NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY
Vodovodna 95, 1000 LJUBLJANA, SLOVENIA
E: nfl@policija.si

EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

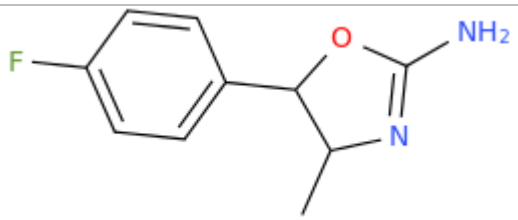
ANALYTICAL REPORT

pF-4-methylaminorex (C10H11FN2O)

5-(4-fluorophenyl)-4-methyl-4,5-dihydro-1,3-oxazol-2-amine

Remark – other NPS detected: none

Sample ID:	1940-18
Sample description:	powder
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (DD/MM/YYYY):	24/04/2018
Date of entry (DD/MM/YYYY) into NFL database:	18/07/2018
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	5-(4-fluorophenyl)-4-methyl-4,5-dihydro-1,3-oxazol-2-amine
Other names	4'-Fluoro-4-methylaminorex; para-fluoro-4-methylaminorex; p-F-4-methylaminorex
Formula (per base form)	C10H11FN2O
M_w (g/mol)	194,21
Salt form/anions detected	base
StdInChIKey (per base form)	UYKYWISHPDEDRQ-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	two isomers

[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 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

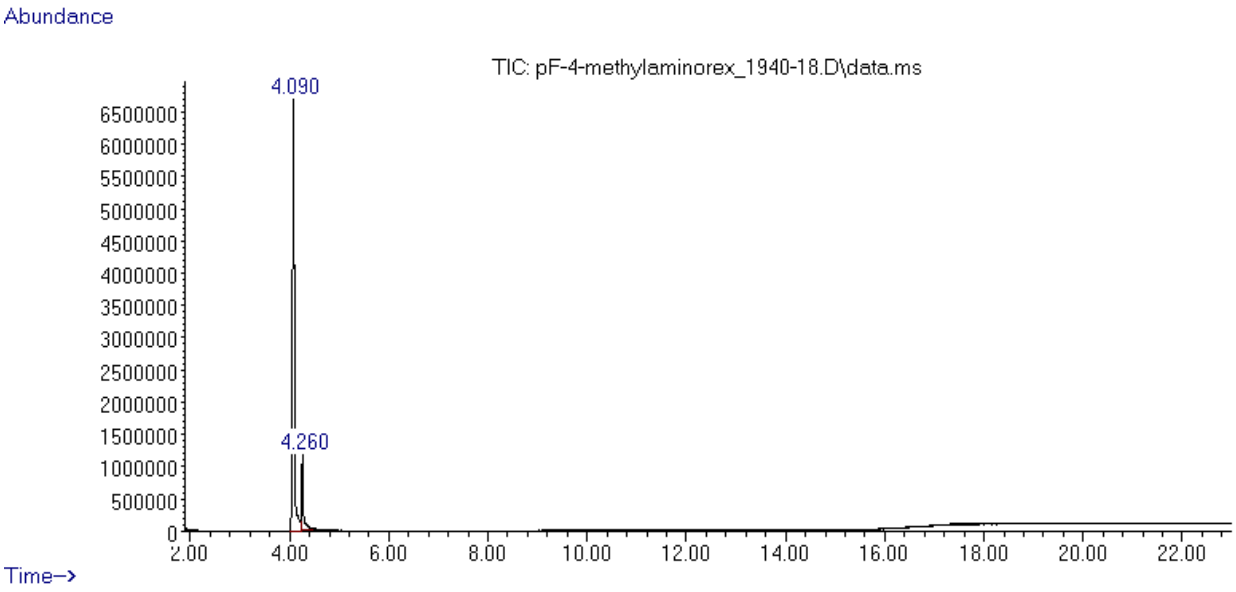
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	partially

