

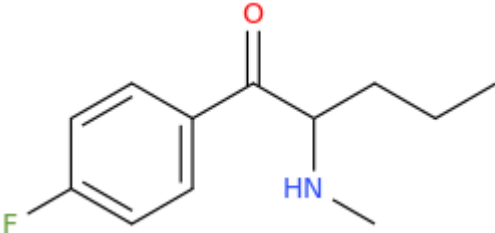
## ANALYTICAL REPORT

4-FPD (C<sub>12</sub>H<sub>16</sub>FNO)

## 1-(4-fluorophenyl)-2-(methylamino)pentan-1-one

Remark – other NPS detected: none

|   |   |
|---|---|
| Sample ID:                                      | 2091-19   |
| Sample description:                             | powder  |
| Sample type:                                    | test purchase /ISF projekt (NFL-SI)   |
| Date of entry (DD/MM/YYYY) into NFL database:   | 14/10/2019  |
| Report updates (if any) will be published here: | <a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a> |

|   |  |
|---|--|
| Substance identified - structure <sup>1</sup> (base form) |  |
| Systematic name   | 1-(4-fluorophenyl)-2-(methylamino)pentan-1-one                                     |
| Other names   | 4F-Pentedrone  |
| Formula (per base form)                                   | C <sub>12</sub> H <sub>16</sub> FNO  |
| M <sub>w</sub> (g/mol)                                    | 209,26   |
| Salt form/anions detected                                 | HCl  |
| StdInChIKey (per base form)                               | QBFXBDUCRNGHSA-UHFFFAOYSA-N  |
| Other NPS detected  | none   |
| Additional info (purity..)                                | >98% pure based on <sup>1</sup> H NMR spectrum                                     |

<sup>1</sup> 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 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm. 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. 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**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 °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<sup>-1</sup>.

**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

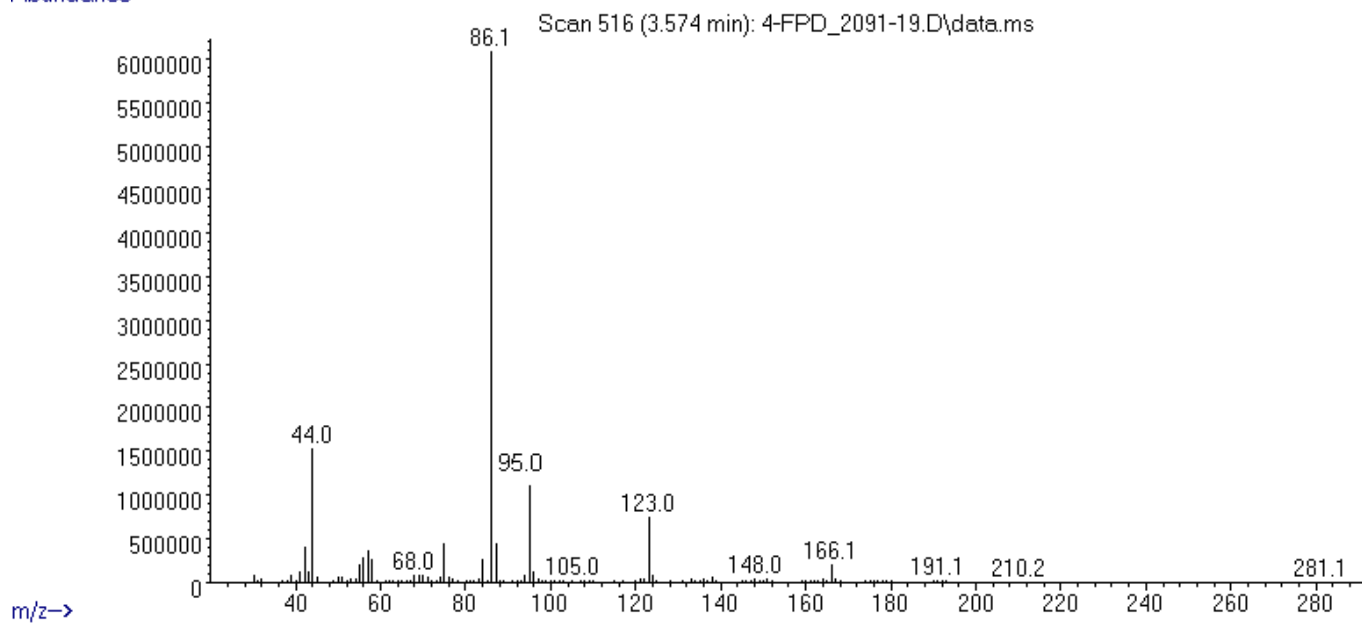
| Solubility in                   | result/remark |
|---------------------------------|---------------|
| CH <sub>2</sub> Cl <sub>2</sub> | partially     |
| MeOH                            | soluble       |
| H <sub>2</sub> O                | partially     |

