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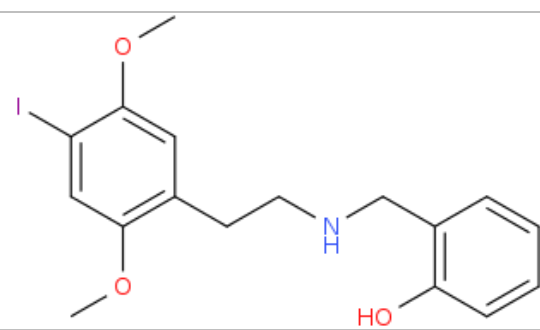
ANALYTICAL REPORT¹

25I-NBOH (C₁₇H₂₀INO₃)

2-([2-(4-iodo-2,5-dimethoxyphenyl)ethyl]amino)methylphenol

Remark – other NPS detected: **none**

Sample ID:	1383-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	12/9/2015
Date of entry (M/D/Y) into NFL database:	2/19/2016
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure ² (base form)	
Systematic name	2-([2-(4-iodo-2,5-dimethoxyphenyl)ethyl]amino)methylphenol
Other names	NBOH-2CI, Cimbi-27, 2C-I-NBOH
Formula (per base form)	C ₁₇ H ₂₀ INO ₃
M _w (g/mol)	413,26
Salt form/anions detected	chloride
StdInChIKey	FEUZHXRQGQTBRO-UHFFFAOYSA-N
Compound Class	Phenethylamines
Other NPS detected	none
Add.info (purity..)	evidence of minor impurities

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 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 (RT=9.53 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 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 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) 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 °C. Chromatographic separation as above **(1)**. Split MS : IR = 1:9.

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 (condensed 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

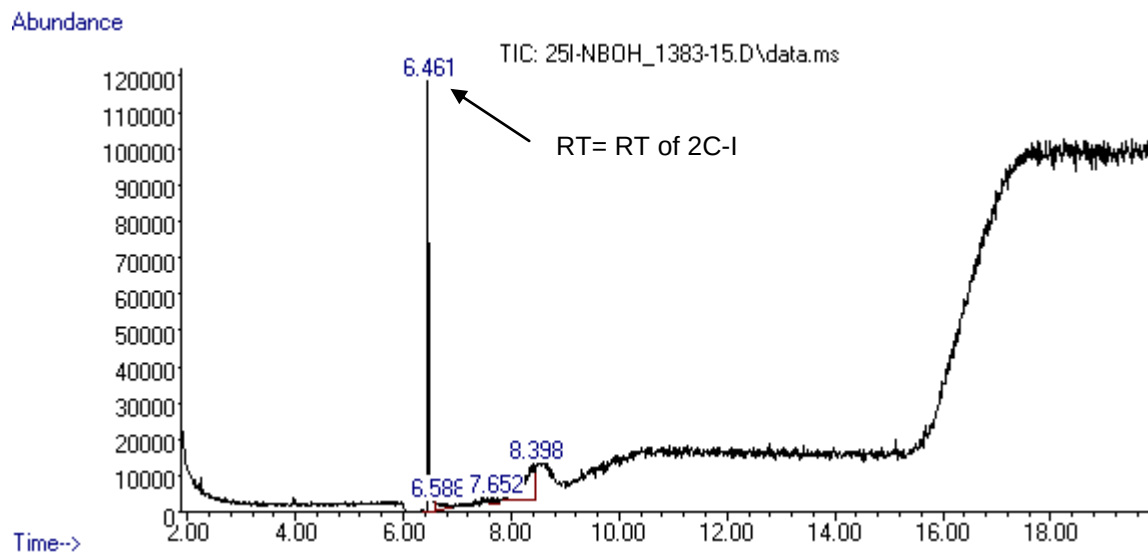
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 12,04 (for di-TMS, i.e. O-TMS and N-TMS derivative) BP(1): 179; BP(2): 280,BP(3) :73,
HPLC-TOF	+	Exact mass (theoretical): 413,0488; measured value Δppm:-0,38; formula:C17H20INO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	-	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		In GC - non derivatized compound decomposes to 2C-I.

