



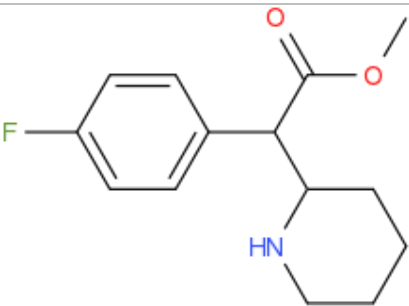
ANALYTICAL REPORT¹

4F-MPH (C₁₄H₁₈FNO₂)

methyl 2-(4-fluorophenyl)-2-(2-piperidyl)acetate

Remark – other NPS detected: **none**

Sample ID:	1352-15
Sample description:	crystalinic - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	11/13/2015
Date of entry (M/D/Y) into NFL database:	12/16/2015
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	methyl 2-(4-fluorophenyl)-2-(2-piperidyl)acetate
Other names	4-fluoromethylphenidate
Formula (per base form)	C ₁₄ H ₁₈ FNO ₂
M _w (g/mol)	251,3
Salt form	chloride
StdInChIKey	XISBAJBPDVRSFG-UHFFFAOYSA-N
Compound Class	Piperidines & pyrrolidines
Other NPS detected	none
Add.info (purity..)	2 peaks (isomers)

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.

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

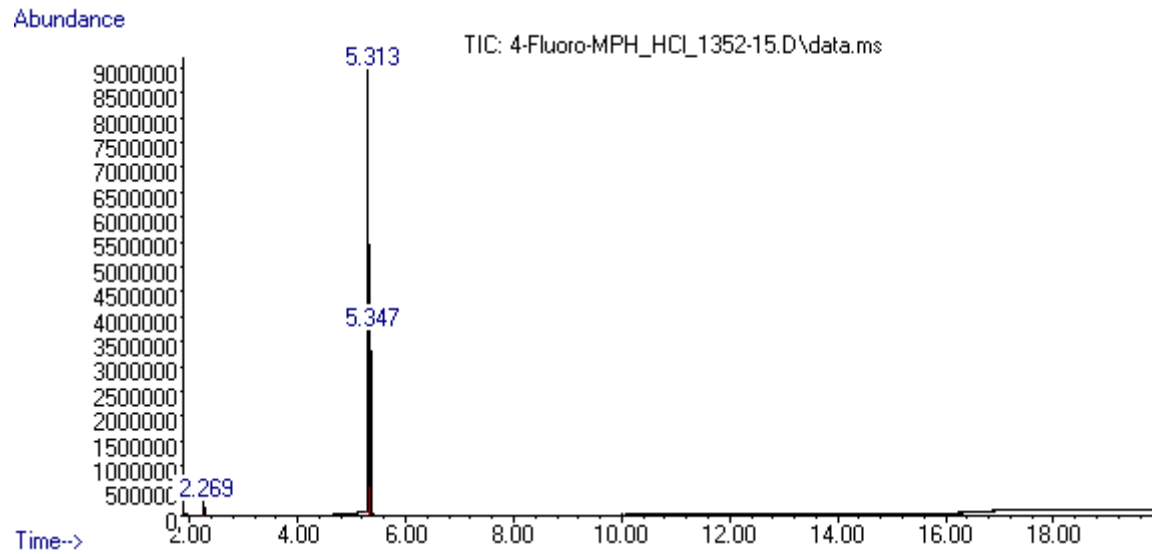
Supporting information

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

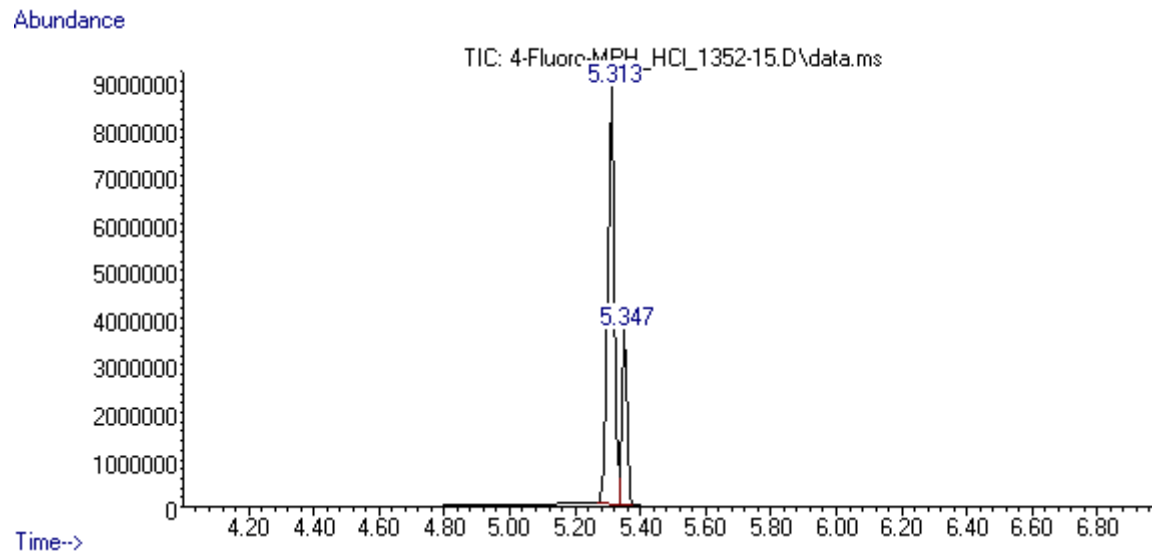
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 5,3 BP(1): 84; BP(2): 109,BP(3) :56,
HPLC-TOF	+	Exact mass (theoretical): 251,1322; measured value Δppm:-1,23; formula:C14H18FNO2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form		
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

