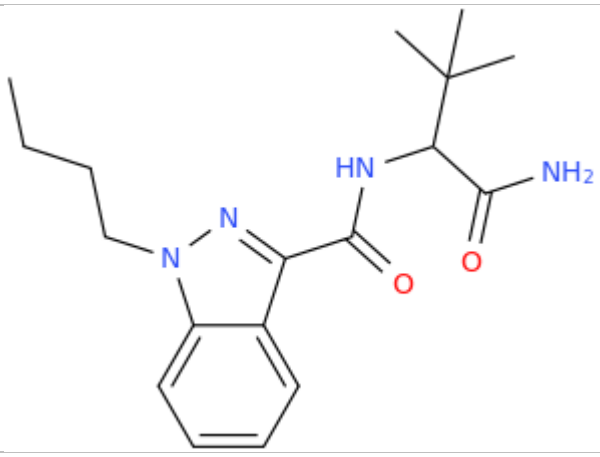
ANALYTICAL REPORT

ADB-BUTINACA (C18H26N4O2)

2-[(1-butyl-1H-indazol-3-yl)formamido]-3,3-dimethylbutanamide

Remark – other NPS detected: none

Sample ID:	2082-19
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of entry (DD/MM/YYYY) into NFL database:	14/10/2019
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[1] (base form)	
Systematic name	2-[(1-butyl-1H-indazol-3-yl)formamido]-3,3-dimethylbutanamide
Other names	ADB-BUTINACA; N-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-1-butyl-1H-indazole-3-carboxamide
Formula (per base form)	C18H26N4O2
M_w (g/mol)	330,43
Salt form/anions detected	base
StdInChIKey (per base form)	GPWADXHYJAZPAX-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>90% pure based on 1H NMR spectrum

[1] 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 7.1 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 KOH 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

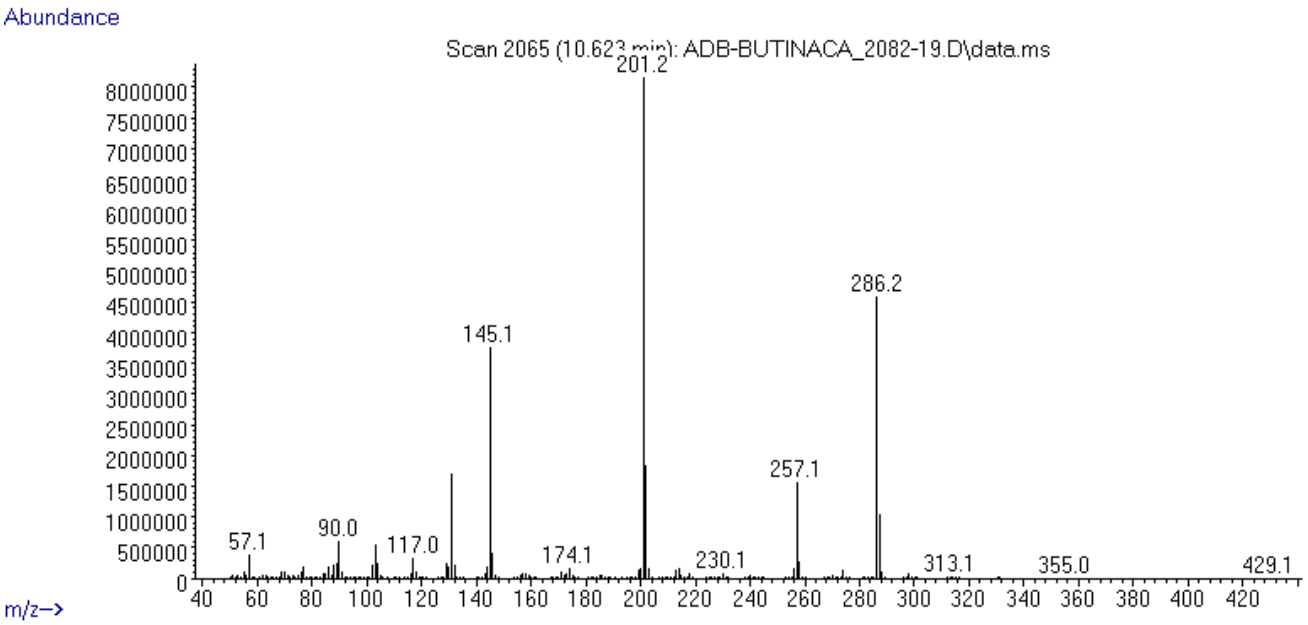
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	patially

