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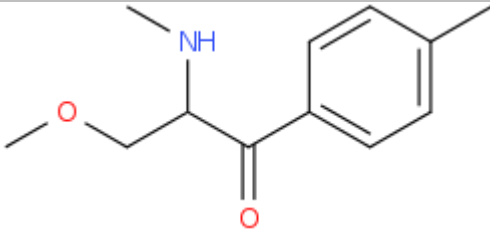
ANALYTICAL REPORT¹

Mexedrone (C₁₂H₁₇N₂O₂)

3-methoxy-2-(methylamino)-1-(4-methylphenyl)propan-1-one

Remark – other NPS detected: **none**

Sample ID:	1519-16	lab note: 233-5622/15 spl.27)
Sample description:	crystallinic - white	
Sample type:	collected /NGO-collected	
Date of sample receipt (M/D/Y):	12/22/2015	
Date of entry (M/D/Y) into NFL database:	2/10/2016	
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php	

Substance identified - structure ² (base form)	
Systematic name	3-methoxy-2-(methylamino)-1-(4-methylphenyl)propan-1-one
Other names	
Formula (per base form)	C ₁₂ H ₁₇ N ₂ O ₂
M _w (g/mol)	207,27
Salt form/anions detected	HCl
StdInChIKey	JHGDCSPZKQLBOP-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	none
Add.info (purity..)	traces of organic impurities by GC-MS

¹ Sample was collected and GC-MS screened in the frame of EU co-funded project I-SEE (JUST/2013/ISEC/DRUGS/AG/6426) and the compound structure was further verified in the frame of the RESPONSE project (JUST/2013/ISEC/DRUGS/AG/6413). Both projects run with the financial support of the Prevention of and fight against crime Programme of the European Union. The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

² Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

Report updates

date	comments (explanation)

Instrumental methods (if applied) in NFL

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (RT=9.53 min). Injection volume 1 ml and split mode (1:50) . Injector temperature: 280 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.

2. HPLC-TOF (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; Injection volume 1 µl. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N2) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V.

3.FTIR-ATR (Perkin Elmer): scan range 4000-400 cm⁻¹; resolution 4cm⁻¹

4. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 °C. Chromatographic separation as above (1). Split MS : IR = 1:9.

MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.

IR (condensed phase): IR scan range 4000 to 650, resolution 4 cm⁻¹.

5. IC (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30°C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

Supporting information

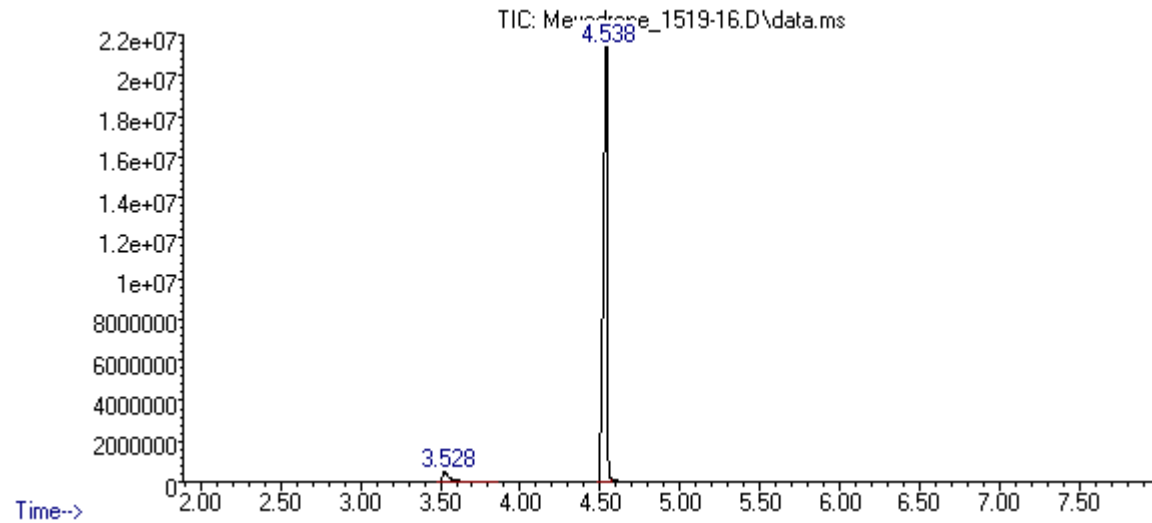
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,54 BP(1): 88; BP(2): 56,BP(3) :91,
HPLC-TOF	+	Exact mass (theoretical): 207,1259; measured value Δppm:0,17; formula:C12H17NO2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

