



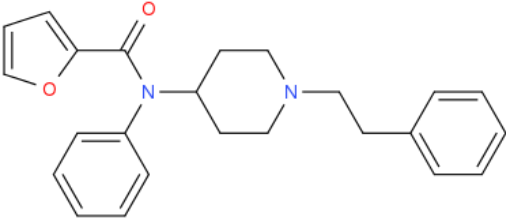
ANALYTICAL REPORT¹

FU-F (
C24H26N2O2)

N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]furan-2-carboxamide

Remark – other NPS detected: **none**

Sample ID:	1394-15
Sample description:	powder - white-off
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	12/9/2015
Date of entry (M/D/Y) into NFL database:	12/18/2015
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	N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]furan-2-carboxamide
Other names	
Formula (per base form)	C24H26N2O2
M _w (g/mol)	374,48
Salt form/anions detected	chloride
StdInChIKey	FZJVHWISUGFFQV-UHFFFAOYSA-N
Compound Class	Opioids
Other NPS detected	none
Add.info (purity..)	Substantial amount of Furancarboxylic acid detected by GC-MS, sample is not pure 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)
29/03/16	Substance class corrected.

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 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 (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

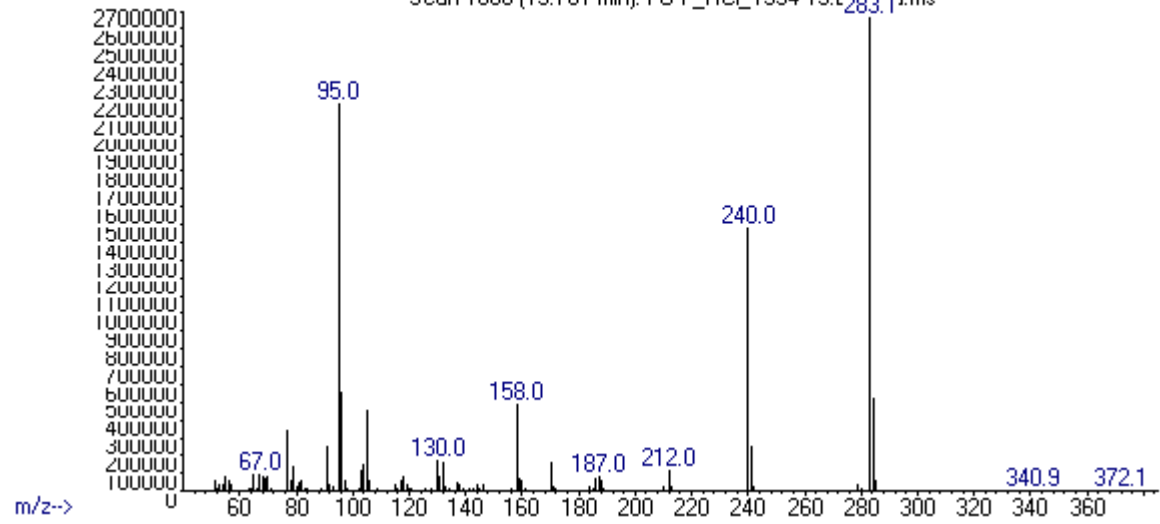
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,7 BP(1): 283; BP(2): 95,BP(3) :240,
HPLC-TOF	+	Exact mass (theoretical): 374,1994; measured value Δppm:-0,06; formula: C ₂₄ H ₂₆ N ₂ O ₂
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



