European Commission

Joint Research Centre

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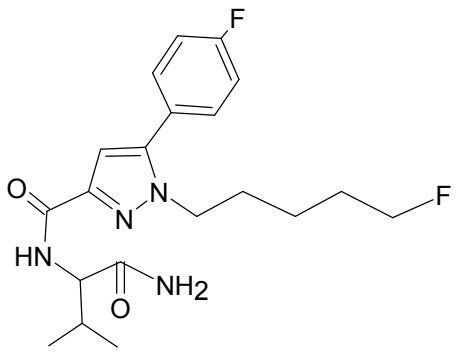
ADMINISTRATIVE ARRANGEMENT JRC-Nr 33619-CLEN2SAND-DG TAXUD-Nr TAXUD/2014/DE/315 BETWEEN DG TAXATION AND CUSTOMS UNION (DG TAXUD) AND THE JOINT RESEARCH CENTRE (JRC) for fast recognition of New Psychoactive Substances (NPS) and identification of unknown chemicals

This report was generated automatically by Kamil Kolar on 27/05/2015 based on data from the European Customs laboratories and the Joint Research Centre. This report includes sample and molecular information, spectra data and associated tables and figures. The chemical structure(s) was/were identified by BCIM group chemists on the basis of analytical data available. The assignments proposed below were performed by using ACD labs tools in agreement with the chemical structure identified by analytical experts. Reported data are connected to the sample in the following table:

Eurodat number	15040014
Received on	13 April 2015
PACKAGING	in 1 glass vial
Registration date	23 April 2015
Name of customer	Service commun des laboratoires France
Customer's identification	2015-25717
Customer's information	2015-25717 white powder, SCL Paris, GC-MSAgilent, FT-IRThermo Nicolet iS10, LC-MSBruker Impact LC-Q-Tof
Customer's comments	hypothetic molecular weight[MH]+ 393.21Hypothetic raw formulaC23H26FN4Ocannabinoid ?

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



Data of identified compound(s)

FW	392.4428
IUPAC Name (v.14.01)	N-(1-amino-3-methyl-1-oxobutan-2-yl)-1-(5-fluoropentyl)-5-(4-fluorophenyl)-1H-pyrazole-3-carboxamide (named manually)
InChI (v.1.04)	InChI=1S/C20H26F2N4O2/c1-13(2)18(19(23)27)24-20(28)16-12-17(14-6-8-15(22)9-7-14)26(25-16)11-5-3-4-10-21/h6-9, 12-13,18H,3-5,10-11H2,1-2H3,(H2,23,27)(H,24,28)
InChI Key (v.1.04)	GSXRDTDYPSATDE-UHFFFAOYSA-N
SMILES (v.14.01)	NC(=O)C(NC(=O)c2cc(c1ccc(F)cc1)n(CCCCCF)n2)C(C)C
Formula	$C_{20}H_{26}F_2N_4O_2$

