European Commission

Joint Research Centre

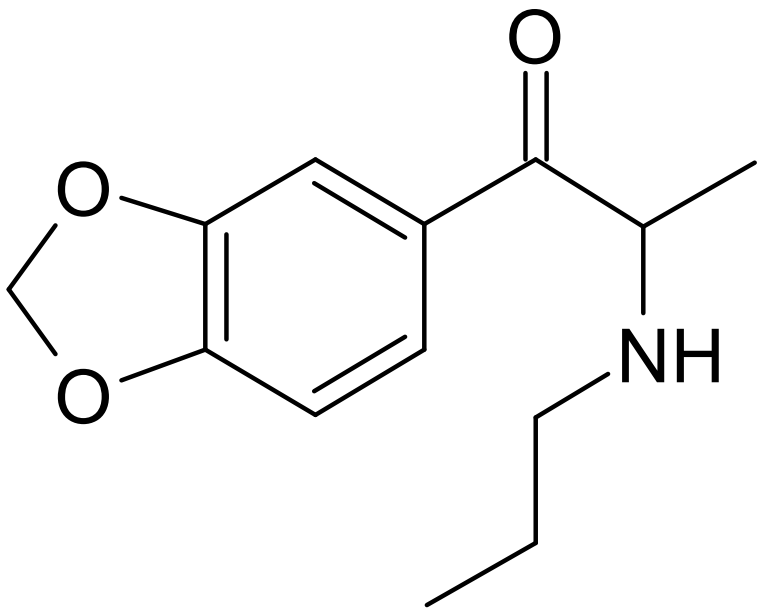
The European Commission's in-house science service

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 02/03/2016 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	16020009	***Received on***	22 February 2016
PACKAGING	1* glass vial 50 mg white powder		
Name of customer	Belgian Customs Laboratory		
Customer's identification	15BD-00431-01		

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



Data of identified compound(s)

IUPAC Name (v.14.02)	1-(2*H*-1,3-benzodioxol-5-yl)-2-(propylamino)propan-1-one		
Formula	$C_{13}H_{17}NO_3$	***FW***	235.2790
Monoisotopic Mass	235.120843		
InChI (v.1.04)	InChI=1S/C13H17NO3/c1-3-6-14-9(2)13(15)10-4-5-11-12(7-10)17-8-16-11/h4-5,7,9,14H,3,6,8H2,1-2H3		
InChI Key (v.1.04)	YFVKHKCZBSGZPE-UHFFFAOYSA-N	***SMILES (v.14.02)***	CC(NCCC)C(=O)c1ccc2OCOc2c1

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

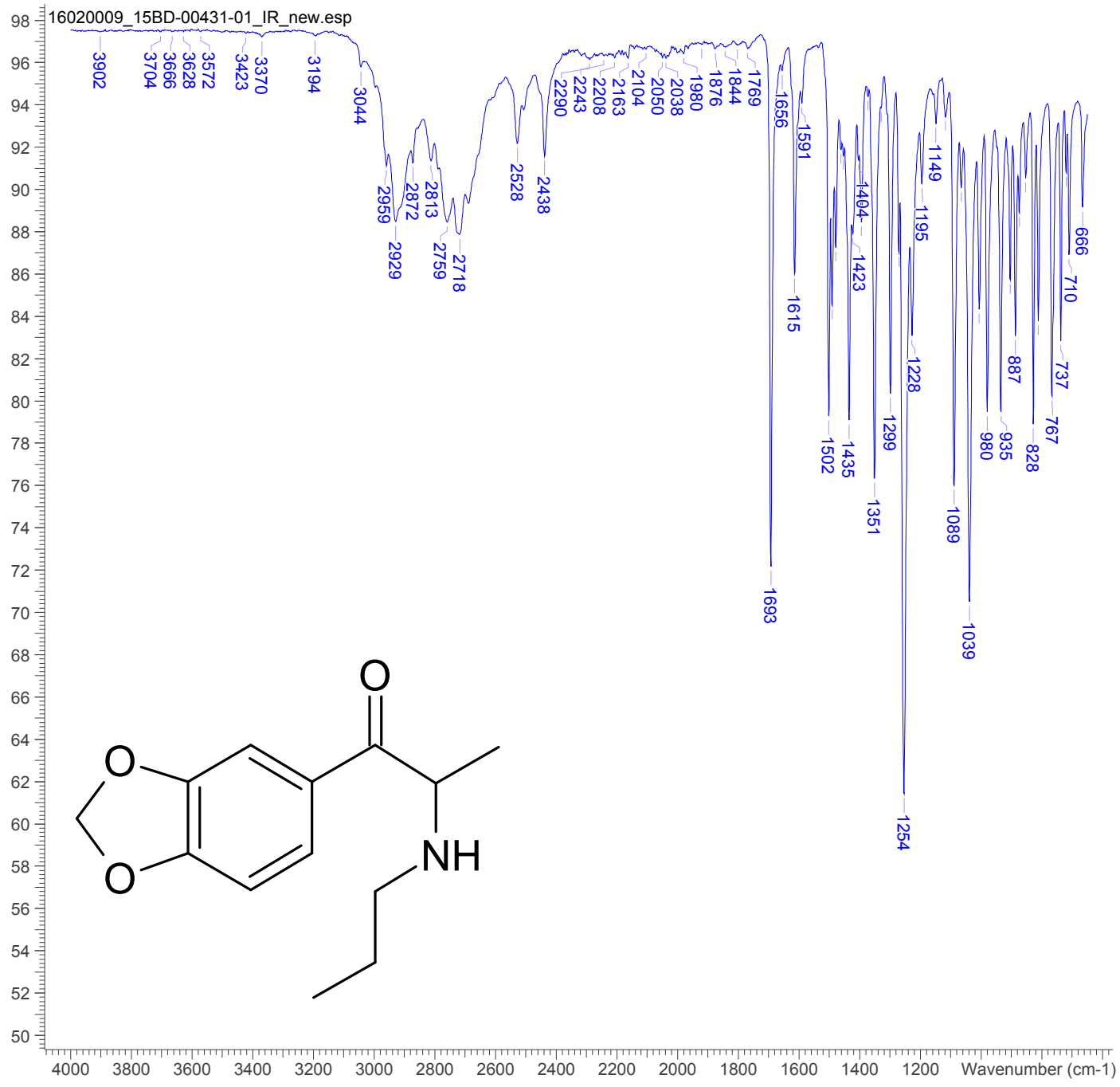
European Commission, Joint Research Centre