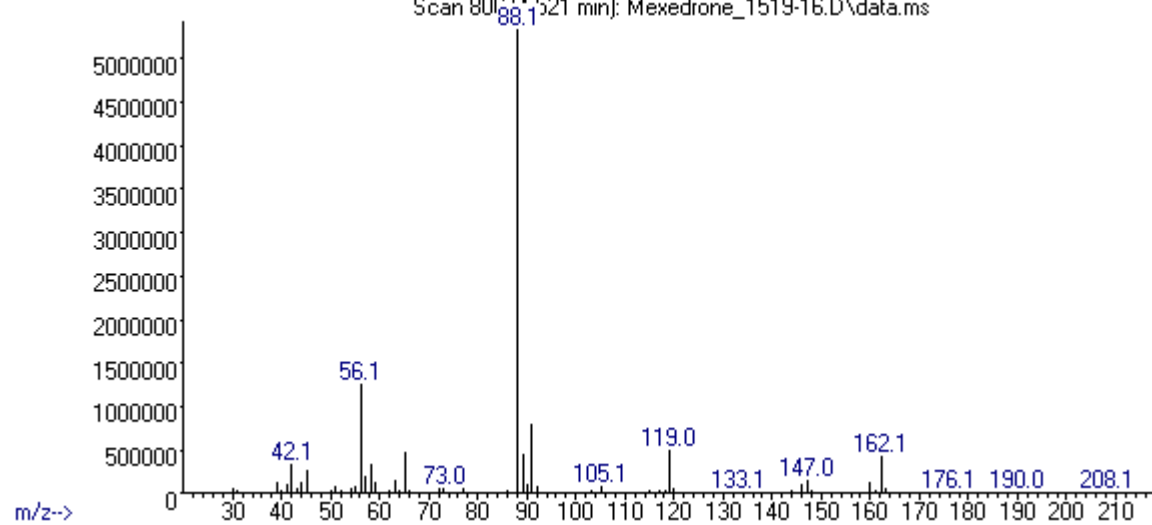
GC-MS (EI)

Abundance



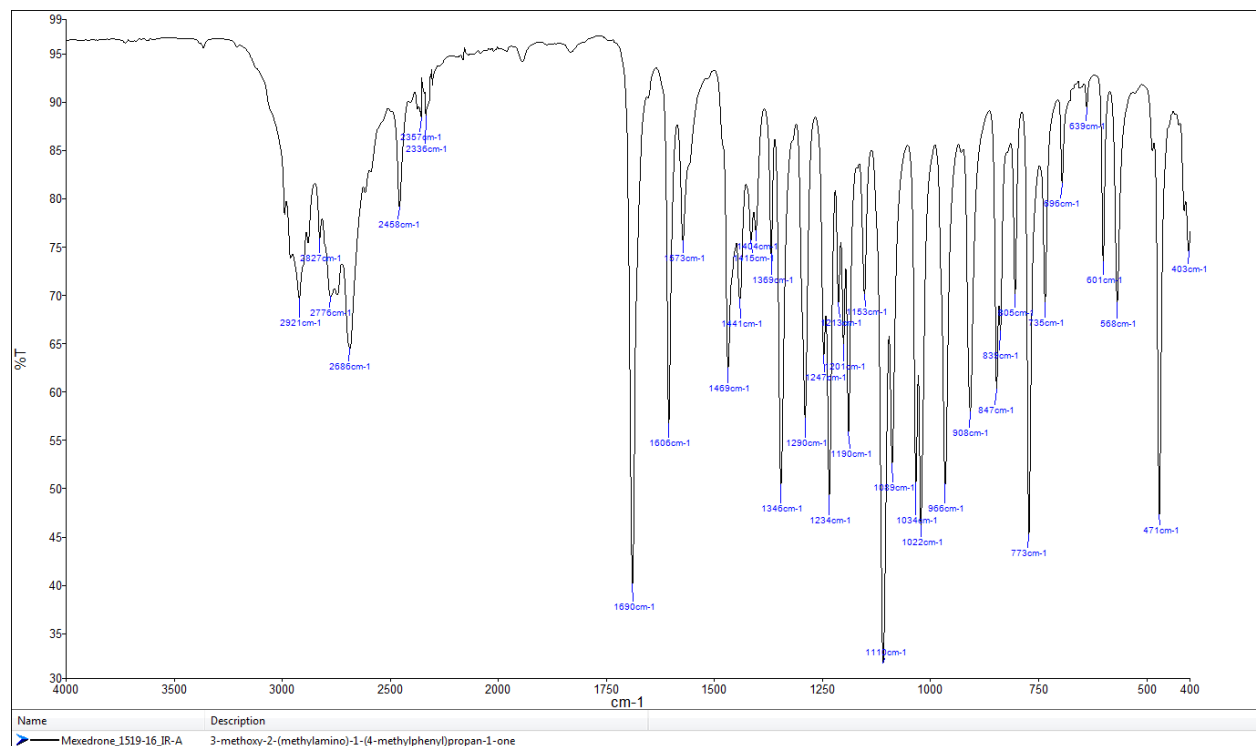
Remark. Unidentified compound at 3.53 min. Mexedrone at RT=4.54 min.

