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RESPONSE 2
to challenges in forensic drugs analyses

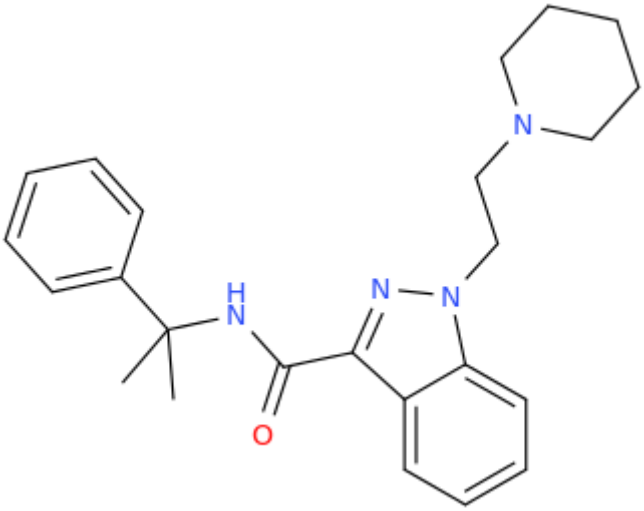
ANALYTICAL REPORT

CUMYL-PIPETINACA (C24H30N4O)

N-(2-phenylpropan-2-yl)-1-[2-(piperidin-1-yl)ethyl]-1H-indazole-3-carboxamide

Remark – other active cpd. detected none

Sample ID:	2001-18
Sample description:	liquid - yellow
Sample type:	RM-reference material
Comments:	CHI Lot# 20 907,
Date of entry (DD/MM/YYYY):	22/01/2019

Substance identified-structure[1] (base form)	
Systematic name:	N-(2-phenylpropan-2-yl)-1-[2-(piperidin-1-yl)ethyl]-1H-indazole-3-carboxamide
Other names:	SGT-234; N-(2-Phenylpropan-2-yl)-1-[2-(piperidin-1-yl)ethyl]-1H-indazole-3-carboxamide
Formula (per base form)	C24H30N4O
M_w (g/mol)	390,53
Salt form:	base
StdInChIKey (per base form)	MTMVUBQQIGZSQB-UHFFFAOYSA-N
Other active cpd. detected	none
Add.info (purity..)	97,8 %

[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): 14,6 BP(1): 98; BP(2): 99,BP(3) :103,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 390,242 / measured Δppm: -0,82

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.

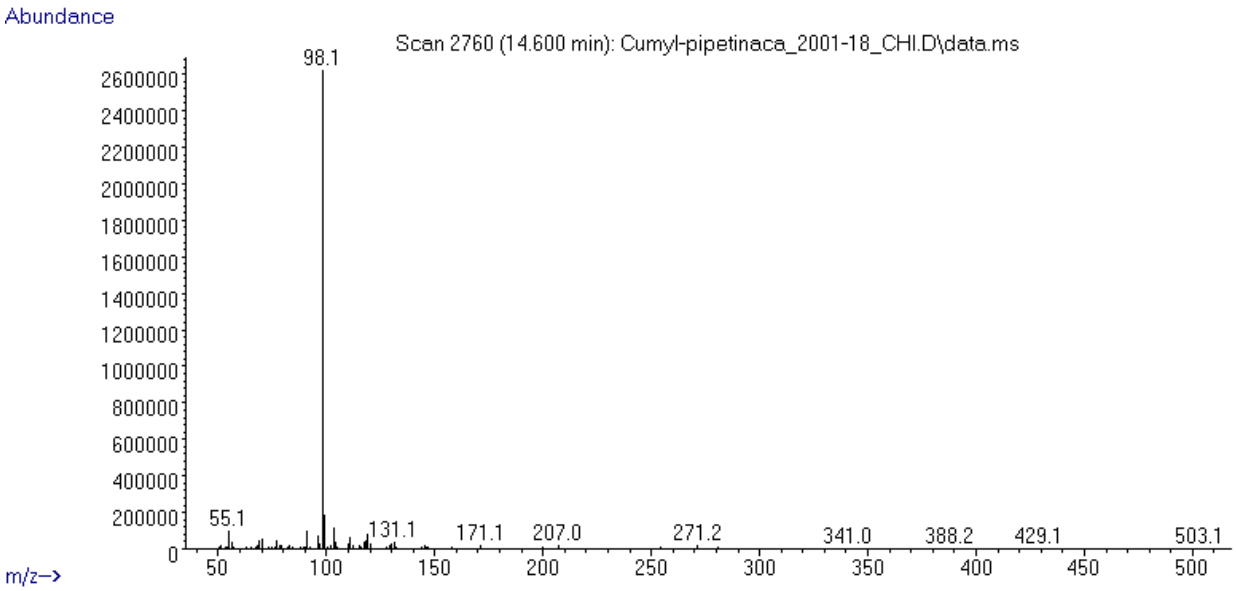
2. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1}; resolution 4cm^{-1}

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}.