GC-MS-data



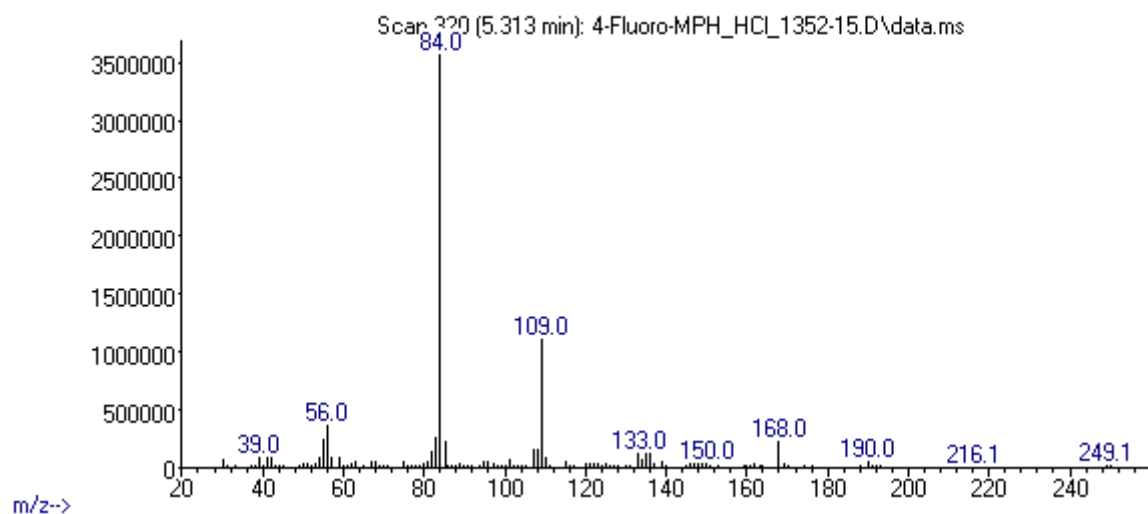
Chromatogram



Chromatogram-zoomed

