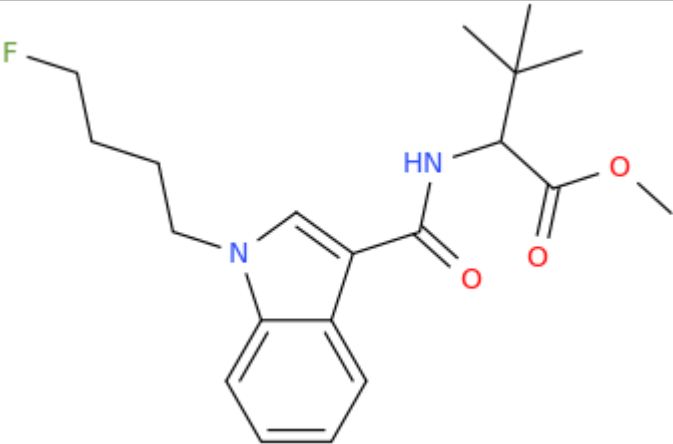


## ANALYTICAL REPORT

4F-MDMB-BICA (C<sub>20</sub>H<sub>27</sub>FN<sub>2</sub>O<sub>3</sub>)**methyl 2-([1-(4-fluorobutyl)indol-3-yl]formamido)-3,3-dimethylbutanoate; Methyl 2-(1-(4-fluorobutyl)-1H-indole-3-carboxamido)-3,3-dimethylbutanoate**Remark – other compounds detected: **unidentified impurities**

Sample ID:	2189-20
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of entry (DD/MM/YYYY) into NFL database:	17/09/2020
Report updates (if any) will be published here:	<a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a>

Substance identified - structure <sup>1</sup> (base form)	
Systematic name	methyl 2-([1-(4-fluorobutyl)indol-3-yl]formamido)-3,3-dimethylbutanoate; Methyl 2-(1-(4-fluorobutyl)-1H-indole-3-carboxamido)-3,3-dimethylbutanoate
Other names	MDMB-4F-BICA; 4F-MDMB-BUTICA; MDMB-4F-BUTICA; methyl N-([1-(4-fluorobutyl)-1H-indol-3-yl]carbonyl)-3-methylvalinate; methyl 2-([1-(4-fluorobutyl)-1H-indol-3-yl]carbonyl)amino)-3,3-dimethylbutanoate
Formula (per base form)	C <sub>20</sub> H <sub>27</sub> FN <sub>2</sub> O <sub>3</sub>
M <sub>w</sub> (g/mol)	362,45
Salt form/anions detected	base
StdInChIKey (per base form)	QIKHYQCWGUGFBB-UHFFFAOYSA-N
Other NPS detected	unidentified impurities
Additional info (purity..)	>95% purity of a sample based on 1H NMR

<sup>1</sup> 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  $\mu$ l 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  $\mu$ m. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 7.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 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 (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  $\mu$ l. 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 (N<sub>2</sub>) 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**4. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)**

GC-method: Injection volume 1  $\mu$ l 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 (30 until 6 min.) to 550 (300) amu.

IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm<sup>-1</sup>.

