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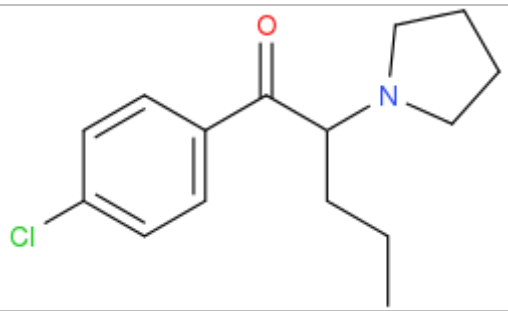
ANALYTICAL REPORT[1]

4Cl-PVP (C15H20ClNO)

1-(4-chlorophenyl)-2-(pyrrolidin-1-yl)pentan-1-one

Remark – other NPS detected: none

Sample ID:	1279-15
Sample description:	powder - beige
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:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[2] (base form)	
Systematic name	1-(4-chlorophenyl)-2-(pyrrolidin-1-yl)pentan-1-one
Other names	
Formula (per base form)	C15H20ClNO
M_w (g/mol)	265.78
Salt form	HCl
StdInChIKey	NIGBFBTVONRYQN-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	none
Add.info (purity..)	Pure by NMR

[1] 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.

[2] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Report updates

date	comments (explanation)
08/12/2016	compound class changed

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 ^{0}C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 293 ^{0}C at a rate of 18 ^{0}C/min, hold for 6.1 min, than heating at 50 ^{0}C/min up to 325 ^{0}C and finally 2.8 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}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 ^{0}C. Chromatographic separation as above **(1).** Split MS : IR = 1:9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.
IR (condesed 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

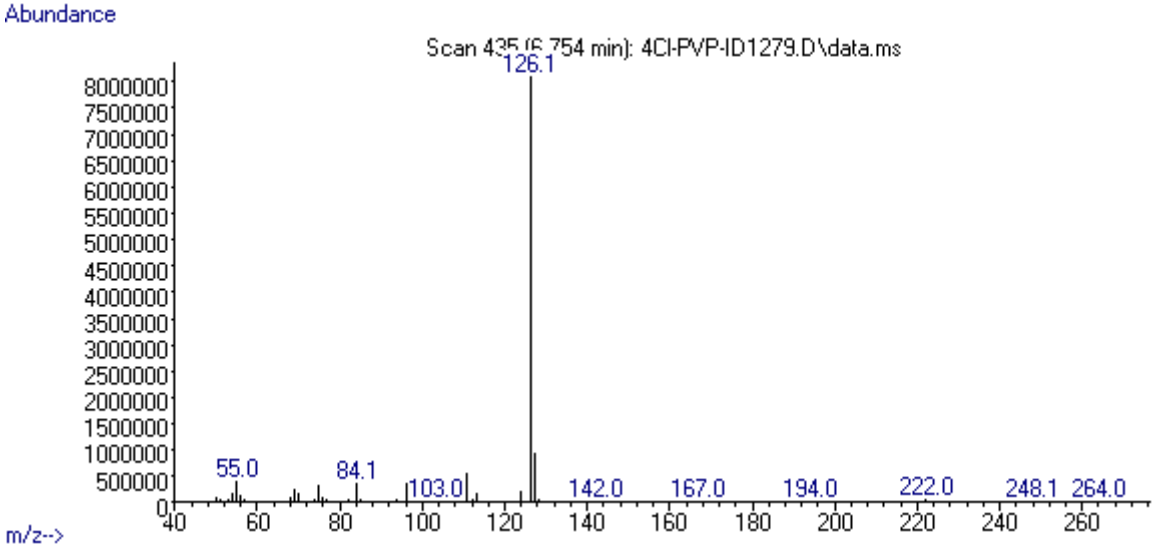
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	

