30>	***Points Count***	65536	***Pulse Sequence***	zg30
Receiver Gain	1.91	***SF***	600.13	***SFO1***	600.133706048414
SI	65536	***SSB***	0	***SW(cyclical) (Hz)***	12019.23
SWH	12019.2307692308			***Solvent***	DMSO-d6
Spectrum Offset (Hz)	3706.0515	***Spectrum Type***	standard	***Sweep Width (Hz)***	12019.05
TD	65536	***TD0***	1	***TE***	299.9993
Temperature (degree C)	26.999	***UNC1***	<1H>	***WDW***	0

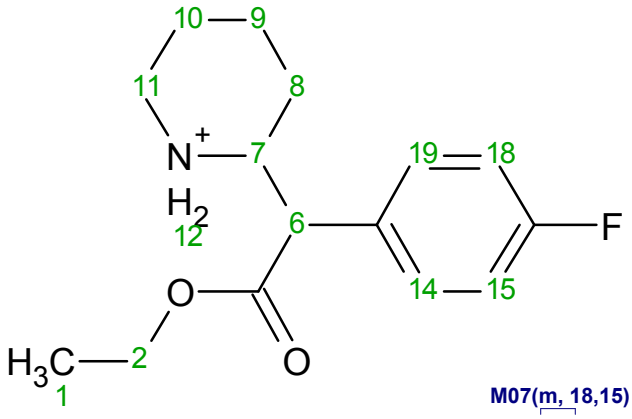
16020005.001.001.1r.1H.esp