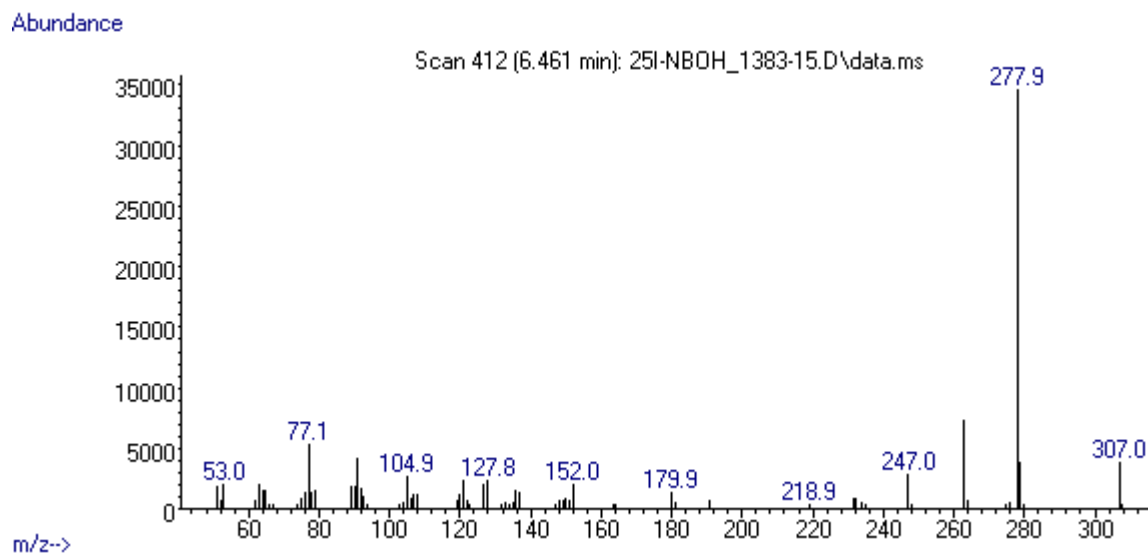
ANALYTICAL RESULTS

Non derivatized compound decomposes to 2C-I under our chromatographic conditions. The same effect we observed for 2C-NBOH.

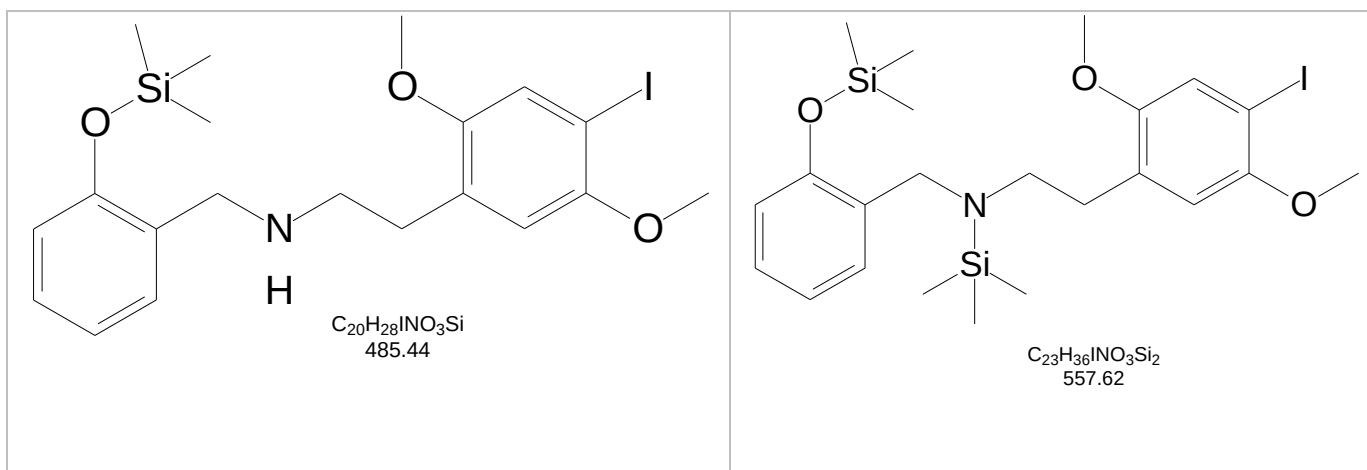
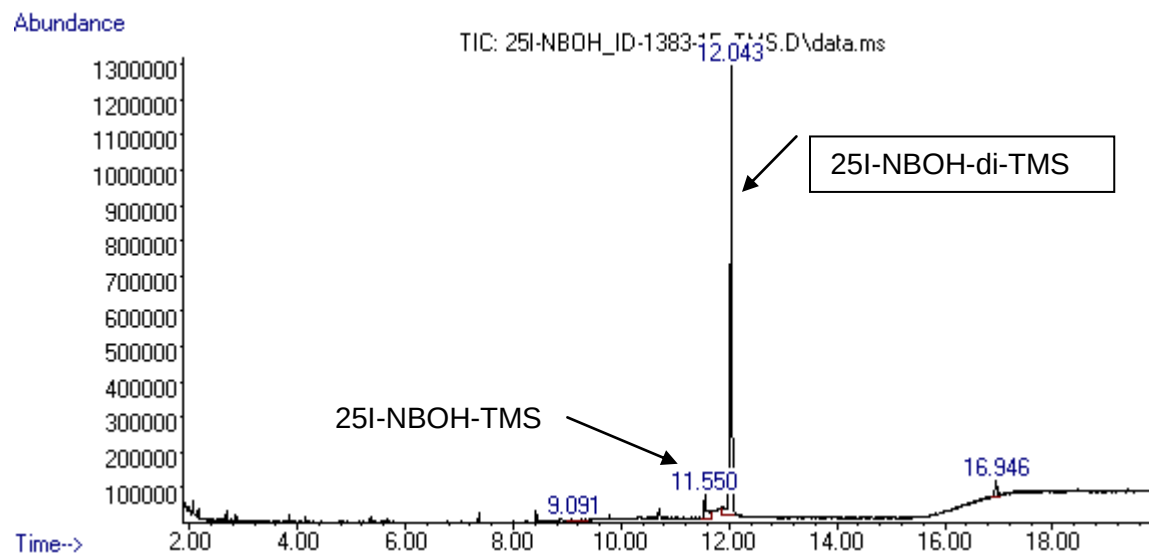
GC-MS (EI) of the **decomposition** product, i.e. 2C-I.



