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EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

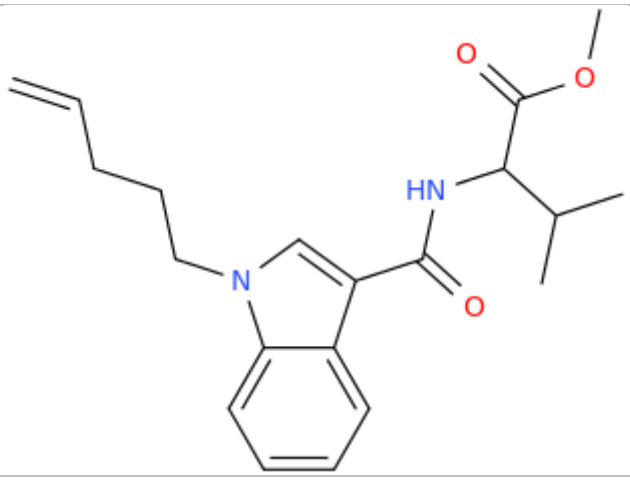
ANALYTICAL REPORT

MMB-022 (MMB-4en-PICA) (C20H26N2O3)

methyl 3-methyl-2-{[1-(pent-4-en-1-yl)-1H-indol-3-yl]formamido}butanoate

Remark – other NPS detected: none

Sample ID:	1955-18
Sample description:	crystalinic
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of sample receipt (DD/MM/YYYY):	26/07/2018
Date of entry (DD/MM/YYYY) into NFL database:	13/08/2018
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	methyl 3-methyl-2-{[1-(pent-4-en-1-yl)-1H-indol-3-yl]formamido}butanoate
Other names	methyl 3-methyl-2-{[1-(pent-4-en-1-yl)-1H-indole-3-carbonyl]amino}butanoate; Methyl 3-methyl-2-(1-(pent-4-en-1-yl)-1H-indole-3-carboxamido)butanoate; methyl (1-(pent-4-en-1-yl)-1H-indole-3-carbonyl)valinate, MMB-022; MMB-4en-PICA;
Formula (per base form)	C20H26N2O3
M_w (g/mol)	342,44
Salt form/anions detected	base
StdInChIKey (per base form)	RQVMOMGJHFPBNR-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	> 96 % based on H1 NMR

[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

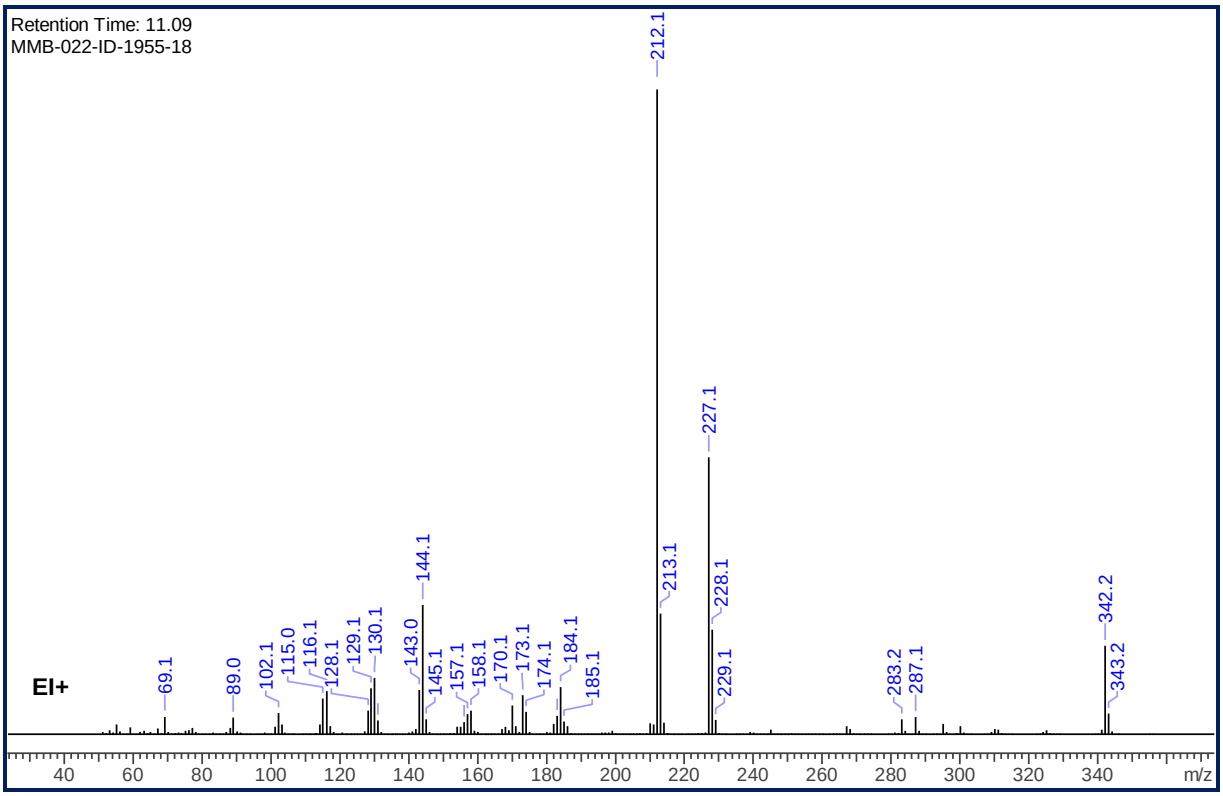
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	partially