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

European Commission, Joint Research Centre

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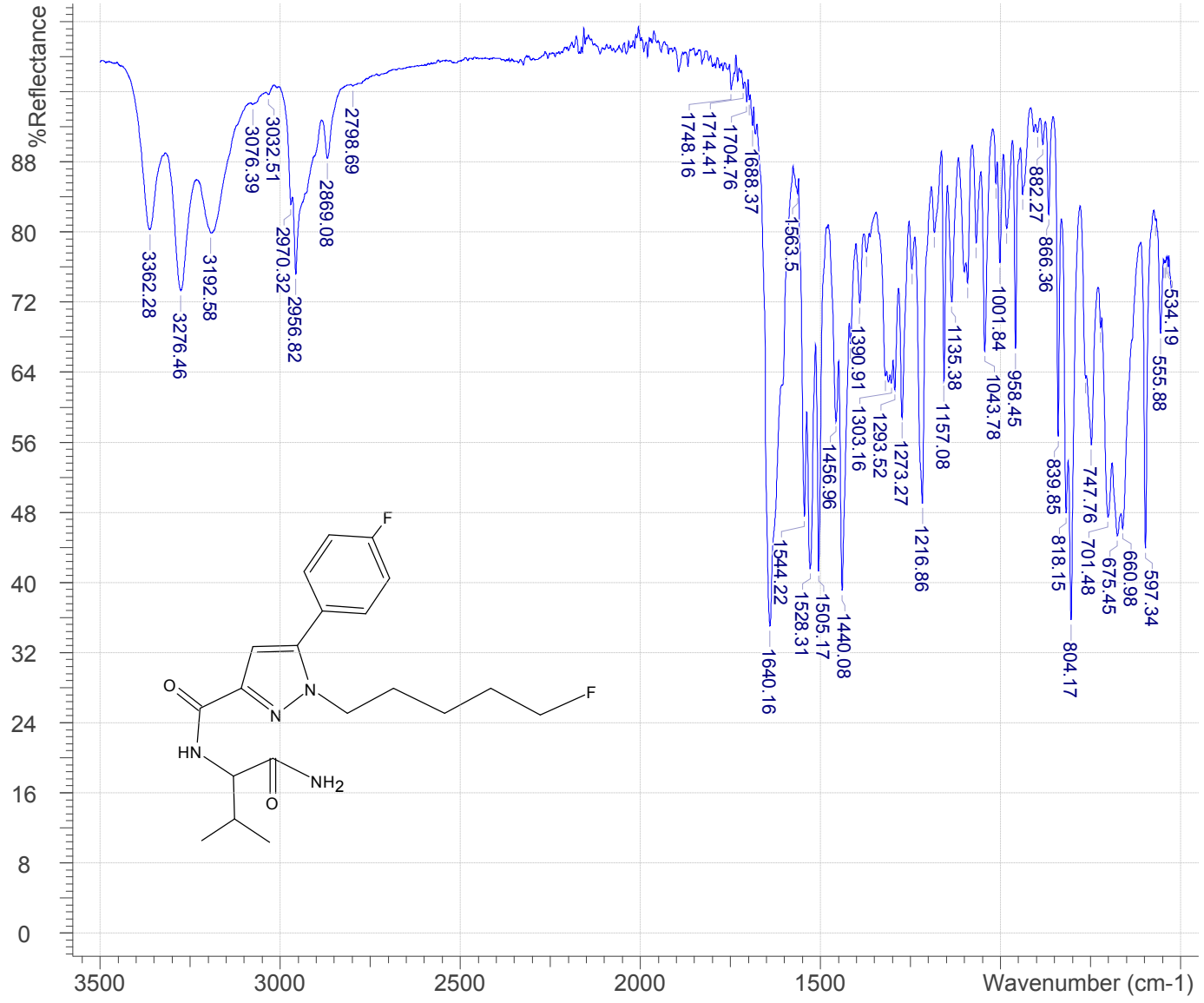
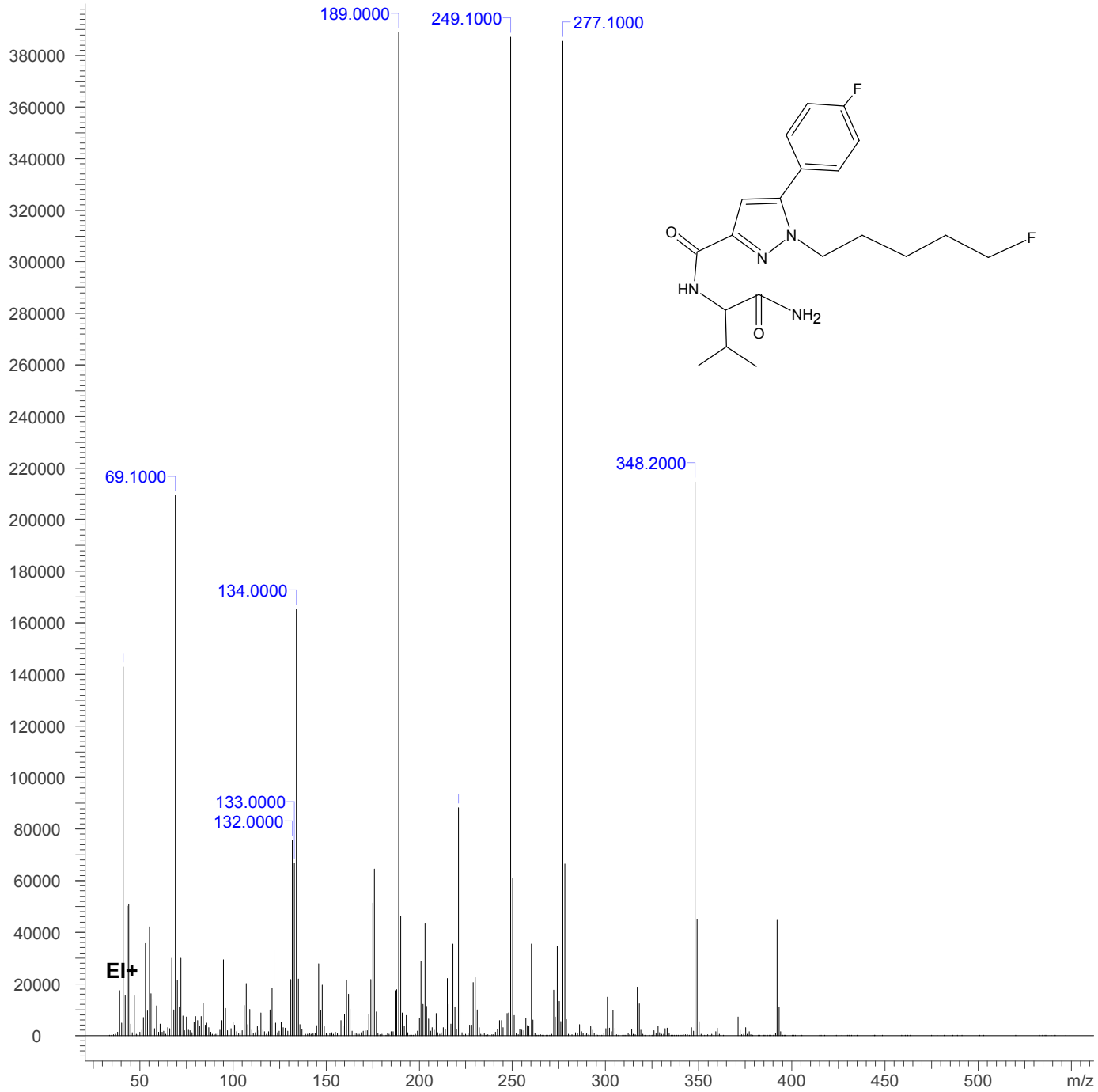
Data measured by SCL Paris:



Title	25717		
File Name	\\s-jrciprna004p-fs-vihcp.jrc.it\ihcp-I01\BIO_CHEMICAL_INTERACTION_METABONOMICS\CLEN2SAND\!PROPOSED-STRUCTURE\CLEN_NAS\15040014\2015-25717\IR.SPA		
Date Stamp	23 Feb 2015 09:01:49	***Date***	23 Feb 2015 11:02:04
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		



Comment	eb	***Count***	394	***Data Type***	Condensed
Date	18 Feb 2015 22:59:16			***Date Stamp***	18 Feb 15 9:47 pm
Estimated Molecular Ion	547.000	***File Name***	2015-25717_Centroid_EI+		
Inlet Model	GC	***Ion Mode***	EI+	***Mass Spec Model***	STUP1
Plot Type	Stick	***Retention Time***	6.770	***Sample***	Ds CH2Cl2/CH3OH : 2015-25717
Scan	1726	***Scan Mode***	Centroid	***Spectrum Assigned***	41.0% [34-549/0-100]
TIC	1074.62	***Total Signal***	4180201		

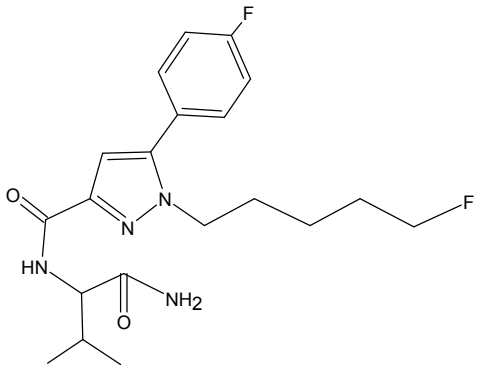
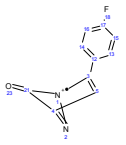
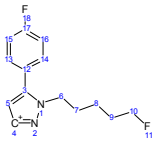
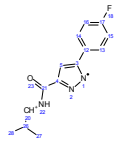
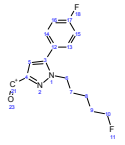
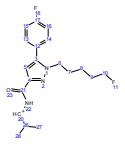
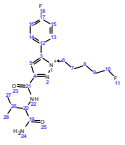


