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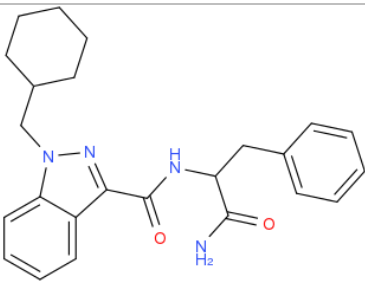
ANALYTICAL REPORT¹

APP-CHMINACA_(PX3) (C₂₄H₂₈N₄O₂)

N-(1-amino-1-oxo-3-phenylpropan-2-yl)-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide

Remark – other NPS detected: possibly reaction by-product Mw=500 (not identified)

Sample ID:	1280-15
Sample description:	powder - yellowish
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	9/18/2015
Date of entry (M/D/Y) into NFL database:	10/4/2015
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-(1-amino-1-oxo-3-phenylpropan-2-yl)-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide
Other names	PX3
Formula (per base form)	C ₂₄ H ₂₈ N ₄ O ₂
M _w (g/mol)	404,5
Salt form	base
StdInChIKey	DMHWDSGURMXMGE-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	?
Add.info (purity..)	two additional organic compounds detected (one by TOF and MS:Mw=500; aprox. 15 %, and one by TOF Mw=263 approx. 5%); For the compound at Mw=500 - empirical formula C ₃₁ H ₄₀ N ₄ O ₂ we suspect that additional -CH ₂ -cyclohexan group is attached to PX3 molecule. Substance Mw=263 with the (suggested formula C ₁₆ H ₁₃ N ₄ O ₂

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

	(unidentified).
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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 °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 2.8 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. (method run time enlarged for 10 minutes).

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

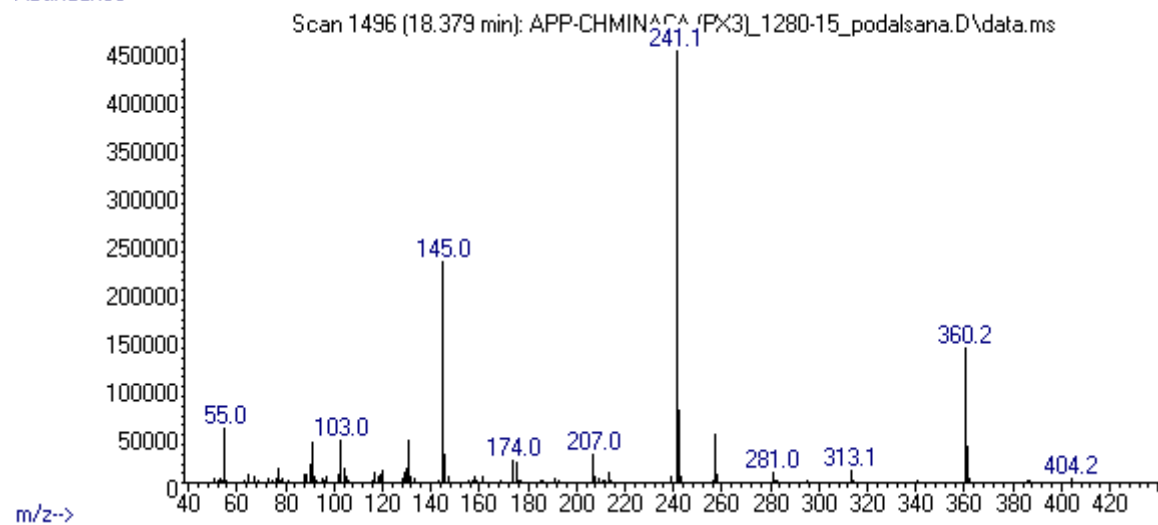
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 18,45 BP(1): 241; BP(2): 145,BP(3) :360,
HPLC-TOF	+	Exact mass (theoretical): 404,2212; measured value Δppm:-2,81; formula:C ₂₄ H ₂₈ N ₄ O ₂
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

