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RESPONSE 2
to challenges in forensic drugs analyses

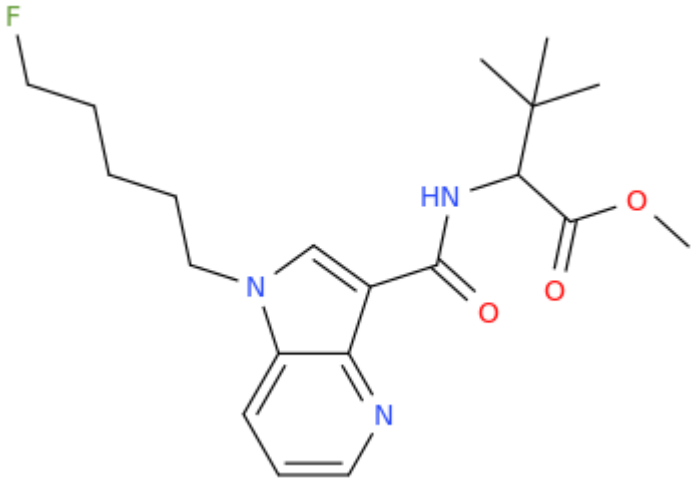
ANALYTICAL REPORT

5F-MDMB-P4AICA (C20H28FN3O3)

methyl 2-{[1-(5-fluoropentyl)-1H-pyrrolo[3,2-b]pyridin-3-yl]formamido}-3,3-dimethylbutanoate

Remark – other active cpd. detected none

Sample ID:	2080-19
Sample description:	liquid - yellow
Sample type:	RM-reference material
Comments:	CHI Lot#21217,
Date of entry (DD/MM/YYYY):	14/08/2019

Substance identified-structure[1] (base form)	
Systematic name:	methyl 2-{[1-(5-fluoropentyl)-1H-pyrrolo[3,2-b]pyridin-3-yl]formamido}-3,3-dimethylbutanoate
Other names:	Methyl 2-(1-(5-fluoropentyl)-1H-pyrrolo[3,2-b]pyridine-3-carboxamido)-3,3-dimethylbutanoate
Formula (per base form)	C20H28FN3O3
M_w (g/mol)	377,46
Salt form:	base
StdInChIKey (per base form)	YUNKAZHULJISDA-UHFFFAOYSA-N
Other active cpd. detected	none
Add.info (purity..)	compound and unknown impurity in molar ratio of 1 : 0.14 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)

Supporting information

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 12,77 BP(1): 233; BP(2): 145,BP(3) :320,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 377,2115 / measured Δppm: -4,7

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.

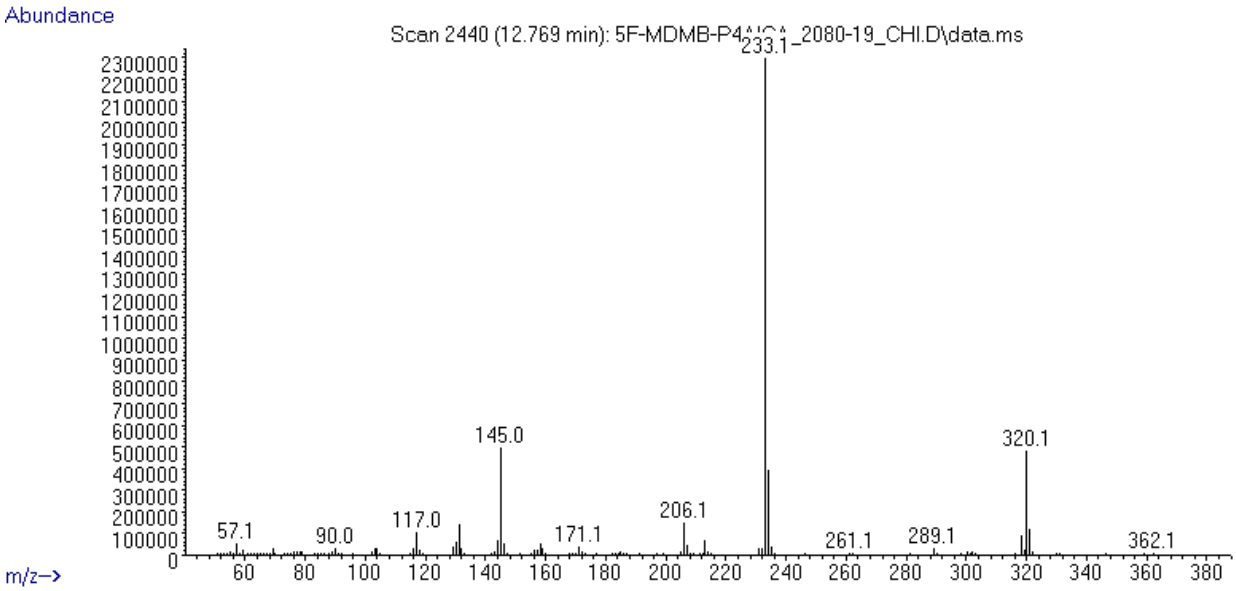
2. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1}; resolution 4cm^{-1}