Table of fragments

N	Fragment	Structure	Formula	Label	*m/z* Calc	TIC Ca (%)	Differe (Da	*m/z* Exp.	RI Exp. (%)	TIC Ex (%)	AQ	RI	Or	Comment
1	1-5,12-18,21,23(+H)		C10H6FN2O	M - C10H20FN2O	189.046	10.499	-0.046	189.000	100.000	10.499	1.0	9.	Ma	al
2	1-18		C14H15F2N2	M - C6H11N2O2	249.120	10.905	-0.020	249.100	99.506	10.905	1.0	7.	Ma	al
3	1-5,12-18,20-23,26-28(+H)		C14H15FN3O	M - C6H11FNO	260.119	1.004	-0.019	260.100	9.105	1.004	1.0	9.	Ma	al
4	1-18,21,23		C15H15F2N2O	M - C5H11N2O	277.115	11.006	-0.015	277.100	99.095	11.006	1.0	8.	Ma	al
5	1-18,20-23,26-28		C19H24F2N3O	M - CH2NO	348.188	6.438	0.012	348.200	55.199	6.431	0.9	8.	Ma	al
6	M		C20H26F2N4O2	M	392.202	1.365	-0.002	392.200	11.500	1.365	1.0	9.	Ma	al

Data measured by BCIM group at IHCP JRC Ispra

NMR spectra

Acquisition Time (sec)	2.7263	***Comment***	25718 (F) 10mg in 600 uL DMSO-d6
Date	14 Apr 2015 17:49:42	***Date Stamp***	14 Apr 2015 17:49:42
File Name	\\s-jrciprna004p-fs-vihcp.jrc.it\ihcp-I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!KK-proposed-structure-temp-dir\BCIM_NAS\BCIM_600\15040003\1\PDATA\1\1r		
Frequency (MHz)	600.13	***Nucleus***	1H
Number of Transients	16	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu
Points Count	65536	***Pulse Sequence***	zg30
Receiver Gain	1.91	***SW(cyclical) (Hz)***	12019.23
Solvent	DMSO-d6	***Spectrum Offset (Hz)***	3706.0515
Spectrum Type	standard	***Sweep Width (Hz)***	12019.05
Temperature (degree C)	27.000		