TOF REPORT

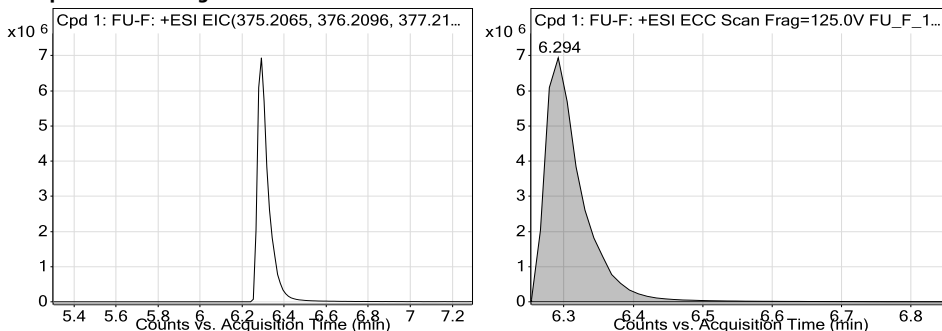
Data File	FU_F_1394-15_TOF.d	Sample Name	ID_1394-15
Sample Type	Sample	Position	P1-C2
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-17112015-XDB-C18-ESI-poz.m	Acquired Time	12/14/2015 2:28:52 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

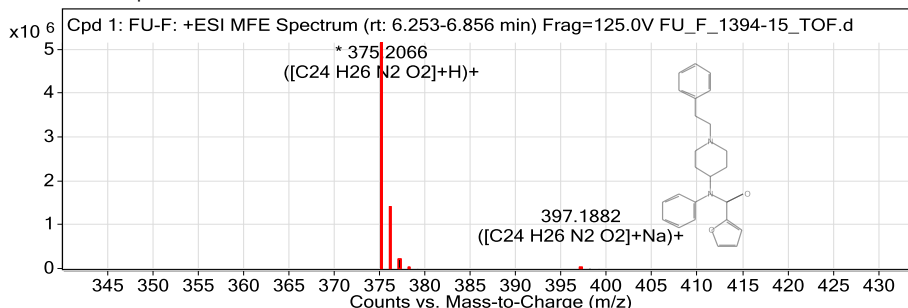
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: FU-F	FU-F	C24 H26 N2 O2	6.294	374.1995

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
FU-F	375.2066	6.294	374.1995	6.29	C24 H26 N2 O2	374.1994	-0.06

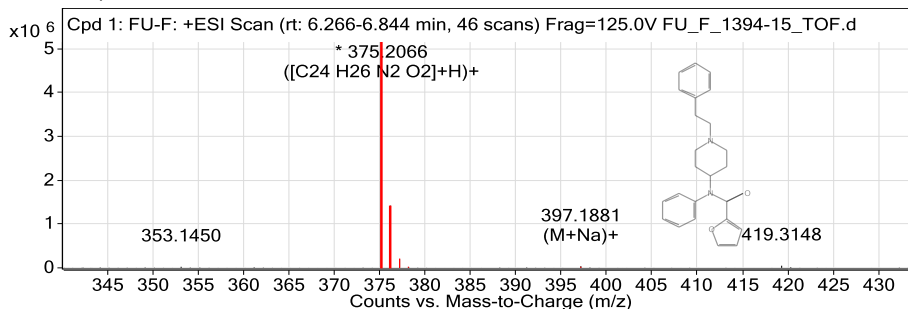
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

