NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY
Vodovodna 95, 1000 LJUBLJANA, SLOVENIA

T: +386 (0)1 428 44 93
E: nfl@policija.si
www.policija.si

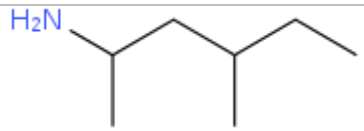
ANALYTICAL REPORT

DMAA (C7H17N)

4-methylhexan-2-amine

Remark – other NPS detected: none

Sample ID:	1834-17
Sample description:	powder
Sample type:	seized /LJ
Date of sample receipt (M/D/Y):	6/8/2017
Date of entry (M/D/Y) into NFL database:	8/18/2017
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	4-methylhexan-2-amine
Other names	1,3-dimethylamylamine, Forthan, Forthane, Floradrene, Geranamine
Formula (per base form)	C7H17N
M_w (g/mol)	115,22
Salt form/anions detected	HCl
StdInChIKey (per base form)	YAHRDLICUYEDAU-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	a mixture of diastereomeres and dimeres (by GC-MS, TOF and 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 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

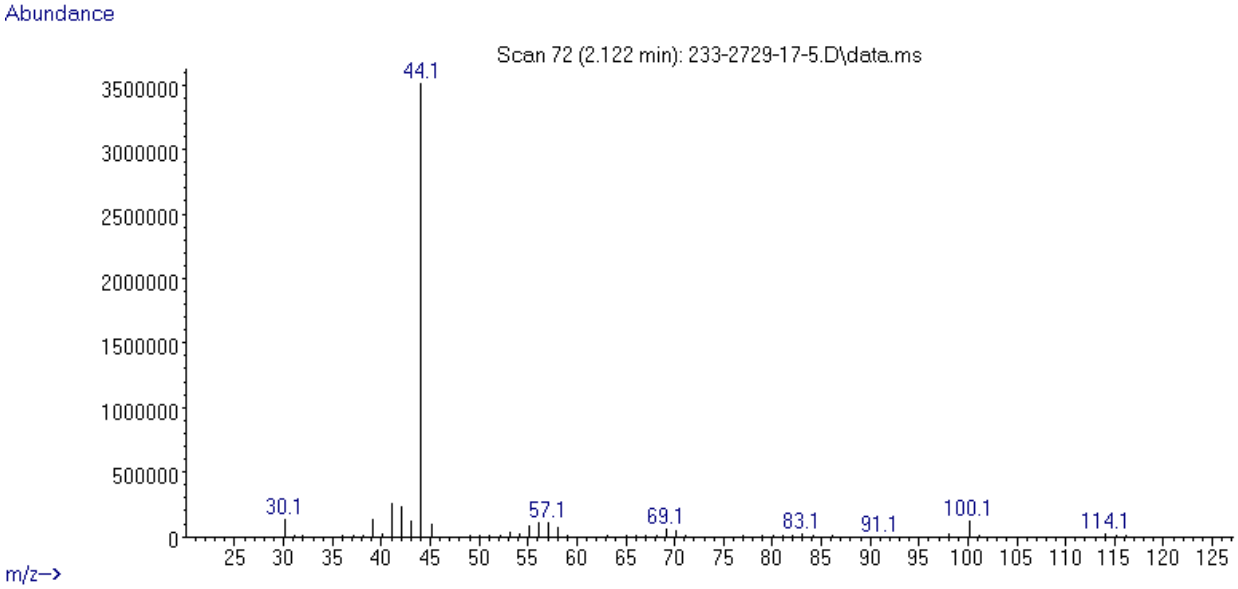
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	soluble

