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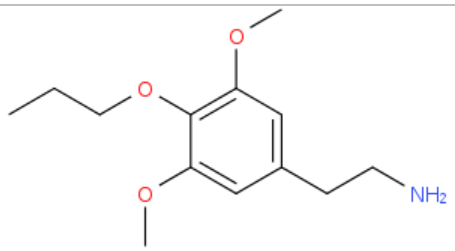
ANALYTICAL REPORT[1]

Proscaline (C13H21NO3)

2-(3,5-dimethoxy-4-propoxyphenyl)ethan-1-amine

Remark – other NPS detected: none

Sample ID:	1731-16
Sample description:	powder - brown
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	11/14/2016
Date of entry (M/D/Y) into NFL database:	12/12/2016
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	2-(3,5-dimethoxy-4-propoxyphenyl)ethan-1-amine
Other names	2-(3,5-dimethoxy-4-propoxyphenyl)ethanamine, 4-propoxy-3,5-DMPEA
Formula (per base form)	C13H21NO3
M_w (g/mol)	239,32
Salt form/anions detected	HCl
StdInChIKey	HYWLMSUAZVDUFW-UHFFFAOYSA-N
Compound Class	Phenethylamines
Other NPS detected	none
Add.info (purity..)	minor impurities by GC-MS, 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)

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 6.1 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 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

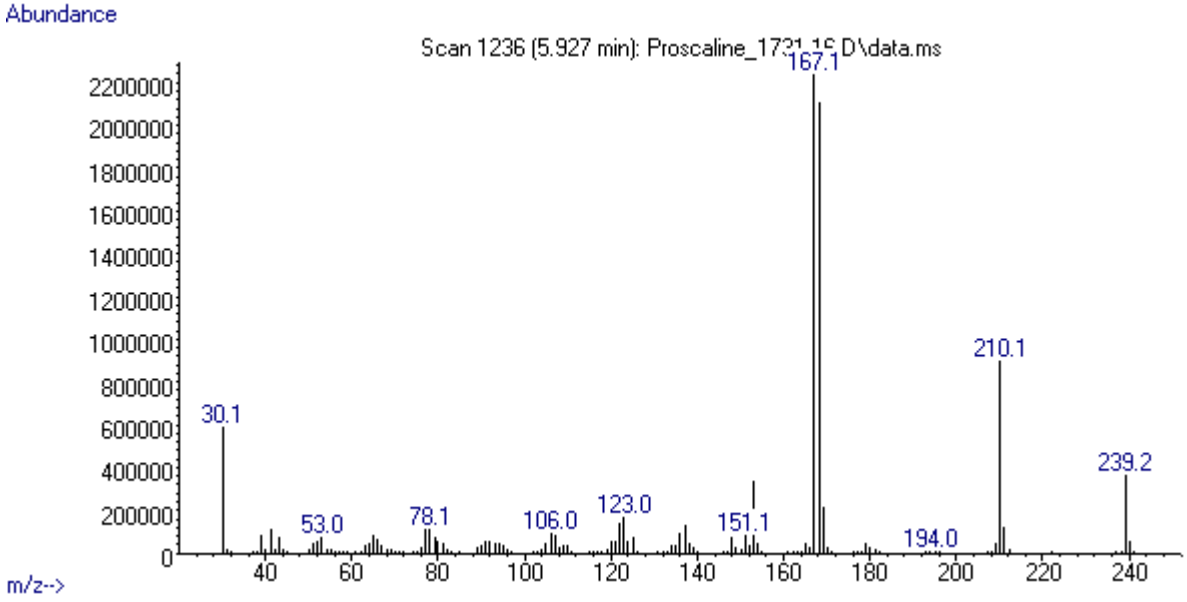
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	soluble