3. 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}.

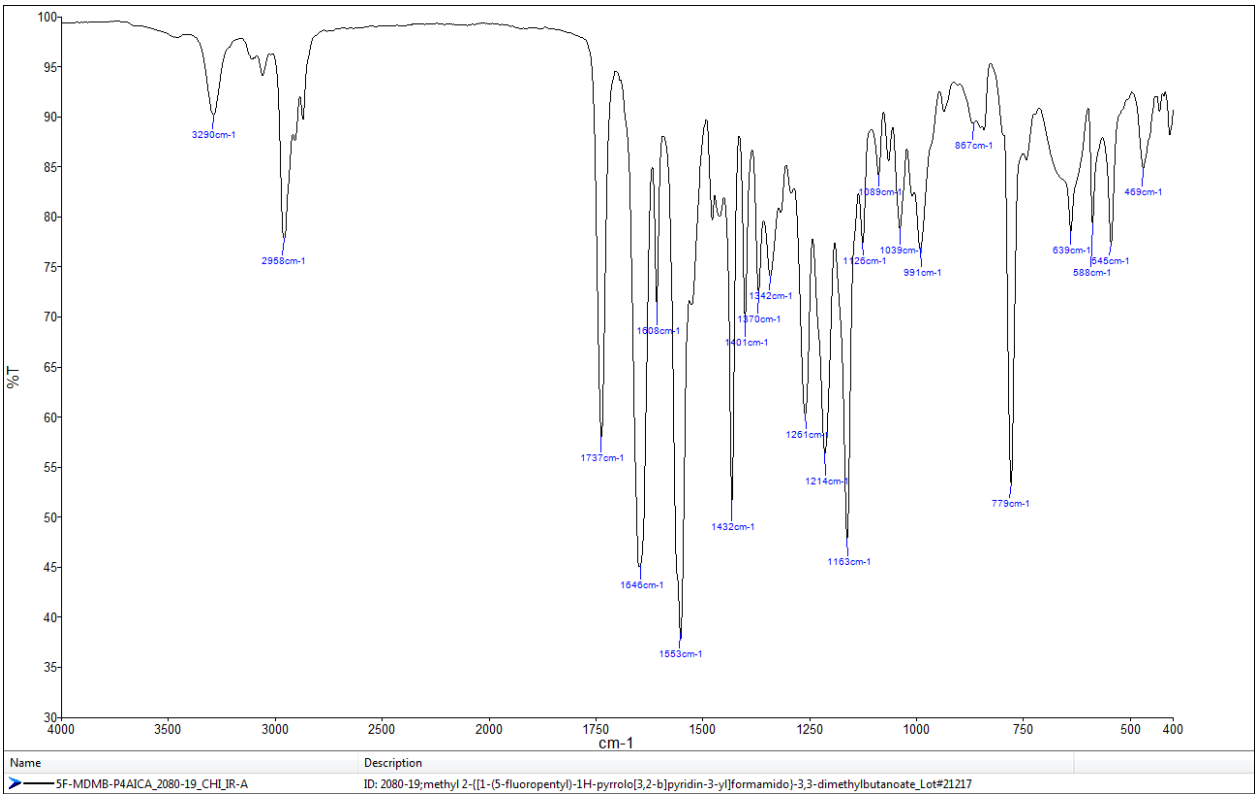
4. **HPLC-TOF** (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 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.

