



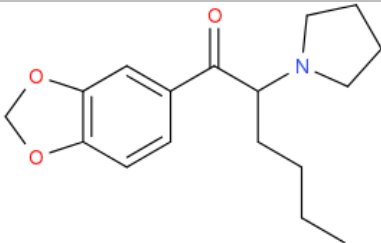
ANALYTICAL REPORT

MDPHP (C17H23NO3)

1-(1,3-benzodioxol-5-yl)-2-pyrrolidin-1-yl-hexan-1-one

Remark – other NPS detected:

Sample ID:	1245-15
Sample description:	powder - brown dark
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-(1,3-benzodioxol-5-yl)-2-pyrrolidin-1-yl-hexan-1-one
Other names	
Formula (per base form)	C17H23NO3
M _w (g/mol)	289.37
Salt form	HCl
StdInChIKey	OBLFRFGUZZRECT-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	
Add.info (purity..)	25% of an unknown organic impurity 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)

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⁻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 °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⁻¹ .

Supporting information

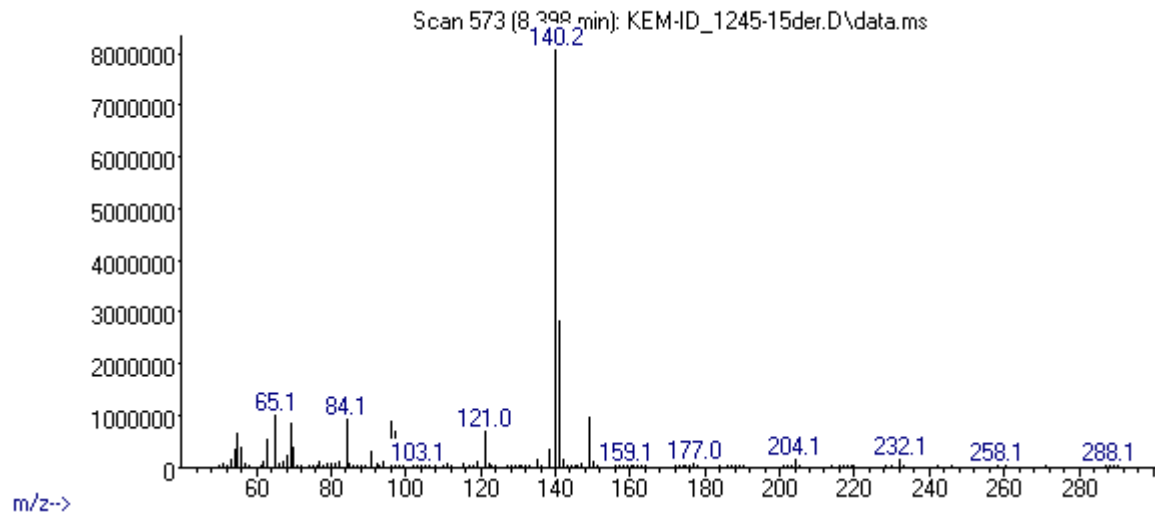
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8.4 BP(1): 140; BP(2): 141, BP(3) :149,
HPLC-TOF	+	Exact mass (theoretical): 289.1687; measured value Δppm:-0.36; formula:C17H23NO3
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
NMR	+	
validation		
other		