Abundance

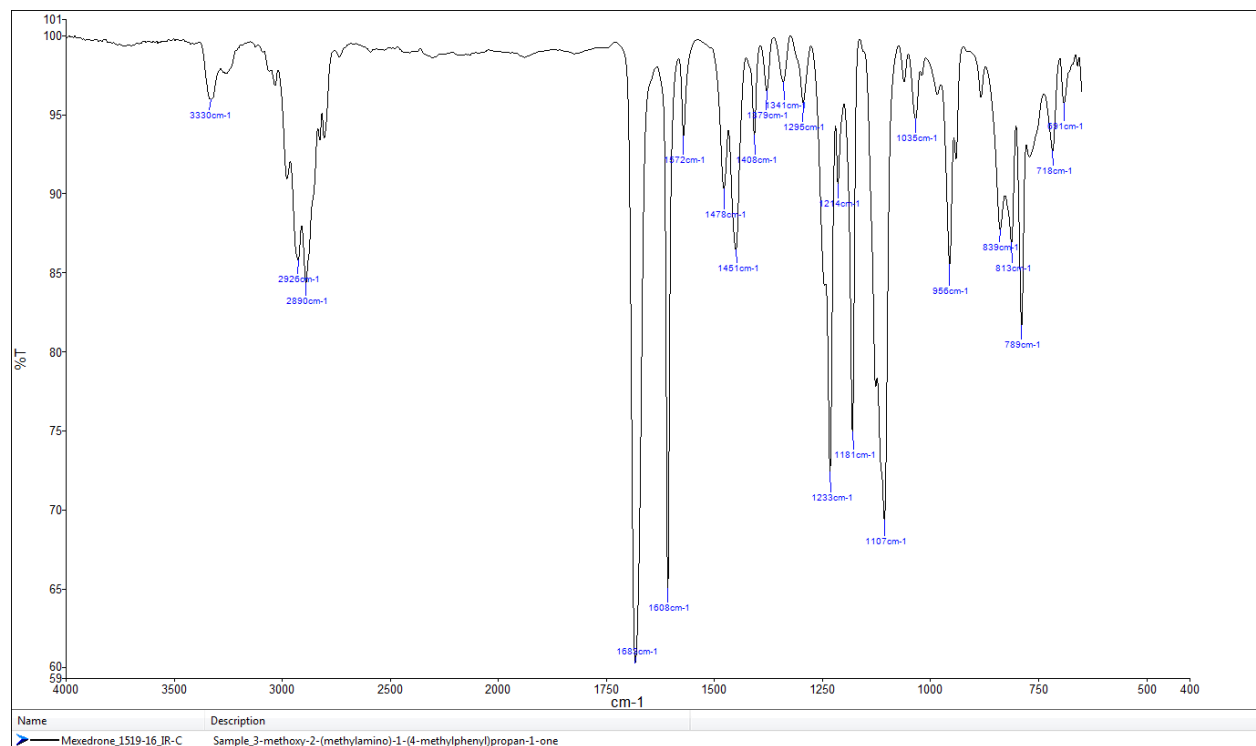


Mexedrone MS spectrum.