ANALYTICAL RESULTS

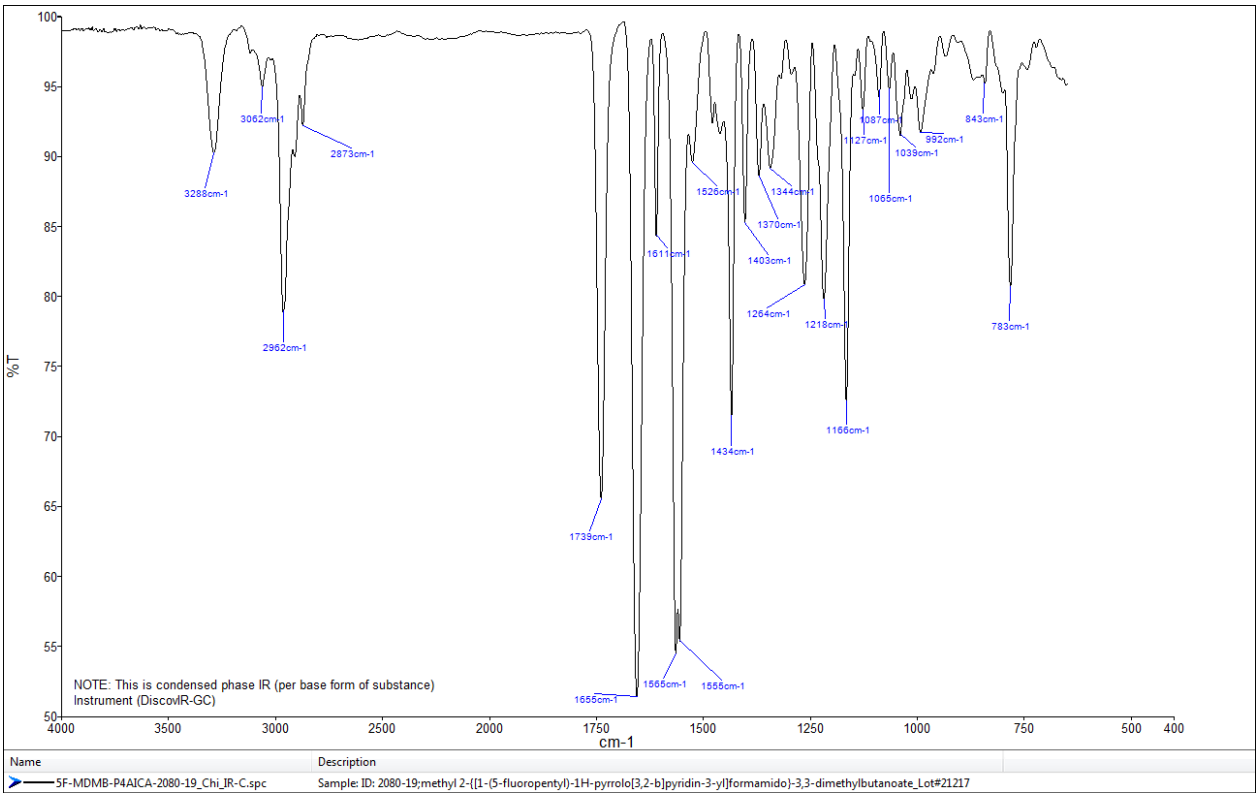
MS (EI)



FTIR-ATR – direct measurement



IR- (condensed (solid) phase – after chromatographic separation) - spectrum per base form