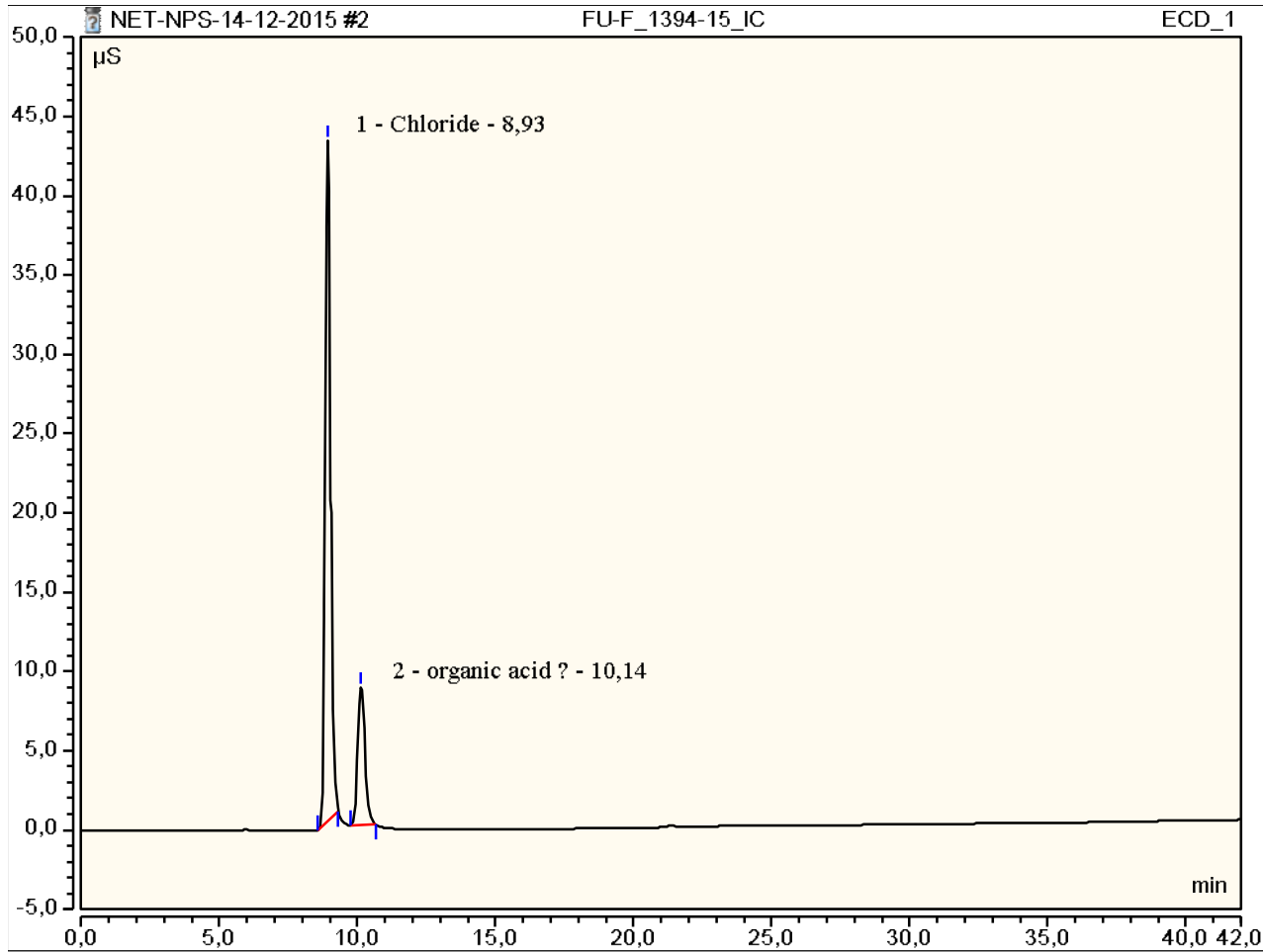
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
375.2066	1	5155182	C24 H26 N2 O2	(M+H)+
376.2104	1	1420864.7	C24 H26 N2 O2	(M+H)+
377.2132	1	197940.11	C24 H26 N2 O2	(M+H)+
378.2154	1	19384.8	C24 H26 N2 O2	(M+H)+
379.2175	1	1705.29	C24 H26 N2 O2	(M+H)+
397.1882	1	33186.7	C24 H26 N2 O2	(M+Na)+
398.1909	1	8136.82	C24 H26 N2 O2	(M+Na)+
399.1954	1	1386.32	C24 H26 N2 O2	(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	FU-F_1394-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	14-dec-2015 / 15:40	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	8,93	Chloride	BMB	9,71	42,88	n.a.
2,00	10,14	organic acid ?	BMB	2,66	8,68	n.a.
TOTAL:				12,37	51,56	0,00





