



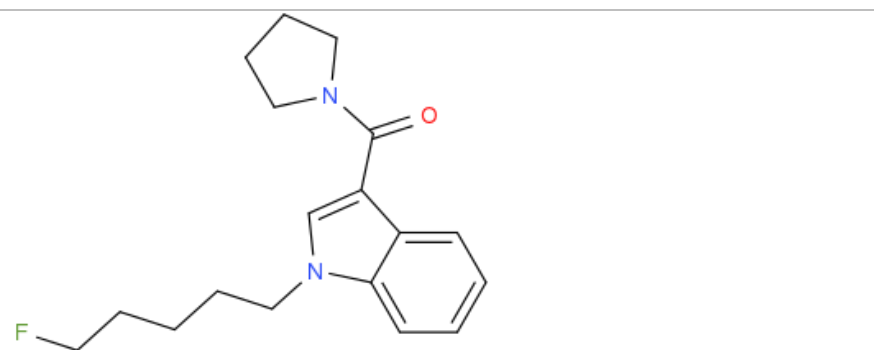
## ANALYTICAL REPORT<sup>1</sup>

### 5F-PY-PICA (C<sub>18</sub>H<sub>23</sub>FN<sub>2</sub>O)

#### 1-(5-fluoropentyl)-3-(pyrrolidine-1-carbonyl)-1H-indole

Remark – other NPS detected: **none**

Sample ID:	1281-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	9/18/2015
Date of entry (M/D/Y) into NFL database:	10/4/2015
Report updates (if any) will be published here:	<a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a>

Substance identified - structure <sup>2</sup> (base form)	
Systematic name	1-(5-fluoropentyl)-3-(pyrrolidine-1-carbonyl)-1H-indole
Other names	(1-(5-fluoropentyl)-1H-indol-3-yl)(pyrrolidin-1-yl)methanone
Formula (per base form)	C <sub>18</sub> H <sub>23</sub> FN <sub>2</sub> O
M <sub>w</sub> (g/mol)	302,39
Salt form	base
StdInChIKey	AJOAHRJLOXOZKX-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	

<sup>1</sup> This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). 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.

<sup>2</sup> 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 2.8 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**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<sup>-1</sup>.

**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

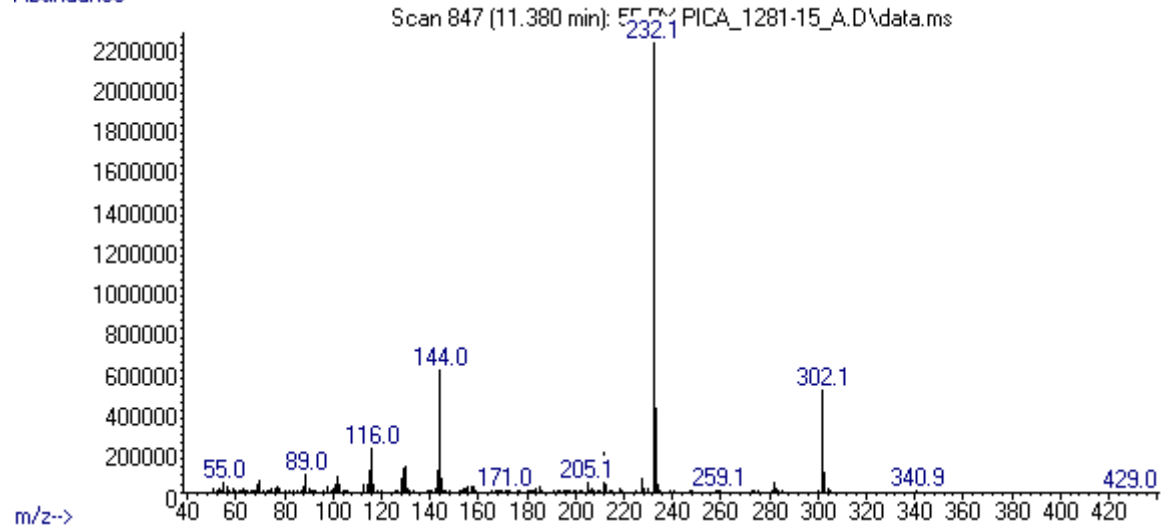
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	soluble
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 11,45 BP(1): 232; BP(2): 144,BP(3) :302,
HPLC-TOF	+	Exact mass (theoretical): 302,1794; measured value Δppm:-0,46; formula:C18H23FN2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