TOF REPORT

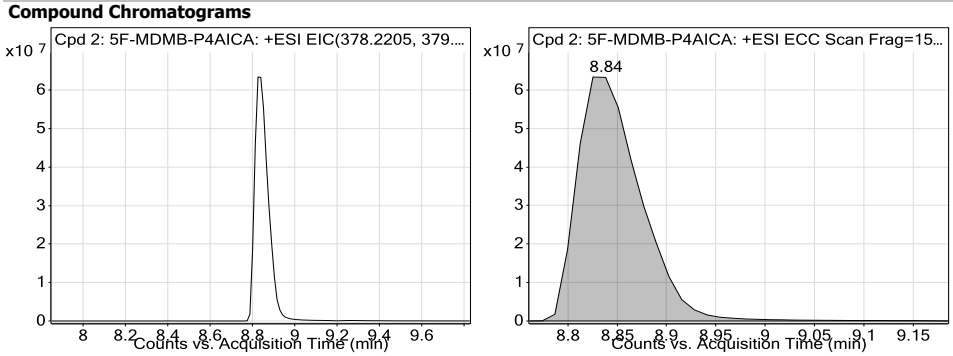
Data File	5F-MDMB-P4AICA_2080-19.d	**Sample Name**	ID_2080-19
Sample Type	Sample	**Position**	P1-F6
Instrument Name	6230B TOF LC-MS	**User Name**	
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	**Acquired Time**	7/15/2019 12:30:16 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment			

Compound Table

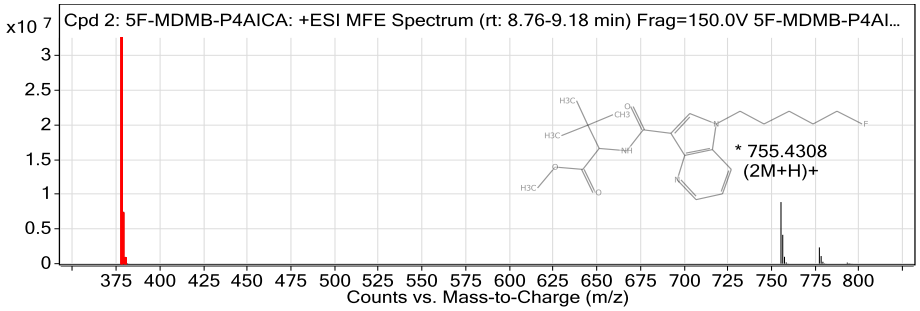
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: 5F-MDMB-P4AICA	5F-MDMB-P4AICA	C20 H28 F N3 O3	8.84	377.2132
Cpd 4: C23 H40 F2 N4 O5		C23 H40 F2 N4 O5	9.2	490.2968

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
5F-MDMB-P4AICA	378.2205	8.84	377.2132	8.84	C20 H28 F N3 O3	377.2115	-4.7

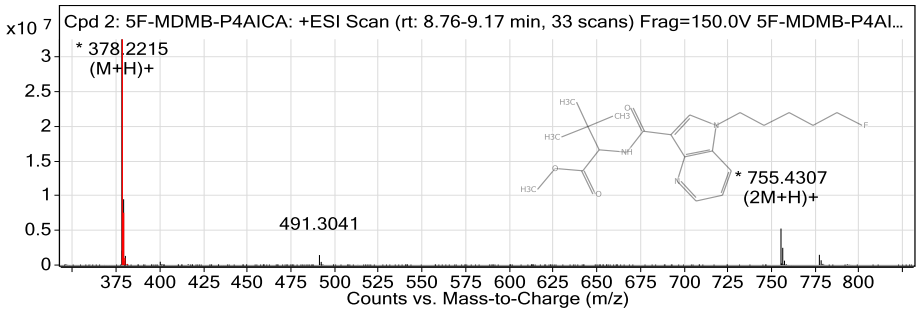
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



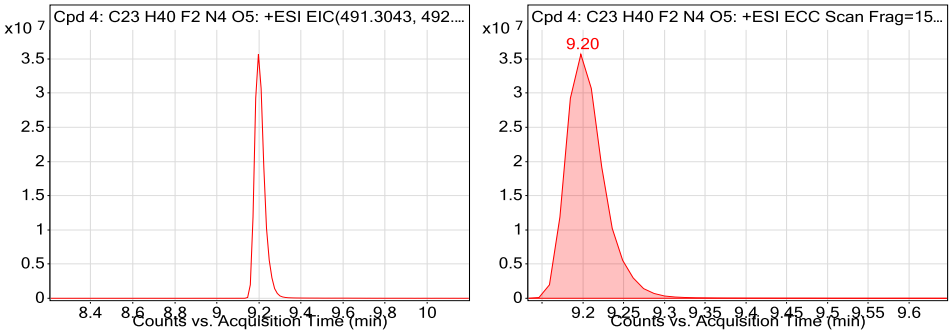
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
378.2205	1	32603538	C20 H28 F N3 O3	(M+H)+
379.2236	1	7494265.42	C20 H28 F N3 O3	(M+H)+
380.2255	1	979724.82	C20 H28 F N3 O3	(M+H)+
755.4308	1	8894818		(2M+H)+
756.4335	1	4168100.2		(2M+H)+
757.4369	1	994421.65		(2M+H)+
758.4395	1	178252.22		(2M+H)+
777.4122	1	2348783.5		(2M+Na)+
778.4163	1	1100321.41		(2M+Na)+
779.4186	1	276741.57		(2M+Na)+