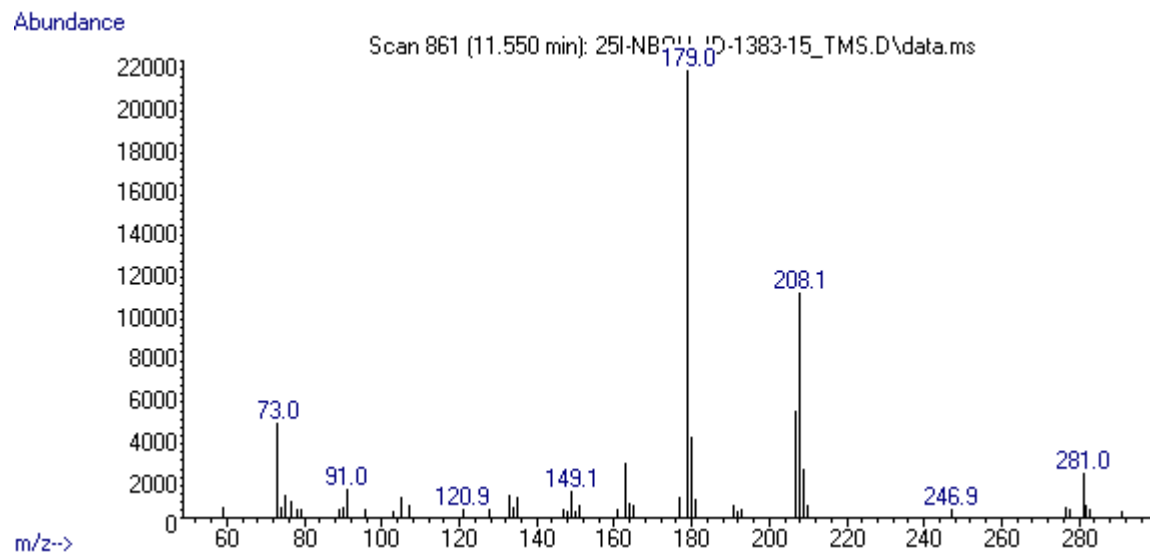
MS (EI) of **decomposition** product **2C-I** (MS consistent by SWGDRUG)



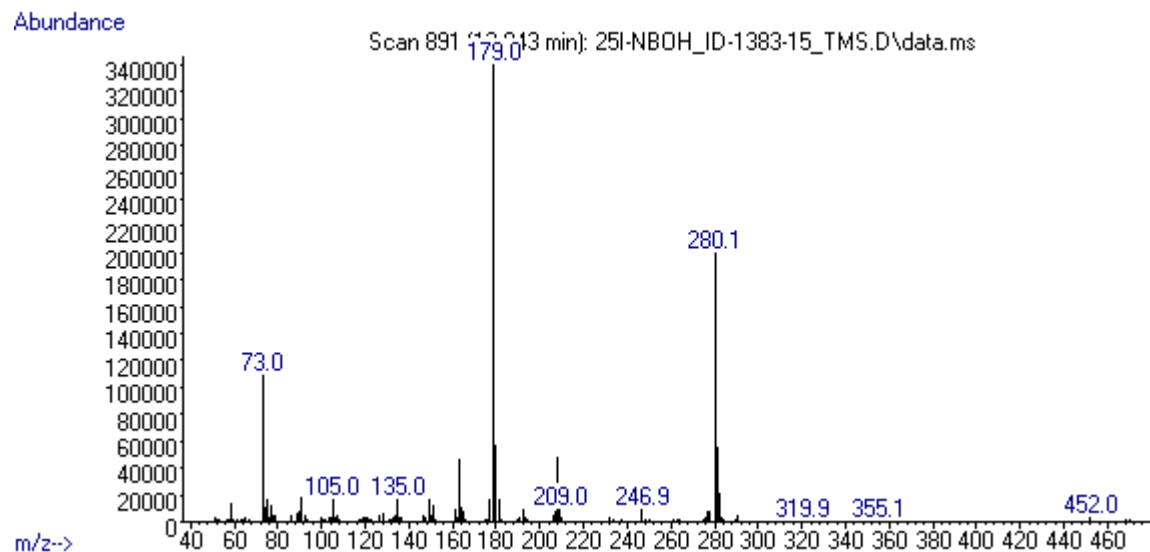
Chromatogram of derivatized sample (by MSTFA):