**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  $\mu$ l

## Supporting information

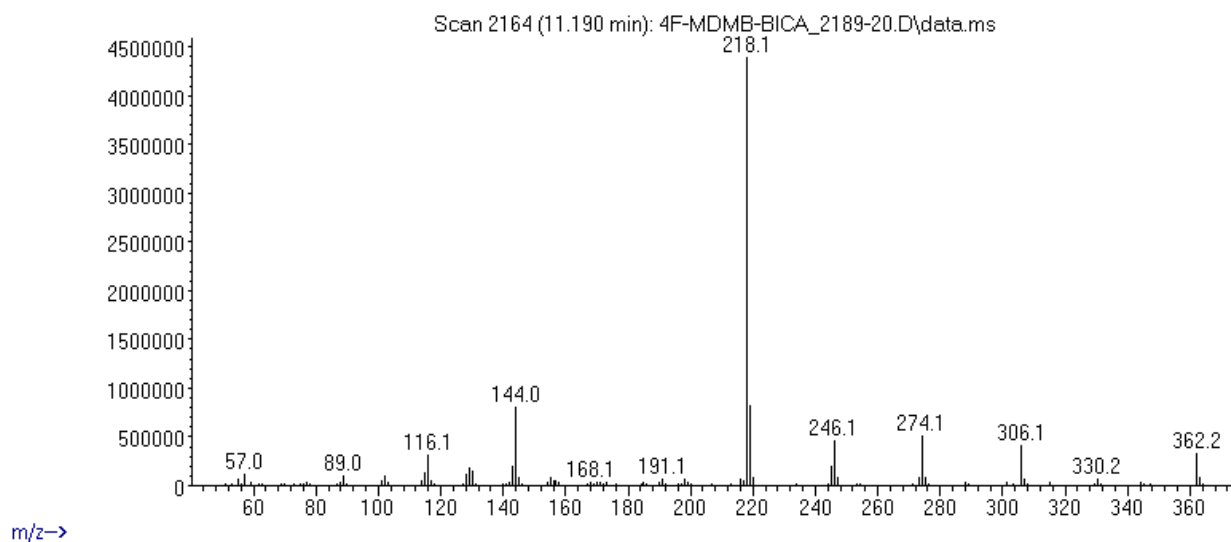
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	
MeOH	
H <sub>2</sub> O	

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 11,2 BP(1): 218; BP(2): 219,BP(3) :144,
HPLC-TOF	+	Exact mass (theoretical): 362,2006; measured value Δppm:-2,08; formula:C20H27FN2O3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

