EUROPEAN PROJECT RESPONSE to challenges in forensic drugs analyses

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

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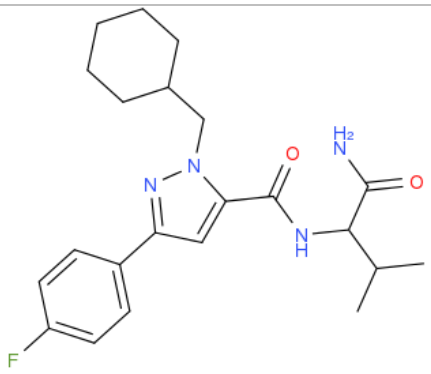
ANALYTICAL REPORT

AB-CHFUPYCA, (C22H29FN4O2)

N-(1-amino-3-methyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-3-(4-fluorophenyl)-1H -pyrazole-5-carboxamide

Remark – other NPS detected: none

Sample ID:	1181-15
Sample description:	powder - white
Sample type:	P- purchased (test purchase of another desired compound from CHINA)
Date of entry:	7/10/2015

Substance identified-structure[i] (base form)	
Systematic name	N-(1-amino-3-methyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-3-(4-fluorophenyl)-1H -pyrazole-5-carboxamide
Other names	
Formula (per base form)	C22H29FN4O2
M_w (g/mol)	400,5
Salt form	base
Smiles	NC(C(C(C)C)NC(=O)C1=CC(=NN1CC1CCCCC1)C1=CC=C(C=C1)F)=O
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	pure

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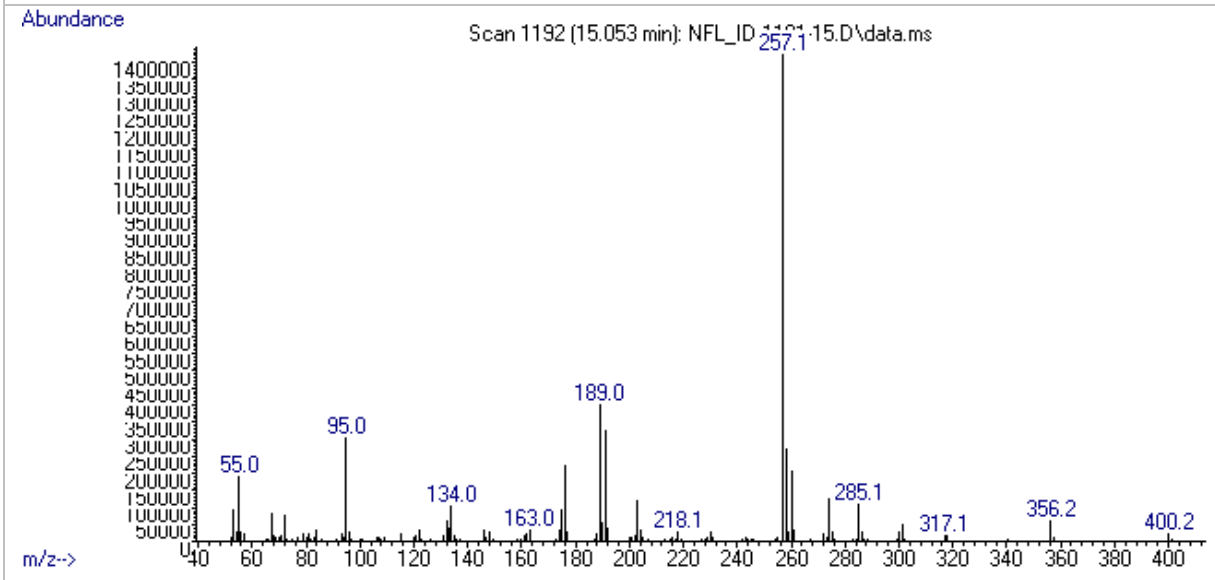
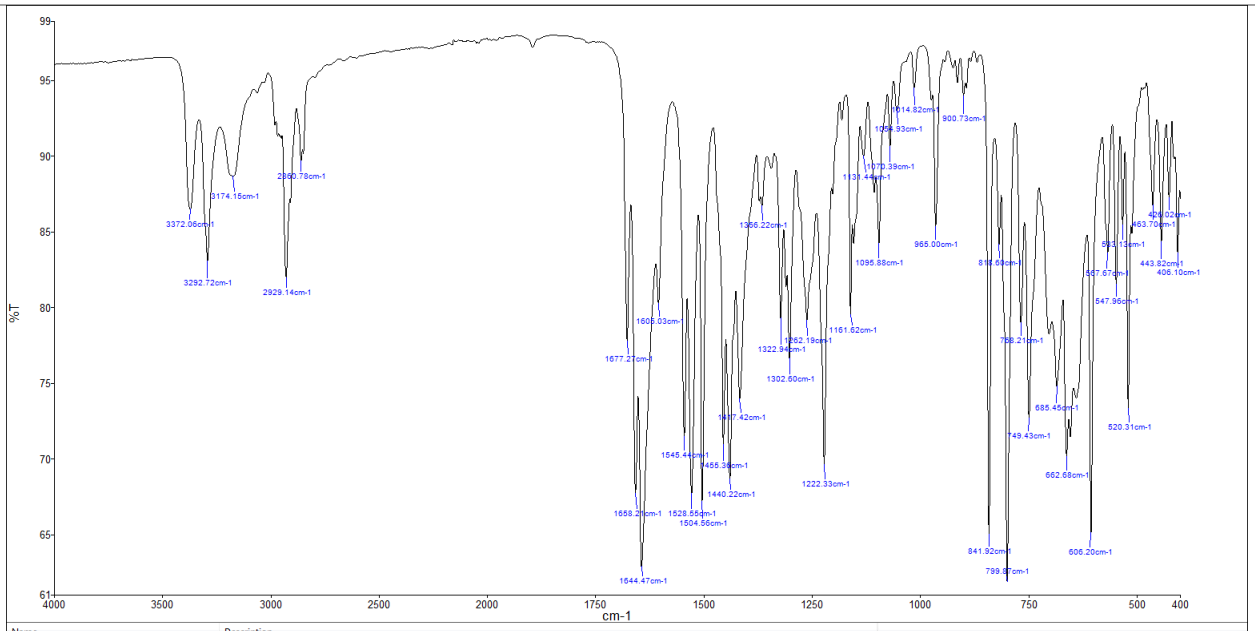
Supporting information

