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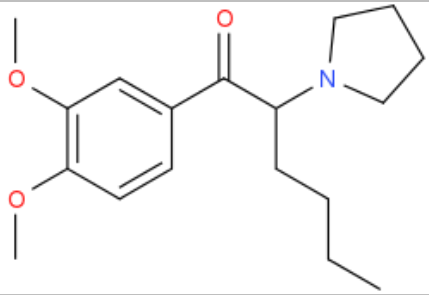
ANALYTICAL REPORT

3,4-dimethoxy-a-PHP (C18H27NO3)

1-(3,4-dimethoxyphenyl)-2-(pyrrolidin-1-yl)hexan-1-one

Remark – other NPS detected: none

Sample ID:	1250-15
Sample description:	powder - yellowish (gently)
Sample type:	test purchase /RESPONSE -purchasing
Comments[1]:	
Date of entry into NFL database: http://www.policija.si/apps/nfl_response_web/seznam.php	8/19/2015

Substance identified-structure[2] (base form)	
Systematic name	1-(3,4-dimethoxyphenyl)-2-(pyrrolidin-1-yl)hexan-1-one
Other names	2,4-DMeO-PHP
Formula (per base form)	C18H27NO3
M_w (g/mol)	305.41
Salt form	HCl
StdInChIKey	KAWWDIAYCGMAPE-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	none
Add.info (purity..)	pure by GC-Ms, HPLC-TOF and 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)
9/11/2015	wrong file name of IR condensed spectra corrected

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}.

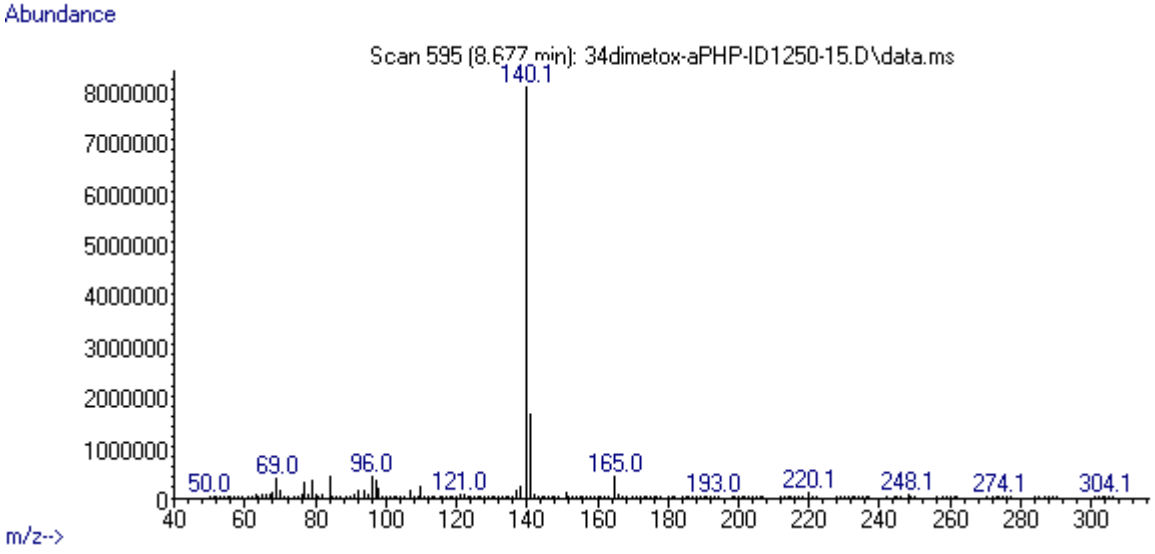
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	soluble