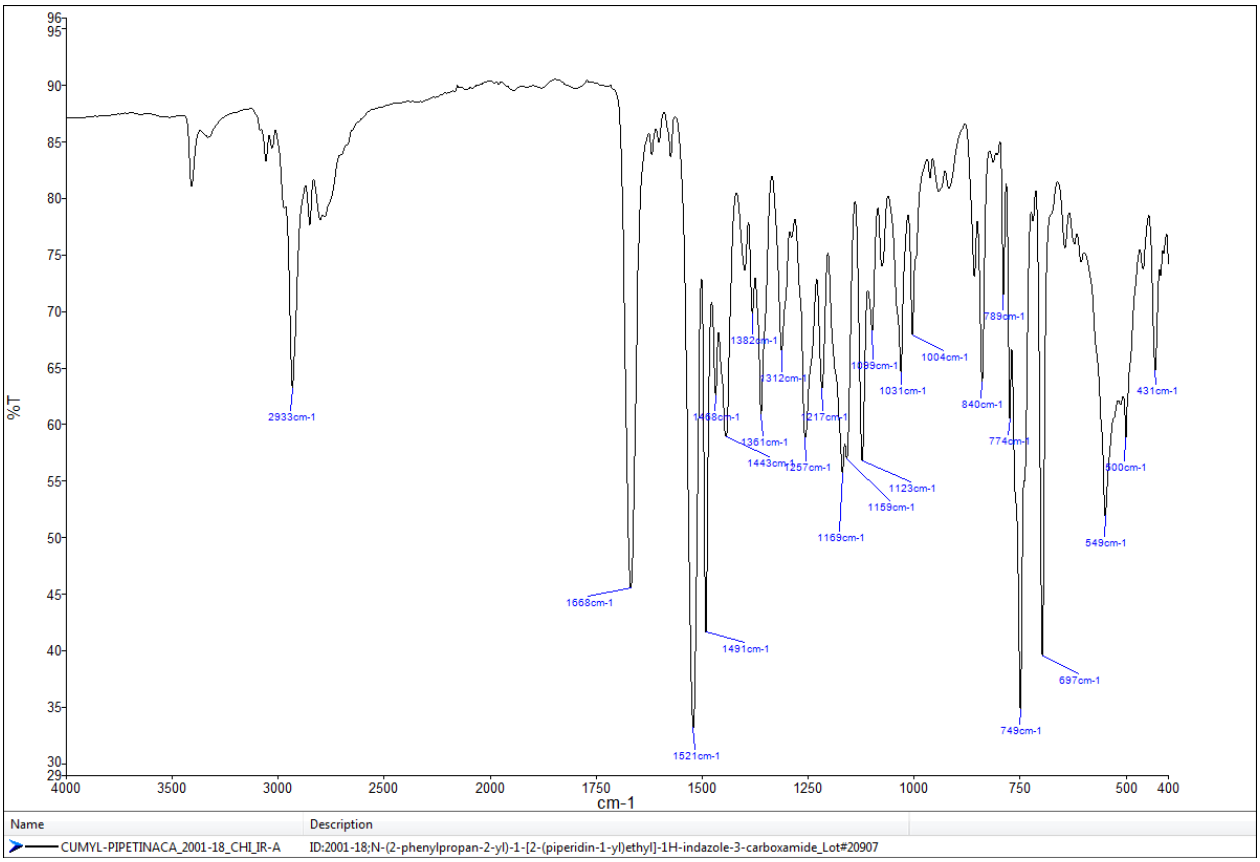
4. **HPLC-TOF** (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over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