Institute for Health and Consumer Protection (IHCP)
via E. Fermi, 2749
TP 281, I-21027 Ispra (VA) - Italy
Phone: +39 0332 785678
Fax: +39 0332 789453
claude.guillou@ec.europa.eu
https://ec.europa.eu/jrc/en/institutes/ihcp

Processing and interpretation based on data provided by:

Belgian Customs Laboratory

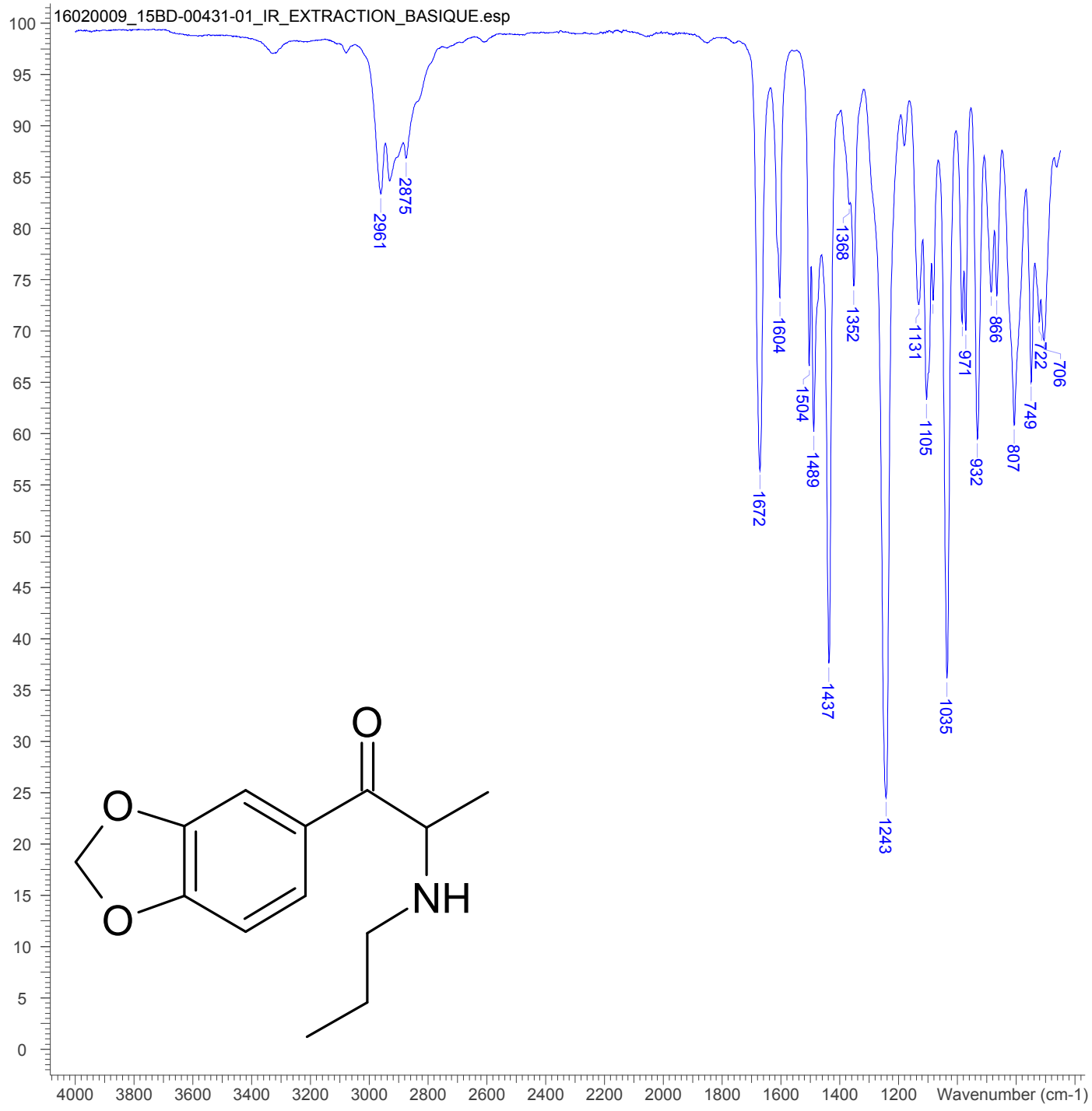
Comment	POWDER as such		
File Name	\\139.191.6.82\ihcp$\I01\BIO_CHEMICAL_INTERACTION_METABONOMICS\CLEN2SAND\!PROPOSED-STRUCTURE\CLEN_NAS\16020009_15BD-00431-01\15BD-00431-01P.SP		
Date Stamp	wed mar 02 09:30:38 2016	***Date***	wed mar 02 09:30:38 2016
Technique	Infrared	***Spectral Region***	IR
X Axis	Wavenumber (cm-1)	***Y Axis***	%Transmittance
Spectrum Range	650.0000 - 4000.0000	***Points Count***	3351
Data Spacing	1.0000		