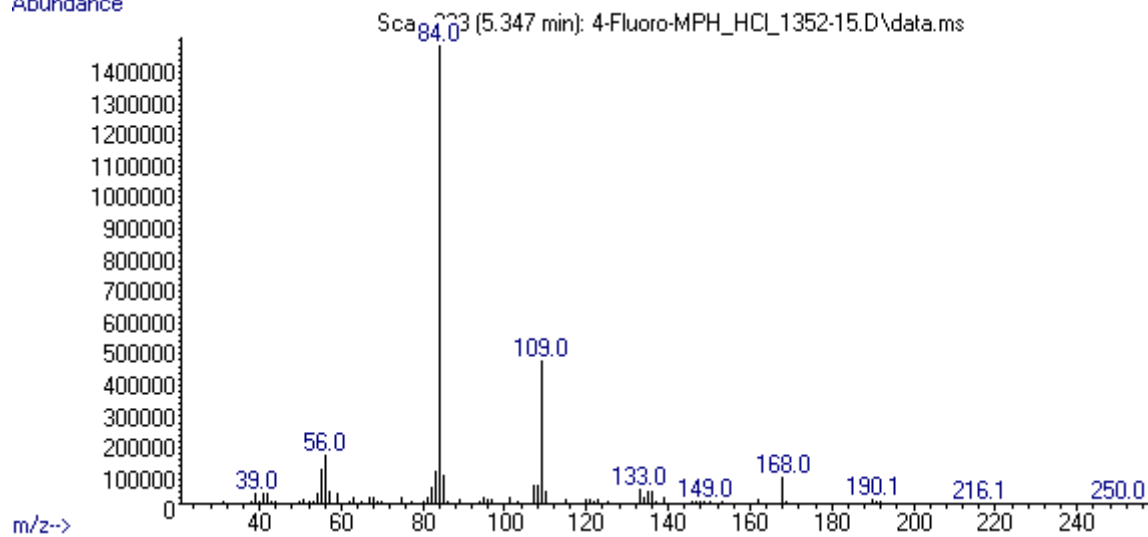
MS (EI)

Abundance



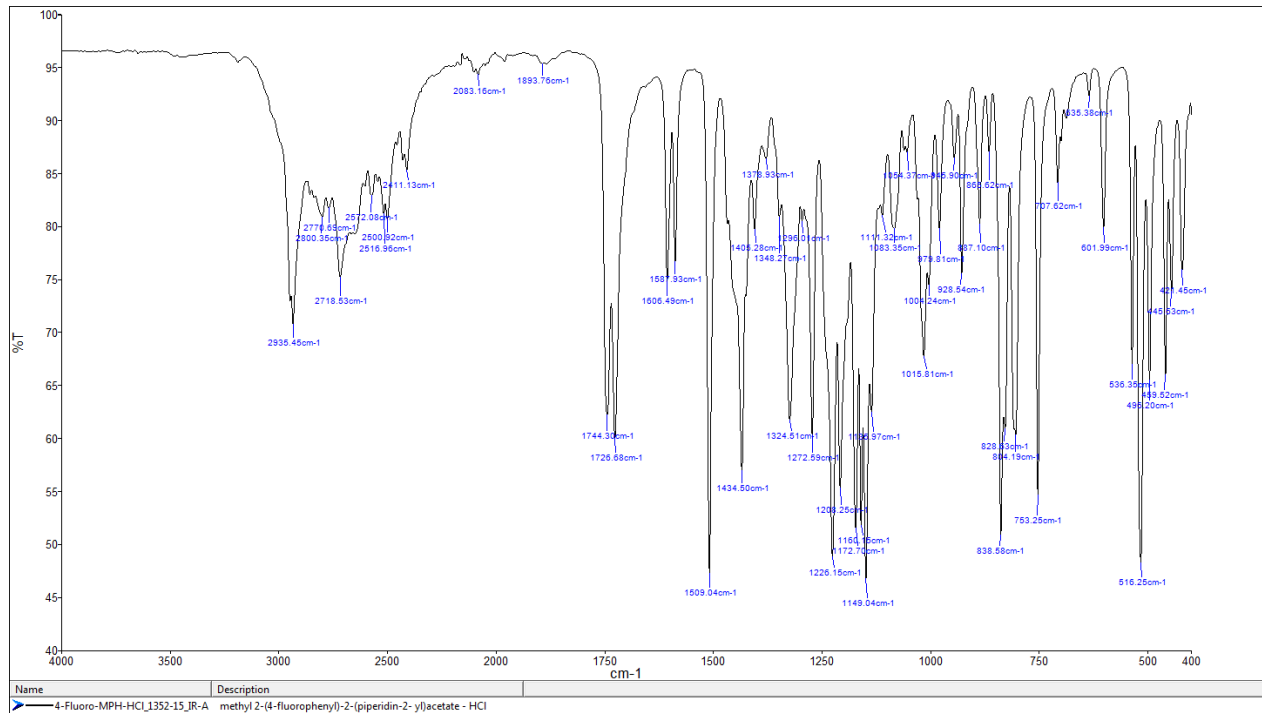
MS(RT=5.31)