| Analytical technique:                  | applied | remarks  |
|--|---------|--|
| GC-MS (EI ionization)                  | +       | NFL GC-RT (min): 3,57<br>BP(1): 86; BP(2): 44,BP(3) :95,                             |
| HPLC-TOF                               | +       | Exact mass (theoretical): 209,1216;<br>measured value Δppm:0,4;<br>formula:C12H16FNO |
| FTIR-ATR                               | +       | direct measurement (sample as received)  |
| FTIR (solid phase) always as base form | +       |  |
| IC (anions)                            | +       |  |
| NMR (in FKKT)                          | +       |  |
| validation                             |         |  |
| other                                  |         |  |

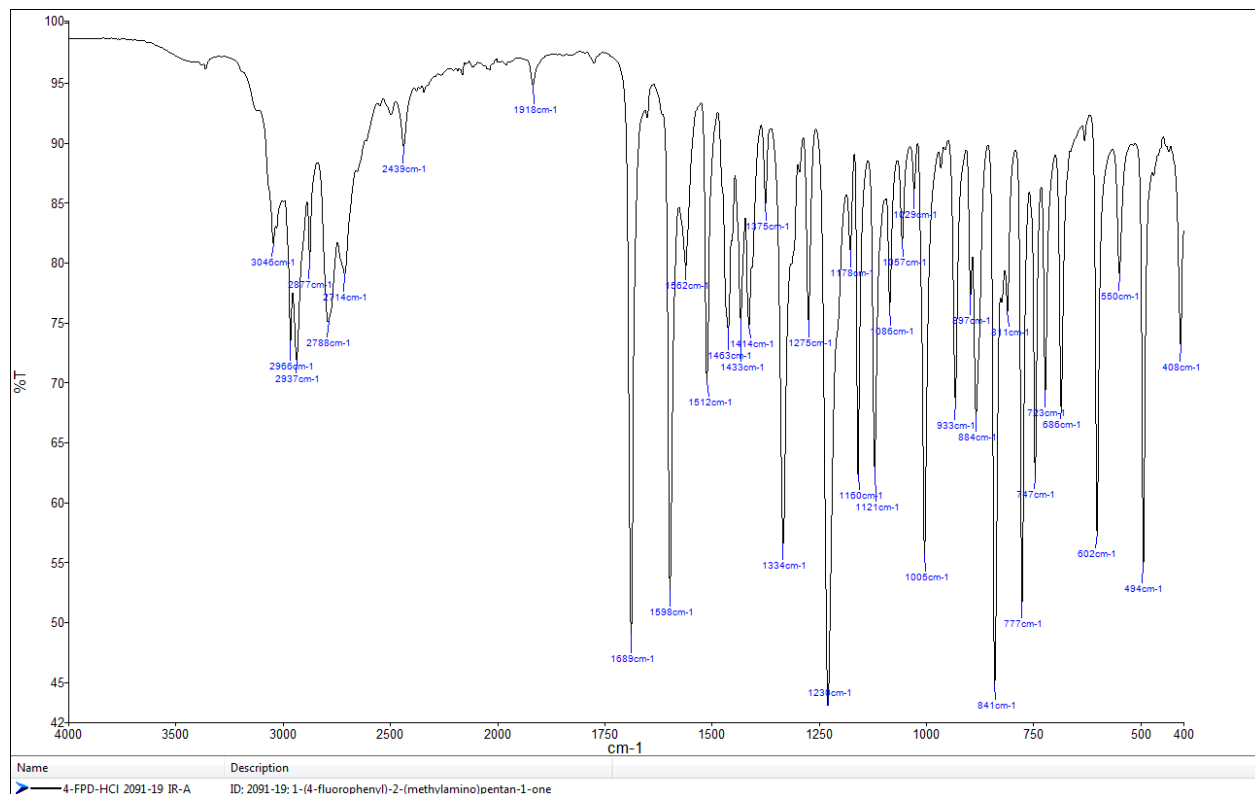
# ANALYTICAL RESULTS

MS (EI)

