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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

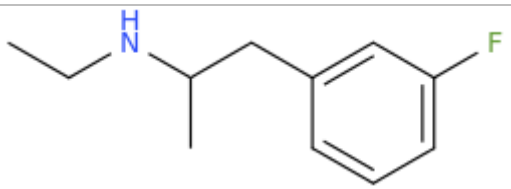
# ANALYTICAL REPORT

## 3-FEA (C11H16FN)

**ethyl[1-(3-fluorophenyl)propan-2-yl]amine**

Remark – other NPS detected: none

| | |
|---|---|
| Sample ID: | 2007-18 |
| Sample description: | powder |
| Sample type: | test purchase /NFL- purchasing |
| Date of sample receipt (DD/MM/YYYY): | 25/10/2018 |
| Date of entry (DD/MM/YYYY) into NFL database: | 22/01/2019 |
| 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 | ethyl[1-(3-fluorophenyl)propan-2-yl]amine |
| Other names | N-ethyl-1-(3-fluorophenyl)propan-2-amine; 3-fluoroethamphetamine; m-fluoro N-ethylamphetamine |
| Formula (per base form) | C11H16FN |
| $M_w$ (g/mol) | 181.25 |
| Salt form/anions detected | HCl |
| StdInChIKey (per base form) | CKPWHLGHHXSVJI-UHFFFAOYSA-N |
| Other NPS detected | none |
| Additional info (purity..) | >95% purity by 1H 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

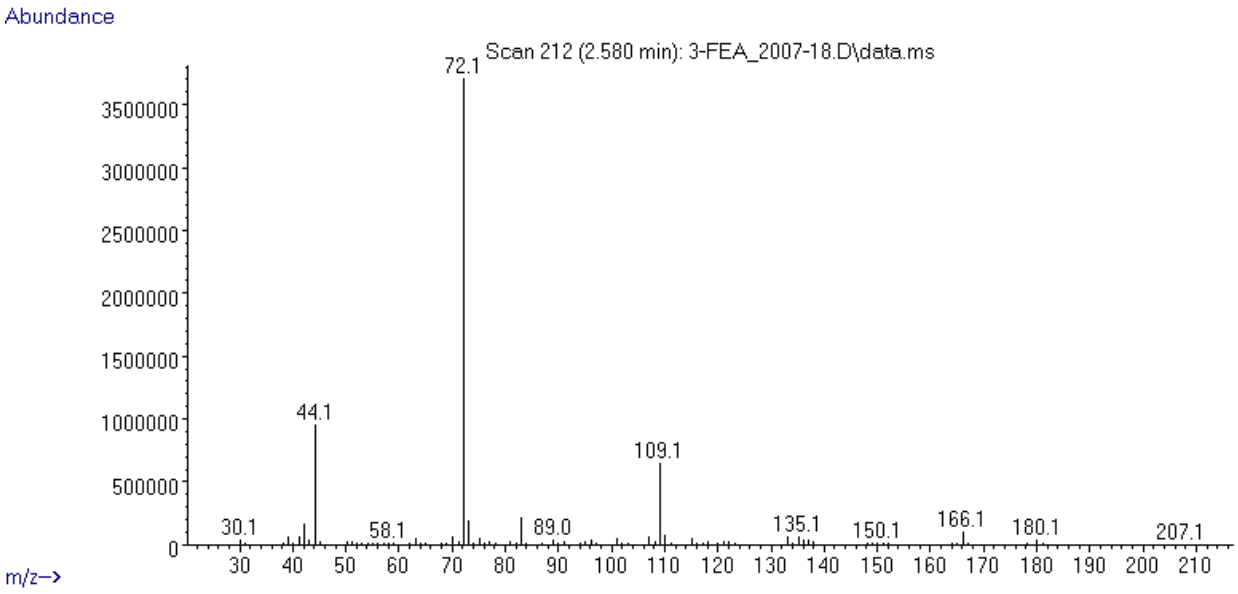
Supporting information

| Solubility in | result/remark |
|---|---|
| $CH_2Cl_2$ | soluble |
| MeOH | soluble |
| $H_2O$ | soluble |