Abundance



MS(RT=5.35)

FTIR-ATR - direct measurement (sample as received)



TOF REPORT

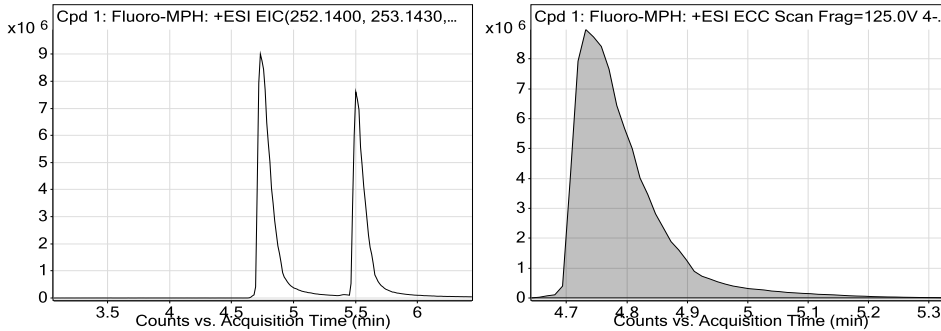
Data File	4-Fluoro-MPH_1352-15_TOF.d	Sample Name	ID_1352-15
Sample Type	Sample	Position	P1-A7
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-17112015-XDB-C18-ESI-poz.m	Acquired Time	11/17/2015 10:28:52 AM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

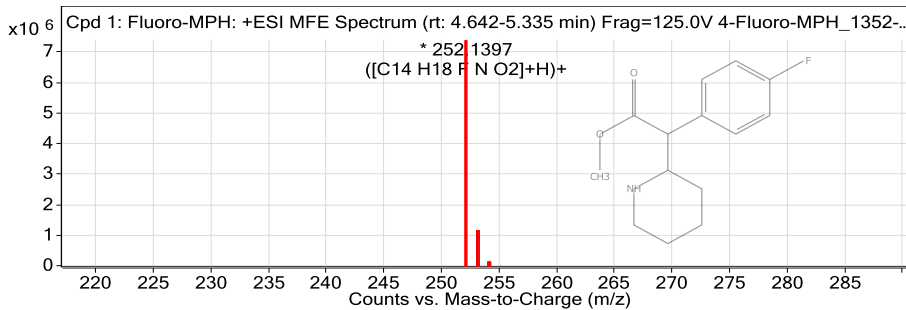
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Fluoro-MPH	Fluoro-MPH	C14 H18 F N O2	4.754	251.1325
Cpd 2: Fluoro-MPH	Fluoro-MPH	C14 H18 F N O2	5.519	251.1323

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Fluoro-MPH	252.1397	4.754	251.1325	4.75	C14 H18 F N O2	251.1322	-1.23

