



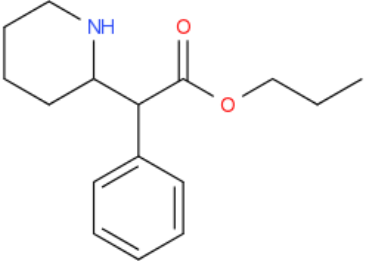
## ANALYTICAL REPORT<sup>1</sup>

### Propylphenidate (C<sub>16</sub>H<sub>23</sub>NO<sub>2</sub>)

#### Propyl-2-phenyl-2-(piperidin-2-yl)acetate

Remark – other NPS detected: **none**

Sample ID:	1246-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	8/18/2015
Date of entry (M/D/Y) into NFL database:	8/19/2015
Report (updates) 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	Propyl-2-phenyl-2-(piperidin-2-yl)acetate
Other names	2-(2-oxo-1-phenyl-2-propoxyethyl)piperidin
Formula (per base form)	C <sub>16</sub> H <sub>23</sub> NO <sub>2</sub>
M <sub>w</sub> (g/mol)	261,37
Salt form	HCl
StdInChIKey	PRMWEANNQSWAR-UHFFFAOYSA-N
Compound Class	Piperidines & pyrrolidines
Other NPS detected	none
Add.info (purity..)	pure by GC-MS, HPLC, NMR

<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

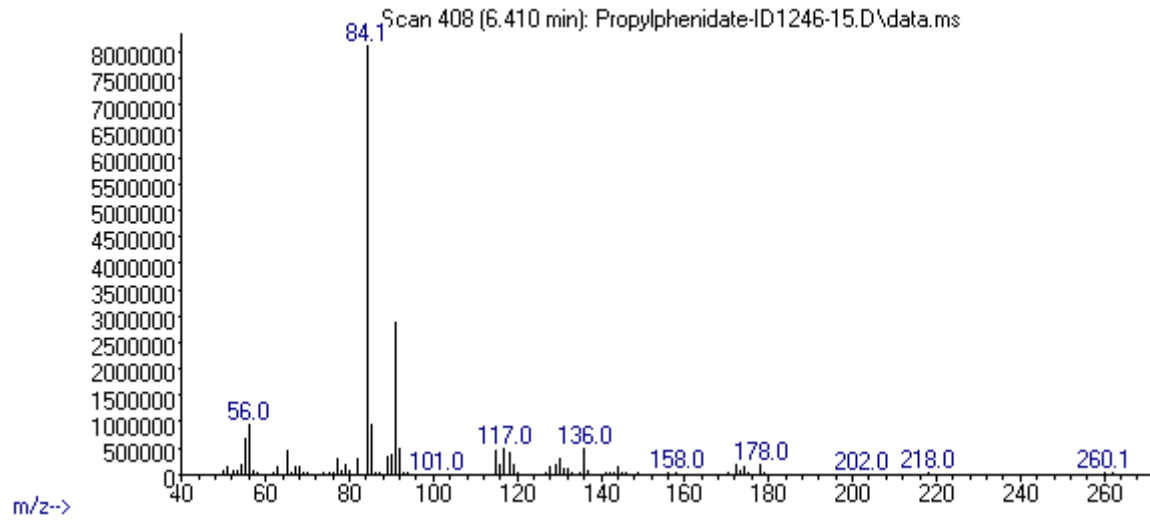
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	soluble
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 6,41 BP(1): 84; BP(2): 91,BP(3) :56,
HPLC-TOF	+	Exact mass (theoretical): 261,1729; measured value Δppm:-0,56; formula:C16H23NO2
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR	+	
validation		
other		