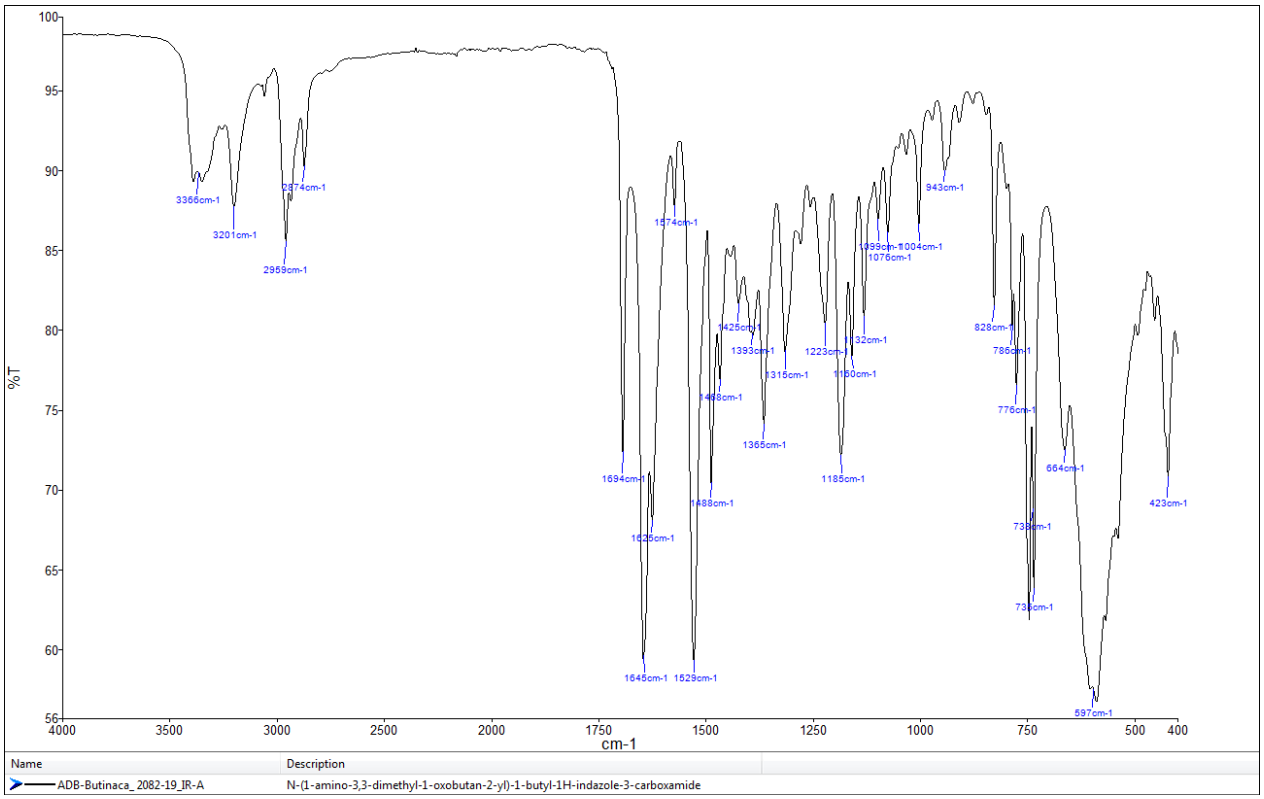
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 10,62 BP(1): 201; BP(2): 286,BP(3) :145,
HPLC-TOF	+	Exact mass (theoretical): 330,2056; measured value Δppm:1,04; formula:C18H26N4O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

