

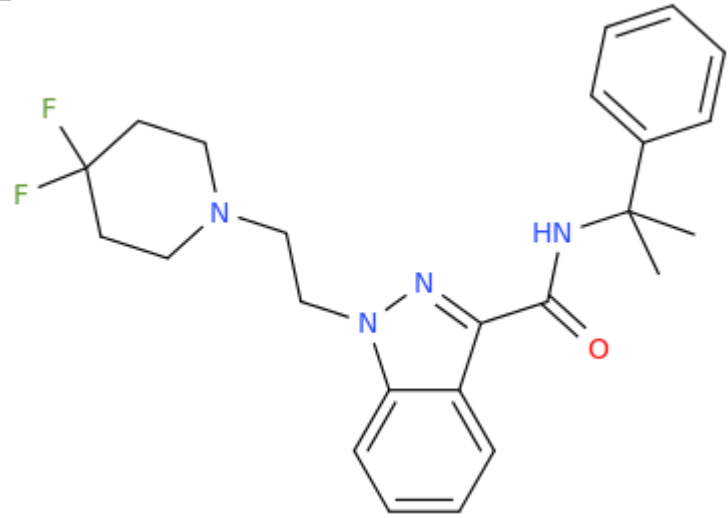
ANALYTICAL REPORT

4,4-F-Cumyl-PIPETINACA (SGT-235) (C₂₄H₂₈F₂N₄O)

1-[2-(4,4-difluoropiperidin-1-yl)ethyl]-N-(2-phenylpropan-2-yl)-1H-indazole-3-carboxamide

Remark – other active cpd. detected **none**

Sample ID:	2000-18
Sample description:	liquid - yellow
Sample type:	RM-reference material
Comments:	CHI Lot# 20 911,
Date of entry (DD/MM/YYYY):	22/01/2019

Substance identified-structure ¹ (base form)	
Systematic name:	1-[2-(4,4-difluoropiperidin-1-yl)ethyl]-N-(2-phenylpropan-2-yl)-1H-indazole-3-carboxamide
Other names:	N-(2-Phenylpropan-2-yl)-1-[2-(4,4-difluoropiperidin-1-yl)ethyl]-1H-indazole-3-carboxamide
Formula (per base form)	C ₂₄ H ₂₈ F ₂ N ₄ O
M _w (g/mol)	426,51
Salt form:	base
StdInChIKey (per base form)	UGIXAWXZCCAHAHAG-UHFFFAOYSA-N
Other active cpd. detected	none
Add.info (purity..)	> 98%

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): 14,38 BP(1): 134; BP(2): 147,BP(3) :135,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 426,2231 / measured Δ ppm: -0,26

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 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 μ 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. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1} ; resolution 4 cm^{-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 °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.

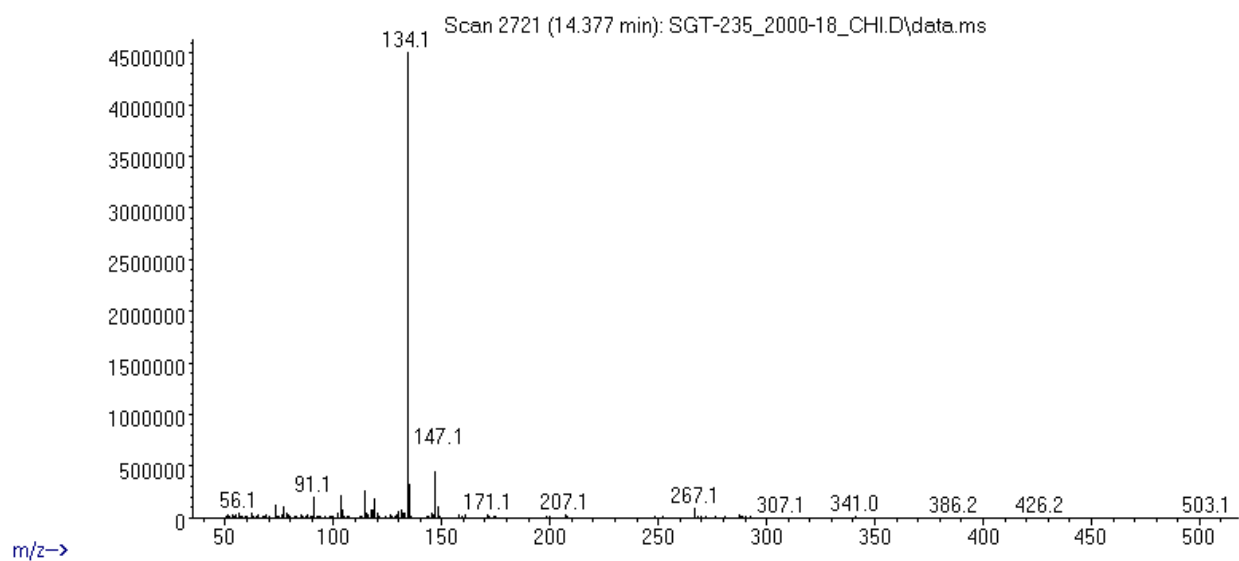
IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm^{-1} .

