



Co-funded by the Prevention of and Fight
against Crime Programme of the European Union

EXPERIENCES WITH NEW SYNTHETIC OPIOIDS OBTAINED IN THE FRAMEWORK OF EU CO-FUNDED PROJECT RESPONSE



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“RESPONSE” project in brief

The main topic of forensic drugs investigations addressed by the RESPONSE project

(<http://www.policija.si/eng/index.php/generalpolicedirectorate/1669-nfl-page-response>)

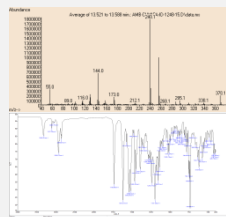
- ❑ **New psychoactive substances (NPS)** identification - challenges
 - ❑ unexpected growth in the number and type of NPSs [1]
 - ❑ easy availability of NPS, which are advertised and sold over the Internet
 - ❑ lack of availability of reference materials (RM) and specific reliable MS and FTIR spectra databases are the main problem for accurate NPS identification
- ❑ **Drugs profiling** where the main problem is the recognized gap between customers (law enforcement, judicial system, EU policy makers) needs and ForL capacities, methodologies and staff competencies.

[1] New Psychoactive substances in Europe, EMCDDA, March 2015, (<http://www.emcdda.europa.eu/publications/2015/new-psychoactive-substances>)

NPS facts & project objectives

FACTS:

- ❑ NPS are mainly **detected** in forensic and customs labs.
- ❑ **Identifying** of NPS means to elucidate compound structure, to give the substance a name and formula
- ❑ To **identify** new substance reference material (RM) or special techniques are needed
- ❑ RM are not available (**in real time**)
- ❑ Most of laboratories have to really:
 - ❑ on comparison of their results to **available MS and/or FTIR** spectra in searchable electronic libraries (not updated in real time)
 - ❑ or **existing information exchange channels and/or open source data**



PROJECT OBJECTIVES:

- ❑ to provide numerous **spectral data** MS, FTIR-ATR and FTIR-condensed phase on newly appearing NPS
- ❑ to **implement spectra into electronic data repositories** of European Network of Forensic Institutes - Drugs Working Group (ENFSI - DWG).
- ❑ to **share analytical data, knowledge and information efficiently through different communication platforms** (SI EWS, EMCDDA, EUROPOL, cooperation /communication with other complementary projects) and open source repositories
- ❑ By internet purchasing of NPSs the project will aim at **implementing a pro-active forensics respond** to the NPS phenomena and form “ NPS material bank”.

Sources of materials & Chemical characterizations methods

Type of material	Analytical methods
Reference materials (Different vendors; around 200)	GC-MS, FTIR-ATR, GC-MS-FTIR-(condensed phase) - optional
Test purchases (Internet based vendors; 200 to 300 samples)	GC-MS FTIR ATR GC-MS-FTIR (Condensed Phase) HPLC-TOF IC NMR (^1H , ^{13}C , $^1\text{H}-^1\text{H}$ <i>gs-COSY</i>)
Seized samples (Police/ Customs)	
Collected samples (NGO – anonymous users, project partners, other)	

The compounds are identified in the Slovenian forensic laboratory (NFL). Structures are confirmed by NMR at the Faculty of Chemistry and Chemical technology (FKKT). Spectra are provided to EI-MS and IR libraries of ENFSI DWG.

Test purchases over the internet -challenges



Detection of new substances [L. Ask Reitzel et al, Systematical methodology for finding novel NPS (New Psychoactive Substances) over the Internet, EAFS-2015, Prague, 2015]



- use a Google engine and simple keyword search and possibly filters
- follow social networks related to recreational drugs (blogs/discussion forums/chats)
- follow already evaluated reliable internet vendors

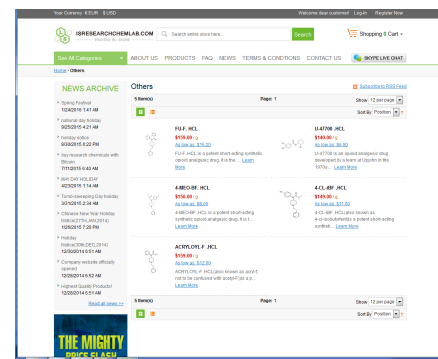
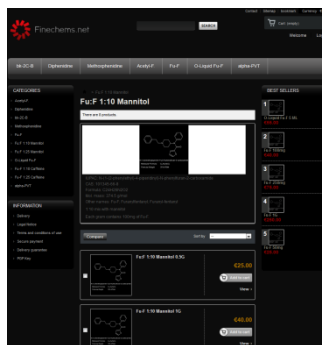
Find a internet vendor(s) and evaluate its reliability



- use information (and updates) from the internet
- check the payment options
- check minimum required order (some only sell bulk quantities)
- test vendors by small orders (few samples) first
- Preferable pure substances



<https://www.reddit.com/r/RCsources/wiki/vendors>



Europe

Belgium

Czech Republic

Germany

Hungary

The Netherlands

Poland

Spain

Sweden

United Kingdom

North America

Canada

USA

Asia

China

India

Japan

Treat with Caution
Scammers

Purchasing procedure:

- shall follow institutional and national rules (quite complicated)
- information exchange system between national authorities concerning internet orders has been established. (Co-operation of the Police (NFL), the Customs, and the Ministry of Health about every purchase in advance and after receiving the samples).

Test purchases - procedures

- ❑ Substances are purchased via four reliable "importing companies" in SI
- ❑ All data about the desired substance, internet links and desired quantities are provided by NFL; further communication with internet vendor is done by importing company
- ❑ First the inquiry at the chosen internet vendor is performed (all the communication is done via e-mail)
- ❑ After a positive response, NFL issues a customer declaration which is forwarded to the importing company in SI and to the liaison officer between the Police and the Customs
- ❑ Importing company orders and pays the substances to the internet vendor
- ❑ All the samples are delivered directly to the NFL
- ❑ NFL confirms the receipt of samples to importing company and to liaison officer
- ❑ Importing company issues the receipt which is paid from the financial department of MNZ
- ❑ In chemistry lab each sample is labeled with a unique identification number and is photographed
- ❑ Identification of the substance by different chemical methods follows
- ❑ In the case of a new substance (first time identified in SI) EMCDDA and SI EWS are informed
- ❑ Chemical characterisation data and analytical report are published in public open database created in the frame of the response project

REPUBLIKA SLOVENIJA
MINISTRSTVO ZA NOTRANJE ZADEVE
POLICIJA
Generálna policijska uprava
Nacionalni forenzični laboratorij
Vodovodna 95
1000 Ljubljana
SLOVENIJA

T: 01 428 44 93
F: 01 428 49 86
E: nfl@policija.si
www.policija.si

IZJAVA KUPCA O NAMENU UPORABE SUBSTANC CUSTOMER DECLARATION OF SPECIFIC USE(S) št./ no: 023-32/2015/38 (2P502-01) datum/date: 18.8.2015

Spodaj podpisani (I, We),

Ime (Name): Dr. SONJA KLEMENC

Naslov (Address):

RS-MNZ-GPU
NACIONALNI FORENZIČNI LABORATORIJ (NFL) /National Forensic Laboratory
(NFL)
Vodovodna 95
1000 LJUBLJANA
SLOVENIA

smo naročili(-i) pri (have ordered from):

Ime (Name):

Naslov (Address):

naslednje snovi (the following substances) iz/from
www.isresearchchemlab.com

4F-BF FREEBASE (1g)

Formula Name: 1-((4-fluorophenyl)(1-phenethylpiperidin-4-yl)amino)butan-2-one
CAS: N/A
Formula: C23H29FN2O
Exact Mass: 368.23
Molecular weight: 368.49
Synonyms: 4-fluoro-butyl-fent
<http://www.isresearchchemlab.com/catalogsearch/result/?q=4F-BF>

I/we hereby certify that the substance(s) referred to above will not be re-sold and will not be used for the further production of any other goods. In Slovenia the ordered substances are not scheduled as illicit drugs.

