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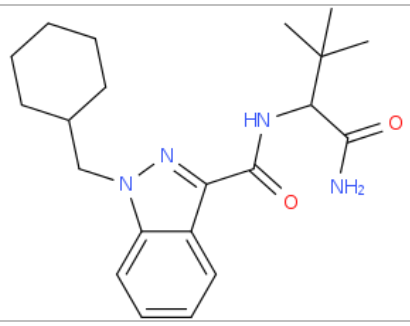
ANALYTICAL REPORT[1]

ADB-CHMINACA (C21H30N4O2)

N-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide

Remark – other NPS detected: ?

Sample ID:	1380-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	11/27/2015
Date of entry (M/D/Y) into NFL database:	2/11/2016
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-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide
Other names	MAB-CHMINACA
Formula (per base form)	C21H30N4O2
M_w (g/mol)	370,5
Salt form/anions detected	base
StdInChIKey	ZWCCSIUBHCZKOY-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	?
Add.info (purity..)	not pure, TOF: 5%-10% C17H23N3O Mw=285.2 and cca 25% C28H42N4O2 Mw= 466.7 (ADB-CHMINACA + possibly cyclohexylmethyl)

[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 ^{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 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) 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 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

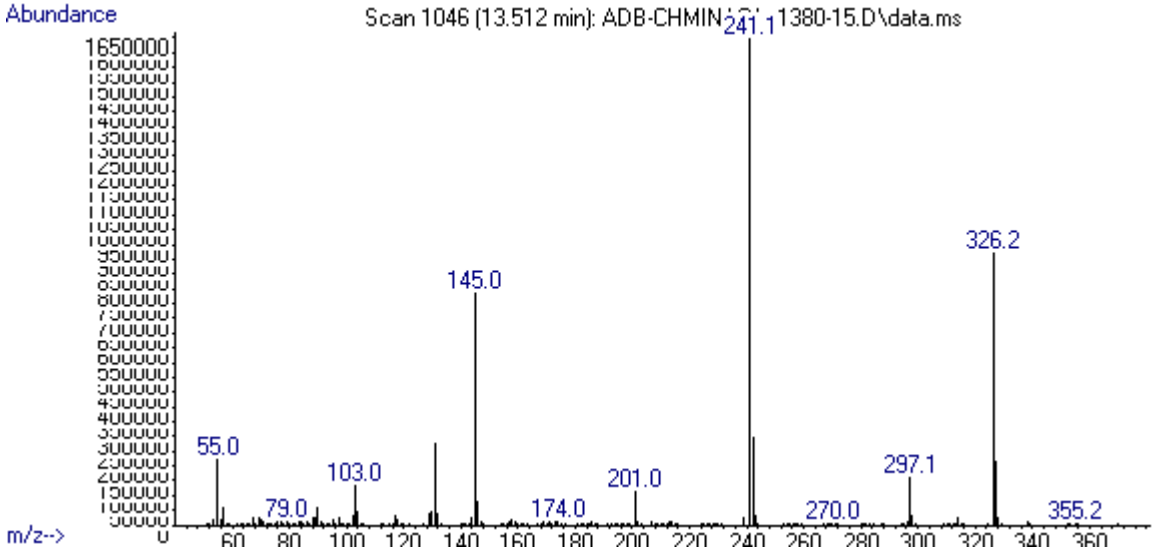
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	low (bad)