REPORT

Sample ID:	1394-15
Our notebook code:	P-1394-15
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:	<p>+ another major component</p>
Chemical name:	1-phenethyl-4-(N-phenylfuran-2-carboxamido)piperidin-1-ium
Comments:	<ul style="list-style-type: none"> - Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure by NMR, it contains some minor impurities (around 5%). The sample, however, besides the compound with the structure proposed above, contains another major component (possible ratio 2:1) - an organic compound, containing at least one N and (hetero)cyclic ring(s) (8 aromatic/carbonyl signals in ^{13}C NMR and signals in ^1H NMR: 8.93, 8.57, 8.05, 7.92, 7.23, 6.65 and possibly others), it seems it has no (or just a few) aliphatic carbons. It is also possible, though unlikely, that the second major constituent is not just one compound, but mixture of more. - NB. In ^1H NMR spectrum below only the signals belonging to the structure proposed above are integrated, whereas in ^{13}C all prominent peaks are peak-picked.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 21, 2016

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P-1394-15

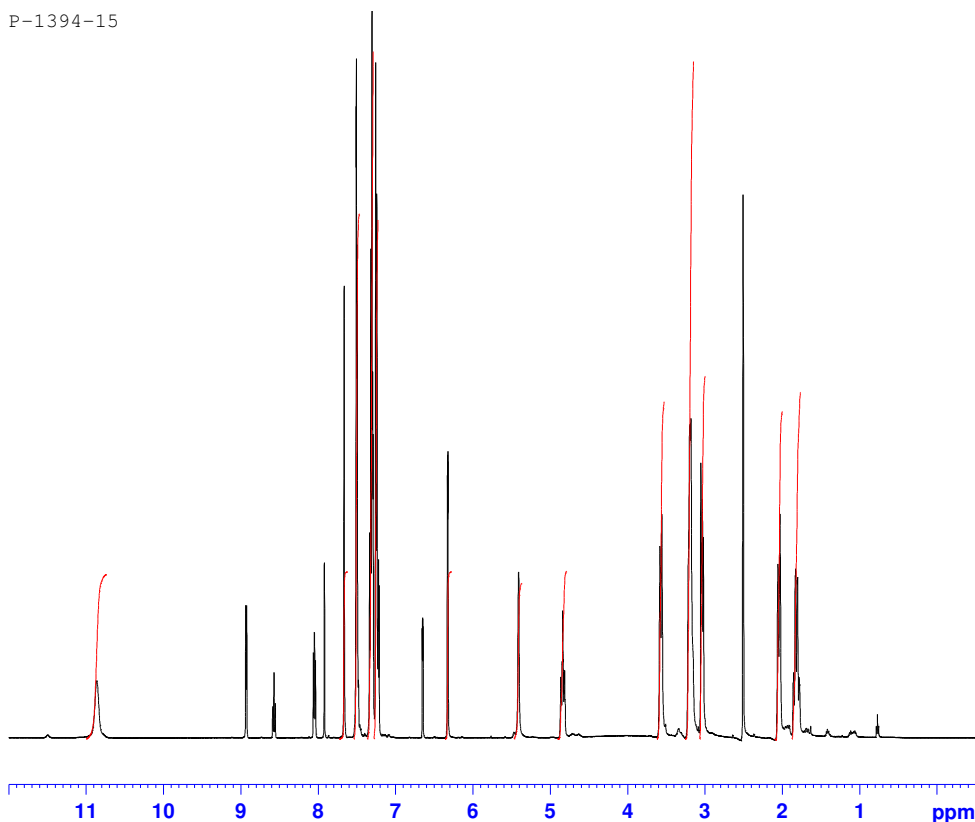


Current Data Parameters
 NAME P-1394-15
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160116
 Time 9.51
 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 32
 DW 50.000 usec
 DE 6.50 usec
 TE 298.0 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 500.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



P-1394-15



Current Data Parameters
 NAME P-1394-15
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160116
 Time 12.26
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 4096
 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 298.0 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
 CPDPRG2 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