Ime (Name)
SONJA KLEMENC

Delovno mesto (Position)/
Vodja oddelka za kemijske preiskave/
Head of chemistry department

Podpis (Signature)

Datum (Date):
18.8.2015



Experiences of test purchases

- ❑ Purchases within EU are relatively easy (no Customs), the substances are delivered with correct invoice and mostly suitable labeling (although often wrong substance)
- ❑ Purchases from Canada run relatively smoothly, appropriate documentation and bills (sometimes wrong substance)
- ❑ Purchases from China:
 - some pages have very good selection (e.g. [Drugs Power Store](#)); but we couldn't establish any contact (they were non responsive)
 - Communications by internet vendor through e-mails
 - Shipments - track&trace (using courier delivery services company, e.g. FEDEX etc.)
 - Sometimes long delivery time
 - Few samples have not been delivered
 - Attached documentation usually describes the shipment as a **free sample** (real price is not given)
 - Substances are wrongly declared (e.g. as **sample of ammonium polyphosphate, acrylic paint etc.**) with different Common Customs Tariff to mislead the Customs (to avoid taxation of goods)
 - "Proforma invoices" do not correspond to the real price, the seller's company data on invoices is mostly "faked" (and this is a problem for the importing company which purchase for NFL)
 - Sample containers (bags) are labeled only by numbers – no chemical information

Shipping Label:

From: 05198199036
 Ship Date: 02DEC15
 Active: 5 YR MAX
 CAD: 6071296050000009

Ship To: HFEA
 HFEA TILES INTERNATIONAL TRADE CO. L.
 ROOM 2041818 HUANING RD HOMEHOUSE
 INTERSECTION RD HUANGSHAN RD
 HFEA, 238037
 CHINA

Country: CN
 Customs Value: 13.00 USD
 Customs Value: 13.00 USD
 Incoterms: DTY: R
 Ship Type: PKG

Tracking: 6396 1209 3151
 Destination: Ljubljana, Slovenia

Commercial Invoice:

INVOICE PAGE 1 OF 1

DATE OF EXPORTATION: 02DEC2015
 INVOICE NUMBER: 639612093151

SHIPPER: EXPORTER
 HFEA TILES INTERNATIONAL TRADE CO. L.
 ROOM 2041818 HUANING RD HOMEHOUSE
 INTERSECTION RD HUANGSHAN RD
 HFEA, 238037 CHN 05198199036

CONSIGNEE: IMPORTER
 Sample Name:
 Ljubljana, Slovenia
 1000 SI 3801428419

TERMS OF SALE: FOB/CA

COUNTRY OF MANUFACTURE	FULL DESCRIPTION OF GOODS	QTY	UNIT VALUE	TOTAL VALUE
CHINA	ACRYLIC PAINT 3213100000 1	1 Piece	13.00	13.00

CHARGES	AMOUNT
TOTAL FREIGHT CHARGES	0.00
TOTAL INSURANCE CHARGES	0.00
OTHER COSTS	0.00
TAX AMOUNT	0.00
TOTAL INVOICE VALUE	13.00
TOTAL NUMBER OF PACKAGES	1
TOTAL WEIGHT	0.1 KG

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE IS TRUE AND CORRECT TO THE BEST OF MY KNOWLEDGE.
 (SIGNATURE REQUIREMENTS MAY VARY PER COUNTRY)

DATE: 02DEC2015
 NAME: HFEA TILES INTERNATIONAL TRADE CO. L. SIGNATURE: TITLE:

Changes on the internet market

- ▶ Chinese Food and Drug Administration announced the control of 116 new psychoactive substances, effective from 1st October 2015 (*Non-medicinal drugs and psychotropic narcotic drug control varieties*).
- ▶ The newly scheduled NPS include also Acetylfentanyl, AH-7921, Butyrylfentanyl, β -Hydroxythiofentanyl, Isobutyrylfentanyl, Ocfentanyl which almost disappeared from China vendors web pages
- ▶ Consequently new synthetic opioids appeared on the China (and EU) market (Acryloil-F, 4Cl-iBF, FU-F, U 47700, etc.)

New synthetic opioids, received within RESPONSE project

The screenshot shows the homepage of ISRESEARCHCHEMLAB.COM. The website has a dark header with navigation links: 'Your Currency: € EUR \$ USD', 'Welcome dear customer!', 'Log-In', and 'Register Now'. Below the header is a search bar with the text 'Search entire store here...' and a 'Search' button. A shopping cart icon shows '0 Cart'. The main navigation bar includes 'See All Categories', 'ABOUT US', 'PRODUCTS', 'FAQ', 'NEWS', 'TERMS & CONDITIONS', and 'CONTACT US'. A 'SKYPE LIVE CHAT' button is also present. The 'NEWS ARCHIVE' section on the left lists various events and notices. The 'Others' section displays a grid of products, each with a chemical structure, name, price, and a 'Learn More' link. The products listed are: FU-F .HCL (\$159.00 / g), U-47700 .HCL (\$140.00 / g), 4-MEO-BF .HCL (\$150.00 / g), 4-CL-iBF .HCL (\$149.00 / g), and ACRYLOYL-F .HCL (\$159.00 / g). The website also features a 'THE MIGHTY PRICE SLASH' banner at the bottom.

ISRESEARCHCHEMLAB.COM
TRUSTED RC STORE

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Home - Others

NEWS ARCHIVE

- Spring Festival 1/24/2016 1:41 AM
- national day holiday 9/25/2015 4:21 AM
- holiday notice 8/30/2015 6:22 PM
- buy research chemicals with Bitcoin 7/11/2015 6:40 AM
- MAY DAY HOLIDAY 4/23/2015 1:14 AM
- Tomb-sweeping Day holiday 3/31/2015 2:34 AM
- Chinese New Year Holiday Notice(27TH JAN,2014) 1/26/2015 7:20 PM
- Holiday Notice(30th DEC,2014) 12/30/2014 6:51 AM
- Company website officially opened 12/28/2014 6:52 AM
- Highest Quality Products! 12/28/2014 6:51 AM

Read all news >>

Others

5 Item(s) Page: 1 Show 12 per page Sort By Position

FU-F .HCL
\$159.00 / g
As low as: \$15.20
FU-F .HCL is a potent short-acting synthetic opioid analgesic drug, It is the ... [Learn More](#)

U-47700 .HCL
\$140.00 / g
As low as: \$6.00
U-47700 is an opioid analgesic drug developed by a team at Upjohn in the 1970s... [Learn More](#)

4-MEO-BF .HCL
\$150.00 / g
As low as: \$8.00
4-MEO-BF .HCL is a potent short-acting synthetic opioid analgesic drug, It is t... [Learn More](#)

4-CL-iBF .HCL
\$149.00 / g
As low as: \$11.00
4-CL-iBF .HCL(also known as 4-cl-isobutyrfent)is a potent short-acting synthe... [Learn More](#)

ACRYLOYL-F .HCL
\$159.00 / g
As low as: \$12.00
ACRYLOYL-F .HCL(also known as acryl-f, not to be confused with acetyl-F)is a p... [Learn More](#)

5 Item(s) Page: 1 Show 12 per page Sort By Position

So far all synthetic opioids samples, purchased within the RESPONSE project, were obtained from one site.

- ❑ 4F-BF
- ❑ 4-MeO-BF
- ❑ FU-F
- ❑ U-47700

All the synthetic opioids have been offered in HCl salt form.

Two were received from FSI Zurich:

- ❑ AH-7921
- ❑ W-15

Two were received from Laboratoire de Police Scientifique de Lyon:

- ❑ Ocfentanil (A-3217)

All these synthetic opioids have been characterized, the data has been included in the online database.

Current situation on new synthetic opioids between users in Slovenia

NFL has not received any sample containing new synthetic opioid seized by the Police or collected via NGO so far.

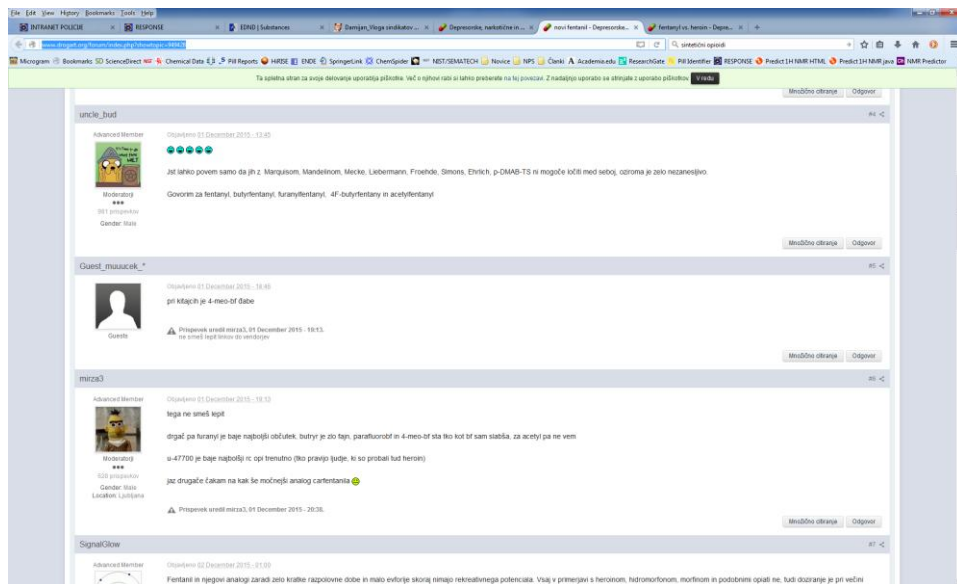
An associated partner DrogArt Association is a private non-profitable volunteer organization with the status of humanitarian organizations. The vision DrogArt Association is to reduce the risks associated with the use of drugs and alcohol in Slovenia.

