



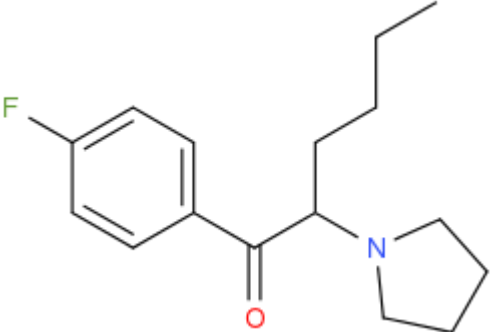
ANALYTICAL REPORT¹

4F-PHP (C₁₆H₂₂FNO)

1-(4-fluorophenyl)-2-(pyrrolidin-1-yl)hexan-1-one

Remark – other NPS detected: **unidentified compounds**

Sample ID:	1302-15
Sample description:	powder - ocker
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	10/2/2015
Date of entry (M/D/Y) into NFL database:	11/6/2015
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	1-(4-fluorophenyl)-2-(pyrrolidin-1-yl)hexan-1-one
Other names	
Formula (per base form)	C ₁₆ H ₂₂ FNO
M _w (g/mol)	263,4
Salt form	HCl
StdInChIKey	BCJXLSGKMNRKO-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	unidentified compounds
Add.info (purity..)	2 peaks by GC-MS, HPLC-TOF, unpure compound, NMR compound is not pure

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)
08/12/2016	Compound class changed

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 quadrupole 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 quadrupole 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.

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

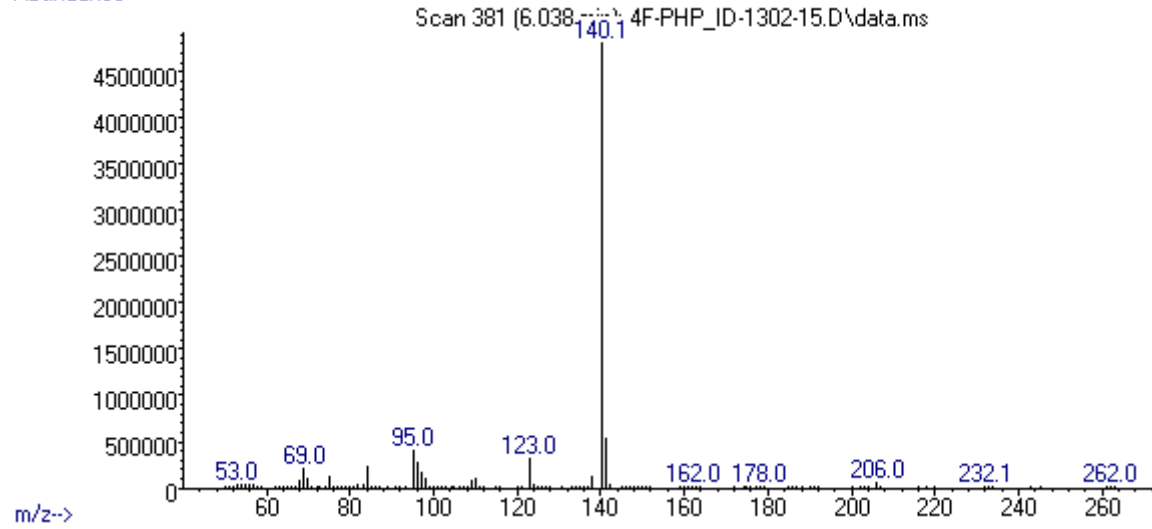
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 6,04 BP(1): 140; BP(2): 141, BP(3) :95,
HPLC-TOF	+	Exact mass (theoretical): 263,1685; measured value Δppm:-0,91; formula:C16H22FNO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