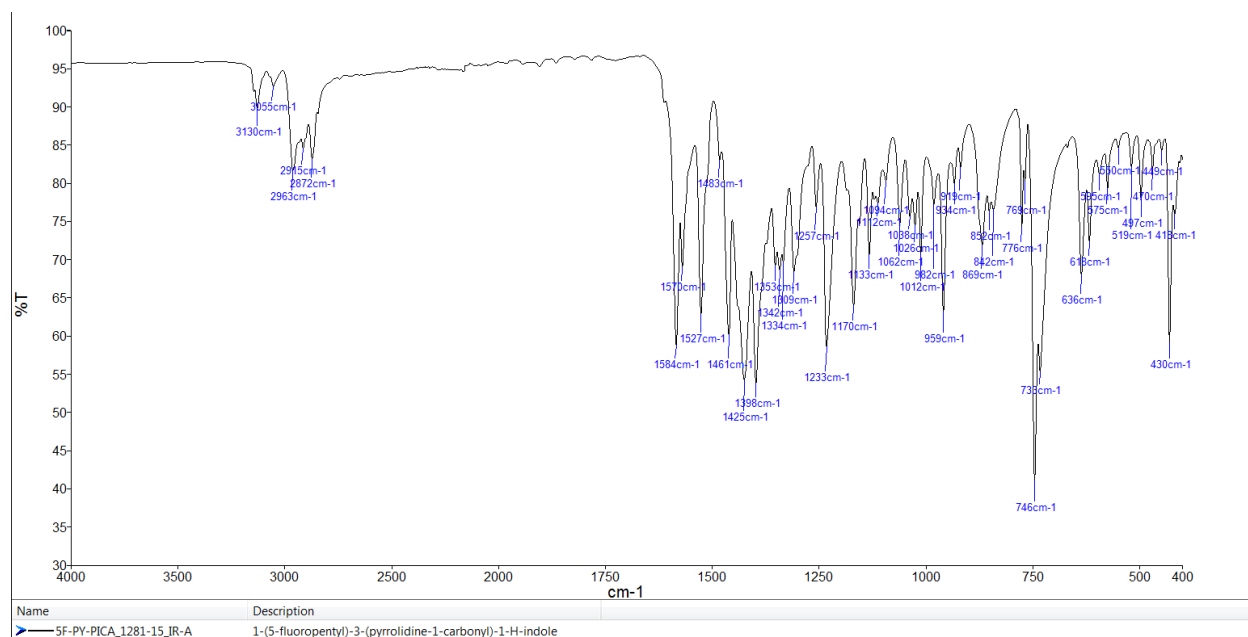
# ANALYTICAL RESULTS

MS (EI)