In DrogArt drug related forums, users also report their experiences with synthetic opioids.



Resume from the forum about new synthetic opioids in Slovenia:

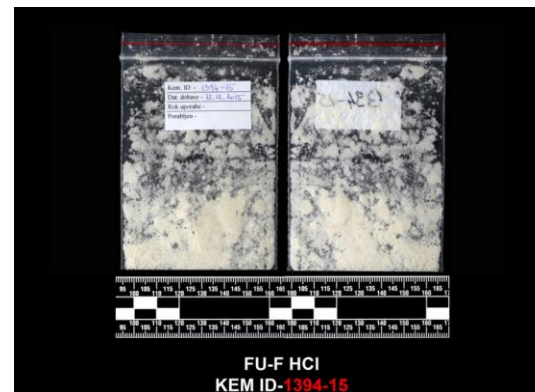
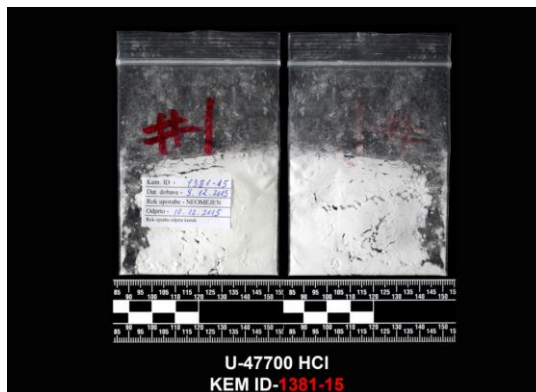
- New synthetic opioids are rarely used, only experimentally
- Users write about experiences with BF series, U-47700, other fentanyles (e.g. furanyl as FU-F, butyryl, acetyl, parafluorobf)
- U-47700 and FU-F are currently considered to be the best
- butyryl + 3fmp is considered to be a good combination
- MT-45 is allegedly poisonous, causes hair loss



Physical appearance of purchased synthetic opioids

Similar physical appearance of two different substances, both purchased from China as 4F-BF and 4-MeO-BF freebase. Zip bags with the substances were marked only with a hand written number on a sticker.

Each sample gets its unique identification number in the NFL laboratory.



Photos of the samples were taken from both sides and merged.

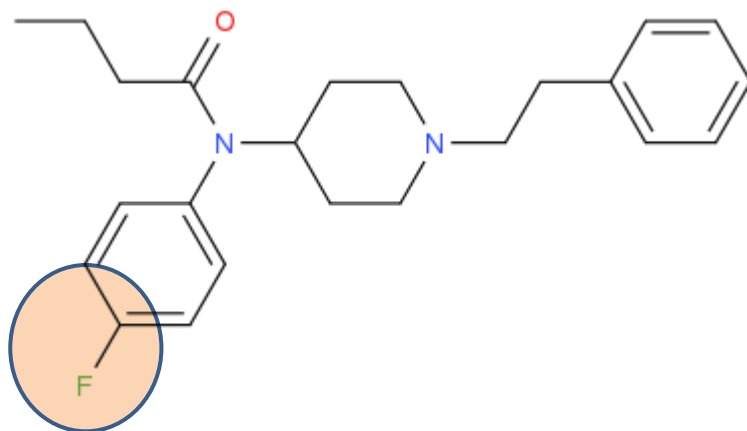
The sample, purchased as U-47700, in a form of white powder, and the sample, purchased as FU-F, in a form of off-white powder.

4F-BF (4-fluoro-butyrfentanyl) and 4-MeO-BF (4-methoxybutyrfentanyl)

Two analogues of butyrfentanyl:

Proposed structures (theroretical):

The structure of 4-MeO-BF given on the web site was different, -CO- was indicated on the position 2

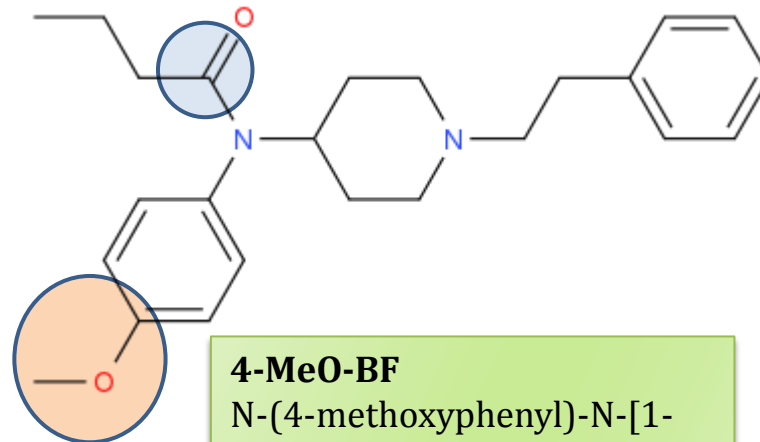


4F-BF

N-(4-fluorophenyl)-N-[1-(2-phenylethyl)piperidin-4-yl]butanamide

Mass: 368,496

Formula: C₂₃H₂₉FN₂O



4-MeO-BF

N-(4-methoxyphenyl)-N-[1-(2-phenylethyl)piperidin-4-yl]butanamide

Mass: 380,532

Formula: C₂₄H₃₂N₂O₂

4-MeO-BF: 2-on or 1-on?



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See All Categories

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SKYPE LIVE CHAT

Home - Others - 4-MEO-BF, HCL

4-MEO-BF, HCL

Formula Name: N-(4-methoxyphenyl)-N-(1-phenethylpiperidin-4-yl)butyramide

CAS: N/A

Formula: C₂₄H₃₂N₂O₂

Exact Mass: 380.25

Molecular weight: 380.52

Availability: In stock

\$150.00 / g

Qty: 1 unit g

Add to Cart

Save to Wishlist

Retail offer of 4-MEO-BF, HCL 1g price per gram

Quantity	Price per gram
1 g	\$150.00 / g
5 g	\$56.00 / g
10 g	\$42.00 / g
25 g	\$28.00 / g
50 g	\$20.00 / g
100 g	\$15.50 / g
250 g	\$11.20 / g
500 g	\$9.20 / g
1000 g	\$8.00 / g

4-MEO-BF, HCL Details

4-MEO-BF, HCL is a potent short-acting synthetic opioid analgesic drug. It is the 4-methoxy group N-butylamide analogue of the potent opioid fentanyl. Use for laboratory research only, not for human consumption.

NEWS ARCHIVE

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- national day holiday 9/25/2015 4:21 AM
- holiday notice 8/30/2015 6:22 PM
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- Holiday Notice(30th DEC,2014) 12/30/2014 6:51 AM
- Company website officially opened 12/28/2014 6:52 AM
- Highest Quality Products! 12/28/2014 6:51 AM

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The 1H-NMR spectrum shows peaks at 7.04, 7.00, 6.96, 6.92, 6.88, 6.84, 6.80, 6.76, 6.72, 6.68, 6.64, 6.60, 6.56, 6.52, 6.48, 6.44, 6.40, 6.36, 6.32, 6.28, 6.24, 6.20, 6.16, 6.12, 6.08, 6.04, 6.00, 5.96, 5.92, 5.88, 5.84, 5.80, 5.76, 5.72, 5.68, 5.64, 5.60, 5.56, 5.52, 5.48, 5.44, 5.40, 5.36, 5.32, 5.28, 5.24, 5.20, 5.16, 5.12, 5.08, 5.04, 5.00, 4.96, 4.92, 4.88, 4.84, 4.80, 4.76, 4.72, 4.68, 4.64, 4.60, 4.56, 4.52, 4.48, 4.44, 4.40, 4.36, 4.32, 4.28, 4.24, 4.20, 4.16, 4.12, 4.08, 4.04, 4.00, 3.96, 3.92, 3.88, 3.84, 3.80, 3.76, 3.72, 3.68, 3.64, 3.60, 3.56, 3.52, 3.48, 3.44, 3.40, 3.36, 3.32, 3.28, 3.24, 3.20, 3.16, 3.12, 3.08, 3.04, 3.00, 2.96, 2.92, 2.88, 2.84, 2.80, 2.76, 2.72, 2.68, 2.64, 2.60, 2.56, 2.52, 2.48, 2.44, 2.40, 2.36, 2.32, 2.28, 2.24, 2.20, 2.16, 2.12, 2.08, 2.04, 2.00, 1.96, 1.92, 1.88, 1.84, 1.80, 1.76, 1.72, 1.68, 1.64, 1.60, 1.56, 1.52, 1.48, 1.44, 1.40, 1.36, 1.32, 1.28, 1.24, 1.20, 1.16, 1.12, 1.08, 1.04, 1.00, 0.96, 0.92, 0.88, 0.84, 0.80, 0.76, 0.72, 0.68, 0.64, 0.60, 0.56, 0.52, 0.48, 0.44, 0.40, 0.36, 0.32, 0.28, 0.24, 0.20, 0.16, 0.12, 0.08, 0.04, 0.00. The spectrum is labeled with chemical shifts in PPM.