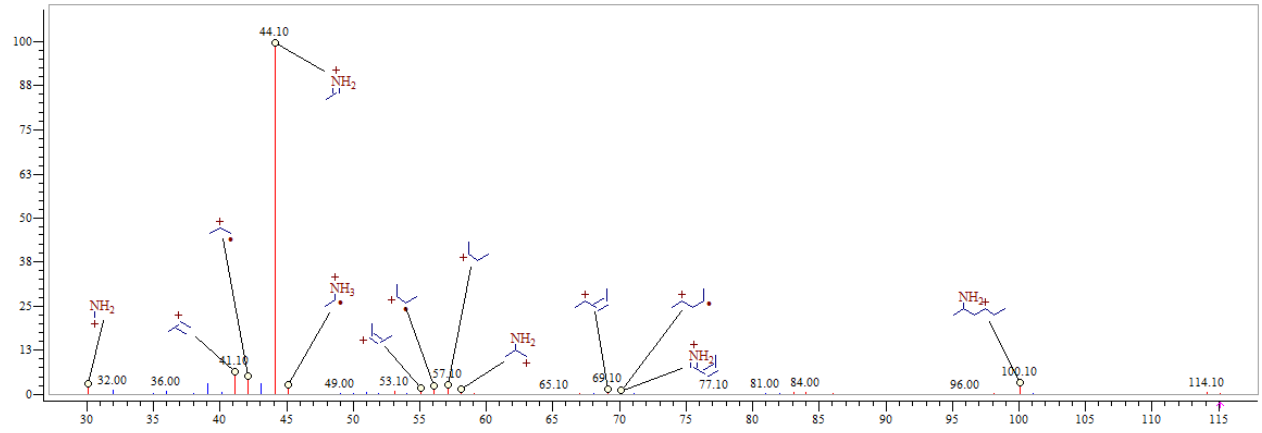
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 2,12 BP(1): 44; BP(2): 41,BP(3) :42,
HPLC-TOF	+	Exact mass (theoretical): 115,1361; measured value Δppm:-4,15; formula:C7H17N
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	-	
IC (anions)	-	
NMR (in FKKT)	+	
validation		MS spectrum consistent by the one in DD2016.L and FTIR match ENFSI IR – ATR 2016 spectrum of DMAA HCl (correlation > 0.997)
other		salt form was confirmed by $AgNO_3$ spot test and by FTIR-ATR

ANALYTICAL RESULTS

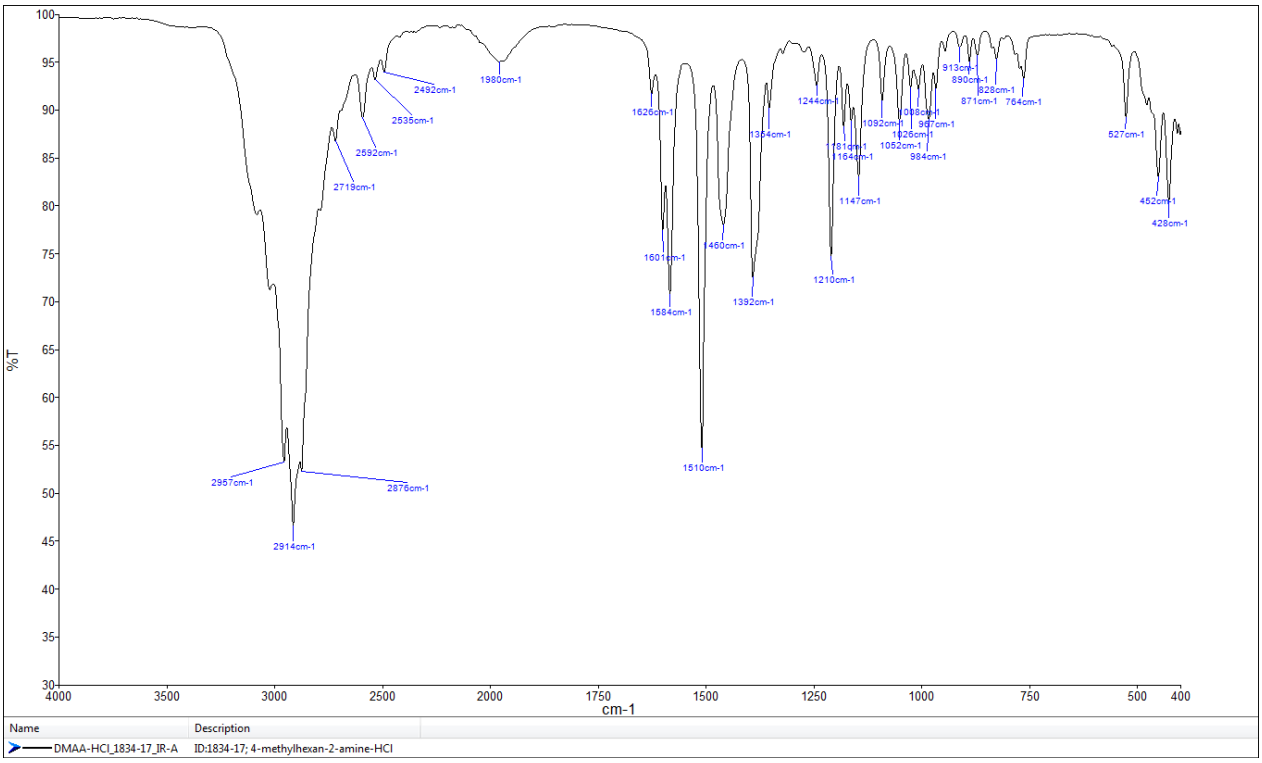
MS (EI)



