



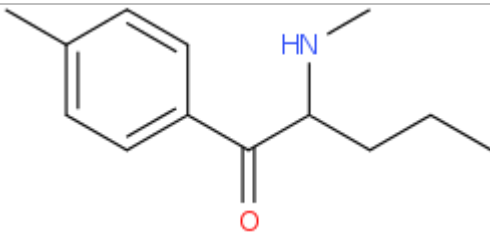
## ANALYTICAL REPORT<sup>1,2</sup>

### 4-MPD (C<sub>13</sub>H<sub>19</sub>NO)

#### 2-(methylamino)-1-(4-methylphenyl)pentan-1-one

Remark – other NPS detected: **none**

Sample ID:	1695-16
Sample description:	powder - white
Sample type:	collected /FSC Ivan Vučetić, Croatia
Date of sample receipt (M/D/Y):	9/21/2016
Date of entry (M/D/Y) into NFL database:	9/30/2016
Report updates (if any) 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>3</sup> (base form)	
Systematic name	2-(methylamino)-1-(4-methylphenyl)pentan-1-one
Other names	4-Methylpentedrone; 1-(4-Methylphenyl)-2-methylamino-pentan-1-one; 2-(methylamino)-1-(p-tolyl)pentan-1-one
Formula (per base form)	C <sub>13</sub> H <sub>19</sub> NO
M <sub>w</sub> (g/mol)	205,3
Salt form/anions detected	chloride
StdInChIKey	AKVKBEDACKJNPO-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	none
Add.info (purity..)	

<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> Acknowledgement: Sample (NMR confirmed) was kindly provided by the FSI Ivan Vučetić, Croatia. NMR is not enclosed.

<sup>3</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 (9.258 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 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then 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 6.1 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 (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 (N<sub>2</sub>) 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 (30 until 6 min.) to 550 (300) amu.

IR (condensed (solid) 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

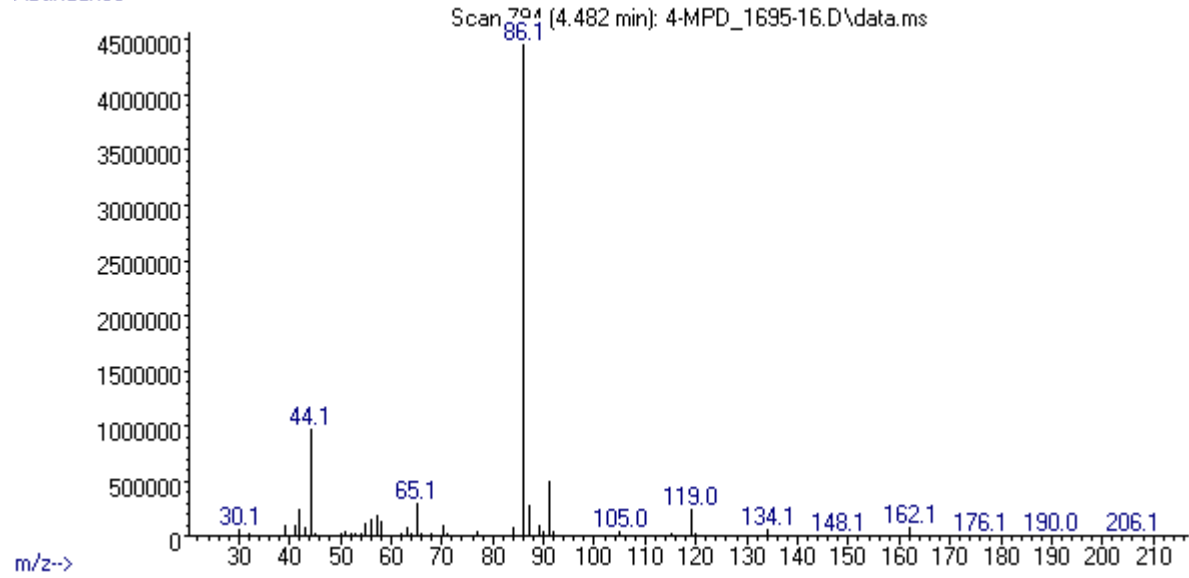
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	partially
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,48 BP(1): 86; BP(2): 44,BP(3) :91,
HPLC-TOF	+	Exact mass (theoretical): 205,1467; measured value Δppm:-0,7; formula:C13H19NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	-	
validation		
other		