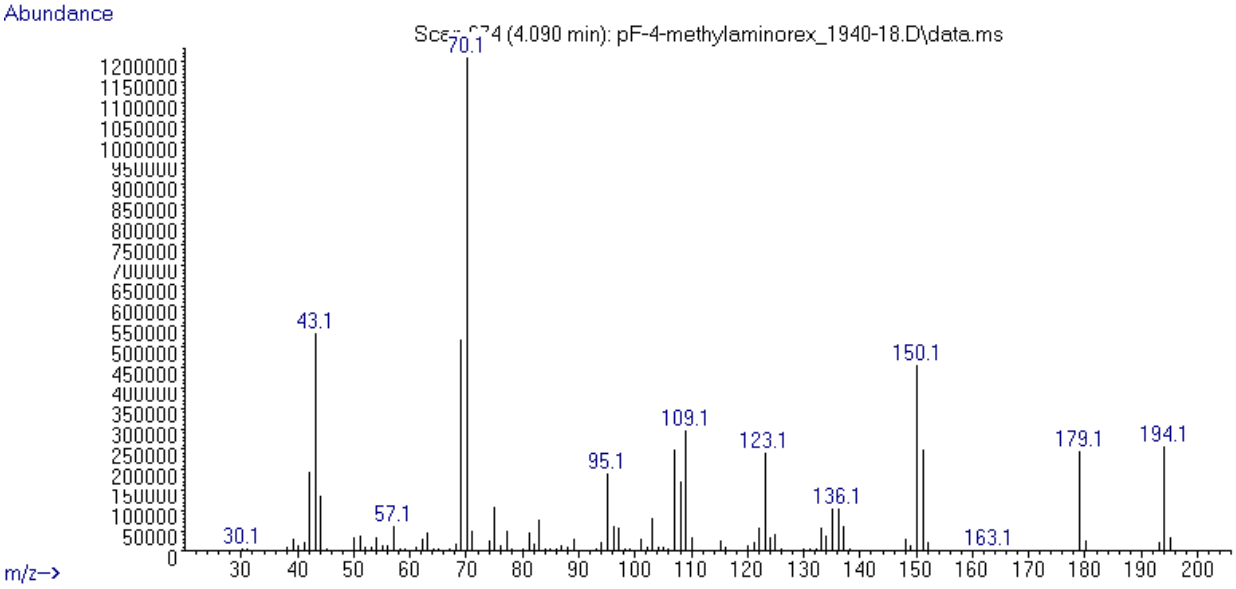
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,09 BP(1): 70; BP(2): 43,BP(3) :69,
HPLC-TOF	+	Exact mass (theoretical): 194,0855; measured value Δppm:-1,3; formula:C10H11FN2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	-	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

