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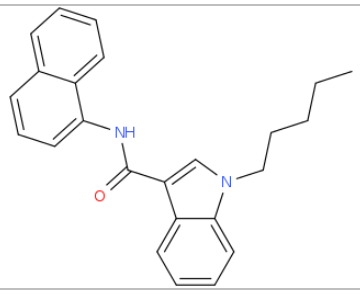
ANALYTICAL REPORT[1,2]

MN24 (C24H24N20)

N-(naphthalen-1-yl)-1-pentyl-1H-indole-3-carboxamide

Remark – other NPS detected: none

Sample ID:	1457-16
Sample description:	powder - brown
Sample type:	collected /Institute of Forensic medicine, University Freiburg, Germany
Date of sample receipt (M/D/Y):	1/14/2016
Date of entry (M/D/Y) into NFL database:	10/25/2016
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[3] (base form)	
Systematic name	N-(naphthalen-1-yl)-1-pentyl-1H-indole-3-carboxamide
Other names	JWH-018 Carboxamide Derivative, MN-24, NNEI, AM-6527
Formula (per base form)	C24H24N20
M_w (g/mol)	356,47
Salt form/anions detected	base
StdInChIKey	GWCQNKRMTGVYIZ-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	pure by GC-MS, minor impurities by NMR and HPLC- TOF

[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] Acknowledgement: Sample (not NMR confirmed) was kindly provided by the Institute of Forensic Medicine, University of Freiburg, Germany. Analytical results shown in this report were done in NFL and FKKT, Slovenia.

[3] 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 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 6.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}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 (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 ^{0}C. Chromatographic separation as above **(1).**
Split MS : IR = 1: 9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.
IR (condesed (solid) 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

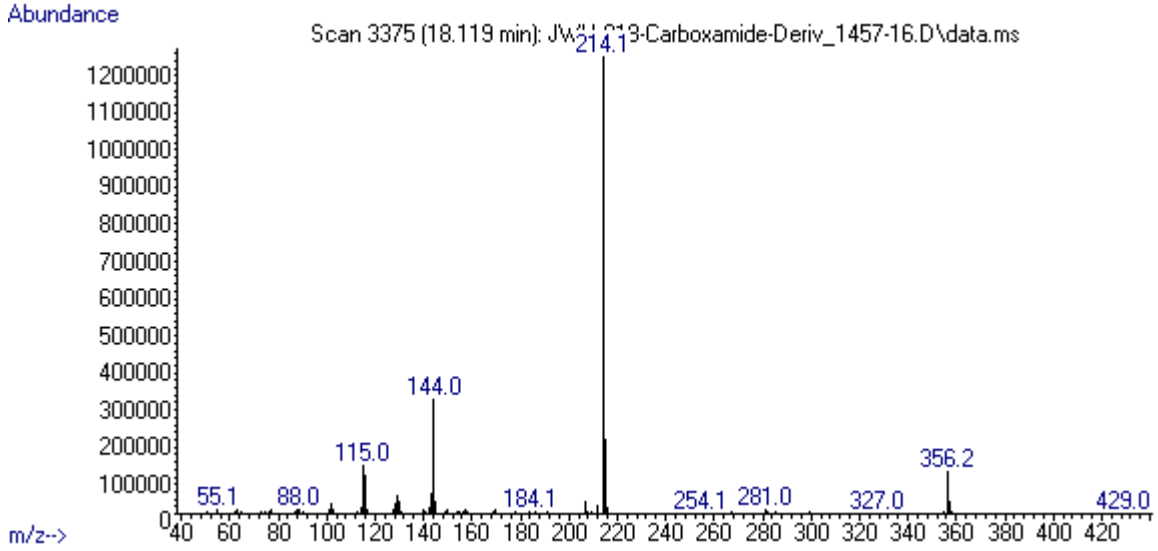
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	partially
H_2O	partially