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 μ 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 (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.

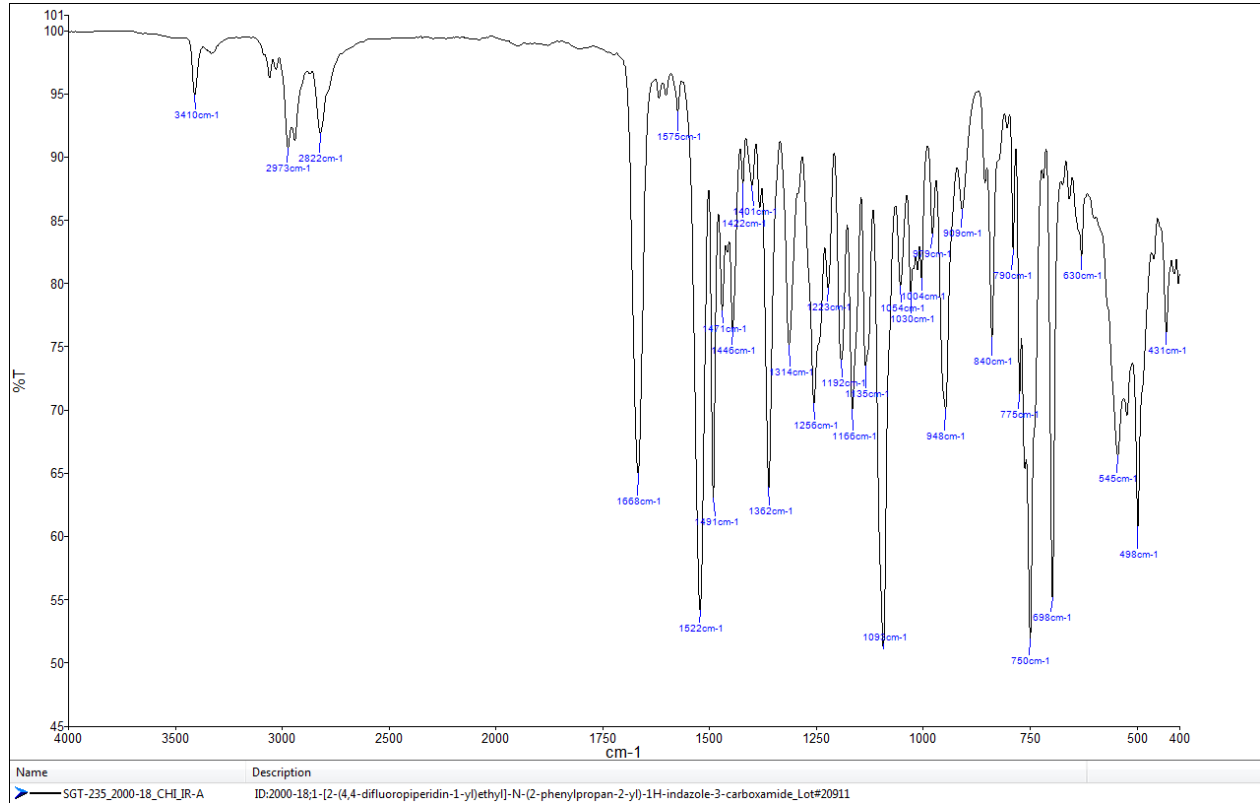
ANALYTICAL RESULTS

MS (EI)