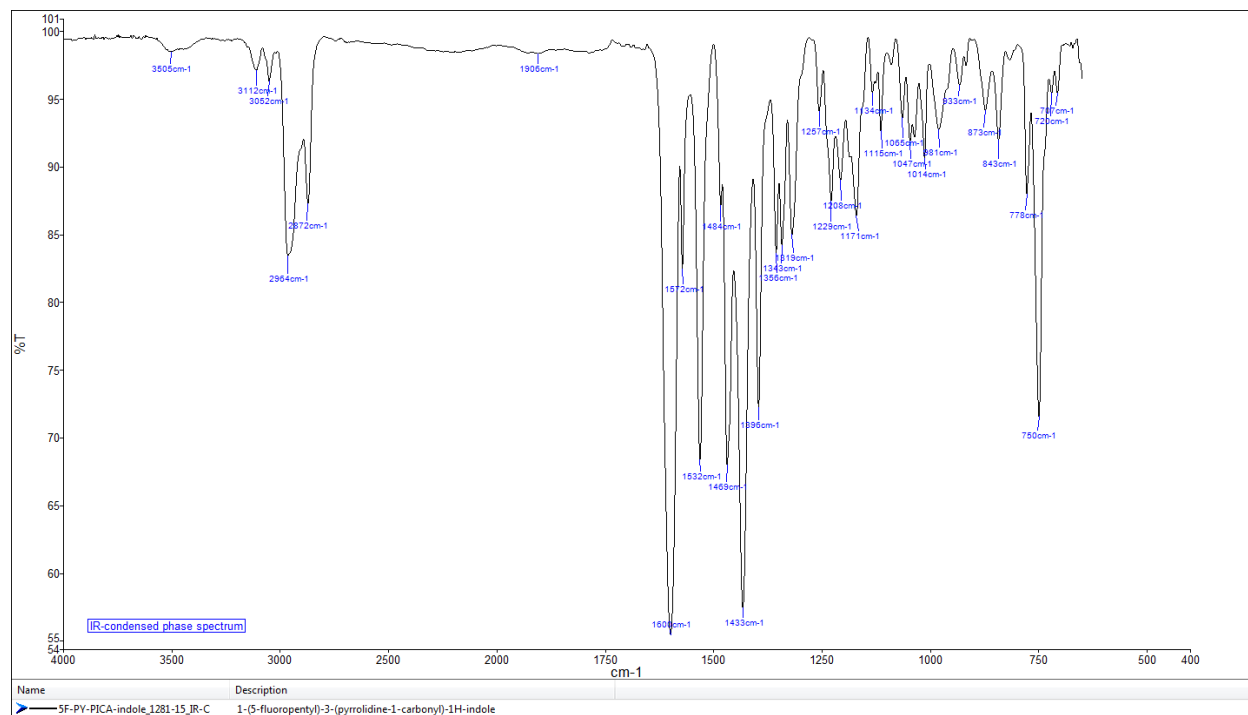
Abundance



## FTIR-ATR - direct measurement (sample as received)



## IR (condensed phase – after chromatographic separation)



# TOF REPORT

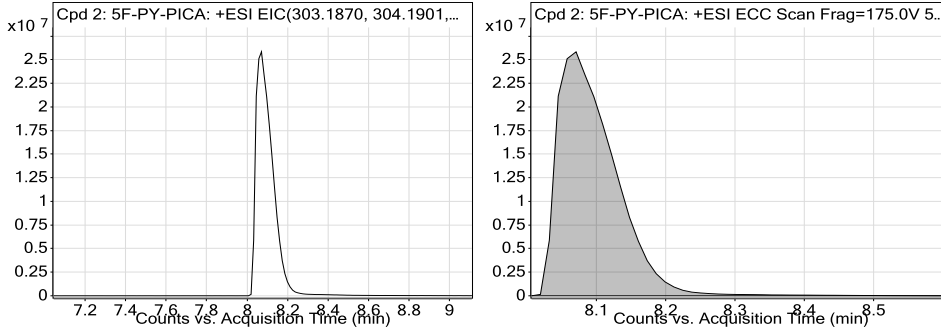
<b>Data File</b>	5F-PY-PICA_1281-15_TOF.d	<b>Sample Name</b>	5F-PY-PICA
<b>Sample Type</b>	Sample	<b>Position</b>	P1-E6
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	droge general-13-5-2015-XDB-C18-ESI-poz.m	<b>Acquired Time</b>	9/21/2015 10:27:05 AM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	Drugs_NFL.m
<b>Comment</b>	extract in MeOH		

## Compound Table

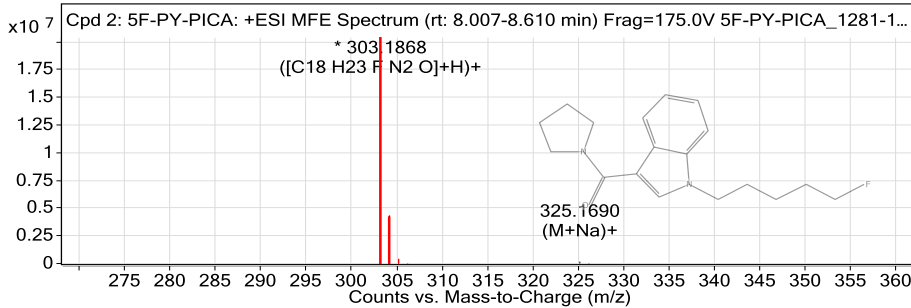
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 2: 5F-PY-PICA	5F-PY-PICA	8.077	302.1796

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
5F-PY-PICA	303.1868	8.077	302.1796	8.1	C18 H23 F N2 O	302.1794	-0.46

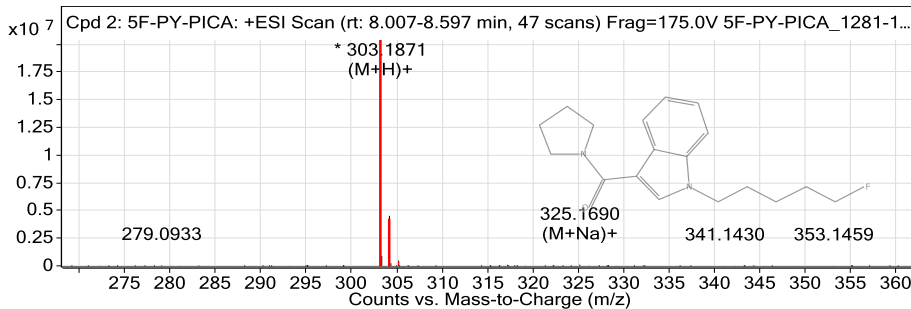
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