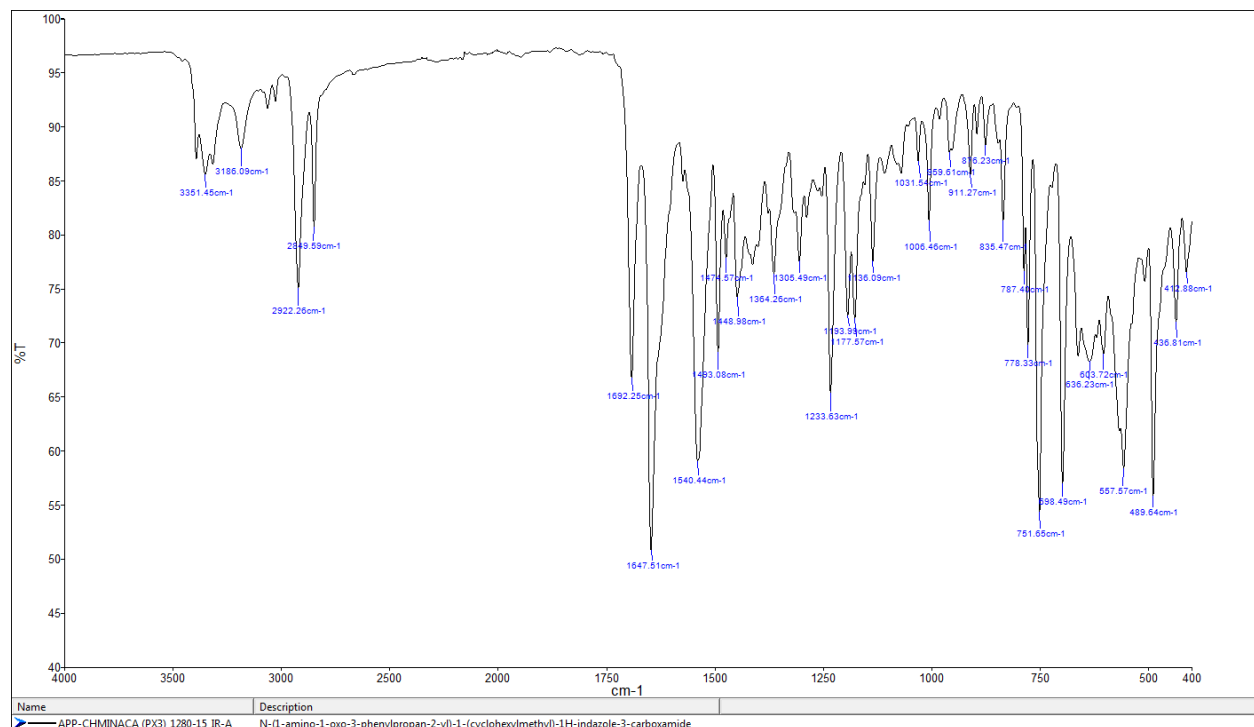
ANALYTICAL RESULTS

MS (EI)

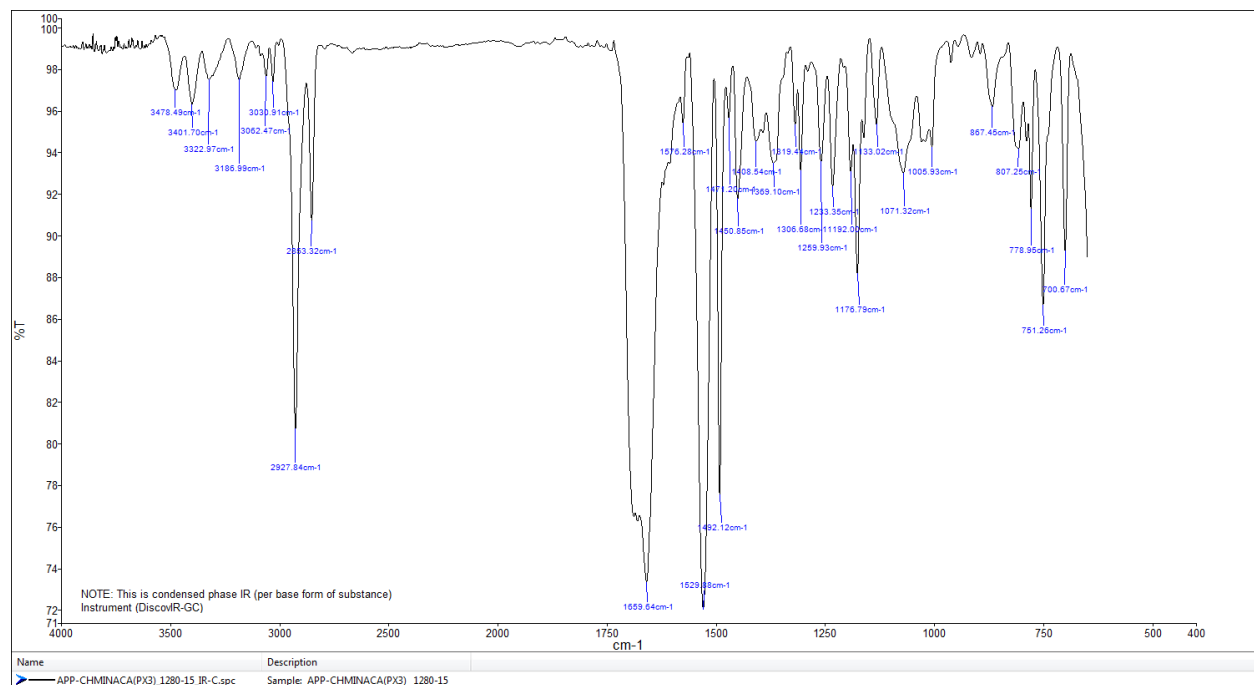
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