MS interpretation



FTIR-ATR - direct measurement (sample as received)



I

TOF REPORT

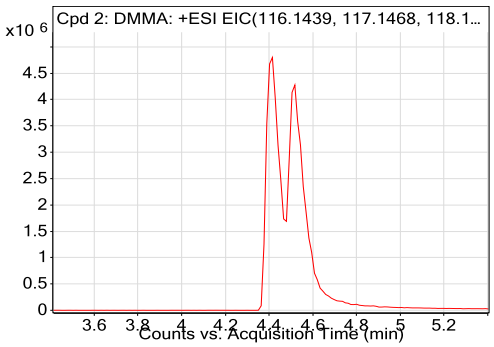
Data File	DMAA_1834-17.d	**Sample Name**	1834-17
Sample Type	Sample	**Position**	P1-C3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-19_07_2017-XDB-C18-ESI-final.m	**Acquired Time**	8/17/2017 12:20:00 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	MeOH		

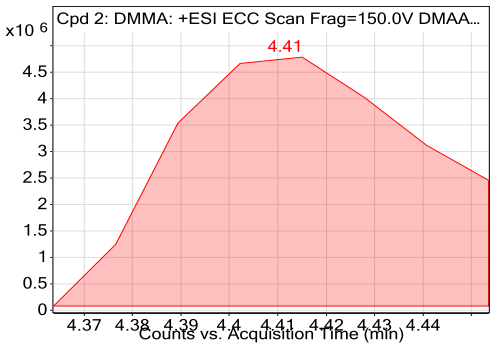
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: DMMA	DMMA	C7 H17 N	4.41	115.1366
Cpd 3: DMMA	DMMA	C7 H17 N	4.52	115.1365
Cpd 5: C14 H31 N		C14 H31 N	7.18	213.2466

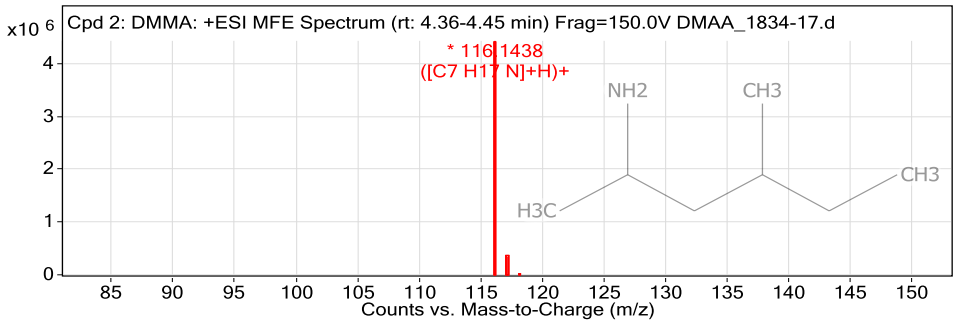
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
DMMA	116.1438	4.41	115.1366	4.38	C7 H17 N	115.1361	-4.15