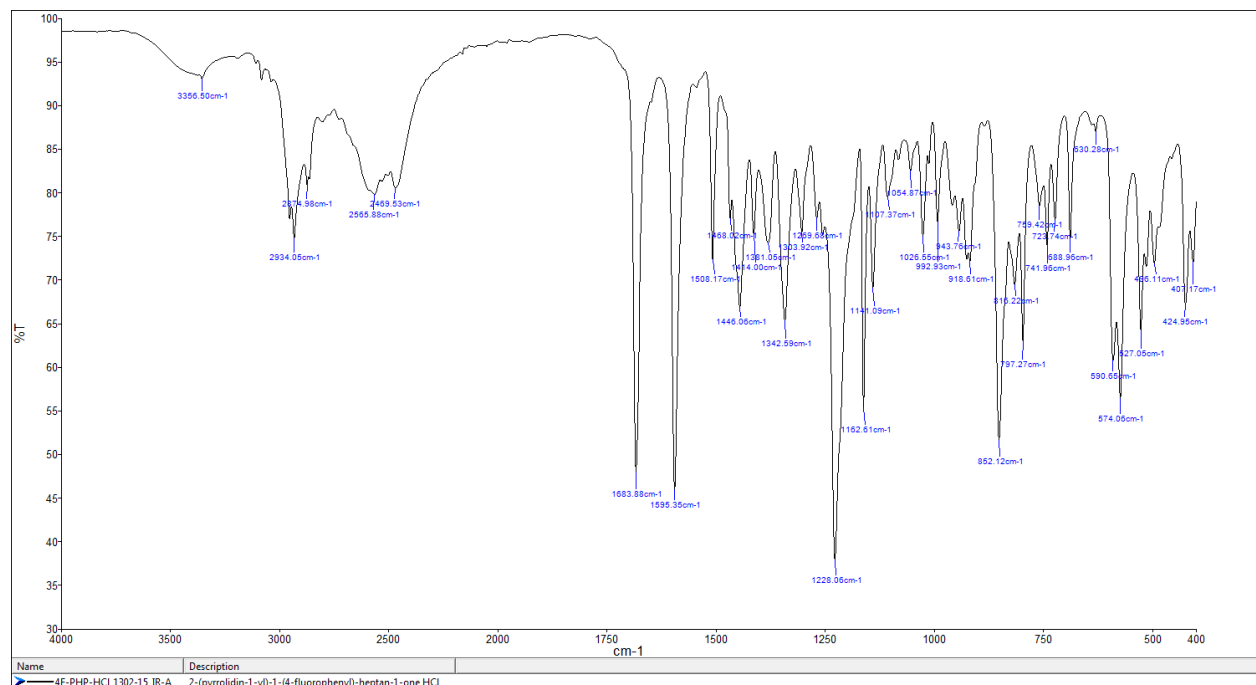
ANALYTICAL RESULTS

MS (EI)

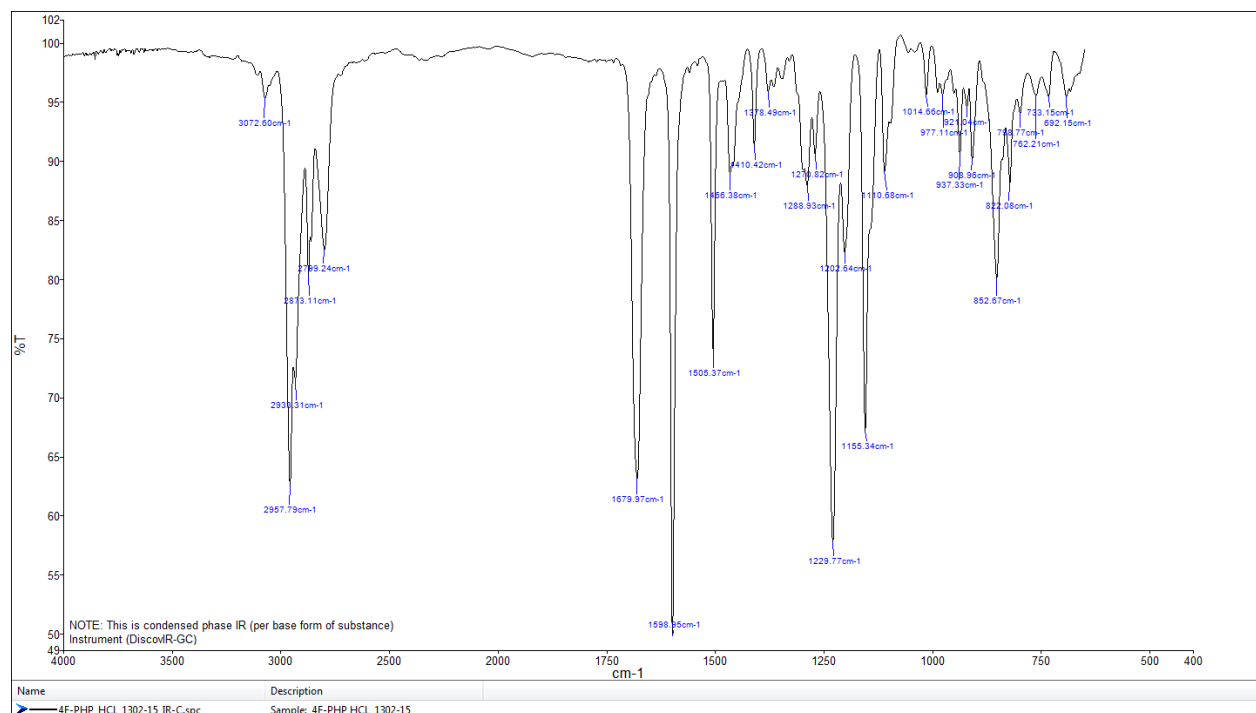
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