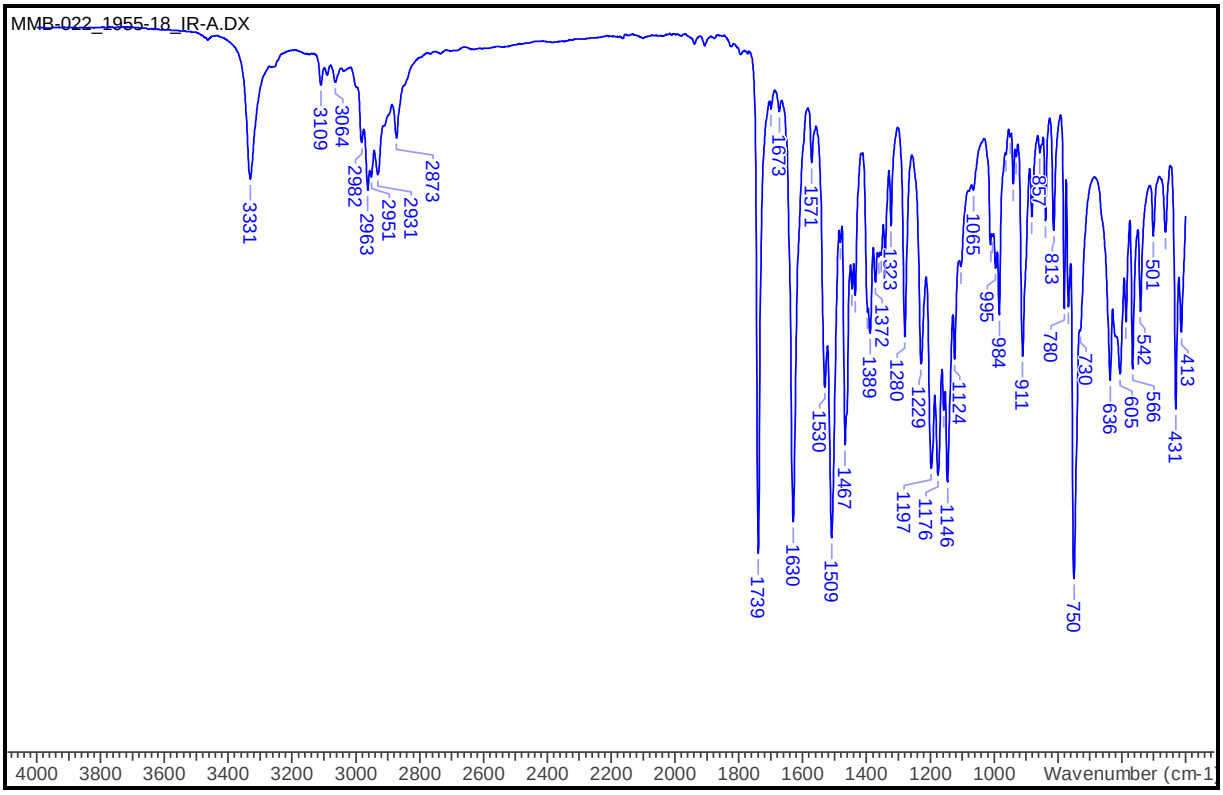
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 11,1 BP(1): 212; BP(2): 227,BP(3) :144,
HPLC-TOF	+	Exact mass (theoretical): 342,1943; measured value Δppm:0,28; formula:C20H26N2O3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	spot tests only
NMR (in FKKT)	+	
validation		
other		