Compound Chromatograms

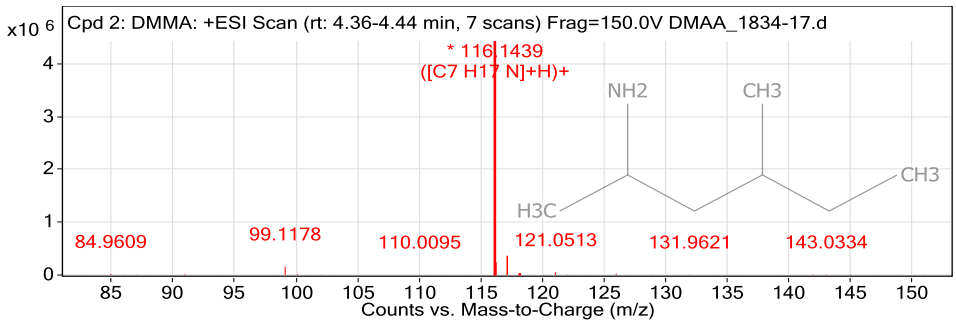




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



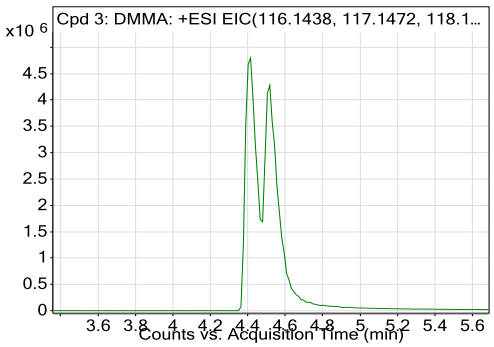
MS Spectrum Peak List

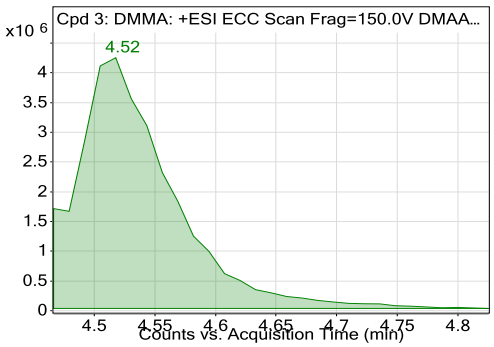
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
116.1438	1	4429150.5	C7 H17 N	(M+H)+
117.1473	1	337698.7	C7 H17 N	(M+H)+
118.1503	1	13654.83	C7 H17 N	(M+H)+

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
DMMA	116.1438	4.52	115.1365	4.49	C7 H17 N	115.1361	-3.77