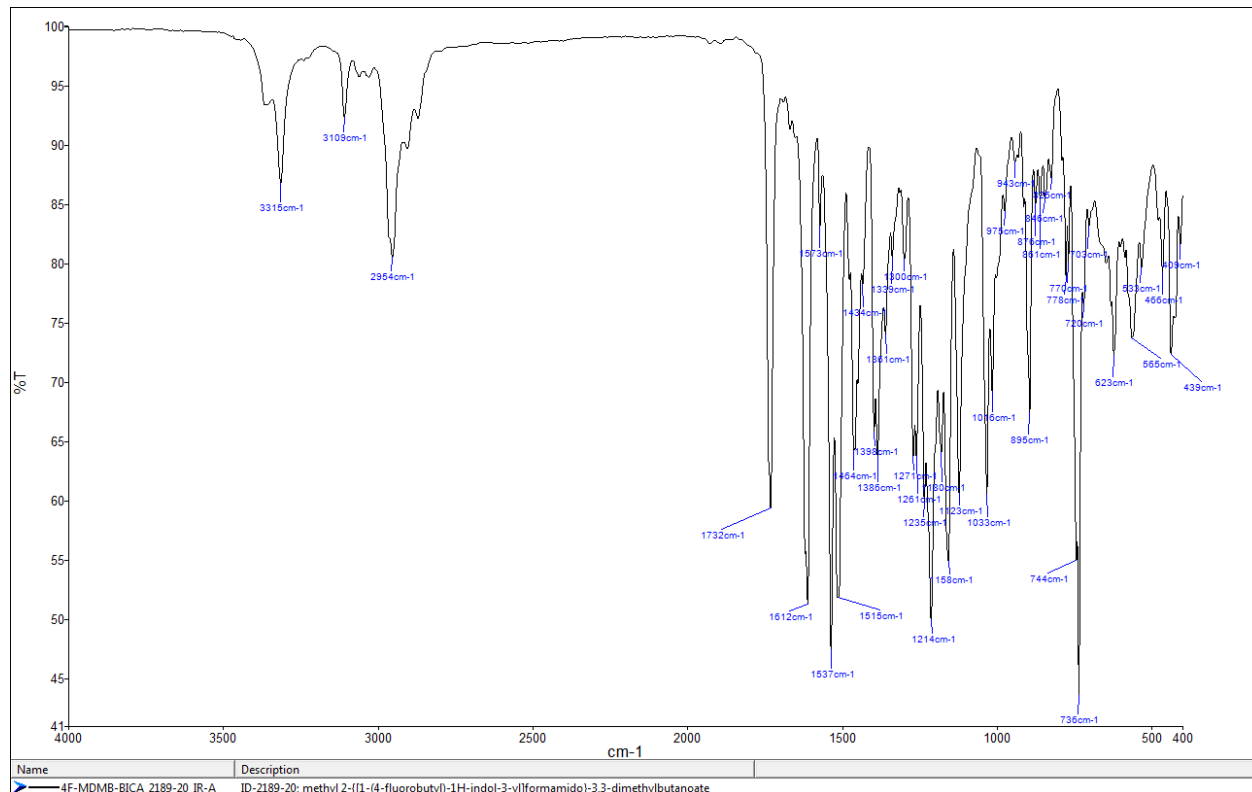
# ANALYTICAL RESULTS

MS (EI)