Processing and interpretation based on data provided by:

Belgian Customs Laboratory

Comment Extracted base			
File Name	\\139.191.6.82\ihcp$\I01\BIO_CHEMICAL_INTERACTION_METABONOMICS\CLEN2SAND\!PROPOSED-STRUCTURE\CLEN_NAS\16020009_15BD-00431-01\15BD-00431-01\15BD-00431 -01 EXTRACTION BASIQUE.SP		
Date Stamp	fri nov 27 14:10:34 2015	***Date***	fri nov 27 14:10:34 2015
Technique	Infrared	***Spectral Region***	IR
X Axis	Wavenumber (cm-1)	***Y Axis***	%Transmittance
Spectrum Range	650.0000 - 4000.0000	***Points Count***	3351
Data Spacing	1.0000		



Processing and interpretation based on data provided by:

Belgian Customs Laboratory

Comment	MTOH/DCM			***Count***	154
Data Type	Condensed	***Date***	3:52:48 PM		
Date Stamp	4 Nov 15 3:22 pm			***Estimated Molecular Ion***	234.2
File Name	15BD431_1_Centroid_EI+			***Inlet Model***	GC
Ion Mode	EI+	***Mass Spec Model***	Instrumen	***Operator***	VH
Plot Type	Stick	***Retention Time***	9.336	***Sample***	15BD-00431-01
Scan	1026	***Scan Mode***	Centroid	***Spectrum Assigned***	69.2% [14-237/0-100]
Spectrum Type	MS	***TIC***	230.85	***Total Signal***	2978550

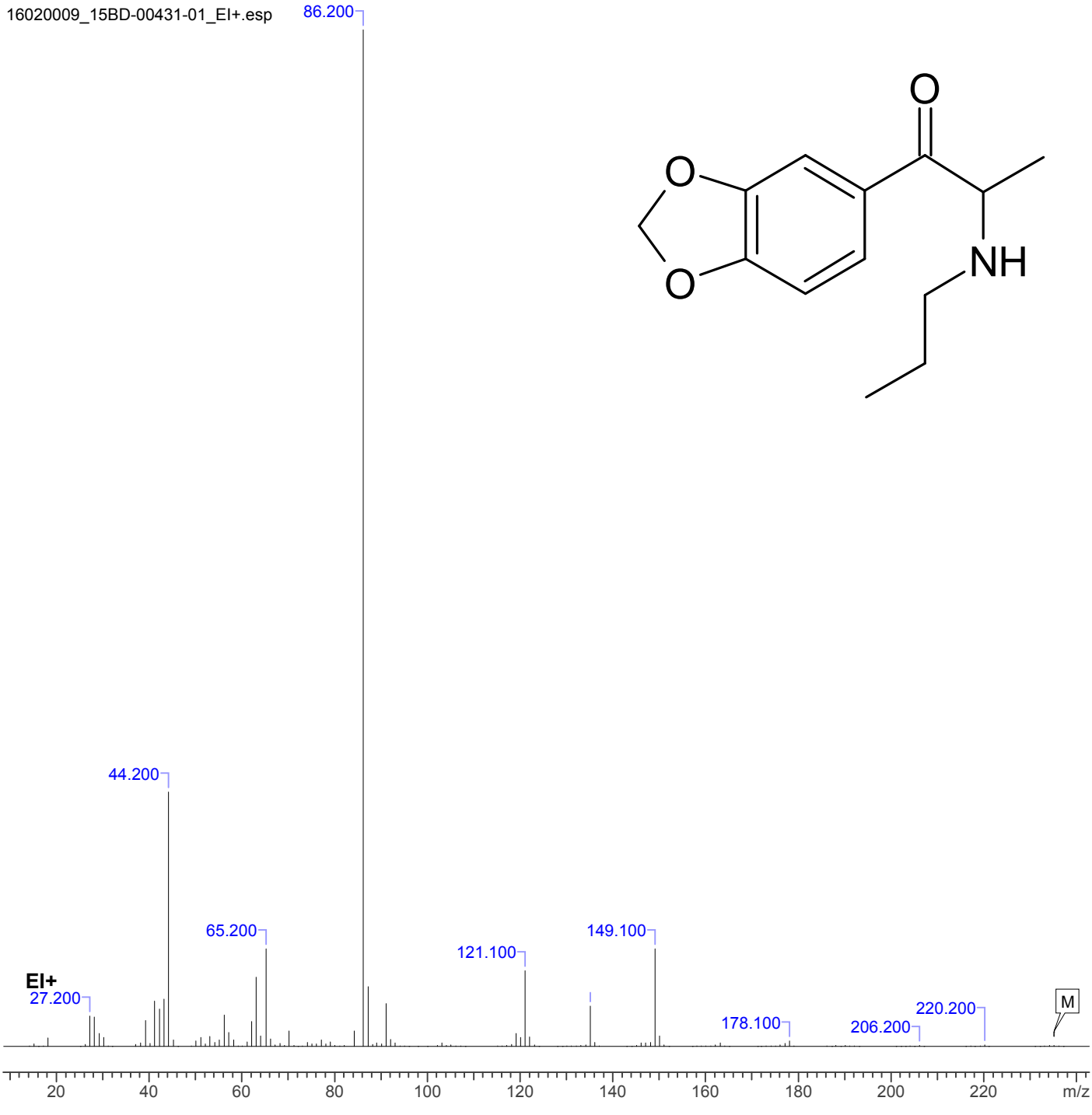
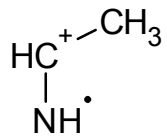
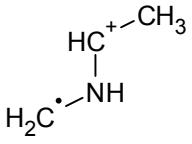
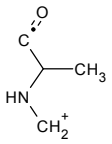
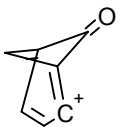
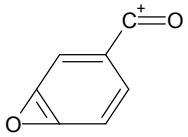
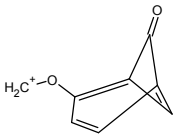
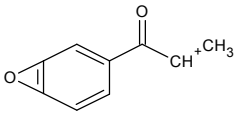
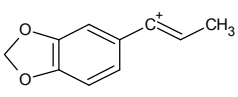
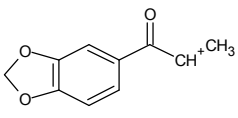
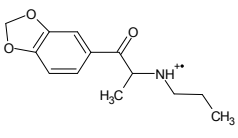