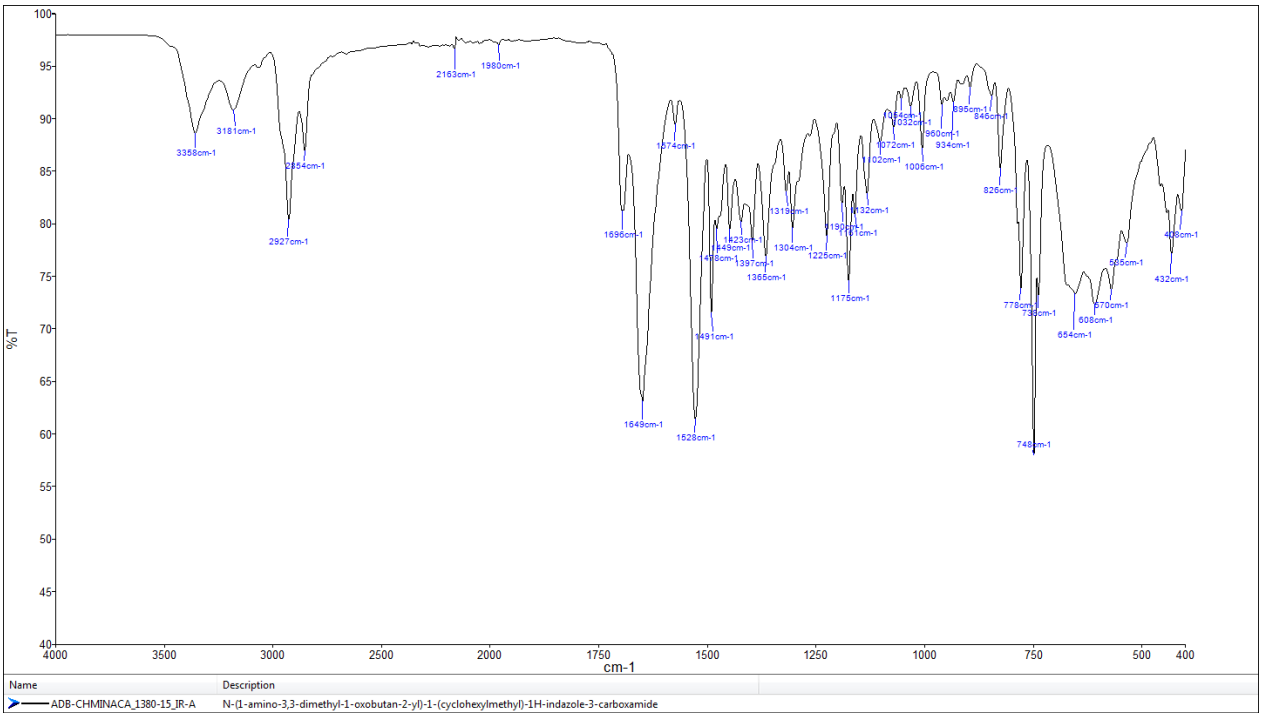
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,51 BP(1): 241; BP(2): 326,BP(3) :145,
HPLC-TOF	+	Exact mass (theoretical): 370,2369; measured value Δppm:0,29; formula:C21H30N4O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form		
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

MS (EI)



FTIR-ATR - direct measurement (sample as received)



TOF REPORT

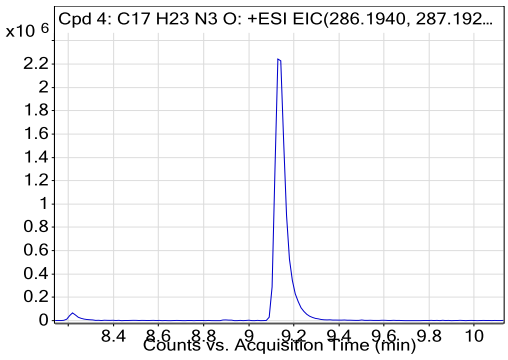
Data File	_1380-15_TOF.d	**Sample Name**	ID_1380-15
Sample Type	Sample	**Position**	P1-E3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-17112015-XDB-C18-ESI-poz.m	**Acquired Time**	11/30/2015 11:39:40 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