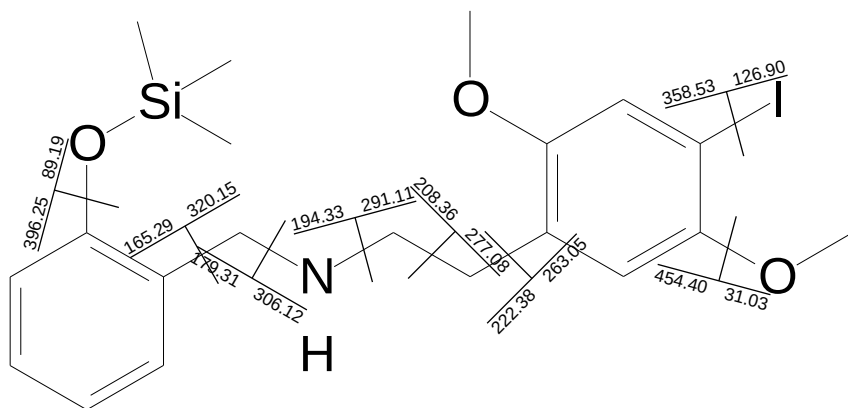
MS (EI) spectrum of 25I-NBOH-TMS (O-TMS-derivative)



MS (EI) spectrum of 25I-NBOH-diTMS (O-TMS, N-TMS derivative)

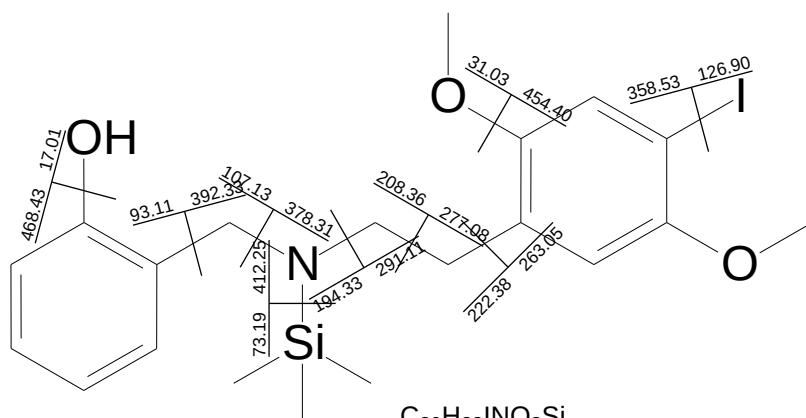


FRAGMENTATION:



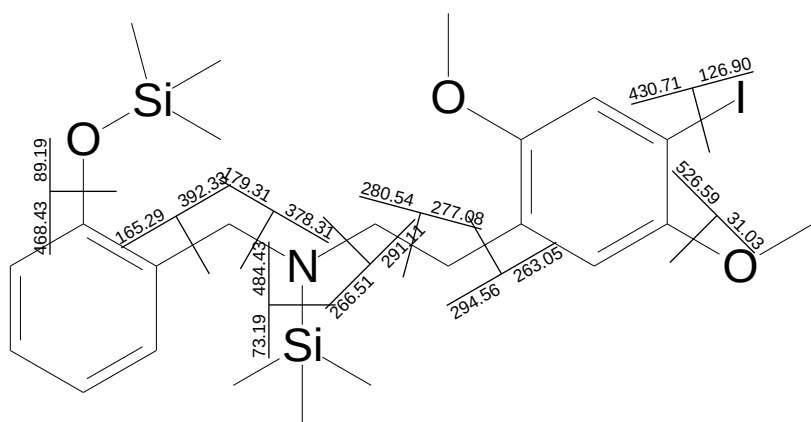
YES

$C_{20}H_{28}INO_3Si$
485.44



NO

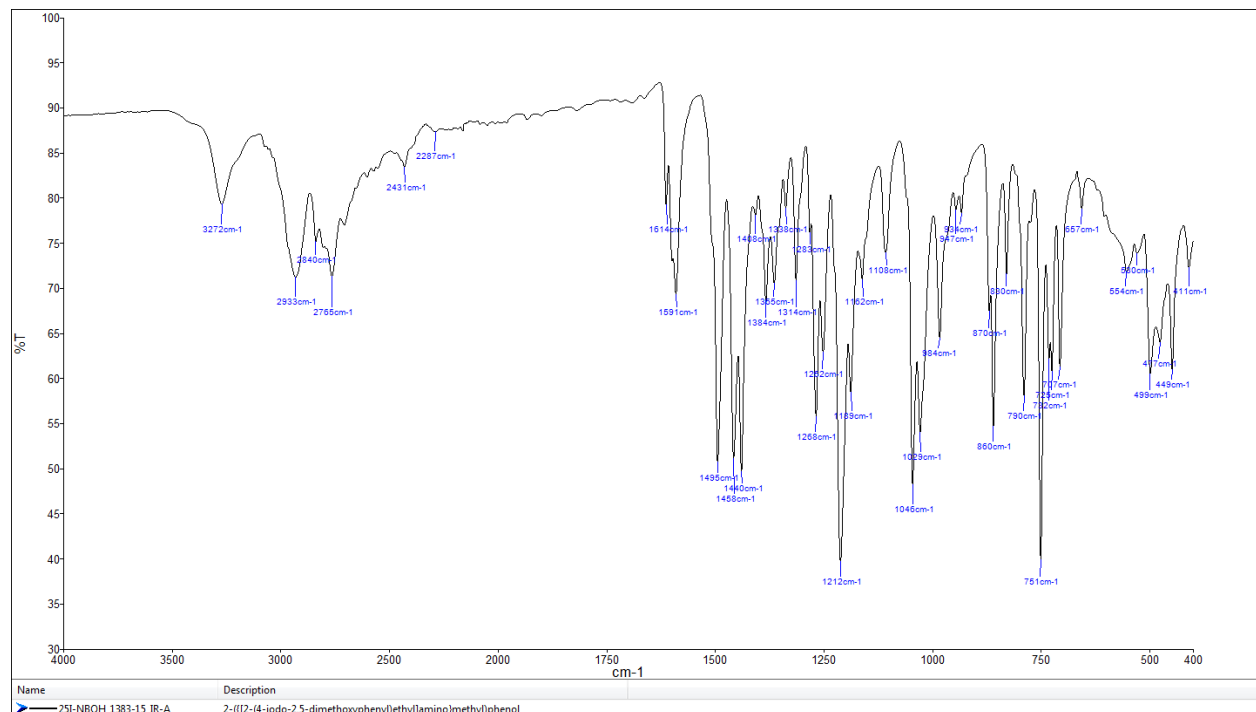
$C_{20}H_{28}INO_3Si$
485.44



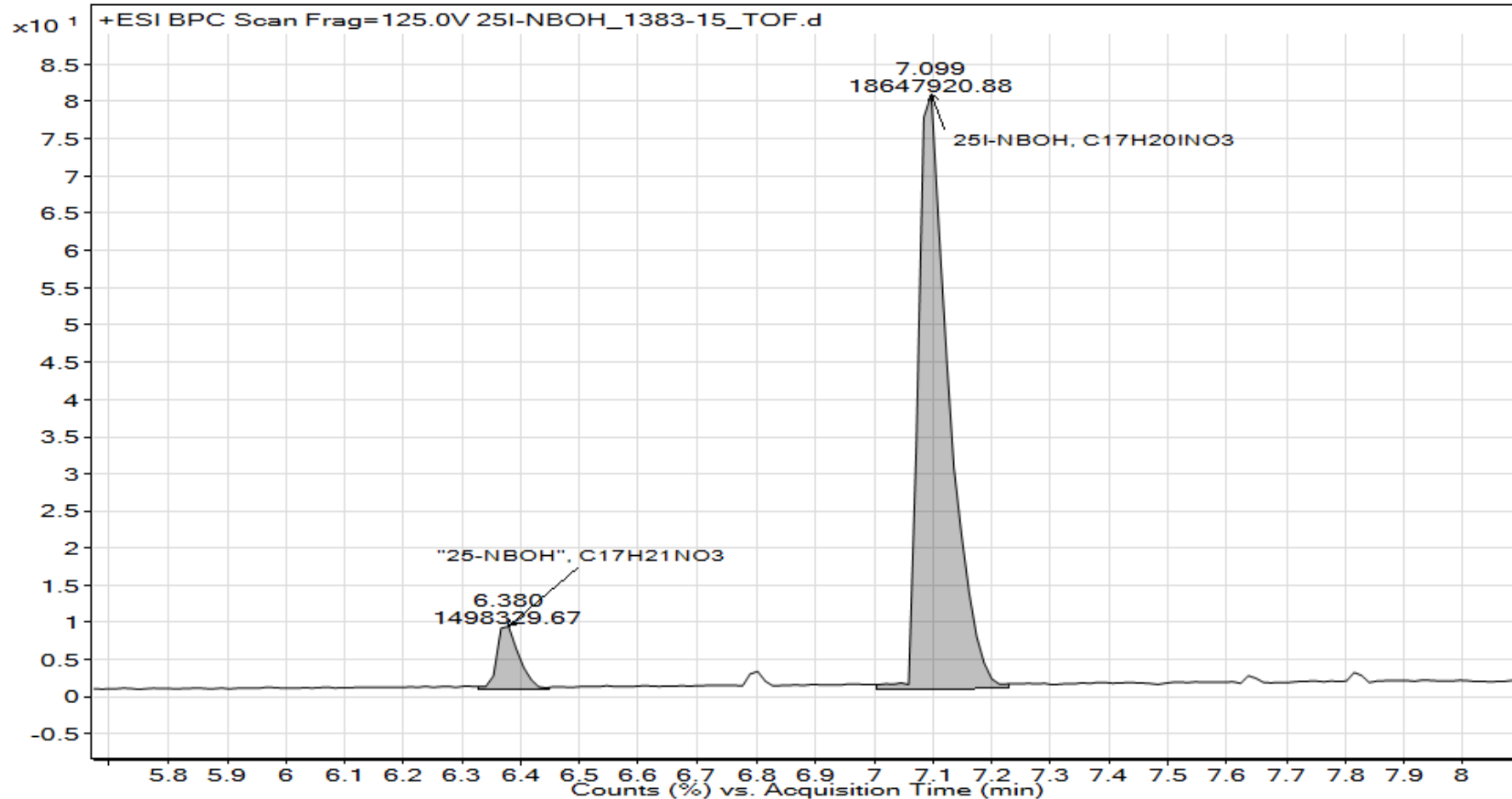
YES

$C_{23}H_{36}INO_3Si_2$
557.62

FTIR-ATR - direct measurement (sample as received)