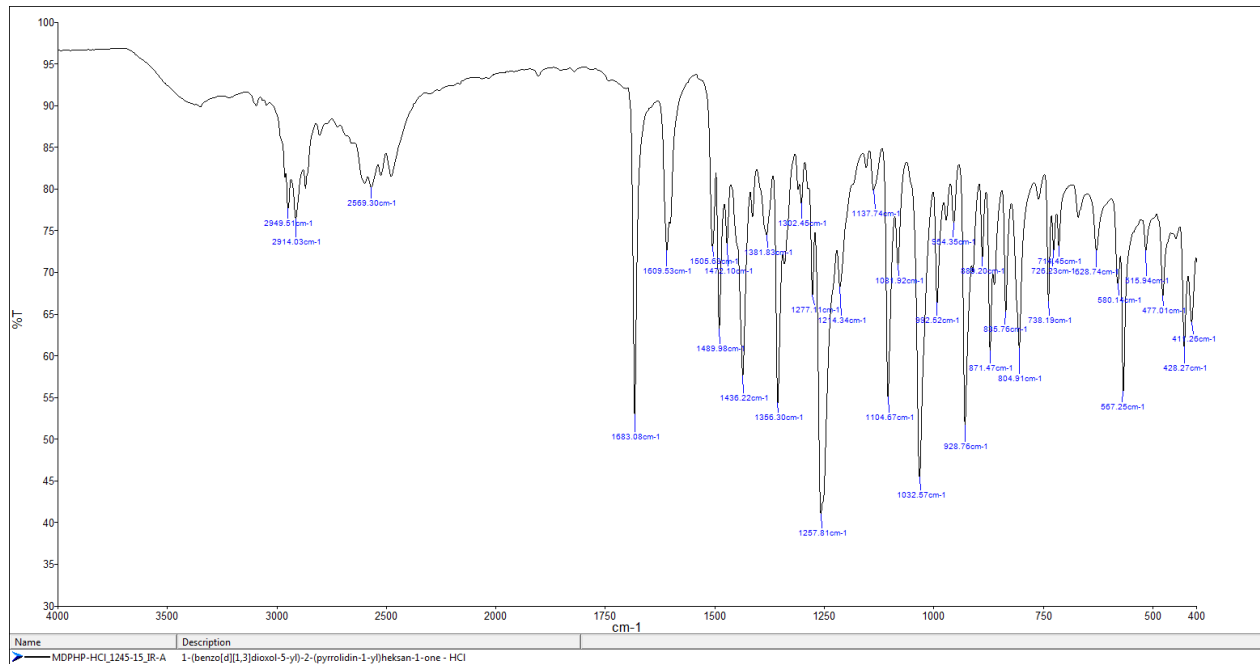
ANALYTICAL RESULTS

MS (EI)