Table of fragments

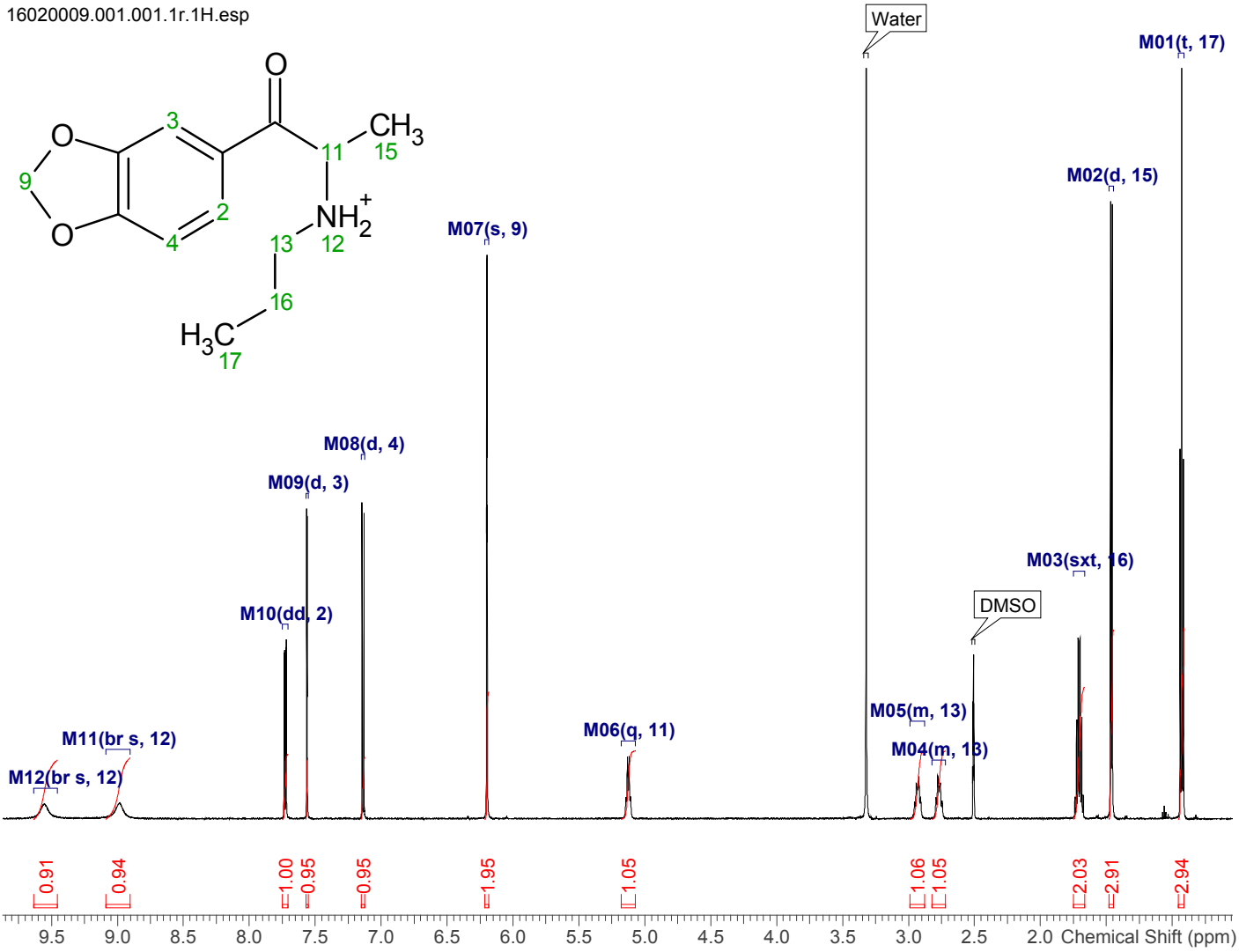
No.	Fragment	Structure	Formula	Label	*m/z* Calc.	TIC Calc (%)	Differe (Da	*m/z* Exp.	RI Exp. (%)	TIC Exp. (%)
1	11-12,17(+H)		C2H6N	M - C11H11O3	44.049	11.121	0.151	44.200	25.005	11.121
2	11-13,17(-H)		C3H6N	M - C10H11O3	56.049	1.377	0.151	56.200	3.061	1.377
3	11-15(-H)		C4H8N	M - C9H9O3	70.065	0.686	0.135	70.200	1.509	0.686
4	10-13,16-17(+H)		C4H8NO	M - C9H9O2	86.060	45.586	0.140	86.200	100.000	45.585
5	1-6,10,16		C7H3O	M - C6H14NO2	103.018	0.149	0.082	103.100	0.317	0.149
6	1-6,8,10,16(+H2)		C7H5O2	M - C6H12NO	121.028	3.493	0.072	121.100	7.424	3.493
7	1-6,8-10,16(+H2)		C8H7O2	M - C5H10NO	135.044	1.887	0.056	135.100	3.966	1.886
8	1-7,10-11,16-17(+H2)		C9H9O2	M - C4H8NO	149.060	4.607	0.040	149.100	9.574	4.607
9	1-11,17(+H2)		C10H11O2	M - C3H6NO	163.075	0.172	0.125	163.200	0.354	0.172
10	1-11,16-17(+H)		C10H10O3	M - C3H7N	178.062	0.252	0.038	178.100	0.518	0.250
11	M		C13H17NO3	M	235.120	0.038	0.080	235.200	0.075	0.038