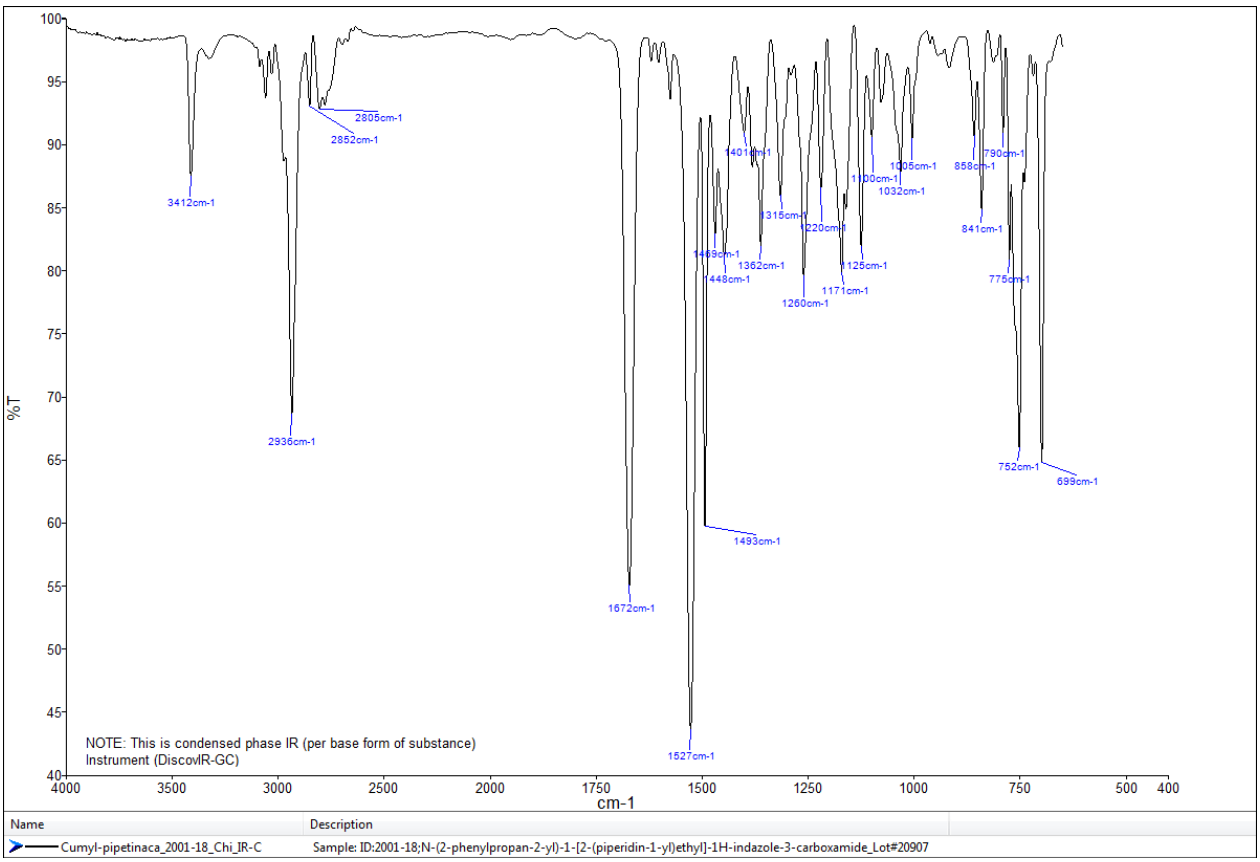
MS (EI)



FTIR-ATR – direct measurement



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


%T
cm-1
3412cm-1
2936cm-1
2852cm-1
2805cm-1
1672cm-1
1527cm-1
1493cm-1
1469cm-1
1448cm-1
1401cm-1
1362cm-1
1315cm-1
1260cm-1
1220cm-1
1171cm-1
1125cm-1
1100cm-1
1032cm-1
1005cm-1
858cm-1
841cm-1
790cm-1
775cm-1
752cm-1
699cm-1
NOTE: This is condensed phase IR (per base form of substance)
Instrument (DiscovIR-GC)
Name
Description
Cumyl-pipetinaca_2001-18_Chi_IR-C
Sample: ID:2001-18;N-(2-phenylpropan-2-yl)-1-[2-(piperidin-1-yl)ethyl]-1H-indazole-3-carboxamide_Lot#20907