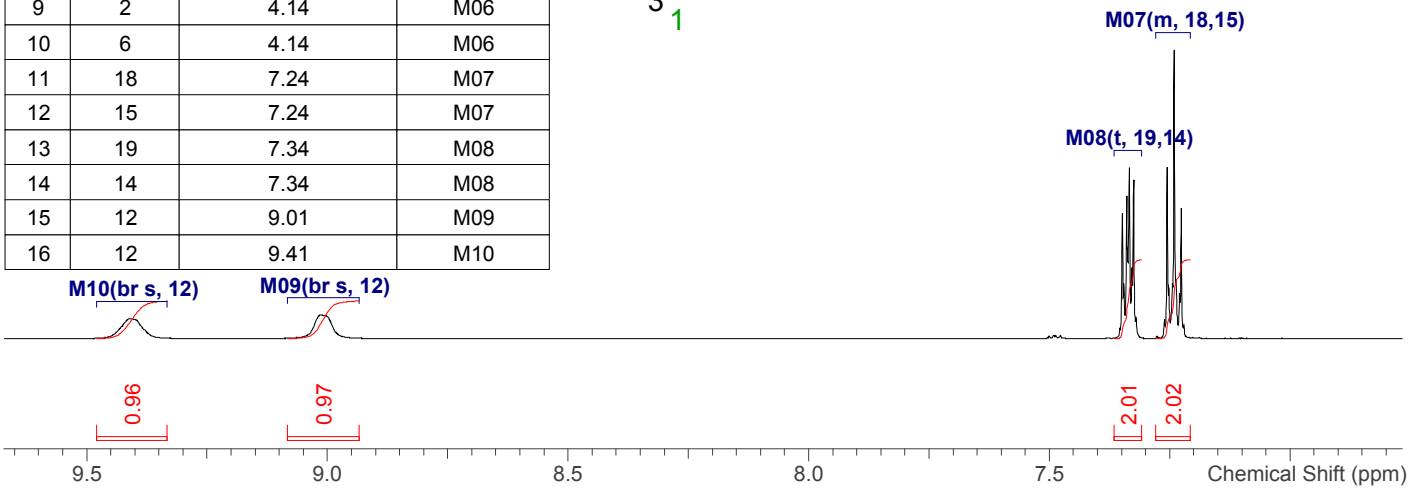


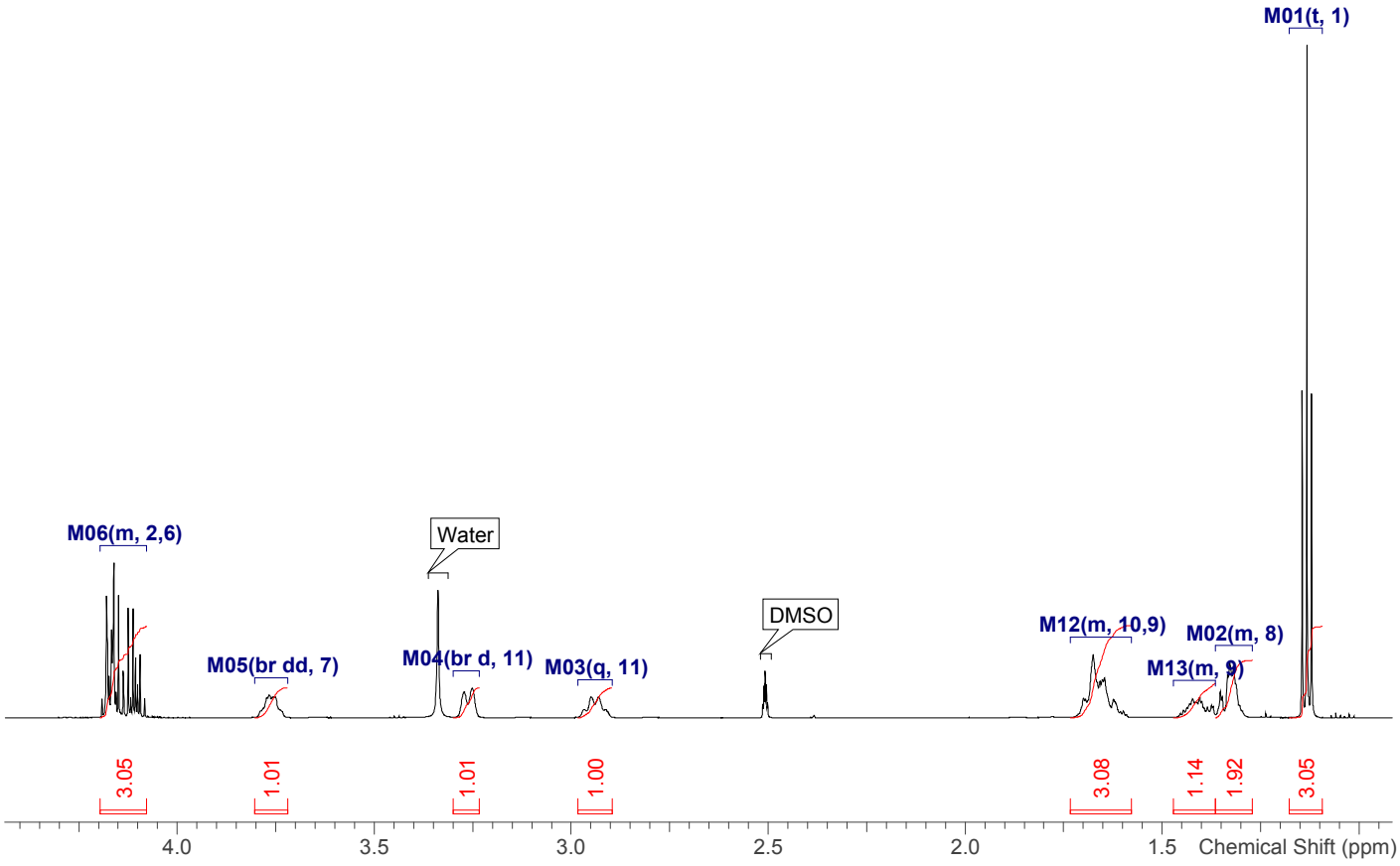
^{1}H NMR (600 MHz, DMSO-d_6) d ppm 1.13 (t, *J*=7.15 Hz, 3 H) 1.27 - 1.36 (m, 2 H) 1.36 - 1.47 (m, 1 H) 1.58 - 1.73 (m, 3 H) 2.94 (q, *J*=10.94 Hz, 1 H) 3.26 (br d, *J*=12.29 Hz, 1 H) 3.76 (br dd, *J*=9.08, 3.39 Hz, 1 H) 4.08 - 4.20 (m, 3 H) 7.21 - 7.28 (m, 2 H) 7.34 (t, *J*=6.70 Hz, 2 H) 9.01 (br s, 1 H) 9.41 (br s, 1 H)

Table of assignments and zoomed parts:

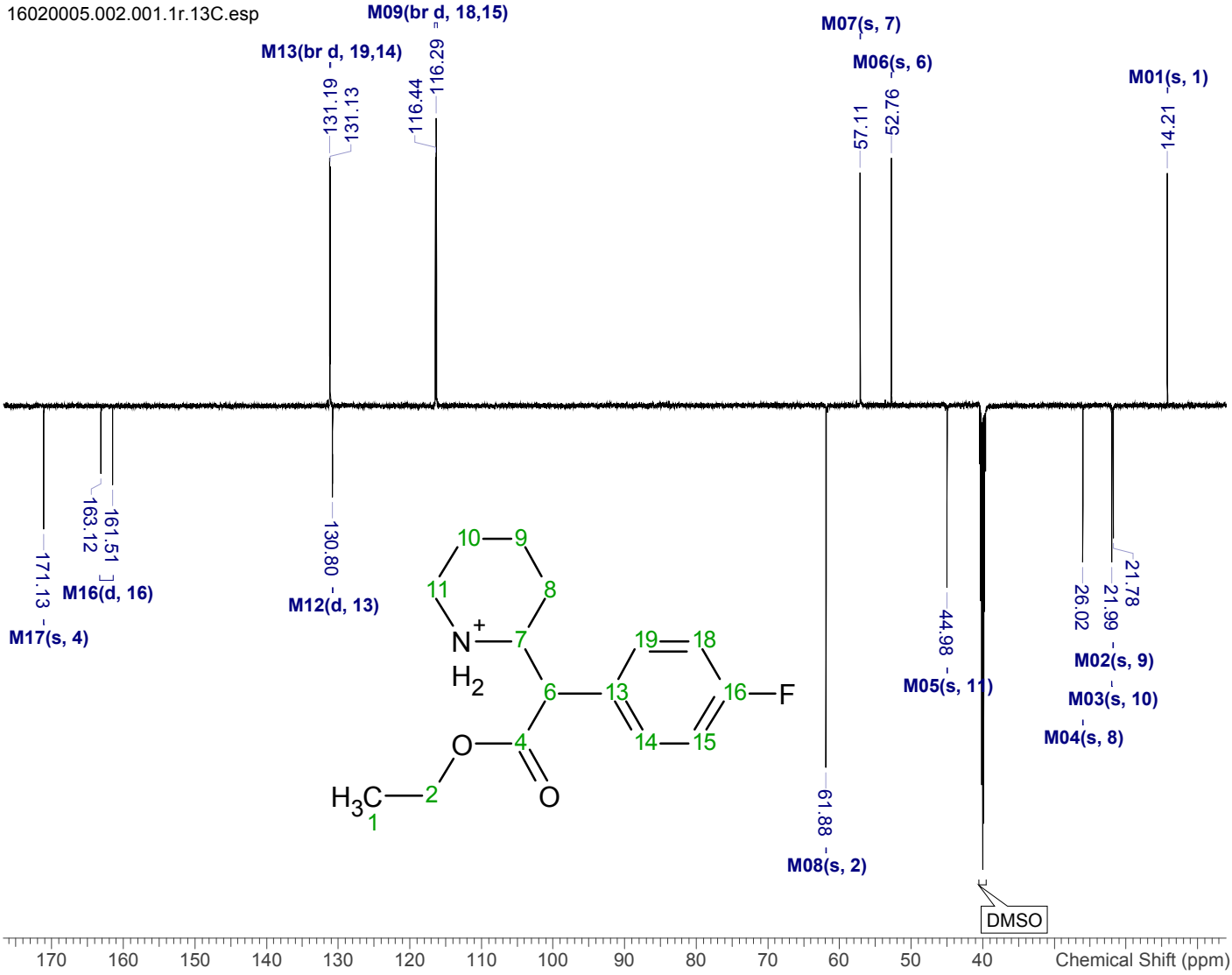
No.	Atom	Exp. Shift (ppm)	Multiplet
1	1	1.13	M01
2	8	1.33	M02
3	9	1.41	M13
4	10	1.66	M12
5	9	1.66	M12
6	11	2.94	M03
7	11	3.26	M04
8	7	3.76	M05
9	2	4.14	M06
10	6	4.14	M06
11	18	7.24	M07
12	15	7.24	M07
13	19	7.34	M08
14	14	7.34	M08
15	12	9.01	M09
16	12	9.41	M10