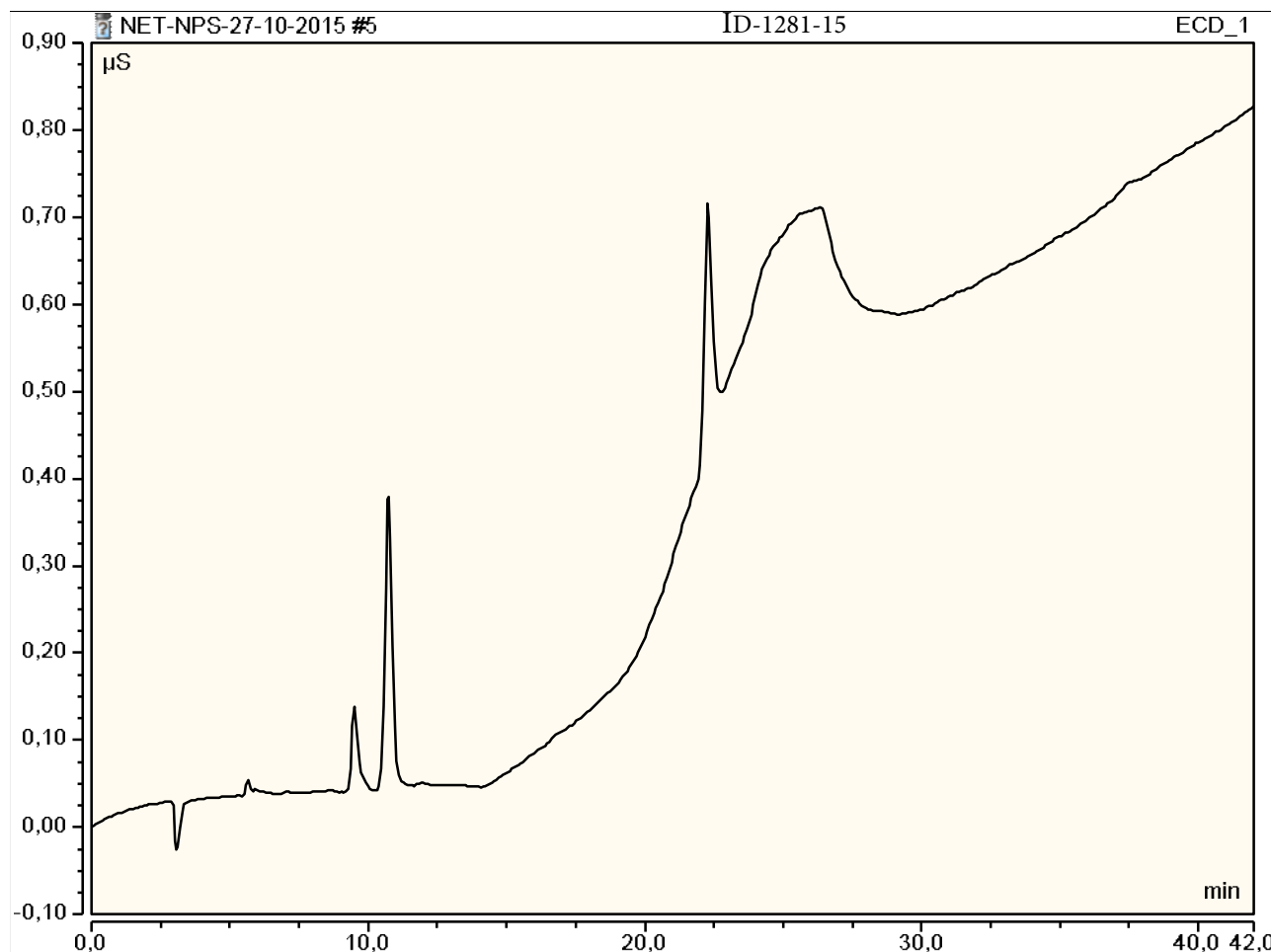
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
303.1868	1	20376238	C18 H23 F N2 O	(M+H)+
304.1902	1	4333842.97	C18 H23 F N2 O	(M+H)+
305.1937	1	407615.89	C18 H23 F N2 O	(M+H)+
306.1961	1	29886.1	C18 H23 F N2 O	(M+H)+
307.1938	1	2732.13	C18 H23 F N2 O	(M+H)+
325.169	1	162829.19		(M+Na)+
326.1721	1	31491.15		(M+Na)+
327.1811	1	2379.34		(M+Na)+