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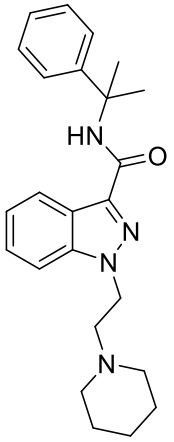
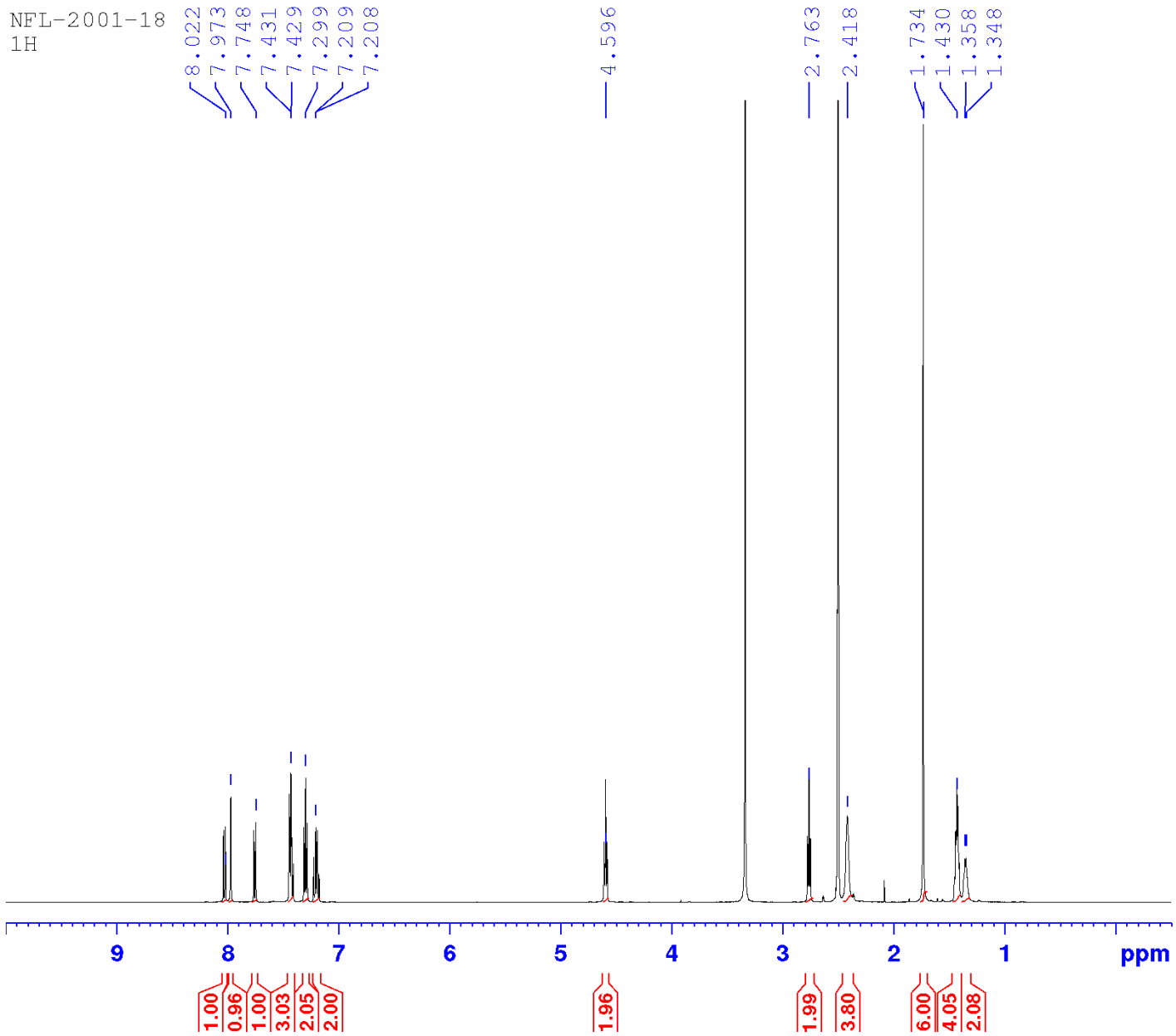
Faculty *of Chemistry*
and Chemical Technology

R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**2001-18**
Received date:	November 29, 2018
Our notebook code:	NFL-2001-18
NMR sample preparation:	5.0 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
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{24}H_{30}N_4O$ Exact Mass: 390,2420 Molecular Weight: 390,5310
Chemical name:	*N*-(2-phenylpropan-2-yl)-1-(2-(piperidin-1-yl)ethyl)-1H-indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >98% 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:	December 13, 2018