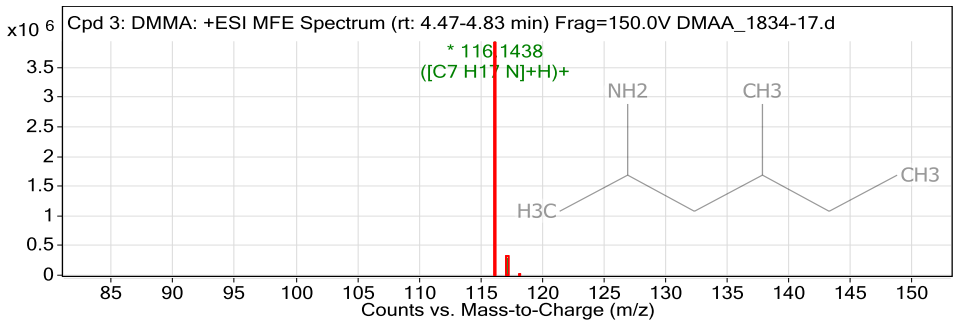
Compound Chromatograms

TOF REPORT

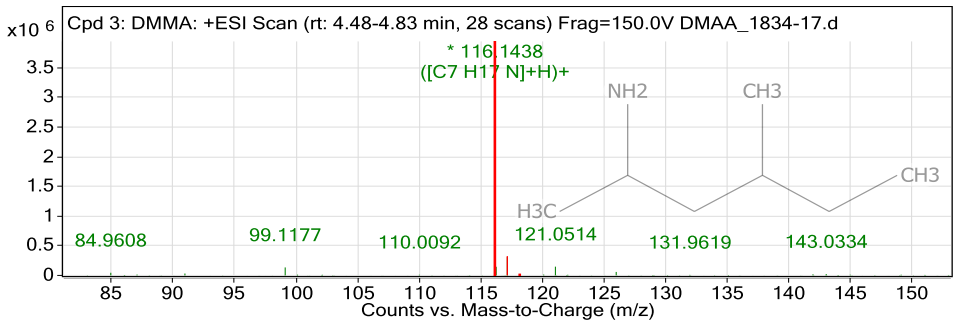




MFE MS Zoomed Spectrum



MS Zoomed Spectrum

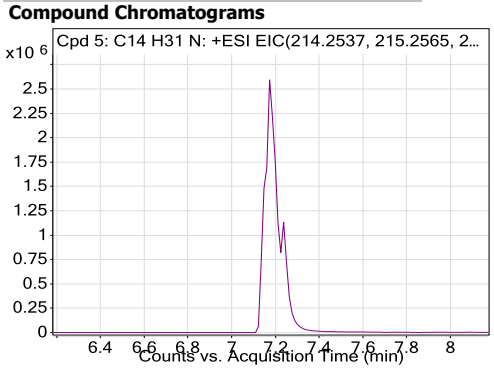


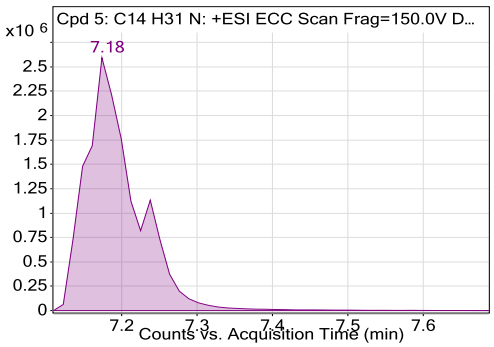
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
116.1438	1	3941153	C7 H17 N	(M+H)+
117.1472	1	295166.56	C7 H17 N	(M+H)+
118.1502	1	12155.35	C7 H17 N	(M+H)+

Obs. m/z	Obs. RT	Obs. Mass
214.2539	7.18	213.2466

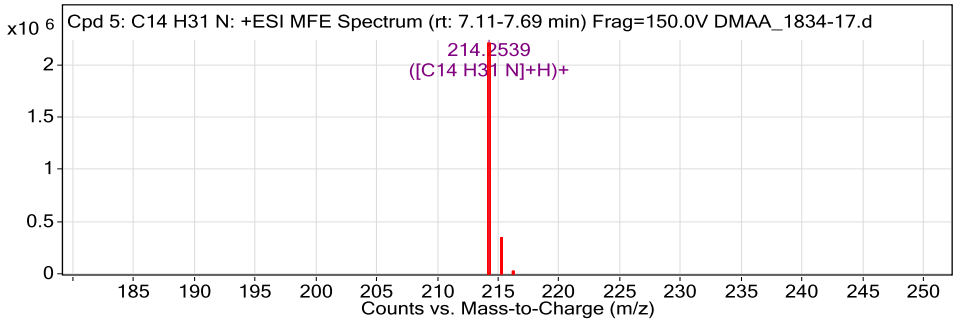
Compound Chromatograms



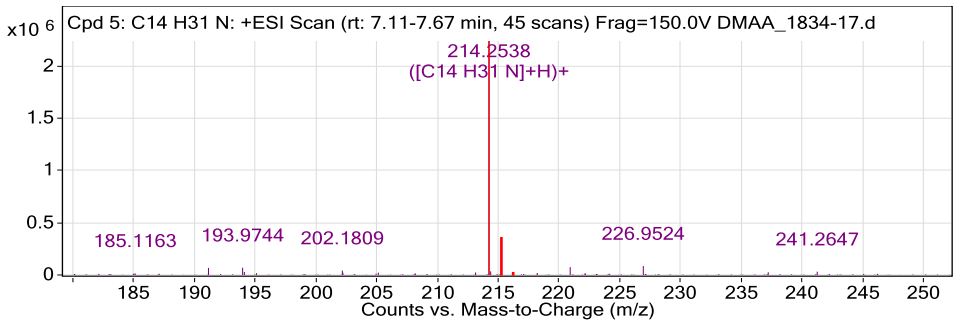


MFE MS Zoomed Spectrum

TOF REPORT



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
214.2539	1	2238120	C14 H31 N	(M+H)+
215.2573	1	318798.3	C14 H31 N	(M+H)+
216.2603	1	23071.4	C14 H31 N	(M+H)+
217.2636	1	818.12	C14 H31 N	(M+H)+