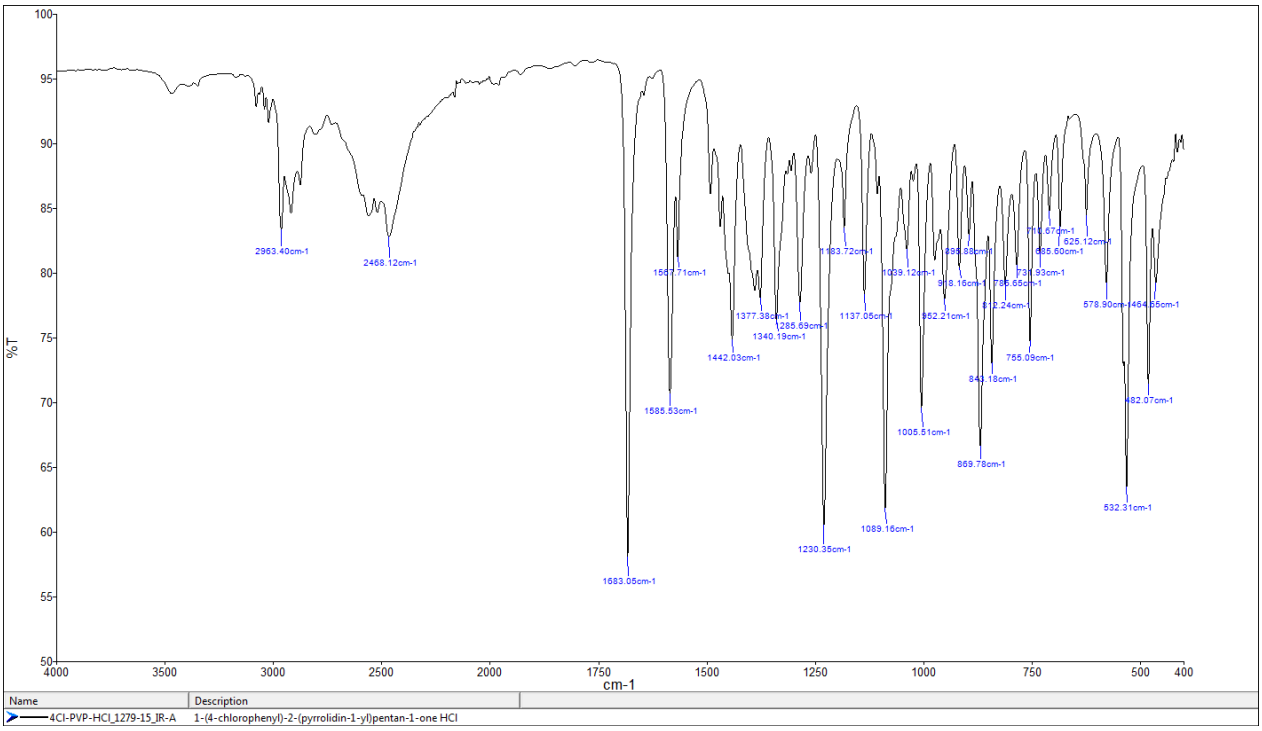
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 6.75 BP(1): 126; BP(2): 127,BP(3) :111,
HPLC-TOF	+	Exact mass (theoretical): 265.1233; measured value Δppm:-0.8; formula:C15H20ClNO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form		
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

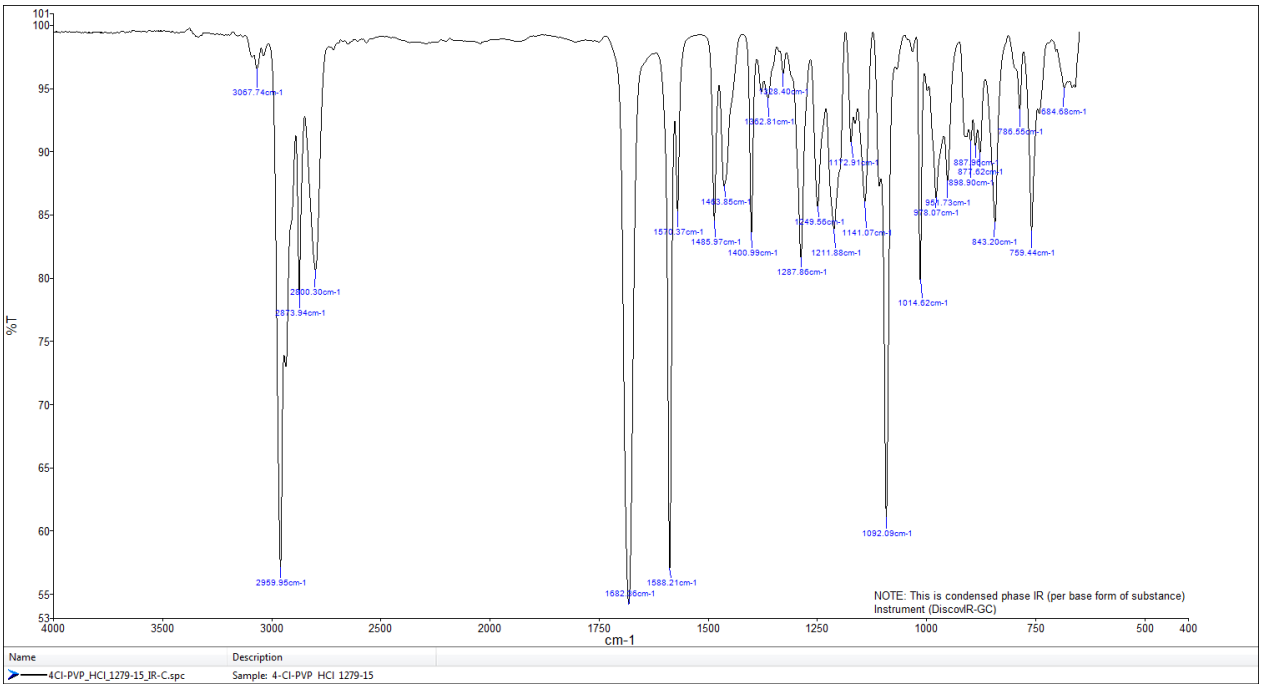
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation, base form of compound)