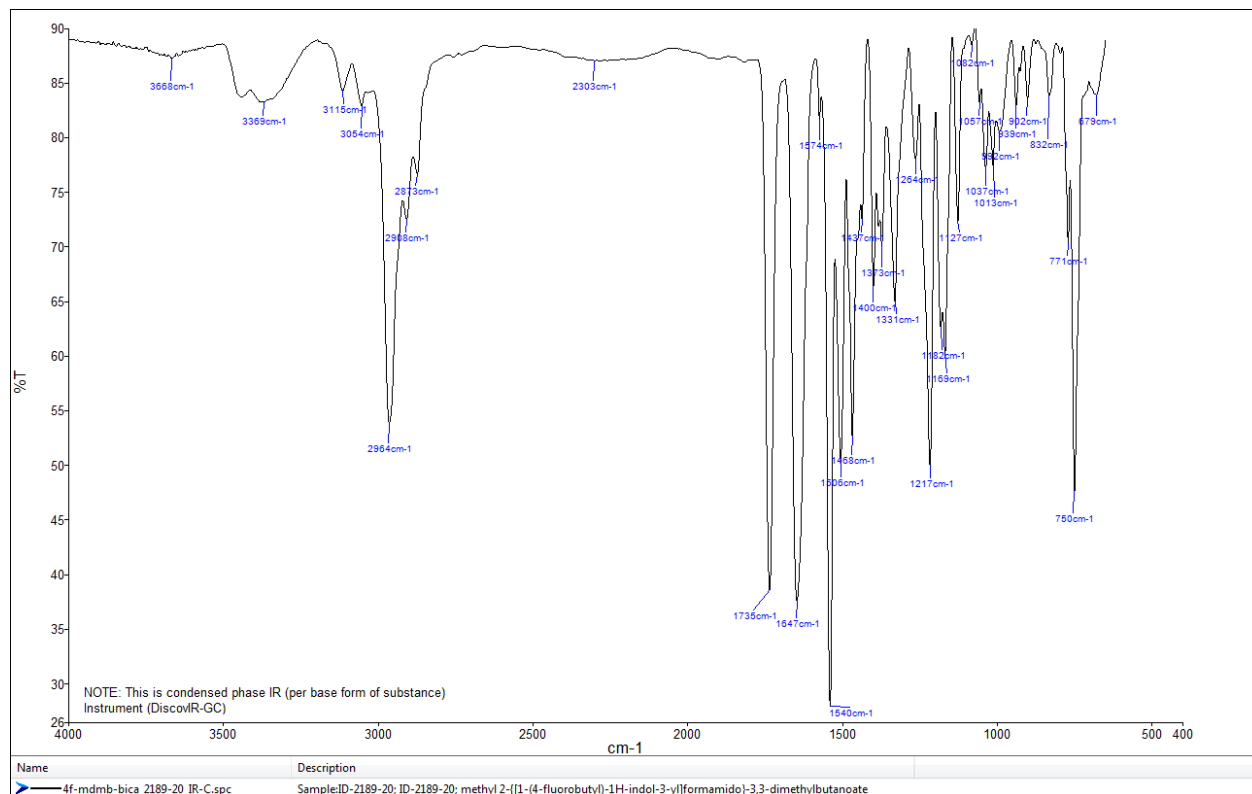
Abundance



## FTIR-ATR - direct measurement (sample as received)



## IR (solid phase – after chromatographic separation)



# TOF REPORT

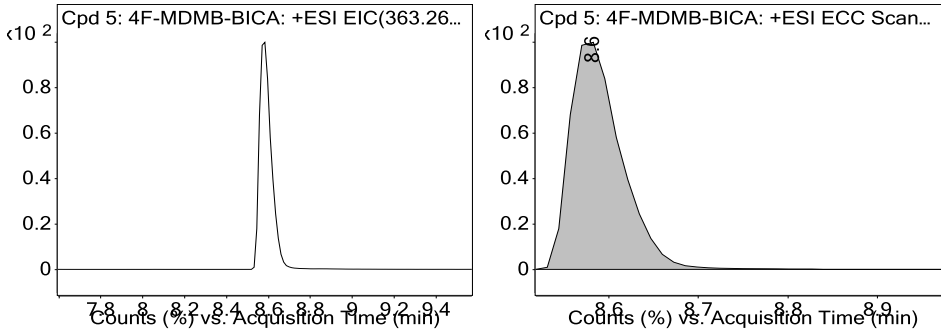
<b>Data File</b>	4F-MDMB-BICA_2189-20.d	<b>Sample Name</b>	ID-2189-20
<b>Sample Type</b>	Sample	<b>Position</b>	P2-A6
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	
<b>Acq Method</b>	general-15_01_2020-XDB-C18-ESI+.m	<b>Acquired Time</b>	9/2/2020 8:47:22 PM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	a-Drugs_NFL.m
