NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY

Vodovodna 95
1000 Ljubljana
SLOVENIJA

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

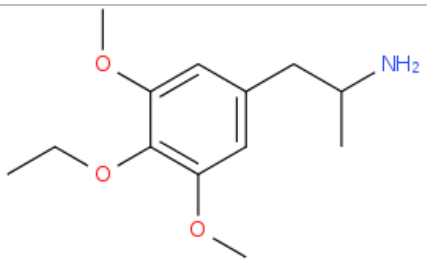
ANALYTICAL REPORT[1,2]

3C-E (C13H21NO3)

1-(4-ethoxy-3,5-dimethoxyphenyl)propan-2-amine

Remark – other NPS detected: none

Sample ID:	1438-16
Sample description:	crystalinic - white
Sample type:	collected /Institute of Forensic medicine, University Freiburg, Germany
Date of sample receipt (M/D/Y):	1/14/2016
Date of entry (M/D/Y) into NFL database:	1/27/2017
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[3] (base form)	
Systematic name	1-(4-ethoxy-3,5-dimethoxyphenyl)propan-2-amine
Other names	3,5-Dimethoxy-4-ethoxy-amphetamine
Formula (per base form)	C13H21NO3
M_w (g/mol)	239,32
Salt form/anions detected	HCl
StdInChIKey (for base form)	AHLXCGRWNKUNTQ-UHFFFAOYSA-N
Other NPS detected	none
Add.info (purity..)	Sample is pure by GC-MS, TOF, and NMR.

[1] This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.
[2] Acknowledgement: Sample (not NMR confirmed) was kindly provided by the Institute of Forensic Medicine, University of Freiburg, Germany (Dr. Verena Angerer). Measurements shown in this report were done in NFL.

[3] 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

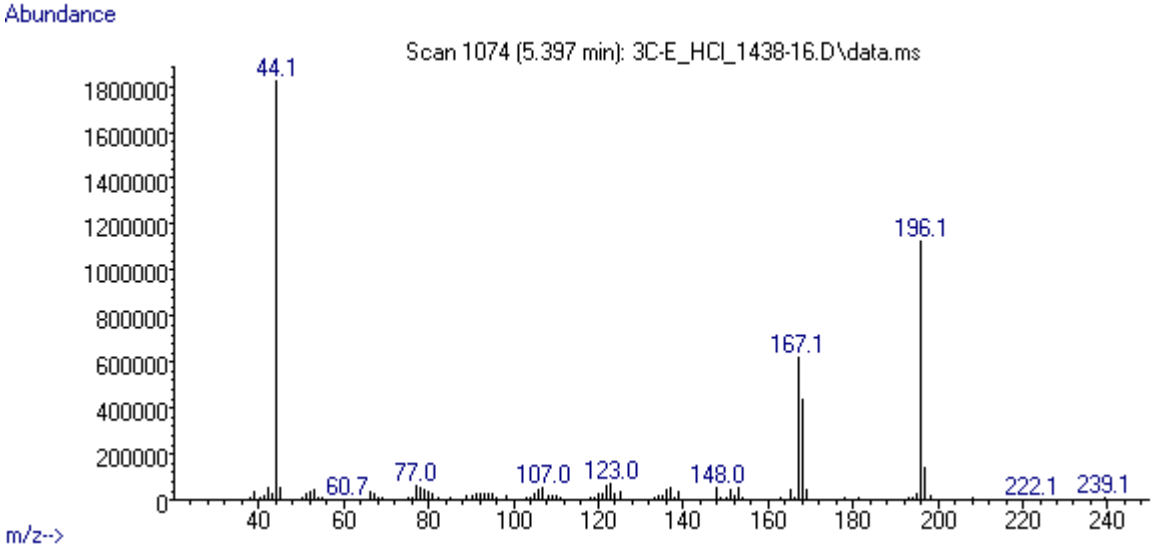
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	soluble