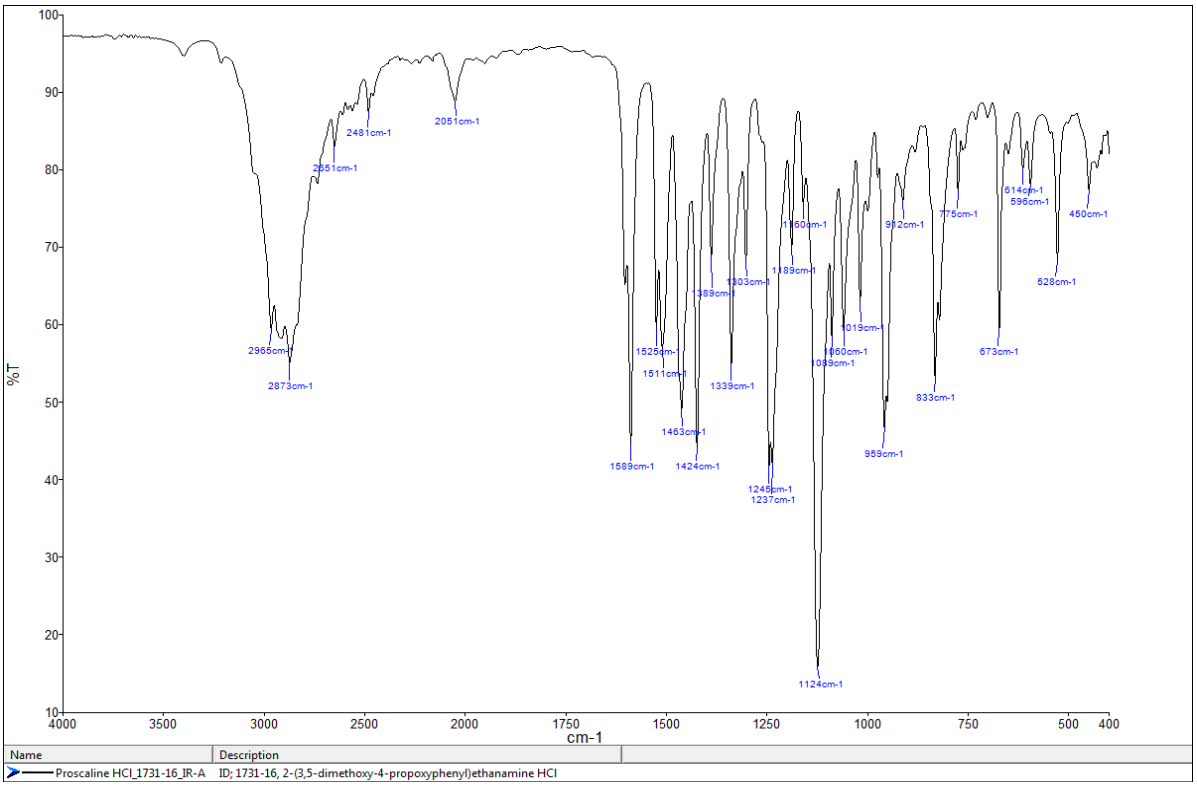
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 5,93 BP(1): 167; BP(2): 168,BP(3) :210,
HPLC-TOF	+	Exact mass (theoretical): 239,1521; measured value Δppm:-0,74; formula:C13H21NO3
FTIR-ATR	+	direct measurement (sample as received)
GC-IR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		MS consistent by SWGDRUG.L and ENFSI.L spectra (QM = 98); GC-IR condensed phase match with the spectrum of Proscaline obtained from FSI Zurich, Switzerland (cosine correlation >0.99).
other		

