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EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

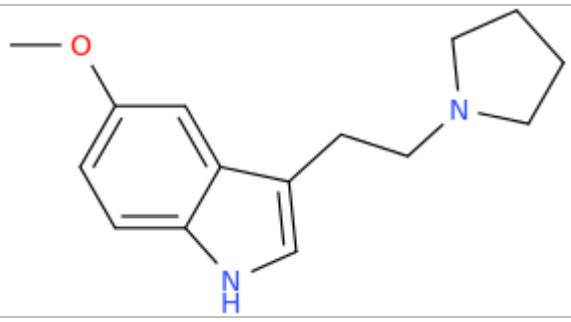
ANALYTICAL REPORT

5-MeO-pyr-T (C15H20N2O)

5-methoxy-3-[2-(pyrrolidin-1-yl)ethyl]-1H-indole

Remark – other NPS detected: none

Sample ID:	2031-18
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of sample receipt (DD/MM/YYYY):	19/11/2018
Date of entry (DD/MM/YYYY) into NFL database:	11/01/2019
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[1] (base form)	
Systematic name	5-methoxy-3-[2-(pyrrolidin-1-yl)ethyl]-1H-indole
Other names	5-methoxy-N,N-tetramethylenetryptamine; 5-methoxy-3-[2-(1-pyrrolidinyl)ethyl]-1H-indole, 5-methoxy-3-[2-(1-pyrrolidyl)ethyl]indole, 5-methoxy-3-(2-pyrrolidinoethyl)indole, methoxy-5-pyrrolidino-2'-ethyl-3-indole; pyrrolidyl-5-methoxytryptamine; 5-Methoxy-
Formula (per base form)	C15H20N2O
M_w (g/mol)	244,34
Salt form/anions detected	oxalate
StdInChIKey (per base form)	KAASYKNZNPWPQG-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	minor impurities observed by GC-MS and NMR (less than 5%)

[1] 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 (9.258 min). Injection volume 1 ml and split mode (1:50). Injector temperature: 280 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 7.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 6.1 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 (30 until 6 min.) to 550 (300 until 6 min) 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 (30 until 6 min.) to 550 (300) amu.
IR (condesed (solid) phase): IR scan range 4000 to 650, resolution 4 cm^{-1}.

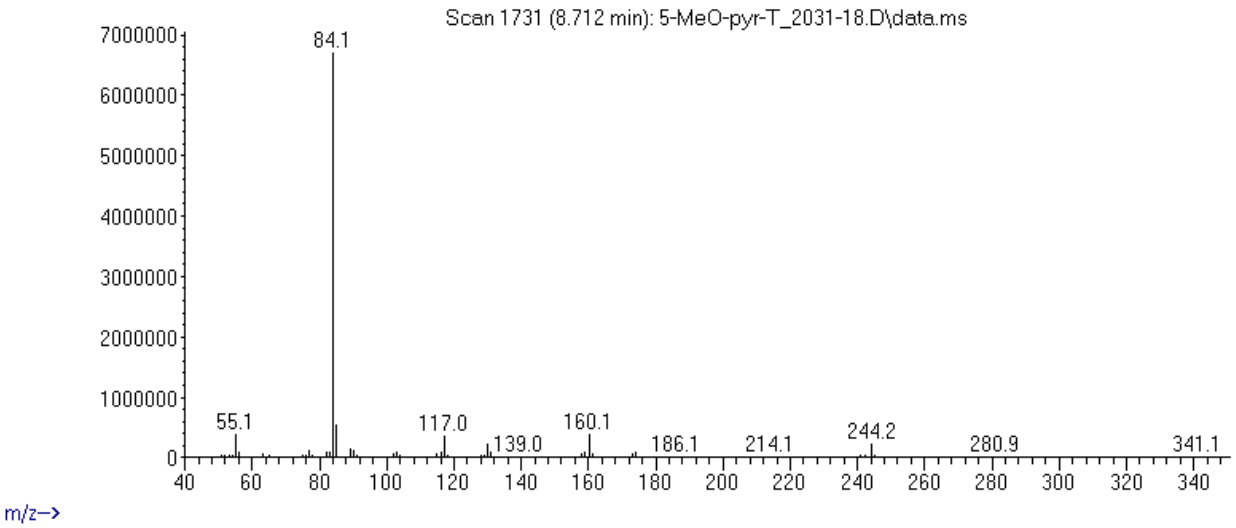
5. **IC** (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM KOH from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30˚C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