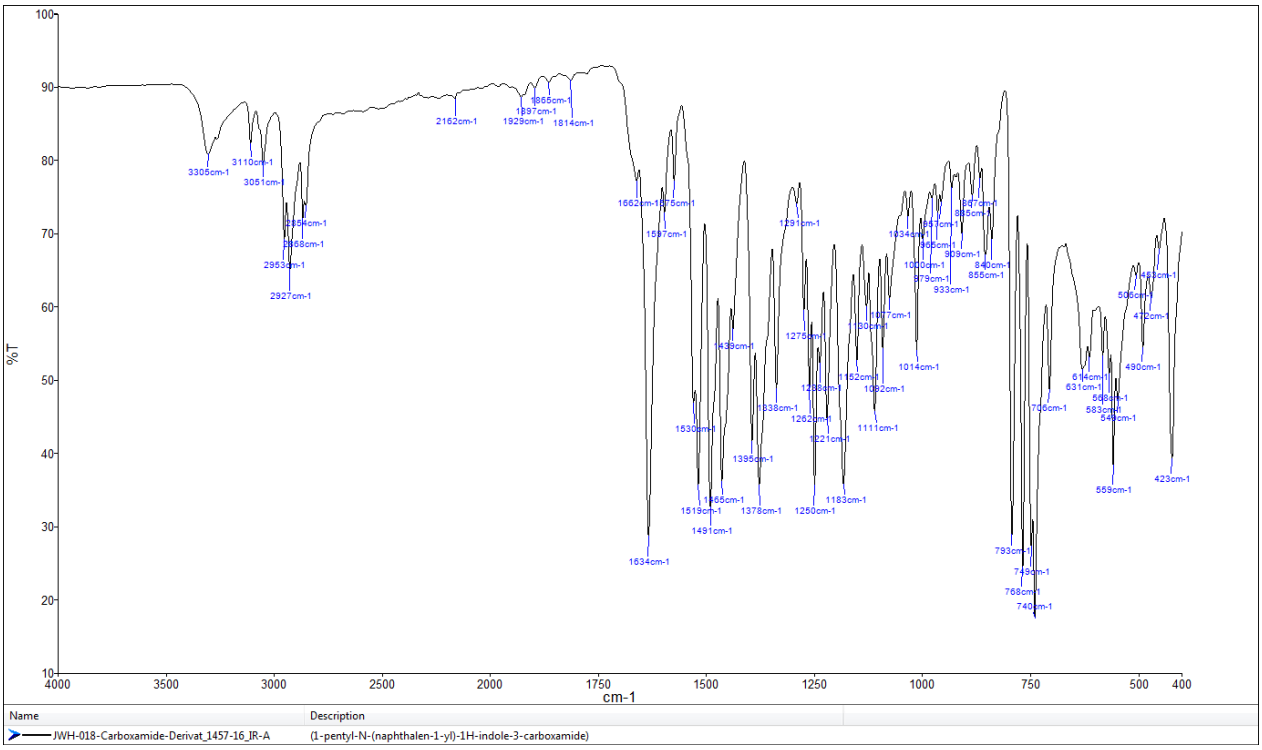
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 18,12 BP(1): 214; BP(2): 144,BP(3) :215,
HPLC-TOF	+	Exact mass (theoretical): 356,1889; measured value Δppm:-0,01; formula:C24H24N20
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		MS consistent by SWGDRUG.L and FTIR-ATR consistent by SWGDRUG-IR library entry
other		