FTIR-ATR - direct measurement (sample as received)



IR condensed phase – after chromatographic separation



TOF REPORT

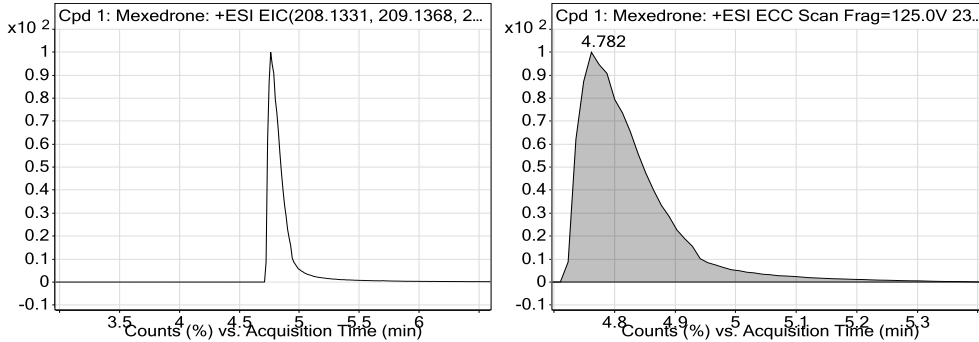
Data File	233-5622-15_vzorec 27.d	Sample Name	vzorec 27
Sample Type	Sample	Position	P1-E3
Instrument Name	6230B TOF LC-MS	User Name	
Acq Method	general-1512015-XDB-C18-ESI-poz.m	Acquired Time	1/12/2016 12:05:46 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	ekstrakcija v MeOH		

Compound Table

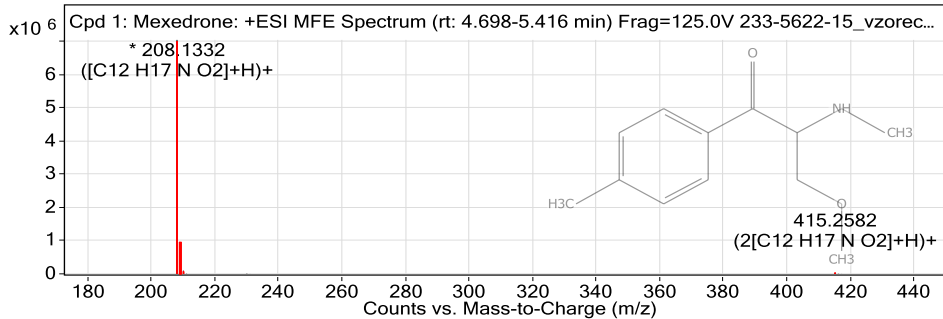
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Mexedrone	Mexedrone	C12 H17 N O2	4.782	207.1259

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Mexedrone	208.1332	4.782	207.1259	4.78	C12 H17 N O2	207.1259	0.17

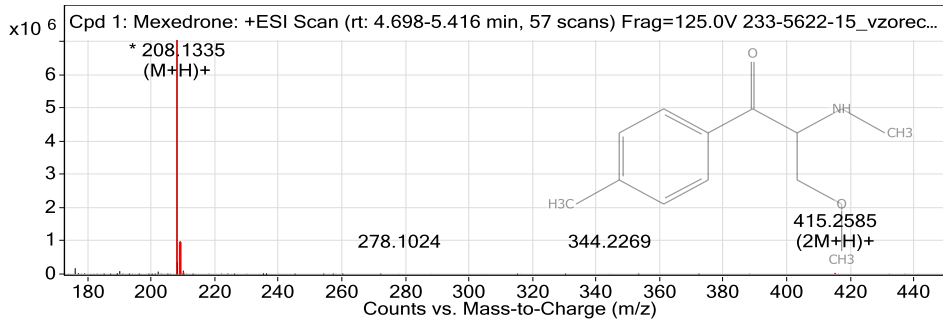
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