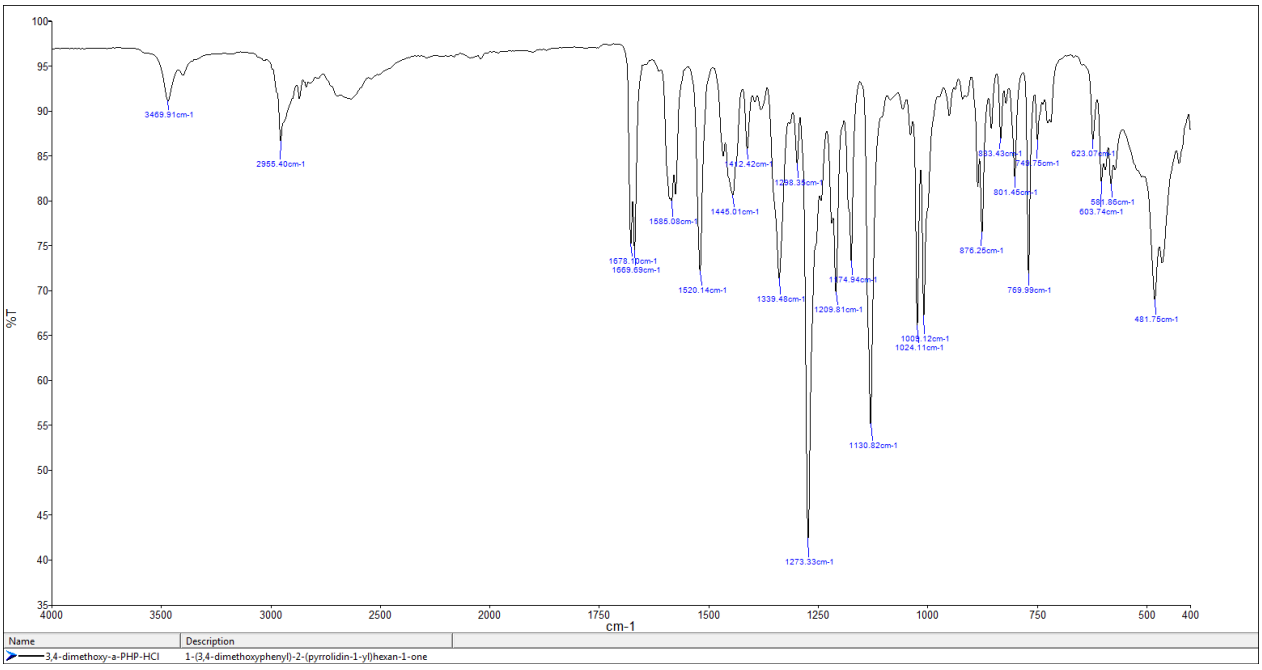
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8.68 BP(1): 140; BP(2): 141,BP(3) :96,
HPLC-TOF	+	Exact mass (theoretical): 305.1992; measured value Δppm:-0.56; formula:C18H27NO3
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
NMR	+	
validation		
other		