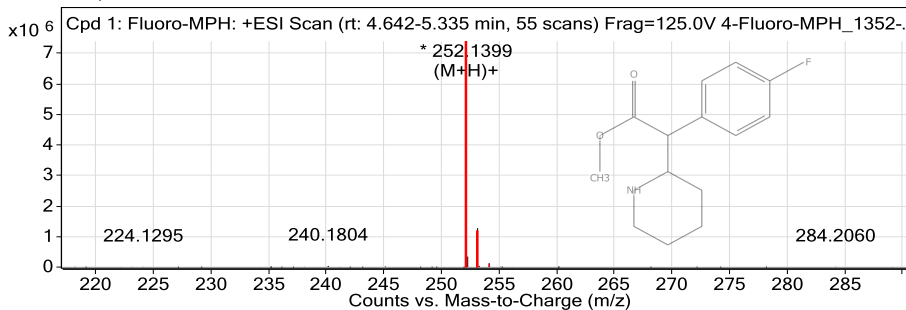
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



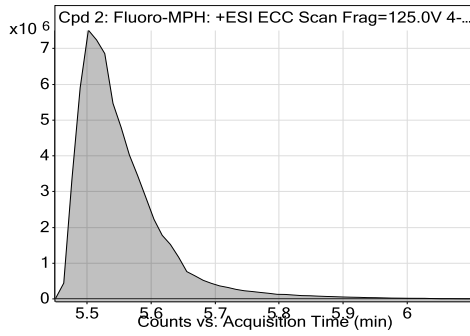
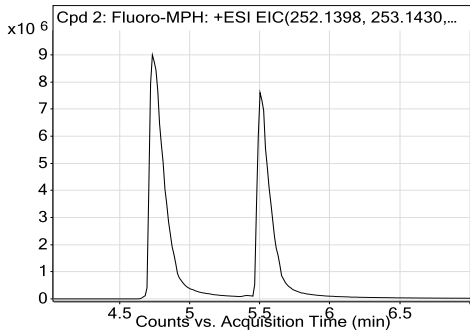
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
252.1397	1	7392479	C14 H18 F N O2	(M+H)+
253.1433	1	1098887.01	C14 H18 F N O2	(M+H)+
254.1459	1	122428.41	C14 H18 F N O2	(M+H)+
255.1485	1	8919.21	C14 H18 F N O2	(M+H)+
256.1609	1	384.06	C14 H18 F N O2	(M+H)+

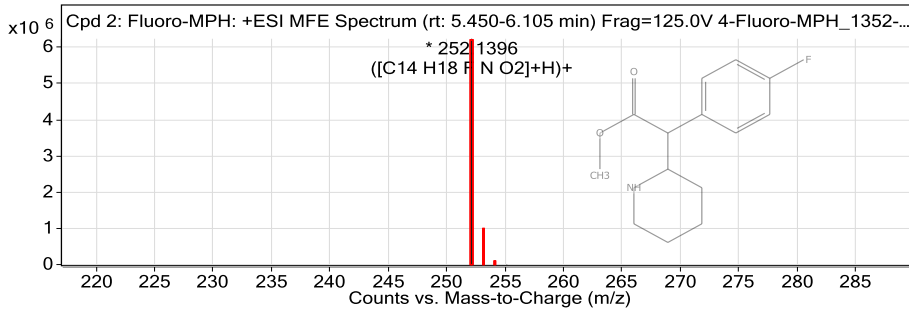
Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Fluoro-MPH	252.1396	5.519	251.1323	5.52	C14 H18 F N O2	251.1322	-0.72