The web site describes 4-MeO-BF as N-(4-methoxyphenyl)-N-(1-phenylpiperidin)-4-butylamide with the structure as ketone (as -2-on substance).

Web sites often describe the substances with incomplete or wrong names, empirical formulas, and/or structures.

This web site even displays ¹H-NMR of the offered substance.

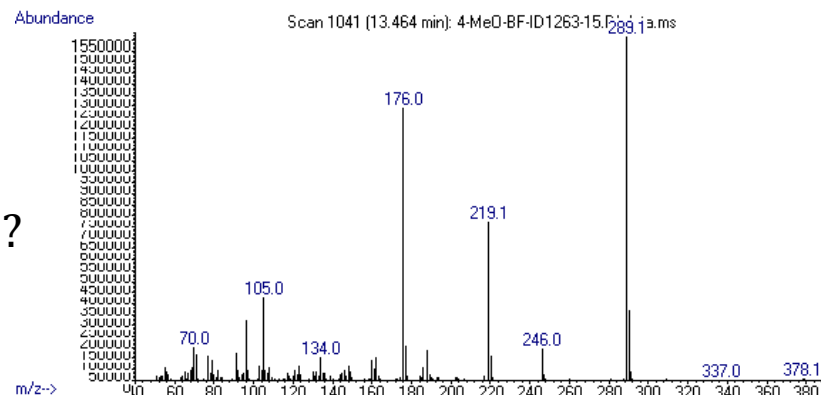
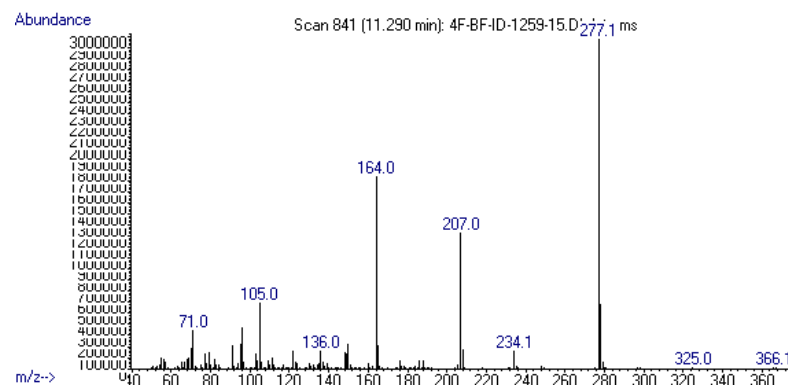
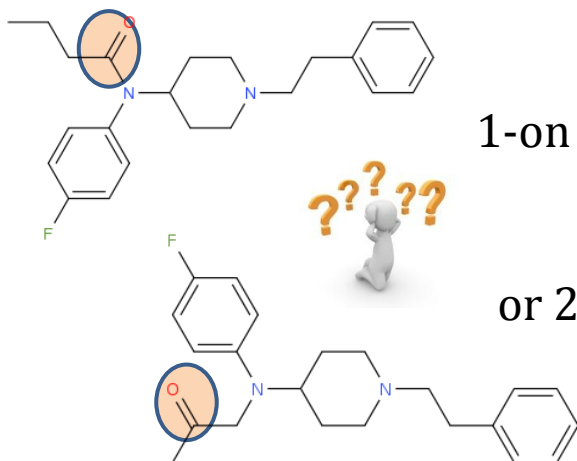
-CO- position can be defined by NMR.

Mass spectra of 4F-BF and 4-MeO-BF

No match with any of available ms libraries
(CaymanSpectraLibrary, SWGDRUG, NIST4, etc.)

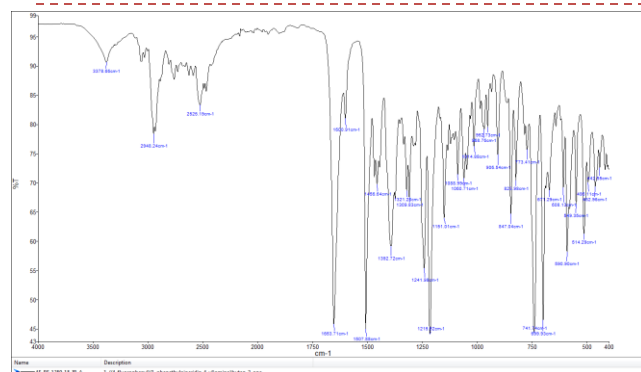
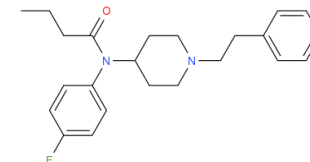
It was not possible to determine the position of -CO-
group without reference material or additional
analytical method

Problem:
-CO- position?

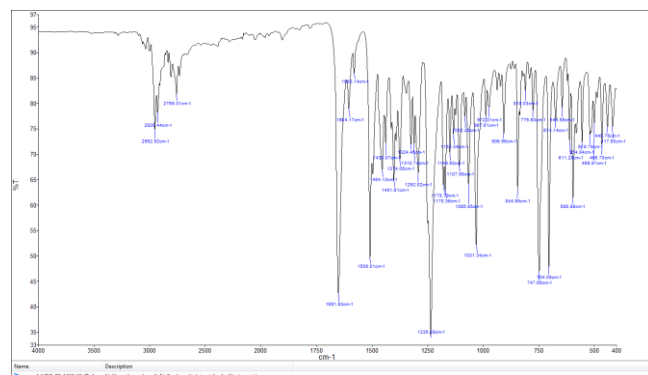


The difference between mayor peaks of both compounds is 12:
the difference between mass of MeO group(31) and fluoride (19)

Analytical data of 4F-BF



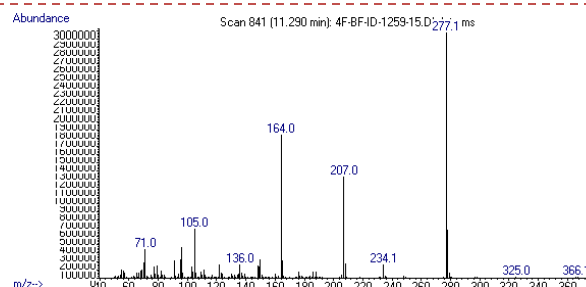
FTIR-ATR: 4F-BF as hydrochloride



FTIR-Condensed Phase:
4F-BF as base (always)

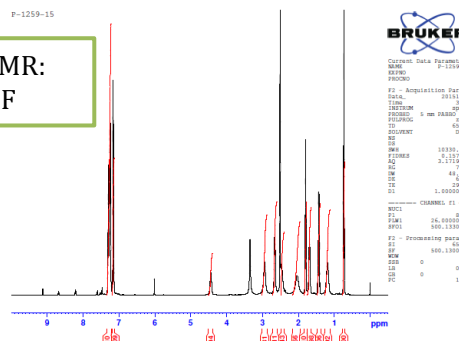
Result:
N-(4-fluorophenyl)-N-[1-(2-phenylethyl)piperidin-4-yl]
butanamide hydrochloride

Mass: 368,496 (as base)
Formula: C₂₃H₂₉FN₂O (as base)

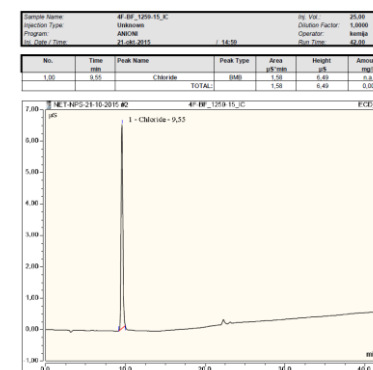


Ms spectrum of a single compound in GC:
no match with any of available ms library
(CaymanSpectraLibrary, SWGDRUG, NIST4,
etc.)

