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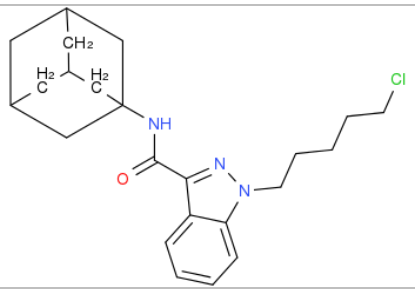
ANALYTICAL REPORT

5C-AKB48 (C23H30ClN3O)

N-(1-adamantyl)-1-(5-chloropentyl)-1H-indazole-3-carboxamide

Remark – other NPS detected: none

Sample ID:	1186-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Comments[1]:	
Date of entry into NFL database: http://www.policija.si/apps/nfl_response_web/seznam.php	7/13/2015

Substance identified-structure[2] (base form)	
Systematic name	N-(1-adamantyl)-1-(5-chloropentyl)-1H-indazole-3-carboxamide
Other names	
Formula (per base form)	C23H30ClN3O
M_w (g/mol)	399,96
Salt form	base
StdInChIKey	LGENBCKWDLSPTD-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	pure,

[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)
16/10/2015	Picture of structure and chemical name at page 1 corrected, "Smiles" replaced by StdInChlKey. Instrumental method (description) section amended,FTIR-condensed phase added, IC (anions) results added.

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 ^{0}C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 293 ^{0}C at a rate of 18 ^{0}C/min, hold for 6.1 min, than heating at 50 ^{0}C/min up to 325 ^{0}C and finally 2.8 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 (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 ^{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 (40) to 550 amu.
IR (condesed 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

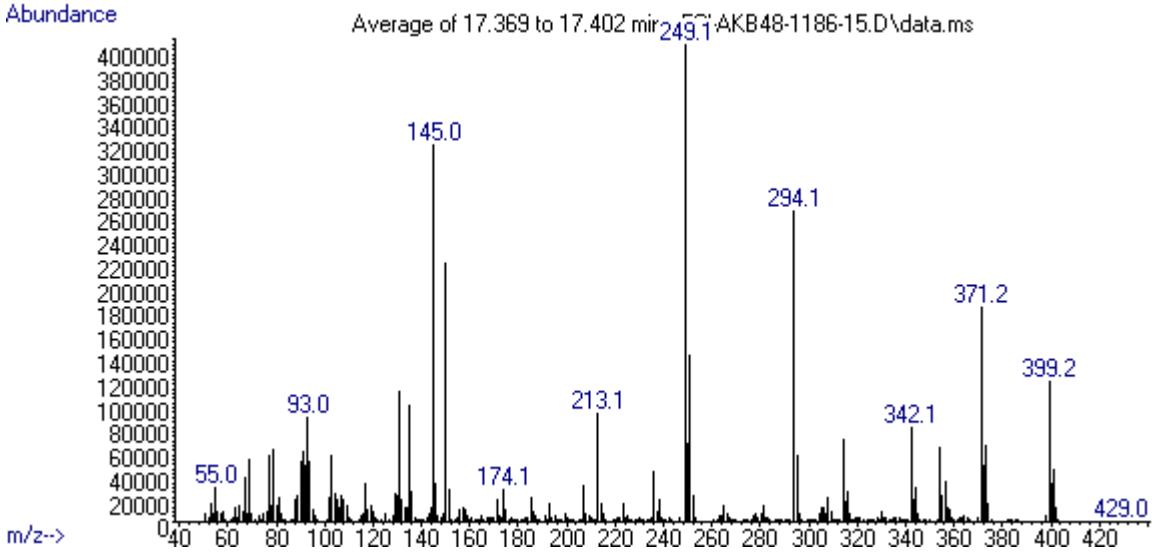
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	partial
H_2O	low