ANALYTICAL RESULTS

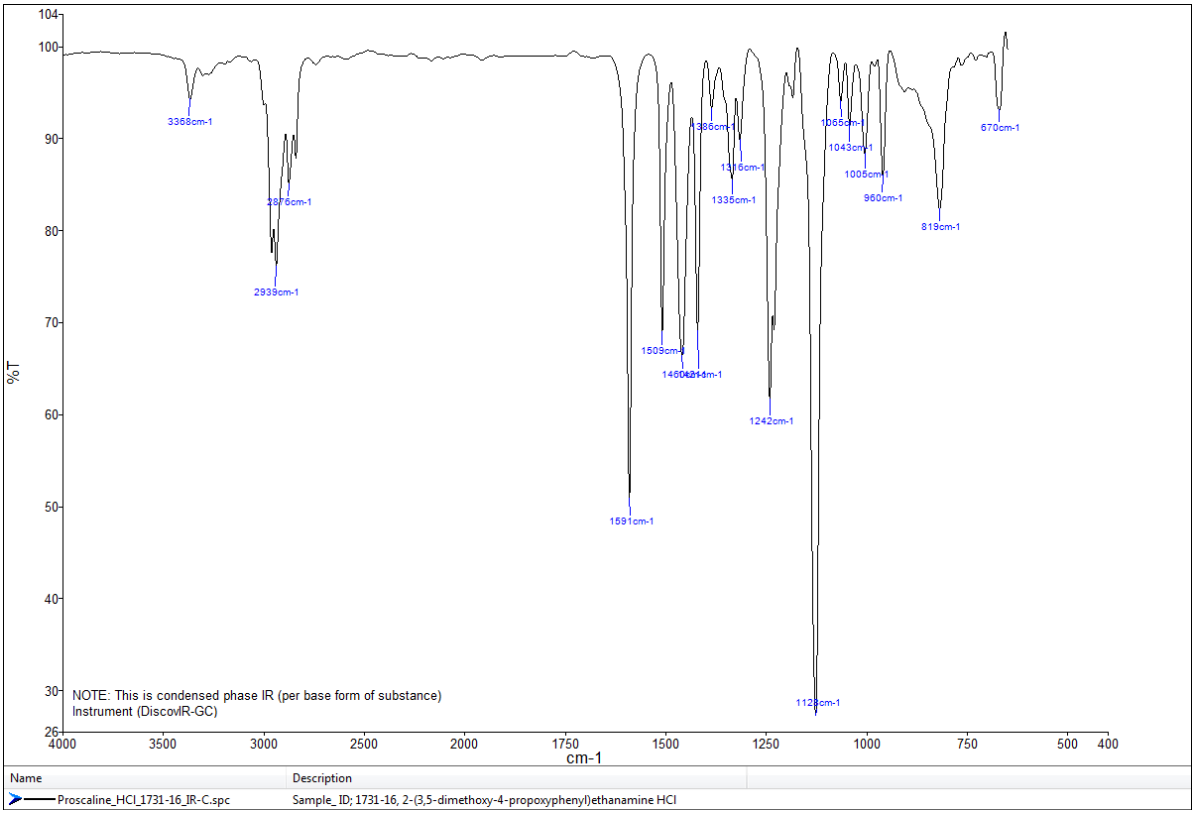
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

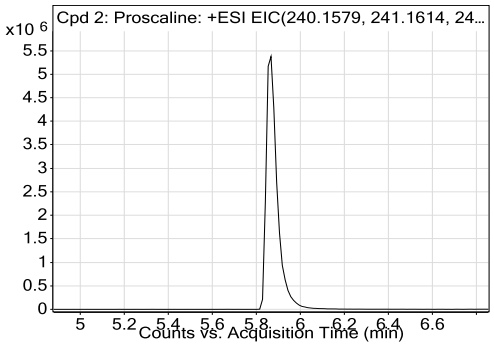
Data File	Proscaline_1731-16.d	**Sample Name**	ID_1731-16
Sample Type	Sample	**Position**	P1-C2
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-10_10_2016-XDB-C18-ESI-poz-soft.m	**Acquired Time**	11/16/2016 12:28:24 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

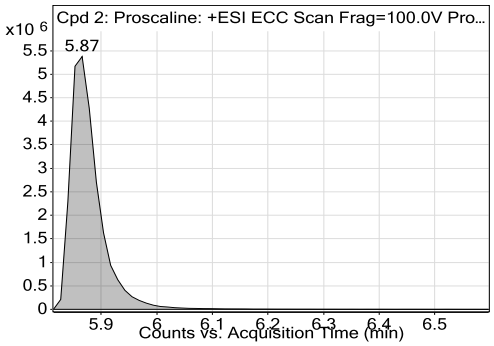
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: Proscaline	Proscaline	C13 H21 N O3	5.87	239.1523

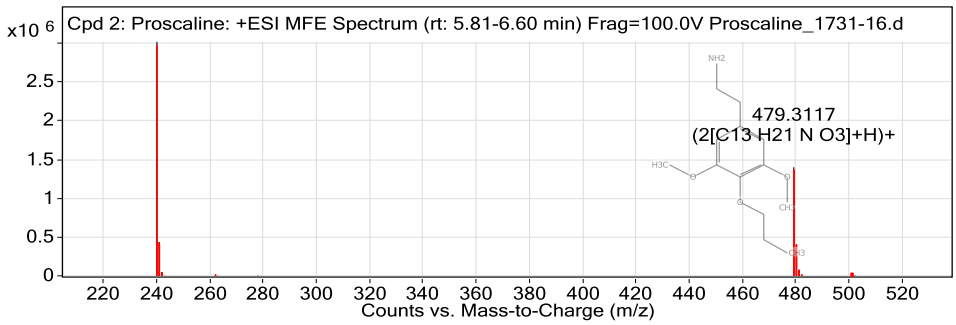
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Proscaline	240.1596	5.87	239.1523	5.87	C13 H21 N O3	239.1521	-0.74