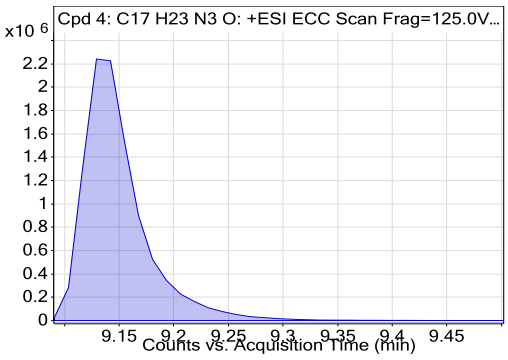
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 4: C17 H23 N3 O		C17 H23 N3 O	9.138	285.1842
Cpd 8: ADB-CHMINACA	ADB-CHMINACA	C21 H30 N4 O2	9.735	370.2368
Cpd 9: ADB-CHMINACA+cyclohexylmethyl	ADB-CHMINACA+cyclohexylmethyl	C28 H42 N4 O2	11.482	466.3306

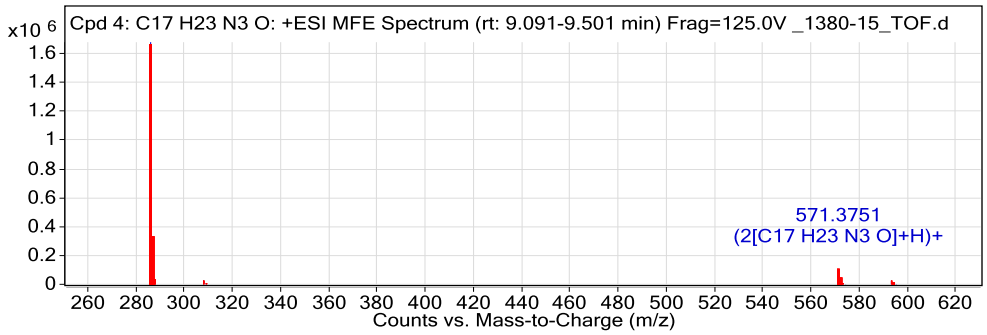
Obs. m/z	Obs. RT	Obs. Mass	DB Formula
286.1915	9.138	285.1842	C17 H23 N3 O