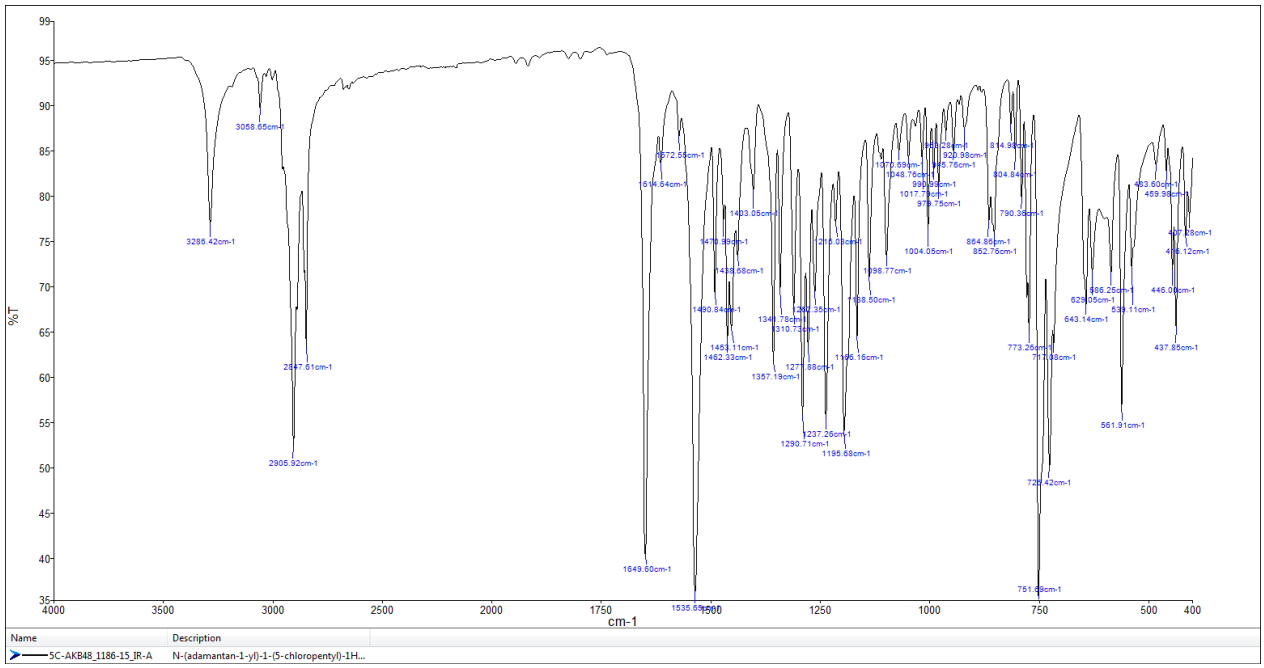
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 17,39 BP(1): 249; BP(2): 145,BP(3) :294,
HPLC-TOF	+	Exact mass (theoretical): 399,2077; measured value Δppm:-0,07; formula:C23H30ClN3O
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR	+	
validation		
other		

ANALYTICAL RESULTS

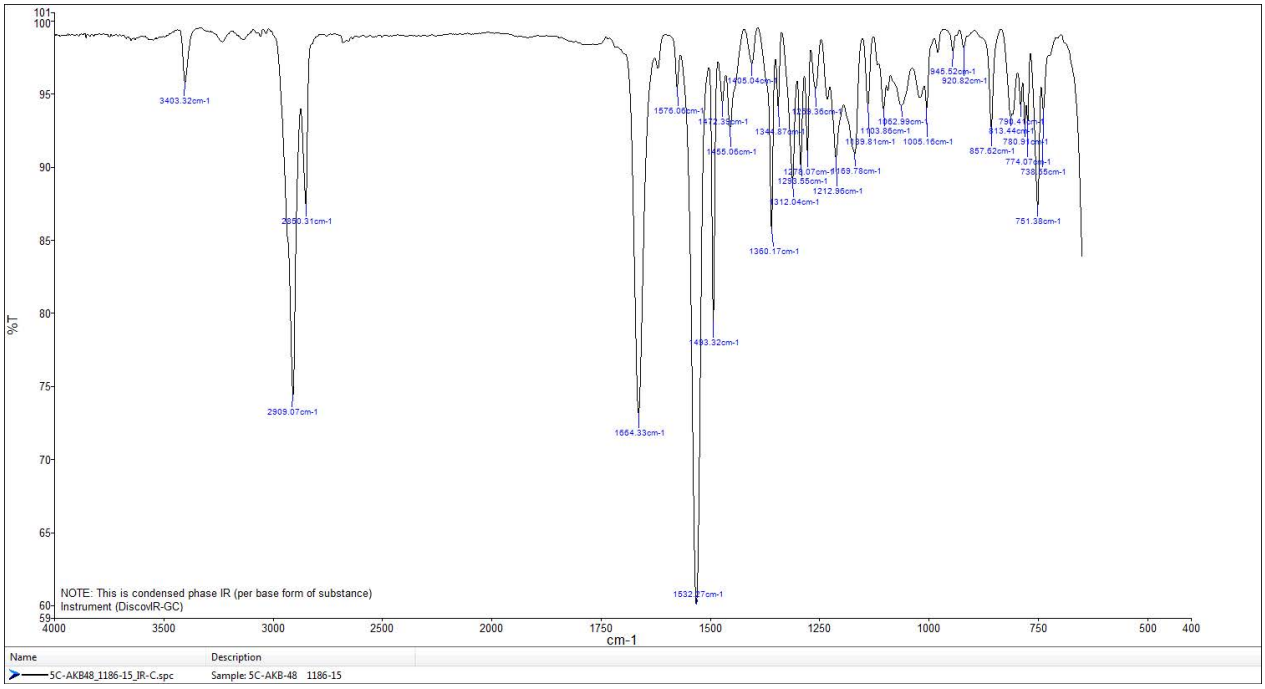
MS (EI)