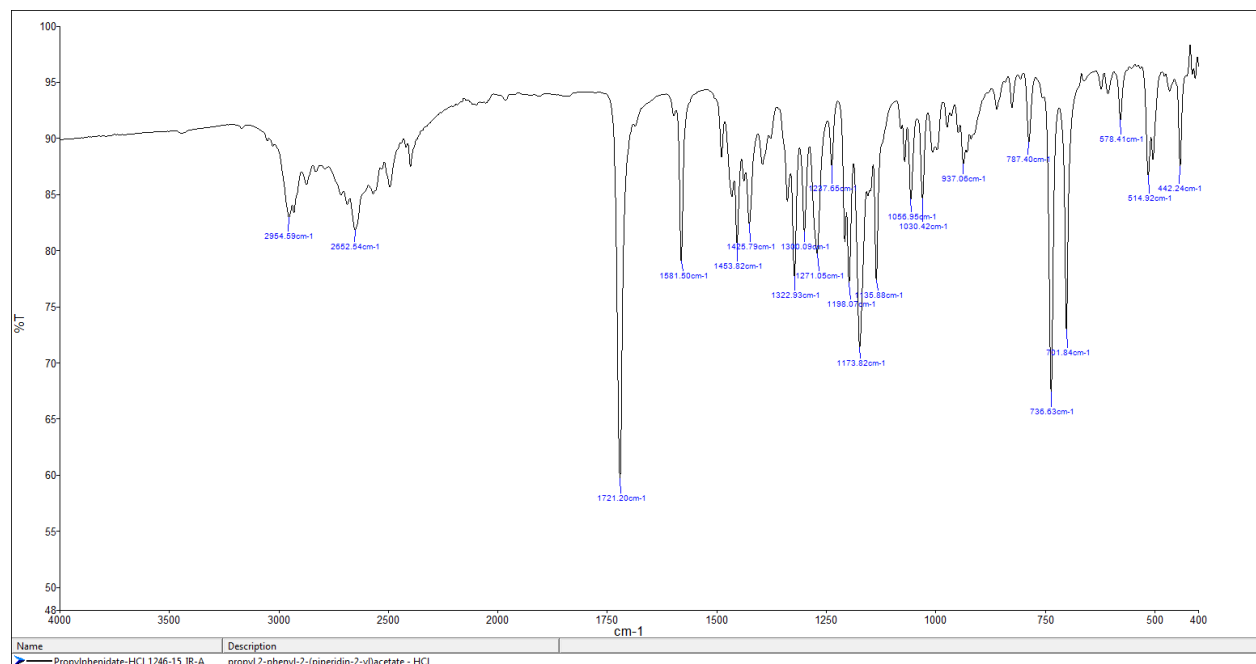
# ANALYTICAL RESULTS

MS (EI)