Compound Chromatograms

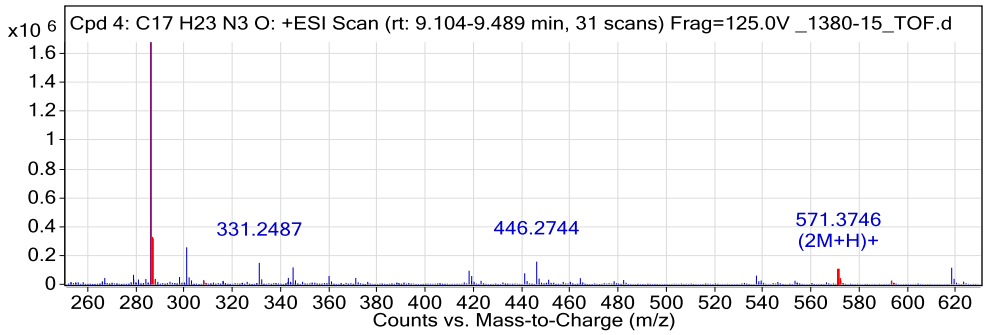




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



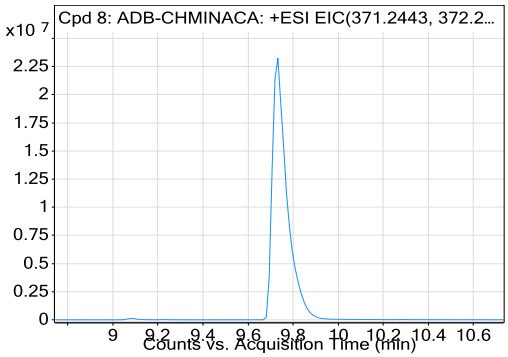
MS Spectrum Peak List

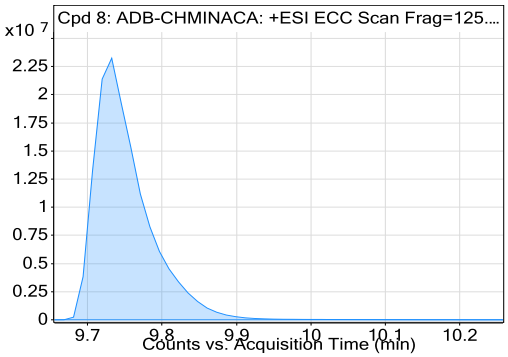
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
286.1915	1	1678717.13	C17 H23 N3 O	(M+H)+
287.1946	1	314255.17	C17 H23 N3 O	(M+H)+
288.197	1	31688.55	C17 H23 N3 O	(M+H)+
308.1729	1	26911.19	C17 H23 N3 O	(M+Na)+
309.1873	1	7148.36	C17 H23 N3 O	(M+Na)+
571.3751	1	103151.05	C17 H23 N3 O	(2M+H)+

572.3778	1	38200.15	C17 H23 N3 O	(2M+H)+
573.3797	1	7498.02	C17 H23 N3 O	(2M+H)+
593.3563	1	25585.18	C17 H23 N3 O	(2M+Na)+
594.3588	1	10046.94	C17 H23 N3 O	(2M+Na)+

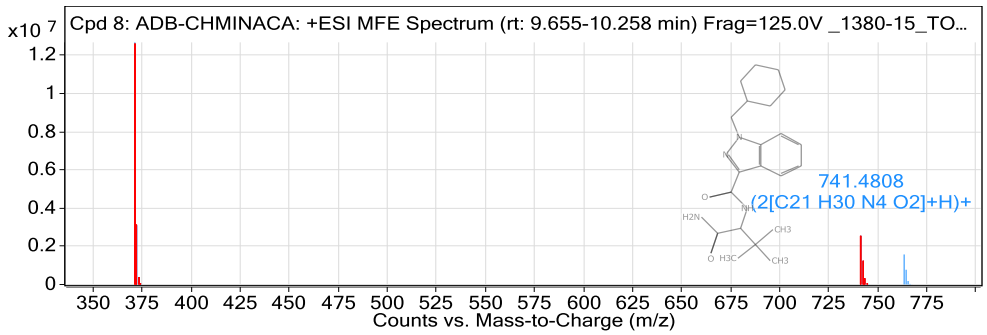
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)
ADB-CHMINACA	371.244	9.735	370.2368	C21 H30 N4 O2	370.2369	0.29