FTIR-ATR - direct measurement



IR (condensed phase)



Target Compound Screening Report

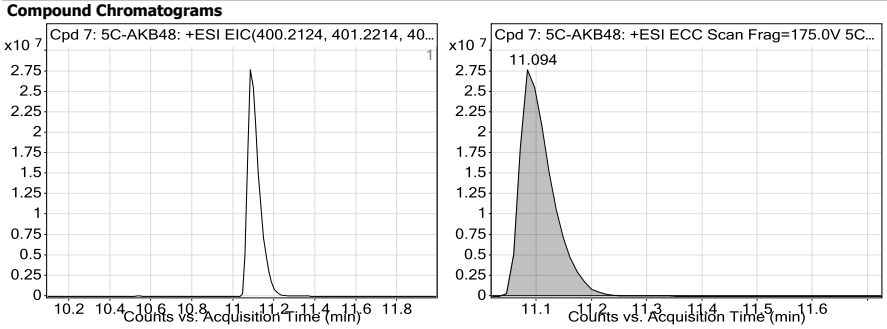
Data File	5C_AKB48_1186-15_TOF.d	**Sample Name**	5C-AKB48
Sample Type	Sample	**Position**	P1-C8
Instrument Name	SG13170002	**User Name**	
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	7/8/2015 10:44:06 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

Compound Table

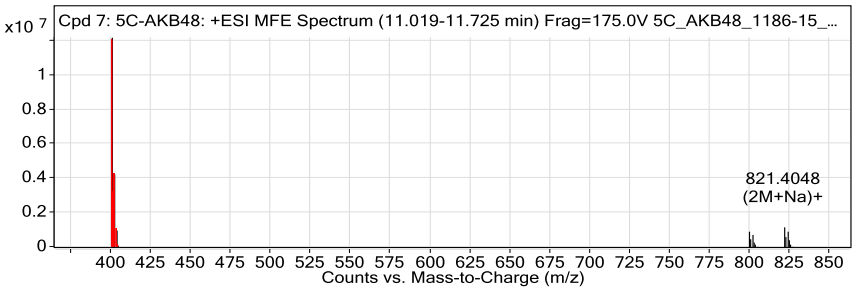
Label	Tgt Name	MFG Formula	Tgt Formula	Obs. RT	Obs. Mass
Cpd 7: 5C-AKB48	5C-AKB48	C23 H30 Cl N3 O	C23 H30 Cl N3 O	11.094	399.2078

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error	Tgt Formula	Find Cpds Algorith
5C-AKB48	400.215	11.094	399.2078	11.094	C23 H30 Cl N3 O	399.2077	-0.07	C23 H30 Cl N3 O	Find by Molecular Feature