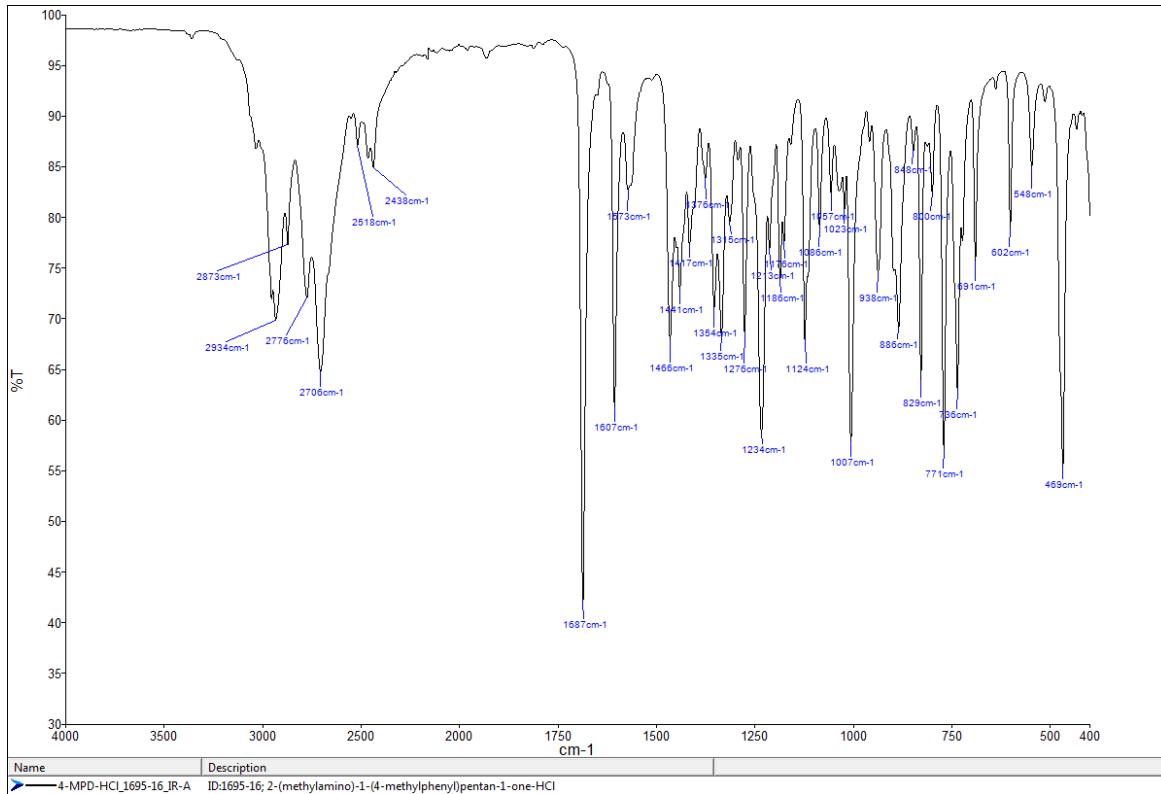
# ANALYTICAL RESULTS

MS (EI)