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	BP(1): 257; BP(2): 189,BP(3) :95,
FTIR-ATR	+	base
FTIR (condensed phase)	/	not scanned
HPLC-TOF	+	Exact mass: 400,2275, measured/ Δppm:-0,57; formula:C22H29FN4O2
NMR-confirmed		pending
validation		MS consistent with see article: http://link.springer.com/article/10.1007%2Fs11419-015-0283-8#page-1
other		RT (NFL)= 15,05 min

MS spectrum (EI)



FTIR - ATR



[i] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Target Compound Screening Report

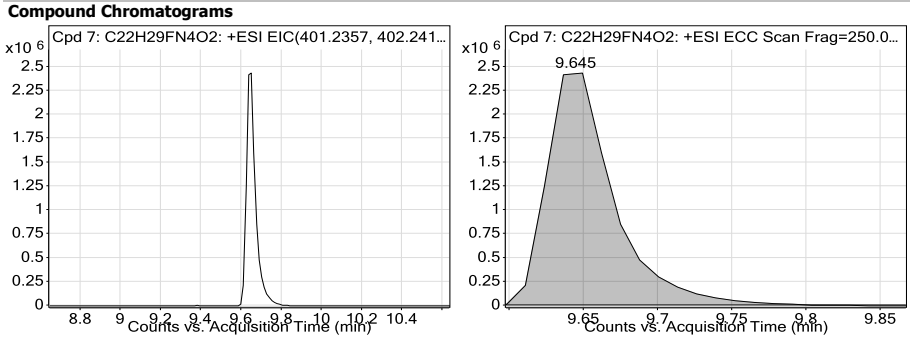
Data File	ID_1181-15_TOF.d	**Sample Name**	ID 1181
Sample Type	Sample	**Position**	P1-E2
Instrument Name	SG13170002	**User Name**	
Acq Method	droge general-19-6-2015-XDB-C18-ESI-poz-frag250.m	**Acquired Time**	6/19/2015 11:55:52 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

Compound Table

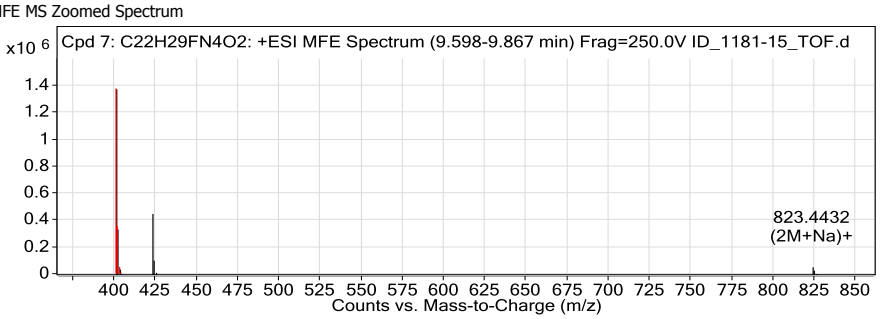
Label	Tgt Name	MFG Formula	Tgt Formula	Obs. RT	Obs. Mass
Cpd 7: C22H29FN4O2	C22H29FN4O2	C22 H29 F N4 O2	C22 H29 F N4 O2	9.645	400.2277