¹H NMR:
4F-BF

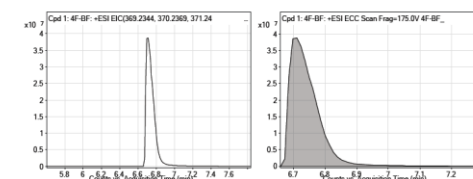


Ion Chromatography:
4F-BF as HCl

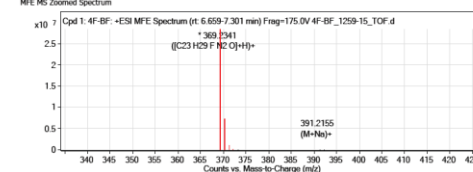


Name	Obs. m/z	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)	Found Cyclic Aliphatic
4F-BF	368.2341	6.719	368.2267	C23H29FN2O	368.2264	-0.91	Find by Molecular Feature

Compound Chromatograms

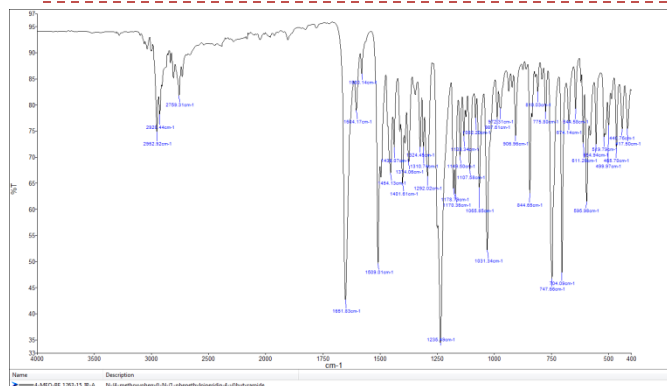
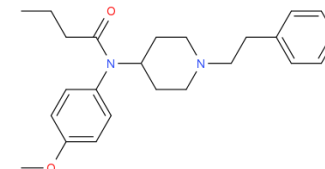


MFE MS Zoomed Spectrum

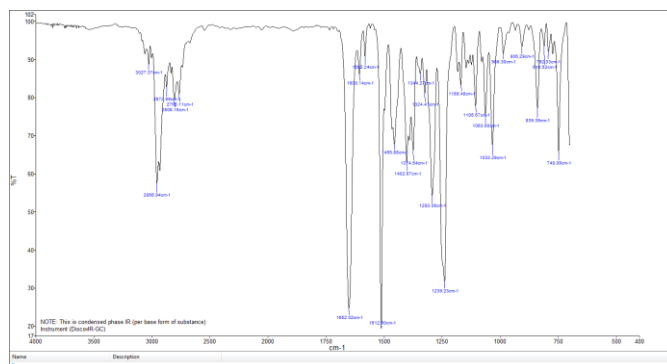


HPLC-TOF: exact mass: 368,2267
(DB Mass error -0,91ppm)
Formula: C₂₃H₂₉FN₂O

Analytical data of 4-MeO-BF



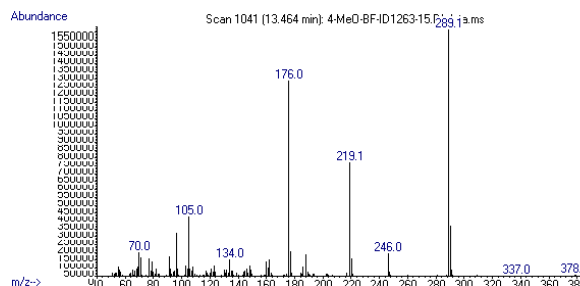
FTIR-ATR: 4-MeO-BF as base



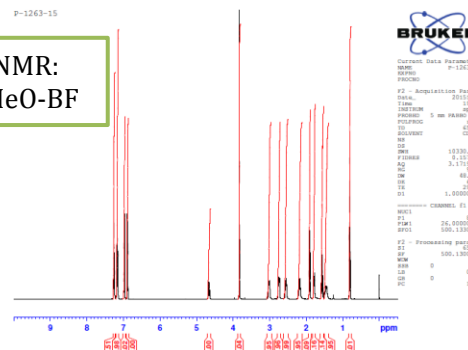
FTIR-Condensed Phase:
4-MeO-BF as base
(always)

Result:
N-(4-methoxyphenyl)-N-[1-(2-phenylethyl)piperidin-4-yl]butanamide base

Mass: 380,532
Formula: C₂₄H₃₂N₂O₂

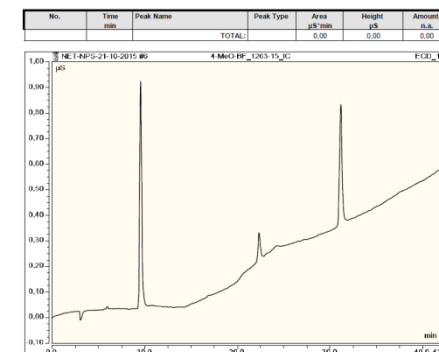


Ms spectrum of a single compound in GC:
no match with any of available ms library
(CaymanSpectralLibrary, SWGDRUG,
NIST4, etc.)



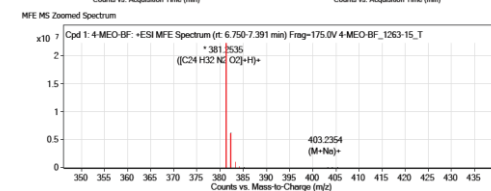
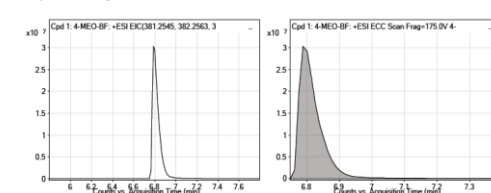
¹H NMR:
4-MeO-BF

Ion Chromatography:
4-MeO-BF as base



No.	Time (min)	Peak Name	Peak Type	Area (µV·min)	Height (µV)	Amount (nA)
1	10.9	4-MEO-BF	1	0.00	0.00	0.00
TOTAL:				0.00	0.00	0.00

Compound Chromatograms



HPLC-TOF: exact mass: 380,2463
(DB Mass error +0,26ppm)
Formula: C₂₄H₃₂N₂O₂

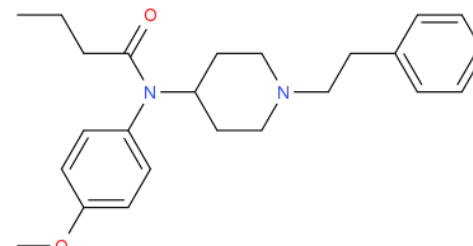


Current Data Parameters
 NAME P-1263-15
 EXPNO 20
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20151031
 Time 19.32
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 90.5
 DW 48.400 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 8.90 usec
 P1M1 26.00000000 W
 SFO1 500.1330885 MHz
 F2 - Processing parameters
 SI 65536
 SF 500.1300118 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