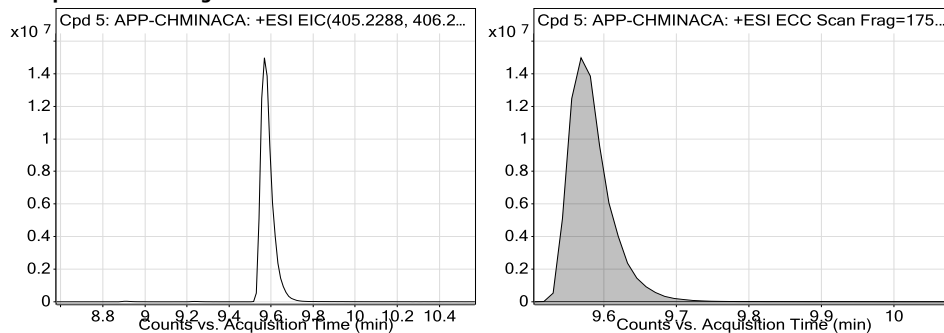
Data File	APP-CHMINACA_1280-15_TOF.d	Sample Name	APP-CHMINACA (PX3)
Sample Type	Sample	Position	P1-E5
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	Acquired Time	9/21/2015 10:12:32 AM
IRM Calibration Status	Success	DA Method	Droge_Default.m
Comment	extract in MeOH		

Compound Table

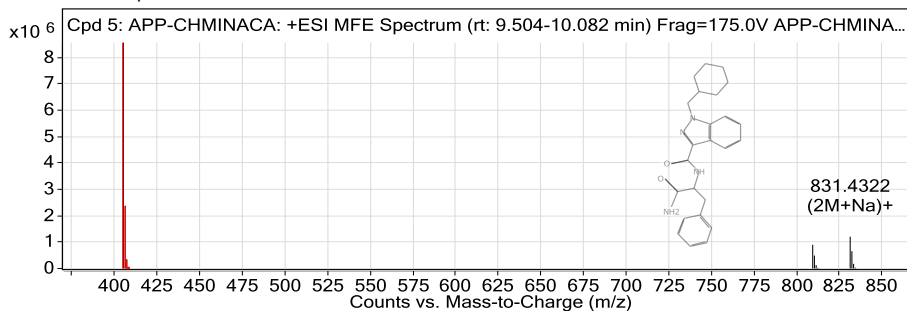
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 5: APP-CHMINACA	APP-CHMINACA	9.573	404.2224

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
APP-CHMINACA	405.2298	9.573	404.2224	9.6	C24 H28 N4 O2	404.2212	-2.81