<b>Comment</b>			

## Compound Table

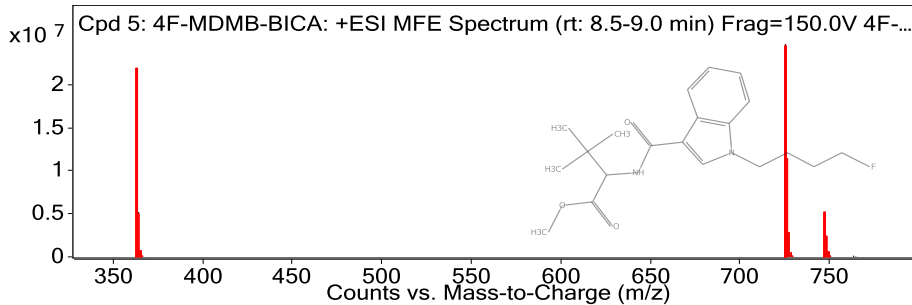
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 5: 4F-MDMB-BICA	4F-MDMB-BICA	C20 H27 F N2 O3	8.6	362.2013

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4F-MDMB-BICA	725.4092	8.6	362.2013	8.6	C20 H27 F N2 O3	362.2006	-2.08

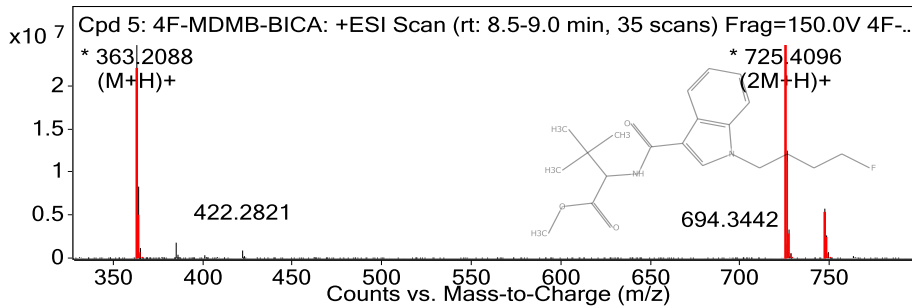
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