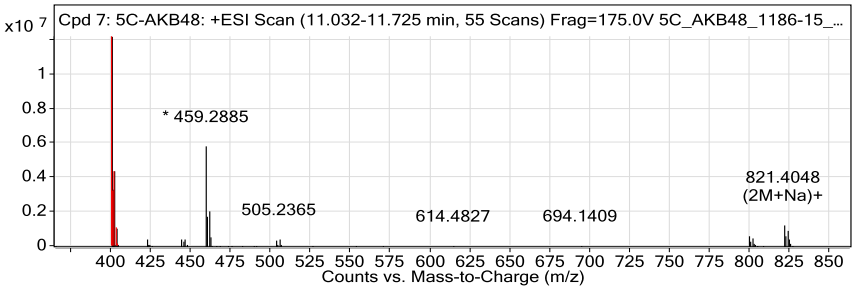
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

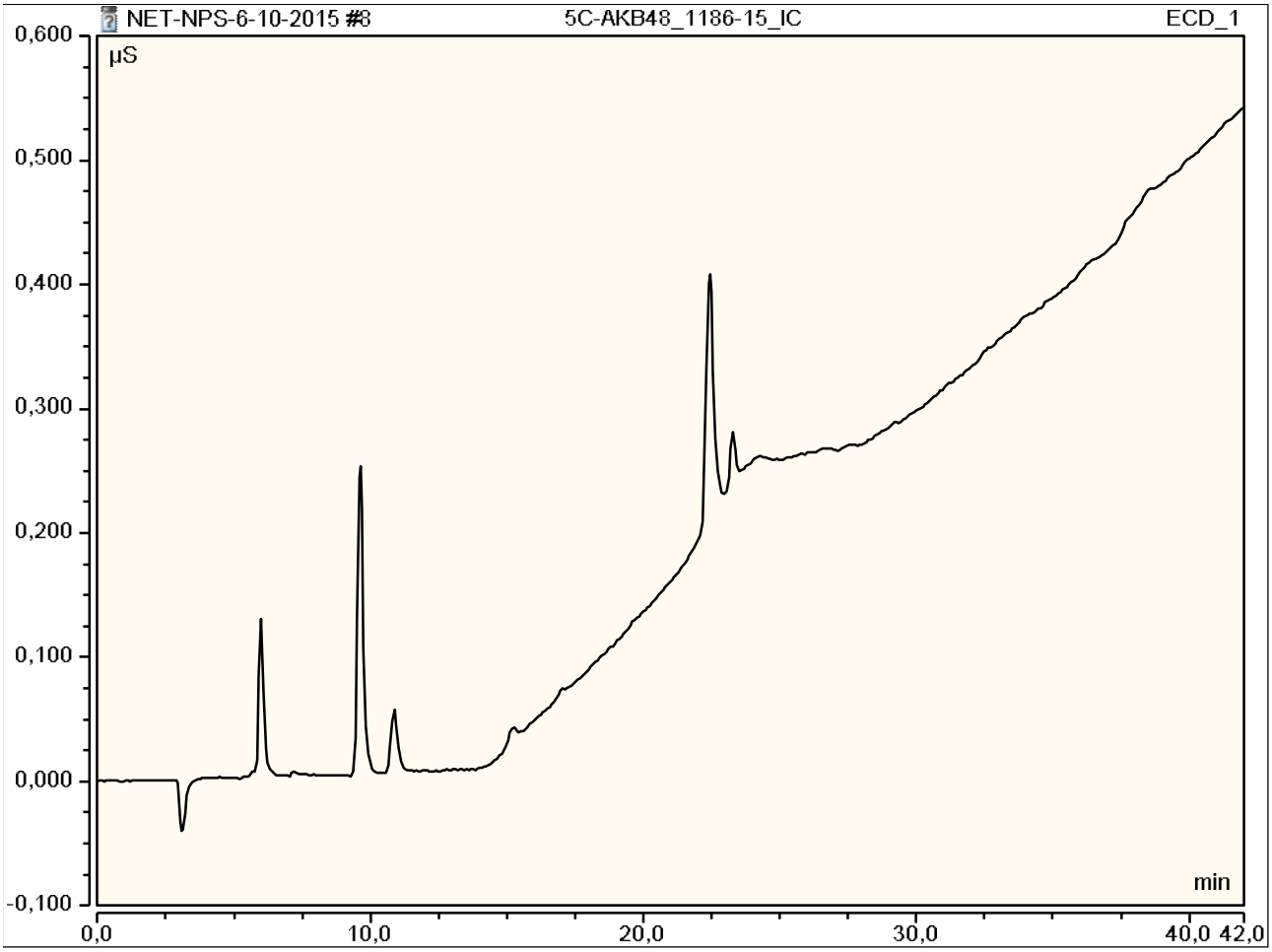
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
400.215	1	12185254	C23 H30 Cl N3 O	(M+H)+
401.2182	1	3188757.28	C23 H30 Cl N3 O	(M+H)+
402.2131	1	4261161.38	C23 H30 Cl N3 O	(M+H)+
403.216	1	988630.58	C23 H30 Cl N3 O	(M+H)+
799.4232	1	952235.31		(2M+H)+
800.4264	1	486076.77		(2M+H)+
801.4221	1	730796.16		(2M+H)+
821.4048	1	1214960.5		(2M+Na)+
822.4083	1	618783.8		(2M+Na)+
823.404	1	935063.99		(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**5C-AKB48_1186-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**16-okt-2015 / 12:34**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount n.a.
		TOTAL:		0,00	0,00	0,00