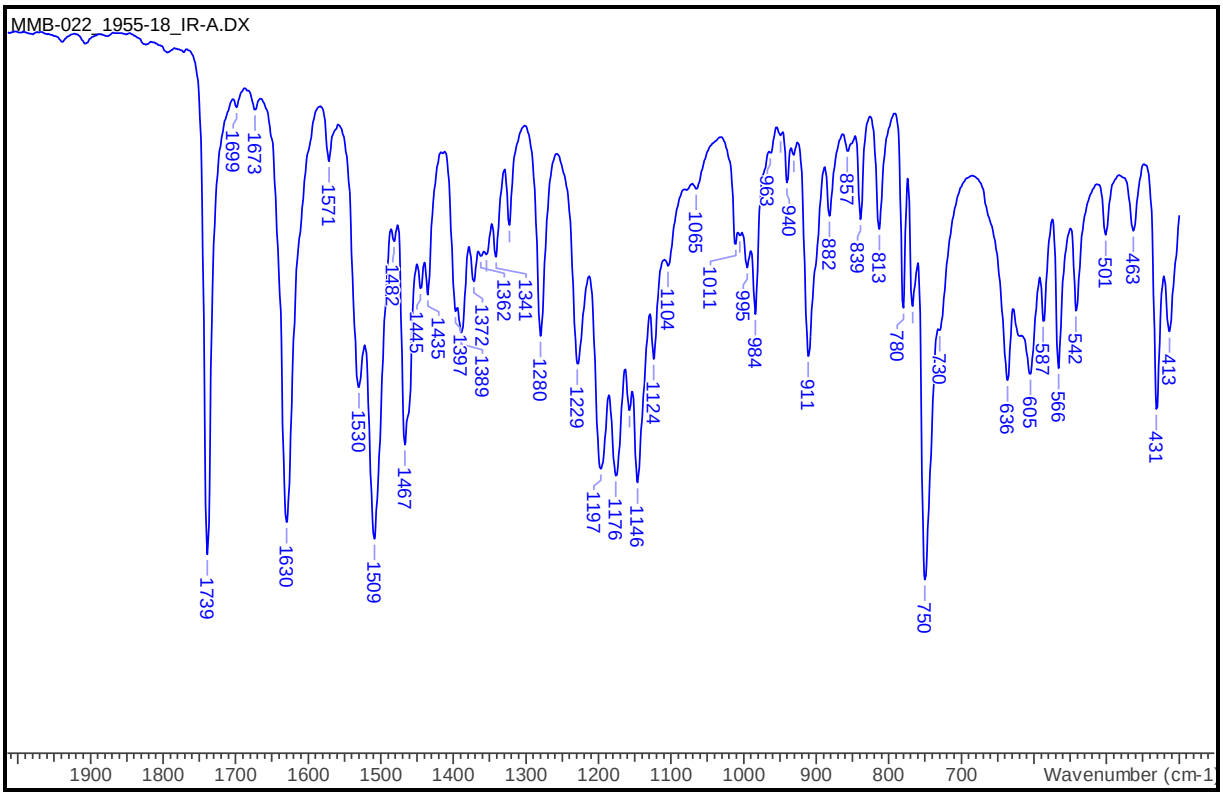
ANALYTICAL RESULTS

MS (EI 70 eV)