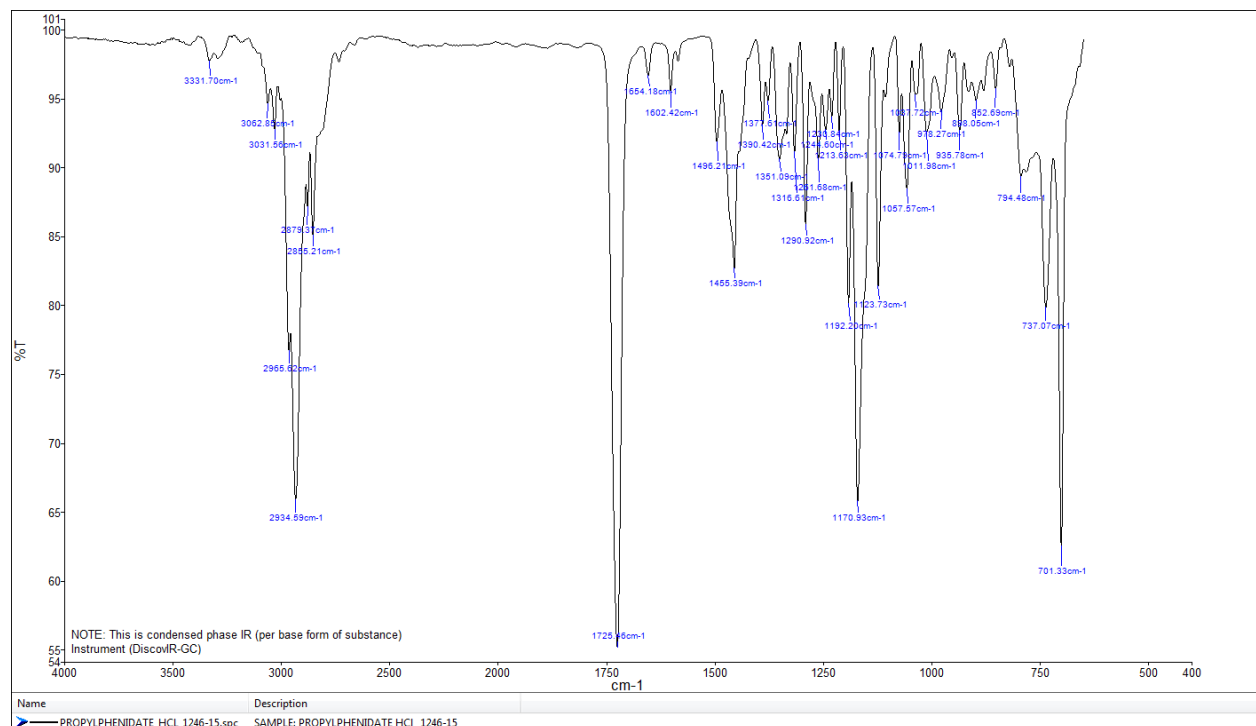
Abundance



## FTIR-ATR - direct measurement



## IR (condensed phase)



# Target Compound Screening Report

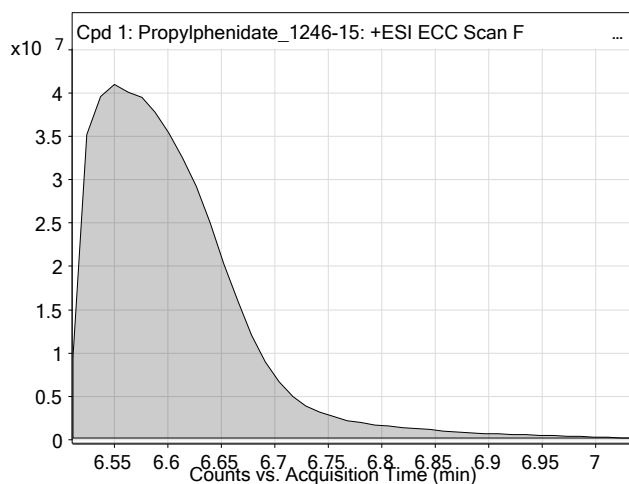
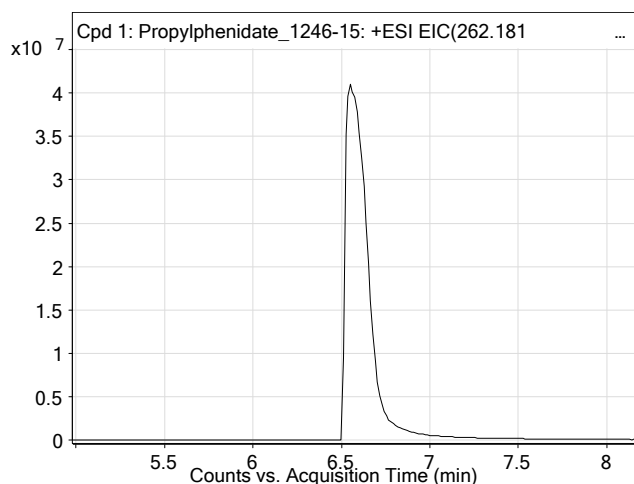
<b>Data File</b>	Propylphenidate_1246-15_TOF.d	<b>Sample Name</b>	Propylphenidate
<b>Sample Type</b>	Sample	<b>Position</b>	P2-E4
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	droge general-13-5-2015-XDB-C18-ESI-poz.m	<b>Acquired Time</b>	8/19/2015 1:12:27 PM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	Droge_Default.m
<b>Comment</b>	extract in MeOH		