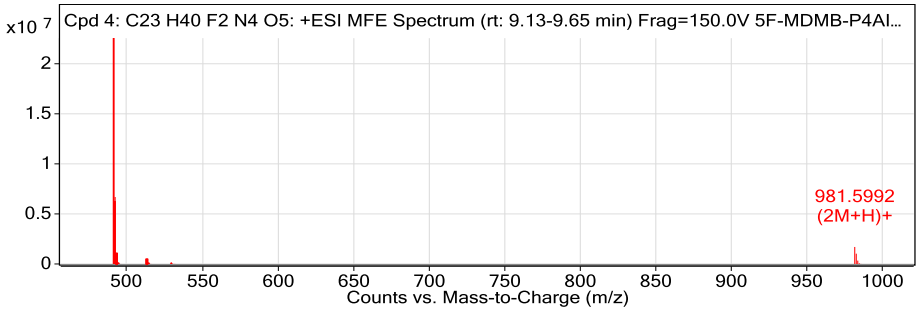
Obs. m/z	Obs. RT	Obs. Mass
491.3045	9.2	490.2968

TOF REPORT

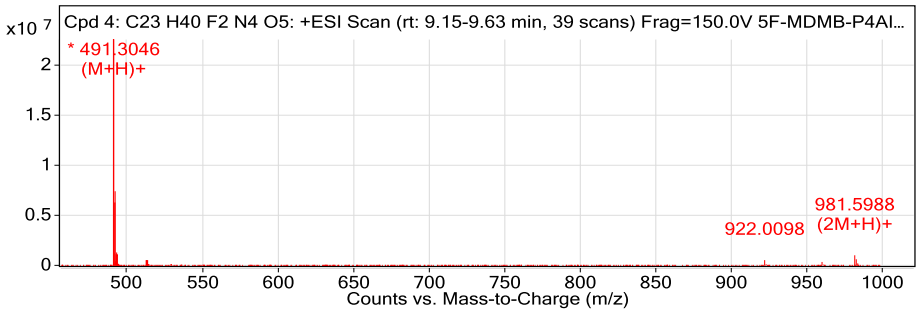
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
491.3045	1	22510084	C23 H40 F2 N4 O5	(M+H)+
492.3076	1	6675707.01	C23 H40 F2 N4 O5	(M+H)+
493.3102	1	1127573.17	C23 H40 F2 N4 O5	(M+H)+
494.3128	1	138417.17	C23 H40 F2 N4 O5	(M+H)+
513.2861	1	454760.31	C23 H40 F2 N4 O5	(M+Na)+
514.2889	1	129107.56	C23 H40 F2 N4 O5	(M+Na)+
529.2597	1	118700.27	C23 H40 F2 N4 O5	(M+K)+
981.5992	1	1695042.13		(2M+H)+
982.6026	1	1025016.56		(2M+H)+
983.6051	1	334577.3		(2M+H)+