Sample Name	ID_1383-15	Position	P1-C5	Instrument Name	6230B TOF LC-MS	User Name	TG
Inj Vol	1	Inj Position		Sample Type	Sample	IRM Calibration Status	Success
Data Filename	25I-NBOH_1383-15_TOF	ACQ Method	general-1512015-XDB-	Comment	extract in MeOH	Acquired Time	12/15/2015 1:18:16 PM



TOF REPORT

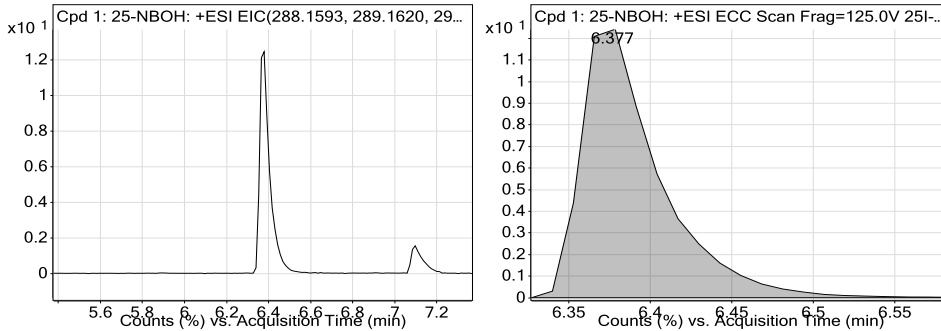
Data File	25I-NBOH_1383-15_TOF.d	Sample Name	ID_1383-15
Sample Type	Sample	Position	P1-C5
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-1512015-XDB-C18-ESI-poz.m	Acquired Time	12/15/2015 1:18:16 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

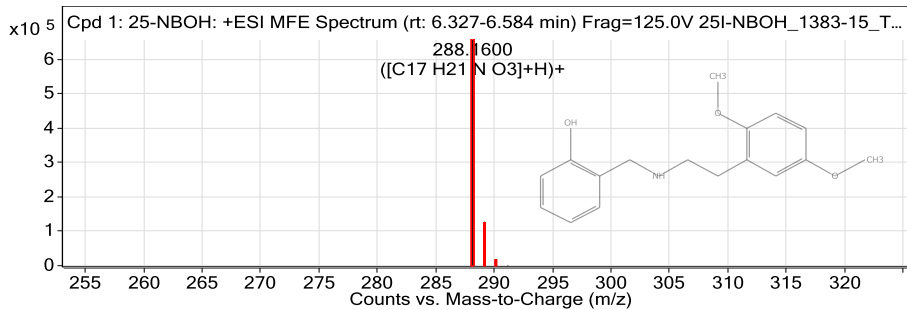
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: 25-NBOH	25-NBOH	C17 H21 N O3	6.377	287.1527
Cpd 2: 25I-NBOH	25I-NBOH	C17 H20 I N O3	7.097	413.0487

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
25-NBOH	288.16	6.377	287.1527	6.35	C17 H21 N O3	287.1521	-1.87

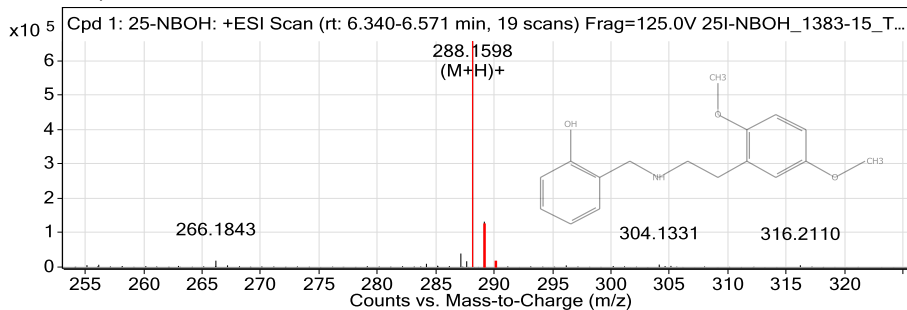
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