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Current Data Parameters
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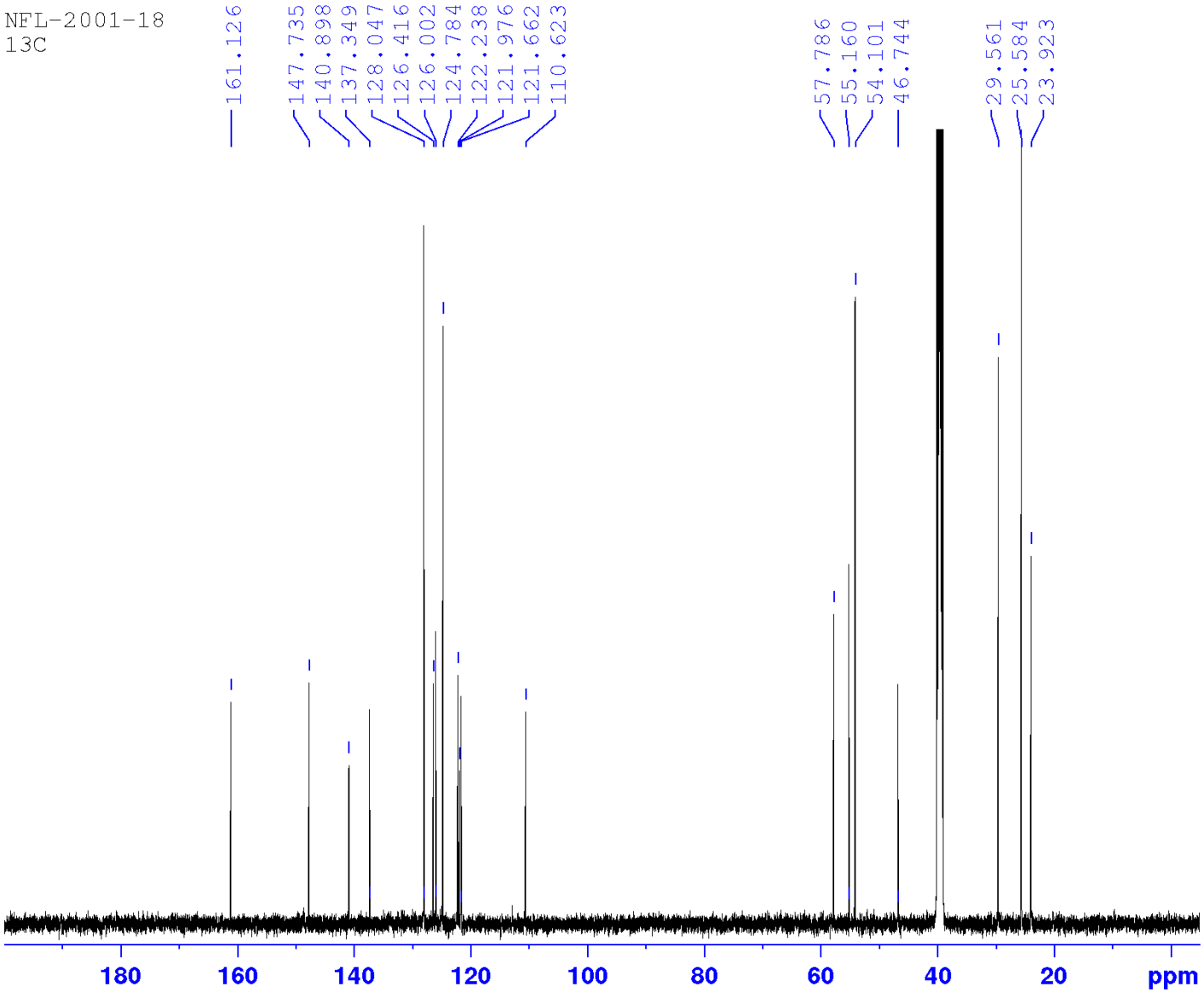
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181130
Time                0.03
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                   114
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           500.1300041 MHz
WDW                  EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

NFL-2001-18
13C




```
Current Data Parameters
NAME         NFL-2001-18
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181130
Time                4.01
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            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           125.7578462 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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