Supporting information

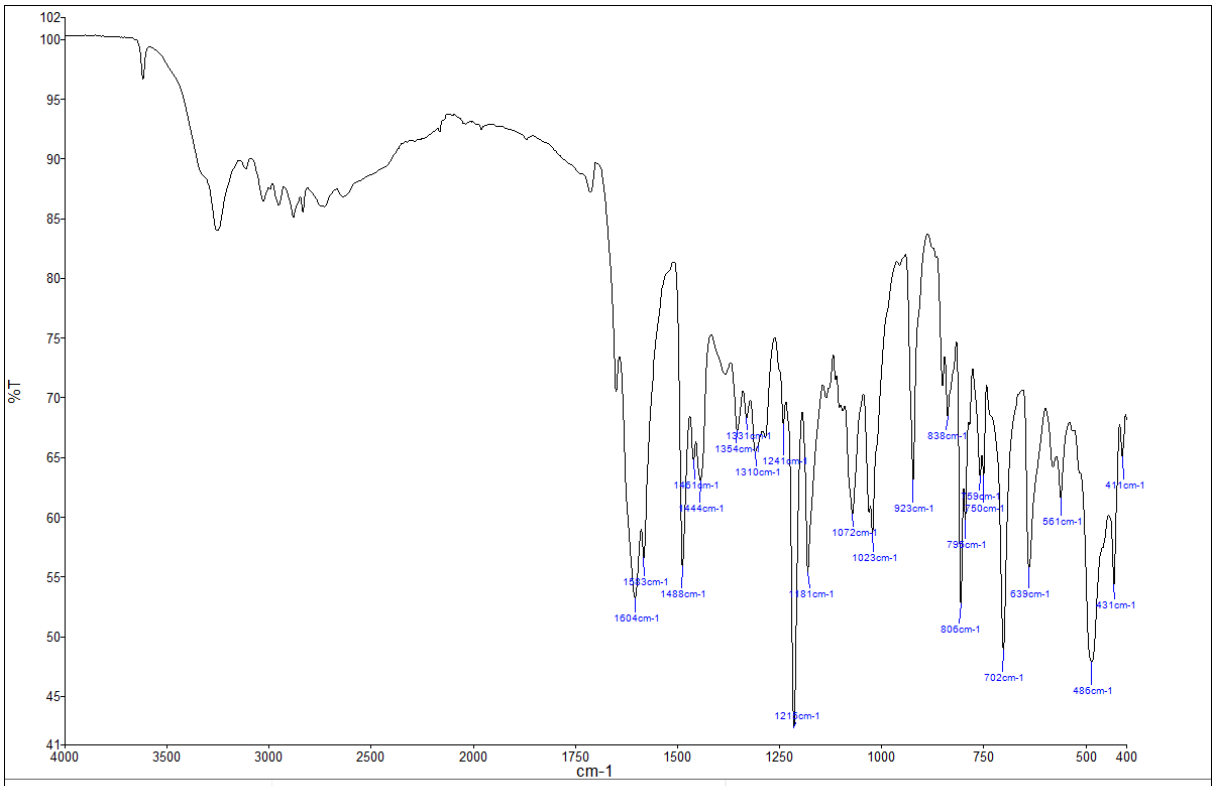
Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	soluble