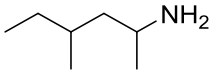
--- End Of Report ---

University
of Ljubljana

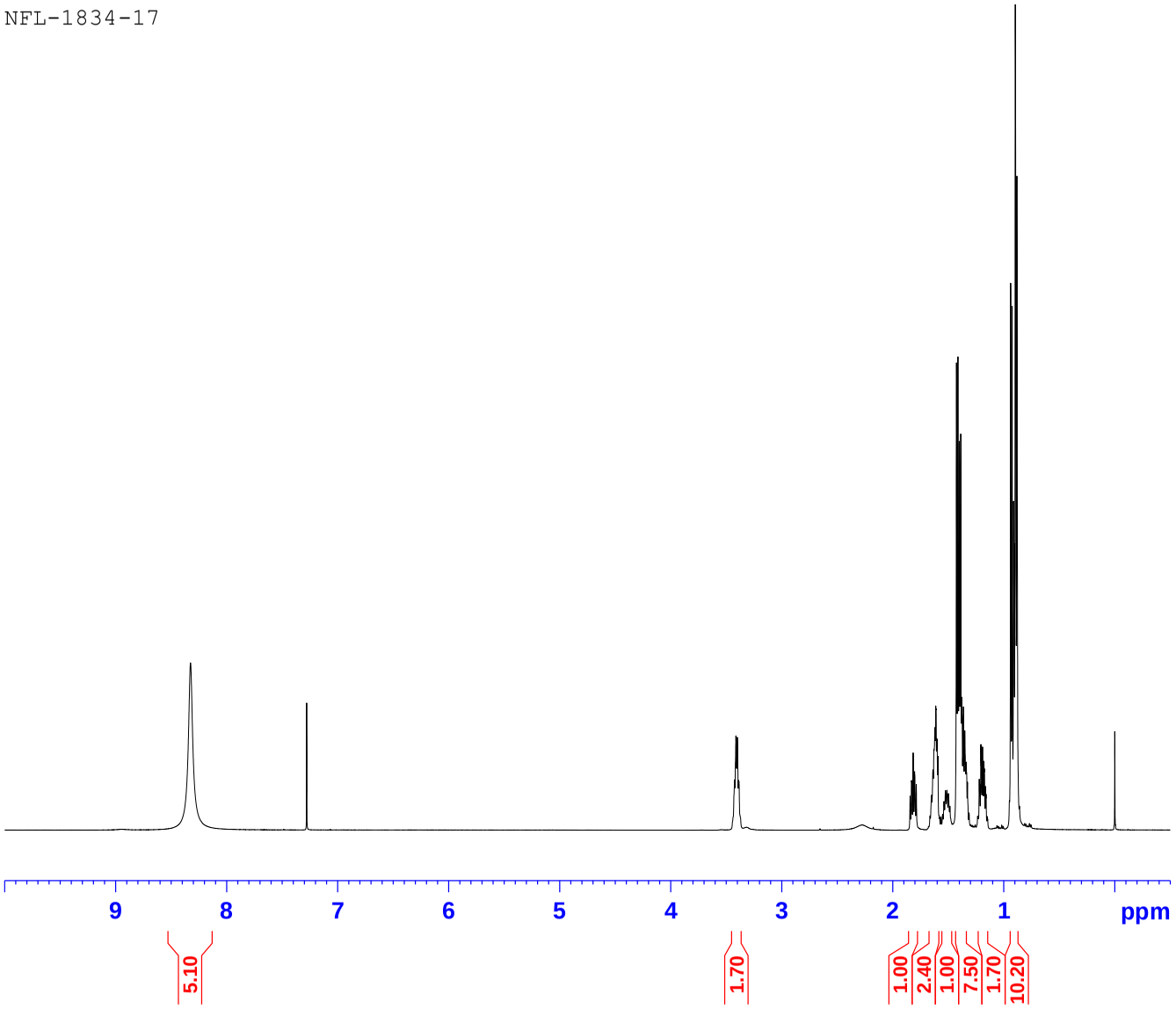
Faculty *of Chemistry and Chemical Technology*

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:	**1834-17**
Received date:	July 12, 2017
Our notebook code:	NFL-1834-17
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
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
Proposed structure, chemical formula, exact mass, molecular weight:	 Chemical Formula: $C_7H_{17}N$ Exact Mass: 115,1 Molecular Weight: 115,2
Chemical name:	4-methylhexan-2-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra. - Compound consists of two diastereomers by NMR (ratio 1 : 0.75).
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Authors:	Marko Krivec, Martin Gazvoda, Janez Košmrlj
Date of report:	July 25, 2017