## Compound Table

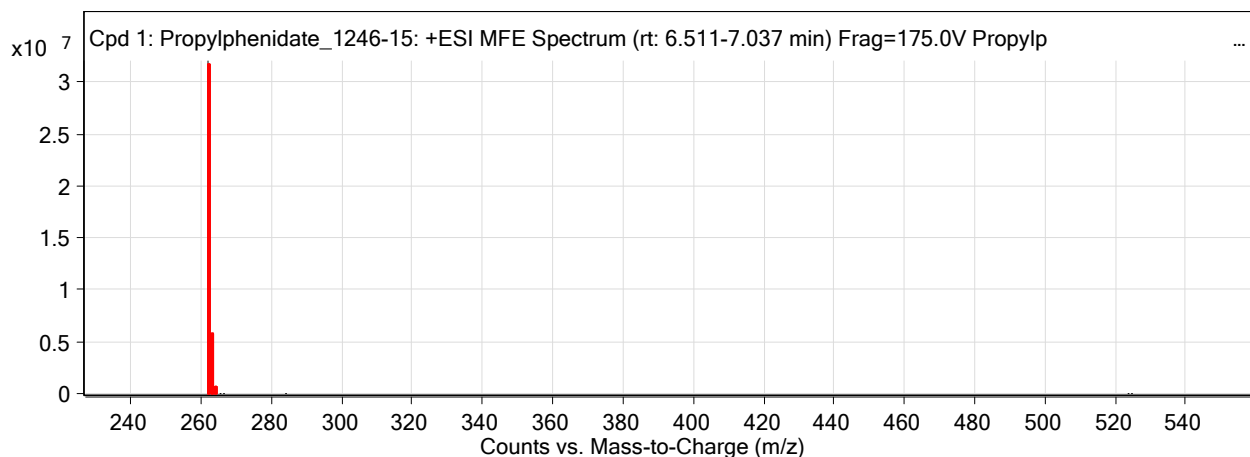
Label	Tgt Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Propylphenidate_1246-15	Propylphenidate_1246-15	C16 H23 N O2	6.576	261.173

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cmps Algorithm
Propylphenidate_1246-15	262.1803	6.576	261.173	6.576	C16 H23 N O2	261.1729	-0.56	Find by Molecular Feature

## Compound Chromatograms



## MFE MS Zoomed Spectrum



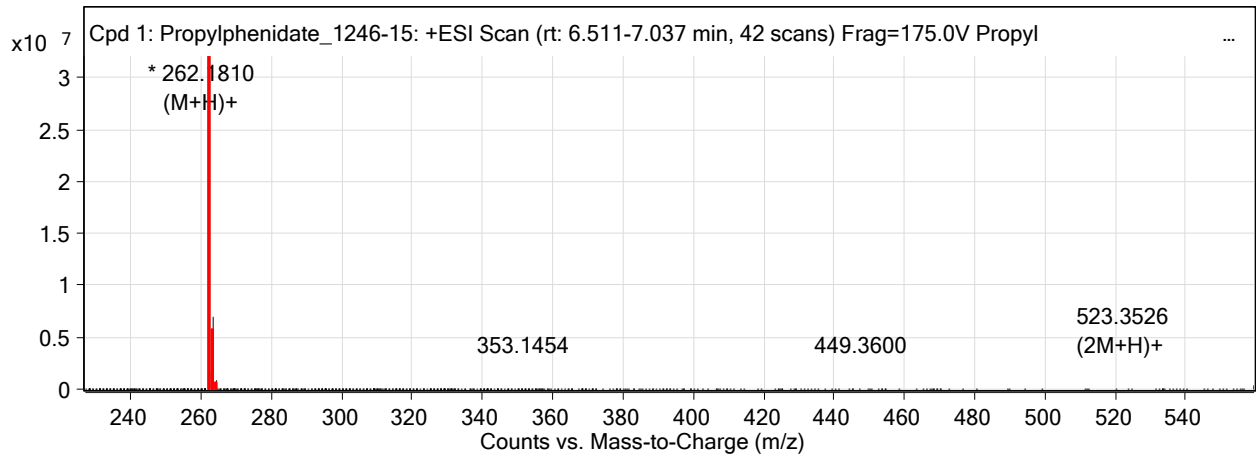
## MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
262.1803	1	32064764	C16 H23 N O2	(M+H)+
263.1837	1	5532229.61	C16 H23 N O2	(M+H)+
264.187	1	560878.45	C16 H23 N O2	(M+H)+
265.1894	1	43412.48	C16 H23 N O2	(M+H)+
266.1916	1	3991.95	C16 H23 N O2	(M+H)+
284.1624	1	15995.35	C16 H23 N O2	(M+Na)+
523.3531	1	26323.13	C16 H23 N O2	(2M+H)+