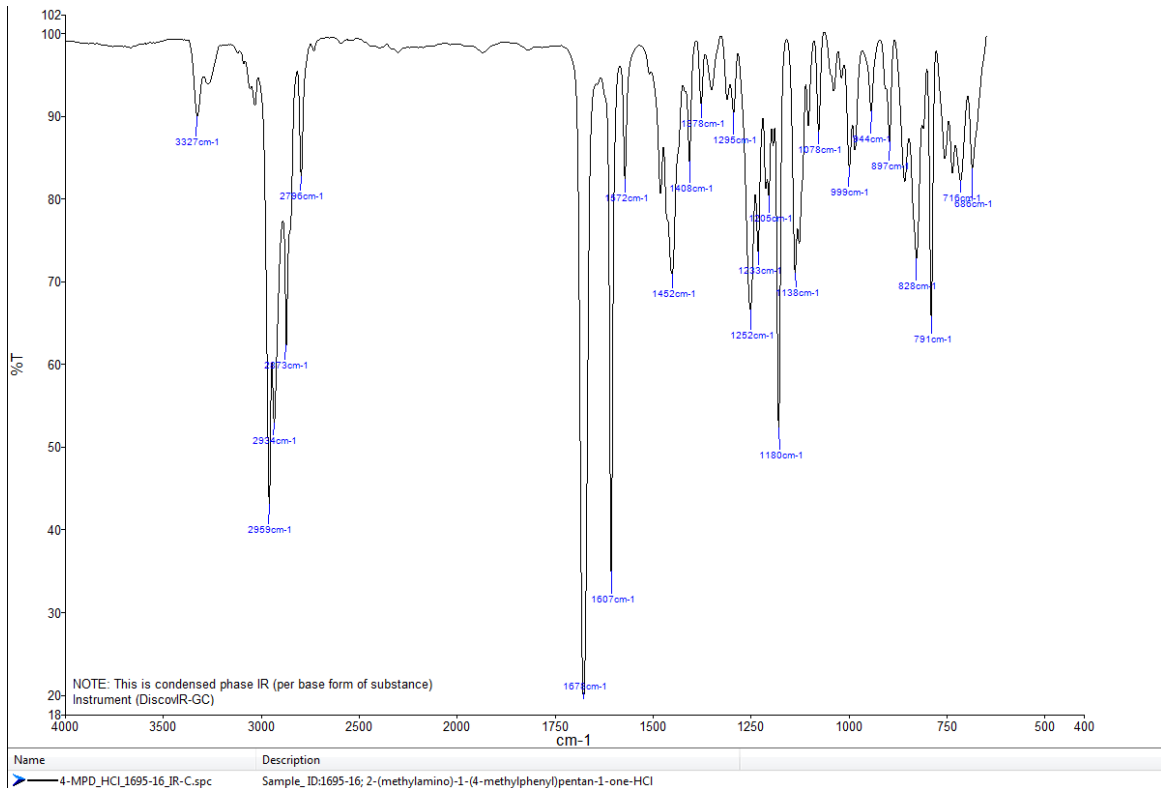
Abundance



### FTIR-ATR - direct measurement (sample as received)



### IR (condensed phase – after chromatographic separation)





# TOF REPORT

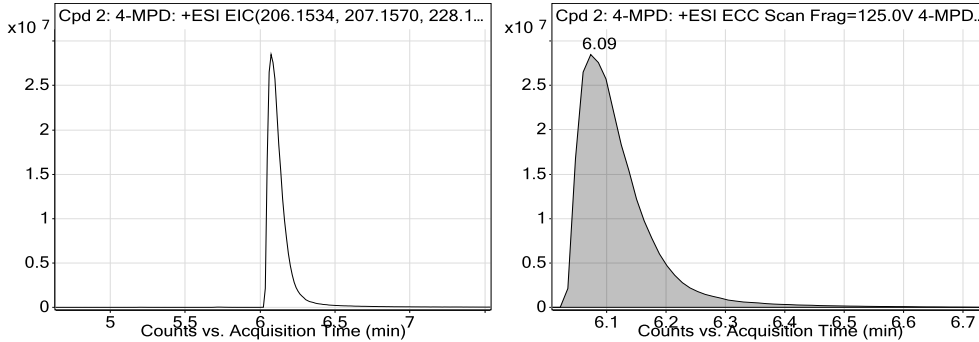
<b>Data File</b>	4-MPD_1695-16_TOF.d	<b>Sample Name</b>	ID_1695-16
<b>Sample Type</b>	Sample	<b>Position</b>	P1-D3
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	general-24_08_2016-XDB-C18-ESI-poz-soft.m	<b>Acquired Time</b>	9/26/2016 11:39:22 AM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	Drugs_NFL.m
<b>Comment</b>	extract in MeOH		