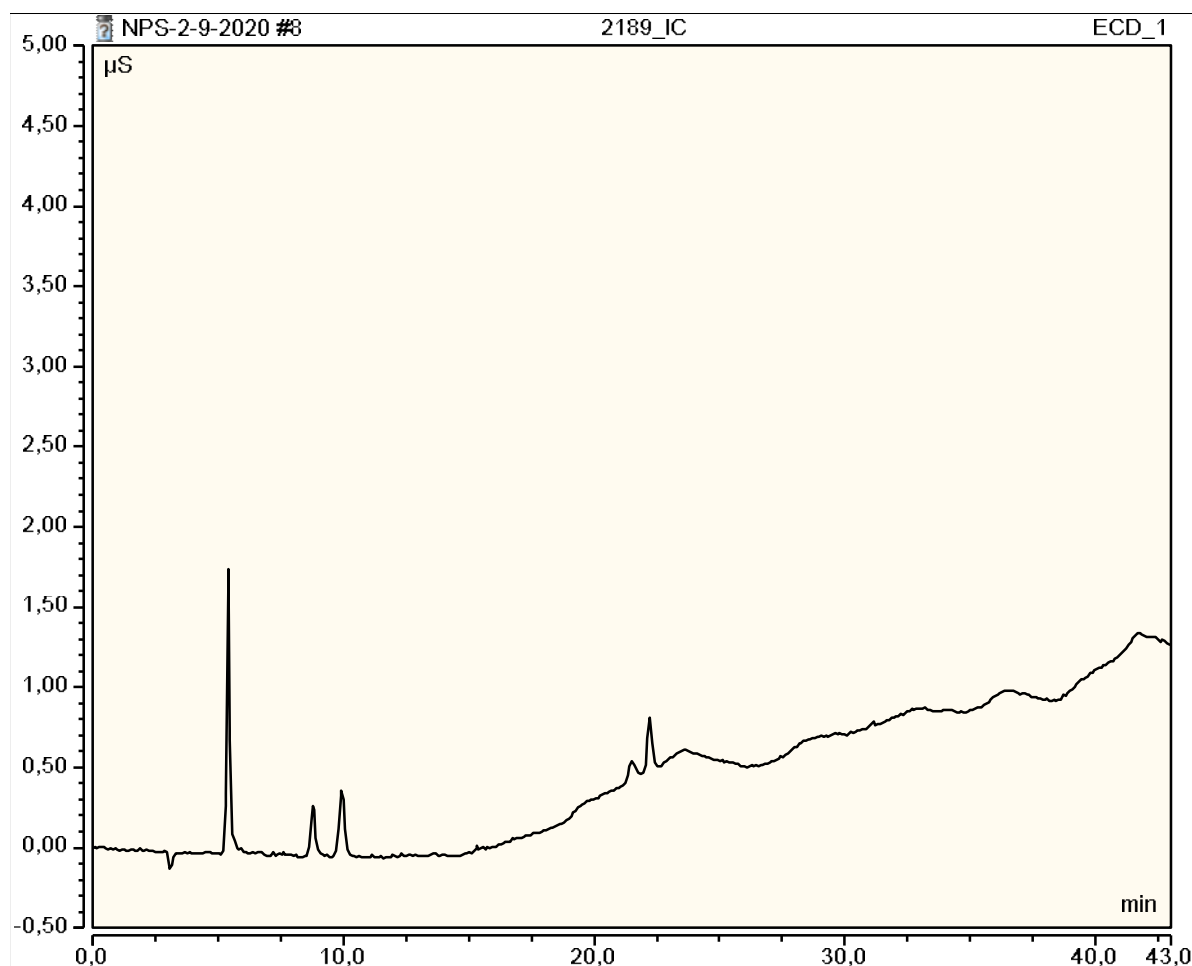
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
363.2086	1	21797818	C20 H27 F N2 O3	(M+H)+
364.2119	1	5197025.96	C20 H27 F N2 O3	(M+H)+
365.2149	1	643717.32	C20 H27 F N2 O3	(M+H)+
725.4092	1	24707428	C20 H27 F N2 O3	(2M+H)+
726.4126	1	11270407.26	C20 H27 F N2 O3	(2M+H)+
727.4152	1	2830880.69	C20 H27 F N2 O3	(2M+H)+
728.4185	1	438660.28	C20 H27 F N2 O3	(2M+H)+
747.3912	1	5214114.5	C20 H27 F N2 O3	(2M+Na)+
748.3941	1	2390630.13	C20 H27 F N2 O3	(2M+Na)+
749.3975	1	624581.68	C20 H27 F N2 O3	(2M+Na)+