Data from BCIM group at JRC, IHCP Ispra

NMR spectra

Acquisition Time (sec)	2.7263	***Comment***	15BD-00431-01 Belgium Custom		
D	0.0002	***D1***	1	***DE***	10
DS	2	***Date***	23 Feb 2016 18:09:33		
Date Stamp	23 Feb 2016 18:09:33				
File Name	\\139.191.6.82\ihcp$\l01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_600\16020009_15BD-00431-01\1\PDATA\1\1r				
Frequency (MHz)	600.1300	***GB***	0	***INSTRUM***	<spect>
LB	0	***NS***	32	***Nucleus***	1H
Number of Transients	32	***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***	300.0009
Temperature (degree C)	27.001	***UNC1***	<1H>	***WDW***	0

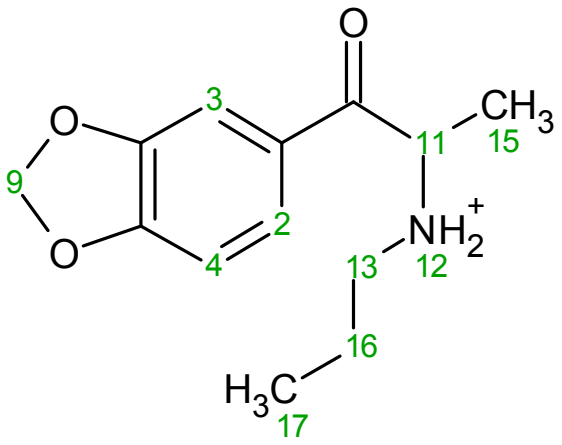