--- End Of Report ---

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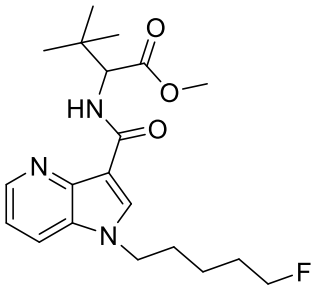
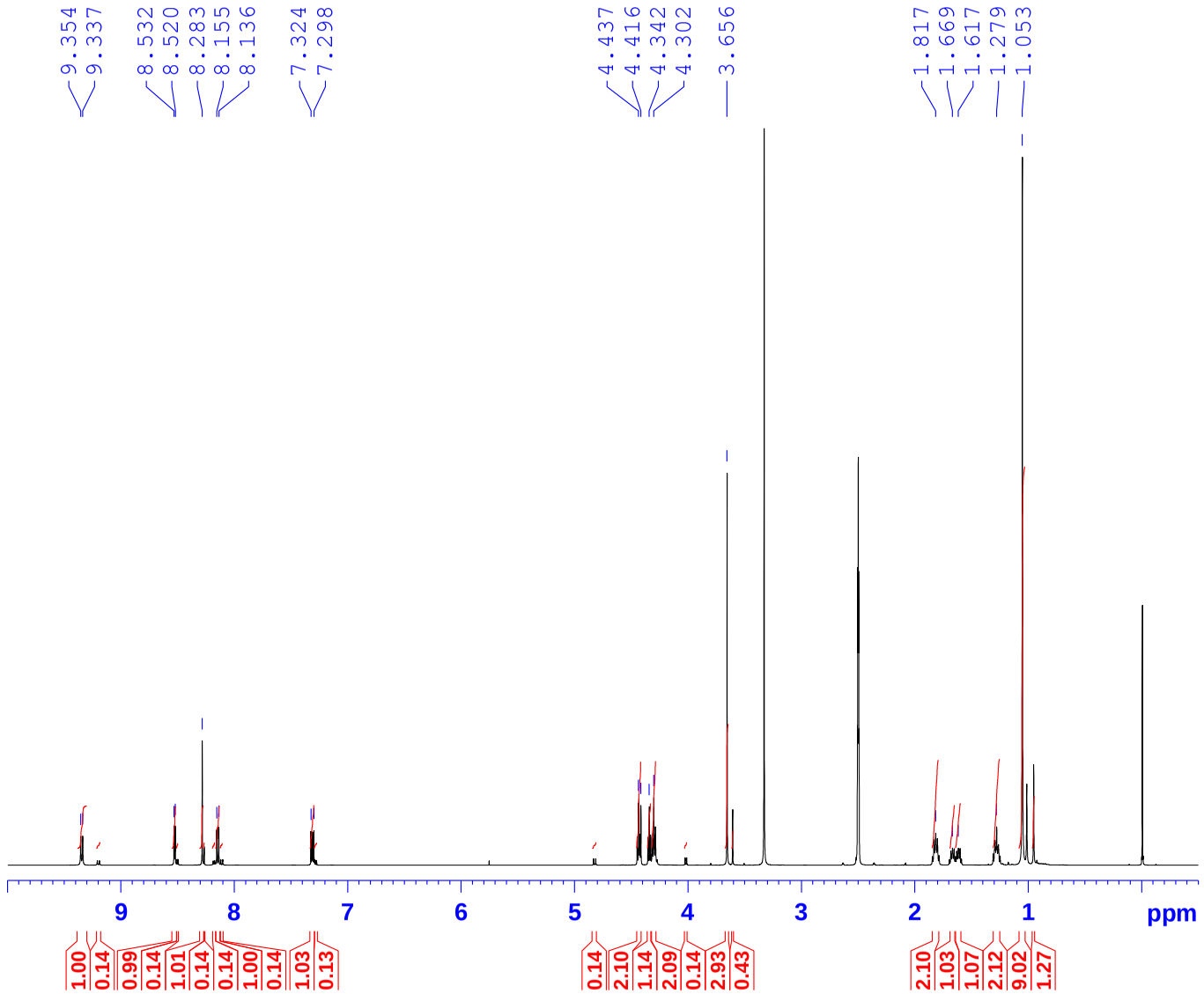
Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**2080-19**
Received date:	July 30, 2019
Our notebook code:	NFL-2080-19
NMR sample preparation:	7.0 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, ^{19}F, *gs*-NOESY
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{20}H_{28}FN_3O_3$ Exact Mass: 377,2115 Molecular Weight: 377,4604
Chemical name:	Methyl 2-(1-(5-fluoropentyl)-1*H*-pyrrolo[3,2-*b*]pyridine-3-carboxamido)-3,3-dimethylbutanoate
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - The sample consists of herein identified compound and unknown impurity in molar ratio of 1.00:0.14, 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:	August 12, 2019