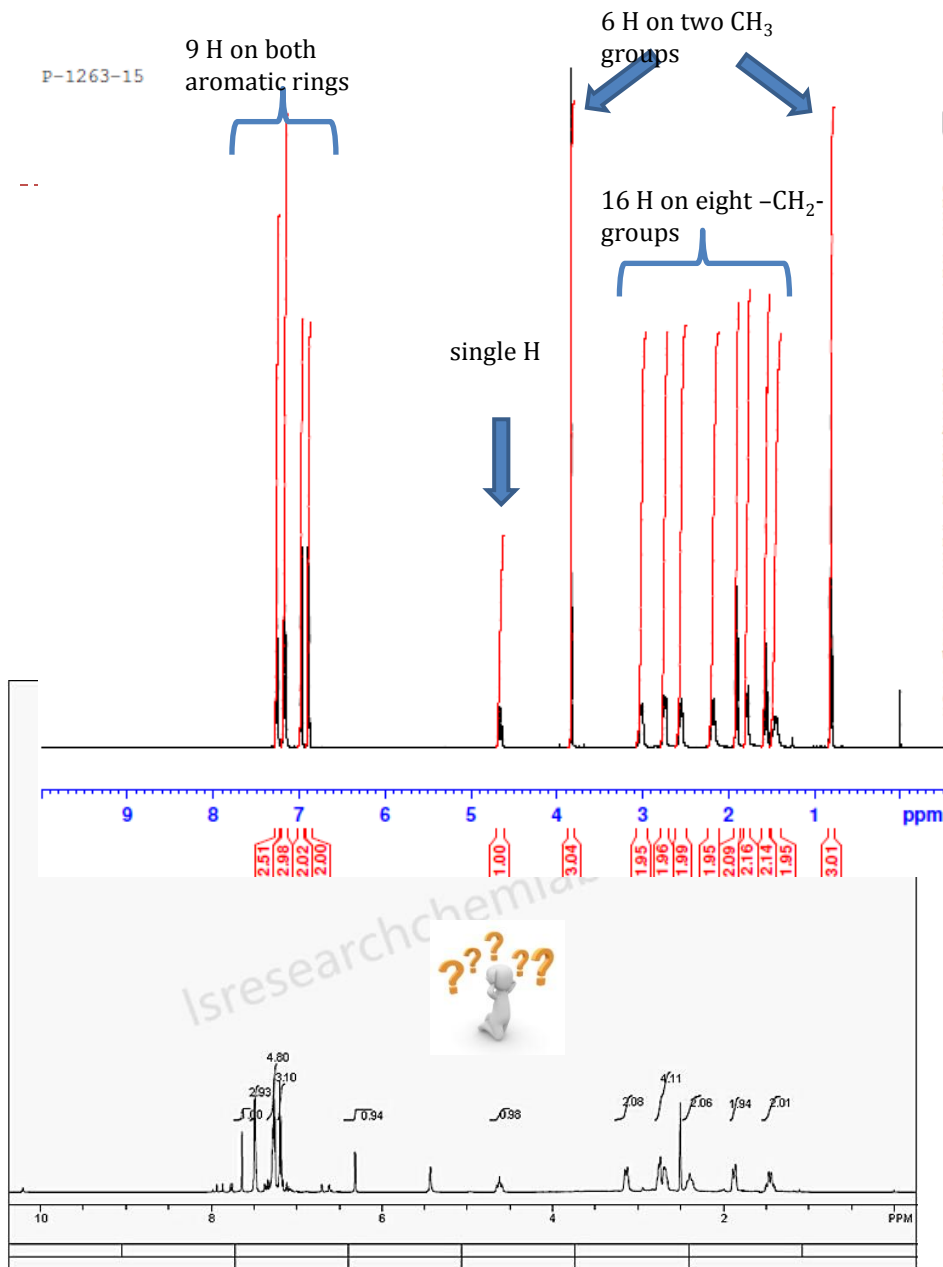
4-MeO-BF ¹H-NMR compared

¹H-NMR spectrum of 4-MeO-BF, obtained from FKKT (above) compared with the ¹H-NMR spectrum from the web site (below).

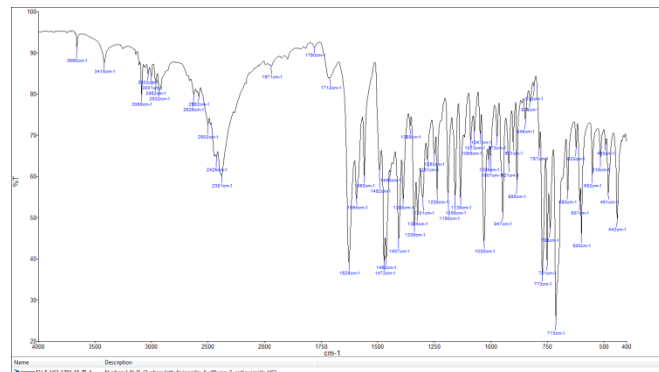
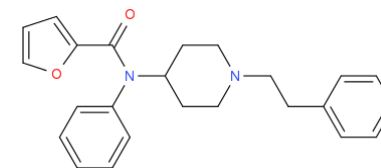


¹H-NMR spectrum clearly confirms 1-on structure (amide), and:

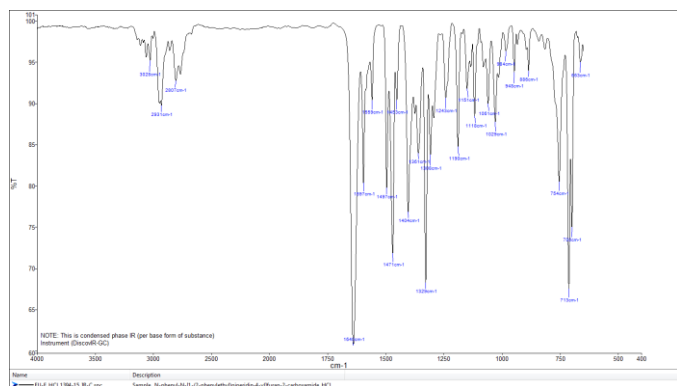
- Structure elucidation based on 1D and 2D NMR spectra
- Compound is pure by NMR.



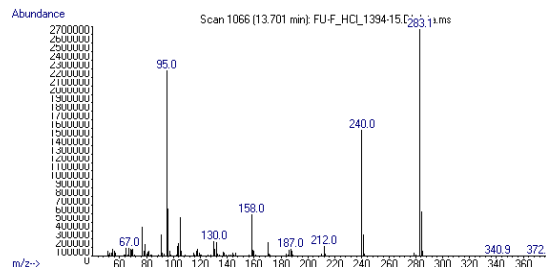
Analytical data of FU-F



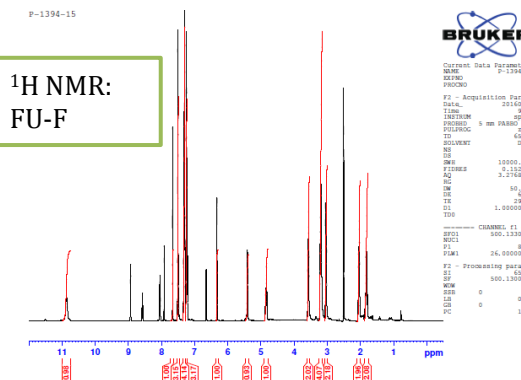
FTIR-ATR: FU-F as hydrochloride



FTIR-Condensed Phase:
FU-F as base (always)



Ms spectrum of a major compound in GC:
no match with any of available ms library
(CaymanSpectralLibrary, SWGDRUG, NIST4,
etc.); 2-Furoic acid was detected as well



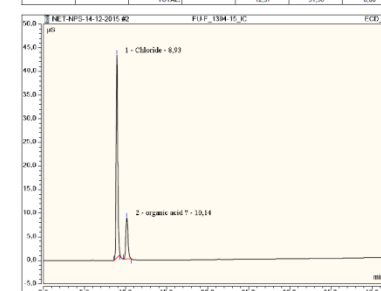
¹H NMR:
FU-F

Result:
N-(1-(2-phenylethyl)-4-piperidinyl)-N-phenylfuran-2-
carboxamide hydrochloride, with impurities

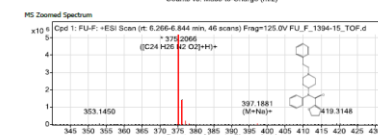
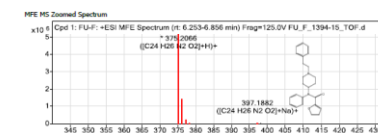
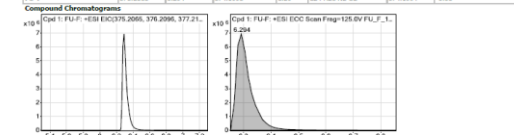
Mass: 374,484 (as base)
Formula: C₂₄H₂₆N₂O₂ (as base)

Ion Chromatography:
FU-F as hydrochloride, presence of
organic acid

No.	Time min	Peak Name	Peak Type	Area µV·min	Height µV	Amount mg/L
1.00	8.93	Chloride	DM	9.71	42.80	n.a.
2.00	10.14	organic acid 2	DM	2.90	6.08	n.a.
		TOTAL		12.37	51.56	6.00



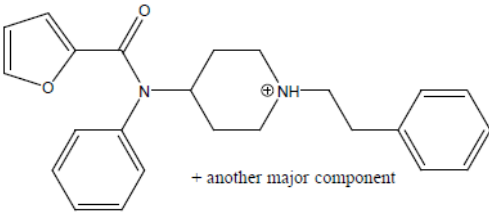
Name	Obs. m/z	Obs. RT	DB Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
FU-F	374.2066	6.294	374.1995	6.29	C ₂₄ H ₂₆ N ₂ O ₂	374.1994	-0.06