16020009.001.001.1r.1H.esp

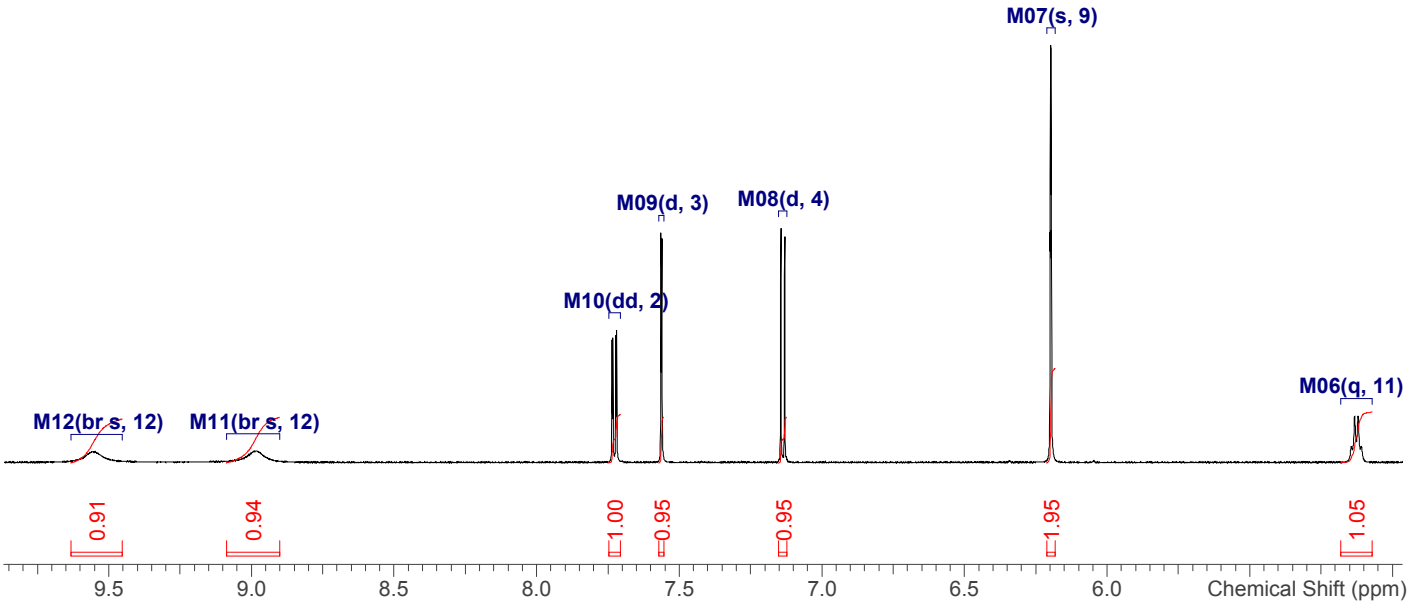


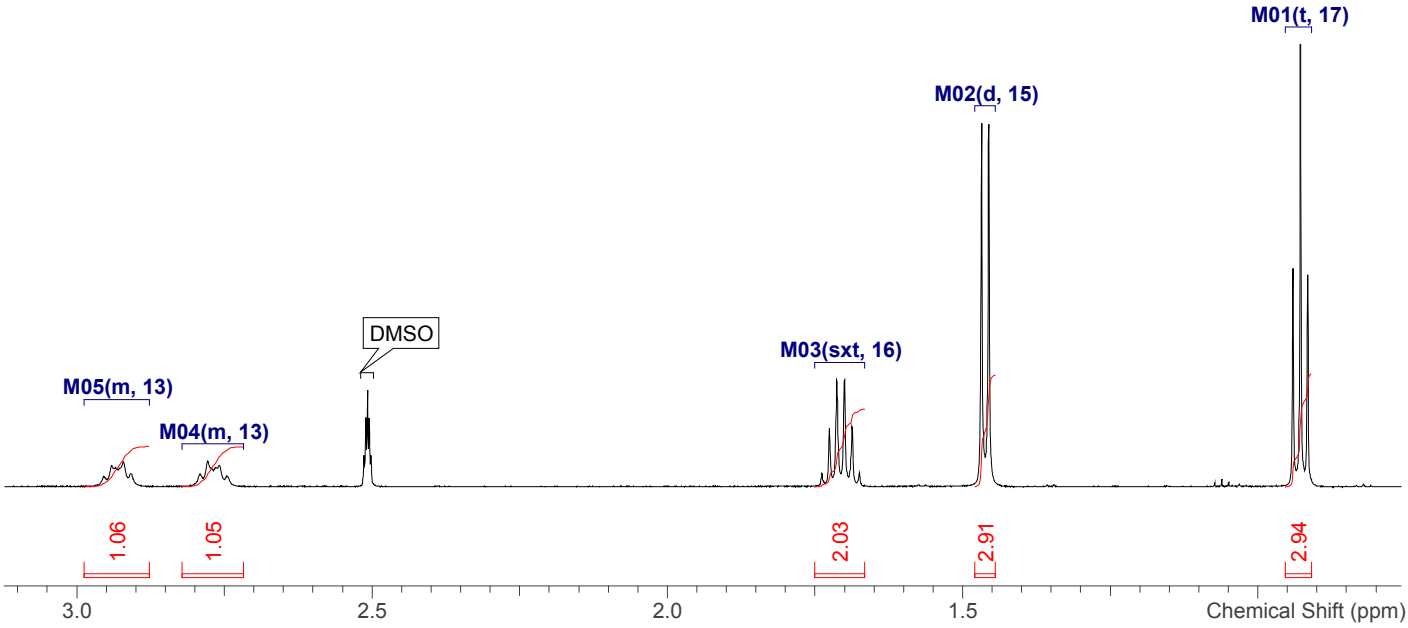
^{1}H NMR (600 MHz, DMSO-d_6) d ppm 0.93 (t, *J*=7.43 Hz, 3 H) 1.46 (d, *J*=7.15 Hz, 3 H) 1.71 (sxt, *J*=7.63 Hz, 2 H) 2.72 - 2.82 (m, 1 H) 2.88 - 2.99 (m, 1 H) 5.13 (q, *J*=7.03 Hz, 1 H) 6.20 (s, 2 H) 7.14 (d, *J*=8.07 Hz, 1 H) 7.56 (d, *J*=1.83 Hz, 1 H) 7.73 (dd, *J*=8.25, 1.83 Hz, 1 H) 8.98 (br s, 1 H) 9.55 (br s, 1 H)

Table of assignments and zoomed parts:

No.	Atom	Exp. Shift (ppm)	Multiplet
1	17	0.93	M01
2	15	1.46	M02
3	16	1.71	M03
4	13	2.77	M04
5	13	2.93	M05
6	11	5.13	M06
7	9	6.20	M07
8	4	7.14	M08
9	3	7.56	M09
10	2	7.73	M10
11	12	8.98	M11
12	12	9.55	M12