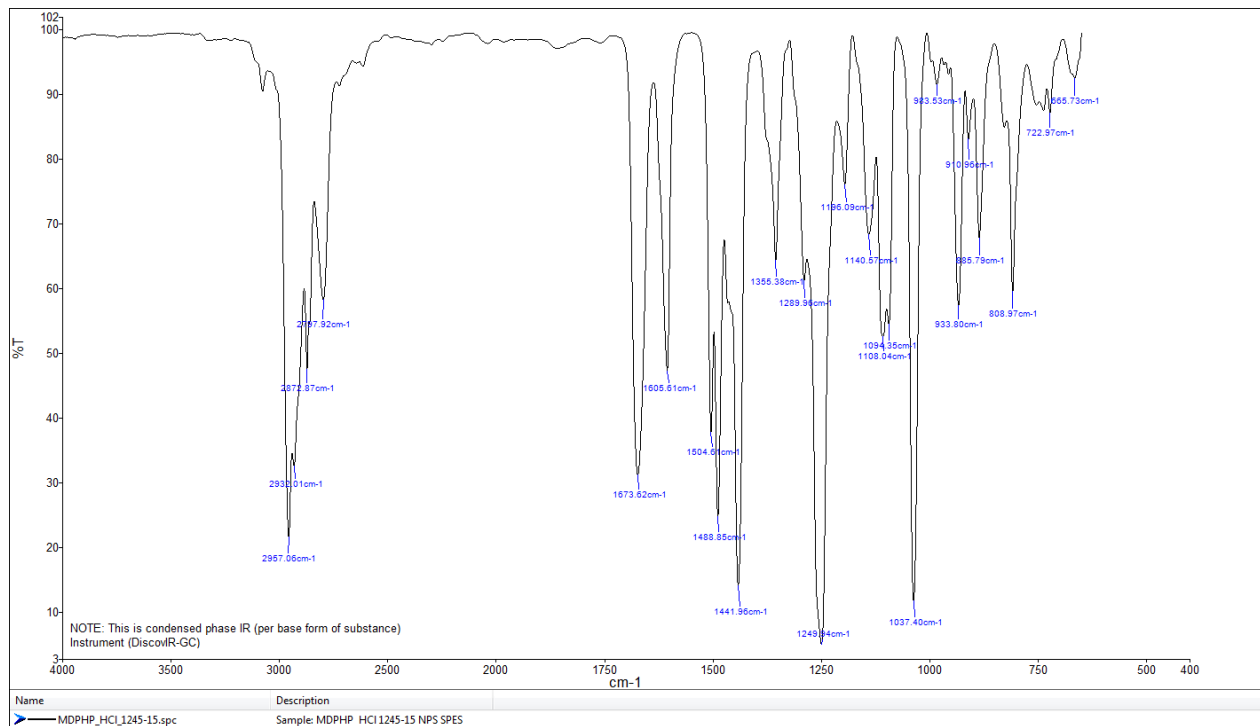
Abundance



FTIR-ATR - direct measurement



IR (condensed phase)



Target Compound Screening Report

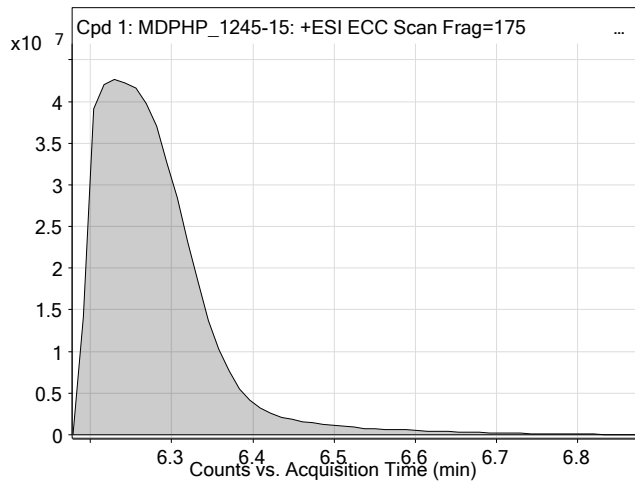
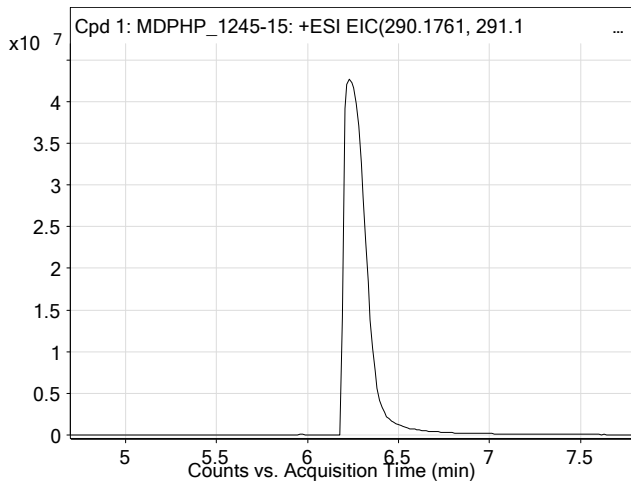
Data File	MDPHP_1245-15_TOF.d	Sample Name	MDPHP
Sample Type	Sample	Position	P2-E3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	Acquired Time	8/19/2015 12:57:54 PM
IRM Calibration Status	Success	DA Method	Droge_Default.m
Comment	extract in MeOH		