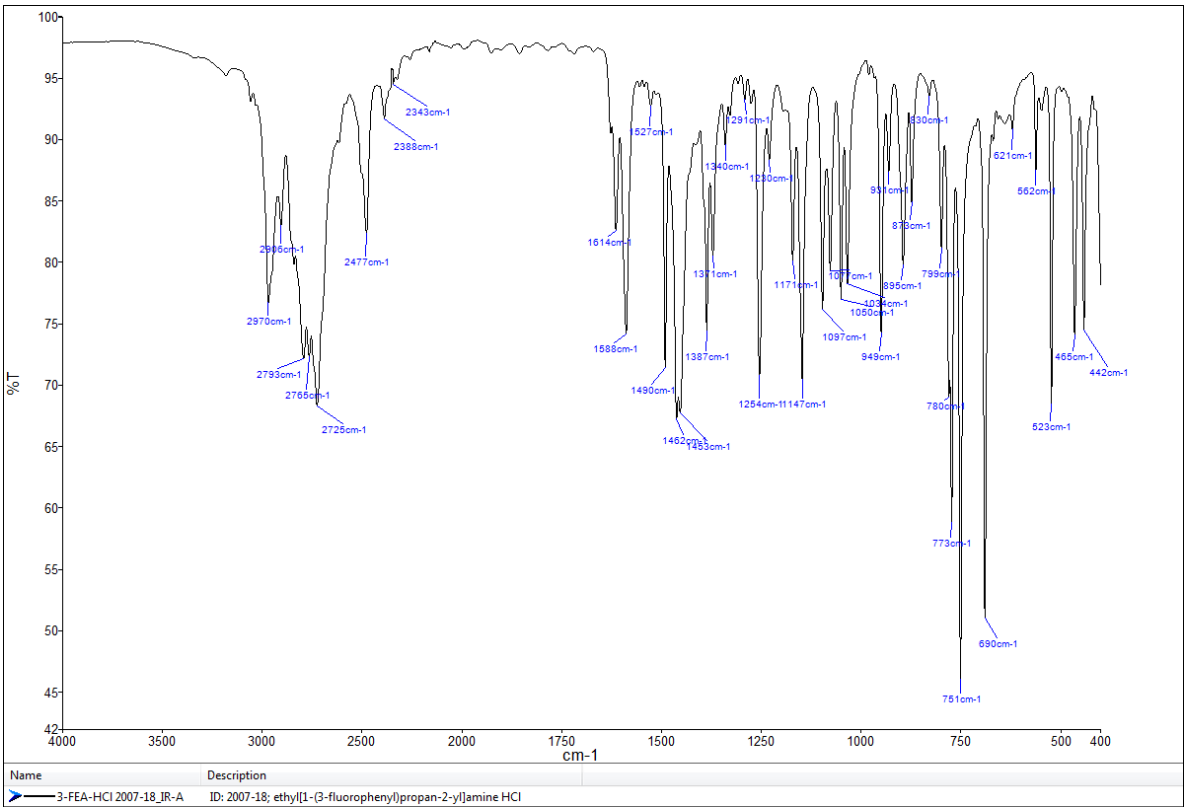
| Analytical technique: | applied | remarks |
|---|---|---|
| GC-MS (EI ionization) | + | NFL GC-RT (min): 2.58<br>BP(1): 72; BP(2): 44,BP(3) :109, |
| HPLC-TOF | + | Exact mass (theoretical): 184.1267;<br>measured value Δppm:-3.42;<br>formula:C11H16FN |
| FTIR-ATR | + | direct measurement (sample as received) |
| FTIR (solid phase) always as base form | + | |
| IC (anions) | + | |
| NMR (in FKKT) | + | |
| validation | | |
| other | | |