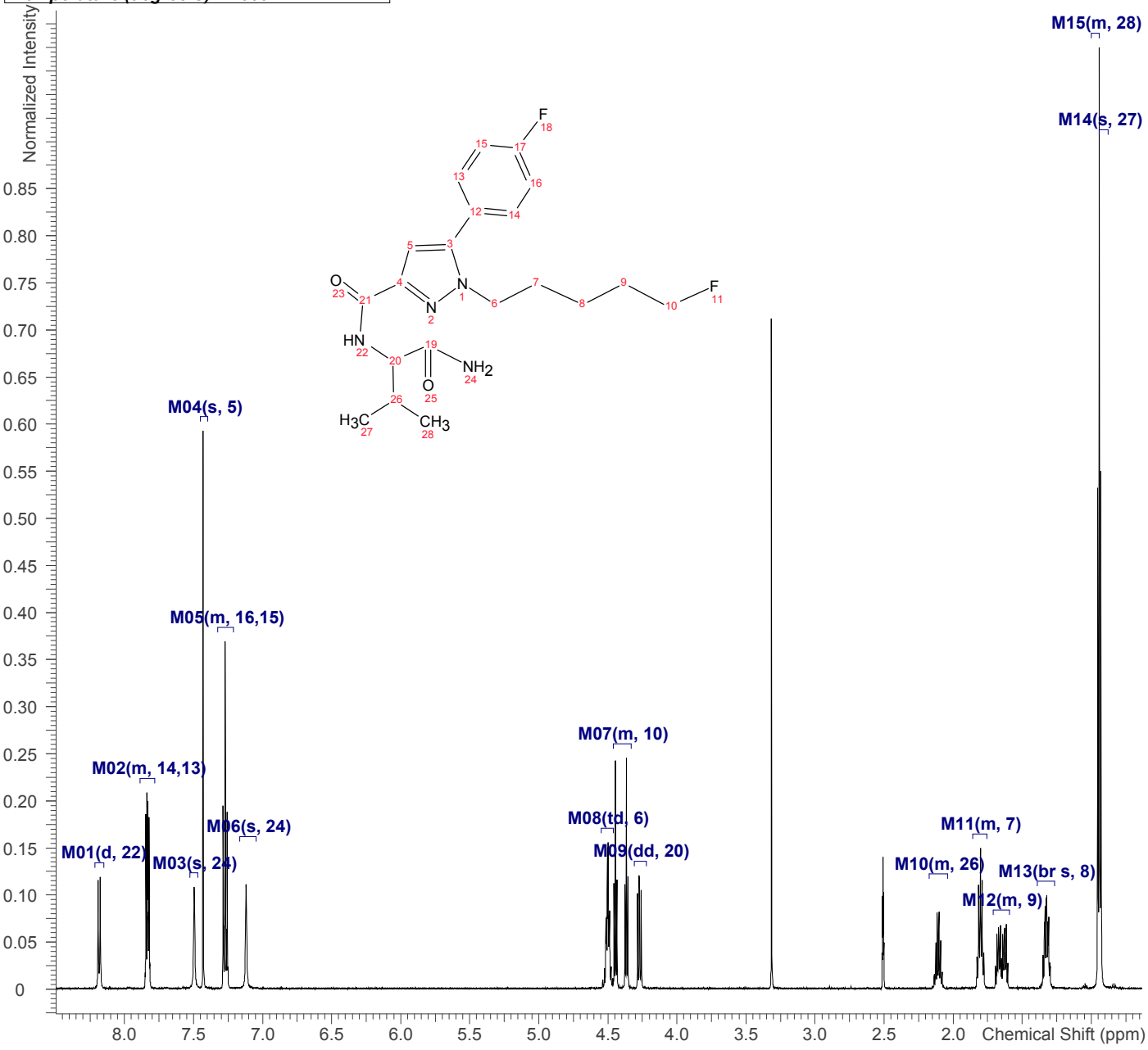
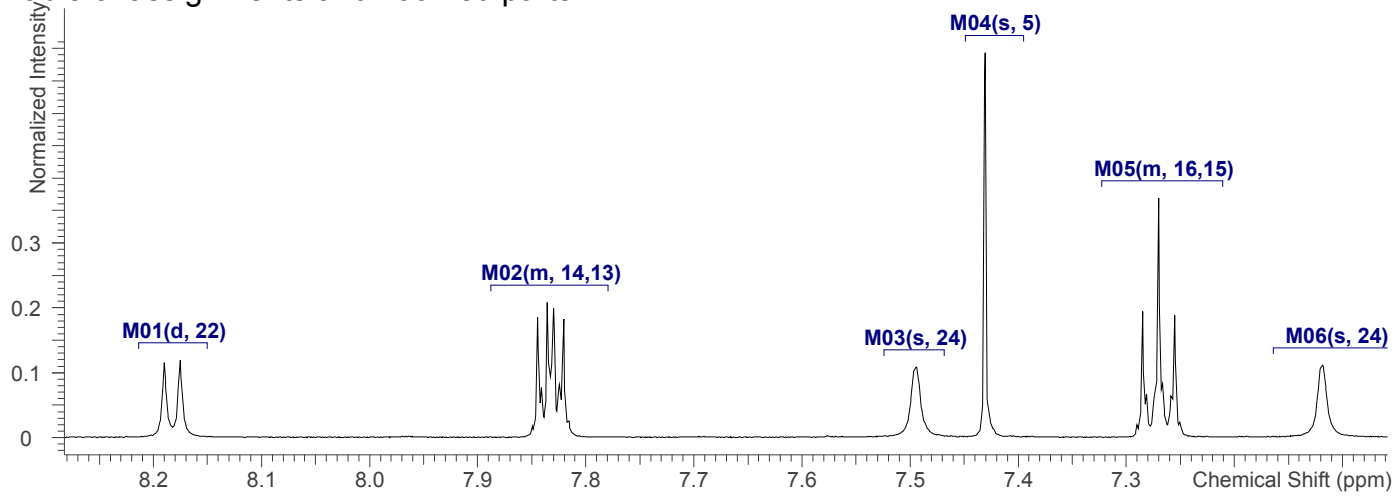
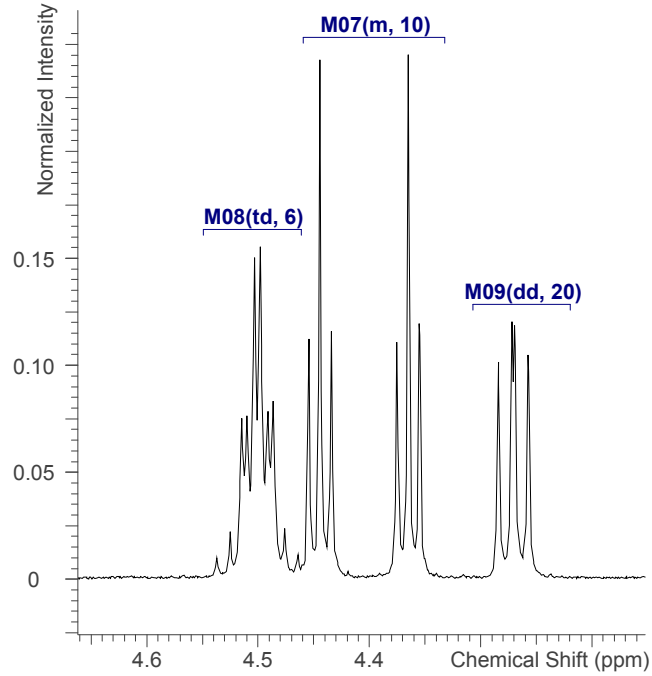
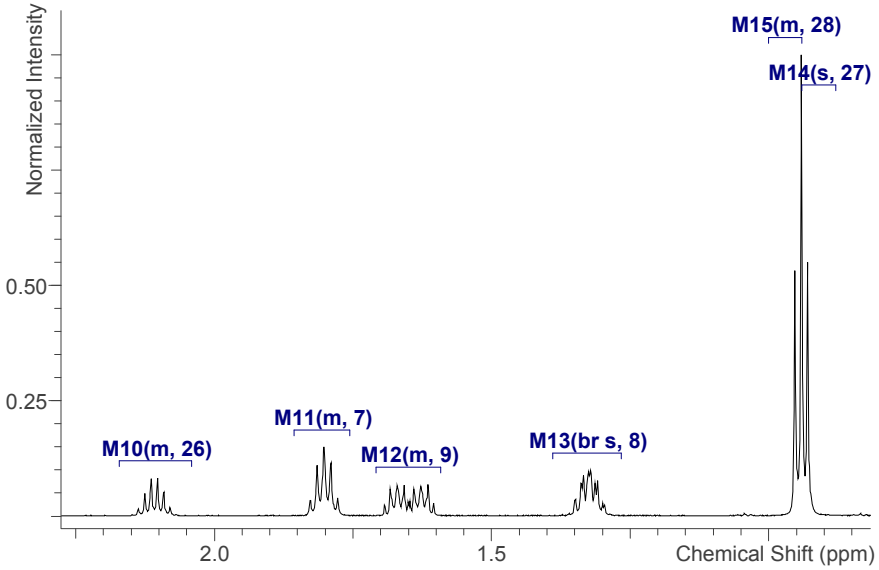


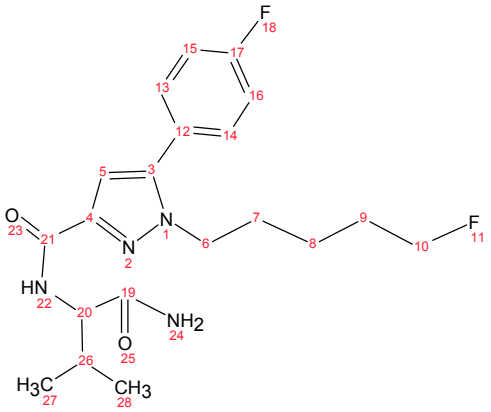
Table of assignments and zoomed parts:





No.	Atom	Exp. Shift (ppm)	Multiplet
1	27	0.93	M14
2	28	0.95	M15
3	8	1.32	M13
4	9	1.64	M12
5	7	1.80	M11
6	26	2.11	M10
7	20	4.27	M09
8	10	4.40	M07
9	6	4.50	M08
10	24	7.12	M06
11	16	7.27	M05
12	15	7.27	M05
13	5	7.43	M04
14	24	7.49	M03
15	14	7.83	M02
16	13	7.83	M02
17	22	8.18	M01