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error	Tgt Formula	Find Cpds Algorith
C22H29FN4O2	401.2349	9.645	400.2277	9.645	C22 H29 F N4 O2	400.2275	-0.57	C22 H29 F N4 O2	Find by Molecular Feature

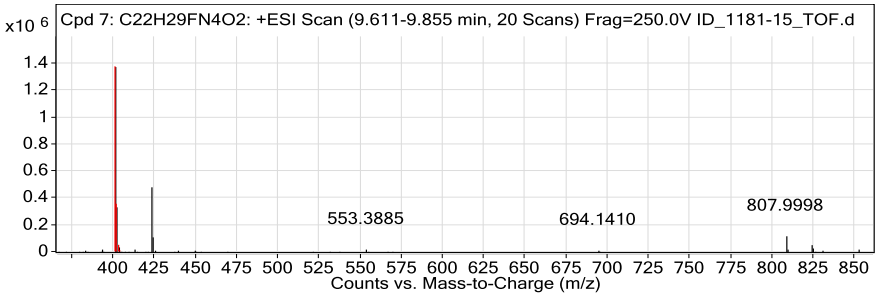
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
401.2349	1	1378105.63	C22 H29 F N4 O2	(M+H)+
402.2381	1	336202.72	C22 H29 F N4 O2	(M+H)+
403.2406	1	42526.46	C22 H29 F N4 O2	(M+H)+
404.2433	1	4483.36	C22 H29 F N4 O2	(M+H)+
423.2169	1	450145.13		(M+Na)+
424.2198	1	105213.93		(M+Na)+
425.2226	1	14894.87		(M+Na)+
823.4432	1	59651.26		(2M+Na)+
824.446	1	29240.72		(2M+Na)+
825.4488	1	7794.32		(2M+Na)+

--- End Of Report ---

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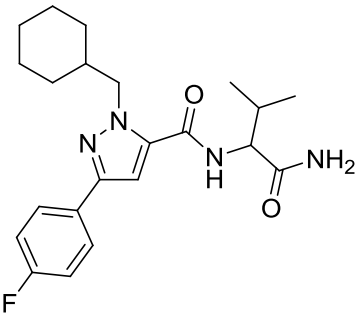
REPORT

Sample ID:	**1181-15**
Our notebook code:	P-1181-15
NMR sample preparation:	15 mg dissolved in 0.7 mL $DMSO_6$
NMR experiments:	^{1}H, ^{13}C, ^{19}F, ^{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 chemical name:	 *N*-(1-amino-3-methyl-1-oxobutan-2-yl)-1-(cyclohexylmethyl)-3-(4-fluorophenyl)-1*H*-pyrazole-5-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj
Date of report:	July 11, 2015