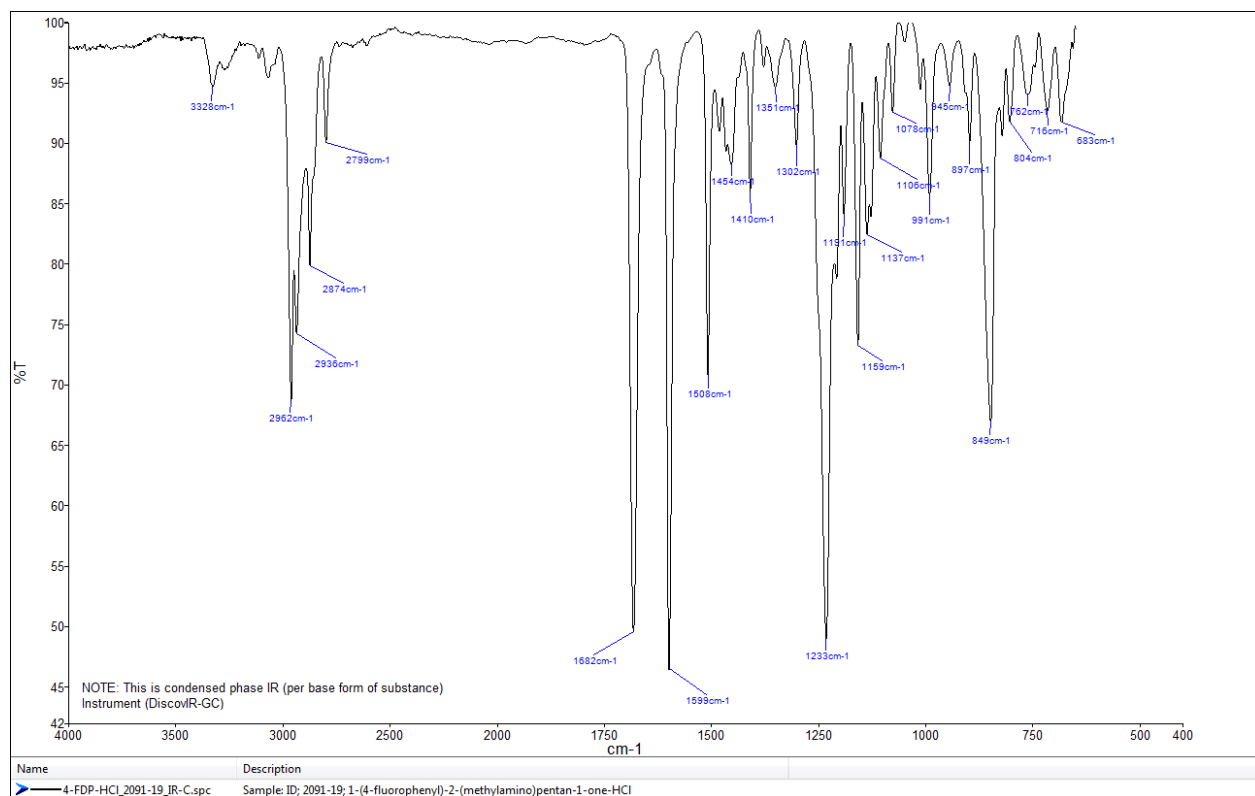
Abundance



## FTIR-ATR - direct measurement (sample as received)



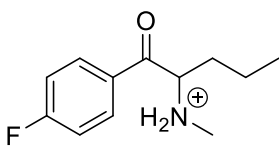
## IR (solid phase – after chromatographic separation)