--- End Of Report ---

### Peak Integration Report

Sample Name:	2189_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	02-sep-2020 / 14:43	Run Time:	43,00

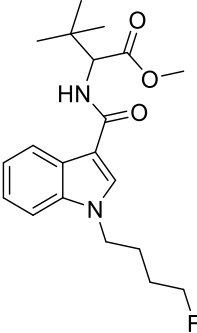
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height $\mu\text{S}$	Amount n.a.
TOTAL:				0,00	0,00	0,0



University  
of Ljubljana  
Faculty of Chemistry  
and Chemical Technology

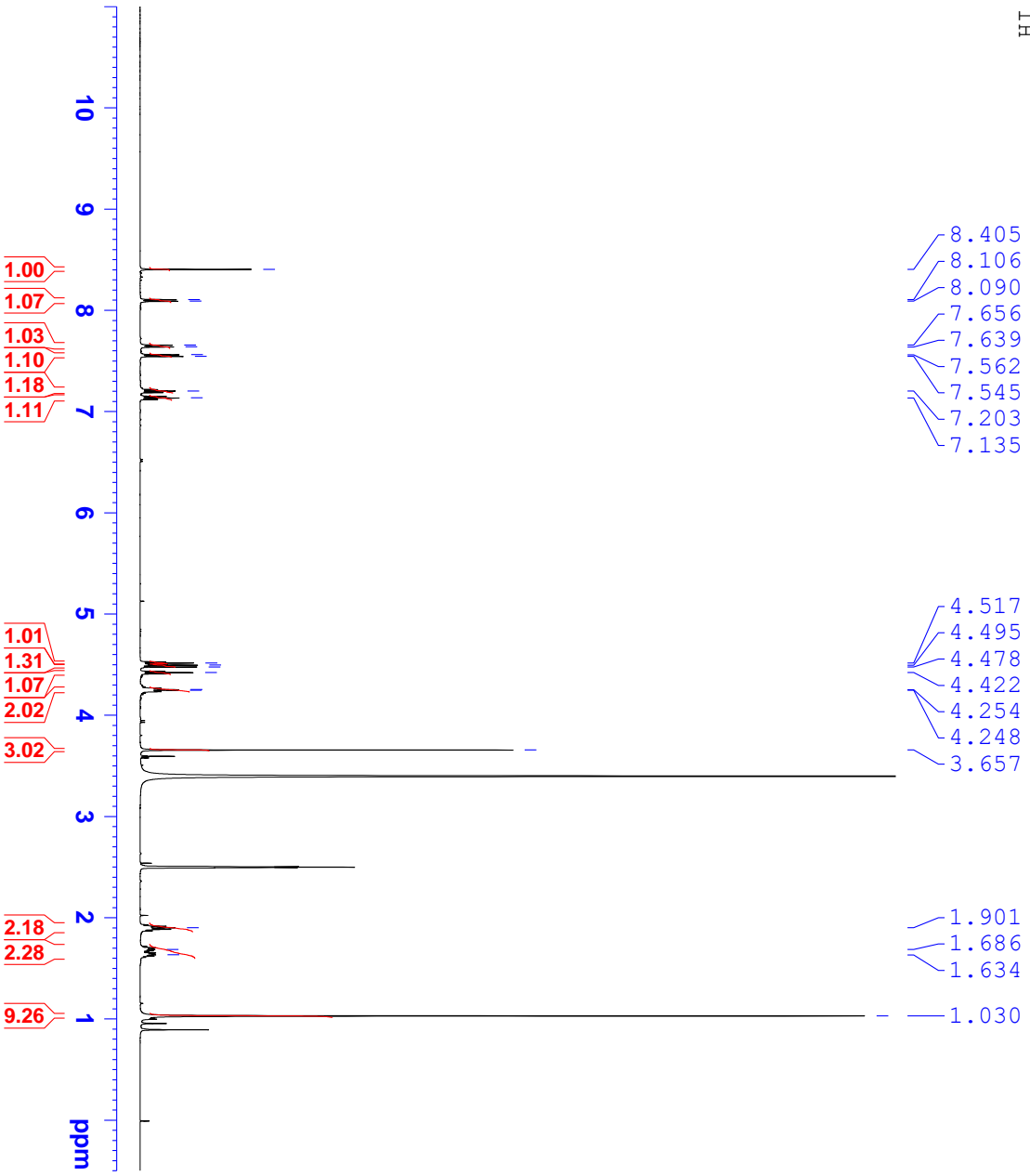


## R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	<b>2189-20</b>
Received date:	September 10, 2020
Our notebook code:	NFL-2189-20
NMR sample preparation:	17.7 mg dissolved in 0.7 mL DMSO- <i>d</i> <sub>6</sub>
NMR experiments:	<sup>1</sup> H, <sup>13</sup> C, <sup>1</sup> H- <sup>1</sup> H <i>gs</i> -COSY, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HSQC, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HMBC, <sup>1</sup> H- <sup>15</sup> N <i>gs</i> -HMBC, <sup>19</sup> F
Proposed structure with formula, exact mass, molecular weight:	 <p>Chemical Formula: C<sub>20</sub>H<sub>27</sub>FN<sub>2</sub>O<sub>3</sub> Exact Mass: 362.2006 Molecular Weight: 362.4454</p>
Chemical name:	Methyl 2-(1-(4-fluorobutyl)-1 <i>H</i> -indole-3-carboxamido)-3,3-dimethylbutanoate
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. ->95% purity of a sample based on <sup>1</sup> H NMR spectrum.
Supporting information:	Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra, <sup>1</sup> H and <sup>13</sup> C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	September 14, 2020



NFL-2189-20  
1H



Current Data Parameters  
NAME NFL-2189-20  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters