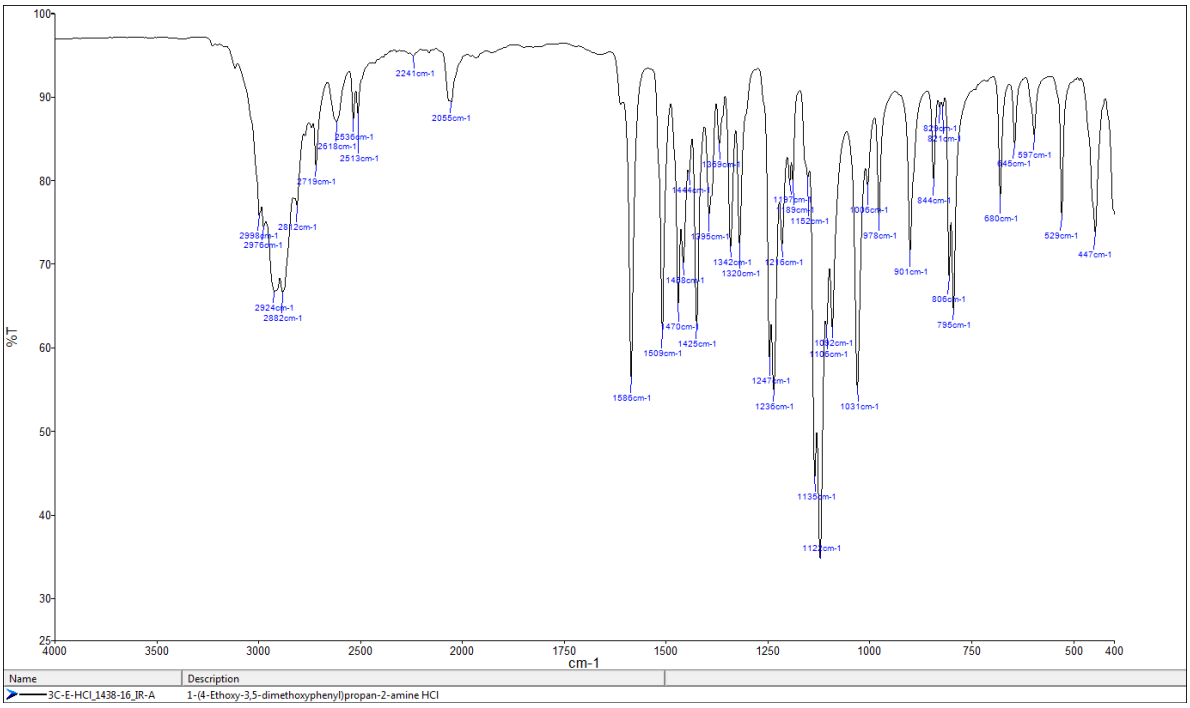
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 5,4 BP(1): 44; BP(2): 196,BP(3) :167,
HPLC-TOF	+	Exact mass (theoretical): 239,1521; measured value Δppm:-0,83; formula:C13H21NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