## ANALYTICAL RESULTS

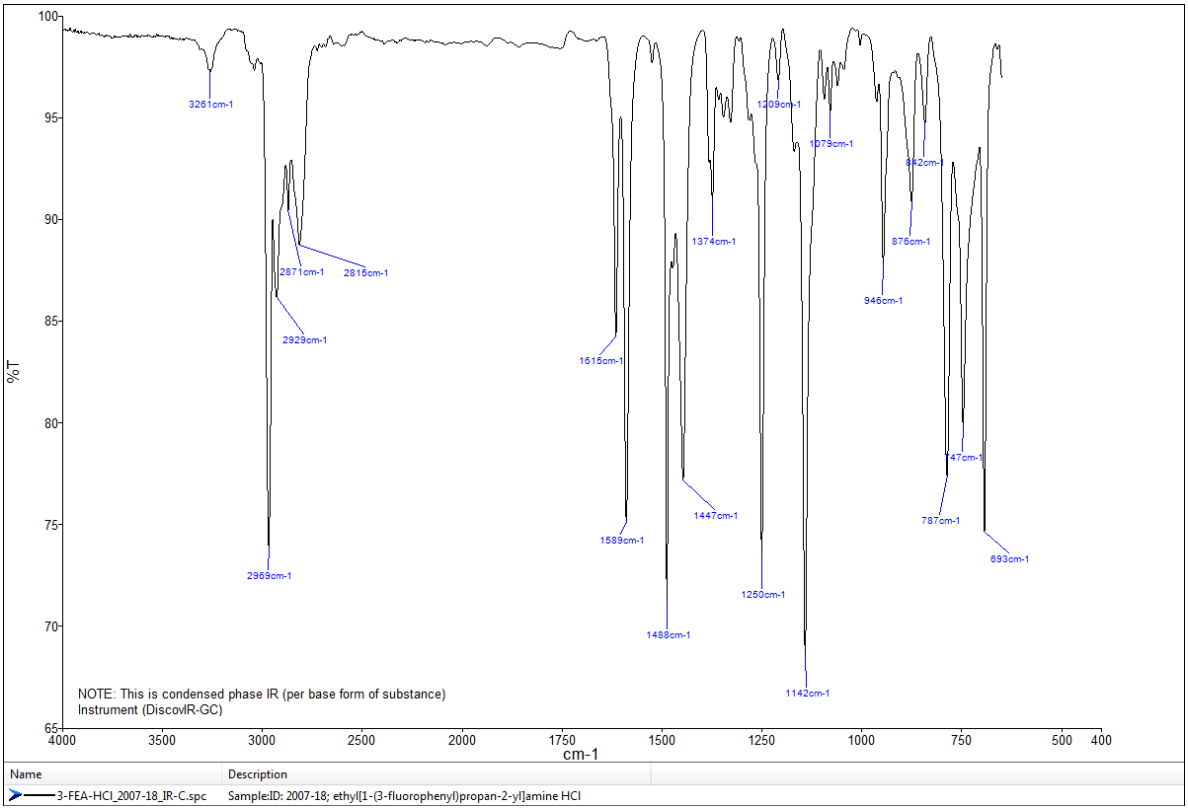
MS (EI)



FTIR-ATR - direct measurement (sample as received)




IR (solid phase – after chromatographic separation)



# TOF REPORT

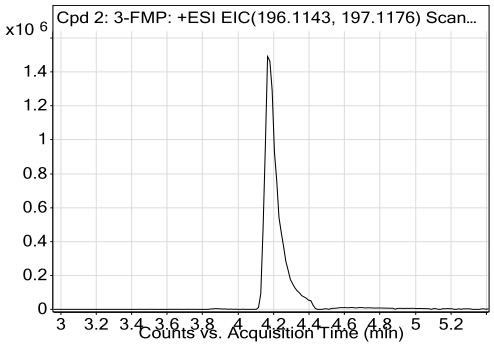
| | | | |
|---|---|---|---|
| **Data File** | 3-FEA_2007-18.d | **Sample Name** | ID 2007-18 |
| **Sample Type** | Sample | **Position** | P2-C6 |
| **Instrument Name** | 6230B TOF LC-MS | **User Name** | TG |
| **Acq Method** | general-19_10_2018-XDB-C18-ESI+.m | **Acquired Time** | 11/1/2018 3:30:35 PM |
| **IRM Calibration Status** | Success | **DA Method** | a-Drugs_NFL.m |
| **Comment** | MeOH | | |