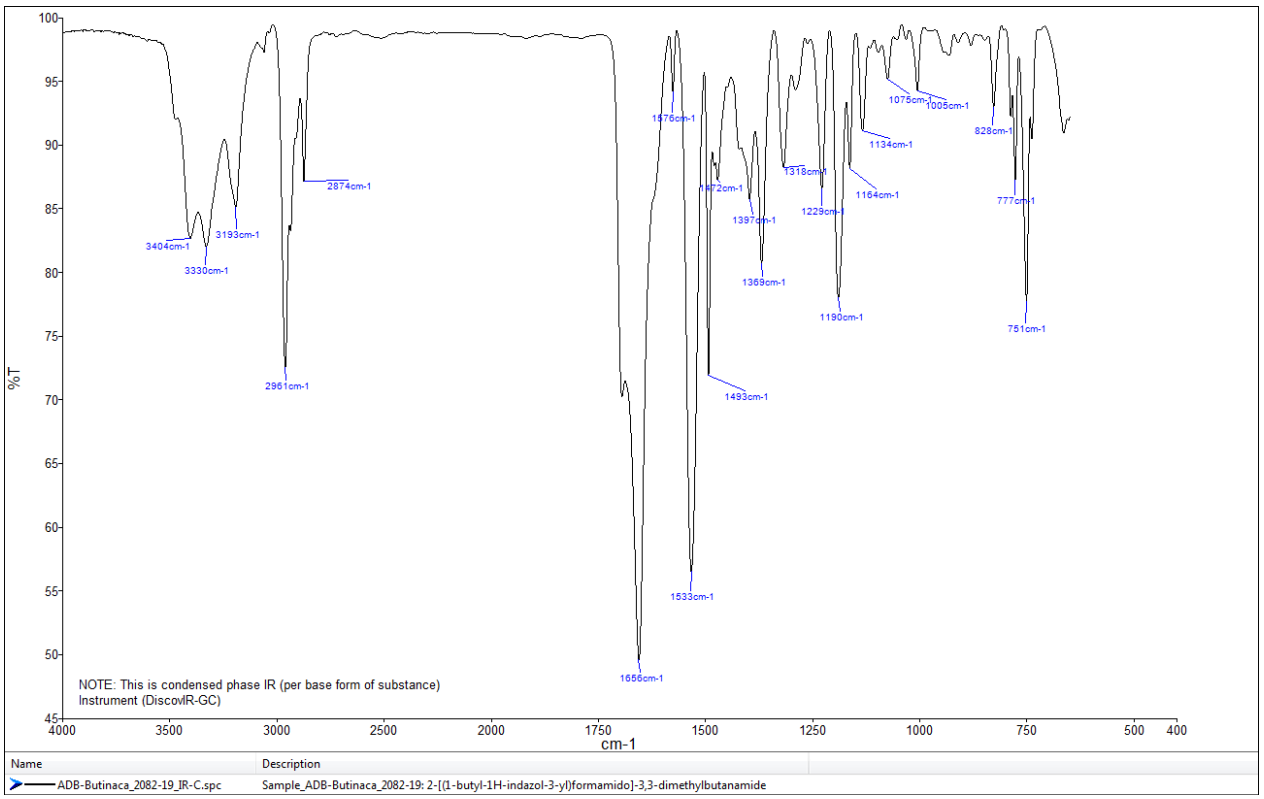
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

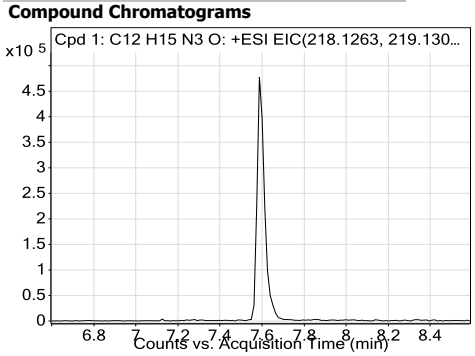
Data File	ADB-Butinaca_2082_pon.d	**Sample Name**	ID-2082-19
Sample Type	Sample	**Position**	P1-C3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	**Acquired Time**	8/5/2019 2:26:46 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