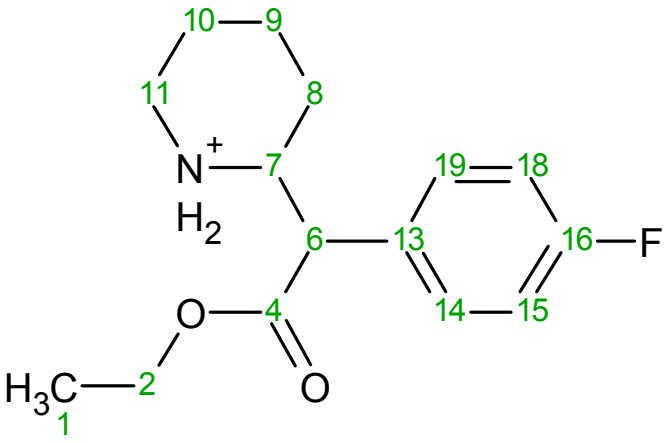
Acquisition Time (sec)	0.9044	***Comment***	5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012		
D	0.0002	***D1***	2	***DE***	18
DS	4	***Date***	19 Feb 2016 12:05:36		
Date Stamp	19 Feb 2016 12:05:36				
File Name	\\139.191.6.82\ihcp$\I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_600\16020005_2016-24417\2\PDATA\1\1r				
Frequency (MHz)	150.9028	***GB***	0	***INSTRUM***	<spect>
LB	1	***NS***	256	***Nucleus***	13C
Number of Transients	256	***Origin***	spect	***Original Points Count***	32768
Owner	nmrsu	***PC***	1		
PROBHD	<5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012 >			***PULPROG***	<jmod>
Points Count	32768	***Pulse Sequence***	jmod	***Receiver Gain***	183.05
SF	150.902809	***SFO1***	150.92091733708		
SI	32768	***SSB***	0	***SW(cyclical) (Hz)***	36231.88
SWH	36231.884057971			***Solvent***	DMSO-d6
Spectrum Offset (Hz)	18108.3359	***Spectrum Type***	APT	***Sweep Width (Hz)***	36230.78
TD	65536	***TD0***	1	***TE***	299.9986
Temperature (degree C)	26.999	***UNC1***	<13C>	***WDW***	1