Acquisition Time (sec)	0.9044	***Comment***	5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012
Date	14 Apr 2015 18:02:01	***Date Stamp***	14 Apr 2015 18:02:01
File Name	\\s-jrciprna004p-fs-vihcp.jrc.it\ihcp·I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_600\15040014\3\PDATA\1\1r		
Frequency (MHz)	150.90	***Nucleus***	13C
Number of Transients	512	***Origin***	spect
Original Points Count	32768	***Owner***	nmrsu
Points Count	32768	***Pulse Sequence***	jmod
Receiver Gain	81.72	***SW(cyclical) (Hz)***	36231.88
Solvent	DMSO-d6	***Spectrum Offset (Hz)***	18108.3359
Spectrum Type	APT	***Sweep Width (Hz)***	36230.78
Temperature (degree C)	27.002		

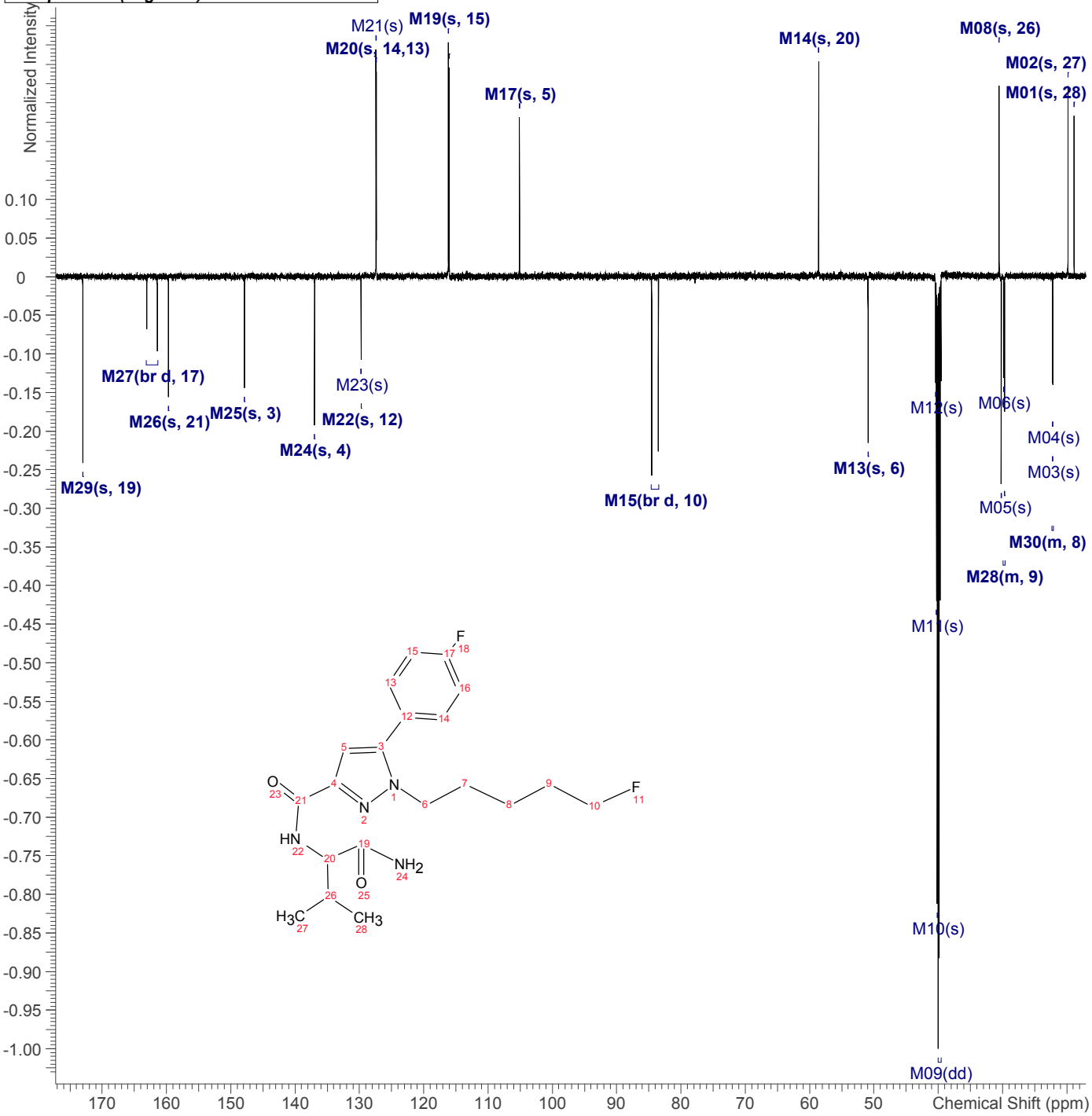
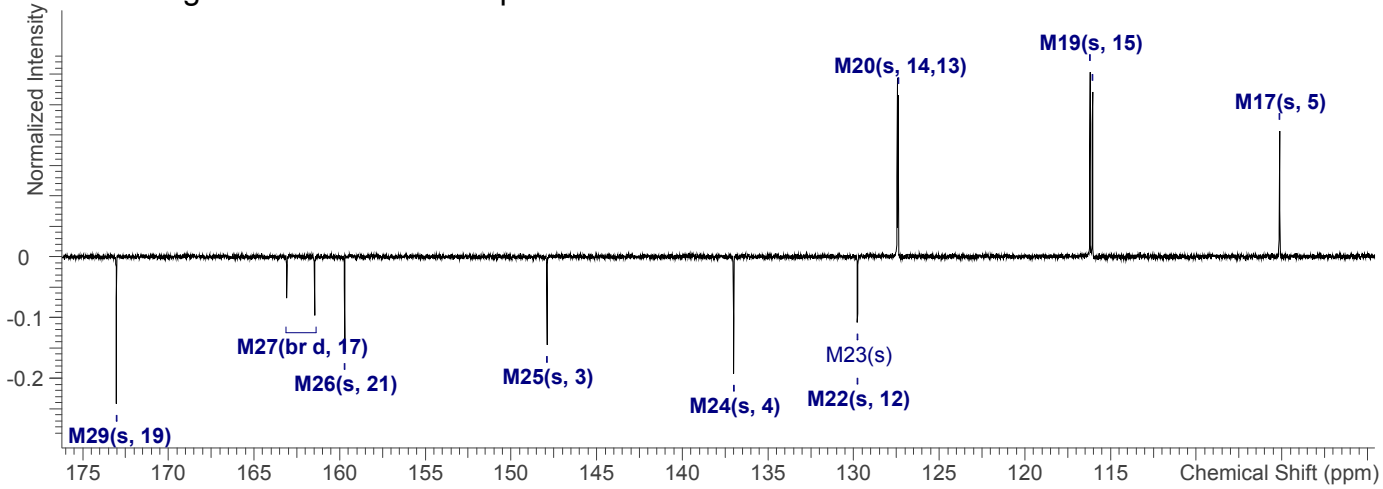
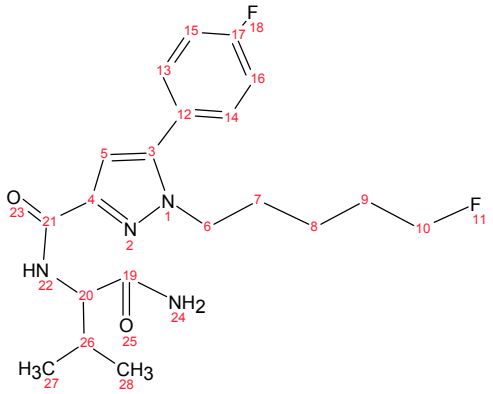
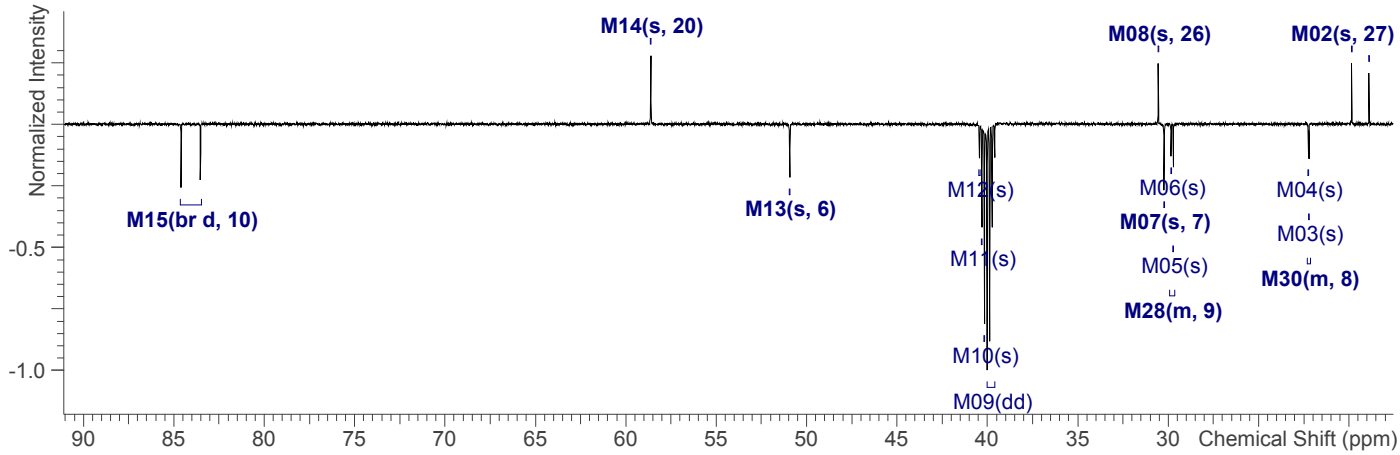