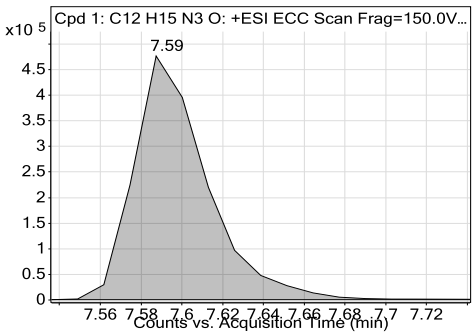
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: C12 H15 N3 O		C12 H15 N3 O	7.59	217.1215
Cpd 7: ADB-Butinaca	ADB-Butinaca	C18 H26 N4 O2	8.73	330.2059

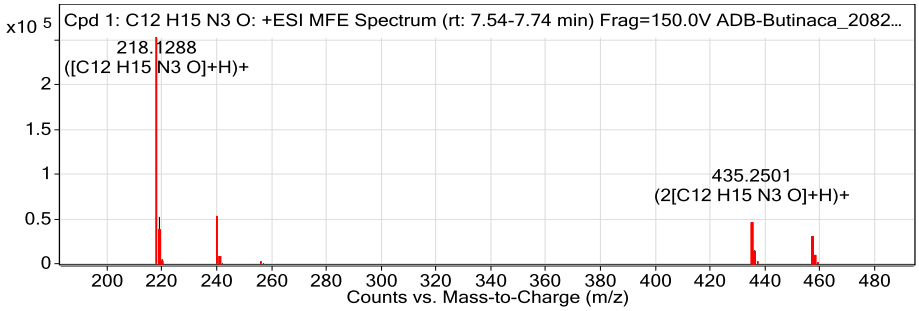
Obs. m/z	Obs. RT	Obs. Mass
218.1288	7.59	217.1215

Compound Chromatograms

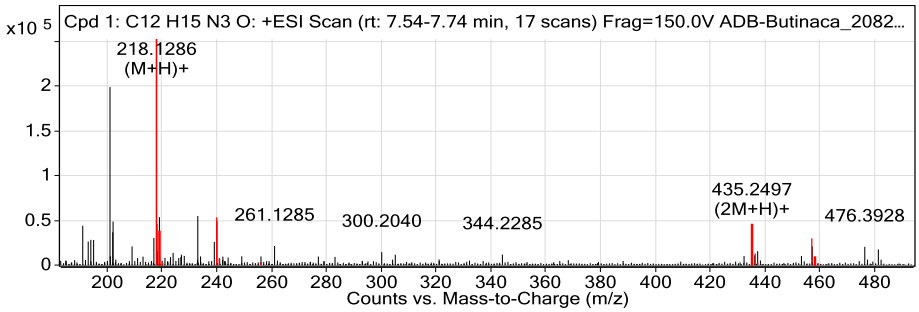




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



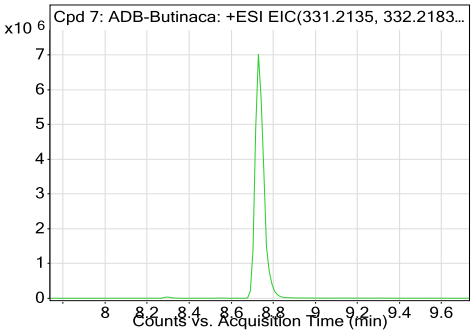
MS Spectrum Peak List

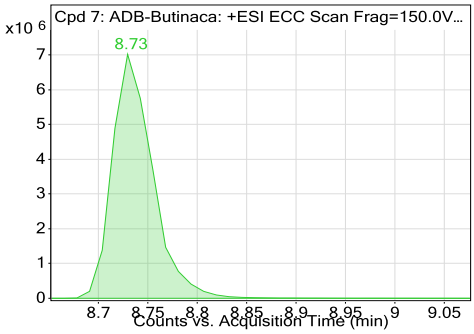
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
218.1288	1	252681.02	C12 H15 N3 O	(M+H)+
219.1309	1	52309.41	C12 H15 N3 O	(M+H)+
220.1332	1	5174.88	C12 H15 N3 O	(M+H)+
240.1106	1	52906.64	C12 H15 N3 O	(M+Na)+
241.1143	1	8114.84	C12 H15 N3 O	(M+Na)+
435.2501	1	41655.86	C12 H15 N3 O	(2M+H)+
436.2525	1	15193.56	C12 H15 N3 O	(2M+H)+
437.2543	1	3022.72	C12 H15 N3 O	(2M+H)+
457.2323	1	28319.77	C12 H15 N3 O	(2M+Na)+
458.2351	1	9509.45	C12 H15 N3 O	(2M+Na)+