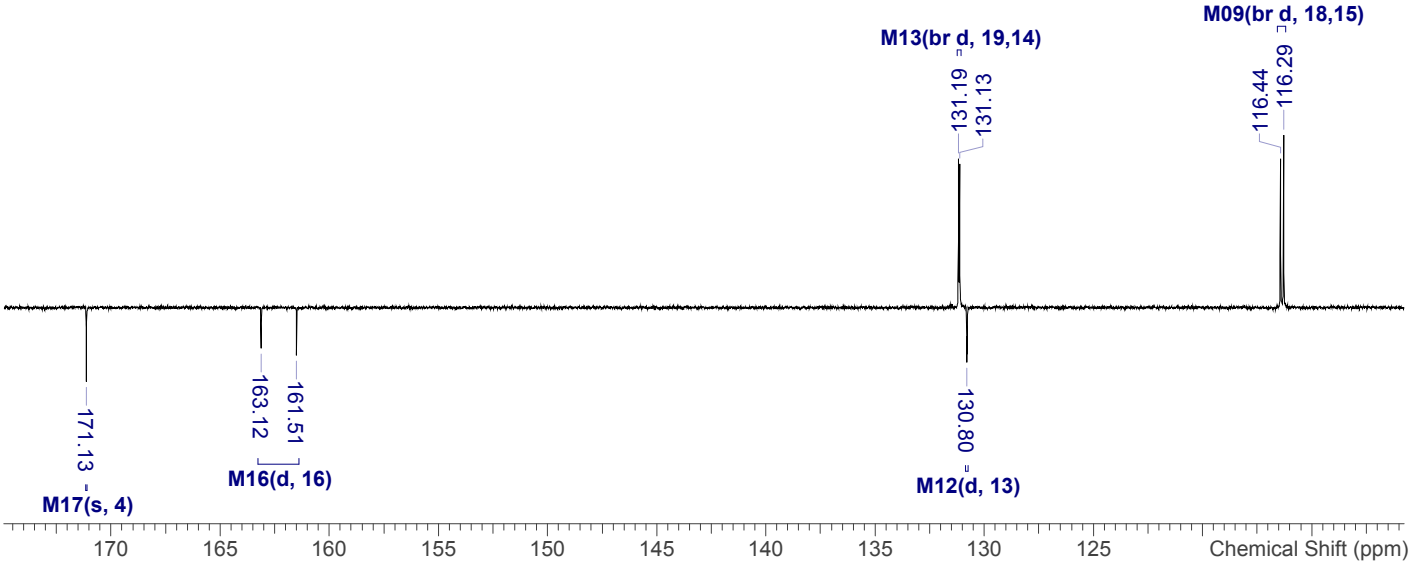


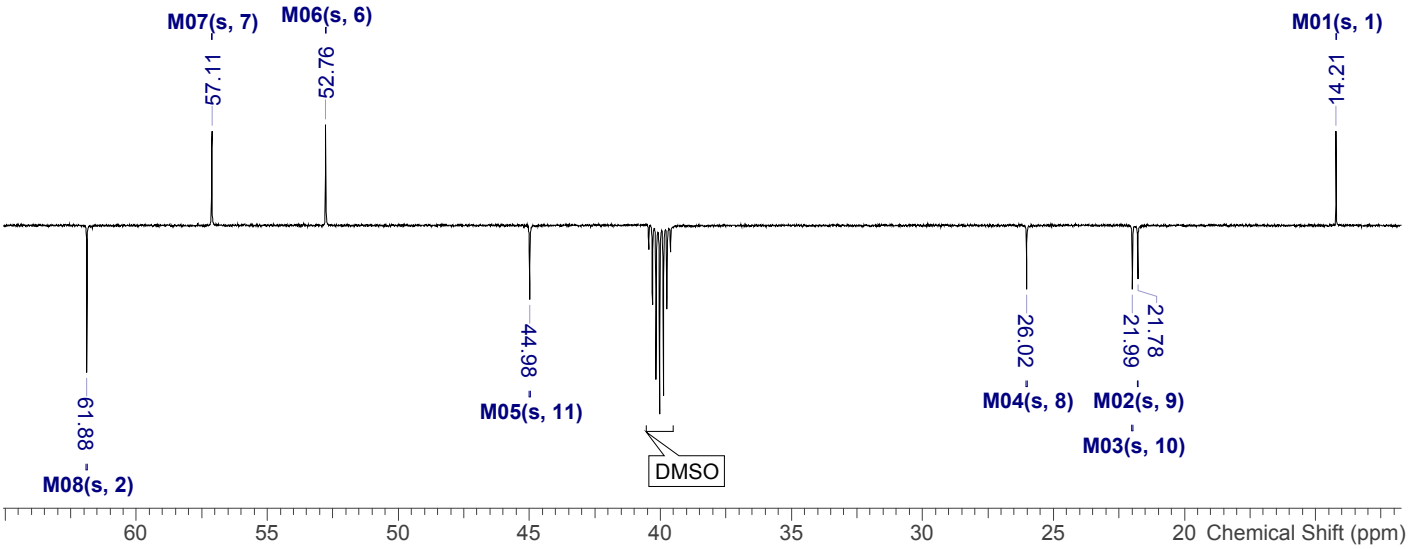
^{13}C NMR (151 MHz, DMSO-d_6) d ppm 14.21 (s, 1 C) 21.78 (s, 1 C) 21.99 (s, 1 C) 26.02 (s, 1 C) 44.98 (s, 1 C) 52.76 (s, 1 C) 57.11 (s, 1 C) 61.88 (s, 1 C) 116.36 (br d, *J*=22.11 Hz, 1 C) 130.79 (d, *J*=3.32 Hz, 1 C) 131.16 (br d, *J*=8.85 Hz, 1 C) 162.32 (d, *J*=244.36 Hz, 1 C) 171.13 (s, 1 C)

Table of assignments and zoomed parts:

No.	Atom	Exp. Shift (ppm)	Multiplet
1	1	14.21	M01
2	9	21.78	M02
3	10	21.99	M03
4	8	26.02	M04
5	11	44.98	M05
6	6	52.76	M06
7	7	57.11	M07
8	2	61.88	M08
9	18	116.36	M09
10	15	116.36	M09
11	13	130.79	M12
12	19	131.16	M13
13	14	131.16	M13
14	16	162.32	M16
15	4	171.13	M17







Fragmentation of the observed molecule and the virtual MS (EI+) spectrum predicted by Thermo Scientific Mass Frontier