Compound Chromatograms

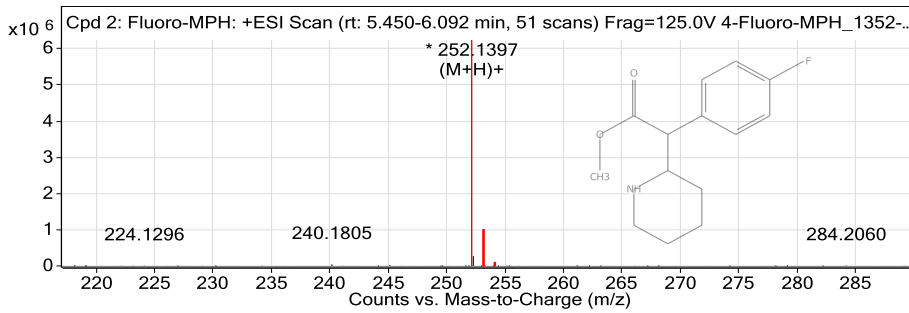
TOF REPORT



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

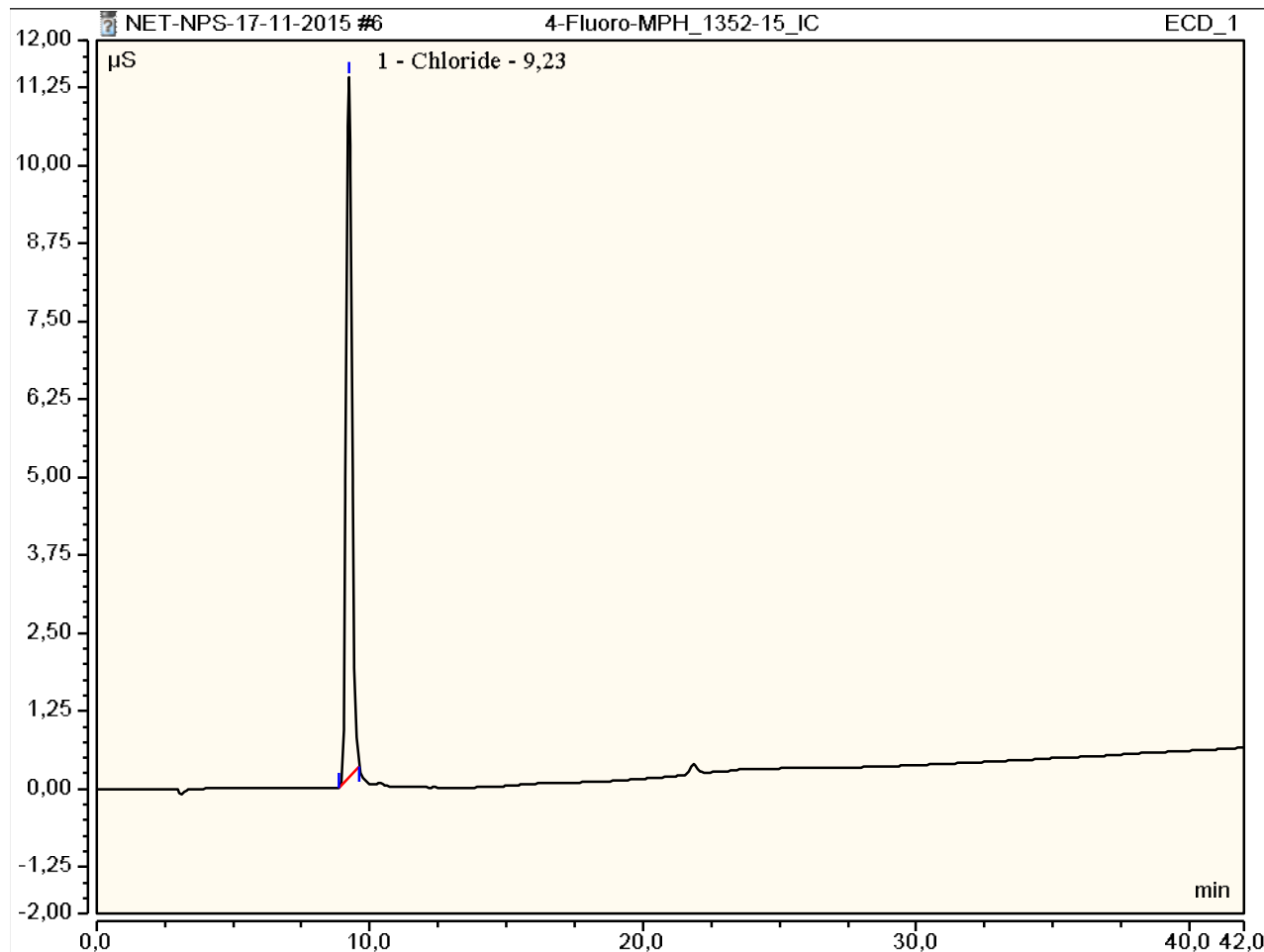
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
252.1396	1	6236341	C14 H18 F N O2	(M+H)+
253.143	1	957825.1	C14 H18 F N O2	(M+H)+
254.1457	1	102019.44	C14 H18 F N O2	(M+H)+
255.1483	1	7798.06	C14 H18 F N O2	(M+H)+