Compound Chromatograms

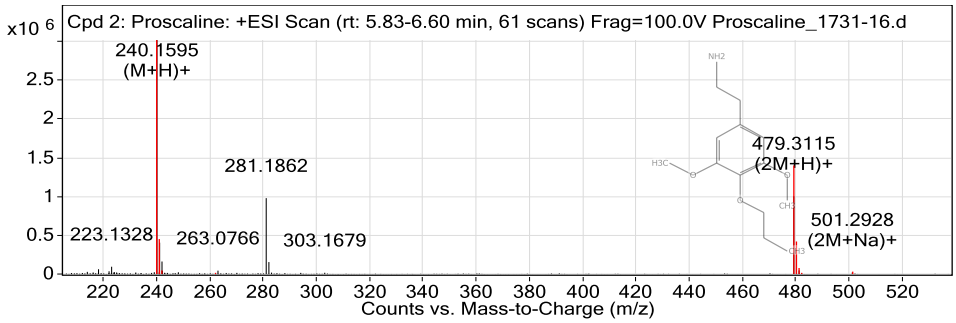




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

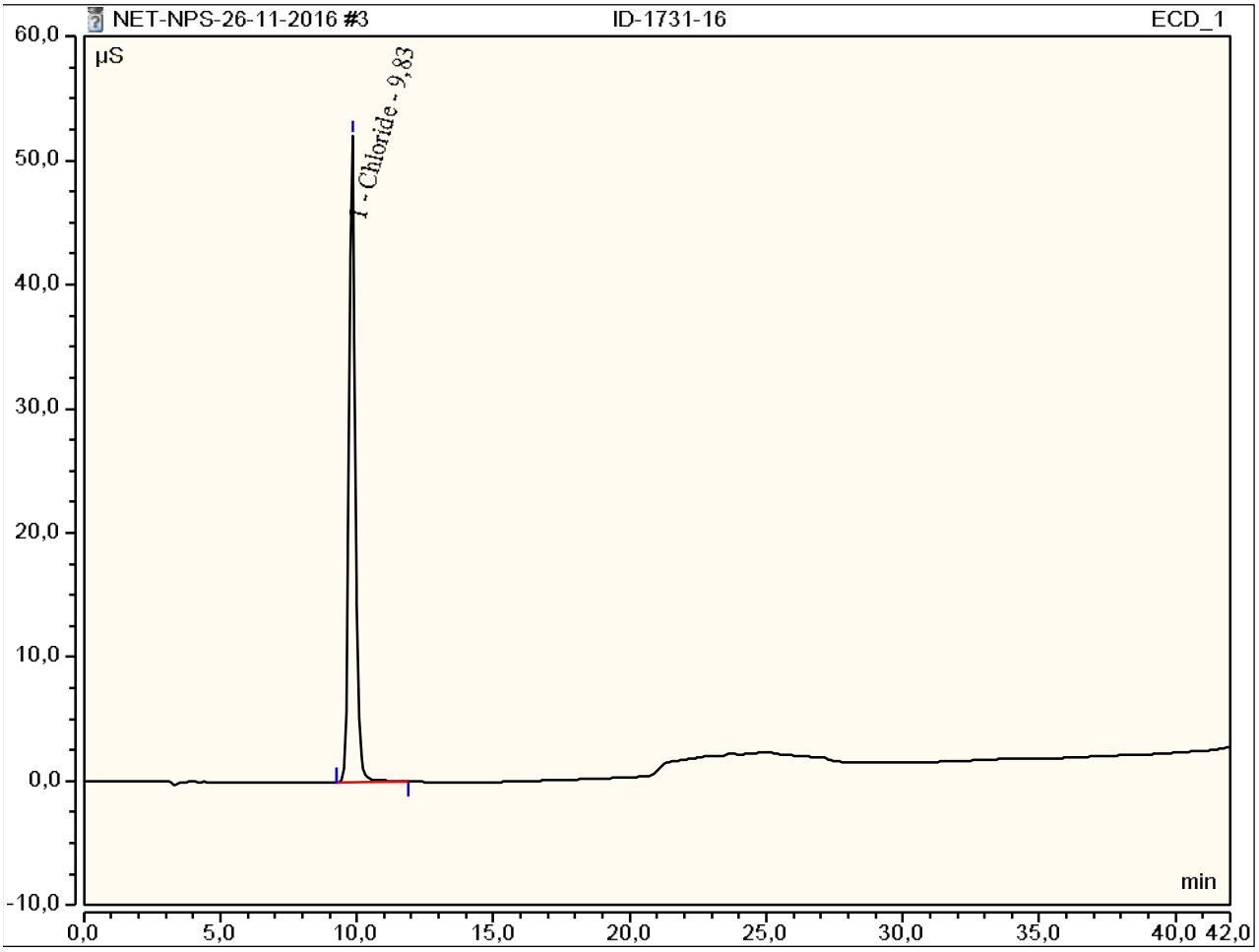
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
240.1596	1	3015598.5	C13 H21 N O3	(M+H)+
241.1632	1	401862.77	C13 H21 N O3	(M+H)+
242.1653	1	40369.67	C13 H21 N O3	(M+H)+
262.1417	1	10971.78	C13 H21 N O3	(M+Na)+
479.3117	1	1400294.5	C13 H21 N O3	(2M+H)+
480.3153	1	383177.18	C13 H21 N O3	(2M+H)+
481.3172	1	64843.93	C13 H21 N O3	(2M+H)+
482.3189	1	8563.03	C13 H21 N O3	(2M+H)+
501.293	1	25561.17	C13 H21 N O3	(2M+Na)+
502.2964	1	7927.18	C13 H21 N O3	(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**ID-1731-16**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**16-nov-2016 / 13:55**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,83	**Chloride**	BMB	14,53	52,14	n.a.
		TOTAL:		14,53	52,14	0,00