ANALYTICAL RESULTS

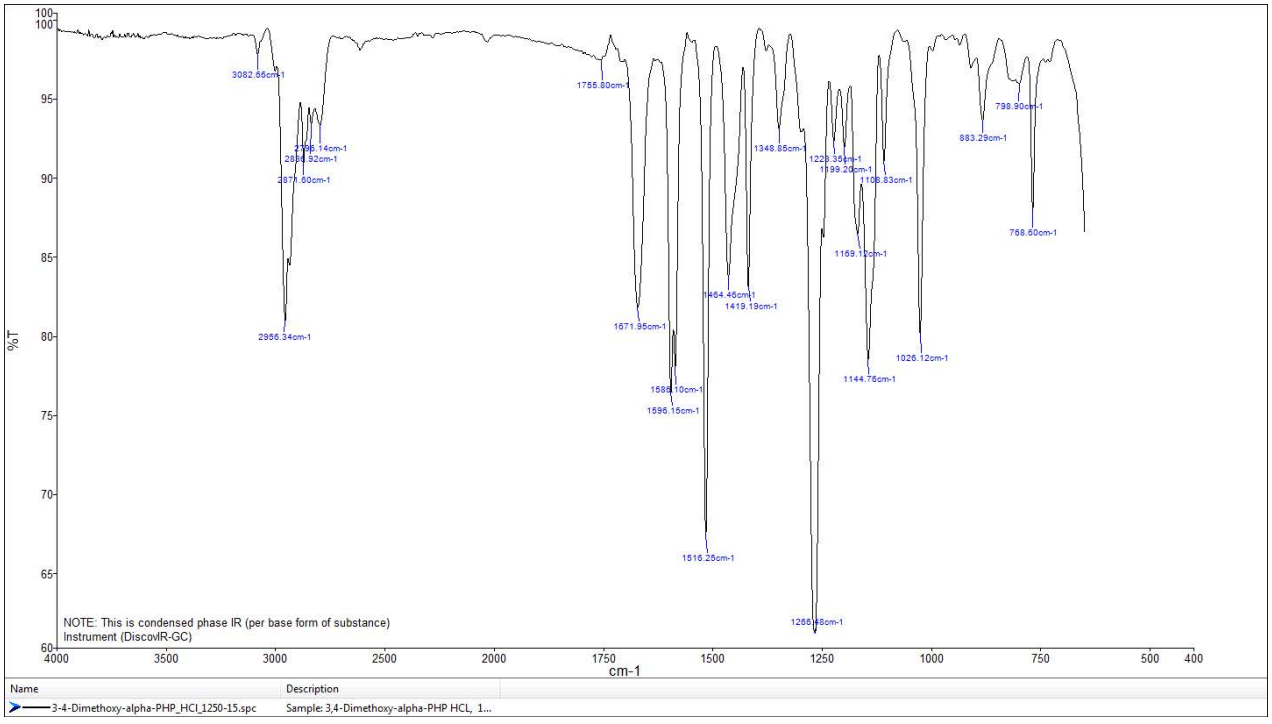
MS (EI)



FTIR-ATR - direct measurement



IR (condensed phase)



Target Compound Screening Report

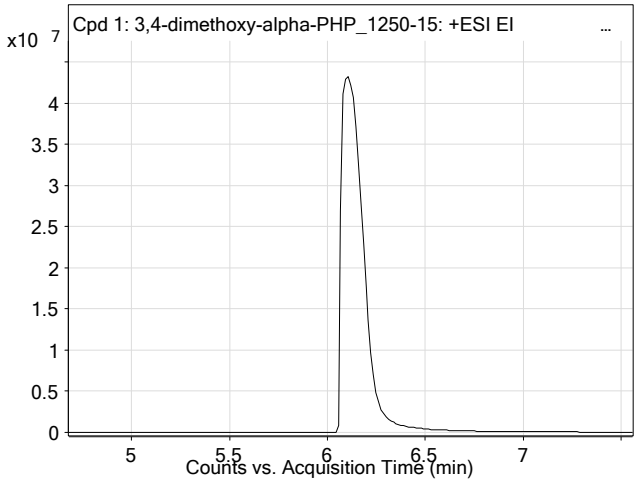
Data File	3_4-dimethoxy-alpha-PHP_1250-15_TOF.d	**Sample Name**	3,4-dimethoxy-alpha-PHP
Sample Type	Sample	**Position**	P2-E8
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	8/19/2015 2:10:39 PM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

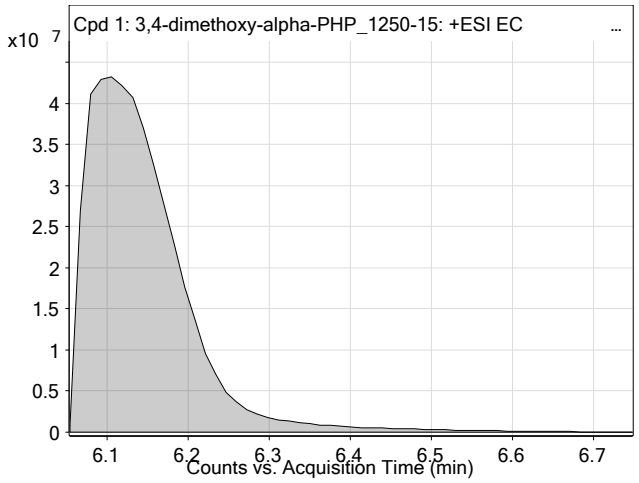
Compound Table

Label	Tgt Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: 3,4-dimethoxy-alpha-PHP_1250-15	3,4-dimethoxy-alpha-PHP_1250-15	C18 H27 N O3	6.117	305.1992