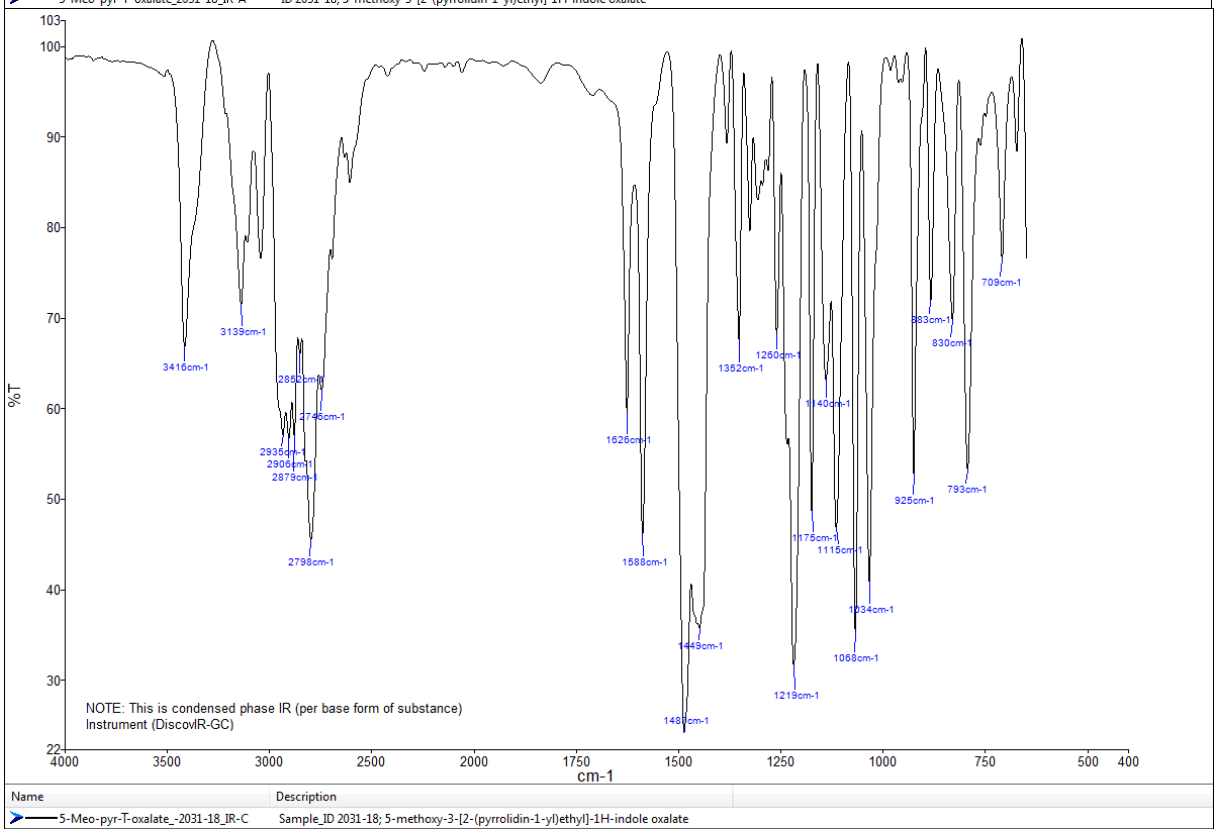
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8,71 BP(1): 84; BP(2): 85,BP(3) :160,
HPLC-TOF	+	Exact mass (theoretical): 244,1576; measured value Δppm:-1,39; formula:C15H20N2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

MS (EI)


Abundance
Scan 1731 (8.712 min): 5-MeO-pyr-T_2031-18.D\data.ms
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
84.1
55.1
117.0
139.0
160.1
186.1
214.1
244.2
280.9
341.1
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
m/z-->

FTIR-ATR - direct measurement (sample as received)





IR (solid phase – after chromatographic separation)

TOF REPORT

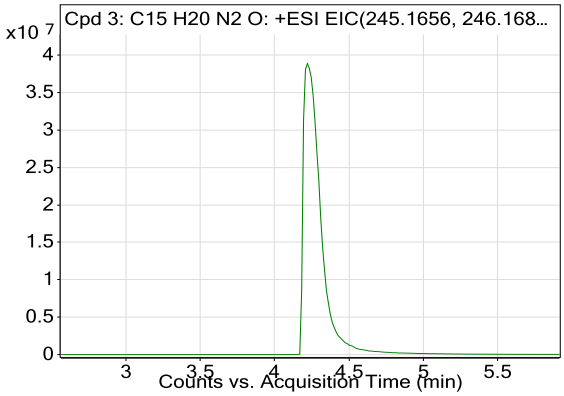
Data File	ID-2031_TOF.d	**Sample Name**	ID 2031-18
Sample Type	Sample	**Position**	P1-E3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-19_10_2018-XDB-C18-ESI+.m	**Acquired Time**	11/23/2018 10:01:20 AM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