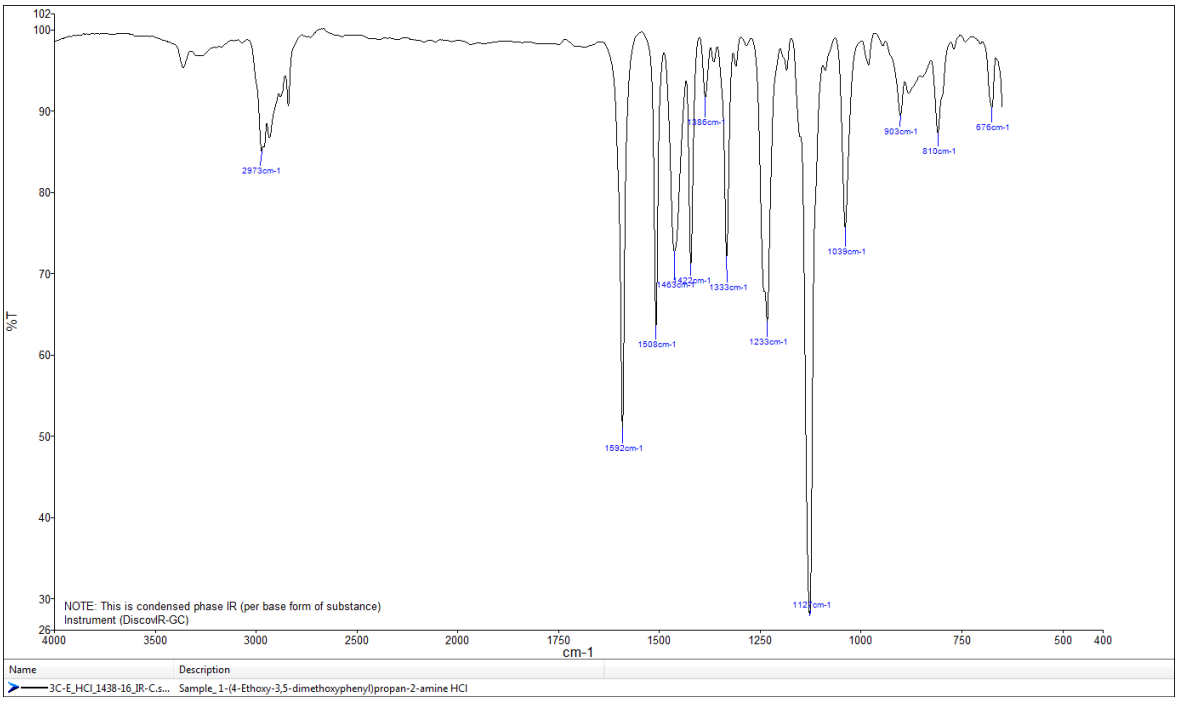
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

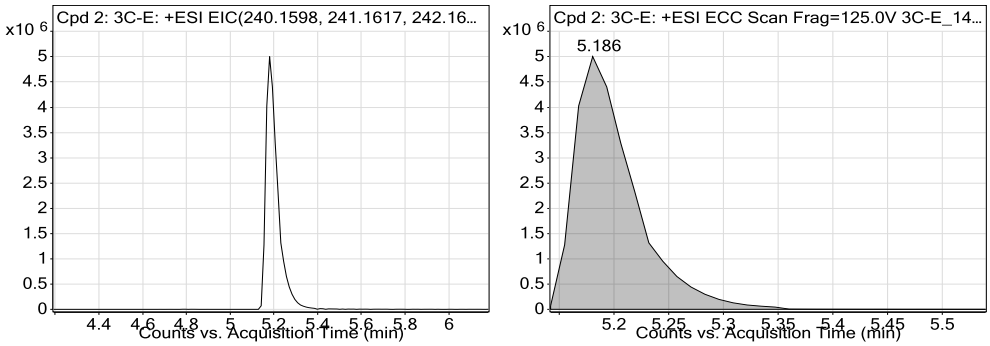
Data File	3C-E_1438-16_TOF.d	**Sample Name**	ID_1438-16
Sample Type	Sample	**Position**	P1-E5
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-1512015-XDB-C18-ESI-poz-pod.m	**Acquired Time**	2/23/2016 9:16:46 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

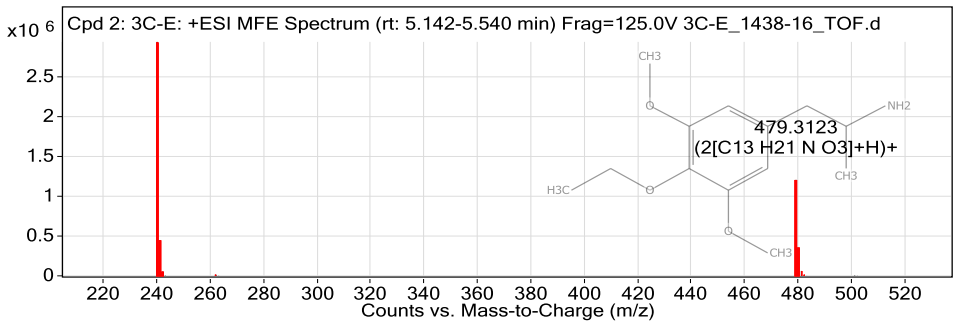
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: 3C-E	3C-E	C13 H21 N O3	5.186	239.1523