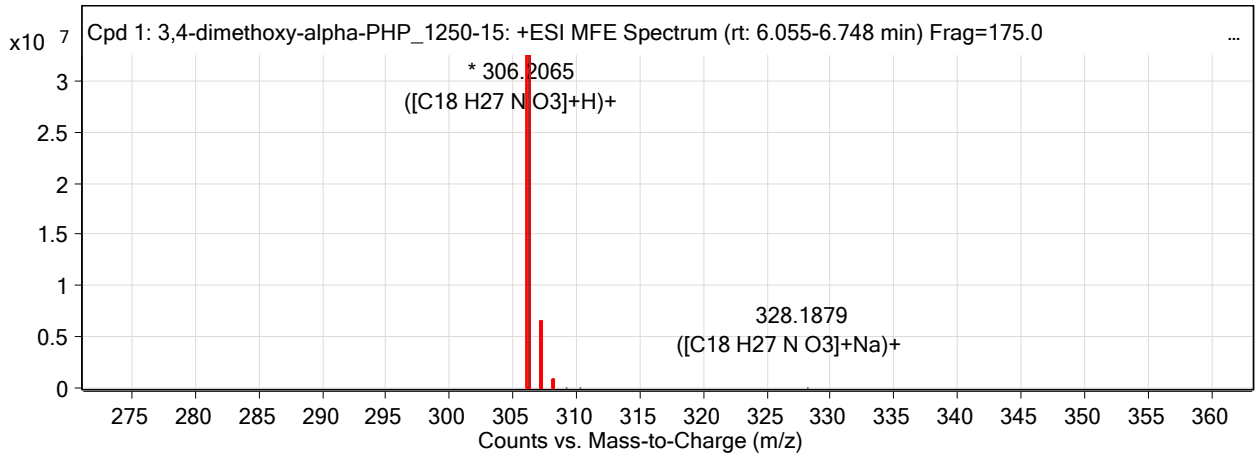
Name	Obs. *m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
3,4-dimethoxy-alpha-PHP_1250-15	306.2065	6.117	305.1992	6.117	C18 H27 N O3	305.1991	-0.51	Find by Molecular Feature