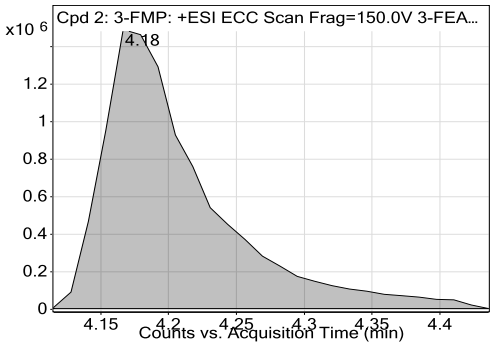
**Compound Table**

| Label | Compound Name | MFG Formula | Obs. RT | Obs. Mass |
|---|---|---|---|---|
| Cpd 2: 3-FMP | 3-FMP | C11 H14 F N O | 4.18 | 195.1065 |

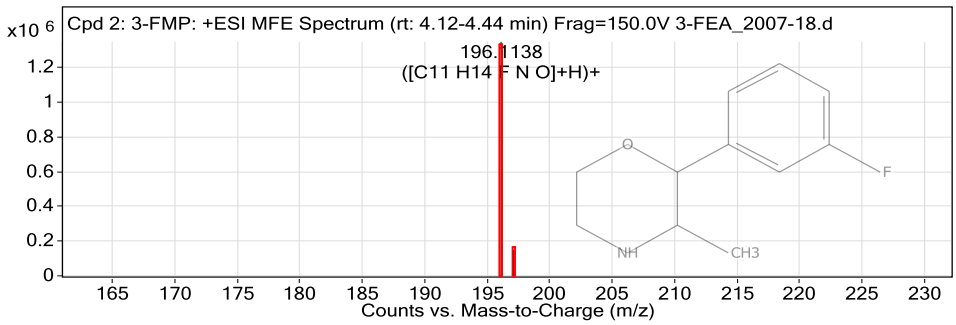
| Name | *Obs. m/z* | Obs. RT | Obs. Mass | DB RT | DB Formula | DB Mass | DB Mass Error (ppm) |
|---|---|---|---|---|---|---|---|
| **3-FMP** | 196.1138 | 4.18 | 195.1065 | 4.21 | C11 H14 F N O | 195.1059 | -2.91 |