ANALYTICAL RESULTS

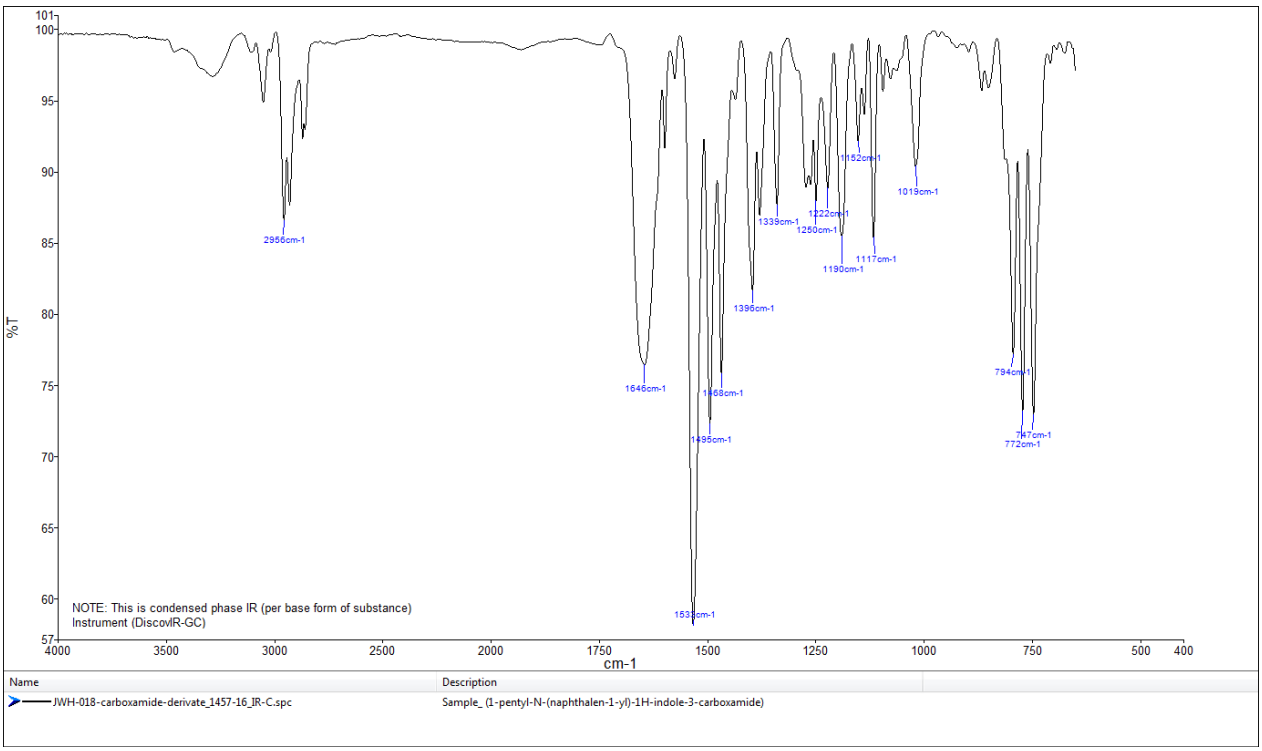
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

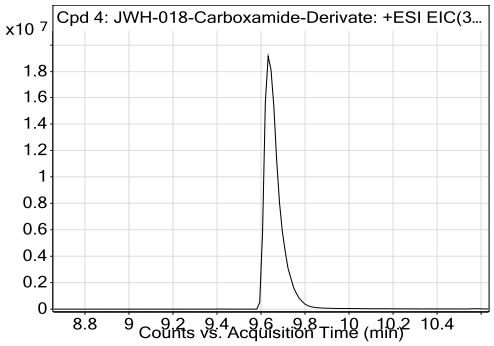
Data File	JWH-018-Carboxamide-Derivate_1457-16_TOF.d	**Sample Name**	ID_1457-16
Sample Type	Sample	**Position**	P1-F9
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-1512015-XDB-C18-ESI-poz-pod.m	**Acquired Time**	2/23/2016 12:51:56 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

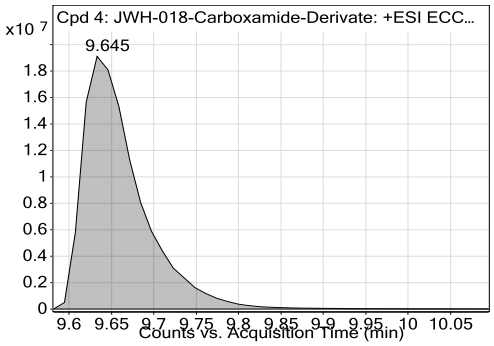
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 4: JWH-018-Carboxamide-Derivate	JWH-018-Carboxamide-Derivate	C24 H24 N2 O	9.645	356.1889
Cpd 5: C38 H39 N3 O2		C38 H39 N3 O2	10.986	569.3041

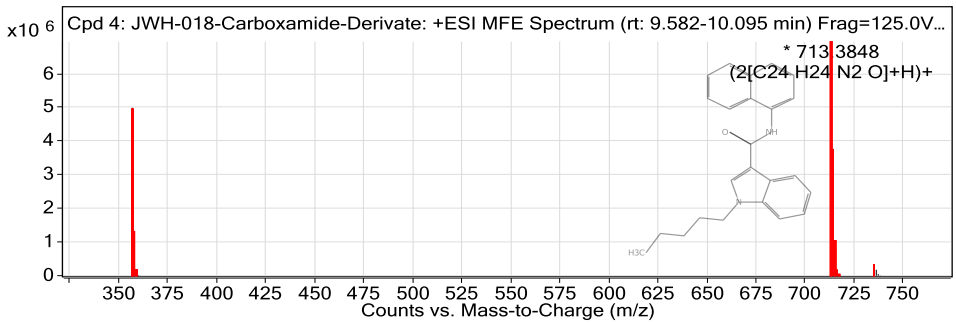
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
JWH-018-Carboxamide-Derivate	713.3848	9.645	356.1889	9.65	C24 H24 N2 O	356.1889	-0.01