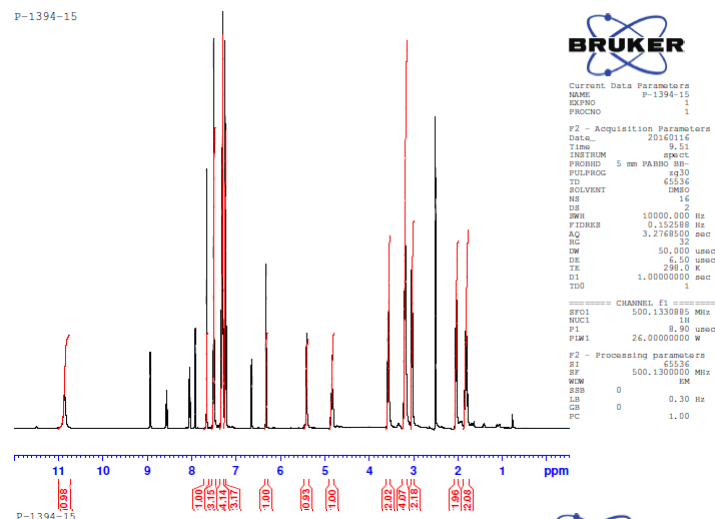
HPLC-TOF: exact mass: 374,1995
(DB Mass error -0,06ppm)
Formula: C₂₄H₂₆N₂O₂

FU-F NMR results

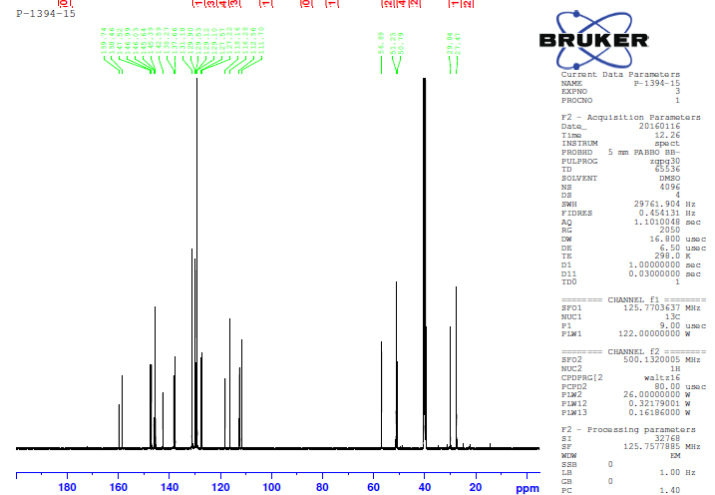
REPORT

Sample ID:	1394-15
Our notebook code:	P-1394-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
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, ¹ H- 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:	<p>- Structure elucidation based on 1D and 2D NMR spectra</p> <p>- 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 ¹³C 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.</p> <p>- NB. In ¹H NMR spectrum below only the signals belonging to the structure proposed above are integrated, whereas in ¹³C all prominent peaks are peak-picked.</p>
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 21, 2016

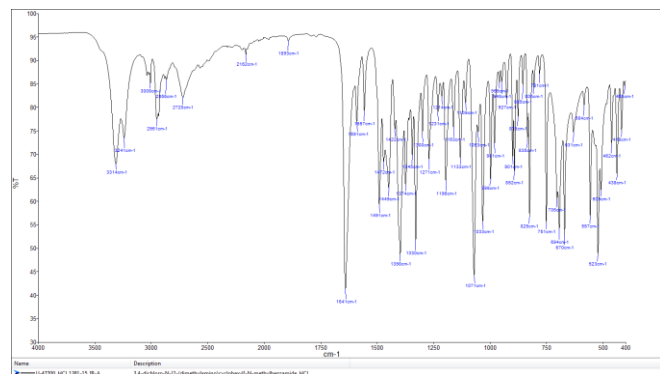
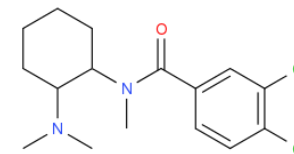
P-1394-15



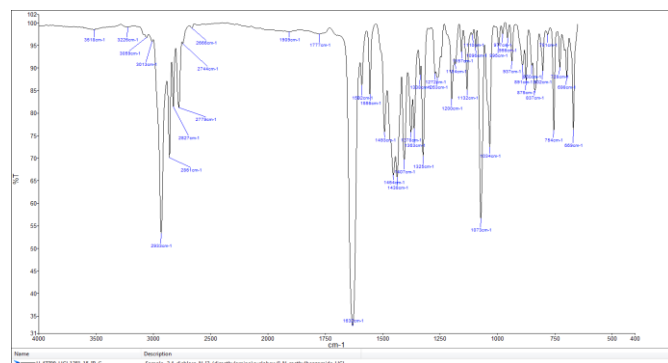
P-1394-15



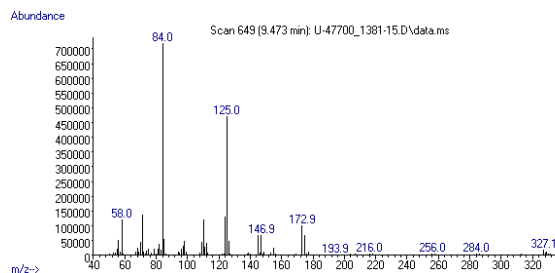
Analytical data of U-47700



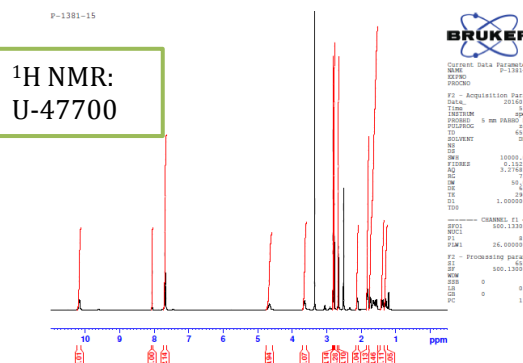
FTIR-ATR: U-47700 as hydrochloride



FTIR-Condensed Phase:
U-47700 as base
(always)



Ms spectrum of a single compound in GC:
no match with any of available ms library
(CaymanSpectralLibrary, SWGDRUG,
NIST4, etc.)



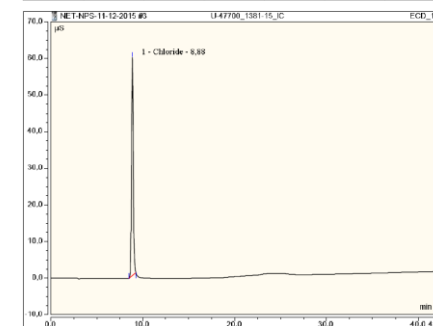
¹H NMR:
U-47700

Result:
3,4-dichloro-N-[2-(dimethylamino)cyclohexyl]-N-
methylbenzamide hydrochloride

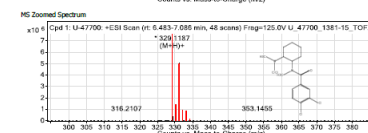
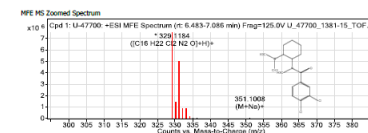
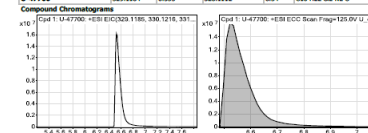
Mass: 329,27 (as base)
Formula: C₁₆H₂₂Cl₂N₂O (as base)

Ion Chromatography: U-47700 as hydrochloride

No.	Time min	Peak Name	Peak Type	Area μS*min	Height μS	Amount mg/L
1.00	8.88	Chloride	BMB	13.22	58.49	n.a.
		TOTAL		13.22	58.49	0.00



Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
U-47700	328.1114	6.53	6.53		C16H22Cl2N2O	328.1109	-0.93



HPLC-TOF: exact mass: 328,1112
(DB Mass error -0,93ppm)
Formula: C₁₆H₂₂Cl₂N₂O₂

“Drugs monographs” database

- ▶ Chemical information on NPSs and related compounds collected in the frame of RESPONSE is joined in the “Drugs Monographs” database – an interactive table searchable along different criteria
 - ▶ http://www.policija.si/apps/nfl_response_web/seznam.php
- ▶ The free access database already enhanced communications and information exchange between forensic and customs community. (example on the next slide)

Recently some new contributors have enriched the database:

- ▶ CLEN2SAND project data on novel opioids (kindly provided by the European Commission Joint Research Centre (JRC), DG TAXUD and the CLEN)
- ▶ Analytical data obtained on samples kindly provided by Forensic Institute Zurich Switzerland , HIFS Hungary , FSI Sweden and some other contributors have been implemented into the RESPONSE project database .