--- End Of Report ---

Peak Integration Report

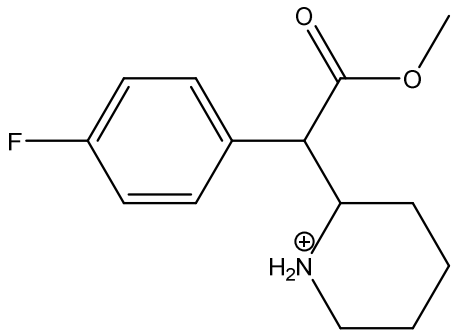
Sample Name:	4-Fluoro-MPH_1352-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	17-nov-2015 / 13:50	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	9,23	Chloride	BMB	2,65	11,22	n.a.
		TOTAL:		2,65	11,22	0,00





REPORT

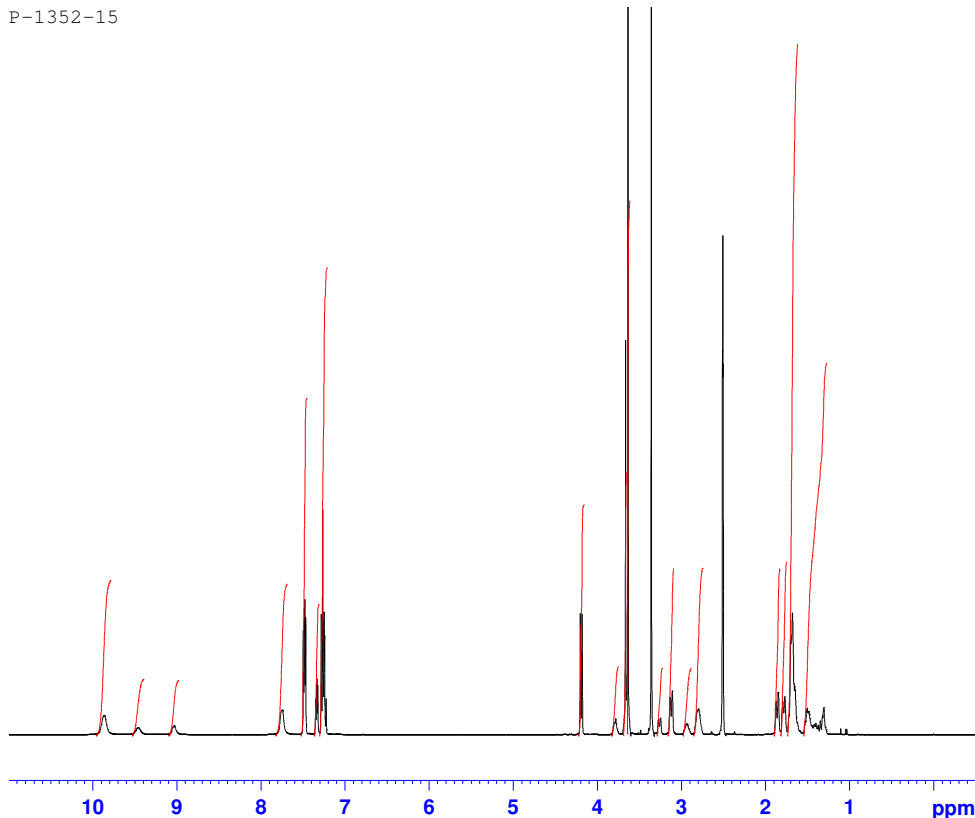
Sample ID:	1352-15
Our notebook code:	P-1352-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	2-(1-(4-fluorophenyl)-2-methoxy-2-oxoethyl)piperidin-1-ium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure by NMR, it is a mixture of two major components (plus possibly additional impurities) in approx. ratio 1 : 0.4. Both compounds contain para-fluorophenyl group. The major components could be diastereoisomers or possibly differ even more, maybe regarding the position of the NH ₂ group in the six-membered ring.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 24, 2015