Compound Table

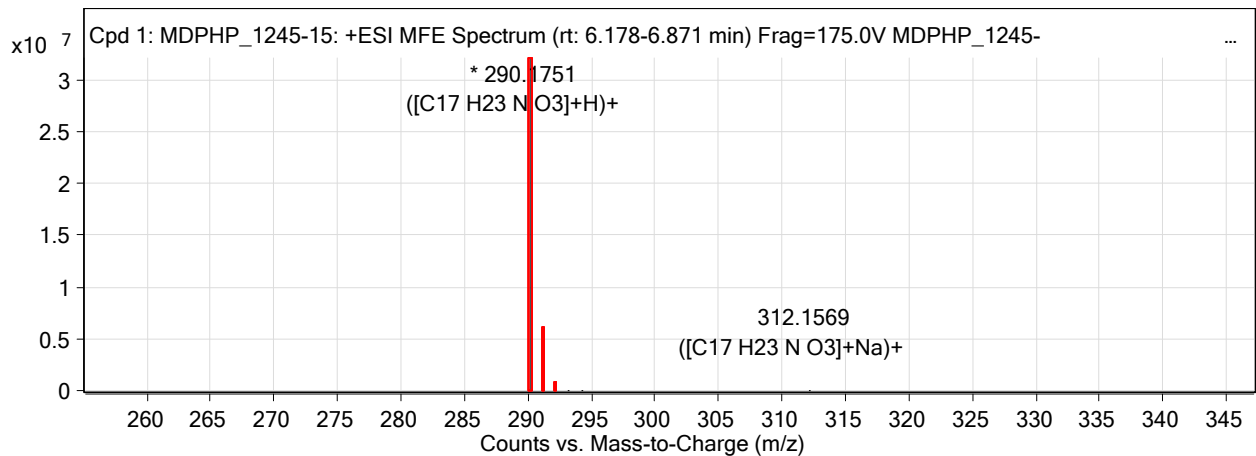
Label	Tgt Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: MDPHP_1245-15	MDPHP_1245-15	C17 H23 N O3	6.249	289.1679

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cmps Algorithm
MDPHP_1245-15	290.1751	6.249	289.1679	6.249	C17 H23 N O3	289.1678	-0.36	Find by Molecular Feature

Compound Chromatograms



MFE MS Zoomed Spectrum

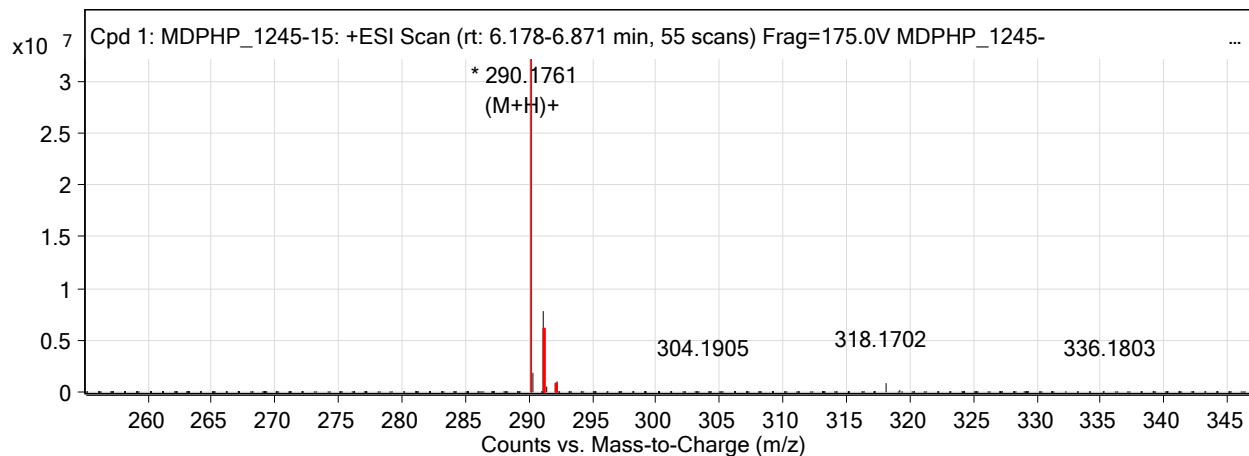


MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
290.1751	1	32135690	C17 H23 N O3	(M+H)+
291.1785	1	6199224.16	C17 H23 N O3	(M+H)+
292.1818	1	674417.23	C17 H23 N O3	(M+H)+
293.1843	1	64502.48	C17 H23 N O3	(M+H)+
294.1862	1	5394.03	C17 H23 N O3	(M+H)+
312.1569	1	9421.59	C17 H23 N O3	(M+Na)+