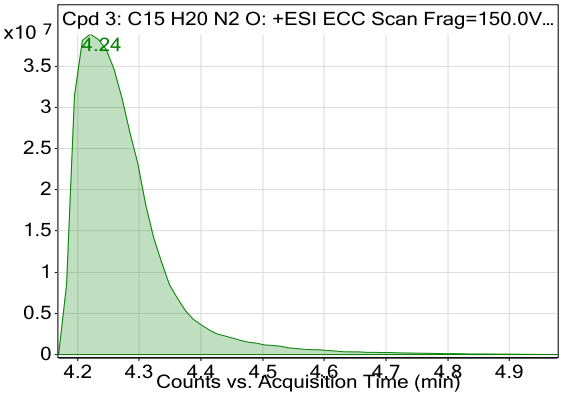
Compound Table

Label	MFG Formula	Obs. RT	Obs. Mass
Cpd 3: C15 H20 N2 O	C15 H20 N2 O	4.24	244.1579

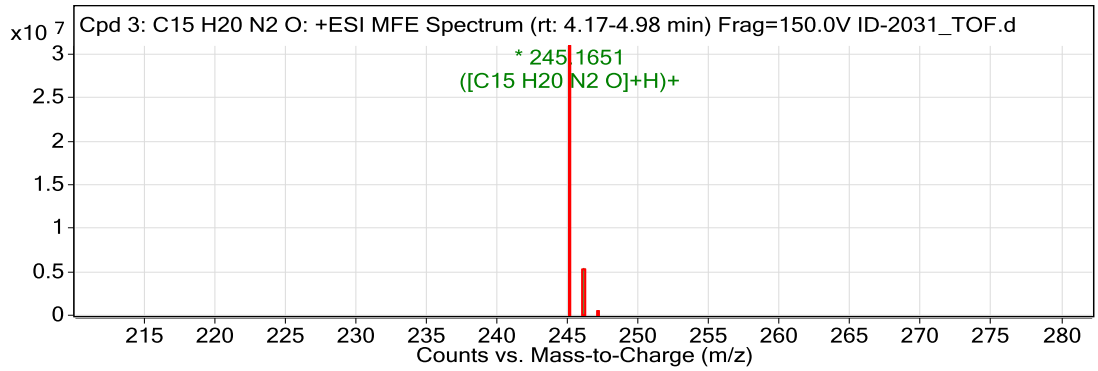
Obs. m/z	Obs. RT	Obs. Mass
245.1651	4.24	244.1579

Compound Chromatograms

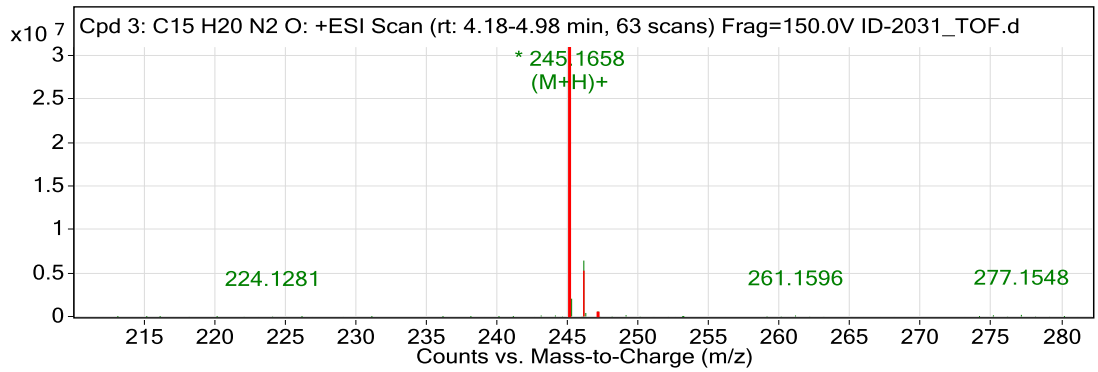




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
245.1651	1	30921624	C15 H20 N2 O	(M+H)+
246.1684	1	5433629.93	C15 H20 N2 O	(M+H)+
247.1717	1	441098.62	C15 H20 N2 O	(M+H)+