Table of assignments and zoomed parts:



No.	Atom	Exp. Shift (ppm)	Multiplet
1	28	18.89	M01
2	27	19.84	M02
3	8	22.21	M30
4	9	29.78	M28
5	7	30.22	M07
6	26	30.54	M08
7	6	50.93	M13
8	20	58.61	M14
9	10	84.06	M15
10	5	105.13	M17
11	16	116.05	M18
12	15	116.20	M19
13	14	127.40	M20
14	13	127.40	M20
15	12	129.76	M22
16	4	137.01	M24
17	3	147.91	M25
18	21	159.72	M26
19	17	162.29	M27
20	19	173.05	M29





Acquisition Time (sec)	(0.2840, 0.0206)	***Comment***	5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012
Date	15 Apr 2015 09:34:54		
File Name	\\s-jrciprna004p-fs-vihcp.jrc.it\ihcp-I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_600\15040014\6\PDATA\1\2rr		
Frequency (MHz)	(600.13, 150.90)	***Nucleus***	(1H, 13C)
Number of Transients	4	***Origin***	spect
Original Points Count	(2048, 512)	***Owner***	nmrsu
Points Count	(1024, 1024)	***Pulse Sequence***	hsqcedetgpsisp2.3
Solvent	DMSO-d6	***Spectrum Type***	HSQC
Sweep Width (Hz)	(7204.50, 24851.33)	***Temperature (degree C)***	27.001