Target Compound Screening Report

MS Zoomed Spectrum

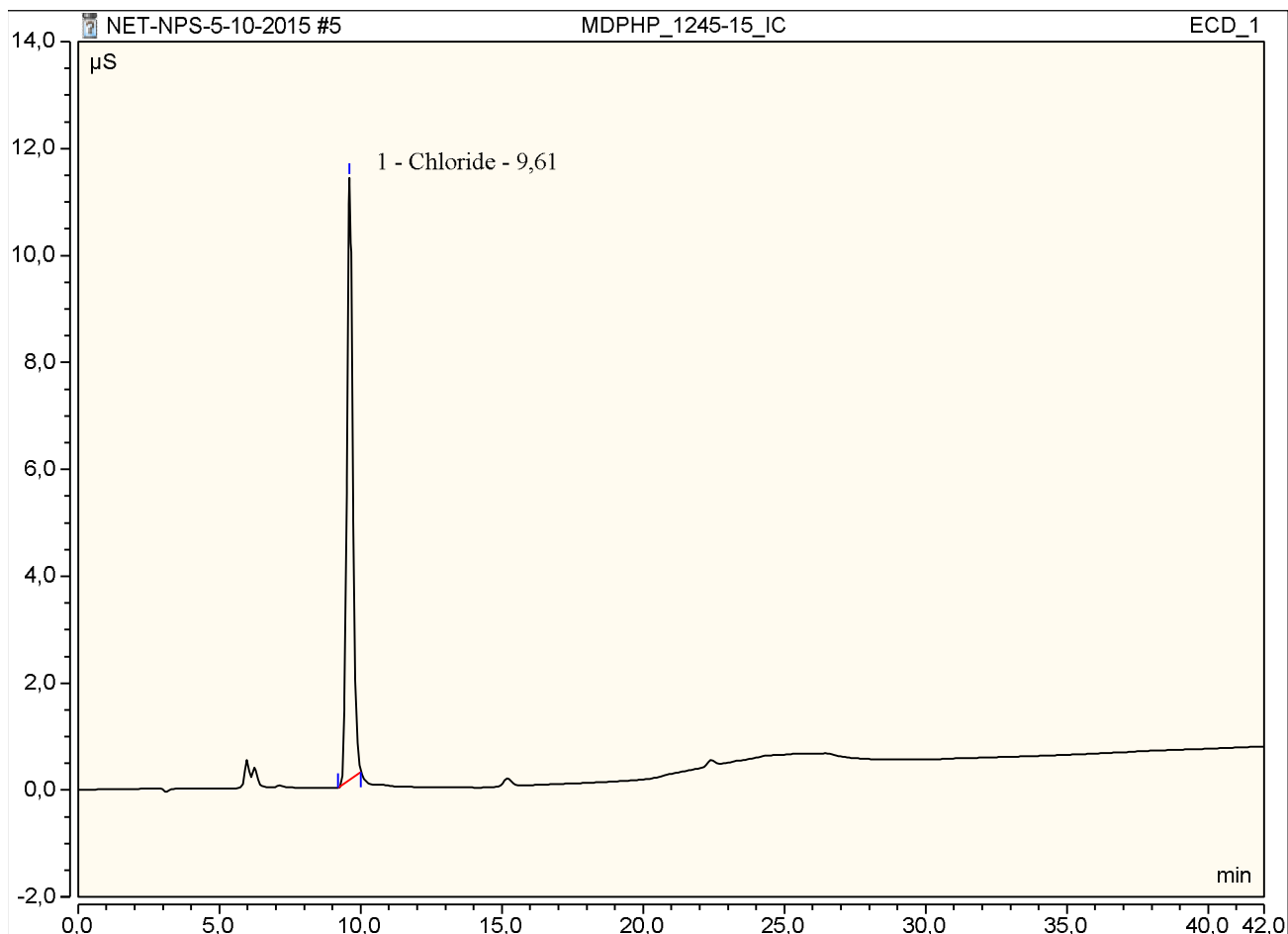


--- End Of Report ---

Peak Integration Report

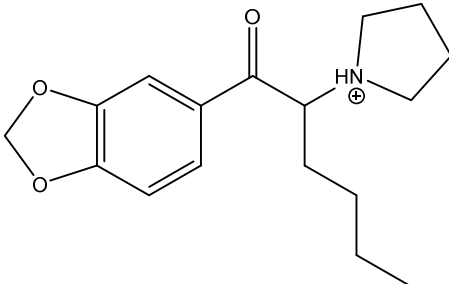
Sample Name:	MDPHP_1245-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	05-okt-2015 / 17:06	Run Time:	41,98

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}^*\text{min}$	Height μS	Amount mg/L
1,00	9,61	Chloride	BMB	2,72	11,27	n.a.
		TOTAL:		2,72	11,27	0,00





REPORT

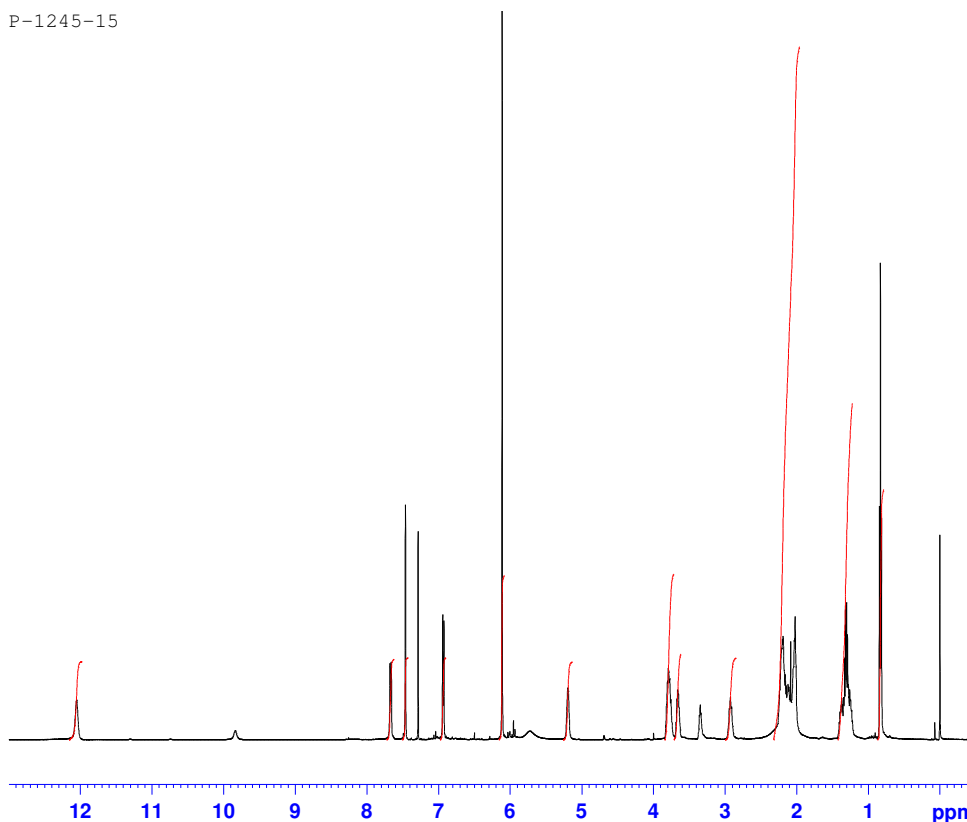
Sample ID:	1245-15
Our notebook code:	P-1245-15
NMR sample preparation:	15 mg dissolved in 0.7 mL CDCl ₃
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC.
Proposed structure with chemical name:	 <p>1-(1-(benzo[d][1,3]dioxol-5-yl)-1-oxohexan-2-yl)pyrrolidin-1-ium</p>
Comments:	<ul style="list-style-type: none"> - Structure elucidation based on 1D and 2D NMR spectra - Compound is not pure by NMR, containing approx. 25% of an unknown organic impurity (¹H NMR signals at 9.83, 5.72, 3.35 and 2.10 (hidden) and ¹³C NMR signals at 44.91 and 24.47). The same impurity is also observed in 1251-51, though in a lesser amount.
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	October 2, 2015