Compound Chromatograms

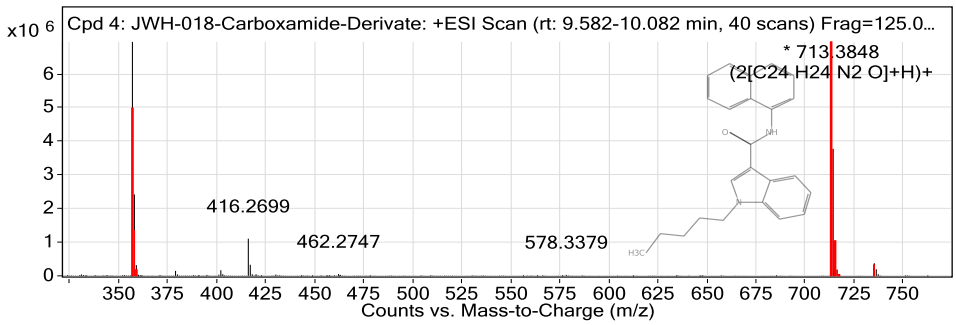




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

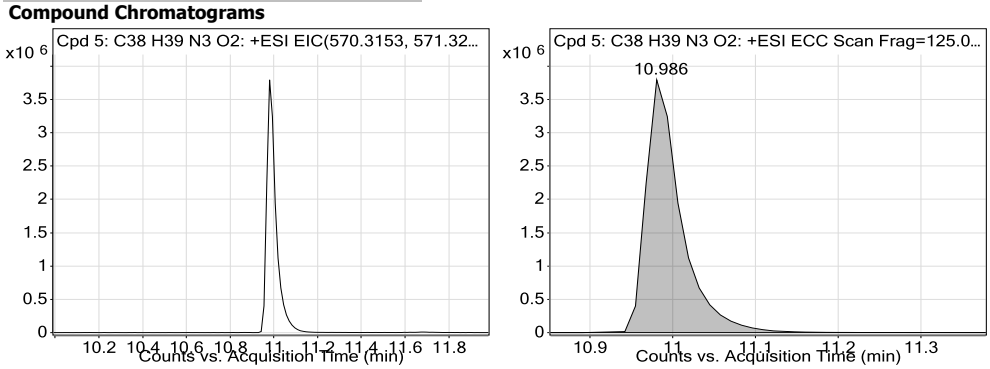
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
357.196	1	4987147.5	C24 H24 N2 O	(M+H)+
358.1997	1	1330453.35	C24 H24 N2 O	(M+H)+
359.2026	1	167737.25	C24 H24 N2 O	(M+H)+
713.3848	1	6949708	C24 H24 N2 O	(2M+H)+
714.3883	1	3766773.62	C24 H24 N2 O	(2M+H)+
715.3923	1	1048332.58	C24 H24 N2 O	(2M+H)+
716.3948	1	177534.46	C24 H24 N2 O	(2M+H)+
735.3672	1	335759.88	C24 H24 N2 O	(2M+Na)+

TOF REPORT

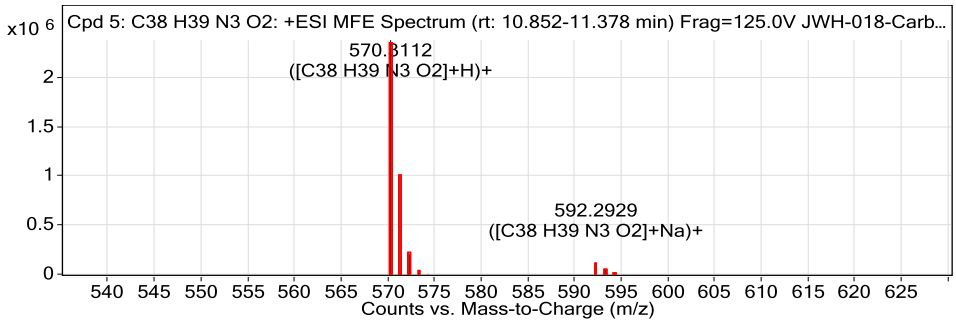
736.3701	1	174039.56	C24 H24 N2 O	(2M+Na)+
737.3729	1	43265.85	C24 H24 N2 O	(2M+Na)+