Compound Chromatograms

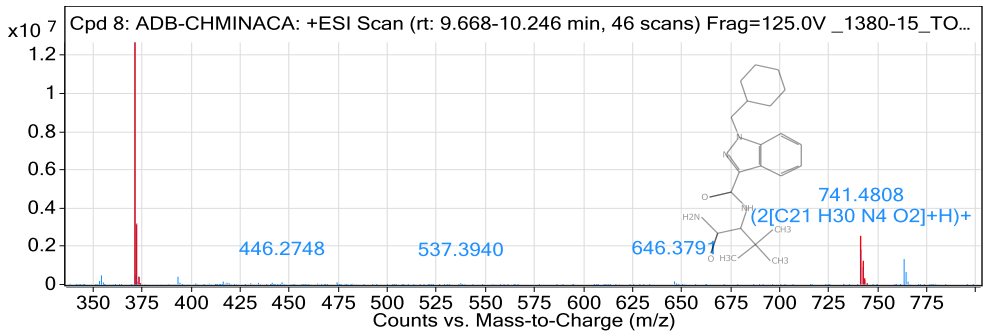




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



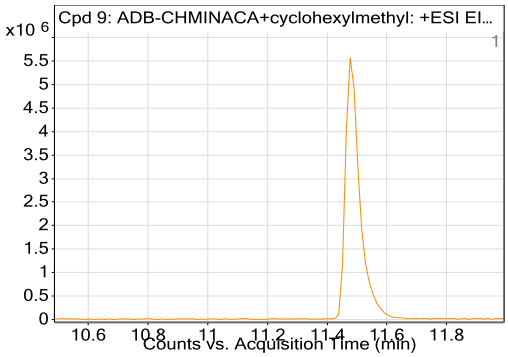
MS Spectrum Peak List

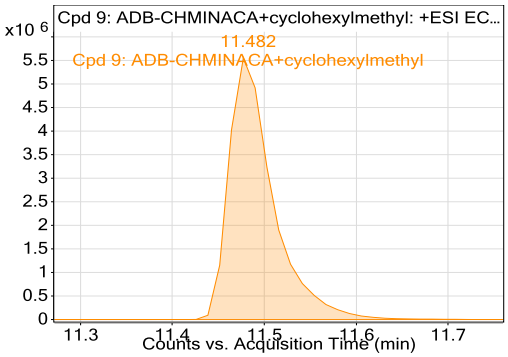
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
371.244	1	12676935	C21 H30 N4 O2	(M+H)+
372.2472	1	3101679.65	C21 H30 N4 O2	(M+H)+
373.2505	1	382224.99	C21 H30 N4 O2	(M+H)+
741.4808	1	2557024	C21 H30 N4 O2	(2M+H)+
742.4846	1	1253035.45	C21 H30 N4 O2	(2M+H)+
743.4873	1	311723.92	C21 H30 N4 O2	(2M+H)+
744.4892	1	48883.96	C21 H30 N4 O2	(2M+H)+
763.4632	1	1572329		(2M+Na)+
764.4666	1	772374.49		(2M+Na)+
765.4689	1	182463.99		(2M+Na)+

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)
ADB-CHMINACA+cyclohexylm	467.3378	11.482	466.3306	C28 H42 N4 O2	466.3308	0.29