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
ADB-Butinaca	331.2125	8.73	330.2059	8.73	C18 H26 N4 O2	330.2056	-0.94

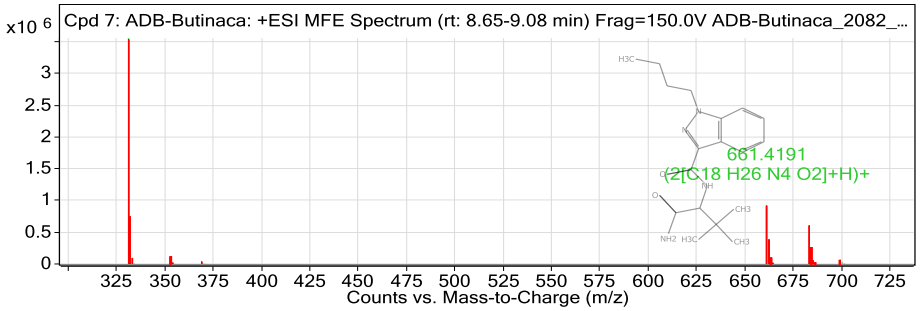
TOF REPORT

Compound Chromatograms

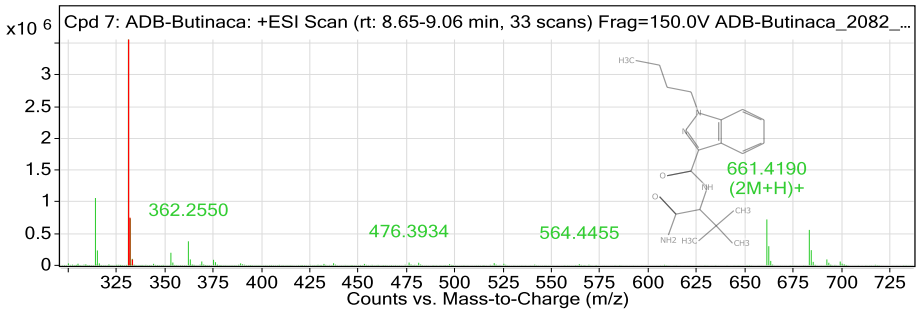




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

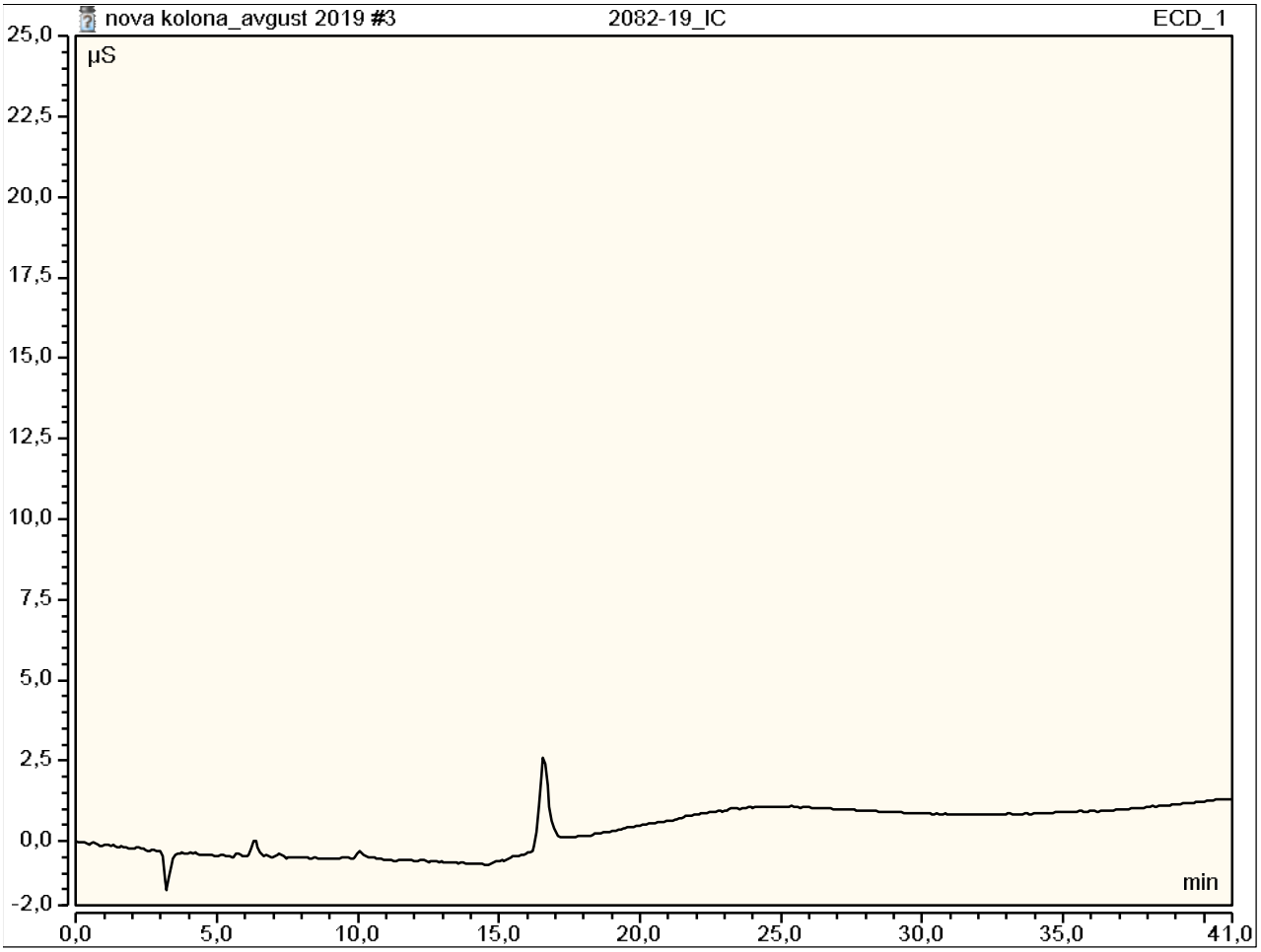
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
331.2125	1	3558912.75	C18 H26 N4 O2	(M+H)+
332.2162	1	732950.71	C18 H26 N4 O2	(M+H)+
333.2186	1	98456.07	C18 H26 N4 O2	(M+H)+
353.1948	1	114191.89	C18 H26 N4 O2	(M+Na)+
661.4191	1	926127.63	C18 H26 N4 O2	(2M+H)+
662.4219	1	385858.47	C18 H26 N4 O2	(2M+H)+
663.4242	1	89178.24	C18 H26 N4 O2	(2M+H)+
683.4009	1	596878	C18 H26 N4 O2	(2M+Na)+
684.4033	1	255637.49	C18 H26 N4 O2	(2M+Na)+
699.374	1	59967.63	C18 H26 N4 O2	(2M+K)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**2082-19_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**31-jul-2019 / 12:41**	*Run Time:*	**43,00**