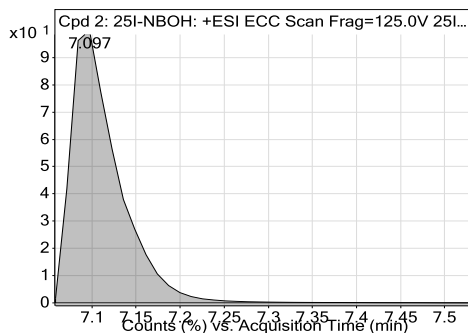
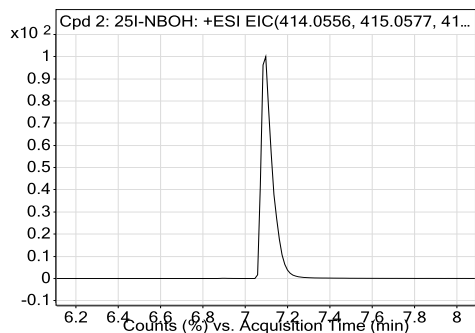
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
288.16	1	657421.75	C17 H21 N O3	(M+H)+
289.1629	1	128114.01	C17 H21 N O3	(M+H)+
290.1653	1	15583.09	C17 H21 N O3	(M+H)+
291.1697	1	1563.67	C17 H21 N O3	(M+H)+

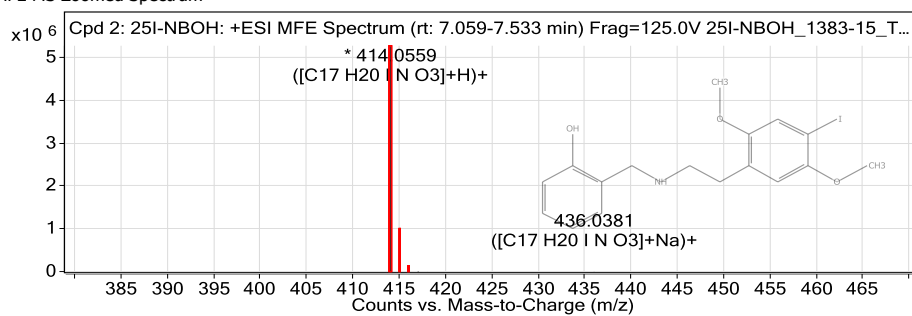
Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
25I-NBOH	414.0559	7.097	413.0487	7.06	C17 H20 I N O3	413.0488	0.23