note: low intensity of peaks observed (the level of ultrapure water for sample preparation)

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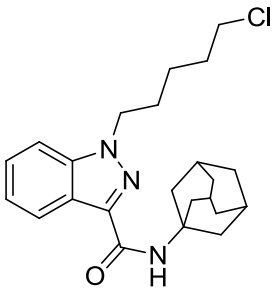
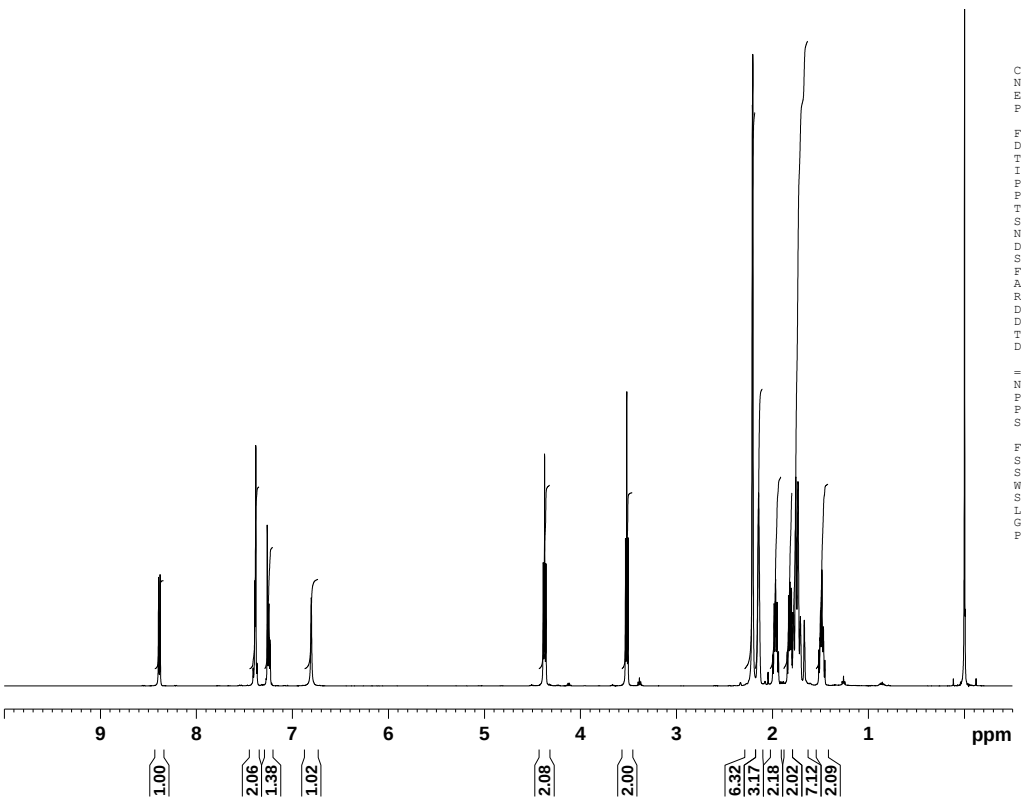
REPORT

Sample ID:	**1186-15**
Our notebook code:	P-1186-15
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 with chemical name:	 *N*-(adamantan-1-yl)-1-(5-chloropentyl)-1*H*-indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj
Date of report:	July 11, 2015

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 publication are the sole responsibility of the Author and can in no way be taken to reflect the views of the European Commission.