Compound Chromatograms





MFE MS Zoomed Spectrum

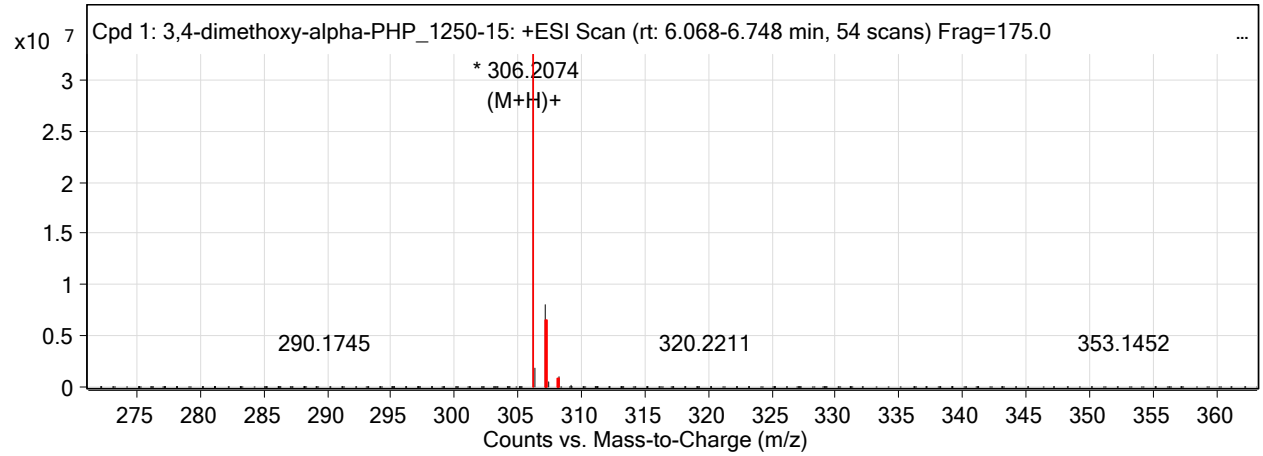


MS Spectrum Peak List

Obs. *m/z*	Charge	Abund	Formula	Ion/Isotope
306.2065	1	32504750	C18 H27 N O3	(M+H)+
307.2098	1	6595074.19	C18 H27 N O3	(M+H)+
308.2126	1	775663.74	C18 H27 N O3	(M+H)+
309.2153	1	72038.45	C18 H27 N O3	(M+H)+
310.2172	1	3618.6	C18 H27 N O3	(M+H)+
328.1879	1	9904.05	C18 H27 N O3	(M+Na)+