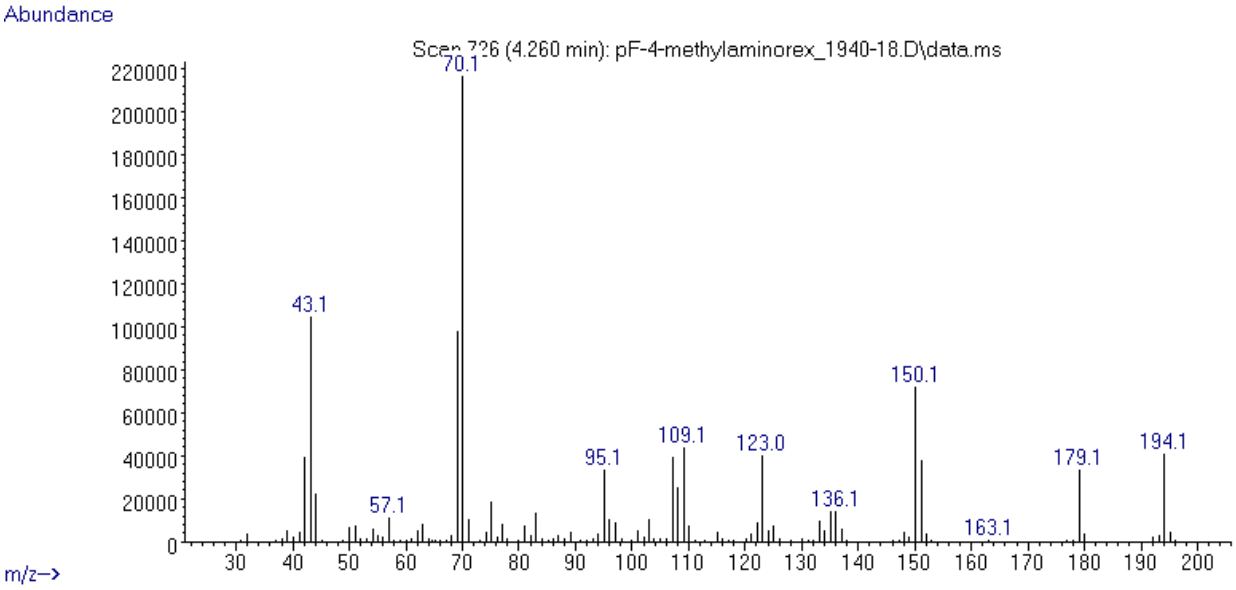
GC



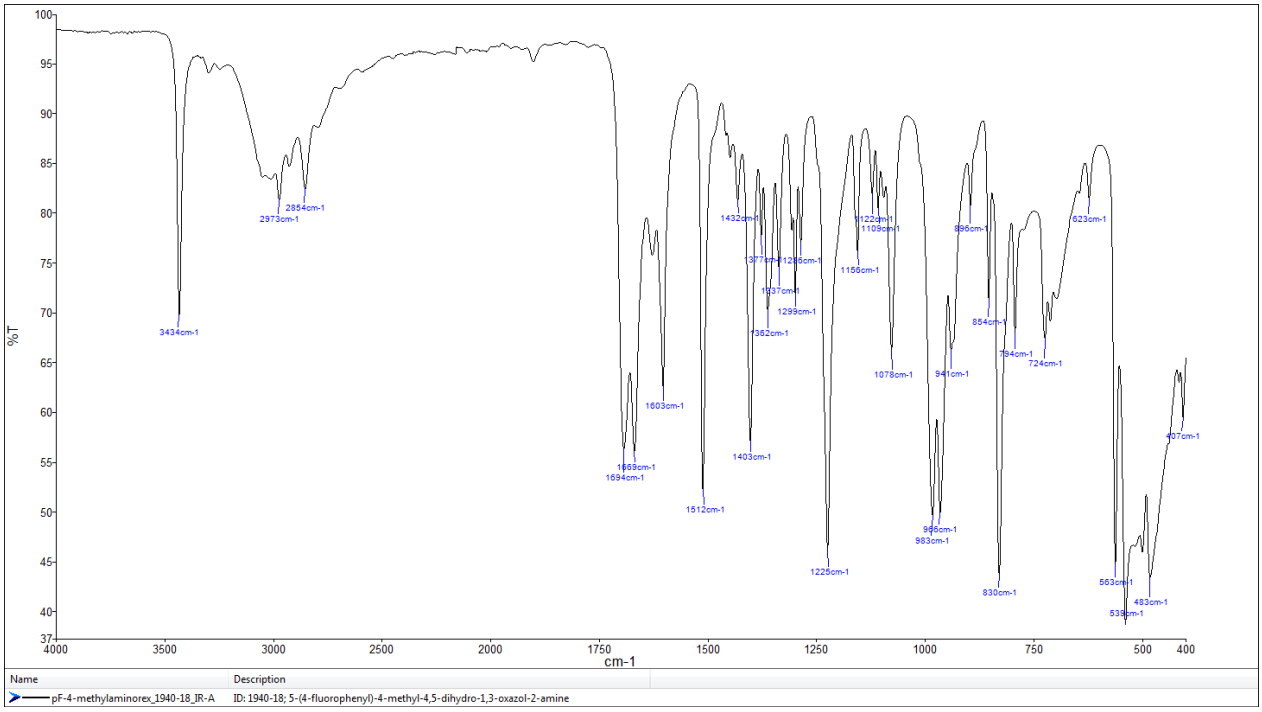
MS (EI): RT 4.09min – isomer1



MS (EI): RT 4.26min – isomer2



FTIR-ATR - direct measurement (sample as received – mix of isomers)