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
3C-E	240.1595	5.186	239.1523	5.19	C13 H21 N O3	239.1521	-0.83

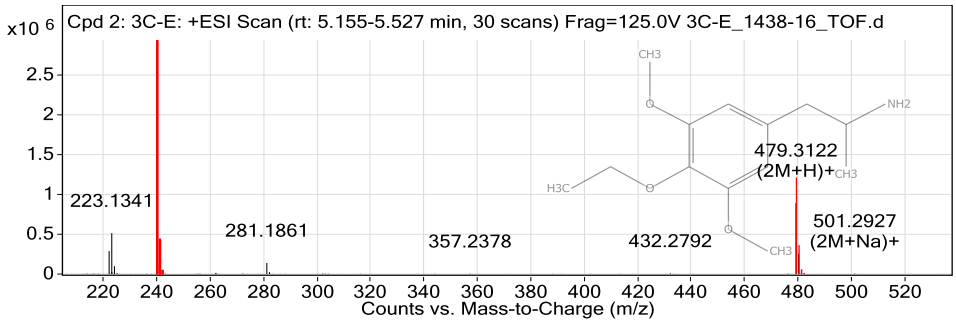
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

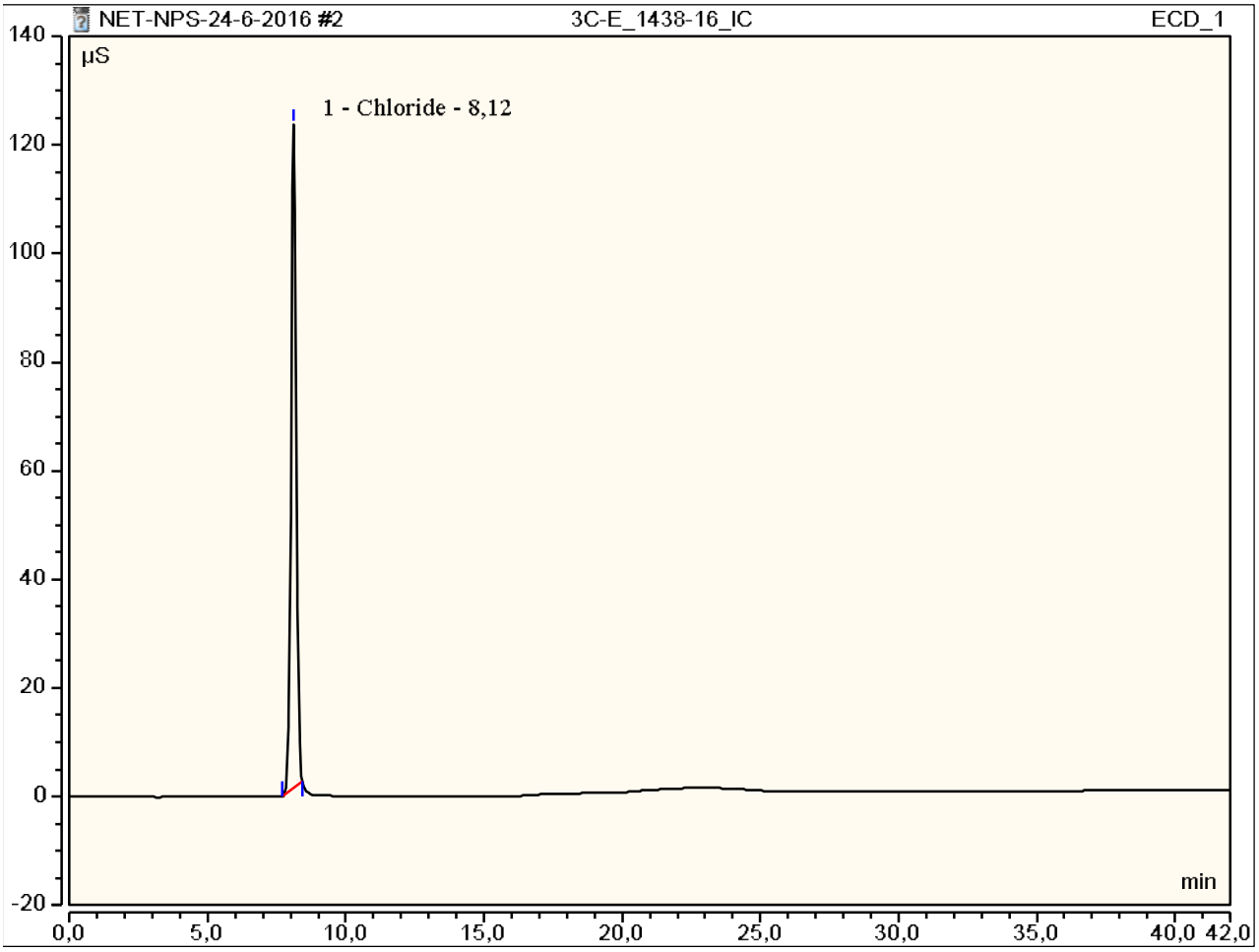
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
240.1595	1	2938373.5	C13 H21 N O3	(M+H)+
241.1635	1	421222.05	C13 H21 N O3	(M+H)+
242.1652	1	52314.68	C13 H21 N O3	(M+H)+
243.1676	1	4227.87	C13 H21 N O3	(M+H)+
262.1414	1	12957.17	C13 H21 N O3	(M+Na)+
479.3123	1	1205278.13	C13 H21 N O3	(2M+H)+
480.3155	1	345807.94	C13 H21 N O3	(2M+H)+
481.3175	1	61564.94	C13 H21 N O3	(2M+H)+
482.3199	1	8117.16	C13 H21 N O3	(2M+H)+
501.2929	1	5114.34	C13 H21 N O3	(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**3C-E_1438-16_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**24-jun-2016 / 11:55**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	8,12	**Chloride**	BMB	25,99	122,20	n.a.
		TOTAL:		25,99	122,20	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