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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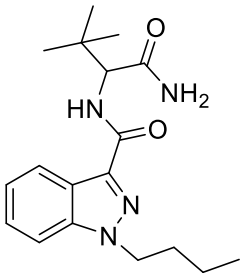
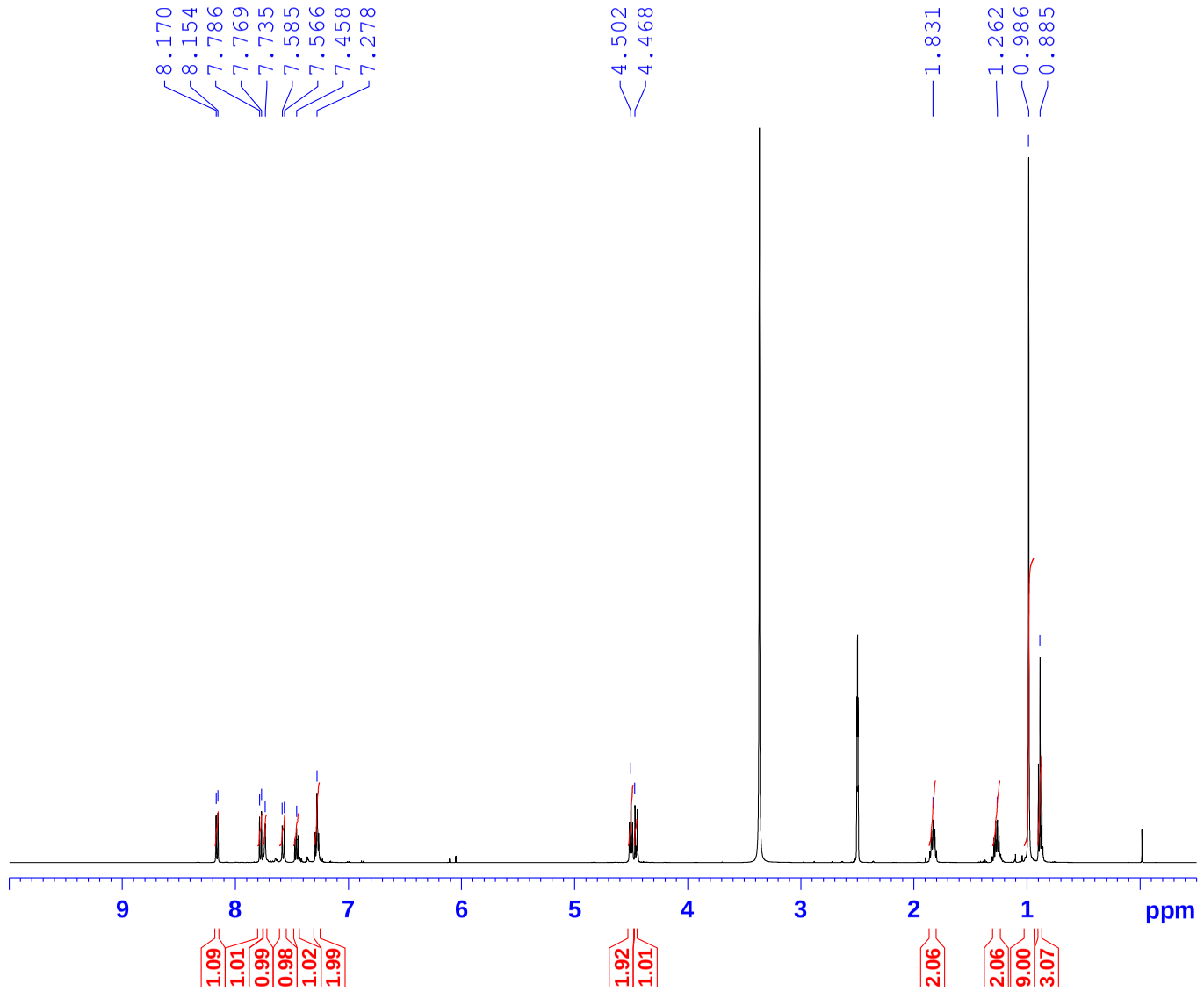
University
of Ljubljana

Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**2082-19**
Received date:	October 1, 2019
Our notebook code:	NFL-2082-19
NMR sample preparation:	16.9 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 with formula, exact mass, molecular weight:	 Chemical Formula: $C_{18}H_{26}N_4O_2$ Exact Mass: 330,2056 Molecular Weight: 330,4320
Chemical name:	*N*-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-1-butyl-1*H*-indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >90% purity of a sample based on ^{1}H NMR spectrum.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra, ^{1}H and ^{13}C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	October 11, 2019