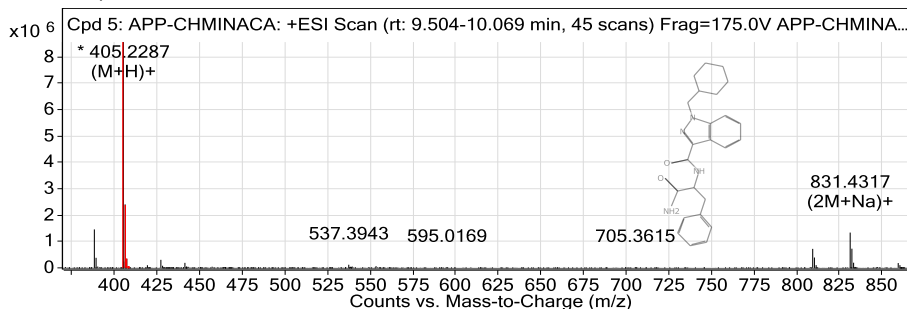
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum

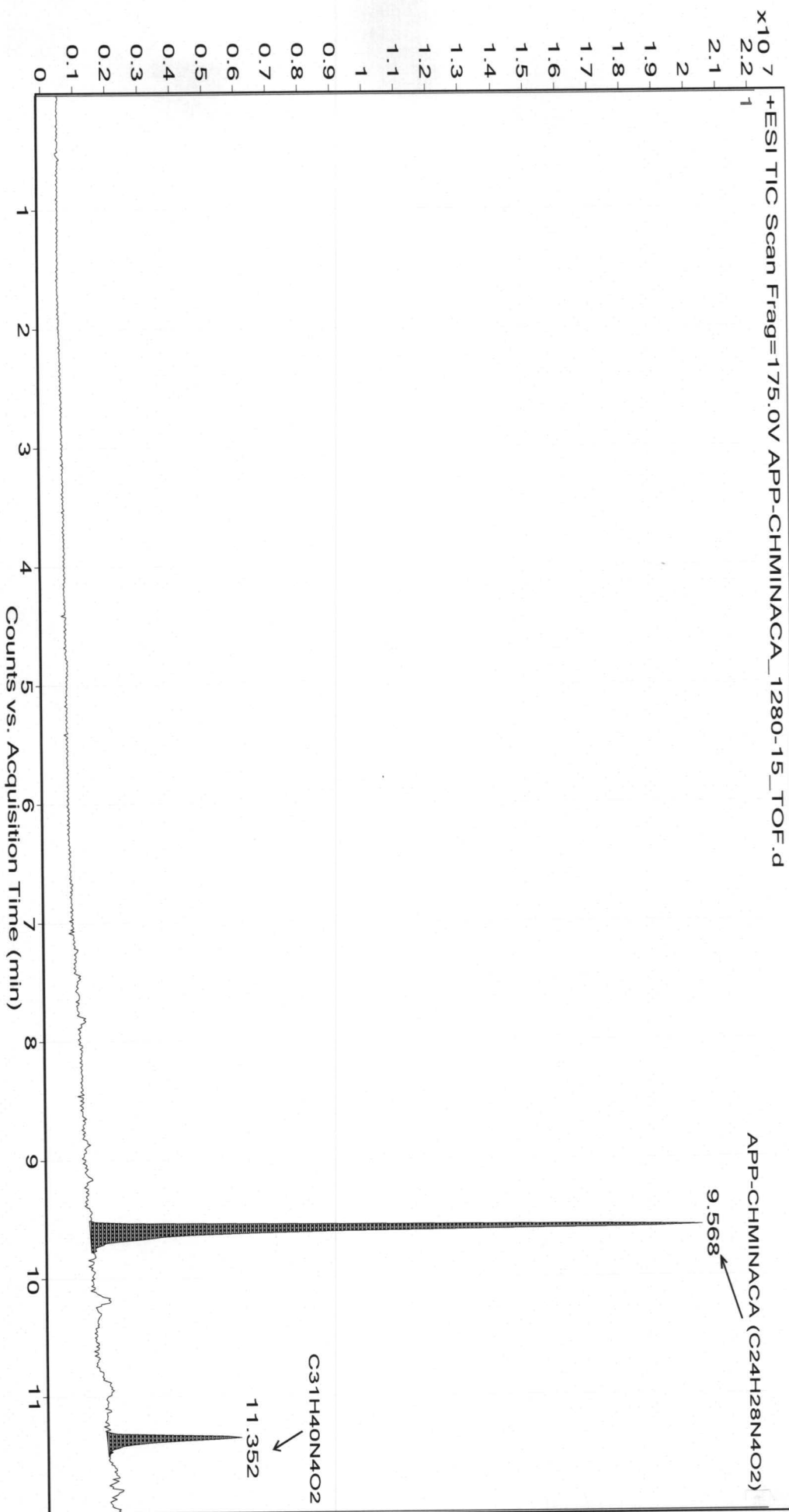


MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
405.2298	1	8562817	C24 H28 N4 O2	(M+H)+
406.2322	1	2369233.93	C24 H28 N4 O2	(M+H)+
407.2354	1	328052.88	C24 H28 N4 O2	(M+H)+
809.4505	1	898492.88		(2M+H)+
810.4535	1	489669.46		(2M+H)+
811.4557	1	129066.25		(2M+H)+
831.4322	1	1205450.25		(2M+Na)+