--- End Of Report ---

### Peak Integration Report

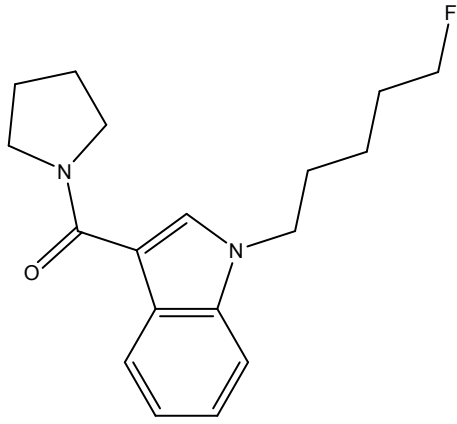
Sample Name:	ID_1281-15_IC Unknown	Inj. Vol.:	25,00
Injection Type:	ANIONI	Dilution Factor:	1,0000
Program:	27-okt-2015	Operator:	kemija
Inj. Date / Time:	/ 14:44	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height $\mu\text{S}$	Amount n.a.
TOTAL:				0,00	0,00	0,00





## REPORT

Sample ID:	<b>1281-15</b>
Our notebook code:	P-1281-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- $d_6$
NMR experiments:	$^1\text{H}$ , $^{13}\text{C}$ .
Proposed structure:	
Chemical name:	(1-(5-fluoropentyl)-1H-indol-3-yl)(pyrrolidin-1-yl)methanone
Comments:	- Structure elucidation based on 1D NMR spectra - Compound is pure by NMR.
Supporting information:	Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	November 30, 2015



P-1281-15



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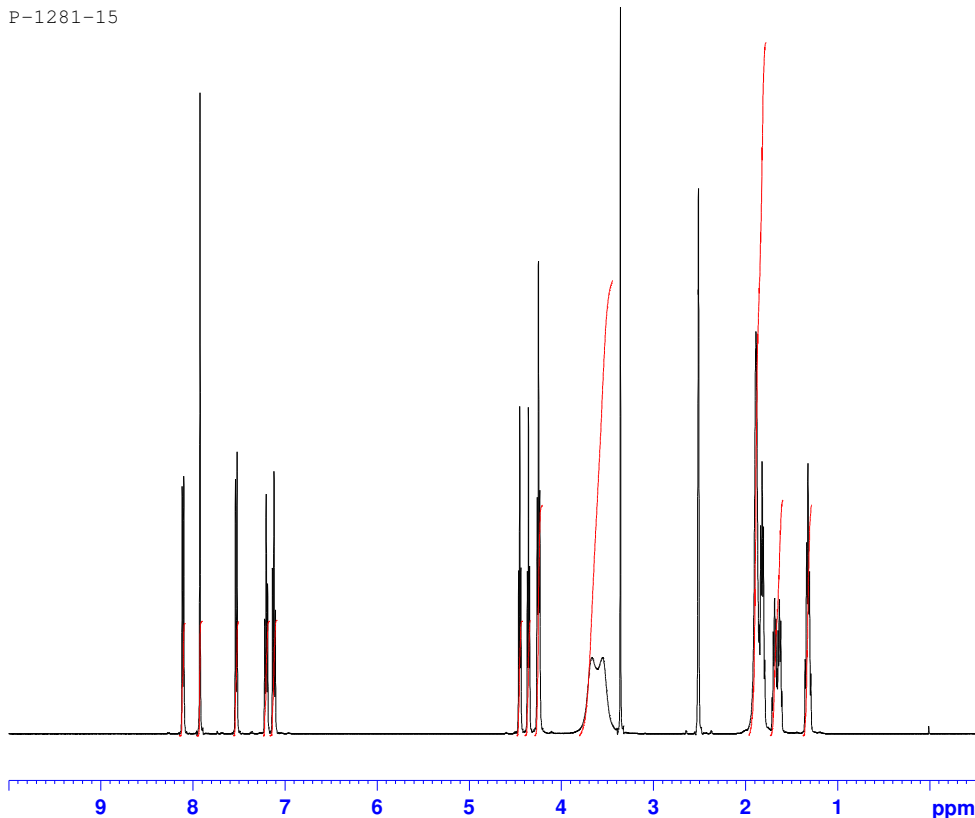
Current Data Parameters
NAME          P-1281-15
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20151119
Time          16.34
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            50.8
DW            48.400 usec
DE            6.50 usec
TE            296.0 K
D1            1.00000000 sec

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

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

```



P-1281-15



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Current Data Parameters
NAME          P-1281-15
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20151119
Time          16.44
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            1024
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            2050
DW            16.800 usec
DE            6.50 usec
TE            296.0 K
D1            2.00000000 sec
D11           0.03000000 sec

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

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PLW2          26.00000000 W
PLW12         0.32179001 W
PLW13         0.20595001 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI            32768
SF            125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

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