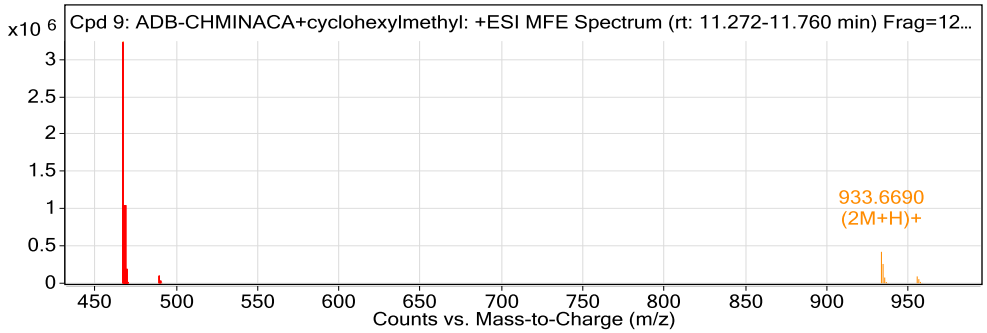
Compound Chromatograms

TOF REPORT

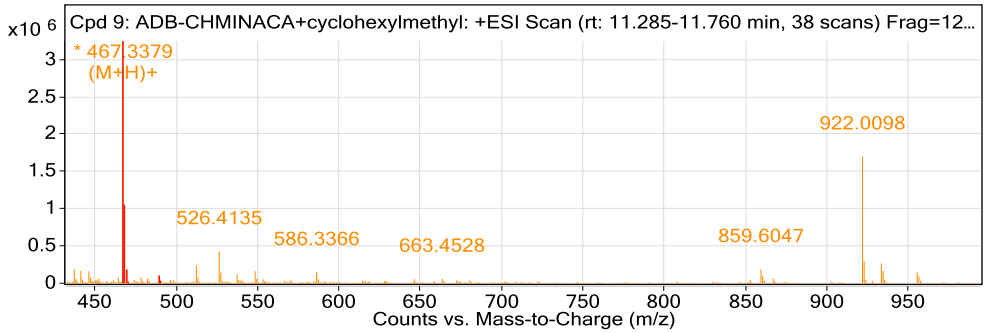




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

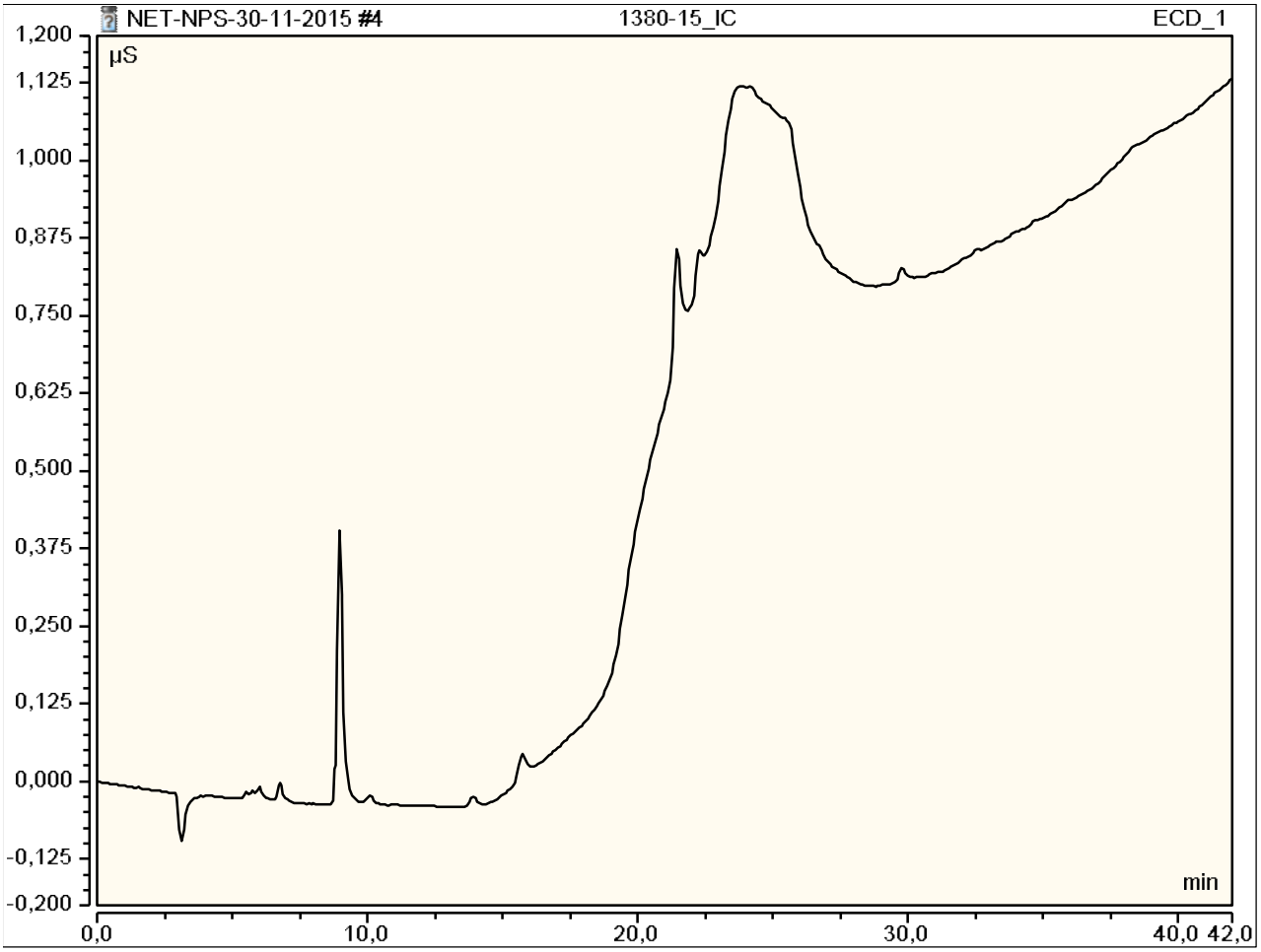
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
467.3378	1	3246845	C28 H42 N4 O2	(M+H)+
468.3417	1	1038963.92	C28 H42 N4 O2	(M+H)+
469.344	1	160312.13	C28 H42 N4 O2	(M+H)+
489.3199	1	96760.49	C28 H42 N4 O2	(M+Na)+
490.3228	1	30438.71	C28 H42 N4 O2	(M+Na)+
933.669	1	421152.34		(2M+H)+
934.6718	1	258315.07		(2M+H)+
935.6742	1	76454.2		(2M+H)+
955.6549	1	91414.07		(2M+Na)+
956.6573	1	54786.01		(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**1380-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**30-nov-2015 / 16:31**	*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