Agilent Technologies

MS Zoomed Spectrum

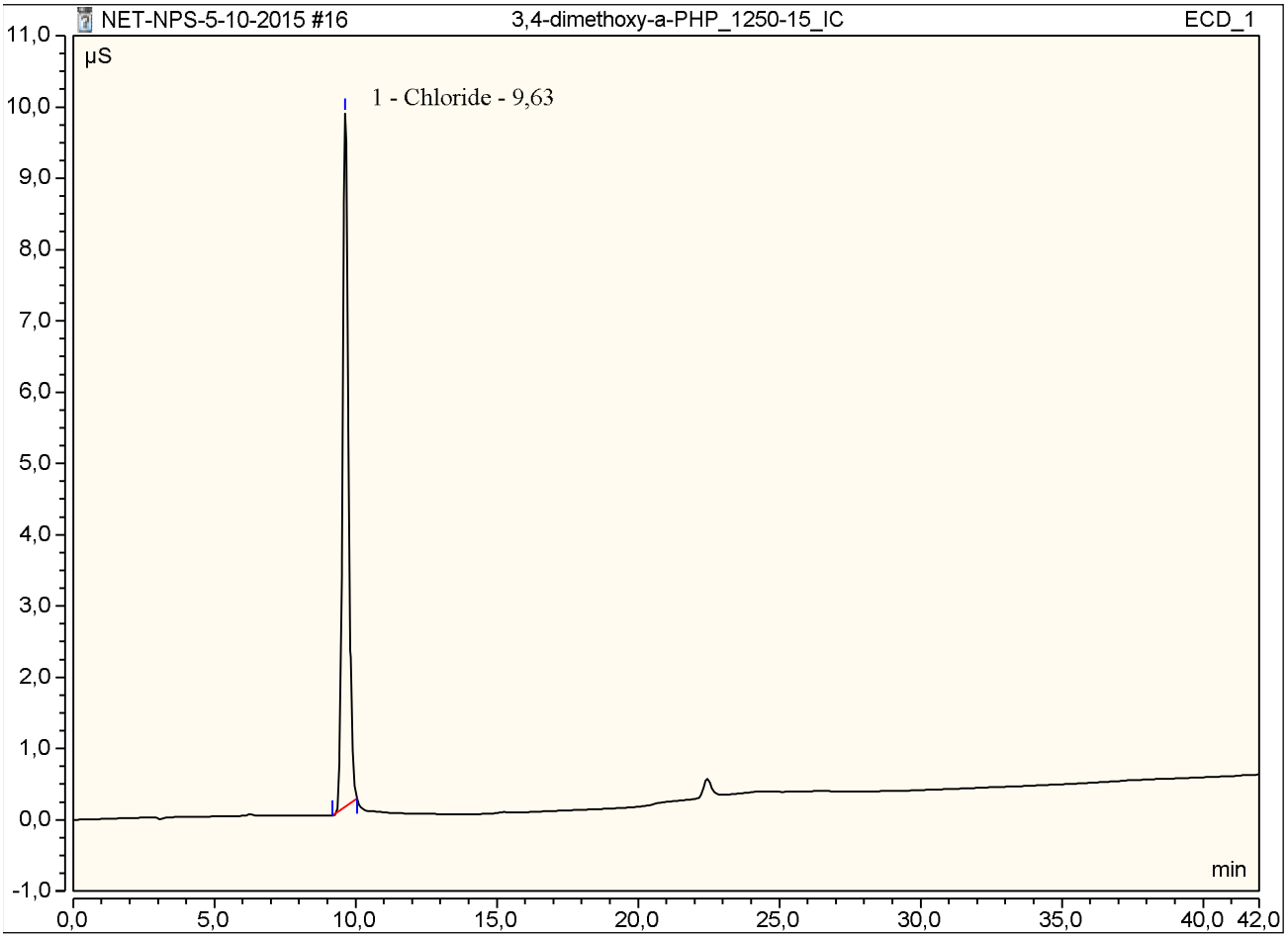


--- End Of Report ---

Peak Integration Report

Sample Name:	**3,4-dimethoxy-a-PHP_1250-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**06-okt-2015 / 02:11**	*Run Time:*	**41,99**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,63	**Chloride**	BMB	2,33	9,72	n.a.
		TOTAL:		2,33	9,72	0,00



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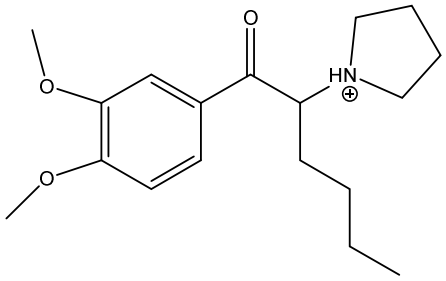
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1250-15**
Our notebook code:	P-1250-15
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 with chemical name:	 1-(1-(3,4-dimethoxyphenyl)-1-oxohexan-2-yl)pyrrolidin-1-ium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound 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:	October 5, 2015

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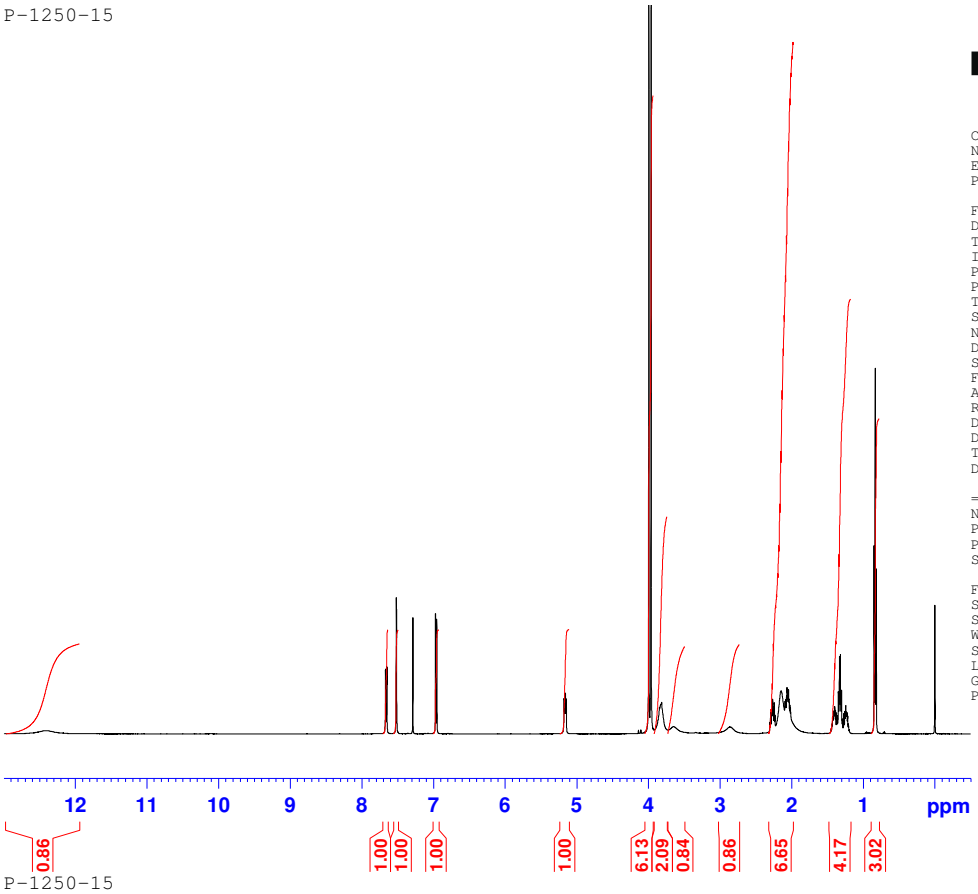
P-1250-15