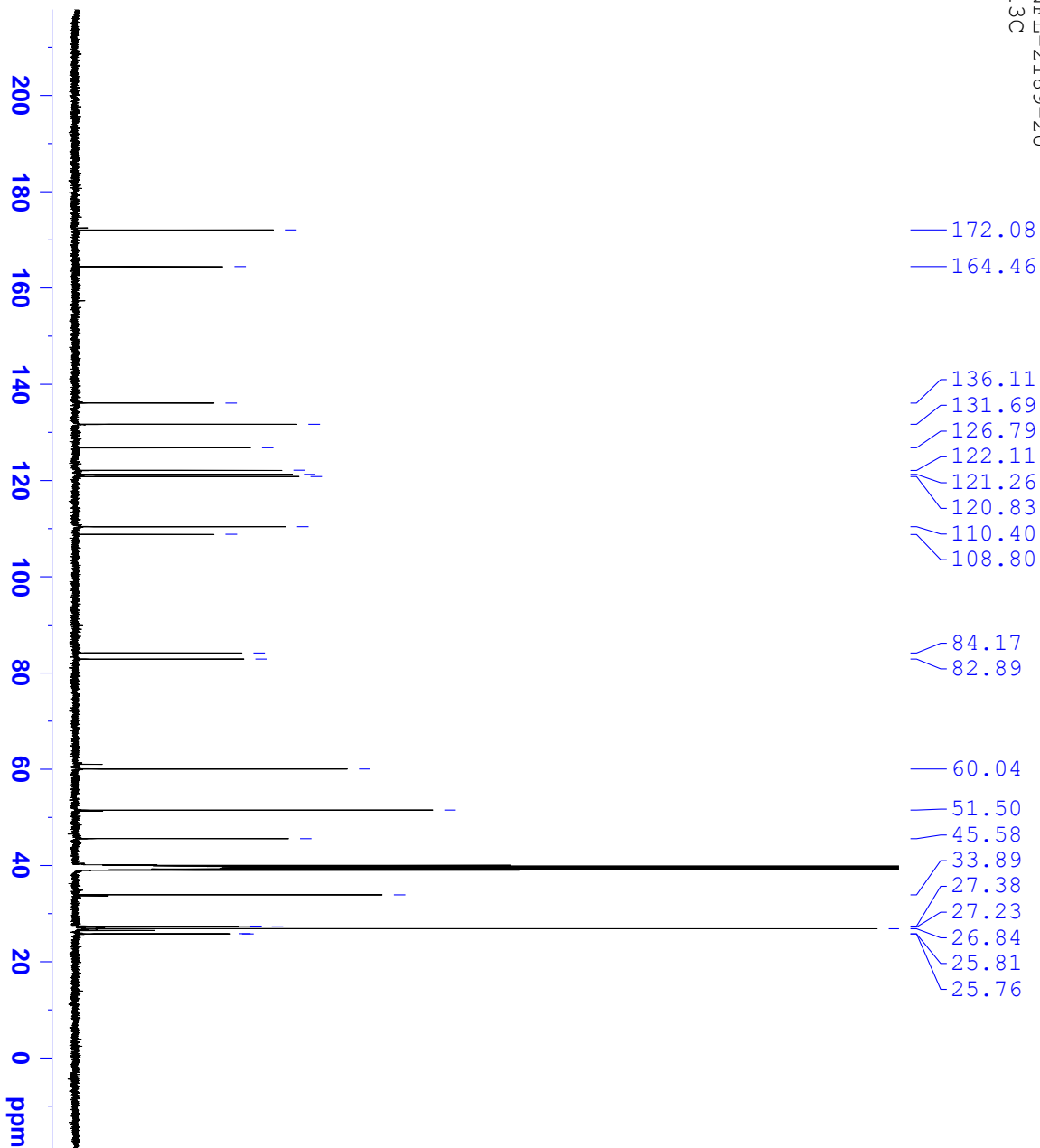
Date\_ 20200911  
Time\_ 16:28  
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PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 57  
DW 50.000 usec  
DE 6.50 usec  
TE 296.0 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 500.130885 MHz  
NUC1 1H  
P1 8.70 usec  
PLM1 26.00000000 W

F2 - Processing parameters

SI 65536  
SF 500.1300046 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

NFL-2189-20  
13C



Current Data Parameters  
NAME NFL-2189-20  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20200911  
Time 16.53

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 236.0 K  
D1 1.0000000 sec  
D11 0.0300000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.70 usec  
PLW1 122.0000000 W

==== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG2 waltz16  
PCPD2 80.00 usec  
PLW2 26.0000000 W  
PLM12 0.30046001 W  
PLM13 0.15113001 W

F2 - Processing parameters  
SI 32768  
SF 125.7578390 MHz  
WDW EM  
SSB 0  
IB 1.00 Hz  
GB 0  
PC 1.40