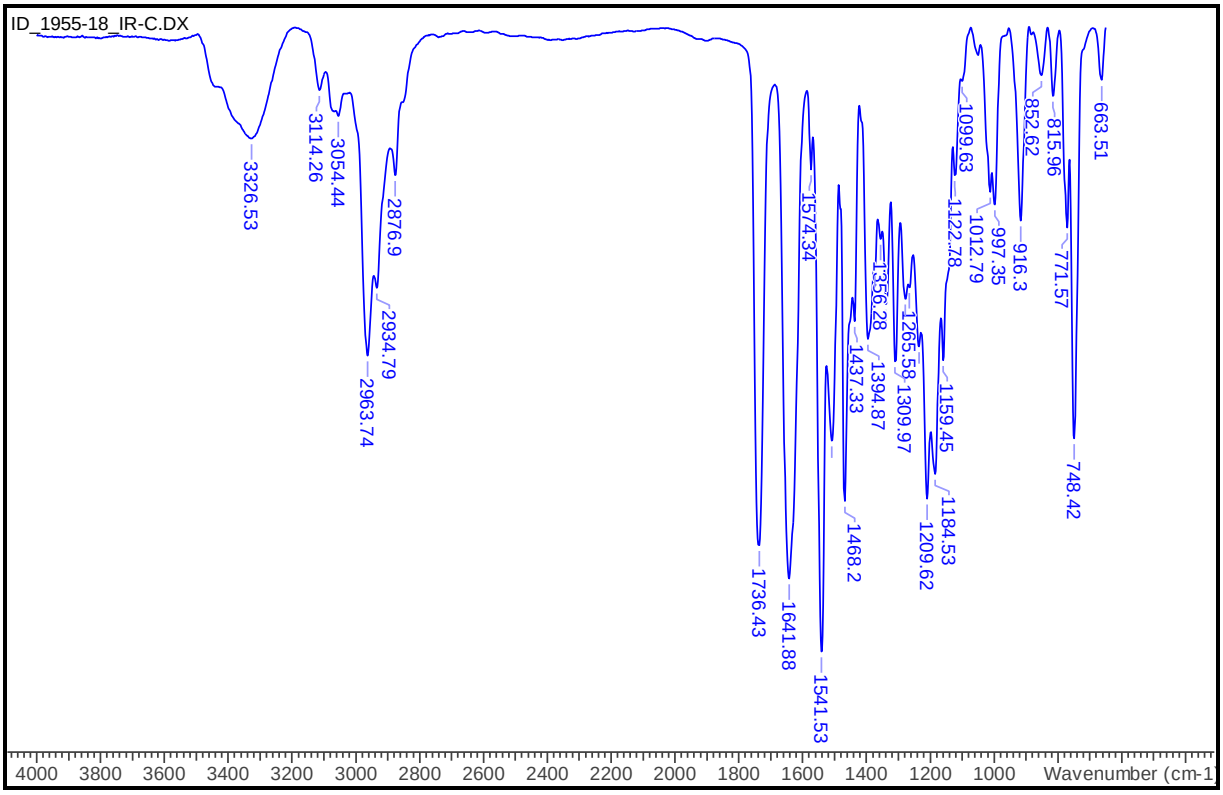


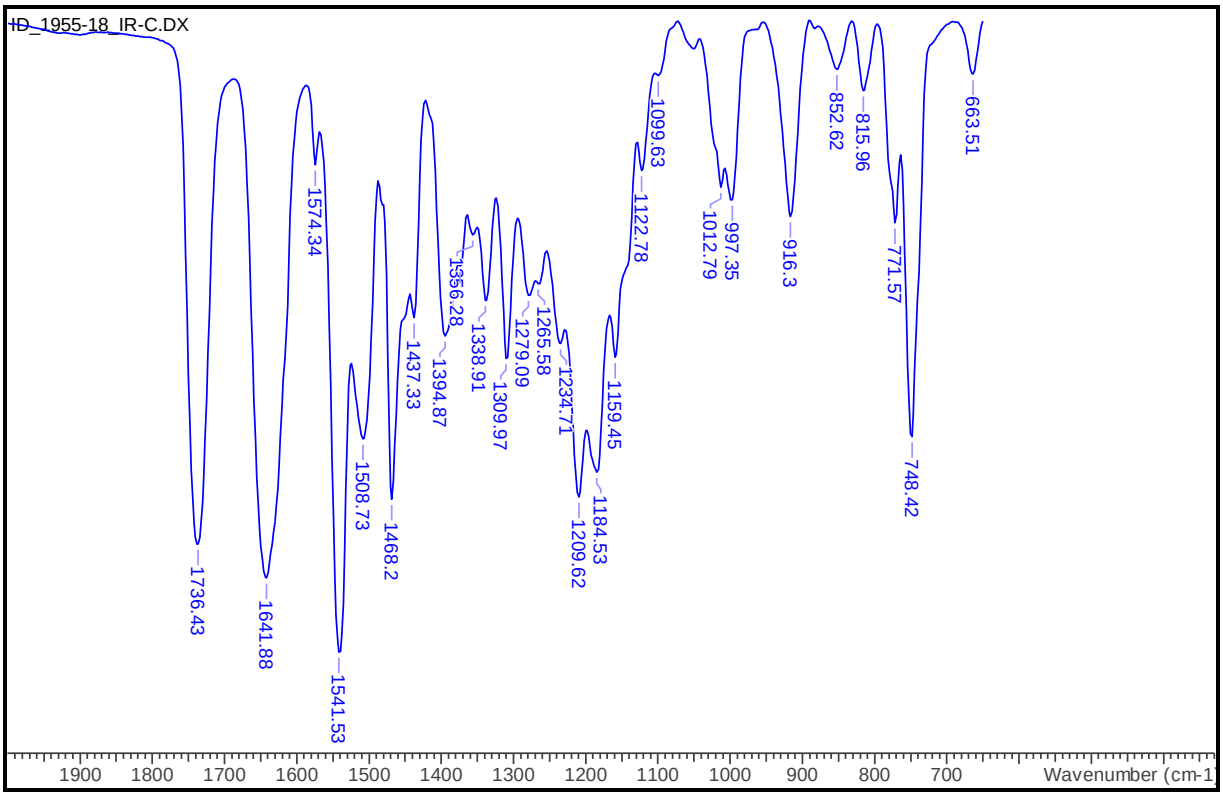
FTIR-ATR - direct measurement (sample as received)





IR (solid phase – after chromatographic separation)