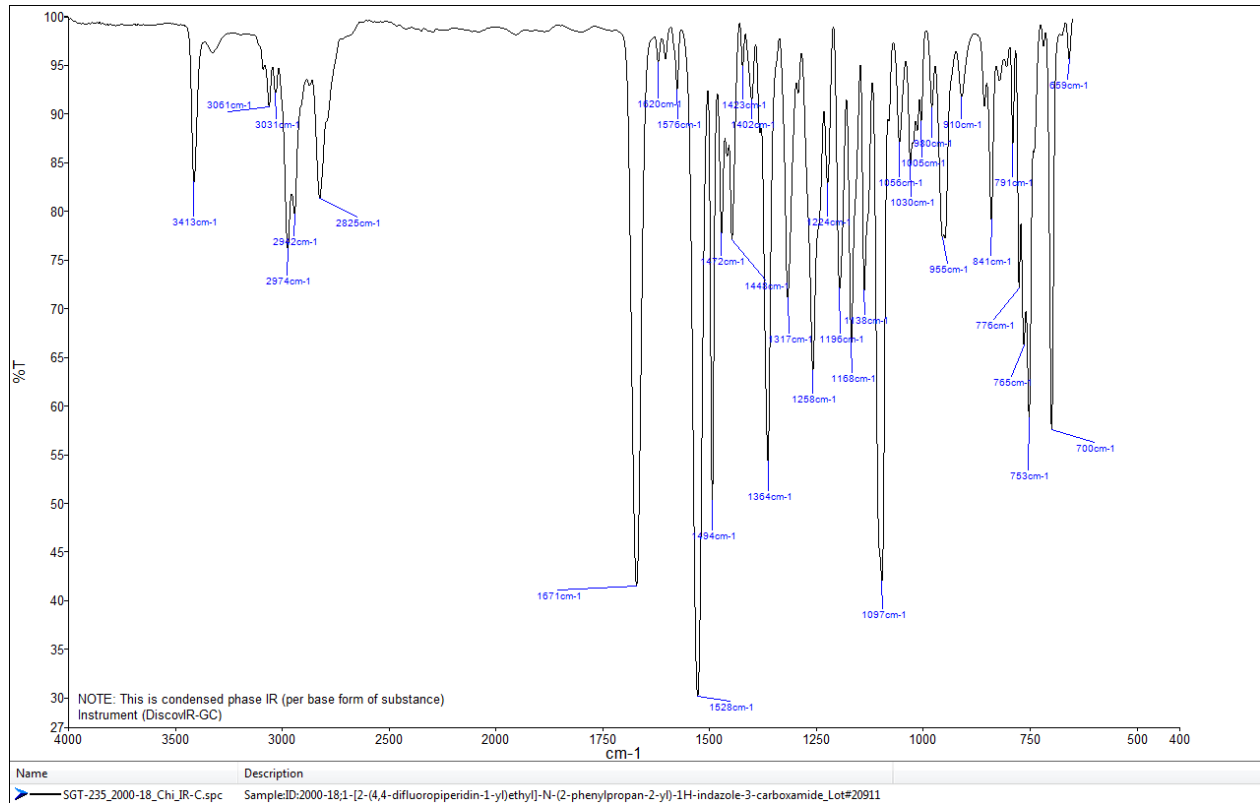
Abundance



FTIR-ATR – direct measurement



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

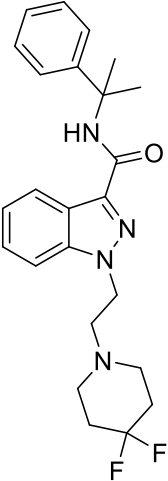


University
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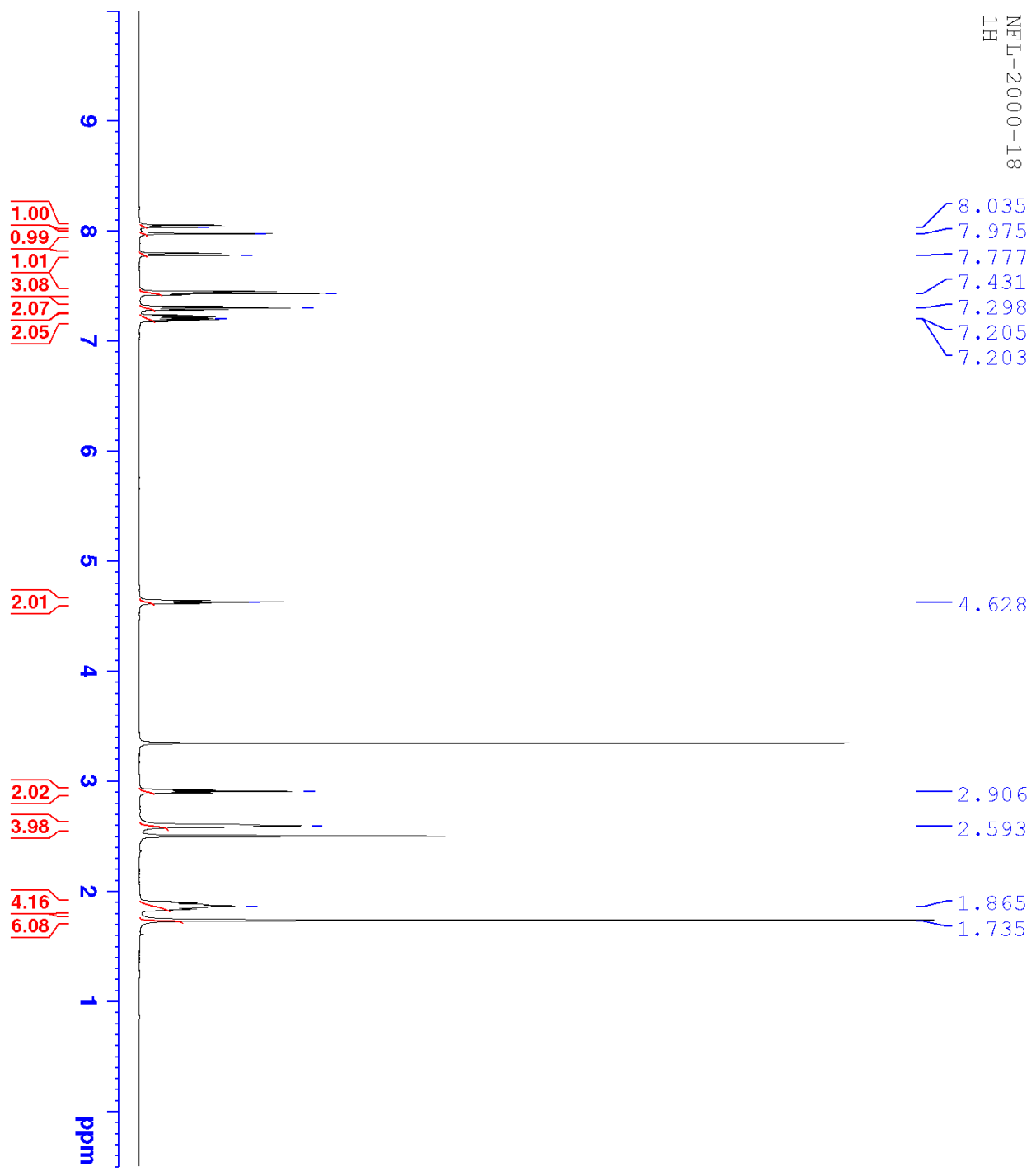
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2000-18
Received date:	November 29, 2018
Our notebook code:	NFL-2000-18
NMR sample preparation:	5.0 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC, ¹⁹ F
Proposed structure with formula, exact mass, molecular weight:	 <p>Chemical Formula: C₂₄H₂₈F₂N₄O Exact Mass: 426,2231 Molecular Weight: 426,5118</p>
Chemical name:	1-(2-(4,4-difluoropiperidin-1-yl)ethyl)-N-(2-phenylpropan-2-yl)-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 12, 2018

NFL-2000-18
1H



8.035
7.975
7.777
7.431
7.298
7.205
7.203
4.628
2.906
2.593
1.865
1.735

Current Data Parameters
NAME NFL-2000-18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20181203
Time 15.55