Obs. m/z	Obs. RT	Obs. Mass
570.3112	10.986	569.3041

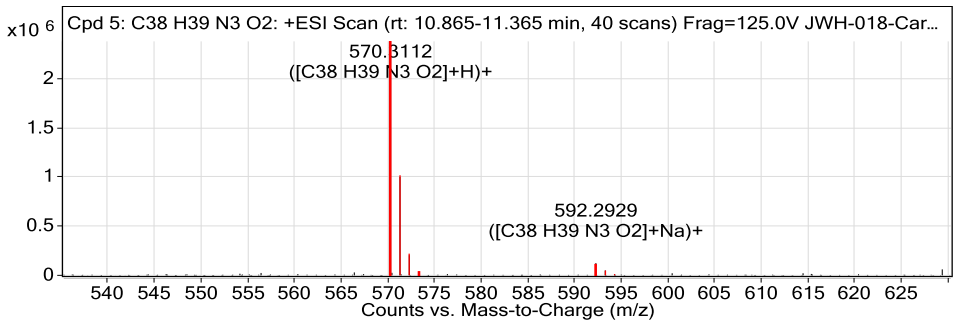
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum

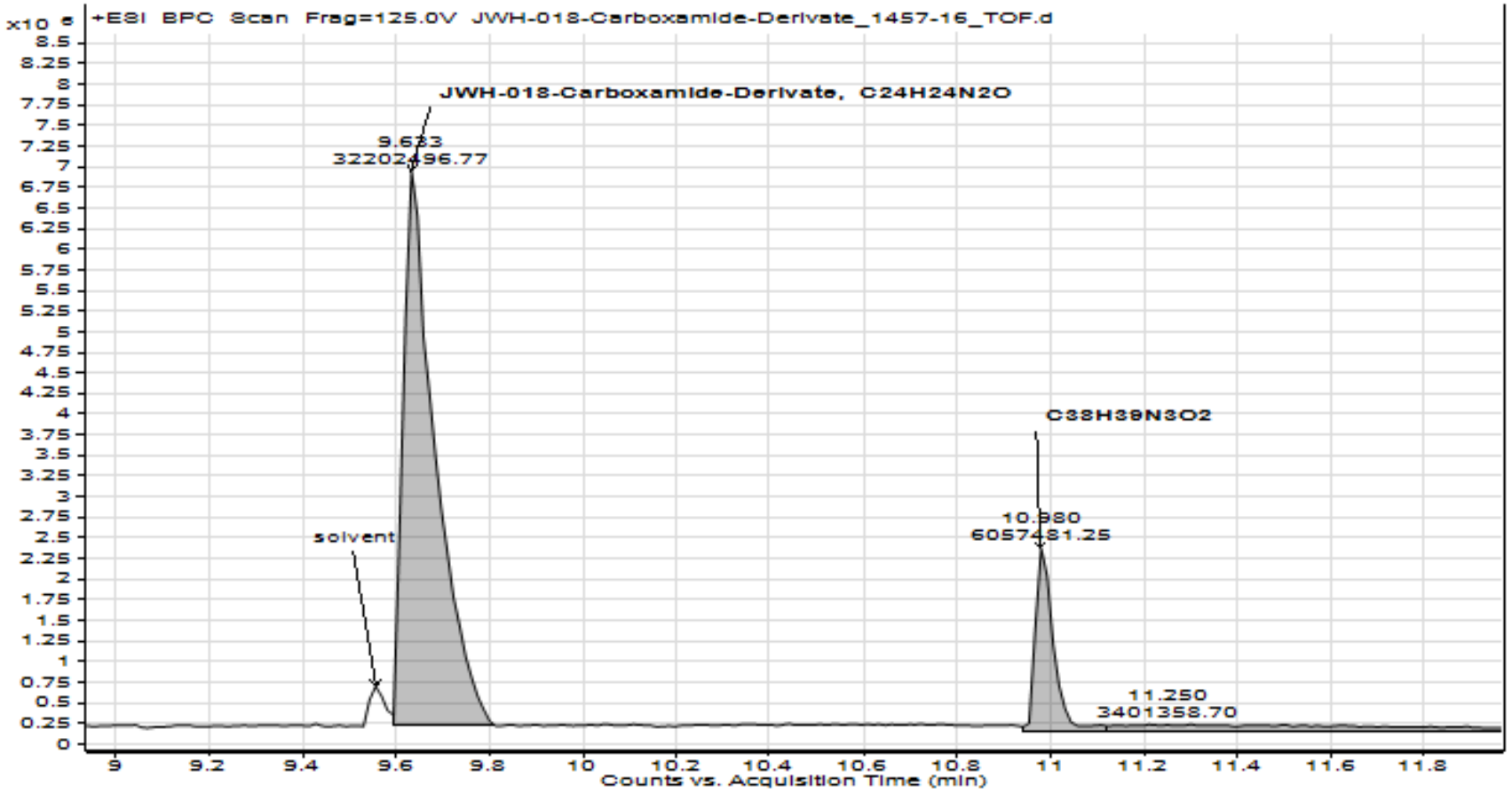


MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
570.3112	1	2377529.25	C38 H39 N3 O2	(M+H)+
571.3151	1	1000608.18	C38 H39 N3 O2	(M+H)+
572.3178	1	202896.29	C38 H39 N3 O2	(M+H)+
573.3205	1	27307.52	C38 H39 N3 O2	(M+H)+
574.3261	1	2842.68	C38 H39 N3 O2	(M+H)+
592.2929	1	113131.6	C38 H39 N3 O2	(M+Na)+