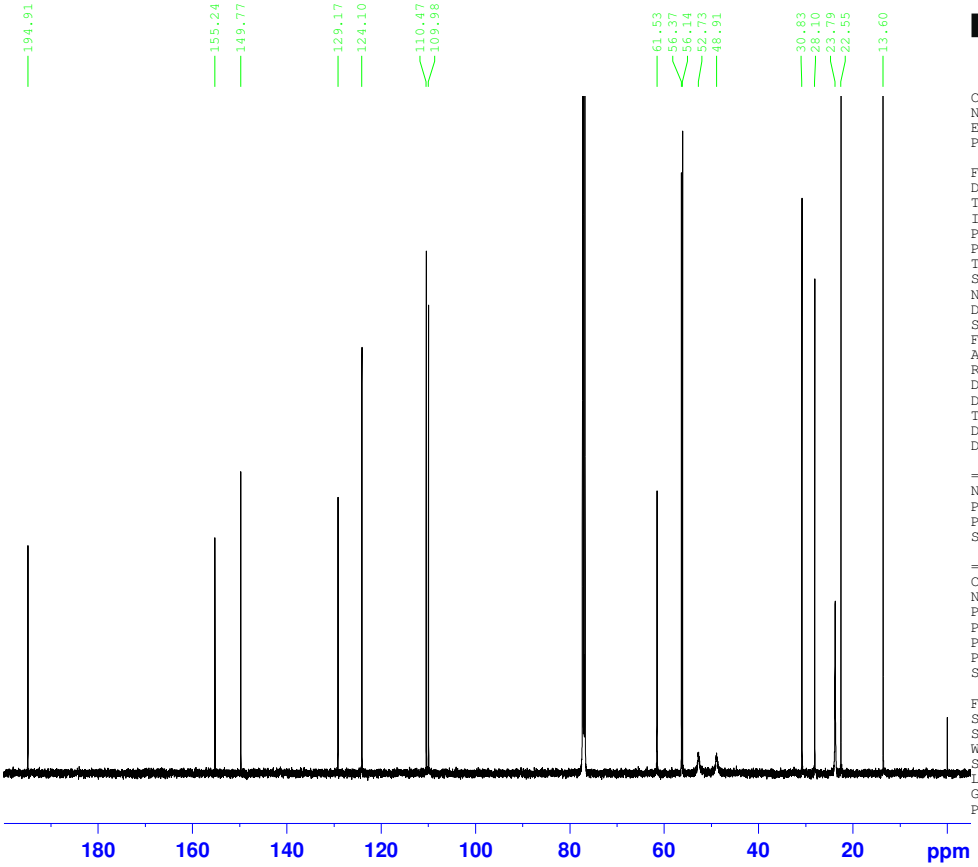

```
Current Data Parameters
NAME                  p-1250-15
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20150928
Time                     17.51
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                  CDC13
NS                          16
DS                           2
SWH                 10330.578 Hz
FIDRES               0.157632 Hz
AQ                   3.1719923 sec
RG                        80.6
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                  500.1299995 MHz
WDW                            EM
SSB                 0
LB                         0.30 Hz
GB                  0
PC                         1.00
```

P-1250-15

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Current Data Parameters
NAME                  p-1250-15
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20150928
Time                     20.57
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                  CDC13
NS                        5120
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                  1.00000000 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                  125.7577880 MHz
WDW                            EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
```