# Target Compound Screening Report

524.3563	1	9296.34	C16 H23 N O2 (2M+H)+
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MS Zoomed Spectrum

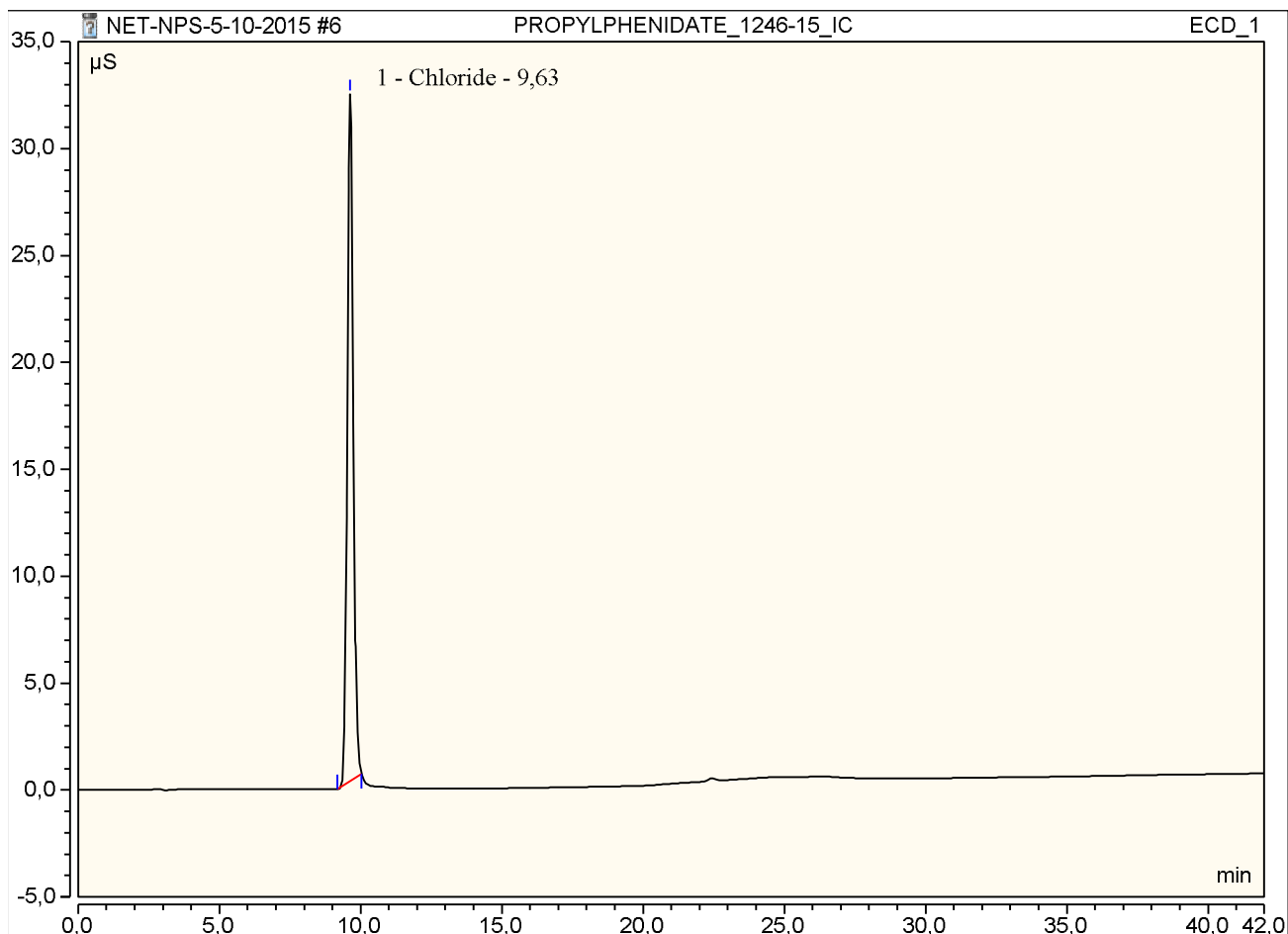


--- End Of Report ---

### Peak Integration Report

Sample Name:	PROPYLPHENIDATE_1246-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	05-okt-2015 / 17:55	Run Time:	41,99