593.2958	1	44534.63	C38 H39 N3 O2	(M+Na)+
594.2997	1	9581.73	C38 H39 N3 O2	(M+Na)+
595.3056	1	1585.87	C38 H39 N3 O2	(M+Na)+

--- End Of Report ---

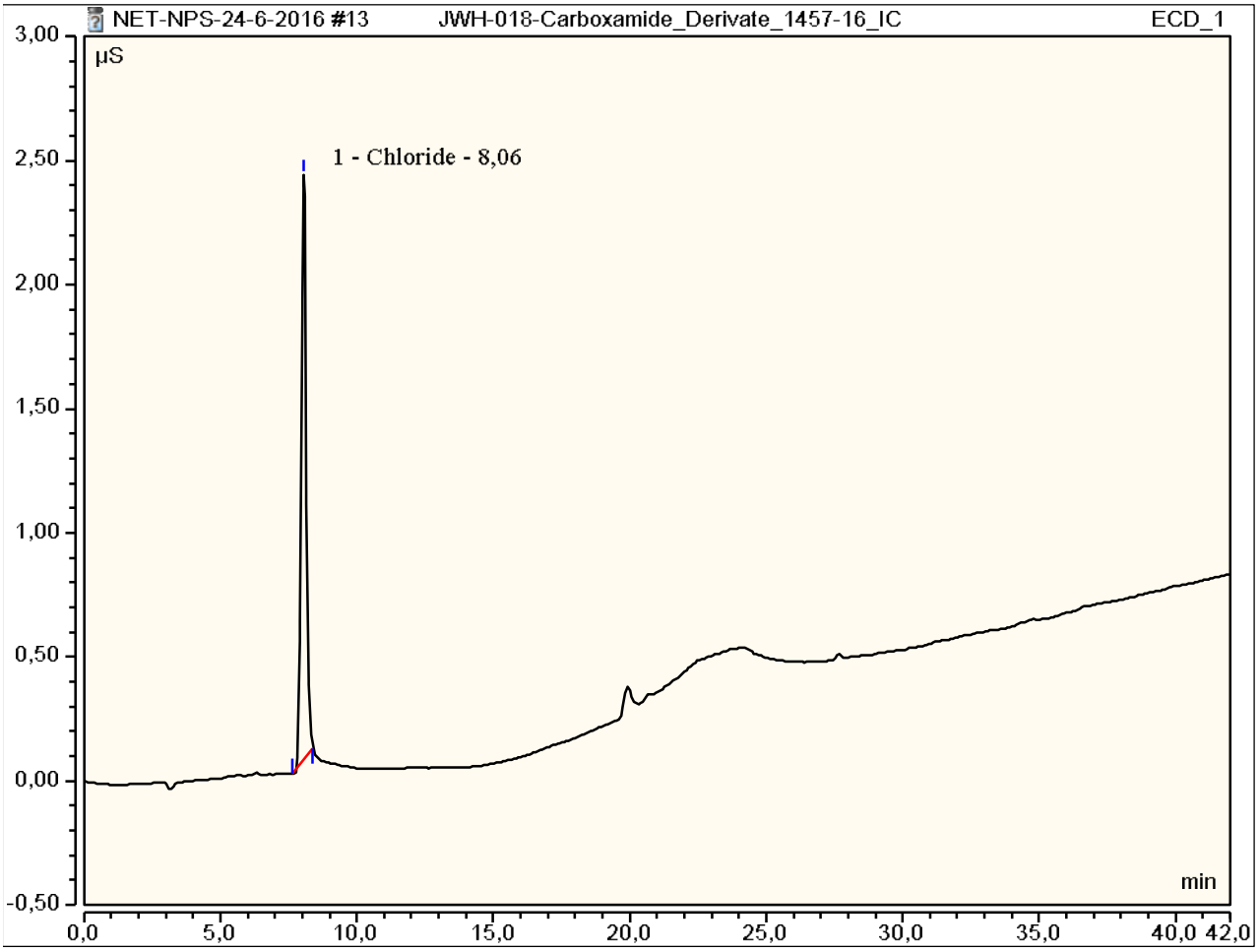
Sample Name	ID_1457-16	**Position**	P1-F9	**Instrument Name**	6230B TOF LC-MS	**User Name**	TG
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	JWH-018-Carboxamide-	**ACQ Method**	general-1512015-XDB-	**Comment**	extract in MeOH	**Acquired Time**	2/23/2016 12:51:56 PM