TOF REPORT

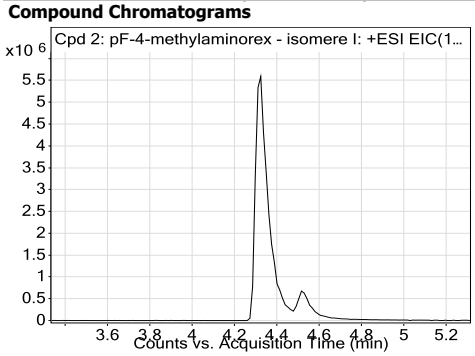
Data File	pF-4-methylaminorex.d	**Sample Name**	ID 1940-18
Sample Type	Sample	**Position**	P1-C3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-04_12_2017-XDB-C18-ESI+.m	**Acquired Time**	4/30/2018 12:10:24 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

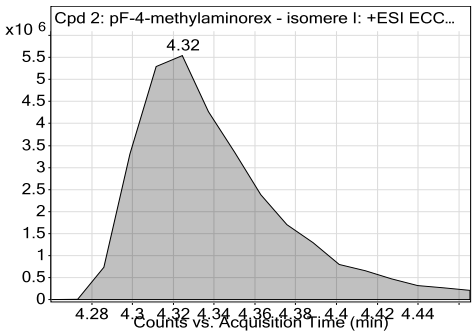
Compound Table

Label	Compound Name	Obs. RT	Obs. Mass
Cpd 2: pF-4-methylaminorex - isomere I	pF-4-methylaminorex - isomere I	4.32	194.0858
Cpd 4: pF-4-methylaminorex - isomere II	pF-4-methylaminorex - isomere II	4.53	194.0858

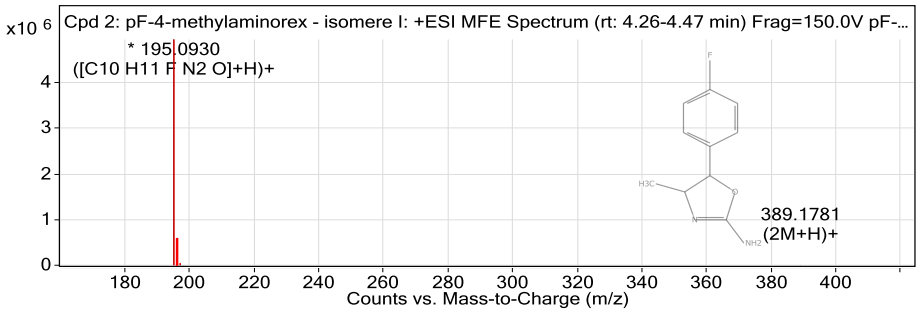
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
pF-4-methylaminorex - isomere I	195.093	4.32	194.0858	4.32	C10 H11 F N2 O	194.0855	-1.3

