ANALYTICAL REPORT

4-FPD (C12H16FNO)

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

Remark – other NPS detected: none

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

<table>
<thead>
<tr>
<th>Substance identified - structure¹ (base form)</th>
</tr>
</thead>
<tbody>
<tr>
<td>![Structure Diagram]</td>
</tr>
</tbody>
</table>

Systematic name 1-(4-fluorophenyl)-2-(methylamino)pentan-1-one

Other names 4F-Pentedrone

Formula (per base form) C12H16FNO

Mₗ (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 1H NMR spectrum

¹ Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ DOI: 10.1021/ci100384d
Report updates

<table>
<thead>
<tr>
<th>date</th>
<th>comments (explanation)</th>
</tr>
</thead>
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</tbody>
</table>

**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 quadrupole 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 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))
   MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadrupole temperatures 280°C and 180°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

<table>
<thead>
<tr>
<th>Substance</th>
<th>Result/Remark</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₂Cl₂</td>
<td>partially</td>
</tr>
<tr>
<td>MeOH</td>
<td>soluble</td>
</tr>
<tr>
<td>H₂O</td>
<td>partially</td>
</tr>
</tbody>
</table>

### Analytical technique:

<table>
<thead>
<tr>
<th>Technique</th>
<th>Applied</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC-MS (EI ionization)</td>
<td>+</td>
<td>NFL GC-RT (min): 3,57 BP(1): 86; BP(2): 44; BP(3): 95,</td>
</tr>
<tr>
<td>HPLC-TOF</td>
<td>+</td>
<td>Exact mass (theoretical): 209,1216; measured value Δppm: 0.4;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>formula: C₁₂H₁₆FNO</td>
</tr>
<tr>
<td>FTIR-ATR</td>
<td>+</td>
<td>direct measurement (sample as received)</td>
</tr>
<tr>
<td>FTIR (solid phase) always</td>
<td>+</td>
<td>as base form</td>
</tr>
<tr>
<td>as base form</td>
<td></td>
<td></td>
</tr>
<tr>
<td>IC (anions)</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>NMR (in FKKT)</td>
<td>+</td>
<td></td>
</tr>
<tr>
<td>validation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>other</td>
<td></td>
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</table>
ANALYTICAL RESULTS

MS (EI)

Abundance

Scan 516 (3.574 min): 4-FPD_2091-19\data.ms

m/z ->
FTIR-ATR - direct measurement (sample as received)

IR (solid phase – after chromatographic separation)

NOTE: This is condensed phase IR (per base form of substance) Instrument (Discover-GC)
**R E P O R T**

<table>
<thead>
<tr>
<th>Contract No.</th>
<th>C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)</th>
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<tbody>
<tr>
<td>Sample ID:</td>
<td><strong>2091-19</strong></td>
</tr>
<tr>
<td>Received date:</td>
<td>October 1, 2019</td>
</tr>
<tr>
<td>Our notebook code:</td>
<td>NFL-2019-19</td>
</tr>
<tr>
<td>NMR sample preparation:</td>
<td>20.1 mg dissolved in 0.7 mL DMSO-$d_6$</td>
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<tr>
<td>NMR experiments:</td>
<td>$^1$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</td>
</tr>
</tbody>
</table>

**Proposed structure with formula, exact mass, molecular weight:**

![Proposed structure](image)

**Chemical Formula:** $C_{12}H_{17}FNO^+$  
**Exact Mass:** 210,1289  
**Molecular Weight:** 210,2719

<table>
<thead>
<tr>
<th>Chemical name:</th>
<th>$N$-protonated 1-(4-fluorophenyl)-2-(methylamino)pentan-1-one</th>
</tr>
</thead>
</table>
| 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: | October 11, 2019                                             |
Current Data Parameters
NAME        NFL-2091-19
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20191002
Time              16.31
INSTRUM           spect
PROBHD   5 mm PABBO BB-
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
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          500.1300044 MHz
WDW                  EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00

1H
Current Data Parameters
NAME        NFL-2091-19
EXPNO                10
PROCNO                1
F2 - Acquisition Parameters
Date_          20191003
Time               8.48
INSTRUM           spect
PROBHD   5 mm PABBO BB-
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 ========
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          125.7578455 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
NFL-2091-19
13C