TOF REPORT

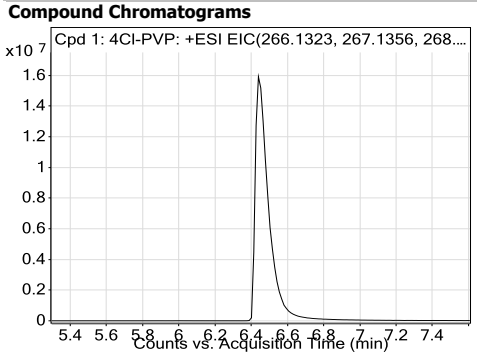
Data File	4-Cl-PVP_1279-15_TOF.d	**Sample Name**	4-Cl-PVP
Sample Type	Sample	**Position**	P1-E4
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	9/21/2015 9:57:59 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

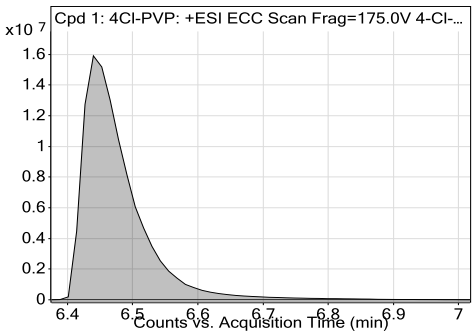
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: 4Cl-PVP	4Cl-PVP	C15 H20 Cl N O	6.452	265.1236

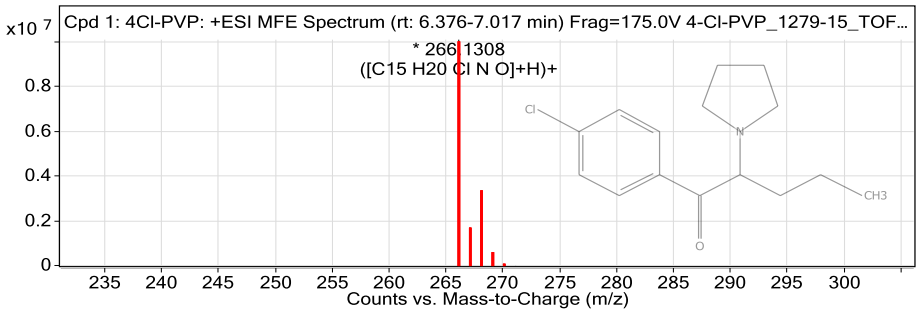
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4Cl-PVP	266.1308	6.452	265.1236	6.45	C15 H20 Cl N O	265.1233	-0.8