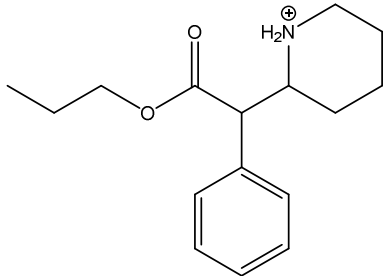
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height $\mu\text{S}$	Amount mg/L
1,00	9,63	Chloride	BMB	7,77	32,11	n.a.
		TOTAL:		7,77	32,11	0,00







## REPORT

Sample ID:	<b>1246-15</b>
Our notebook code:	P-1246-15
NMR sample preparation:	15 mg dissolved in 0.7 mL CDCl <sub>3</sub>
NMR experiments:	<sup>1</sup> H, <sup>13</sup> C, <sup>1</sup> H- <sup>1</sup> H <i>gs</i> -COSY, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HSQC, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HMBC, <sup>1</sup> H- <sup>14</sup> N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	2-(2-oxo-1-phenyl-2-propoxyethyl)piperidin-1-ium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR.
Supporting information:	Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	October 19, 2015

P-1246-15

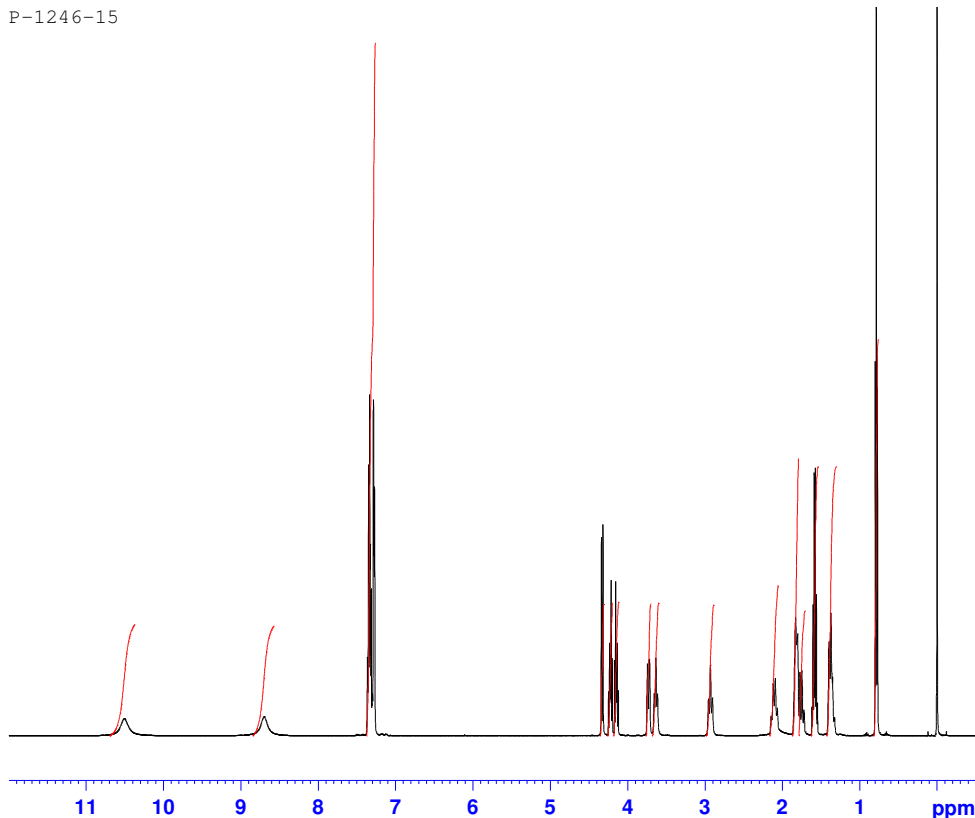


Current Data Parameters  
 NAME P-1246-15  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20151018  
 Time 0.54  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 10330.578 Hz  
 FIDRES 0.157632 Hz  
 AQ 3.1719923 sec  
 RG 71.8  
 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 500.1300040 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



P-1246-15



Current Data Parameters  
 NAME P-1246-15  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20151018  
 Time 3.34  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 4096  
 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 125.7577895 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