P-1245-15



Current Data Parameters
 NAME p-1245-15
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150914
 Time 20.57
 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 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

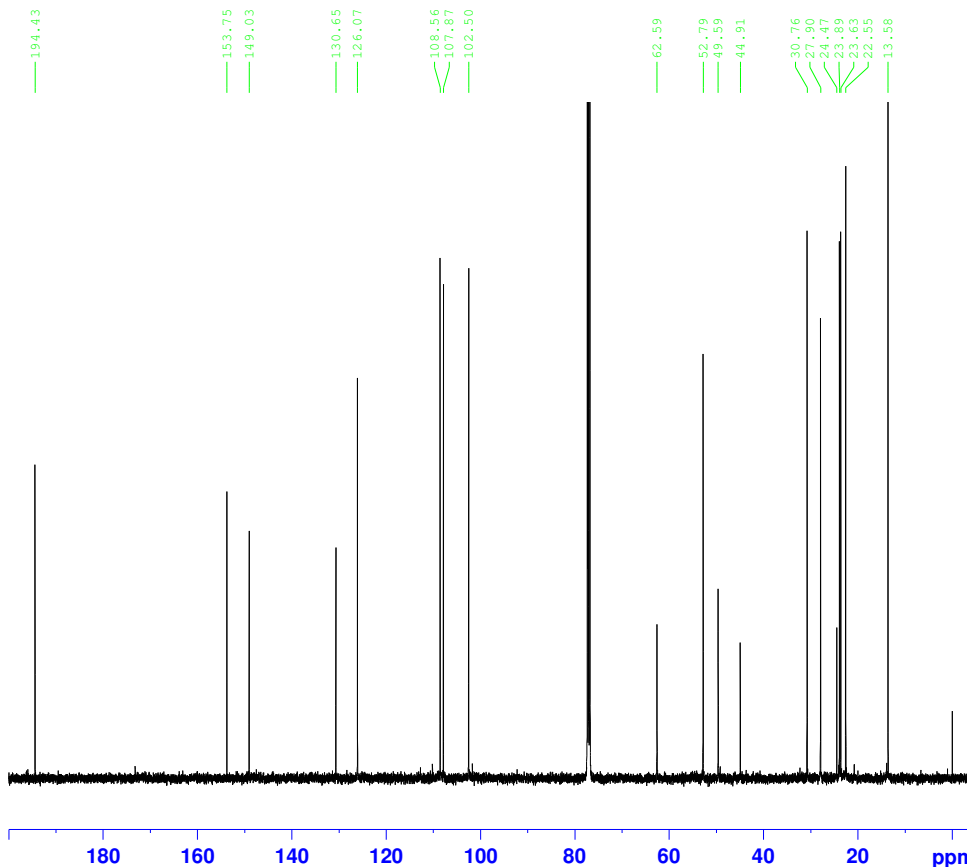
F2 - Processing parameters
 SI 65536
 SF 500.1300017 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

P-1245-15



Current Data Parameters
 NAME p-1245-15
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150915
 Time 0.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 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 125.7577888 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40