832.4355	1	655471.33		(2M+Na)+
833.4377	1	175558.78		(2M+Na)+
834.44	1	31387.51		(2M+Na)+

--- End Of Report ---

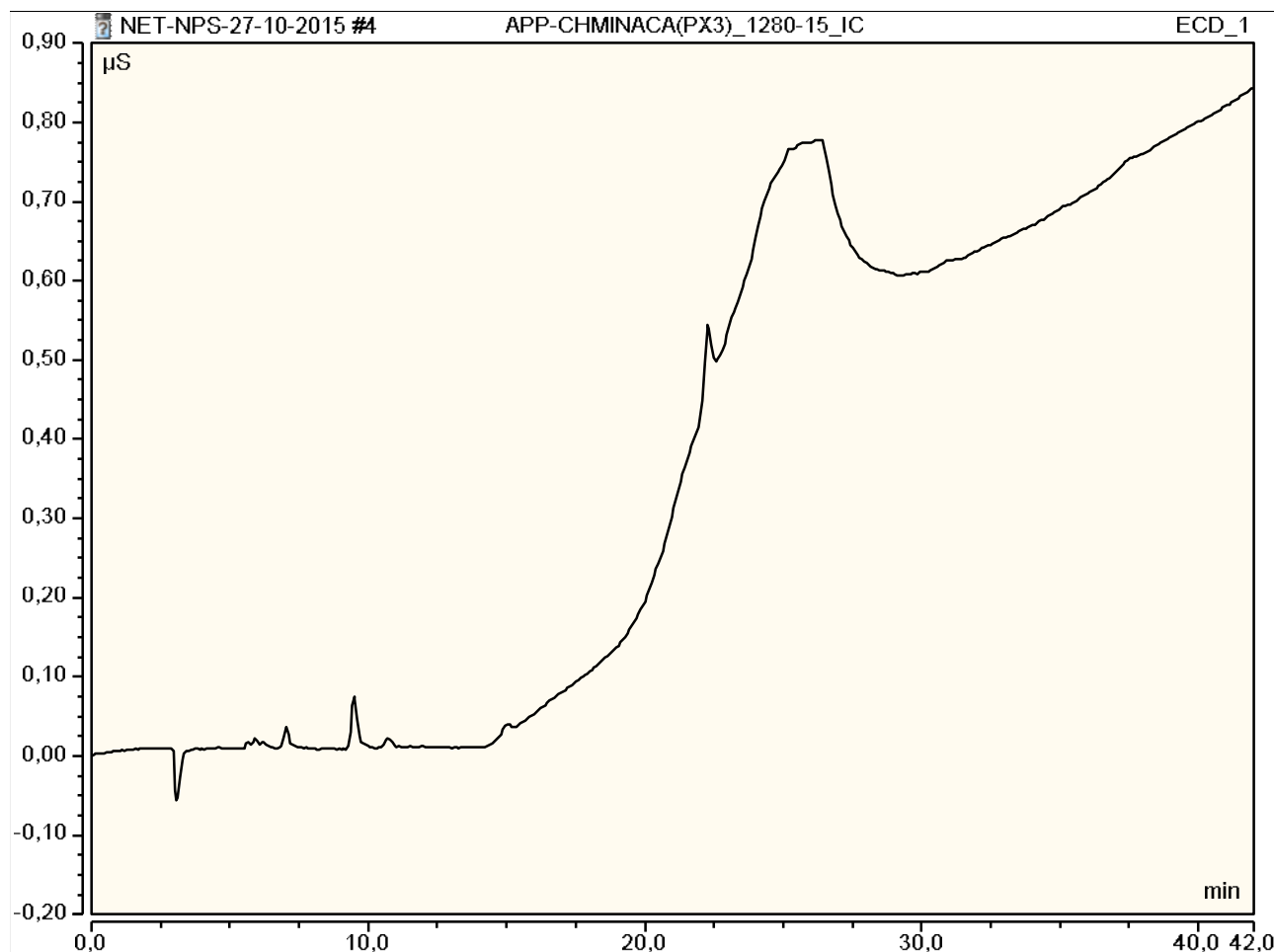
Sample Name	APP-CHMINACA (PX3)	Position	P1-E5	Instrument Name	6230B TOF LC-MS	User Name	TG
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	APP-CHMINACA_1280-15	ACQ Method	droge general-13-5-2	Comment	extract in MeOH	Acquired Time	9/21/2015 10:12:32 AM