P-1186-15



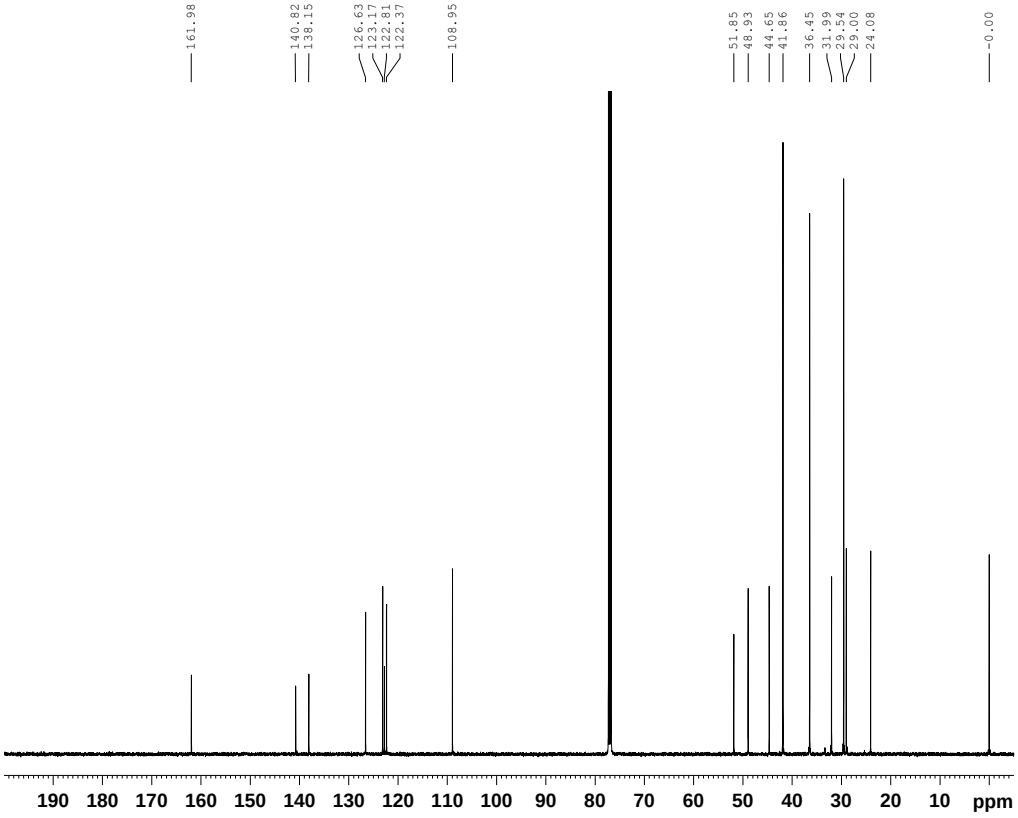

```
Current Data Parameters
NAME                  1186-15
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150707
Time                    3.35
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                     71.8
DW                  48.400 usec
DE                   6.50 usec
TE                  296.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.90 usec
PLW1           26.00000000 W
SFO1           500.1330885 MHz

F2 - Processing parameters
SI                    65536
SF               500.1300119 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

P-1186-15




```
Current Data Parameters
NAME                  1186-15
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150707
Time                    6.07
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     3072
DS                        4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ               1.1010548 sec
RG                     2050
DW                  16.800 usec
DE                   6.50 usec
TE                  296.0 K
D1              1.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.00 usec
PLW1          122.00000000 W
SFO1           125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PLW2           26.00000000 W
PLW12           0.32179001 W
PLW13           0.20595001 W
SFO2           500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF               125.7577915 MHz
WDW                      EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```