Compound Chromatograms

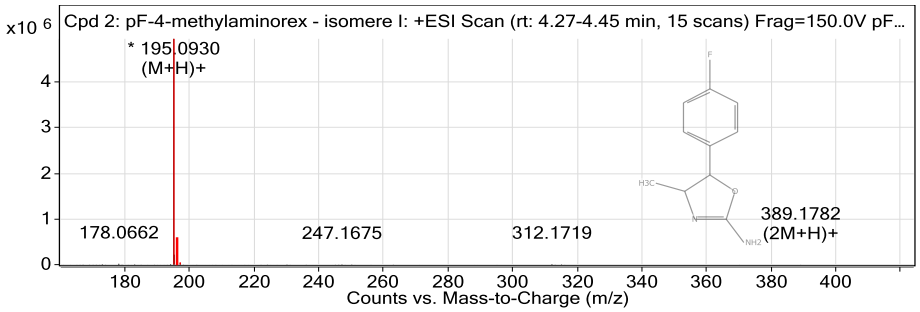




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



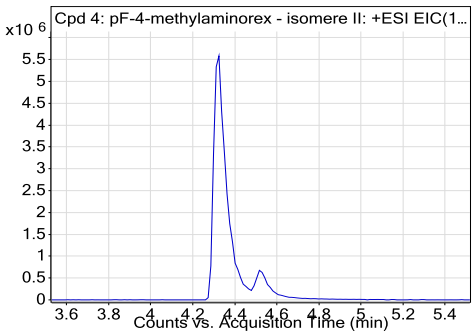
MS Spectrum Peak List

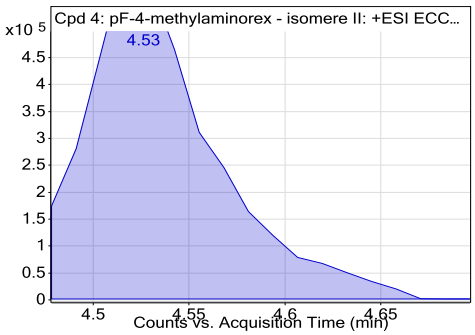
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
195.093	1	4948161	C10 H11 F N2 O	(M+H)+
196.0964	1	537908.75	C10 H11 F N2 O	(M+H)+
197.0986	1	38726.47	C10 H11 F N2 O	(M+H)+
198.1058	1	1932.6	C10 H11 F N2 O	(M+H)+
389.1781	1	2481.61		(2M+H)+

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
pF-4-methylaminorex - isomere II	195.0931	4.53	194.0858	4.53	C10 H11 F N2 O	194.0855	-1.26