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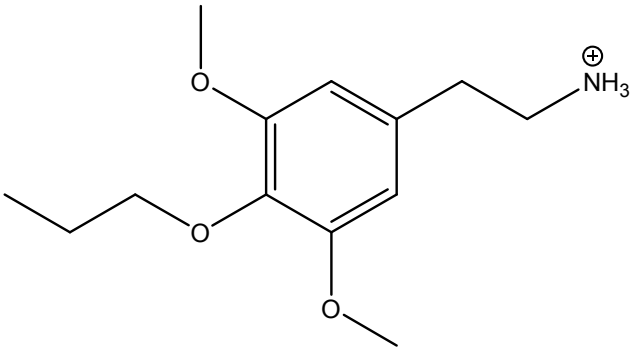
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1731-16**
Our notebook code:	P-1731-16
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
NMR experiments:	^1H, ^{13}C, ^1H–^1H *gs*-COSY, ^1H–^{13}C *gs*-HSQC, ^1H–^{13}C *gs*-HMBC, ^1H–^{15}N *gs*-HMBC.
Proposed structure:	
Chemical name:	2-(3,5-dimethoxy-4-propoxyphenyl)ethan-1-aminium cation
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample contains a minor amount of impurities, according to the NMR.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 12, 2016

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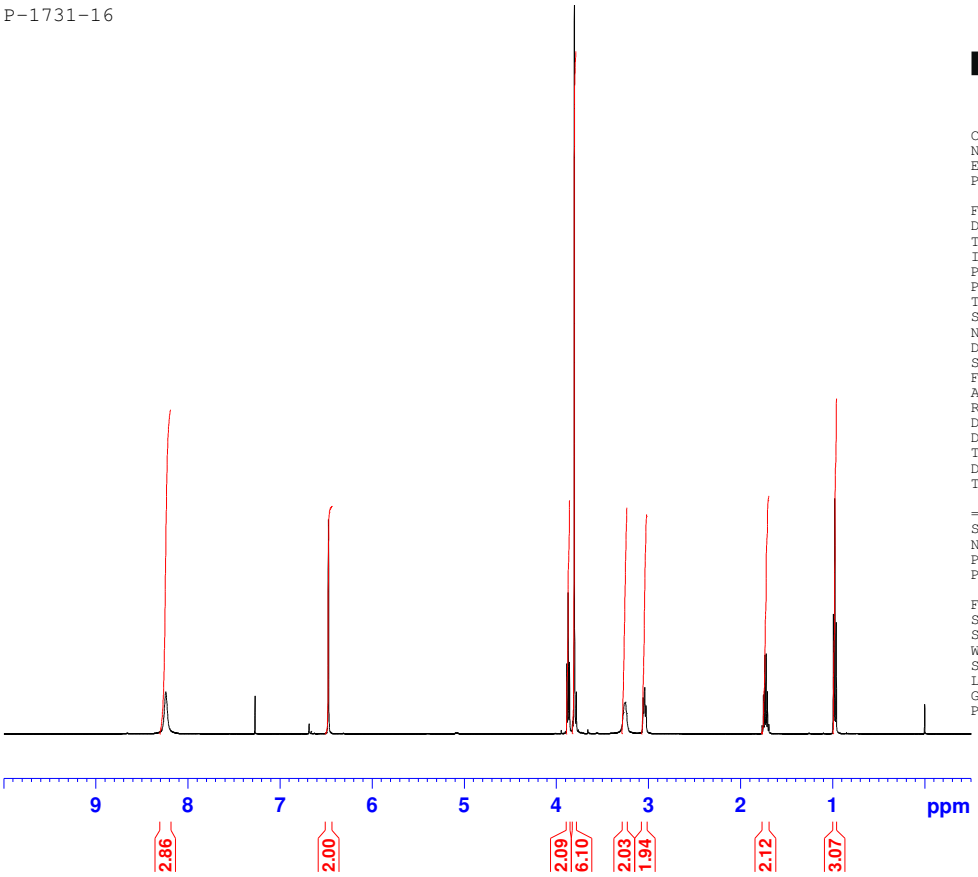