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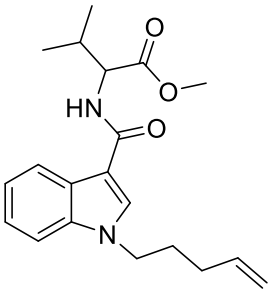
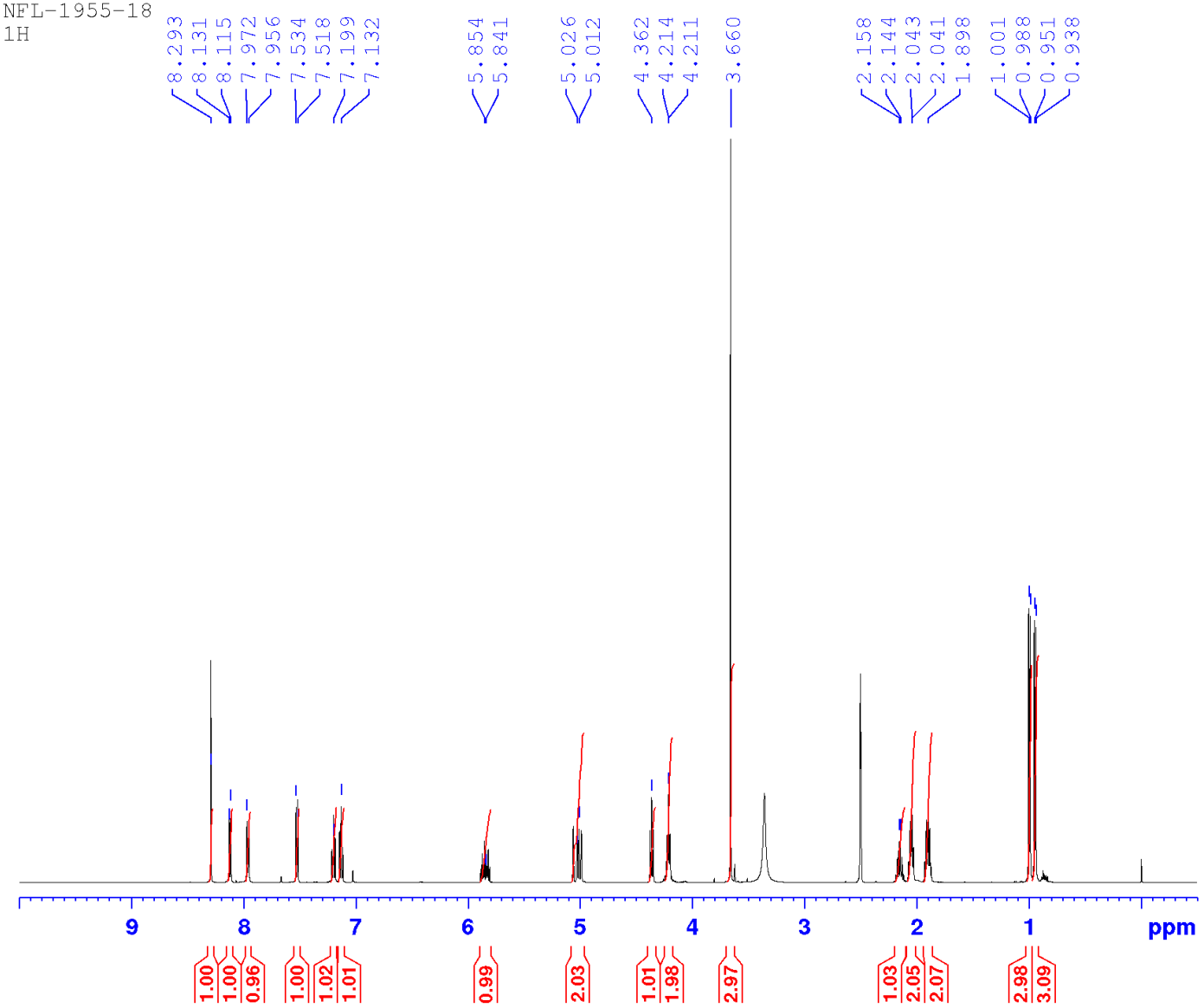
University
of Ljubljana

Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**1955-18**
Received date:	August 6, 2018
Our notebook code:	NFL-1955-18
NMR sample preparation:	26.0 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
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{20}H_{26}N_2O_3$ Exact Mass: 342,1943 Molecular Weight: 342,4390
Chemical name:	methyl (1-(pent-4-en-1-yl)-1*H*-indole-3-carbonyl)valinate
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >96% 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:	August 10, 2018