**Compound Chromatograms**

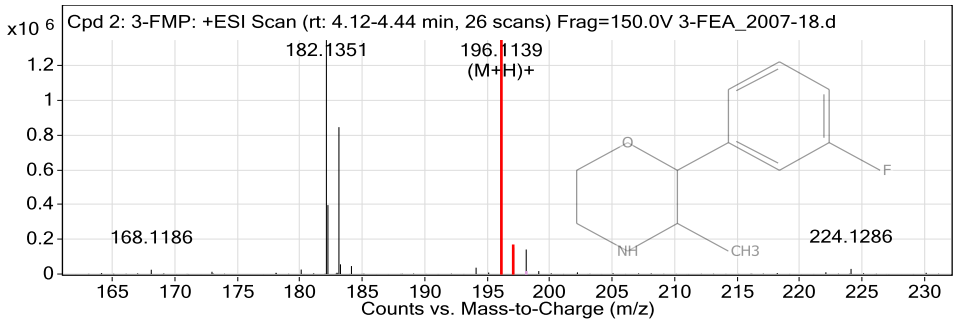



MFE MS Zoomed Spectrum




MS Zoomed Spectrum




**MS Spectrum Peak List**

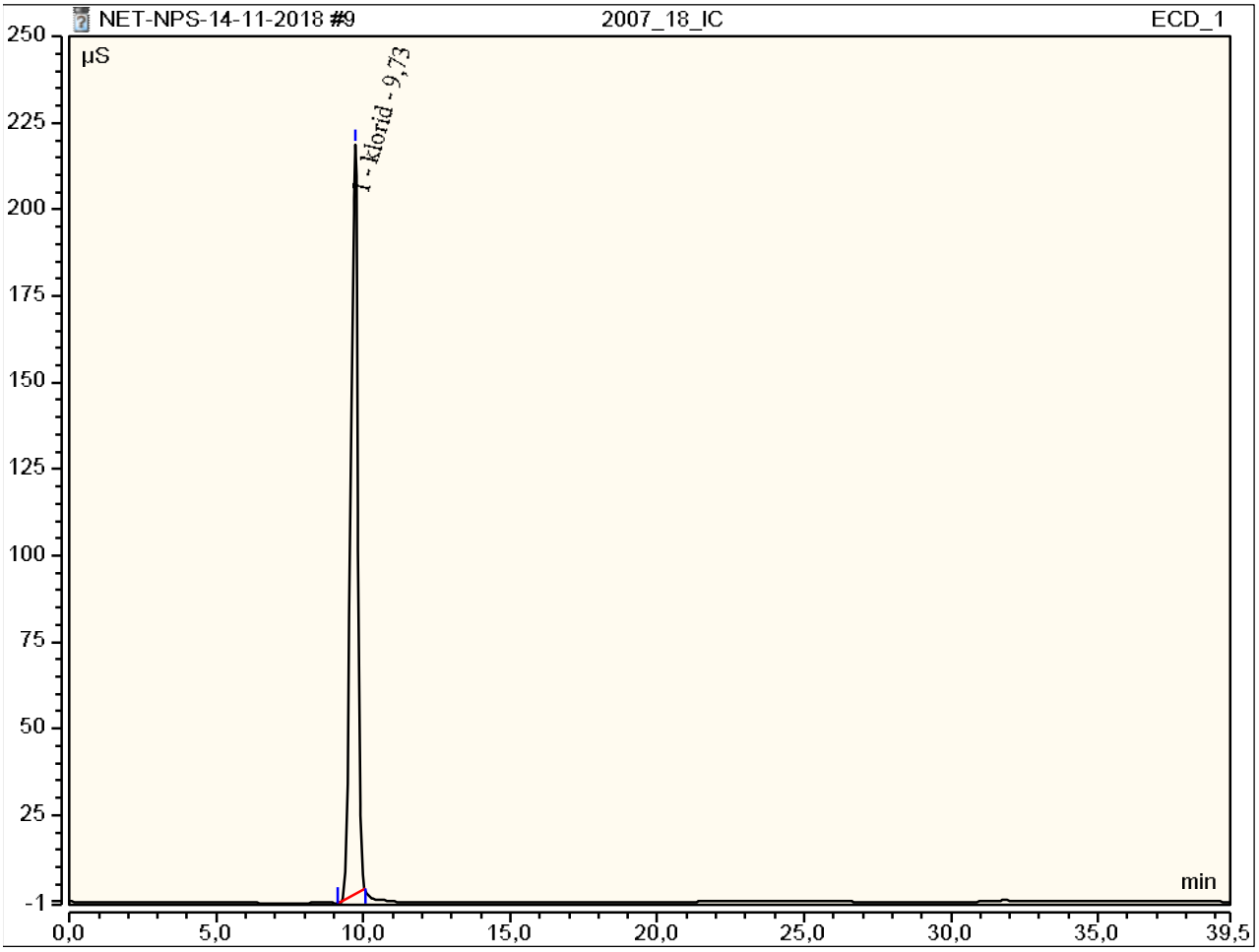
| *Obs. m/z* | Charge | Abund | Formula | Ion/Isotope |
|---|---|---|---|---|
| 196.1138 | 1 | 1348574.88 | C11 H14 F N O | (M+H)+ |
| 197.117 | 1 | 149895.66 | C11 H14 F N O | (M+H)+ |

--- End Of Report ---

# Peak Integration Report

| | | | |
|---|---|---|---|
| *Sample Name:* | **2007_18_IC** | *Inj. Vol.:* | **25,00** |
| *Injection Type:* | **Unknown** | *Dilution Factor:* | **1,0000** |
| *Program:* | **ANIONI** | *Operator:* | **kemija** |
| *Inj. Date / Time:* | **15-nov-2018 / 16:56** | *Run Time:* | **42,00** |

| No. | Time min | Peak Name | Peak Type | Area µS*min | Height µS | Amount mg/L |
|---|---|---|---|---|---|---|
| 1,00 | 9,73 | **klorid** | BMB | 58,94 | 216,53 | n.a. |
| | | **TOTAL:** | | 58,94 | 216,53 | 0,00 |