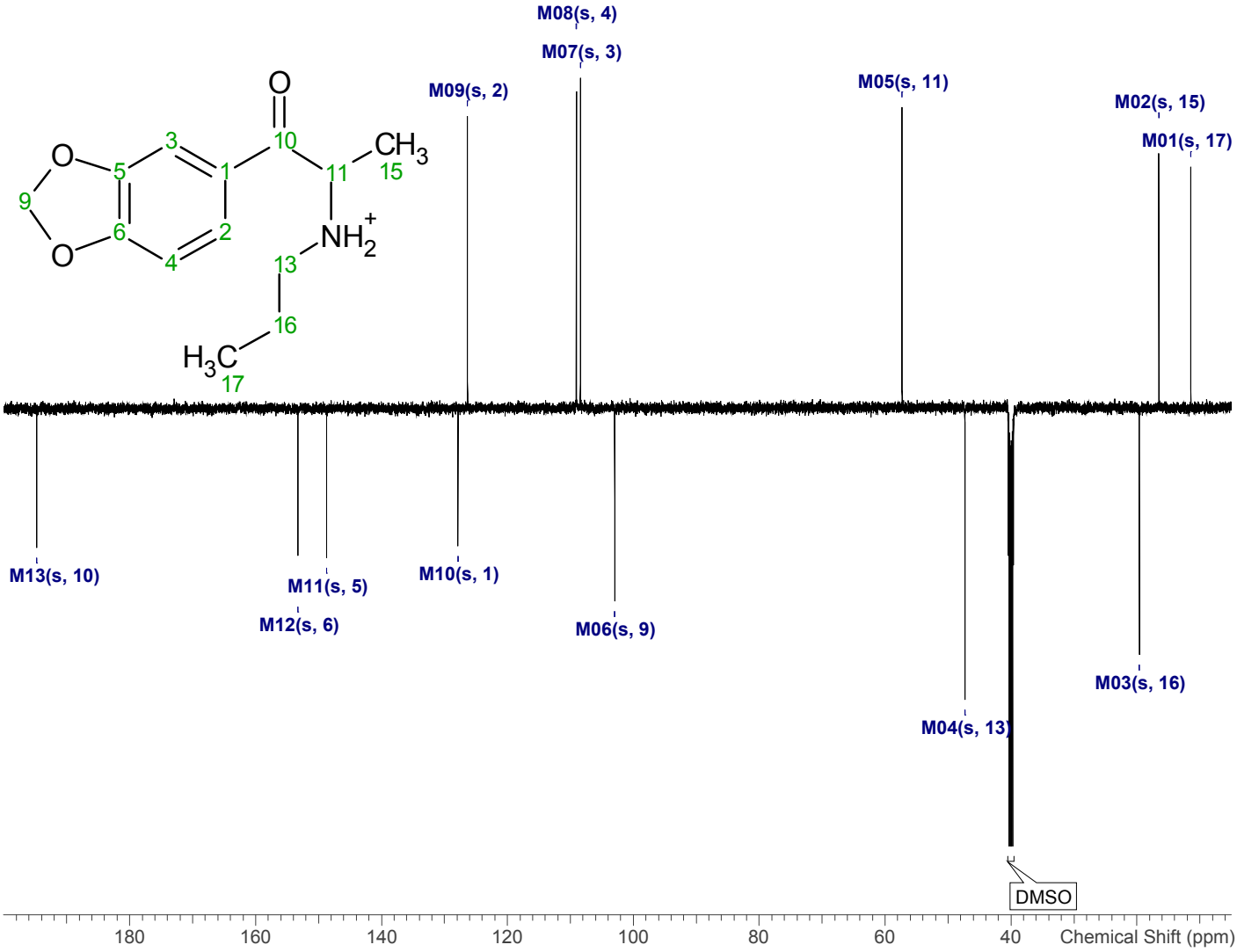






Acquisition Time (sec)	0.9044	***Comment***	5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012		
D	0.0002	***D1***	2	***DE***	18
DS	4	***Date***	23 Feb 2016 18:14:44		
Date Stamp	23 Feb 2016 18:14:44				
File Name	\\139.191.6.82\ihcp$\I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\!proposed-structure\BCIM_NAS\BCIM_600\16020009_15BD-00431-01\2\PDATA\1\1r				
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***	300.0019
Temperature (degree C)	27.002	***UNC1***	<13C>	***WDW***	1