15040014.006.001.2rr.man-assign.esp

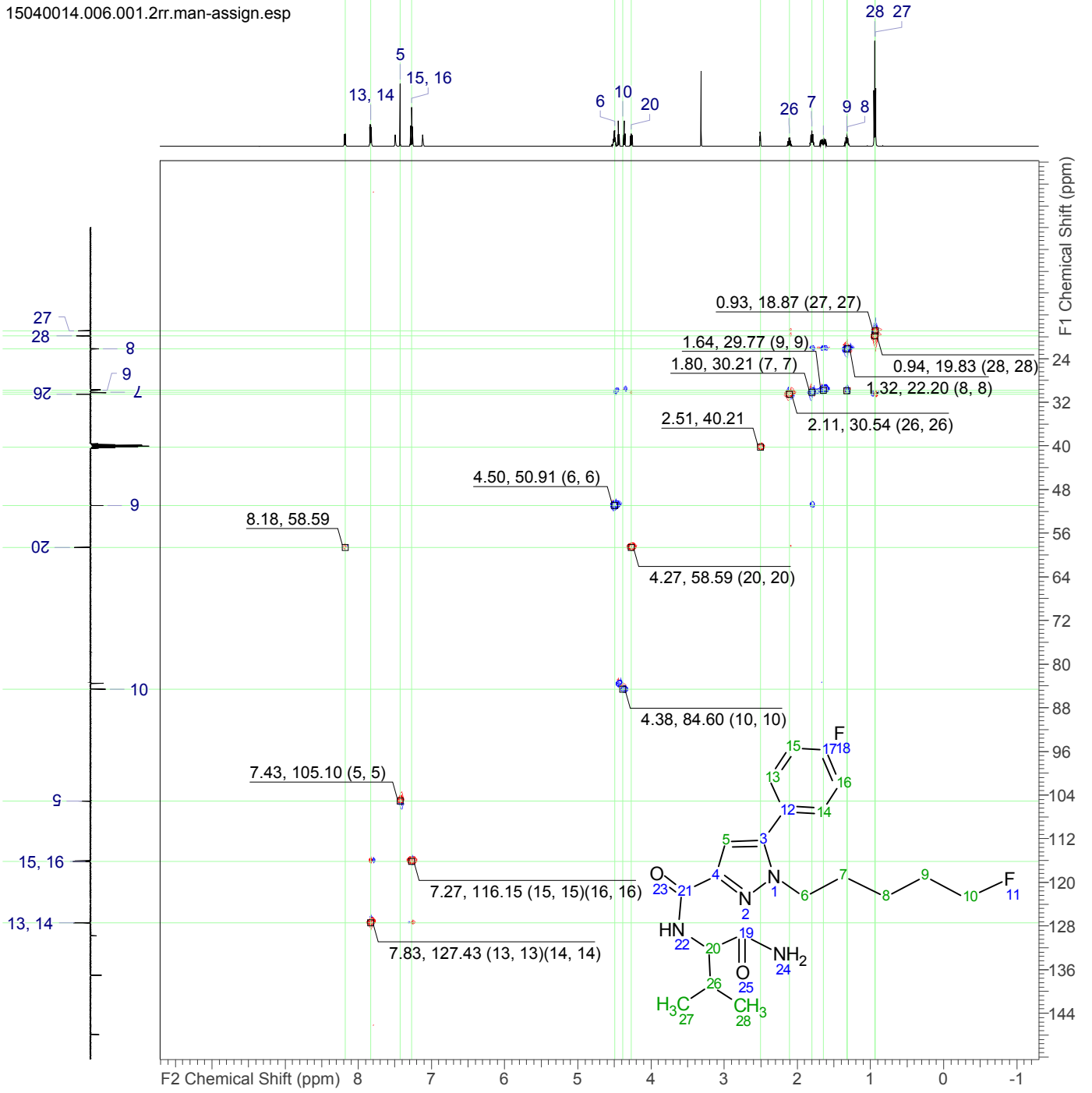
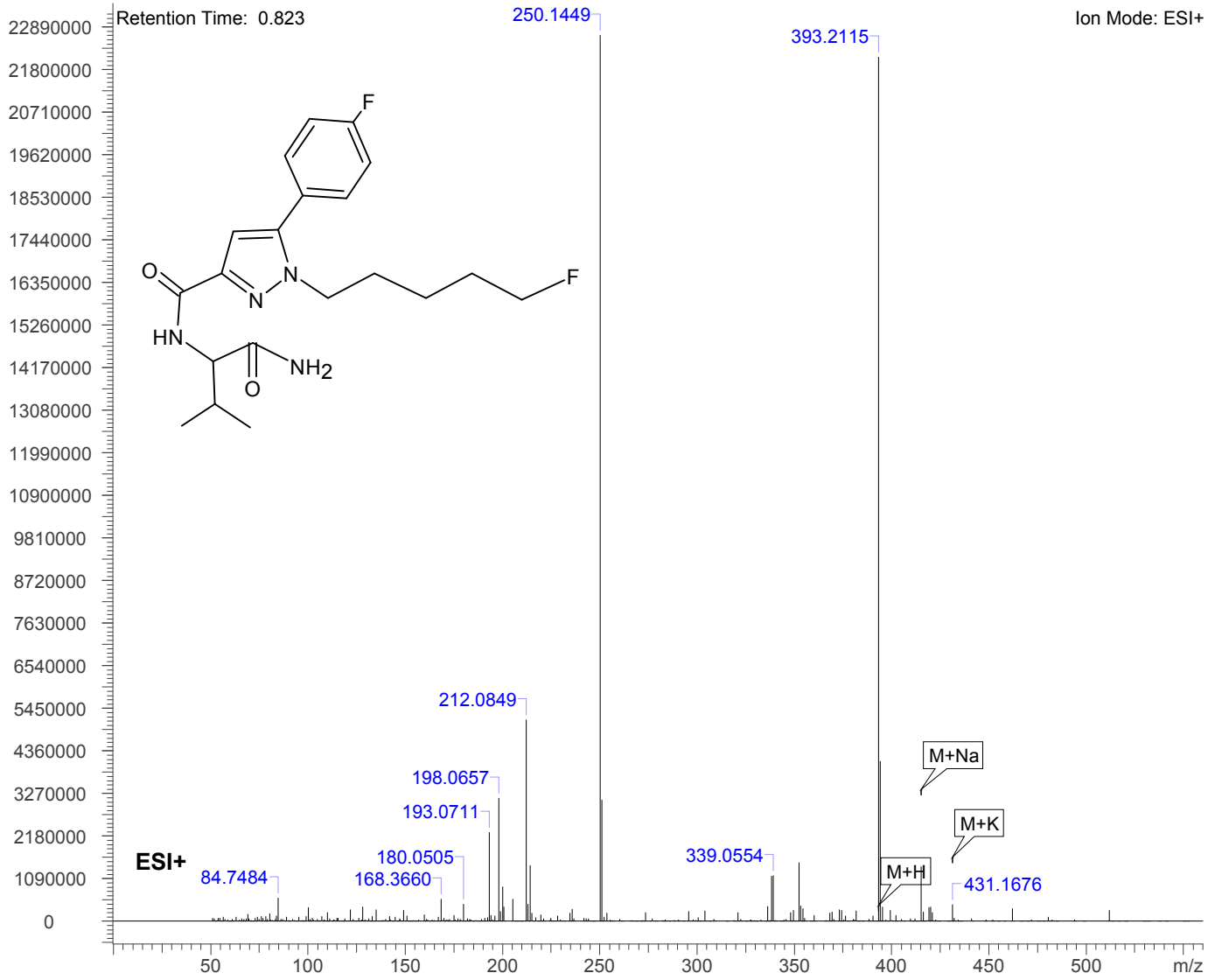


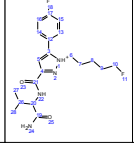
Table of assignments:

No.	F2 Atom	F1 Atom	F2 (ppm)	F1 (ppm)
1	5	5	7.43	105.10
2	6	6	4.50	50.91
3	7	7	1.80	30.21
4	8	8	1.32	22.20
5	9	9	1.64	29.77
6	9	9	1.32	29.87
7	10	10	4.38	84.60
8	13	13	7.83	127.43
9	14	14	7.83	127.43
10	15	15	7.27	116.15
11	16	16	7.27	116.15
12	20	20	4.27	58.59
13	26	26	2.11	30.54
14	27	27	0.93	18.87
15	28	28	0.94	19.83