--- End Of Report ---

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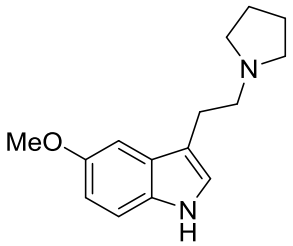
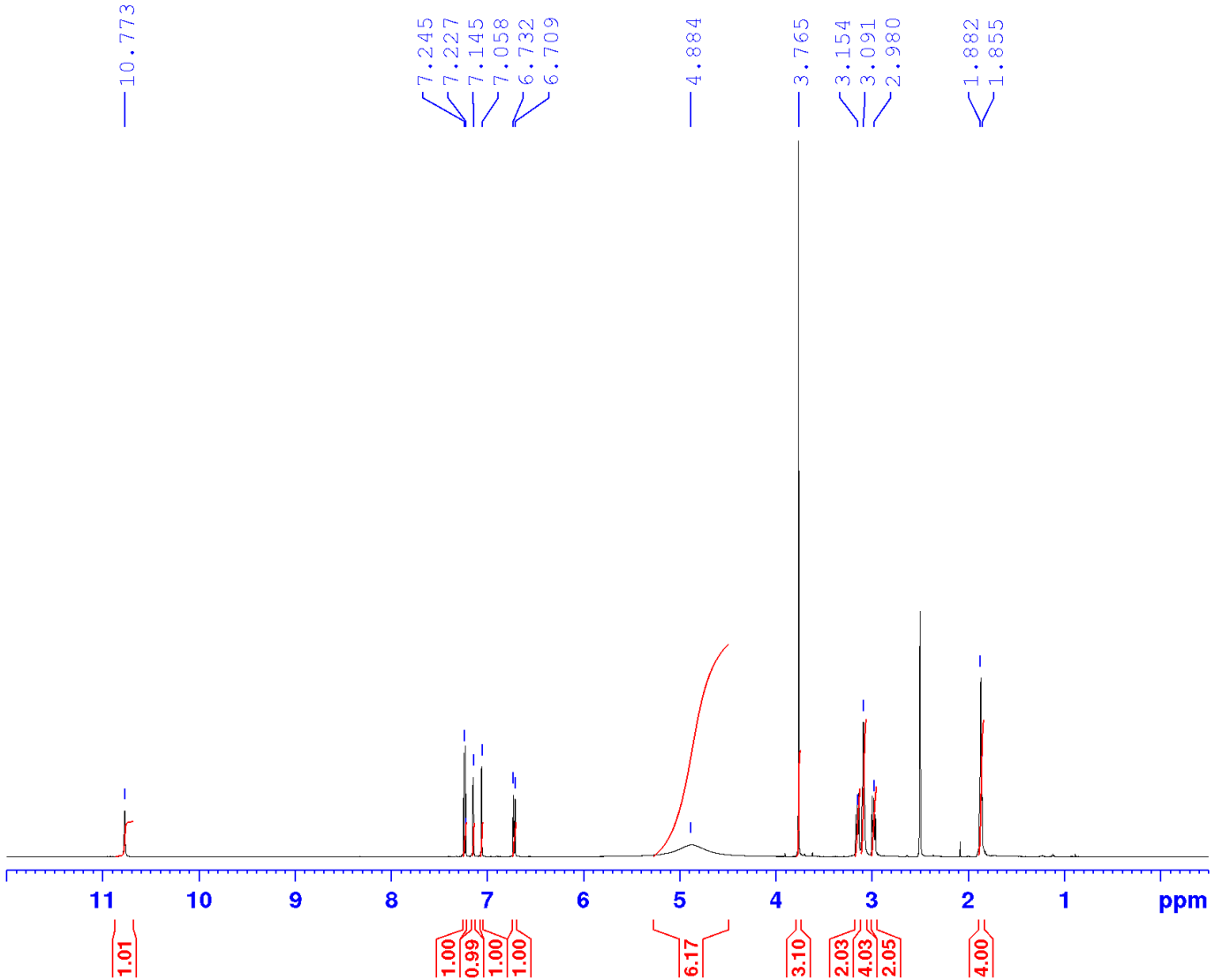
R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**2031-18**
Received date:	January 9, 2019
Our notebook code:	NFL-2031-18
NMR sample preparation:	19.9 mg dissolved in 0.7 mL DMSO-d_6
NMR experiments:	^{1}H, ^{13}C, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC, ^{1}H–^{15}N *gs*-HMBC
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{15}H_{20}N_2O$ Exact Mass: 244,1576 Molecular Weight: 244,3380
Chemical name:	5-methoxy-3-(2-(pyrrolidin-1-yl)ethyl)-1*H*-indole
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - The sample consists of herein identified compound and presumably oxalic acid, the amount of which was not determined. - Some minor impurities are also present (less than 5%).
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra, ^{1}H and ^{13}C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	January 17, 2019