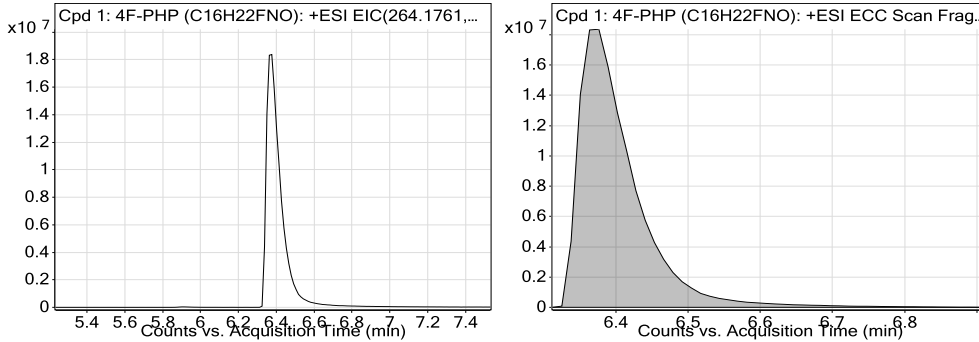
Data File	4-PHP_1302-15_TOF.d	Sample Name	4F-PHP
Sample Type	Sample	Position	P1-B5
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-28052015-XDB-C18-ESI-poz.m	Acquired Time	10/23/2015 1:08:37 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

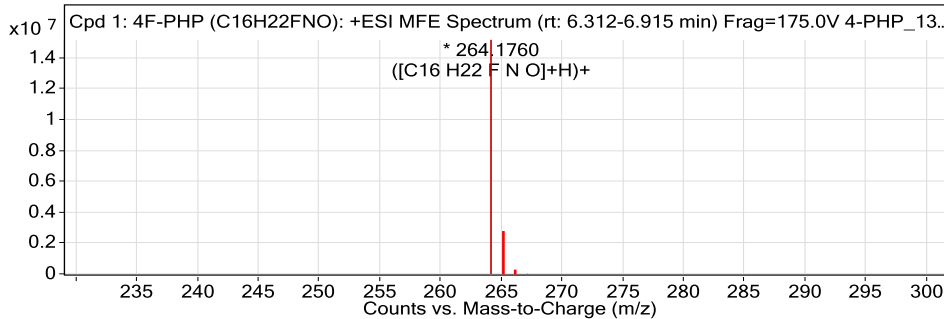
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 1: 4F-PHP (C16H22FNO)	4F-PHP (C16H22FNO)	6.378	263.1688

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4F-PHP (C16H22FNO)	264.176	6.378	263.1688	6.4	C16 H22 F N O	263.1685	-0.91

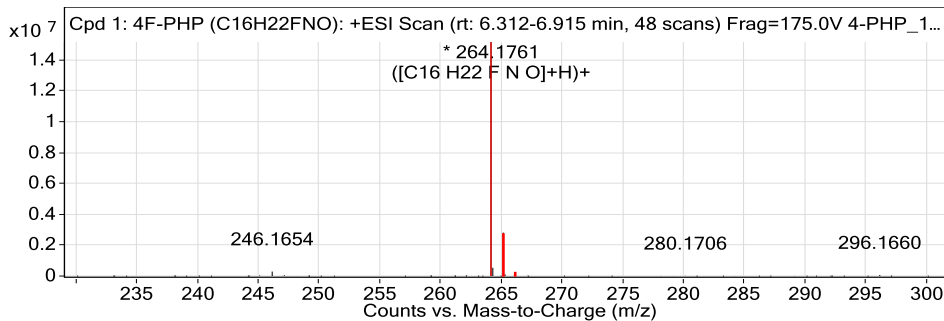
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
264.176	1	15148192	C16 H22 F N O	(M+H)+
265.1794	1	2612805.96	C16 H22 F N O	(M+H)+
266.1827	1	234396.78	C16 H22 F N O	(M+H)+
267.1851	1	17004.21	C16 H22 F N O	(M+H)+