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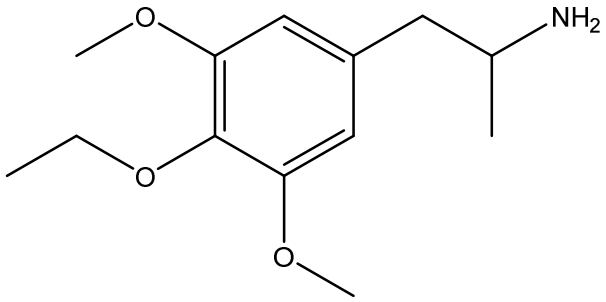
REPORT

Sample ID:	**1438-16**
Our notebook code:	P-1438-16
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
NMR experiments:	^1H, ^{13}C, ^1H–^1H *gs*-COSY, ^1H–^{13}C *gs*-HSQC, ^1H–^{13}C *gs*-HMBC, ^1H–^{15}N *gs*-HMBC.
Proposed structure:	
Chemical name:	1-(4-ethoxy-3,5-dimethoxyphenyl)propan-2-amine
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure according to the NMR.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 23, 2017

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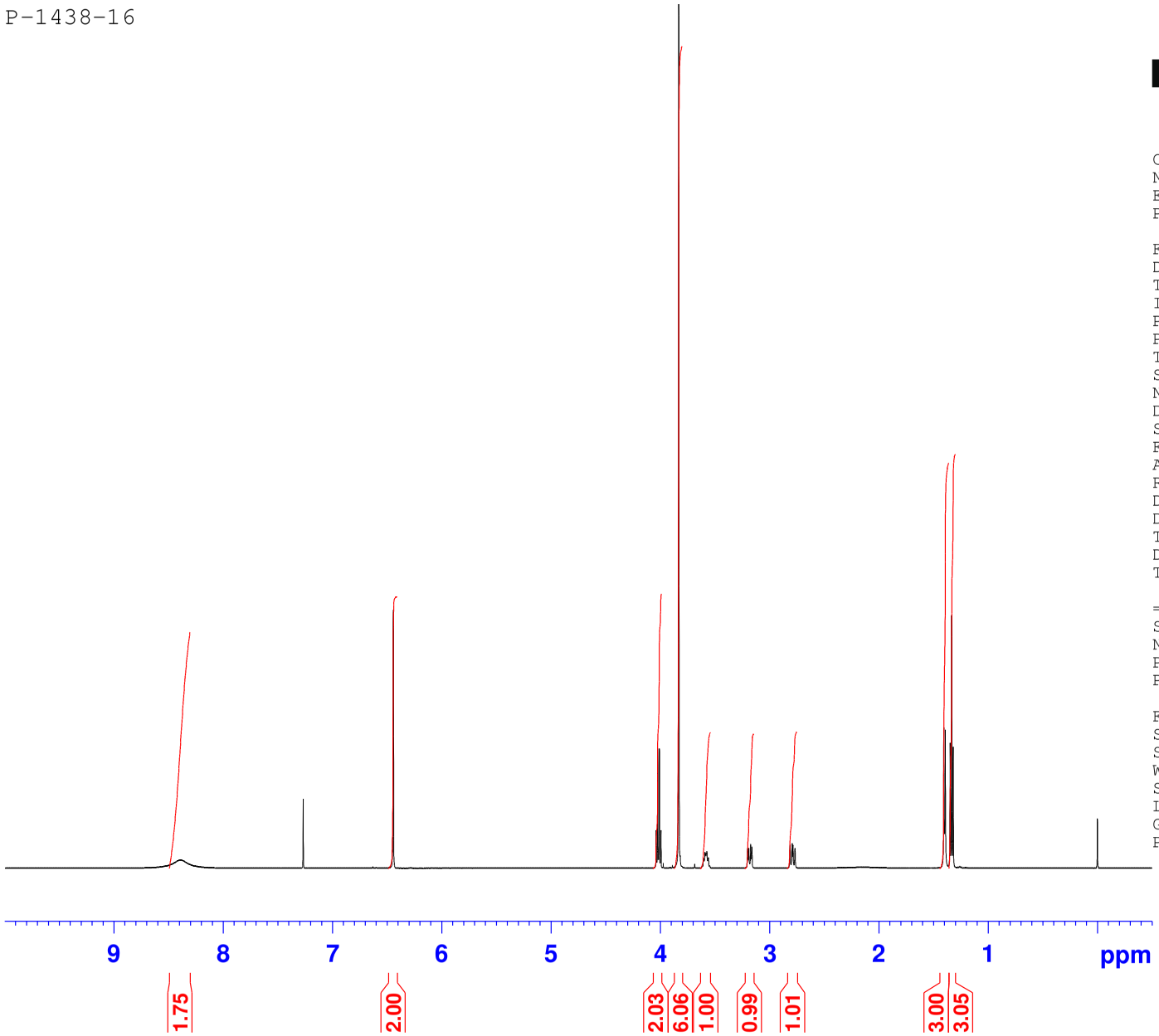
P-1438-16



Current Data Parameters
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EXPNO 1
PROCNO 1