P-1731-16



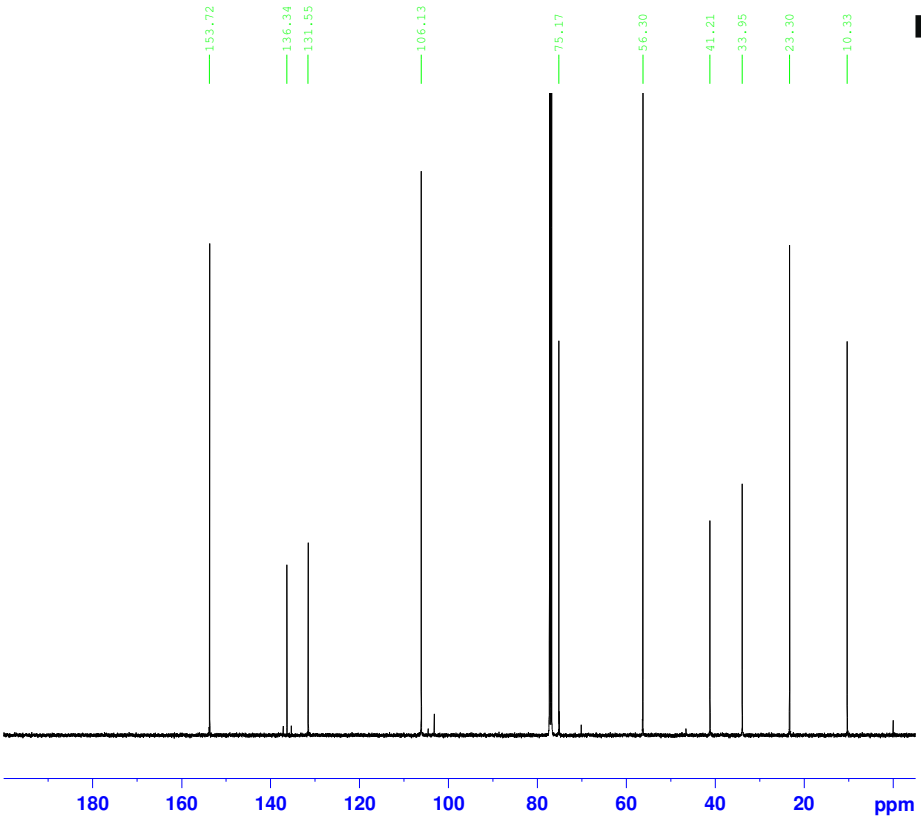
Current Data Parameters
NAME P-1731-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161207
Time 22.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 57
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 8.60 usec
PLW1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1731-16



Current Data Parameters
NAME P-1731-16
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161208
Time 0.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 298.0 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 125.7577880 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40