NFL-1834-17



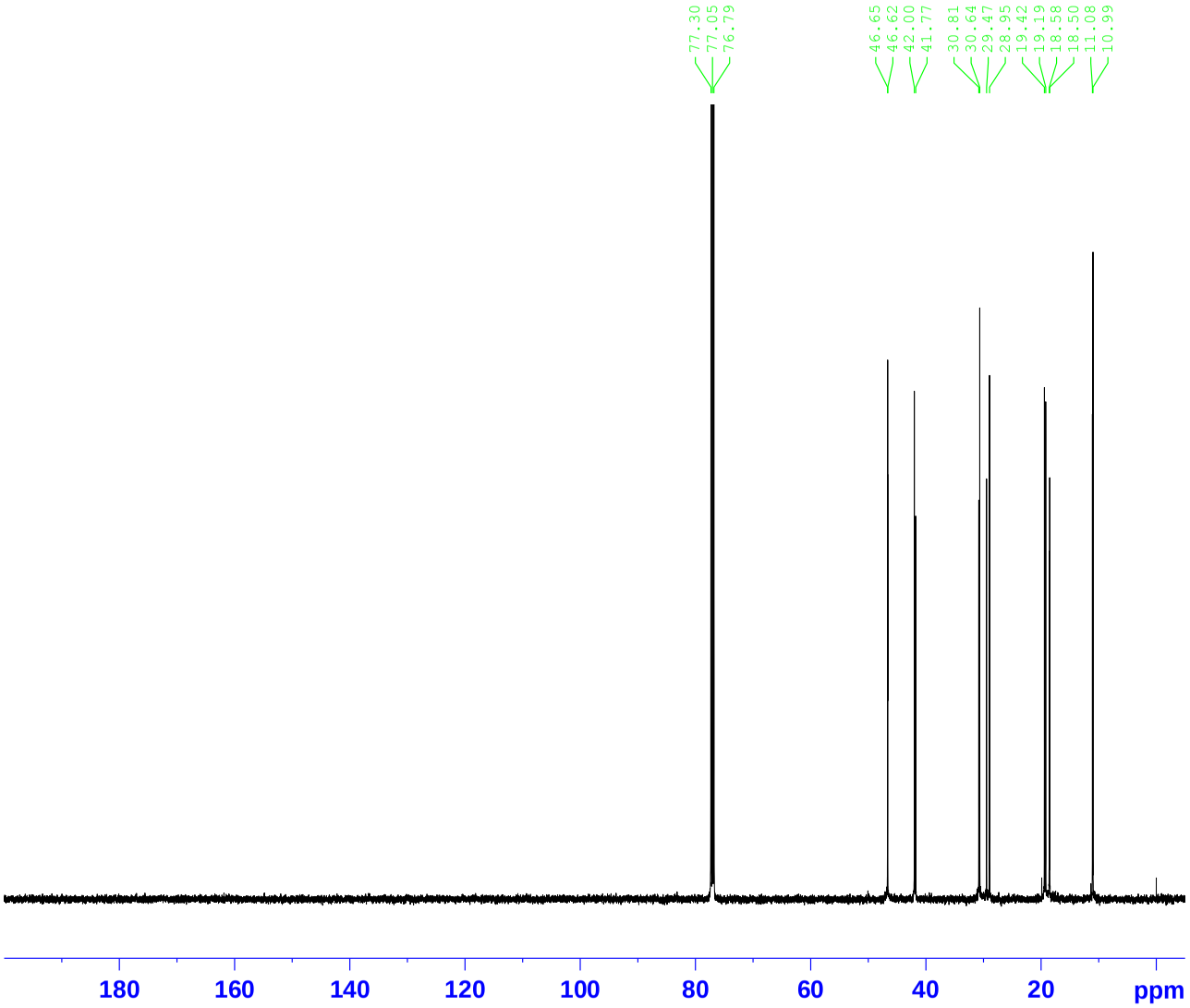
Current Data Parameters
NAME NFL-1834-17
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170716
Time 7.23
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 50.8
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 7.10 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-1834-17



Current Data Parameters
NAME NFL-1834-17
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170716
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5120
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 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 14.00 usec
PLW1 114.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 16.00000000 W
PLW12 0.12250000 W
PLW13 0.06161700 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40