Compound Chromatograms

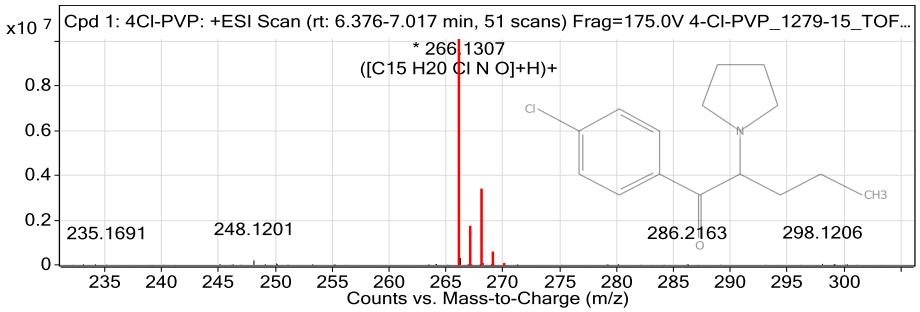




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

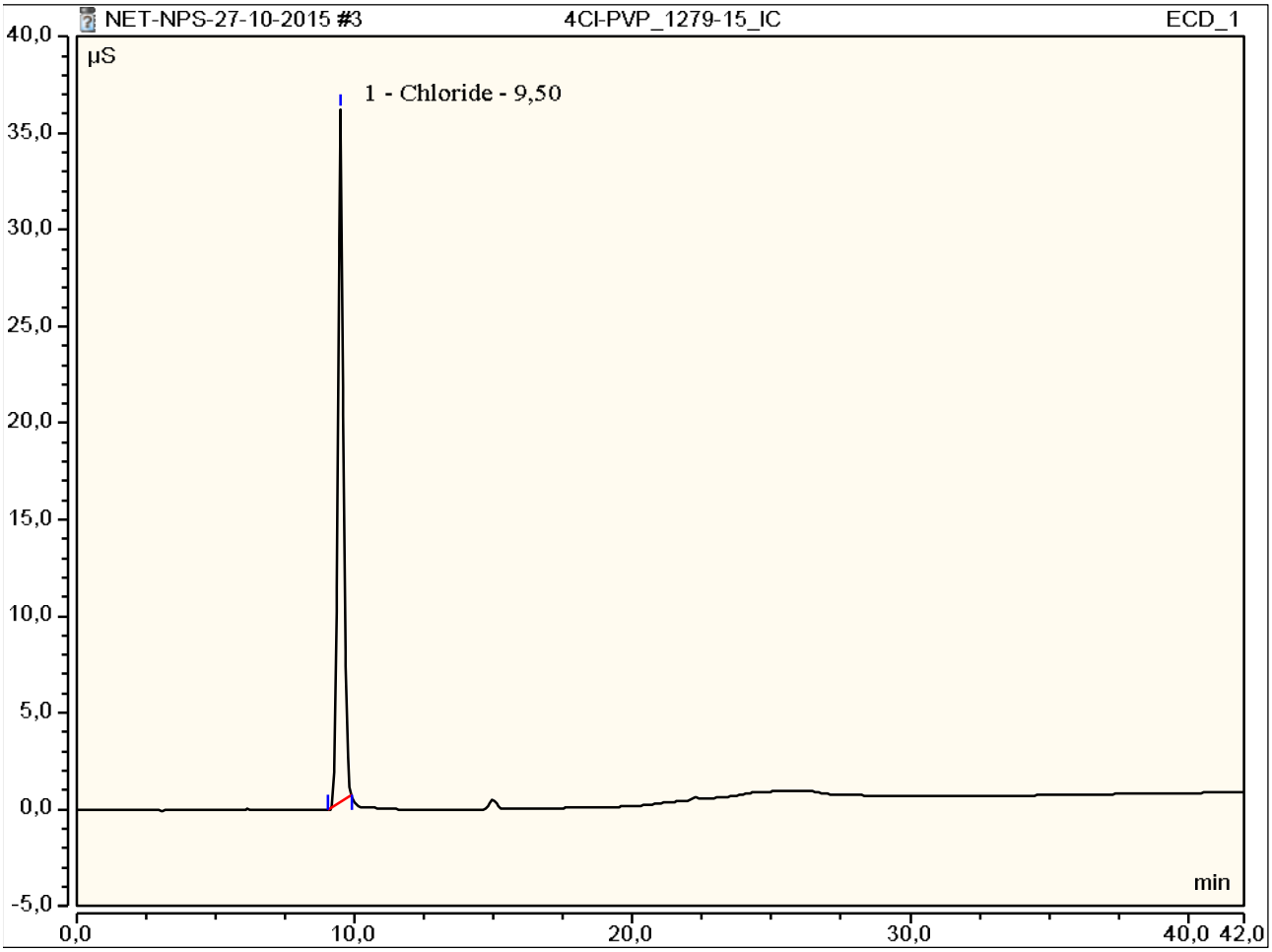
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
266.1308	1	10079695	C15 H20 Cl N O	(M+H)+
267.1341	1	1685641.23	C15 H20 Cl N O	(M+H)+
268.1283	1	3280432.44	C15 H20 Cl N O	(M+H)+
269.1316	1	510969.43	C15 H20 Cl N O	(M+H)+
270.1342	1	43589.41	C15 H20 Cl N O	(M+H)+
271.1371	1	3083.85	C15 H20 Cl N O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**4Cl-PVP_1279-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**27-okt-2015 / 13:05**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,50	**Chloride**	BMB	8,49	35,81	n.a.
		TOTAL:		8,49	35,81	0,00