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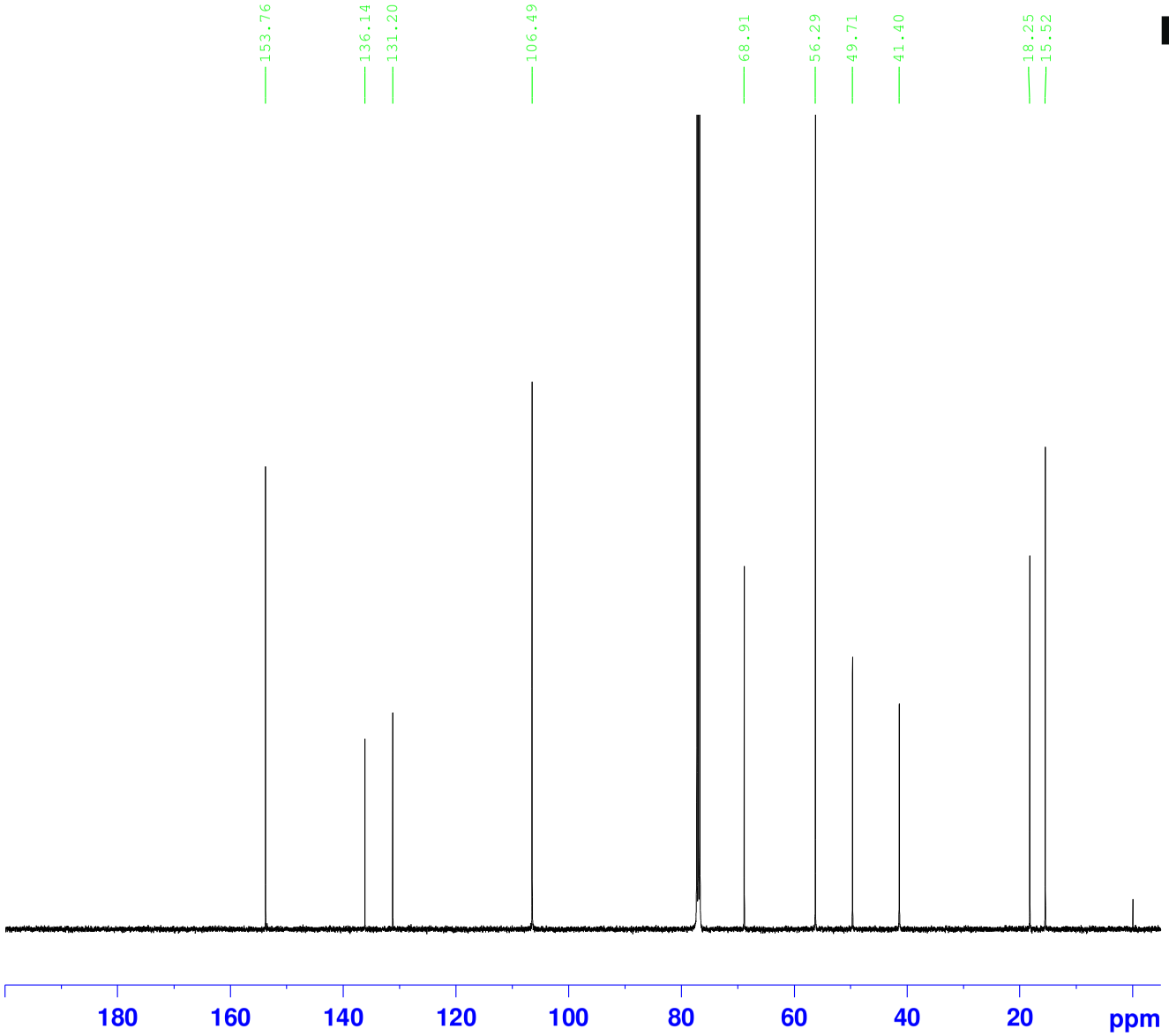
Date_ 20160806
Time 12.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 101
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.90 usec
PLW1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1438-16



BRUKER

```
Current Data Parameters
NAME                  P-1438-16
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160806
Time                      18.33
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        6144
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                      300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               125.7703637 MHz
NUC1                        13C
P1                         9.00 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               0.32179001 W
PLW13               0.16186000 W

F2 - Processing parameters
SI                        32768
SF                 125.7577878 MHz
WDW                          EM
SSB          0
LB                         1.00 Hz
GB           0
PC                         1.40
```