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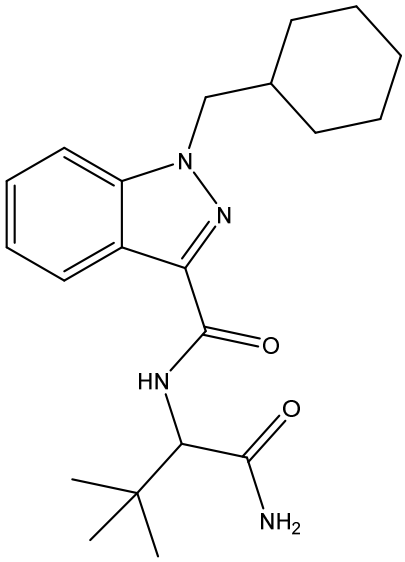
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1380-15**
Our notebook code:	P-1380-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO-d_6
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-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure by NMR, contains the same impurity as P-1280-15 (evident from ^{13}C NMR peaks at 46.1, 31.7 and 26.0) and possibly other impurities as well.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 21, 2015

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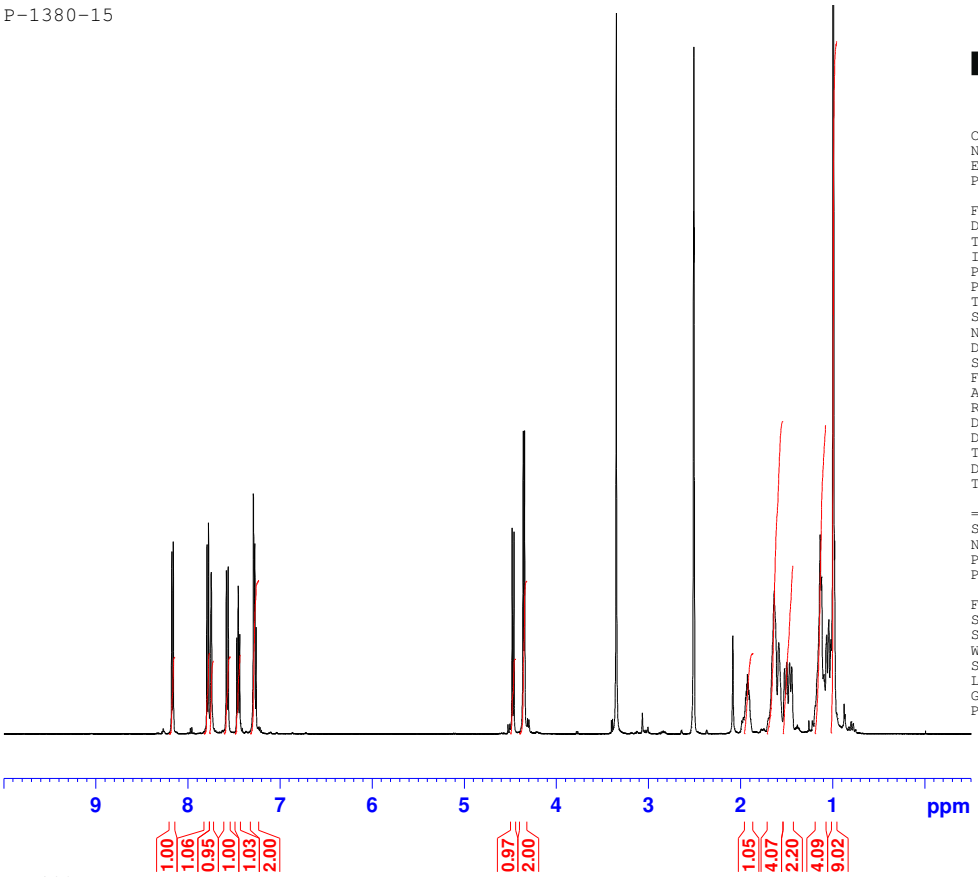
P-1380-15