P-1352-15



Current Data Parameters
 NAME P-1352-15
 EXPNO 1
 PROCNO 1

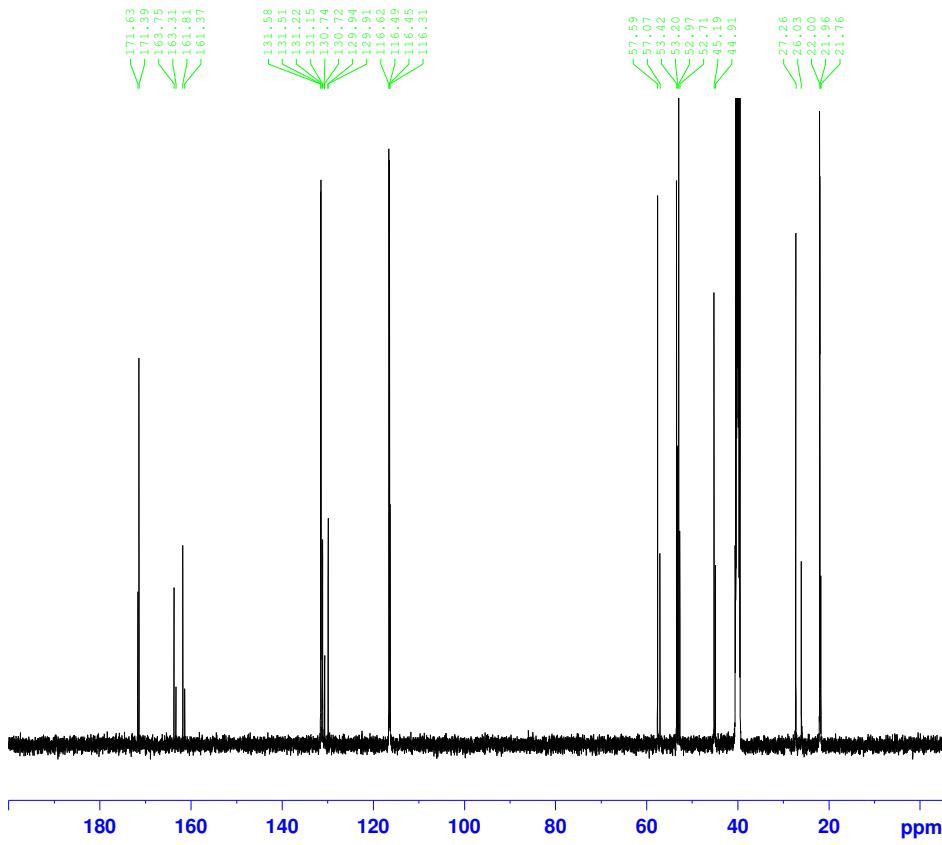
F2 - Acquisition Parameters
 Date_ 20151217
 Time 18.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 80.6
 DW 50.000 usec
 DE 6.50 usec
 TE 295.9 K
 D1 1.00000000 sec
 TD0 1



===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 8.90 usec
 PLW1 26.0000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

P-1352-15



Current Data Parameters
 NAME P-1352-15
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151217
 Time 20.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 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 122.0000000 W

===== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 80.00 usec
 PLW2 26.0000000 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