NFL-1955-18
1H



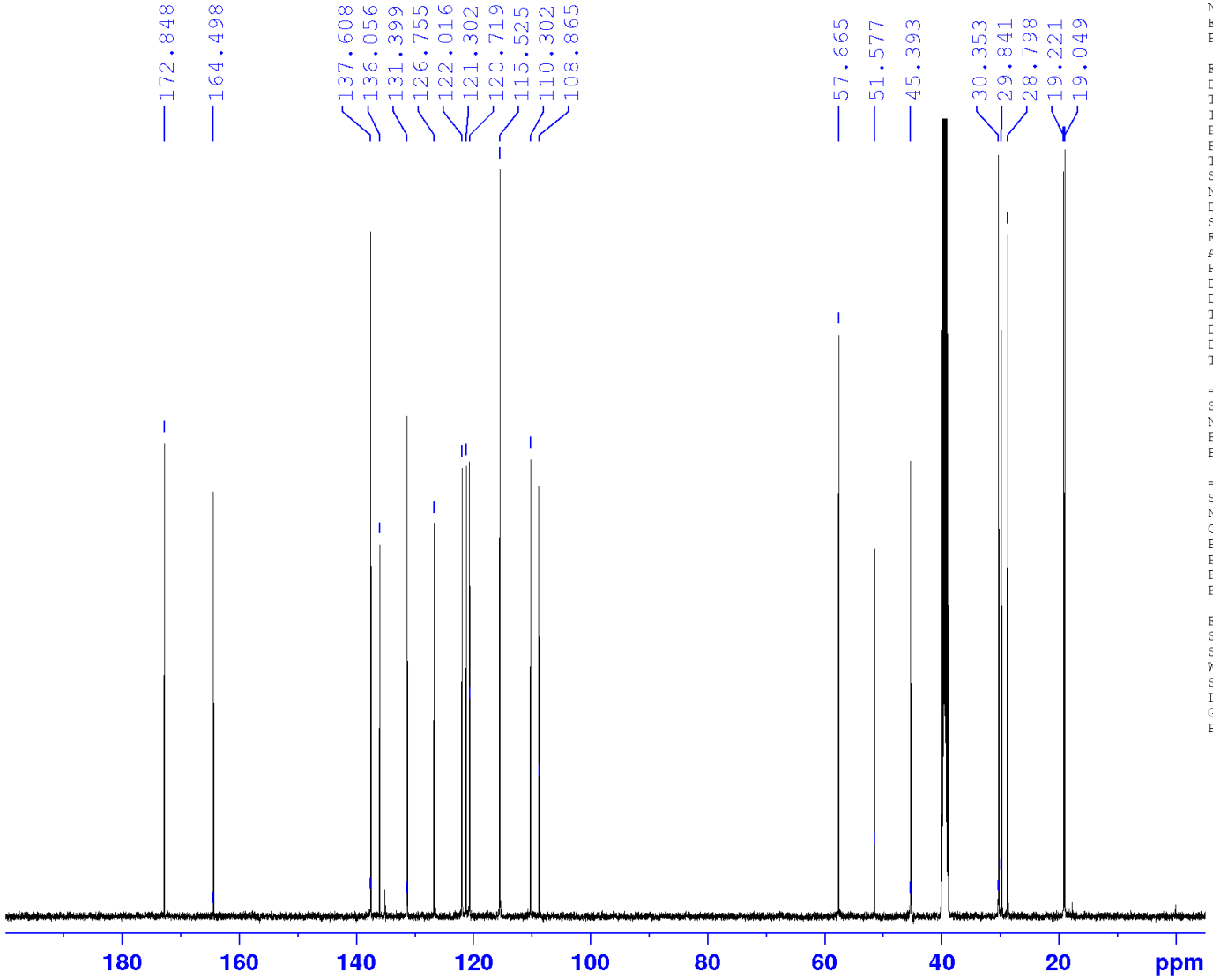

```
Current Data Parameters
NAME         NFL-1955-18
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180806
Time               23.03
INSTRUM            spect
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         500.1330885 MHz
NUC1                   1H
P1                 8.70 usec
PLW1        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-1955-18
13C




```
Current Data Parameters
NAME         NFL-1955-18
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180807
Time                1.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 3072
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           2.00000000 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          125.7578452 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
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