--- End Of Report ---



REPORT

Sample ID:	1302-15
Our notebook code:	P-1302-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C .
Proposed structure:	
Chemical name:	1-(1-(4-fluorophenyl)-1-oxohexan-2-yl)pyrrolidin-1-ium
Comments:	<ul style="list-style-type: none"> - Structure elucidation based on 1D NMR spectra - Sample is not pure as evident by NMR: major impurity (^{13}C NMR signals 132, 115, 44.7 and 24.3 and ^1H signals at 8.10, 7.15, 3.90, 1.83) as well as signals corresponding to other compounds present in the sample.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 8, 2015

P-1302-15

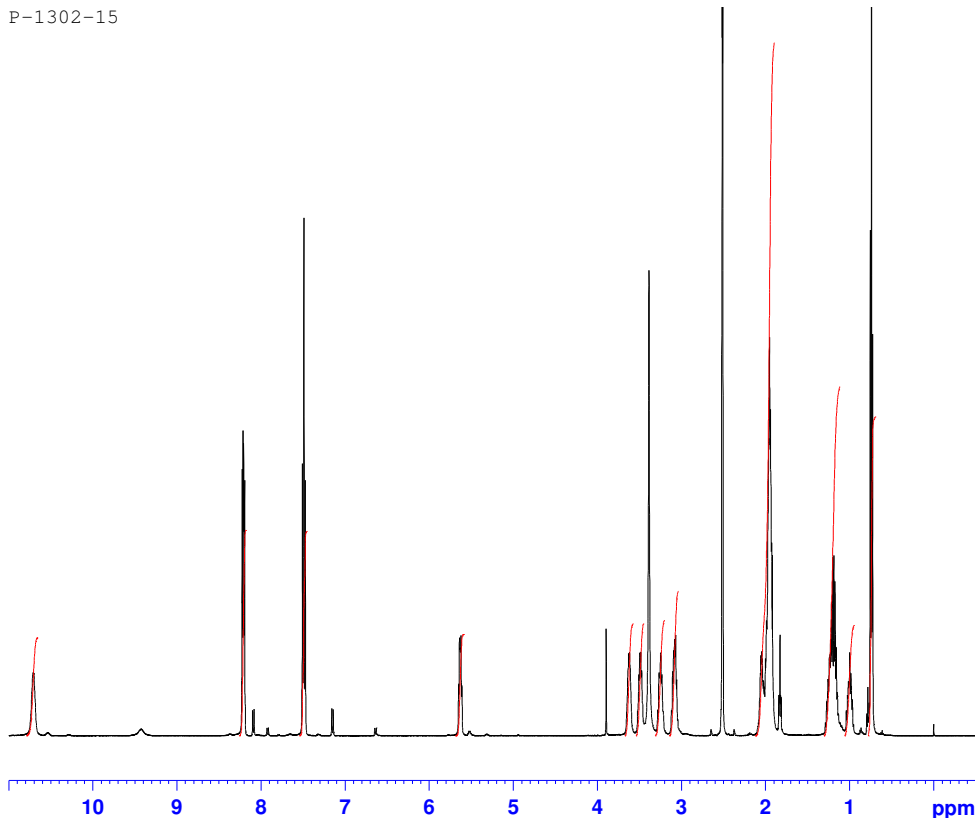


Current Data Parameters
 NAME p-1302-15
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151204
 Time 4.04
 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 3.2768500 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 8.90 usec
 PLW1 26.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1299977 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



P-1302-15



Current Data Parameters
 NAME P-1302-15
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151204
 Time 5.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 2048
 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 9.00 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 0.32179001 W
 PLW13 0.16186000 W

F2 - Processing parameters
 SI 32768
 SF 125.7577885 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