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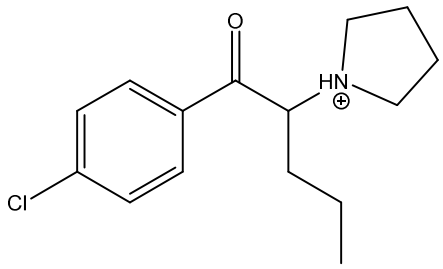
REPORT

Sample ID:	**1279-15**
Our notebook code:	P-1279-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO-d_6
NMR experiments:	^{1}H, ^{13}C.
Proposed structure:	
Chemical name:	1-(1-(4-chlorophenyl)-1-oxopentan-2-yl)pyrrolidin-1-ium
Comments:	- Structure elucidation based on 1D NMR spectra - Compound is pure by NMR.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 1, 2015

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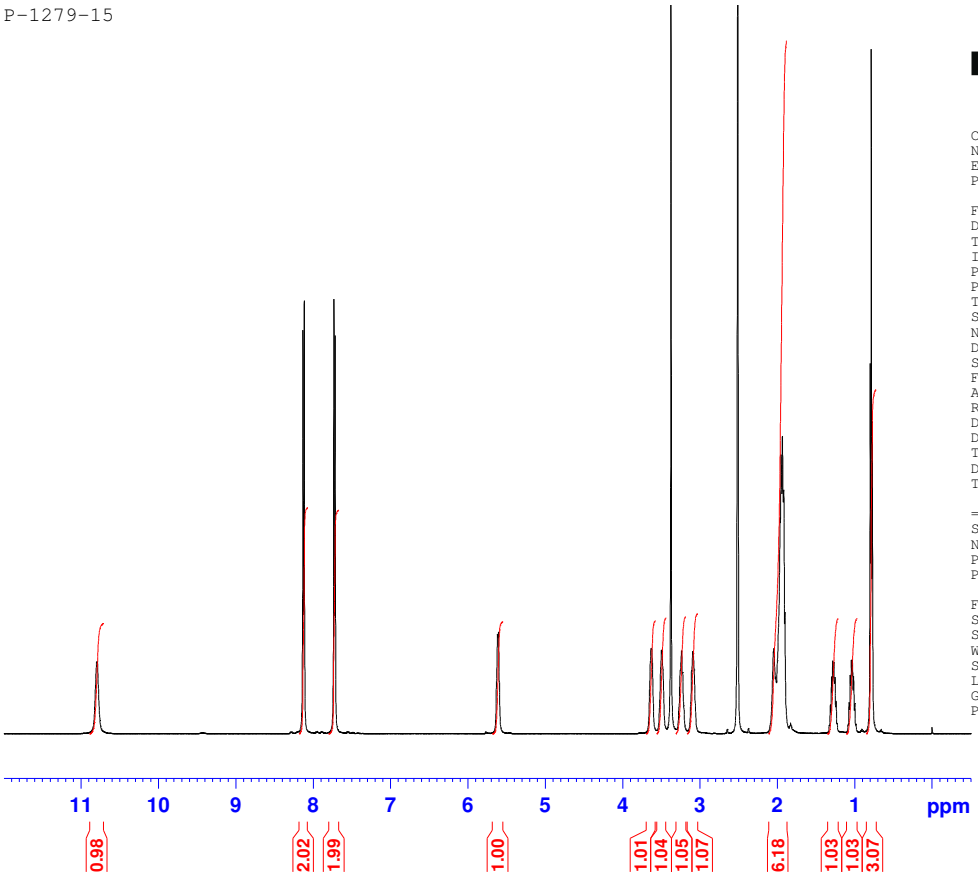


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Current Data Parameters
NAME          P-1279-15
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20151128
Time              16.02
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                296.0 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           500.1299982 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

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