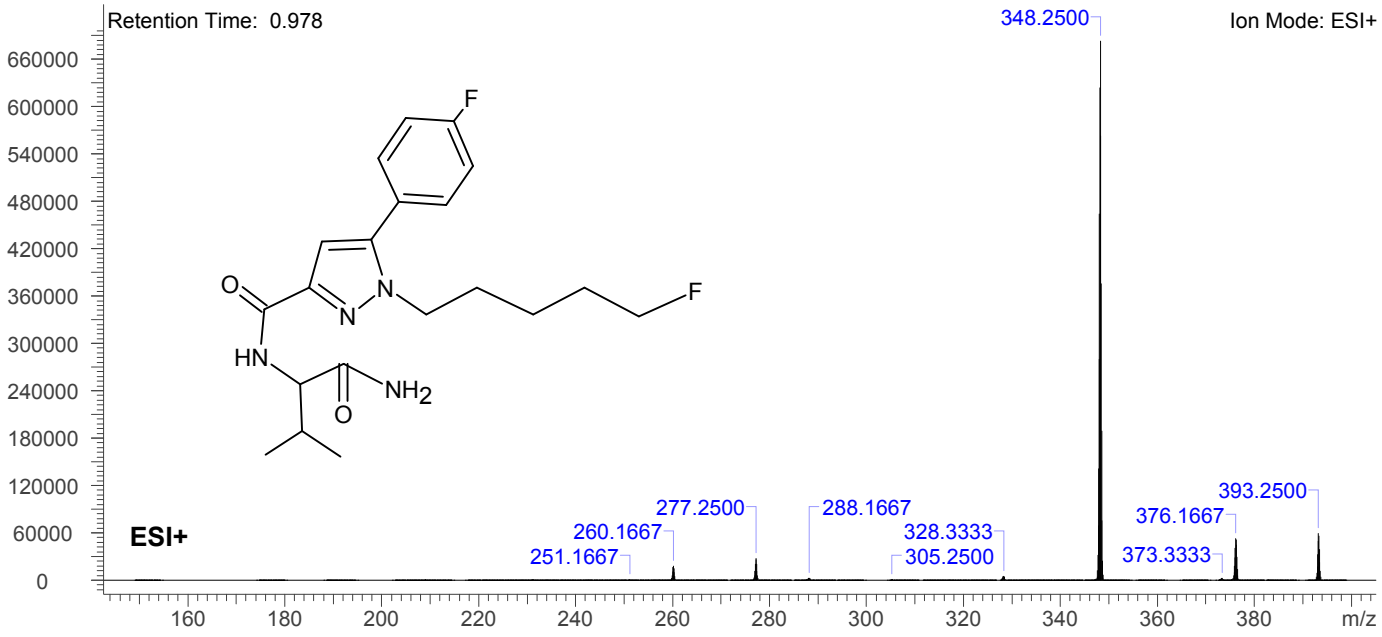
MS spectra

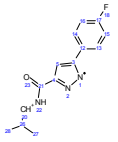
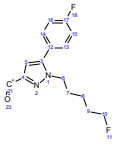
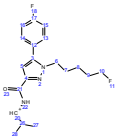
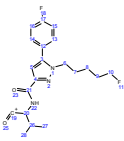
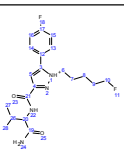
Combine	<84-87>	***Count***	409
Data Source Name	15040014_25717_MS mode_50_1000_RES30000_1		
Data Type	p	***Date***	24 Apr 2015
Estimated Molecular Ion	996.921		
File Name	\\s-jrciprna004p-fs-vihcp.jrc.it\ihcp-I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_Orbi\15040014_25717\15040014_25717_MS mode_50_1000_RES30000.raw		
Ion Mode	ESI+	***Mass Spec Model***	LTQ Orbitrap/LTQ Orbitrap
Plot Type	Profile	***Retention Time***	0.823
Scan	85	***Scan Filter***	FTMS + p ESI Full ms [50.00-1000.00]
Scan Mode	ms	***Scan Type***	Full
Spectrum Assigned	27.8% [50-997/0-100]	***TIC***	421.47
Total Signal	95607024		



No.	Fragment	Structure	Formula	Label	*m/z* Calc.	TIC Calc. (%)	Difference (Da)	*m/z* Exp.	RI Exp. (%)	TIC Exp. (%)	AQI	RDBE	Origin	Comment
1	M(+H)		C20H27F2N4O2	M + H	393.210	29.502	0.002	393.212	97.507	28.402	0.963	8.5	Manual	

Count	1915	***Data Type***	p
Date	20 Apr 2015	***Estimated Molecular Ion***	393.500
File Name	\\s-jrciprna004p-fs-vihcp.jrc.it\ihcp·.I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_Orbi\15040014_25717\15040014_25717_MSMS mode_393_CE 20_105_400.raw		
Ion Mode	ESI+	***Mass Spec Model***	LTQ Orbitrap/LTQ Orbitrap
Plot Type	Profile	***Retention Time***	0.978
Scan	120	***Scan Filter***	ITMS + p ESI Full ms2 393.21@cid20.00 [105.00-400.00]
Scan Mode	ms2	***Scan Type***	Full
Spectrum Assigned	17.5% [149-399/0-100]	***TIC***	654.02
Total Signal	4466182.5		



N	Fragment	Structure	Formula	Label	*m/z* Calc	TIC Ca (%)	Differe (Da	*m/z* Exp	RI Exp. (%)	TIC Ex (%)	AQI	RDI	Origin	Comment
1	1-5,12-18,20-23,26-28(+H)		C14H15FN3O	M - C6H11FNO	260.119	0.427	-0.036	260.083	2.356	0.367	0.861	9.0	Manu	
2	1-18,21,23		C15H15F2N2O	M - C5H11N2O	277.115	0.538	-0.031	277.083	2.946	0.472	0.878	8.5	Manu	
3	1-18,20-23,26-28		C19H24F2N3O	M - CH2NO	348.188	17.513	-0.022	348.167	91.391	14.355	0.820	8.5	Manu	
4	1-23,25-28		C20H24F2N3O2	M - H2N	376.183	1.503	-0.016	376.167	7.737	1.222	0.813	9.5	Manu	
5	M(+H)		C20H27F2N4O2	M + H	393.210	1.693	0.040	393.250	8.683	1.382	0.816	8.5	Manu	

Fragmentation of the observed molecule and the virtual MS and MSMS spectra predicted by Thermo Scientific Mass Frontier