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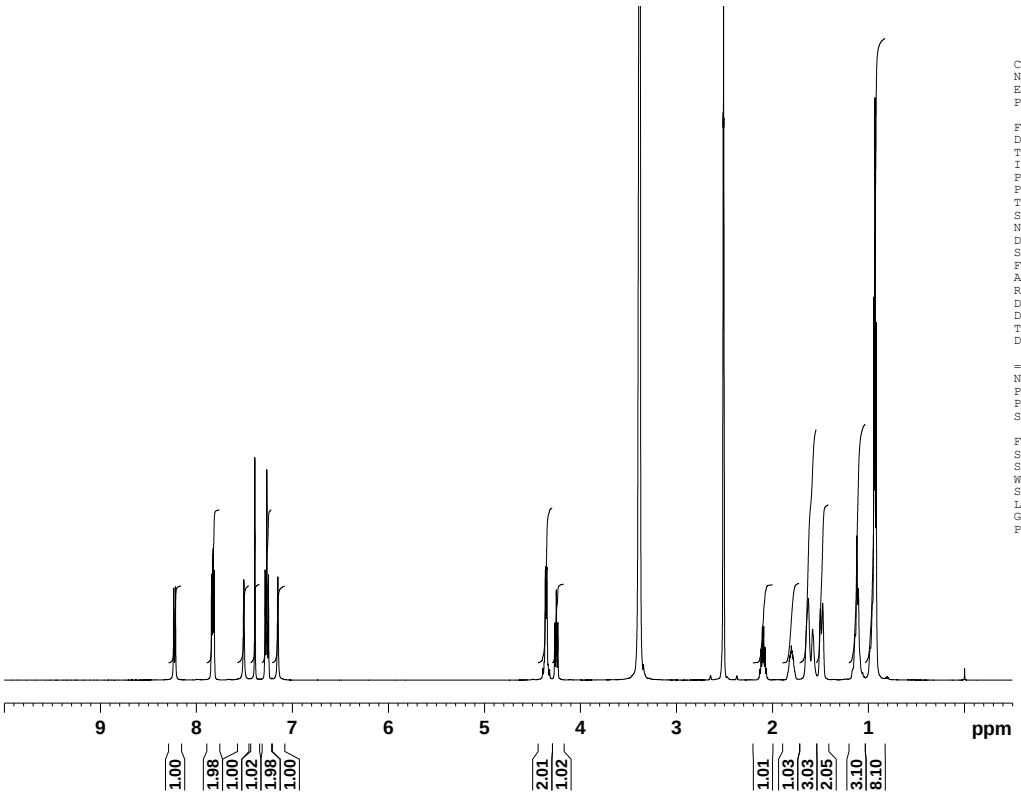


```
Current Data Parameters
NAME                  1181-15
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150708
Time                  21.40
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       32
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ               3.1719923 sec
RG                     71.8
DW                  48.400 usec
DE                   6.50 usec
TE                  296.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.90 usec
PLW1           26.00000000 W
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                    65536
SF              500.1300018 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

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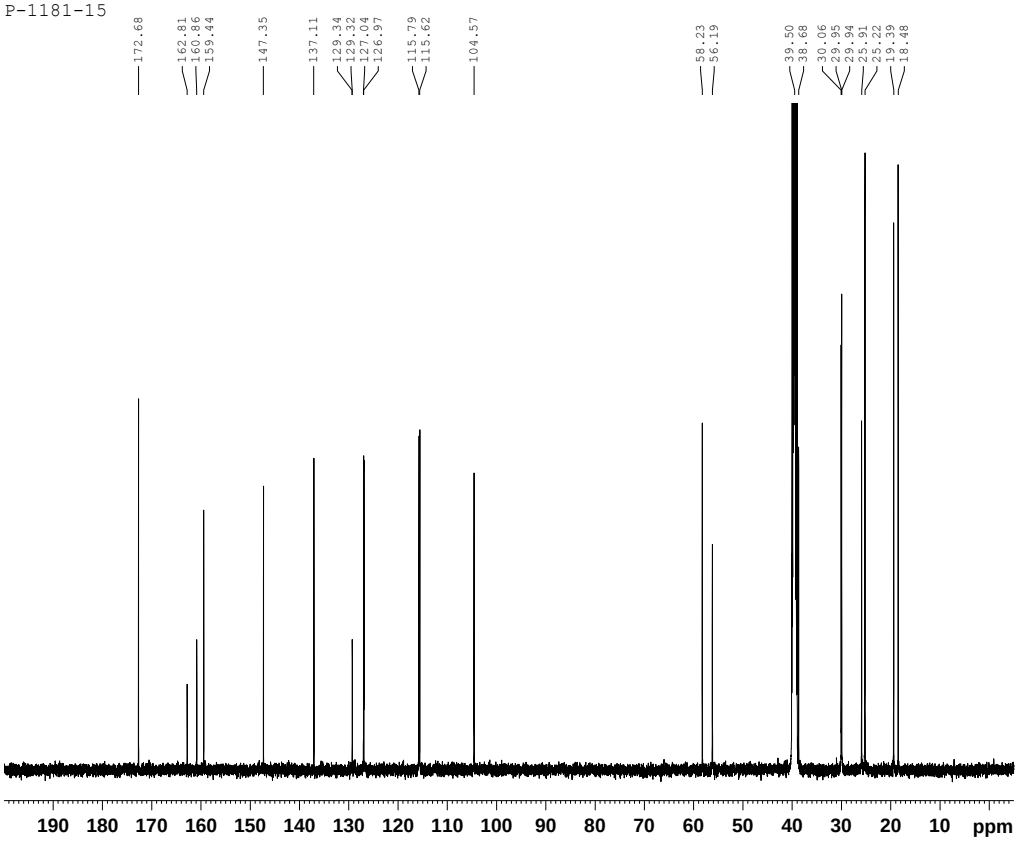


```
Current Data Parameters
NAME                  1181-15
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150709
Time                   1.26
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                      5120
DS                         4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ               1.1010548 sec
RG                      2050
DW                  16.800 usec
DE                   6.50 usec
TE                  296.0 K
D1              1.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.00 usec
PLW1          122.00000000 W
SFO1          125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PLW2           26.00000000 W
PLW12           0.32179001 W
PLW13           0.20595001 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF              125.7578436 MHz
WDW                      EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```