Peak Integration Report

Sample Name:	APP-CHMINACA(PX3)_1280-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	27-okt-2015 / 13:55	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount n.a.
		TOTAL:		0,00	0,00	0,00





REPORT

Sample ID:	1280-15
Our notebook code:	P-1280-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C .
Proposed structure:	
Chemical name:	N-(1-amino-1-oxo-3-phenylpropan-2-yl)-1-(cyclohexylmethyl)-1H-indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D NMR spectra - Compound is not pure by NMR, contains at least one other compound, as evident from an additional set of signals observed in both spectra.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 9, 2015

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P-1280-15

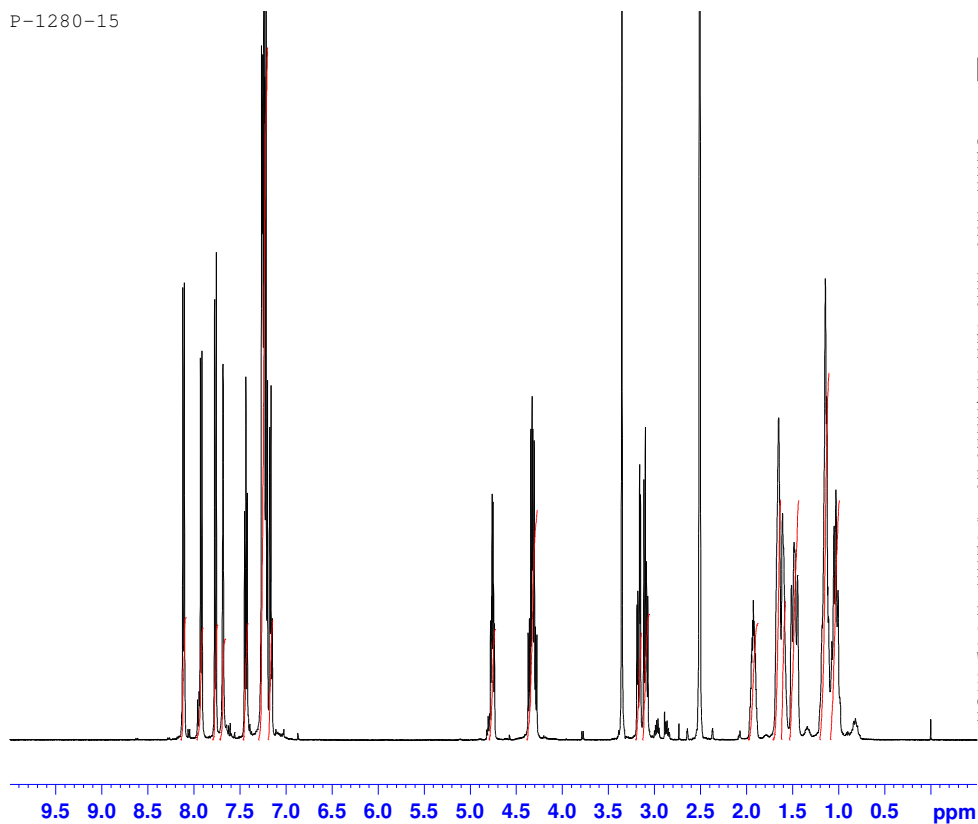


Current Data Parameters
NAME p-1280-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151204
Time 5.23
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 71.8
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 500.1299999 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



P-1280-15



Current Data Parameters
NAME P-1280-15
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151204
Time 6.38
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