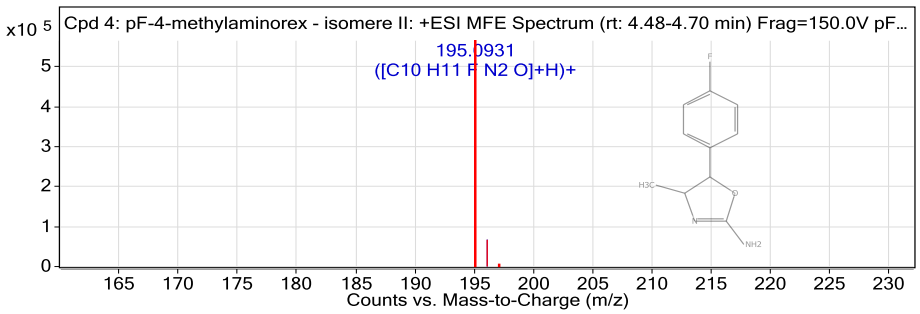
Compound Chromatograms

TOF REPORT

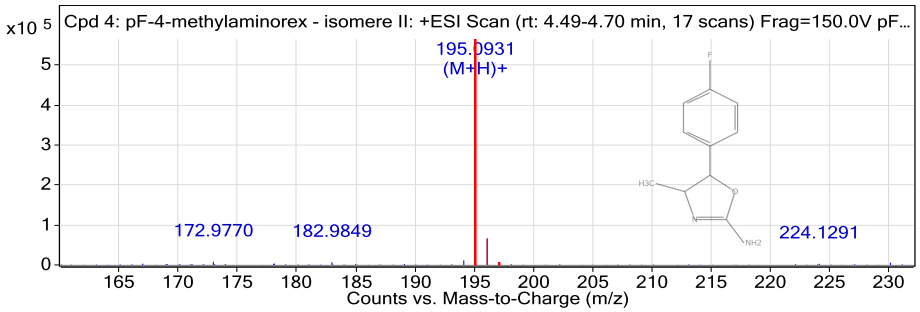




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

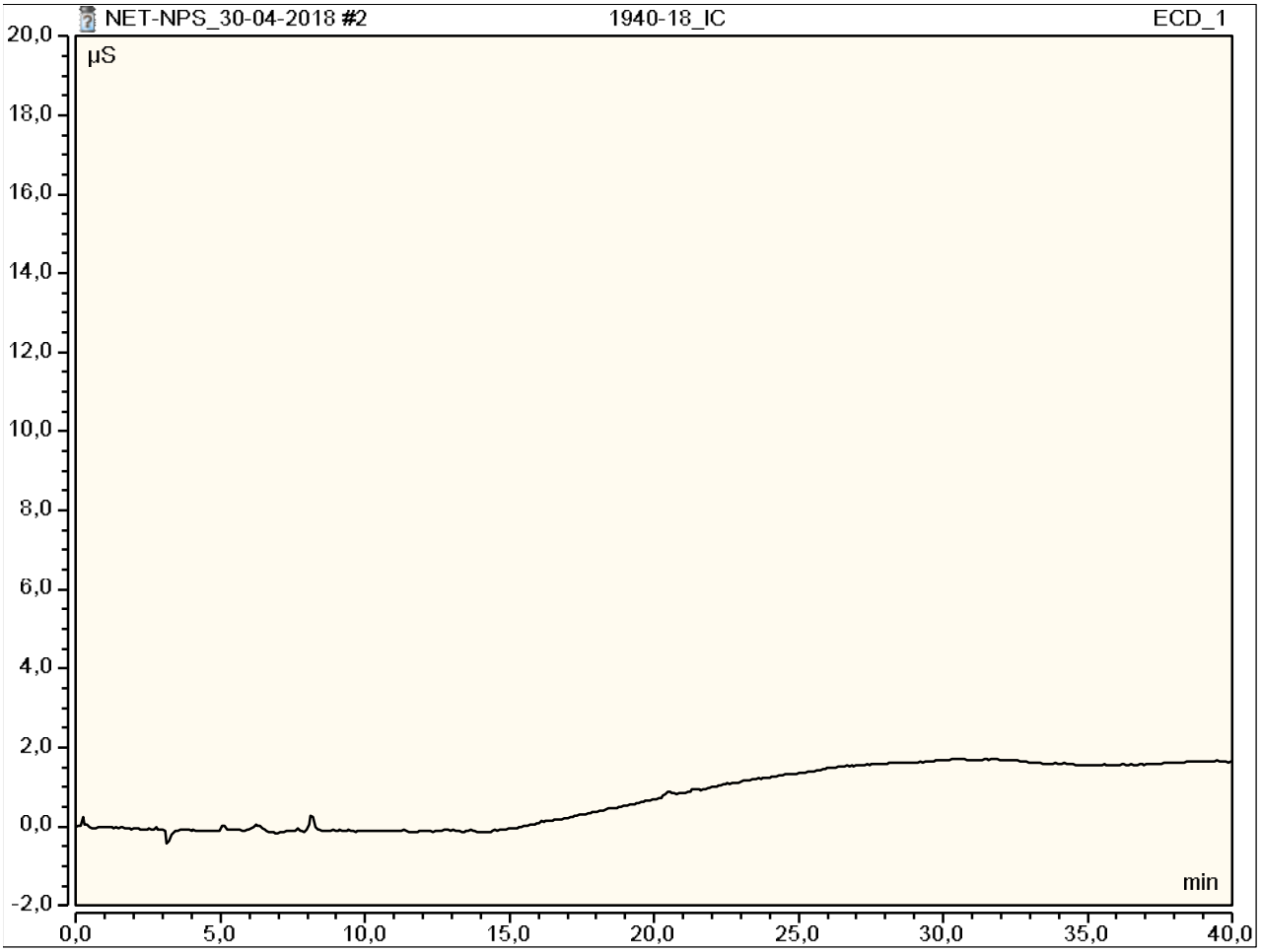
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
195.0931	1	564667.5	C10 H11 F N2 O	(M+H)+
196.096	1	67736.11	C10 H11 F N2 O	(M+H)+
197.0985	1	5842.08	C10 H11 F N2 O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**1940-18_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**30-apr-2018 / 10:01**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount n.a.
		TOTAL:		0,00	0,00	0,00