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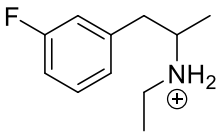
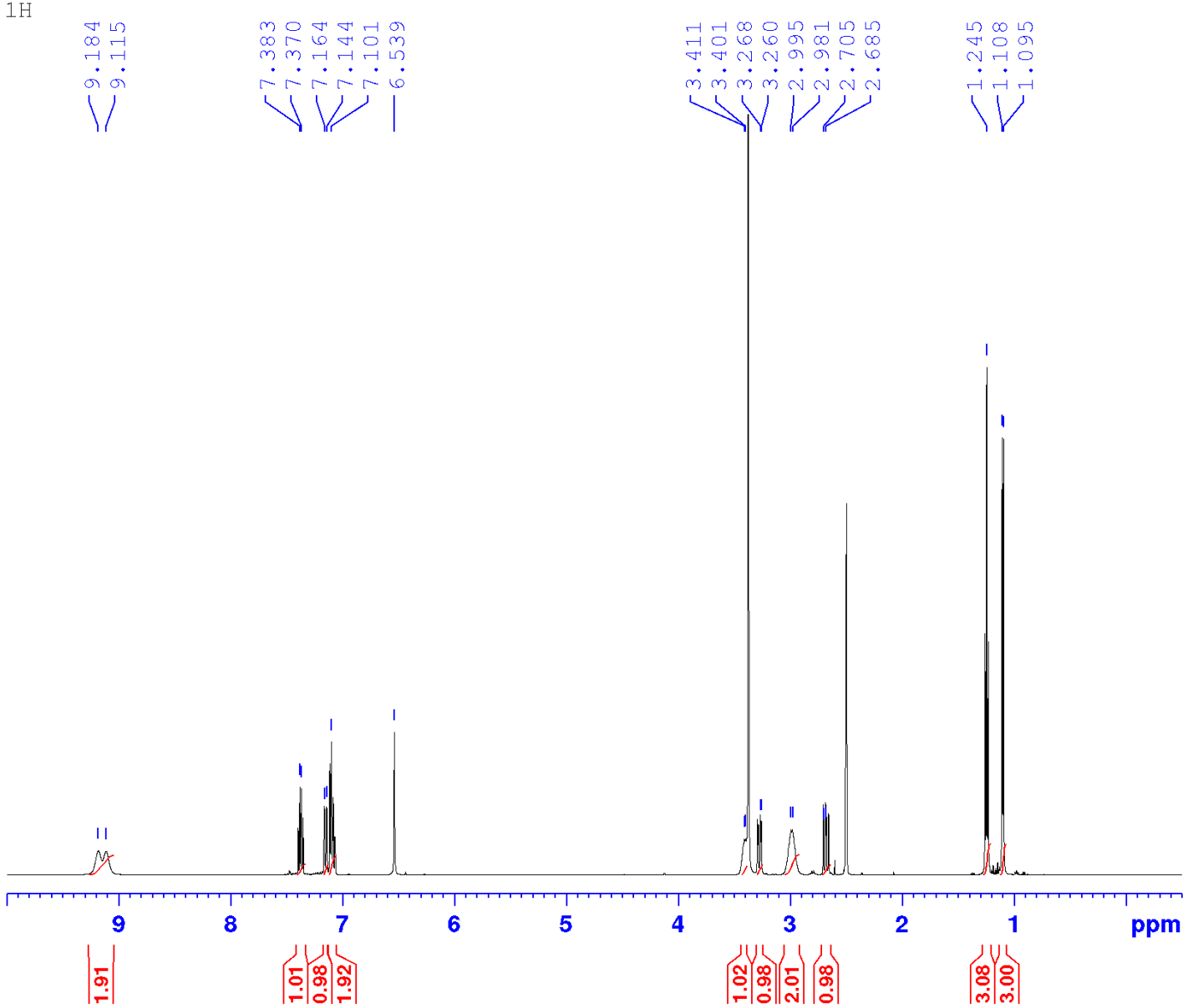
University
*of Ljubljana*

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: | **2007-18** |
| Received date: | November 29, 2018 |
| Our notebook code: | NFL-2007-18 |
| NMR sample preparation: | 17.7 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: $C_{11}H_{17}FN^+$<br>Exact Mass: 182,1340<br>Molecular Weight: 182,2619 |
| Chemical name: | *N*-protonated *N*-ethyl-1-(3-fluorophenyl)propan-2-amine |
| Comments: | - Structure elucidation based on 1D and 2D NMR spectra and HRMS.<br>- >95% 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 18, 2018 |