NFL-2082-19
1H



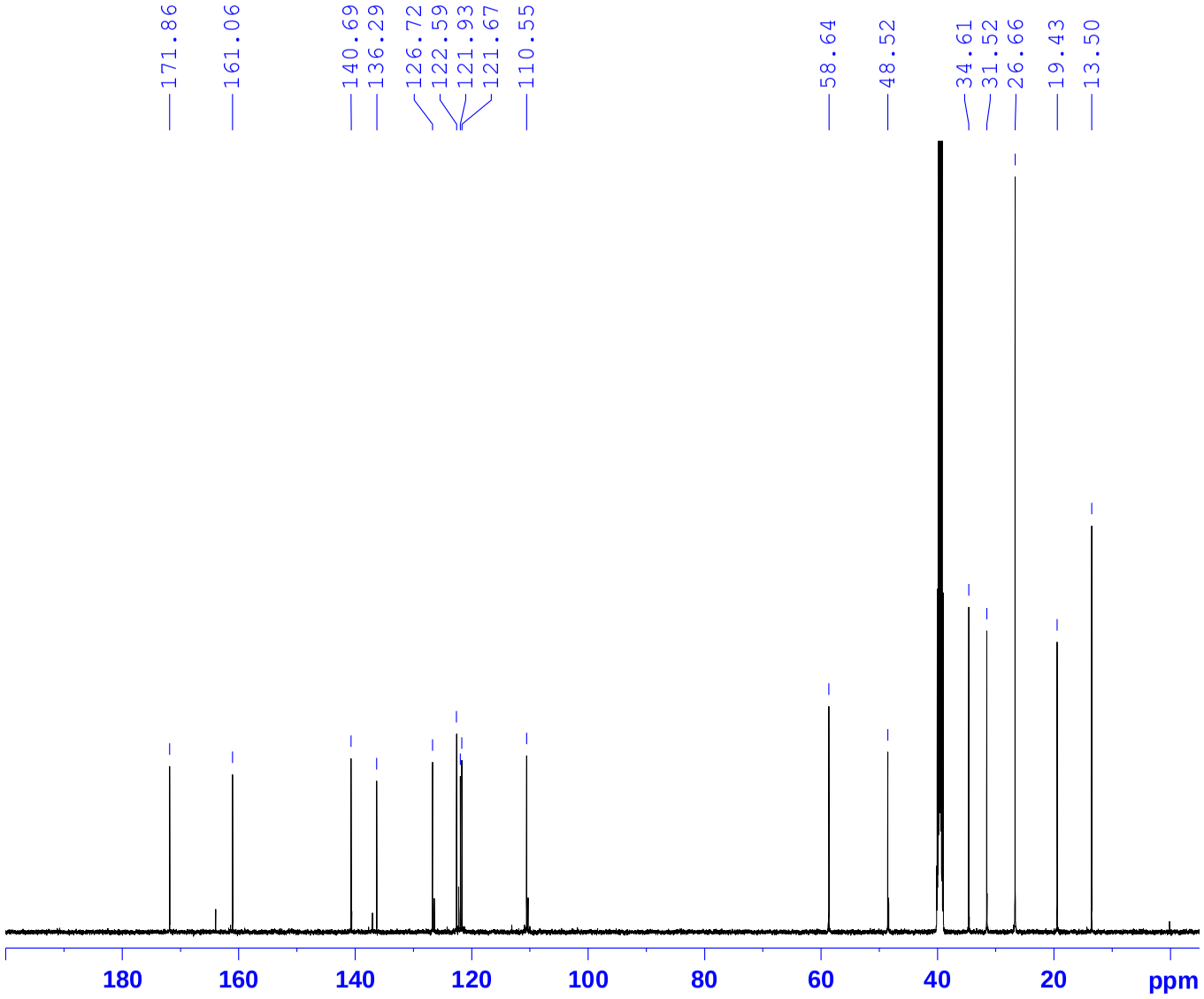

```
Current Data Parameters
NAME         NFL-2082-19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20191001
Time               23.13
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT              DMSO
NS                     64
DS                      2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2767999 sec
RG                     64
DW                50.000 usec
DE                 6.50 usec
TE                296.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                1H
P1                 8.70 usec
PLW1         26.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300042 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

NFL-2082-19
13C

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Current Data Parameters
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EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
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Time                1.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                   4096
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                   8.70 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         0.30046001 W
PLW13         0.15113001 W

F2 - Processing parameters
SI                  32768
SF           125.7578430 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```