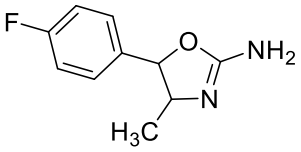


University
of Ljubljana

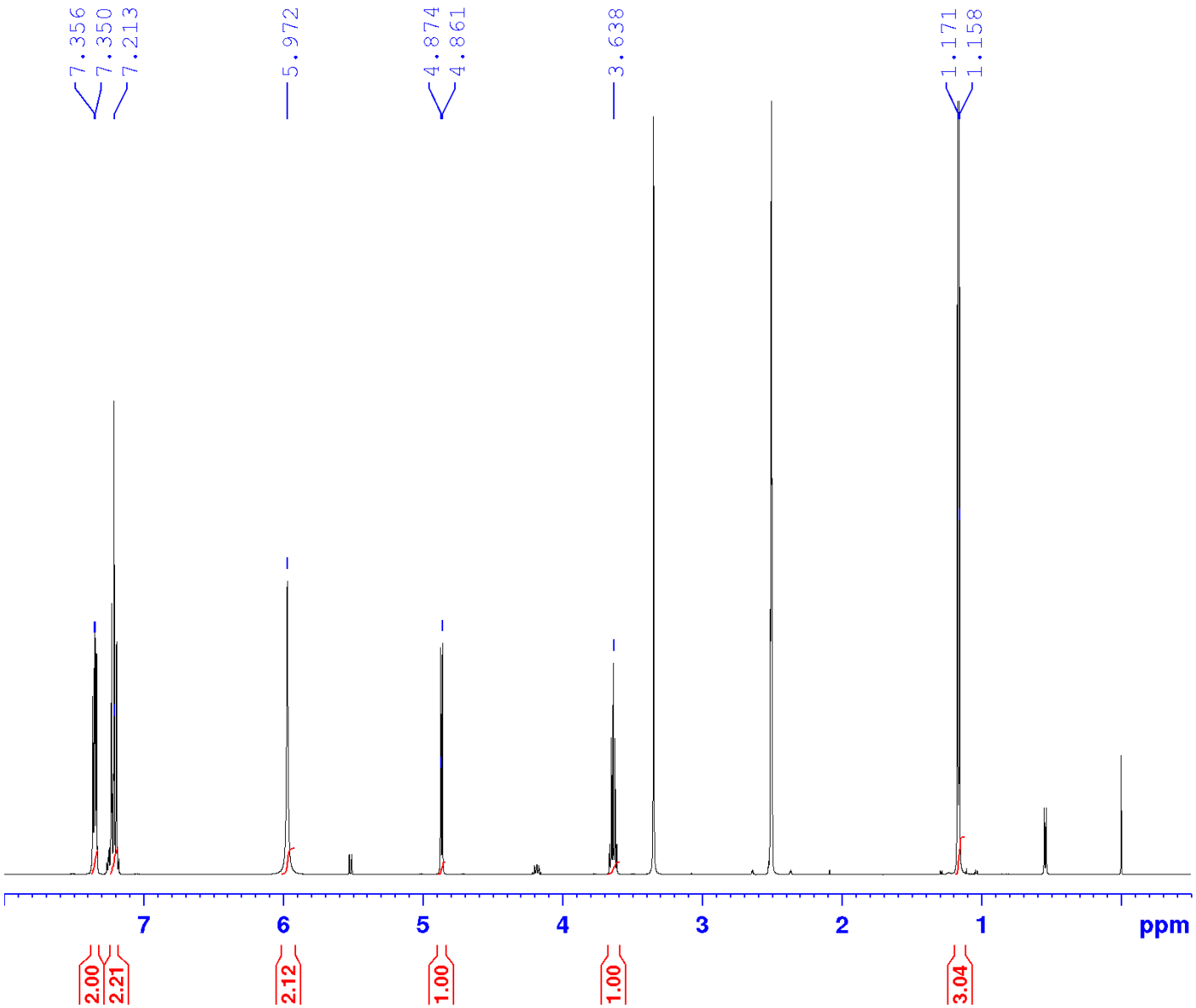
Faculty *of Chemistry and Chemical Technology*

Večna pot 113
P. O. Box 537
SI-1001 Ljubljana
Slovenia
Phone: +386 1 479 8558
janez.kosmrlj@fkkt.uni-lj.si

R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**1940-18**
Received date:	June 4, 2018
Our notebook code:	NFL-1940-18
NMR sample preparation:	10.0 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: $C_{10}H_{11}FN_2O$ Exact Mass: 194,0855 Molecular Weight: 194,2094
Chemical name:	5-(4-fluorophenyl)-4-methyl-4,5-dihydrooxazol-2-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - Based on NMR experiments the sample presumably consist of two isomers, *trans* and *cis* in a molar ratio of 10:1 - >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:	June 21, 2018