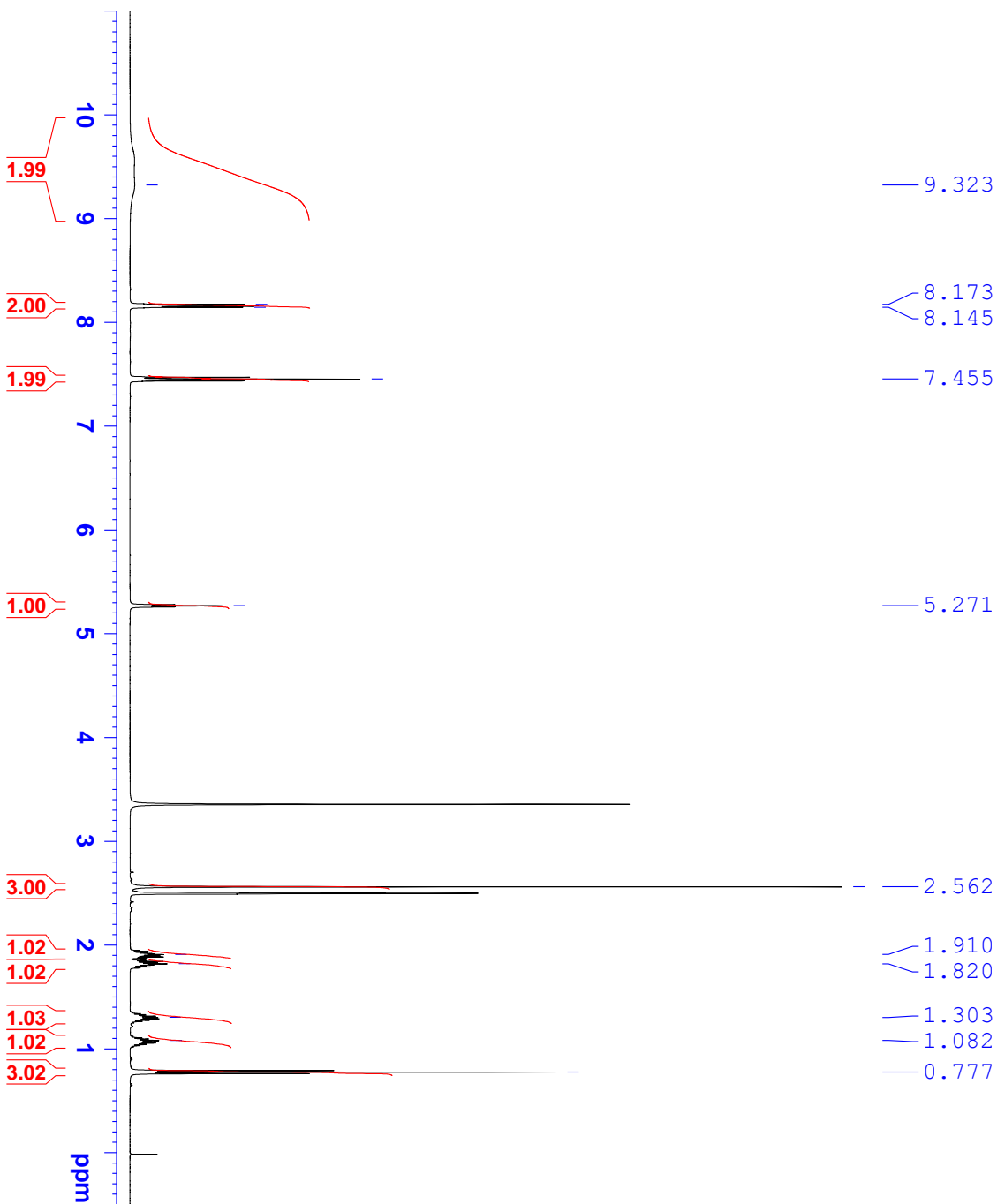
University  
of Ljubljana  
Faculty of Chemistry  
and Chemical Technology



## R E P O R T

|  |   |
|--|---|
| Contract No.   | C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)  |
| Sample ID:   | <b>2091-19</b>  |
| Received date:   | October 1, 2019   |
| Our notebook code:   | NFL-2019-19   |
| NMR sample preparation:  | 20.1 mg dissolved in 0.7 mL DMSO- <i>d</i> <sub>6</sub>   |
| NMR experiments:   | <sup>1</sup> H, <sup>13</sup> C, <sup>1</sup> H- <sup>1</sup> H <i>gs</i> -COSY, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HSQC, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HMBC, <sup>1</sup> H- <sup>15</sup> N <i>gs</i> -HMBC, <sup>19</sup> F |
| Proposed structure with formula, exact mass, molecular weight: |  <p>Chemical Formula: C<sub>12</sub>H<sub>17</sub>FNO<sup>+</sup><br/>Exact Mass: 210,1289<br/>Molecular Weight: 210,2719</p>                                    |
| Chemical name:   | <i>N</i> -protonated 1-(4-fluorophenyl)-2-(methylamino)pentan-1-one   |
| Comments:  | - Structure elucidation based on 1D and 2D NMR spectra and HRMS.<br>- >98% purity of a sample based on <sup>1</sup> H NMR spectrum.   |
| Supporting information:  | Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra, <sup>1</sup> H and <sup>13</sup> C FIDs.  |
| Principal investigator:  | Prof. Dr. Janez Košmrlj   |
| Date of report:  | October 11, 2019  |

NFL-2091-19  
1H



9.323  
8.173  
8.145  
7.455  
5.271  
2.562  
1.910  
1.820  
1.303  
1.082  
0.777