Peak Integration Report

Sample Name:	**JWH-018-Carboxamide_Derivate_1457-16_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**24-jun-2016 / 20:58**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	8,06	**Chloride**	BMB	0,48	2,36	n.a.
		TOTAL:		0,48	2,36	0,00



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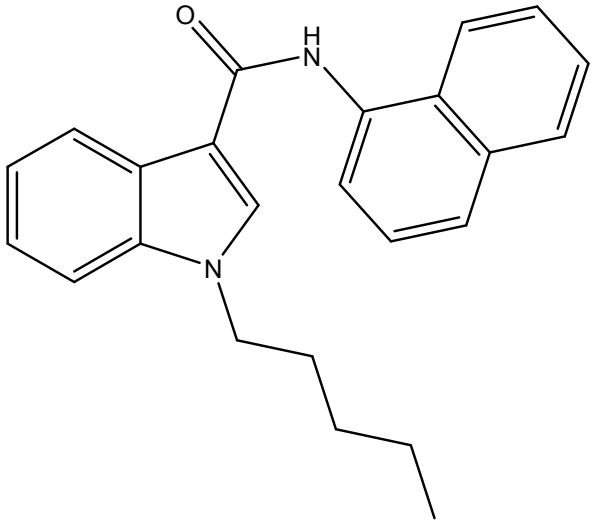
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1457-16**
Our notebook code:	P-1457-16
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
NMR experiments:	^{1}H, ^{13}C, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC, ^{1}H–^{15}N *gs*-HMBC.
Proposed structure:	
Chemical name:	*N*-(naphthalen-1-yl)-1-pentyl-1*H*-indole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure as it contains some minor impurities that are evident from ^{1}H and ^{13}C NMR.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	October 21, 2016