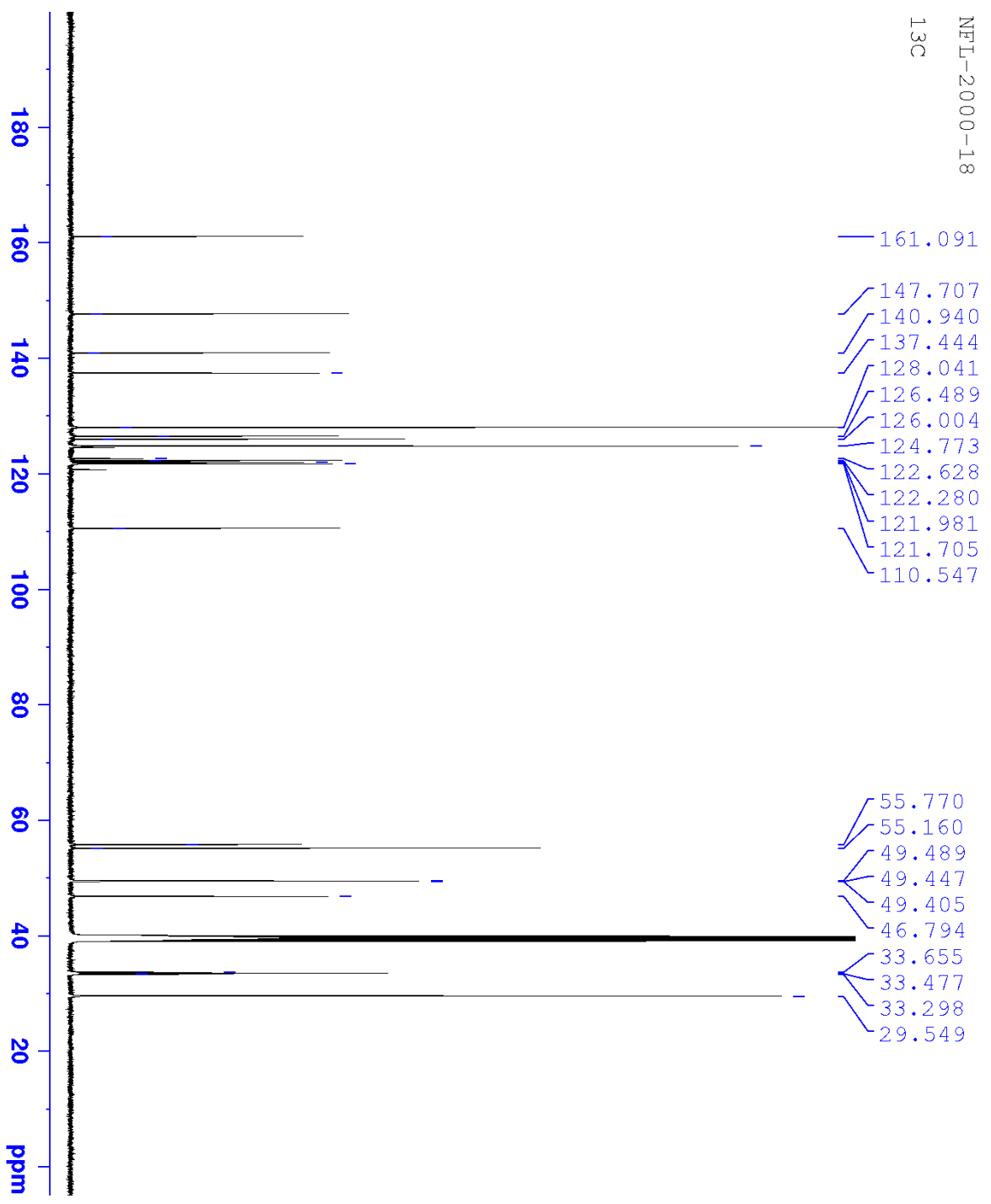
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2

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FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 71.8
DE 50.000 usec
TE 296.0 K
D1 1.00000000 sec
TDO 1

==== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 8.70 usec
PLM1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NEL-2000-18
13C



Current Data Parameters
NAME NEL-2000-18
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
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Time 20.47

INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 5120
DS 4

SWH 29761.904 Hz
FIDRES 0.454131 Hz
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RG 2050
DM 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
PL1 122.00000000 W

CHANNEL F2
SFO2 500.1320005 MHz
NUC2 1H
CDEPRG12 waltz16
PCPDZ 80.00 usec
PLW2 26.00000000 W
PLM12 0.30046001 W
PLM13 0.15113001 W

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