NFL-2080-19
1H



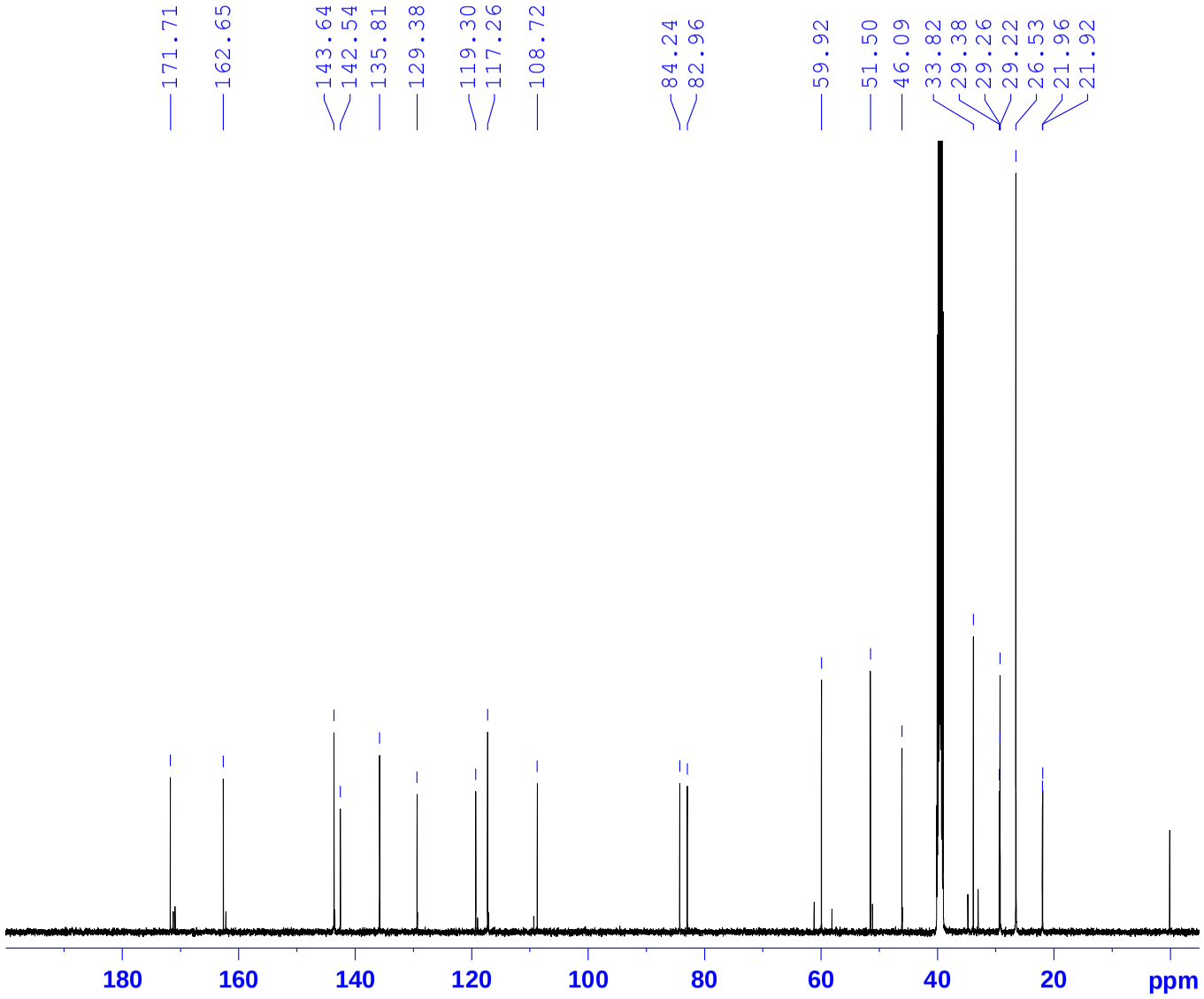

```
Current Data Parameters
NAME         NFL-2080-19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190731
Time              21.18
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                   101
DW               50.000 usec
DE                6.50 usec
TE                297.2 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           500.1300039 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

NFL-2080-19
13C

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Current Data Parameters
NAME         NFL-2080-19
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190801
Time                1.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  7168
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                297.5 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           125.7578489 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```