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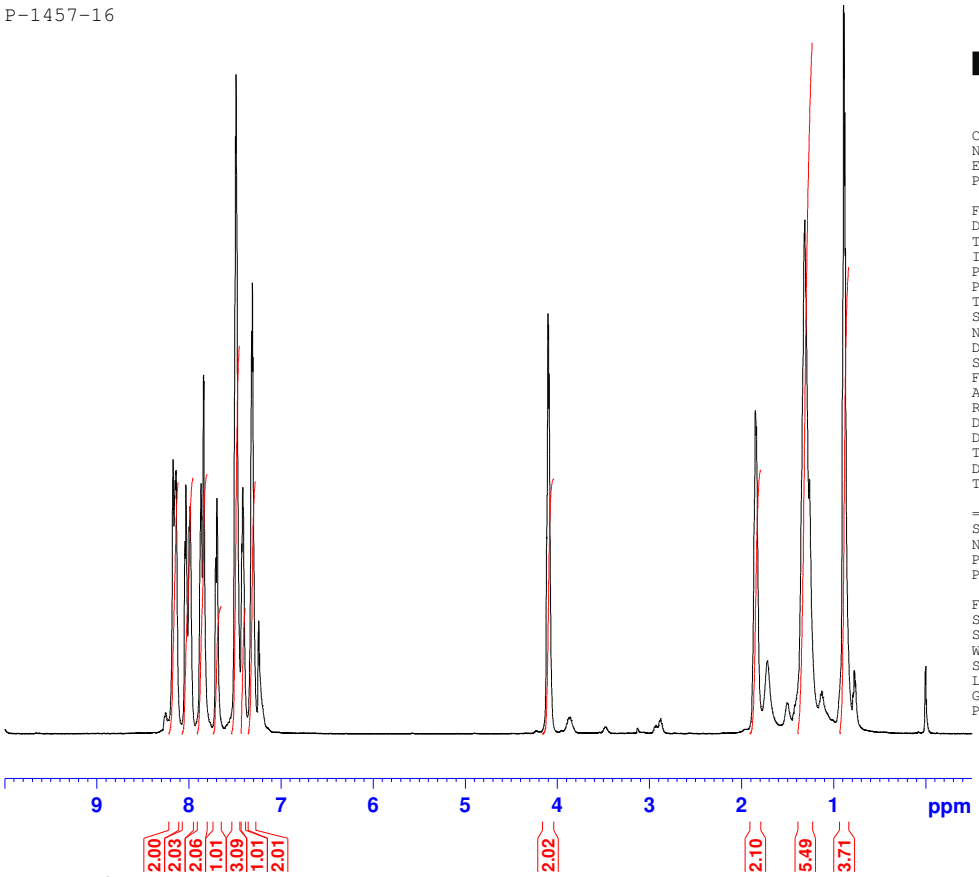
P-1457-16



Current Data Parameters
NAME p-1457-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160811
Time 4.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 64
DW 50.000 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

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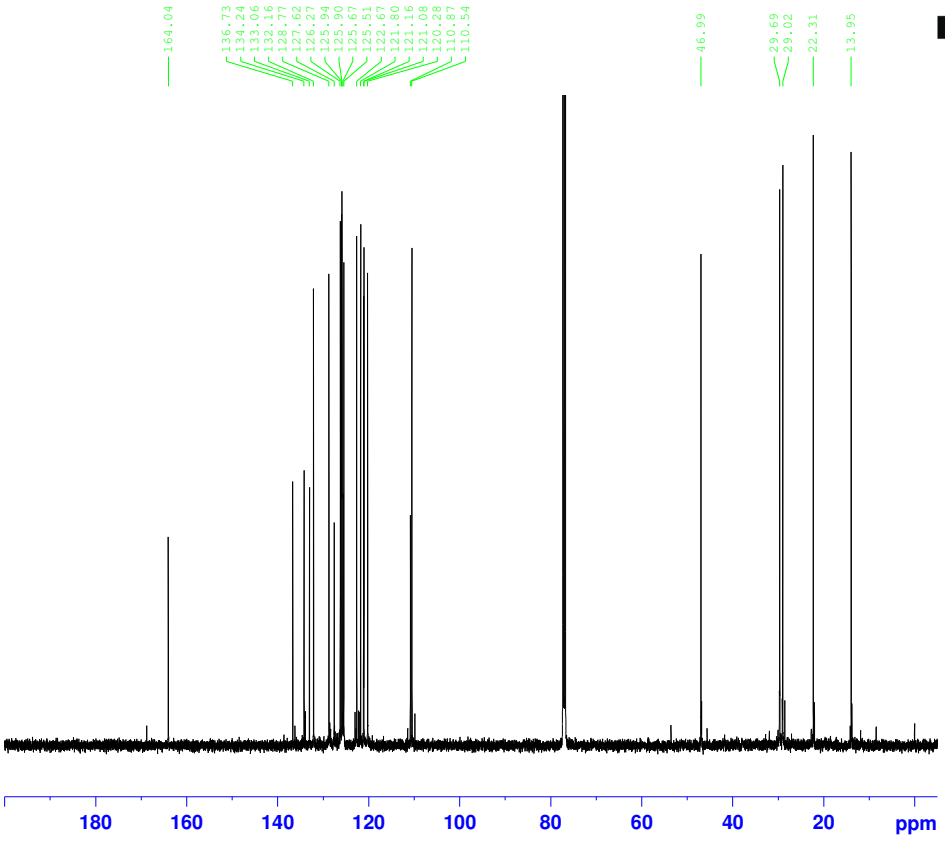
SFO1 500.1330885 MHz
NUC1 1H
P1 8.90 usec
PLW1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1457-16



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EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160811
Time 6.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
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 297.1 K
D1 2.00000000 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