16020009.002.001.1r.13C.esp

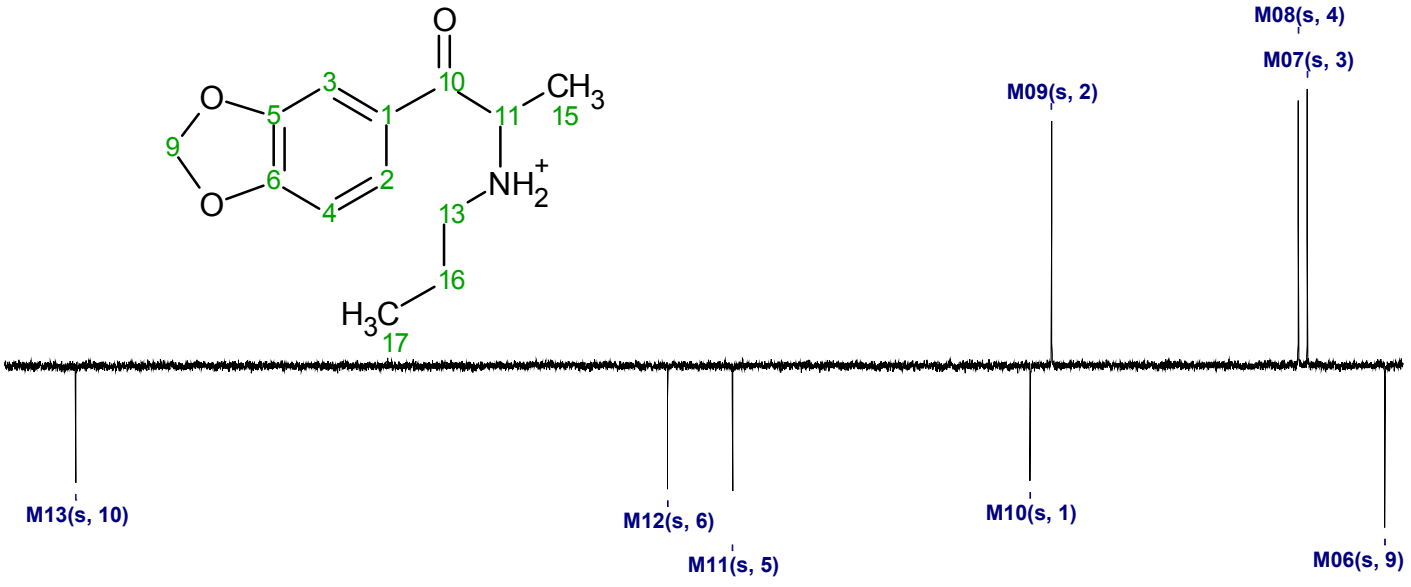


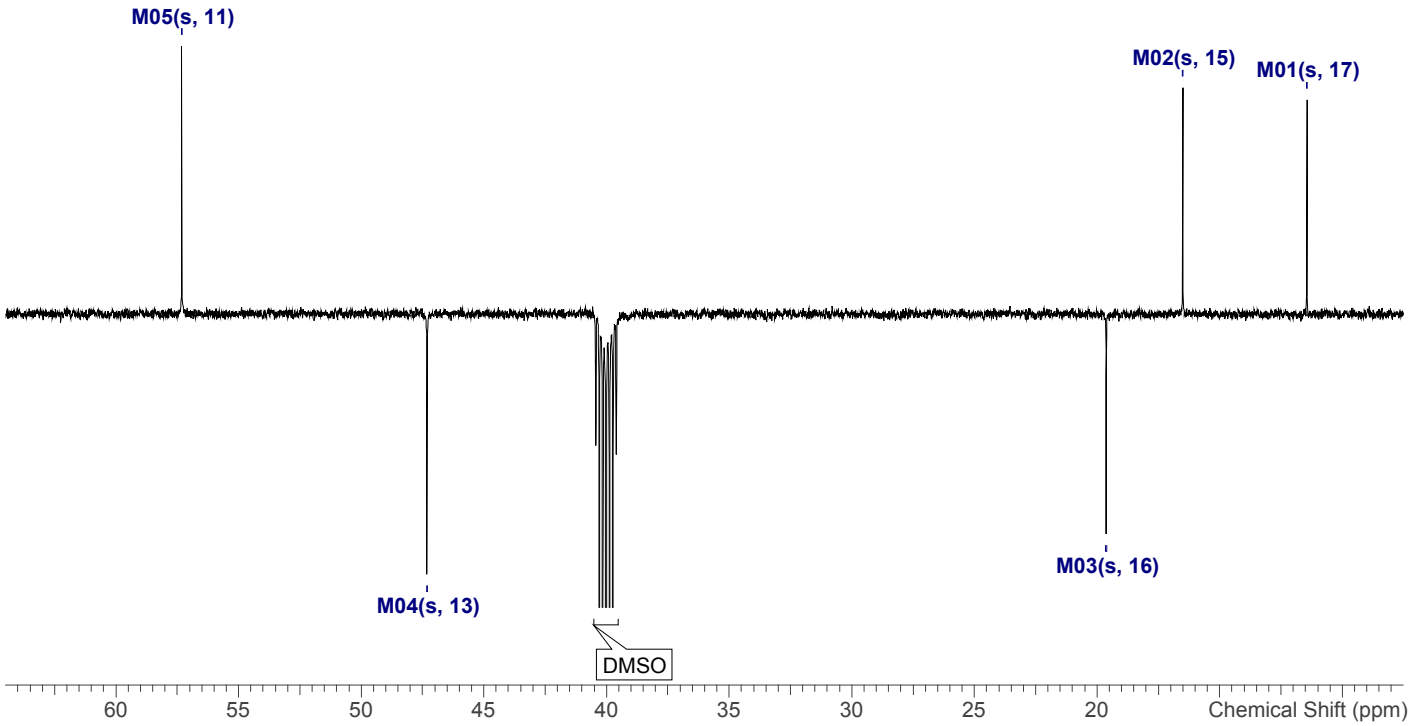
^{13}C NMR (151 MHz, DMSO-d_6) d ppm 11.44 (s, 1 C) 16.51 (s, 1 C) 19.63 (s, 1 C) 47.32 (s, 1 C) 57.32 (s, 1 C) 102.97 (s, 1 C) 108.40 (s, 1 C) 109.04 (s, 1 C) 126.34 (s, 1 C) 127.85 (s, 1 C) 148.72 (s, 1 C) 153.27 (s, 1 C) 194.78 (s, 1 C)

Table of assignments and zoomed parts:

No.	Atom	Exp. Shift (ppm)	Multiplet
1	17	11.44	M01
2	15	16.51	M02
3	16	19.63	M03
4	13	47.32	M04
5	11	57.32	M05
6	9	102.97	M06
7	3	108.40	M07

No.	Atom	Exp. Shift (ppm)	Multiplet
8	4	109.04	M08
9	2	126.34	M09
10	1	127.85	M10
11	5	148.72	M11
12	6	153.27	M12
13	10	194.78	M13





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