NFL-2007-18
1H



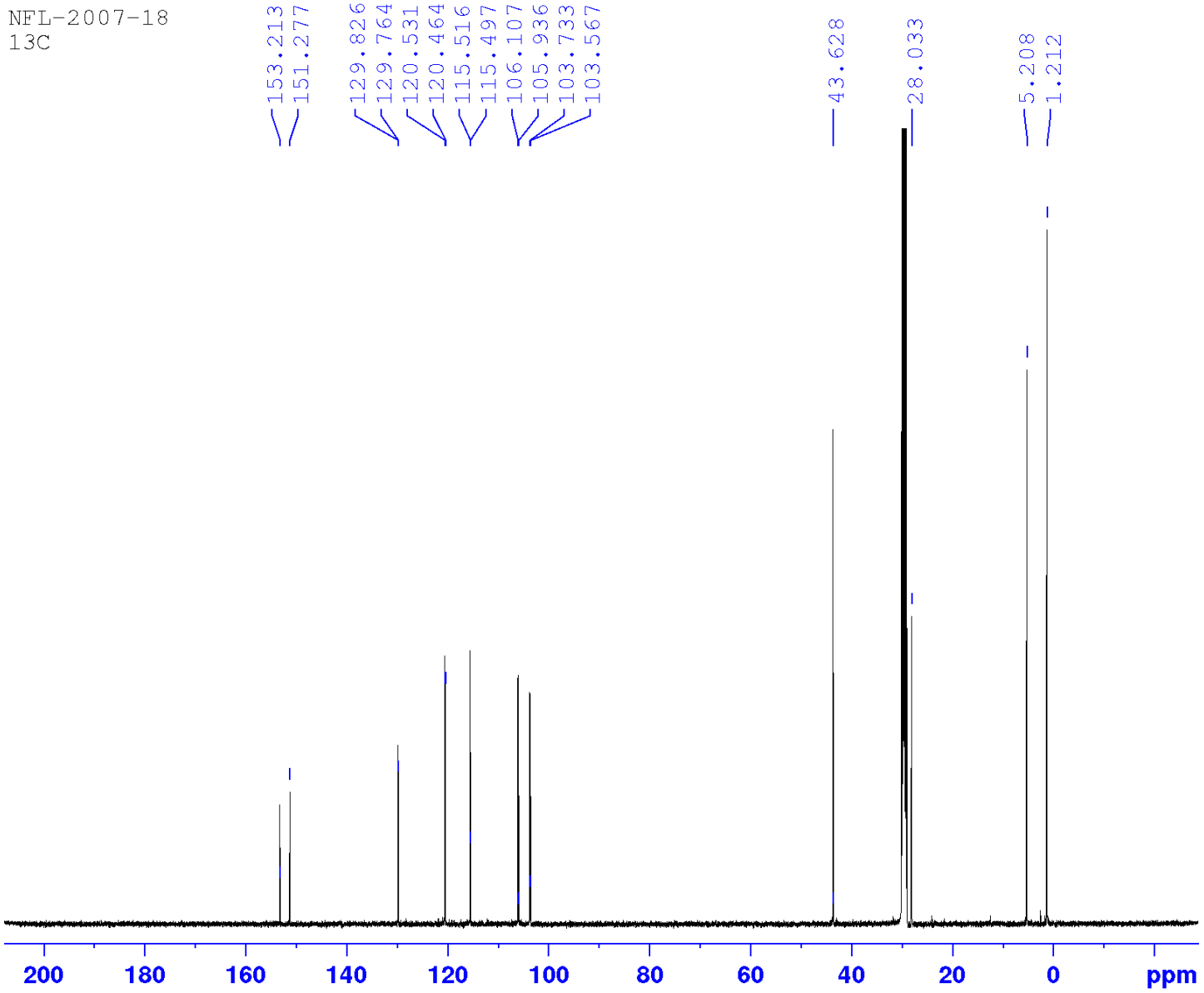

```
Current Data Parameters
NAME         NFL-2007-18
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181214
Time               17.41
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                 71.8
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           500.1300045 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

NFL-2007-18
13C




```
Current Data Parameters
NAME         NFL-2007-18
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181214
Time               23.50
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           125.7591018 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```