## Compound Table

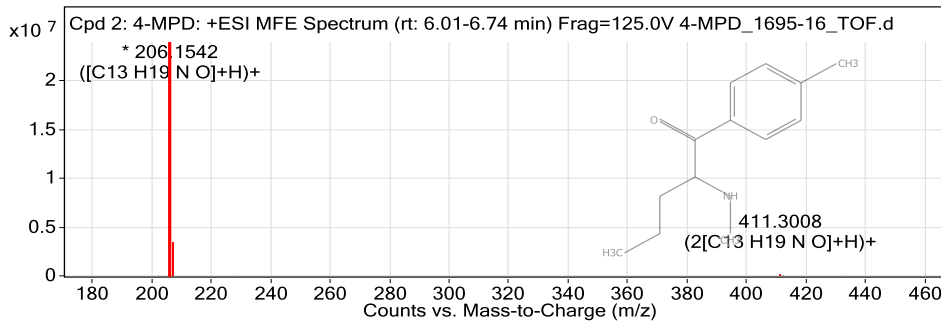
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: 4-MPD	4-MPD	C13 H19 N O	6.09	205.1468

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4-MPD	206.1542	6.09	205.1468	6.09	C13 H19 N O	205.1467	-0.7

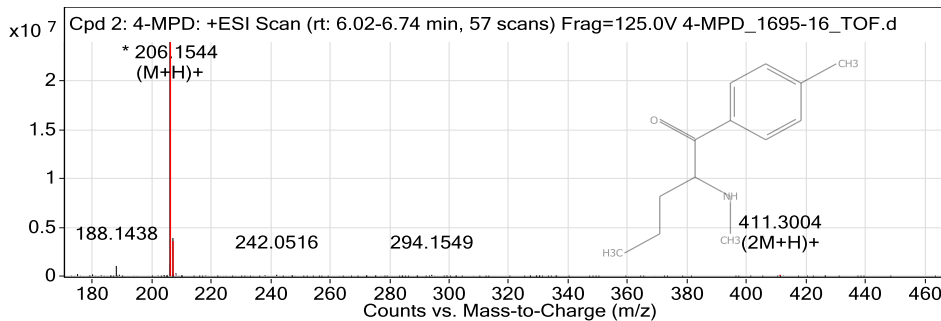
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
206.1542	1	24004940	C13 H19 N O	(M+H)+
207.1575	1	3397597.45	C13 H19 N O	(M+H)+
228.136	1	10271.21	C13 H19 N O	(M+Na)+
411.3008	1	174580.89	C13 H19 N O	(2M+H)+
412.3039	1	49646.24	C13 H19 N O	(2M+H)+
413.307	1	8172.79	C13 H19 N O	(2M+H)+
433.2821	1	9684.02	C13 H19 N O	(2M+Na)+
434.2858	1	2885.97	C13 H19 N O	(2M+Na)+

--- End Of Report ---

### Peak Integration Report

Sample Name:	4-MPD_1695-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	26-sep-2016 / 12:35	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height $\mu\text{S}$	Amount mg/L
1,00	7,55	Chloride	BMB	13,93	66,34	n.a.
TOTAL:				13,93	66,34	0,00