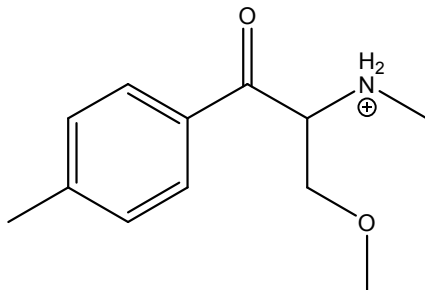
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
208.1332	1	7045393.5	C12 H17 N O2	(M+H)+
209.1361	1	909131.7	C12 H17 N O2	(M+H)+
210.1388	1	97870.55	C12 H17 N O2	(M+H)+
211.1412	1	6826.66	C12 H17 N O2	(M+H)+
212.1434	1	438.13	C12 H17 N O2	(M+H)+
230.1142	1	10032.01	C12 H17 N O2	(M+Na)+
231.1179	1	1519.15	C12 H17 N O2	(M+Na)+
415.2582	1	24732.99	C12 H17 N O2	(2M+H)+
416.2614	1	6535.51	C12 H17 N O2	(2M+H)+
417.2692	1	1868.08	C12 H17 N O2	(2M+H)+

--- End Of Report ---



REPORT

Sample ID:	233-5622-15-27
Our notebook code:	P-233-5622-15-27
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	3-methoxy-N-methyl-1-oxo-1-(p-tolyl)propan-2-aminium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure by NMR.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 26, 2016

P-233-5622-15-27

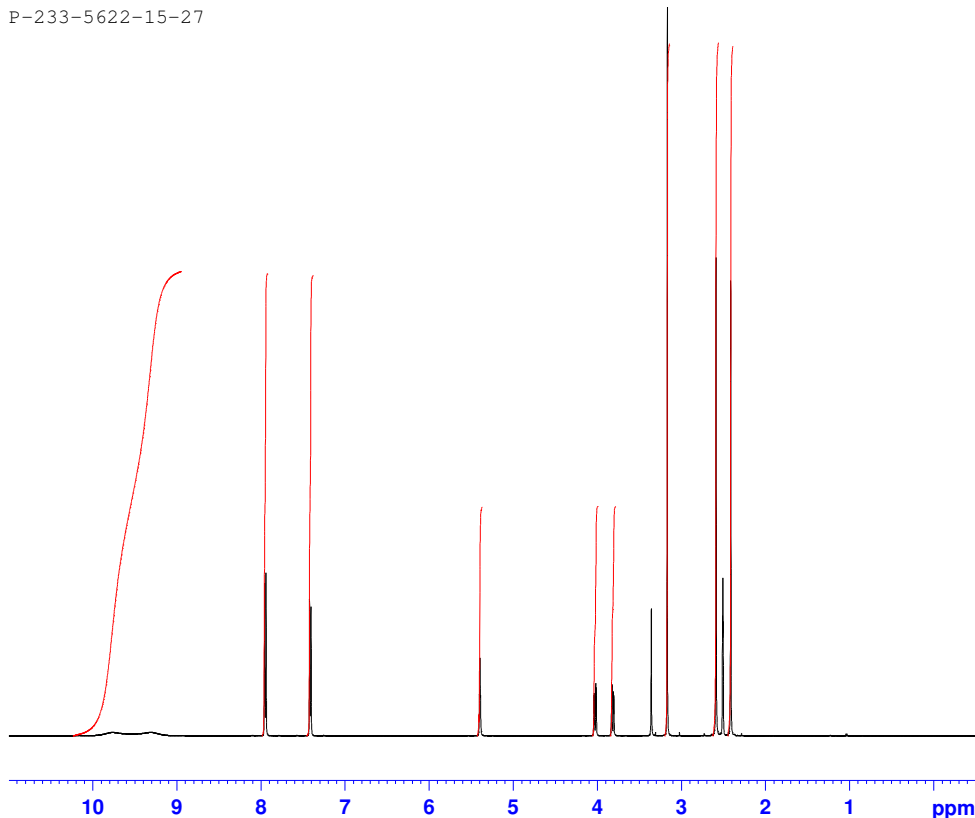


Current Data Parameters
 NAME P-233-5622-15-27
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160123
 Time 22.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 71.8
 DW 50.000 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 8.90 usec
 PLW1 26.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



P-233-5622-15-27



Current Data Parameters
 NAME P-233-5622-15-27
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160124
 Time 1.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 5120
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 298.1 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 9.00 usec
 PLW1 122.00000000 W

===== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 80.00 usec
 PLW2 26.00000000 W
 PLW12 0.32179001 W
 PLW13 0.16186000 W

F2 - Processing parameters
 SI 32768
 SF 125.7577885 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