Current Data Parameters  
NAME NFL-2091-19  
EXPNO 1  
PROCNO 1

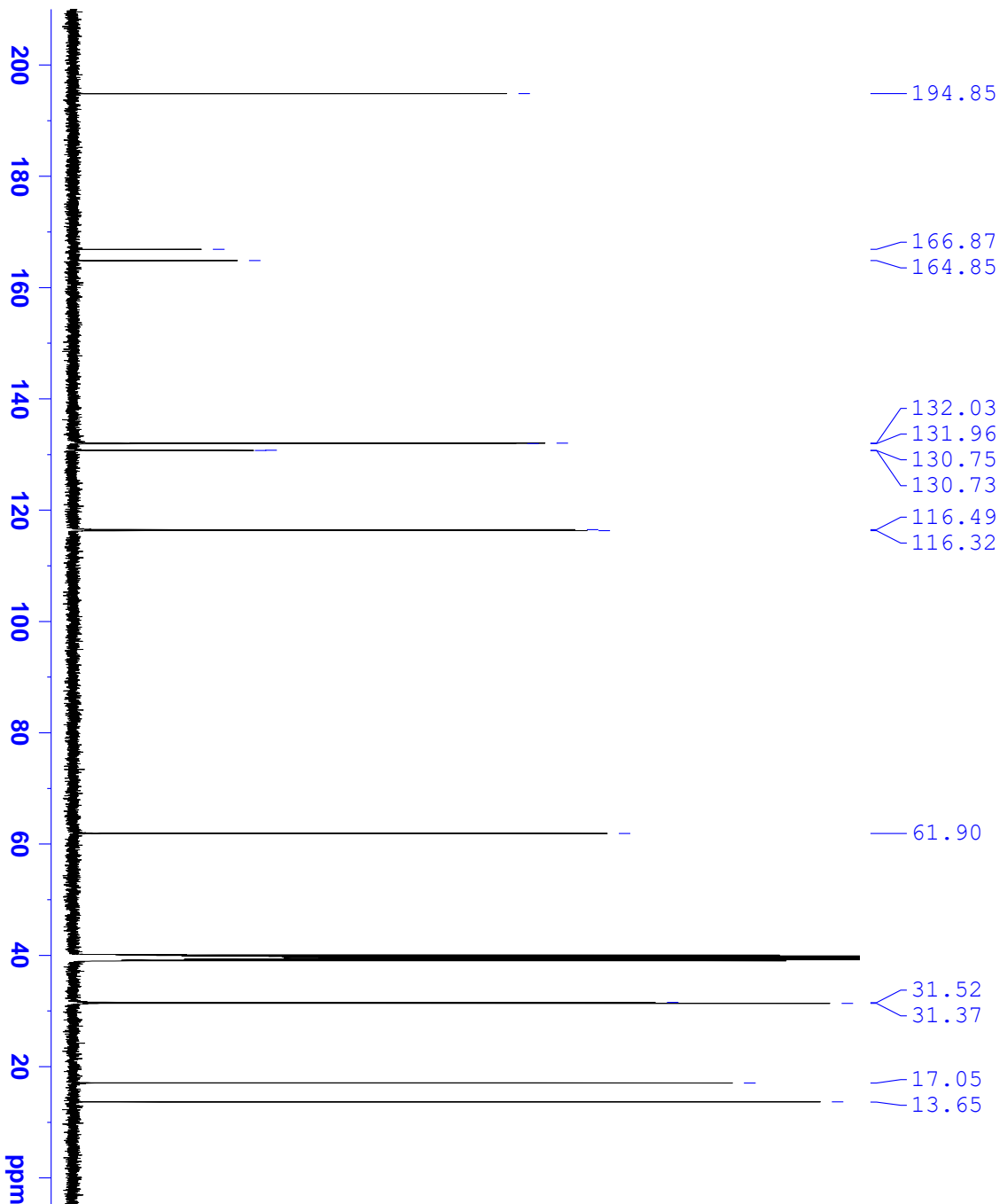
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Time\_ 16.31  
INSTRUM spect  
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PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 16  
DS 2

SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 80.6  
DM 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 500.1300044 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

NFL-2091-19  
13C



Current Data Parameters  
 NAME NFL-2091-19  
 EXPNO 10  
 PROCNO 1

F2 - Acquisition Parameters  
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 Time\_ 8.48  
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 PULPROG zgpg30  
 TD 65536  
 SOLVENT DMSO  
 NS 1024  
 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 =====  
 SF01 125.7703637 MHz  
 NUC1 13C  
 P1 8.70 usec  
 PLW1 122.00000000 W

==== CHANNEL f2 =====  
 SF02 500.1320005 MHz  
 NUC2 1H  
 CPDPRG12 waltz16  
 PCPD2 80.00 usec  
 PLW2 26.00000000 W  
 PLM12 0.30046001 W  
 PLM13 0.15113001 W

F2 - Processing parameters  
 SI 32768  
 SF 125.7578455 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40