NFL-2031-18
1H

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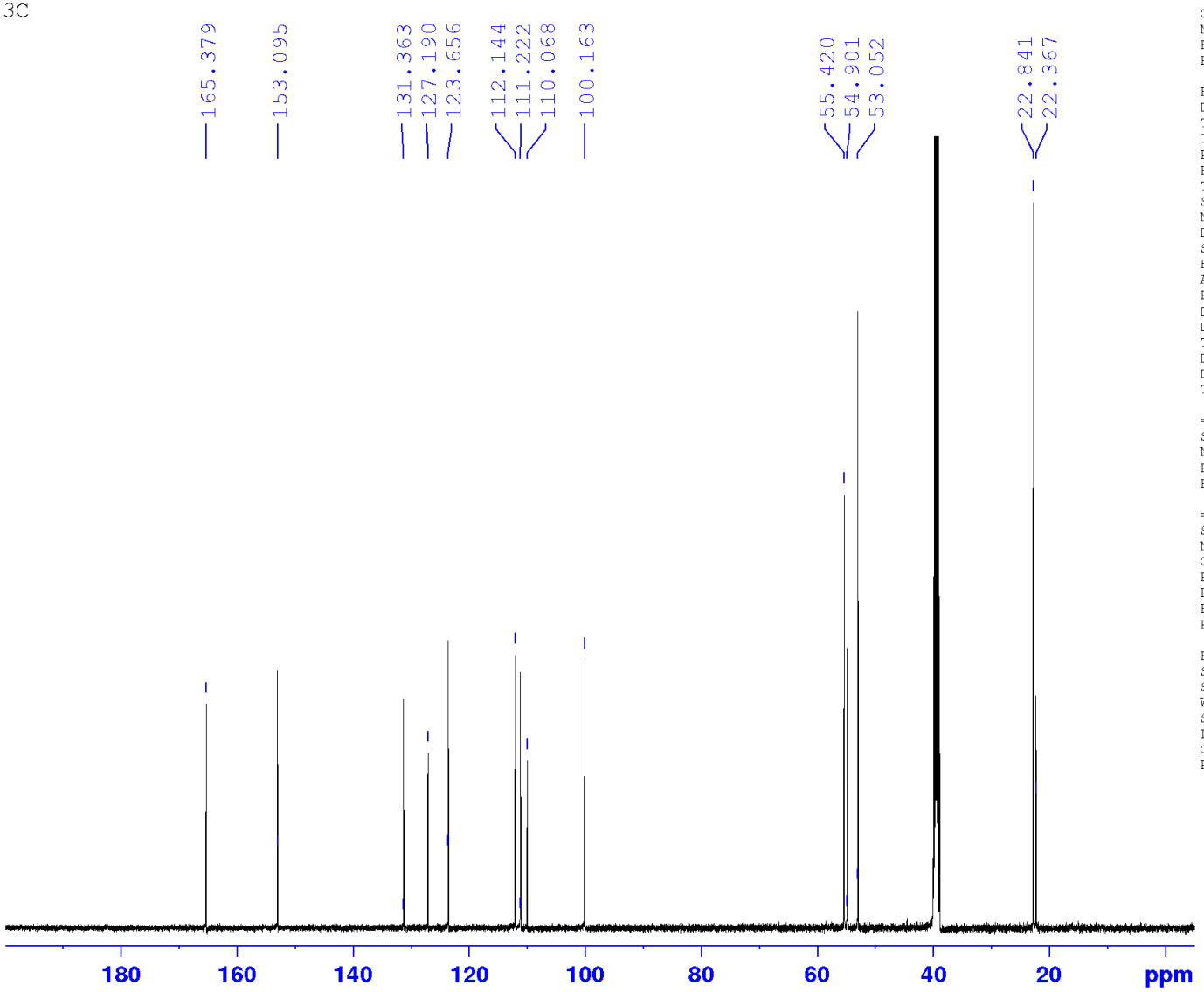
Current Data Parameters
NAME         NFL-2031-18
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181224
Time               13.09
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            3.2767999 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                 8.70 usec
PLW1        26.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300044 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

NFL-2031-18
13C




```
Current Data Parameters
NAME         NFL-2031-18
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181224
Time               15.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  3072
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                296.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                  13C
P1                   8.70 usec
PLW1        122.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         26.00000000 W
PLW12         0.30046001 W
PLW13         0.15113001 W

F2 - Processing parameters
SI                 32768
SF           125.7578443 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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