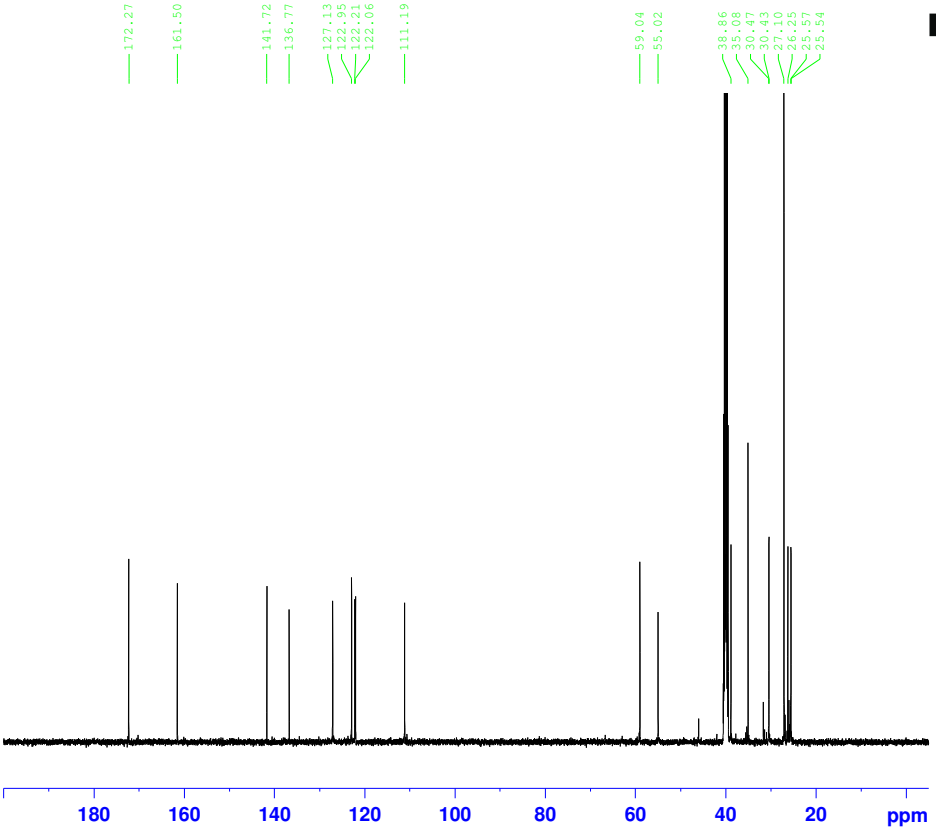

```
Current Data Parameters
NAME          P-1380-15
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20151215
Time               3.51
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                   57
DW              50.000 usec
DE               6.50 usec
TE              296.0 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-1380-15




```
Current Data Parameters
NAME          P-1380-15
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20151215
Time               5.13
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
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              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       122.00000000 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
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