NFL-1940-18



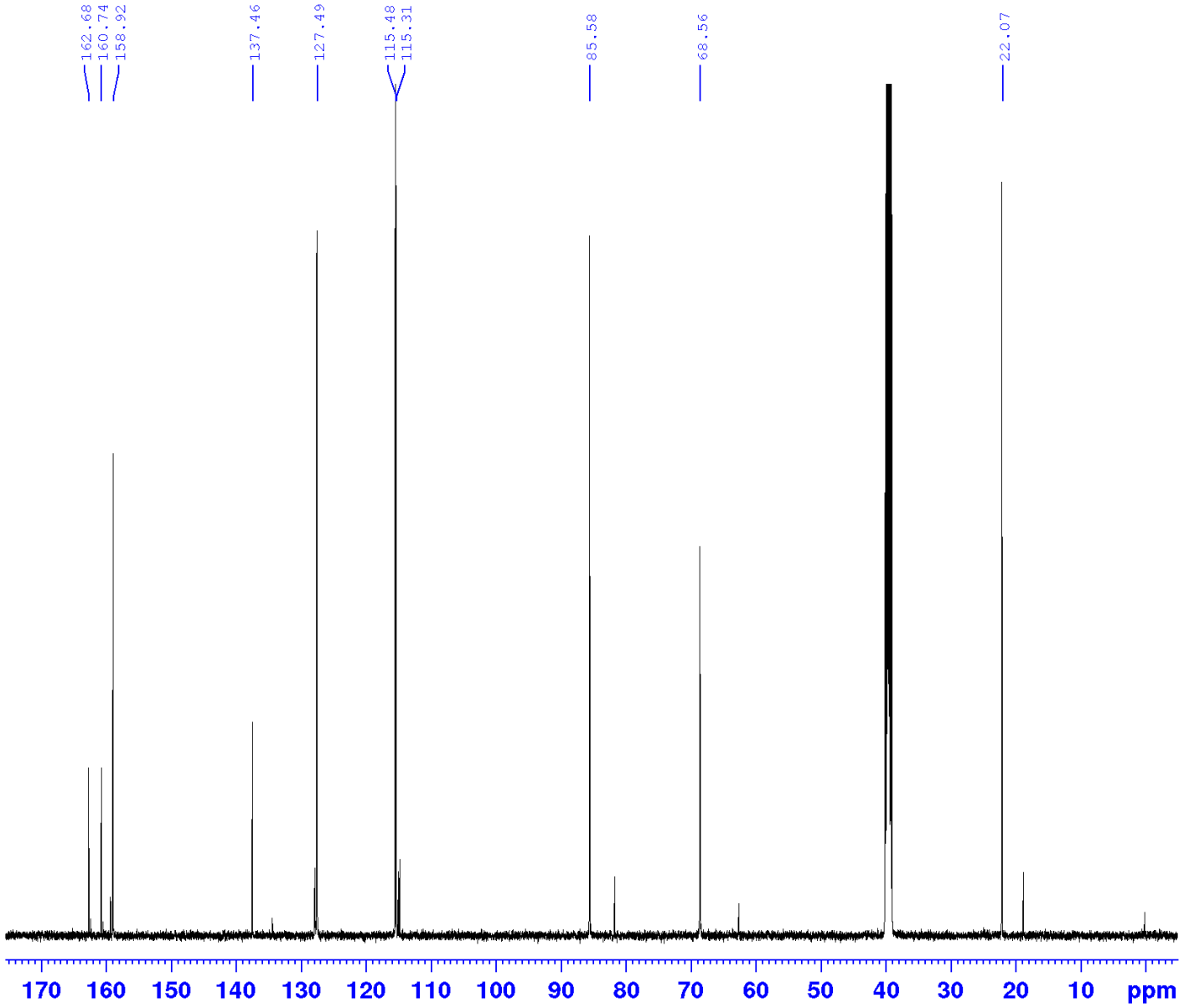

```
Current Data Parameters
NAME                  ok27285
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20180611
Time                  16.12
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                      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             500.1300018 MHz
WDW                      EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```

NFL-1940-18




```
Current Data Parameters
NAME                  ok27285
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20180611
Time                  19.18
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                    5120
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ               1.1010048 sec
RG                    1030
DW                  16.800 usec
DE                   6.50 usec
TE                  296.0 K
D1              1.00000000 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               125.7578460 MHz
WDW                      EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```