Information and data exchange in practice U-47700

- ▶ Preliminary characterisation data on U-47700 compound was published in “Dugs monographs” database 18/12/2015 (compound was not yet NMR confirmed at that time)
- ▶ At 20th January, 2016 we received two and at 27th January the third request from Begium:
 - ▶ From Toxicological Center of University of Antwerpwen (at 10:54 AM) – request for MS spectrum (we delivered the spectrum in electronic format (Agilent) at 11:15 AM)
 - ▶ From Customs lab 27/01/16 at 13:34 PM - request for IR and/or MS (we delivered data at 14:49)
 - ▶ From forensic lab NICC at 15:20 PM request for IR spectrum (we delivered data at 17:18)
- ▶ Belgian colleagues confirmed the substance as U-47700 succesfully



U-47700
INGE M.J. VINCKIER za: sonja.klemenc

Zgodovina: Sporočilo že ima odgovor.

van Nuijs Alexander <alexander.vannuijs@uantwerpen.be>
to me, sonja.klemenc, Maudens ▾

Jan 20 ☆ Reply ▾

Dear Sonja,

We have a possible intoxication with U-47700 but need to confirm this. I see in your analytical reports from the RESPONSE project (http://www.policija.si/apps/nfi_response_web/seznam.php) that there is some information on this compound (BP), but a full mass spectrum is not available. Is it possible to send me a full mass spectrum of this compound or bring me in contact with people who could help me with this?

Thank you very much for your help!
Kind regards,
Prof. dr. Alexander L.N. van Nuijs, PharmD

 **Toxicological Centre**
University of Antwerp

Dear Sonja,

Can you send me a IR spectrum from the U-47700.
This compound is listed on the Response website. We have a mass spectrum but I want to confirm it with a IR spectrum.

Best regards,

Filip VAN DURME, gerechtelijk deskundige drugs
Tel +32 2 240 04 83 – GSM +32 474 87 45 16 – Fax +32 2 243 46 08
Nationaal Instituut voor Criminalistiek en Criminologie – Vilvoordsesteenweg 100, 1120 Brussel

Dear Sonja,

Here I am again with some question about a product in the Response Database: U-47700
Do you have the FTIR spectrum or the MS-spectrum of this product ? Or is it still being analysed?

Thank you in advance,
Inge

Final reports: the web database “Drug Monographs”



NPS AND RELATED COMPOUNDS - ANALYTICAL REPORTS

Co-funded by the Prevention of and Fight
against Crime Programme of the European Union



Show 8 entries

Search:

Substance class	substance (NPS) common name	structure (created by OPSIN free tool)	NPS1 systematic name	other names	Formula per base form	Mw (g/mol) per base form NPS1	MS (BP1)	MS (BP2)	MS (BP3)	GC-MS-RT NFL/min	MS spectrum (picture)	StdIn ChIKey	Type of detection	comments	date of entry	rep up
Opioids	4-MEO-BF		N-(4-methoxyphenyl)-N-(1-phenethylpiperidin-4-yl)butyramide		C24H32N2O2	380.53	289	176	219	13.46	show	FNVSEQCPMXWQKG-UHFFFAOYSA-N	test purchase		2015-09-02	2015-
Opioids	U_47700		3,4-dichloro-N-[2-(dimethylamino)cyclohexyl]-N-methylbenzamide		C16H22Cl2N2O	329.27	84	125	71	9.46	show	JGPNMZWFVRQNGU-UHFFFAOYSA-N	test purchase	Impurity detected by NMR	2015-12-18	2016-
Opioids	AH-7921		3,4-dichloro-N-[(1-(dimethylamino)cyclohexyl)methyl]benzamide		C16H22Cl2N2O	329.27	126	127	173	10.31	show	JMZR0FPPEXCST-UHFFFAOYSA-N	collected	Sample was kindly provided by Forensic Science Institute Zurich, Switzerland (NMR confirmed)	2016-01-15	
Opioids	4F-BF		1-((4-fluorophenyl)(1-phenethylpiperidin-4-yl)amino)butan-1-one	4F-BF/4-fluoro-butyrfentanyl	C23H29FN2O	368.49	277	164	207	11.29	show	QZFMICYBPSLOBP-UHFFFAOYSA-N	test	pure by GC-HPLC		
Opioids	VALERYLFENTANYL		N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]pentanamide		C24H32N2O	364.52	273	146	189		show	VCCPWHJAYWQMR-UHFFFAOYSA-N	anal			
Opioids	W-15		4-chloro-N-[1-(2-phenylethyl)-2-piperidinylidene]-benzenesulfonamide		C19H21ClN2O2S	376.90	207	104	111	15.84	show	VJHXSSVOCBVMU-UHFFFAOYSA-N	coll			

Showing 1 to 8 of 8 entries (filtered from 303 total entries)

Table updated 22.03.2016
826 hits

The analytical results are published on the RESPONSE web page:
http://www.policija.si/apps/nfl_response_web/seznam.php
in a searchable table form containing pdf monographs. The table enables filtering data.
The page is regularly updated.

ANALYTICAL REPORT¹
4-MEO-BF (C24H32N2O2)

N-(4-methoxyphenyl)-N-(1-phenethylpiperidin-4-yl)butyramide

Remark – other NPS detected: none

Sample ID: 1263-15
Sample description: powder – yellowish (gently)
Sample type: test purchase / RESPONSE – purchasing
Date of sample receipt (M/D/Y): 9/2/2015
Date of entry (M/D/Y) into NPS database: 9/2/2015
Report updates (if any) will be published here: http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified – structure* (base form):

Systematic name: N-(4-methoxyphenyl)-N-(1-phenethylpiperidin-4-yl)butyramide

Other names:

Formula (per base form): C24H32N2O2

M_w (g/mol): 380.53

Salt form: base

StdInChIKey: FNVSEQCPMXWQKG-UHFFFAOYSA-N

Compound Class: Opioids

Other NPS detected: none

Add info (purity...): pure by GC-HPLC

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 AUST/2013/105C/DRI/2014/043). 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: 10.1039/c3cc50384a

Stron 1 od 5

ID1263-15



Conclusions and thoughts

- ❑ New synthetic opioids are inexpensive and easily available through Internet
- ❑ Online shop information does not always correspond to the offered substance: incomplete or wrong names, structures and formulas, wrong salts
- ❑ The test purchases performed in the frame of the RESPONSE project so far, show that:
 - drugs users can never be sure of what they get when buying from internet vendors.
 - the rate of the “false advertised” compounds is at approximately 20% - 30%.
 - this poses a serious health risks for the population of NPS users
- ❑ Activities which can rise the awareness are welcomed and should be enhanced at all levels (national and international EWS stakeholders are the key actors).
- ❑ One of the options with regard to harm reduction are also enhanced activity and capacities for the support of "anonymous testing" of samples provided by users (as for example already existing WEDINOS project).
- ❑ Our impression is that the internet market is still growing.
- ❑ China ban of 116 substances was a bit "disturbing" but the supplying chain was not seriously "endangered".
- ❑ What to expect with regard to "UK's Psychoactive Substances Act 2016"? Reduced supply or just movement of the web vendors to new locations?

Acknowledgements

To partners and associate partners

[University of Ljubljana, Faculty of Chemistry and Chemical Technology \(FKKT\), Slovenia](#)

[Ministry of the Interior, National Forensic Institute \(INPS\), France](#)

[Hungarian Institute for Forensic Sciences \(HIFS\), Hungary](#)

[National Institute of Criminalistics and Criminology \(INCC\), Belgium](#)

[Institut for Forensic Medicine, Aarhus \(UNI Aarhus\), Denmark](#)

[University of Copenhagen, Faculty of Health \(UNI CPHG\), Department of Forensic Medicine, Denmark](#)

[Ministry of the Interior of the Republic of Croatia, Forensic Science Centre \(MUP RH\), Croatia](#)

[European Monitoring Centre for Drugs and Drug Addiction \(EMCDDA\), Portugal](#)

[Forensic Science Lab, Dublin \(FSL\), R Ireland](#)

[Forensic Science and Toxicology Lab, State General Laboratory, Ministry of Health \(FSTL\), Cyprus](#)

[Judiciary Police, Scientific Police Laboratory \(SPL\), Portugal](#)

[National Investigation Service Norway \(KRIPOS\), Norway](#)

[National Institute for Public Health \(NIJZ\), Slovenija](#)

[University of Ljubljana, Faculty of Medicine, Department of Toxicology \(UNI LJ FM DT\), Slovenia](#)

[Association DrogArt \(DrogArt\), Slovenia](#)

To EU COMMISSION: The RESPONSE project is financially supported by the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). We kindly acknowledge this! The content of this presentation is the sole responsibility of the authors and can in no way be taken to reflect the views of the European Commission.