Compound Chromatograms

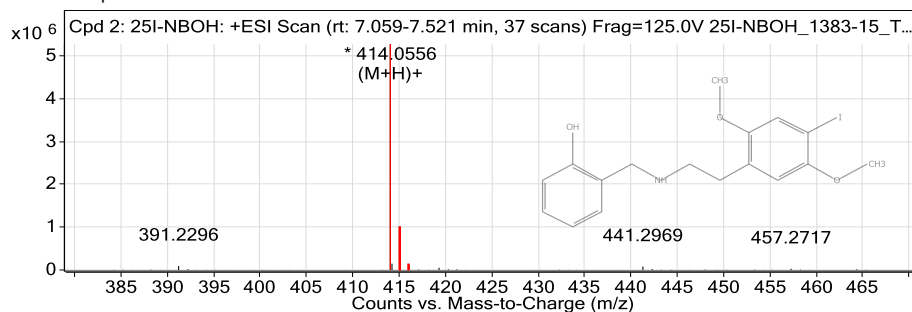
TOF REPORT



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

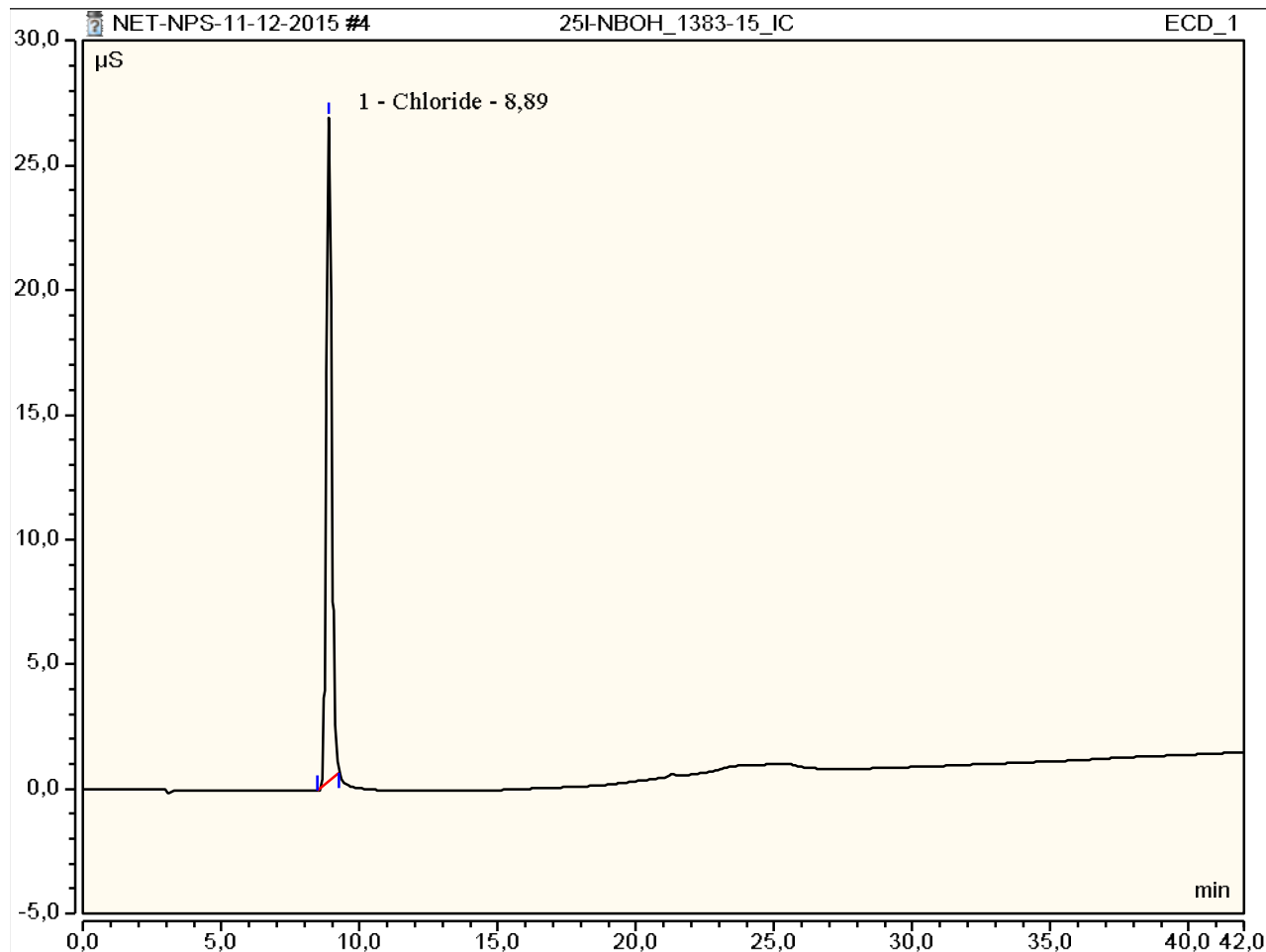
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
414.0559	1	5271175.5	C17 H20 I N O3	(M+H)+
415.0598	1	1013526.87	C17 H20 I N O3	(M+H)+
416.0619	1	112597.37	C17 H20 I N O3	(M+H)+
417.0642	1	9945.9	C17 H20 I N O3	(M+H)+
418.0659	1	498.25	C17 H20 I N O3	(M+H)+
436.0381	1	1672.89	C17 H20 I N O3	(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	25I-NBOH_1383-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	11-dec-2015 / 13:08	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	8,89	Chloride	BMB	5,97	26,60	n.a.
		TOTAL:		5,97	26,60	0,00

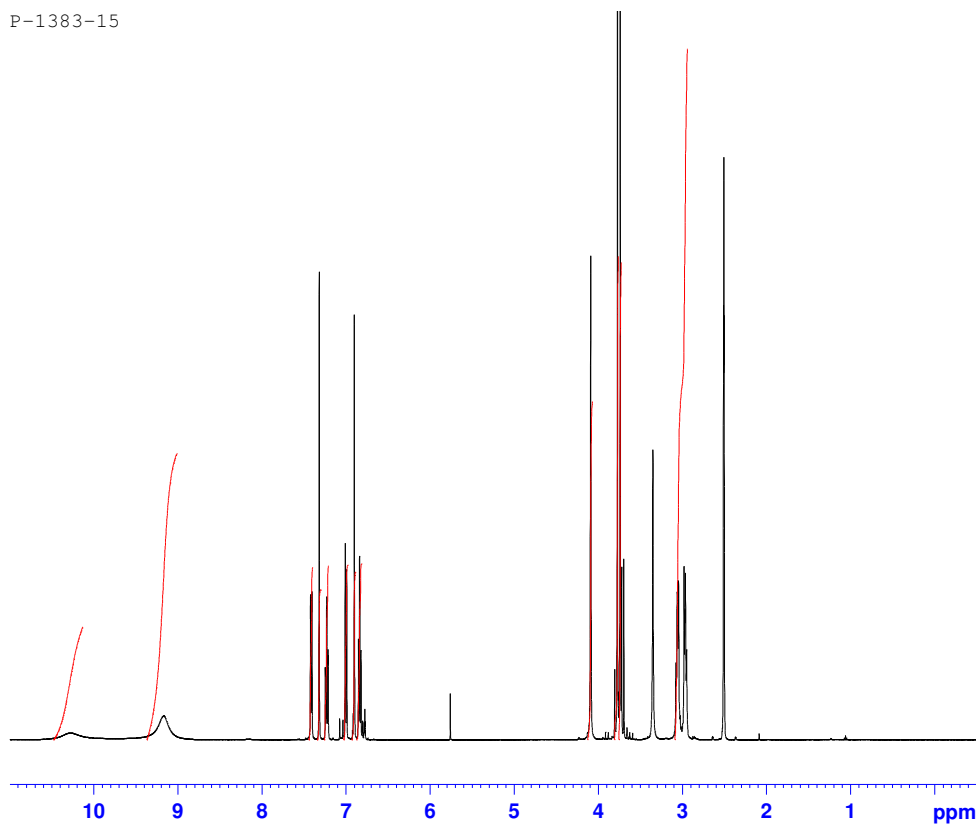




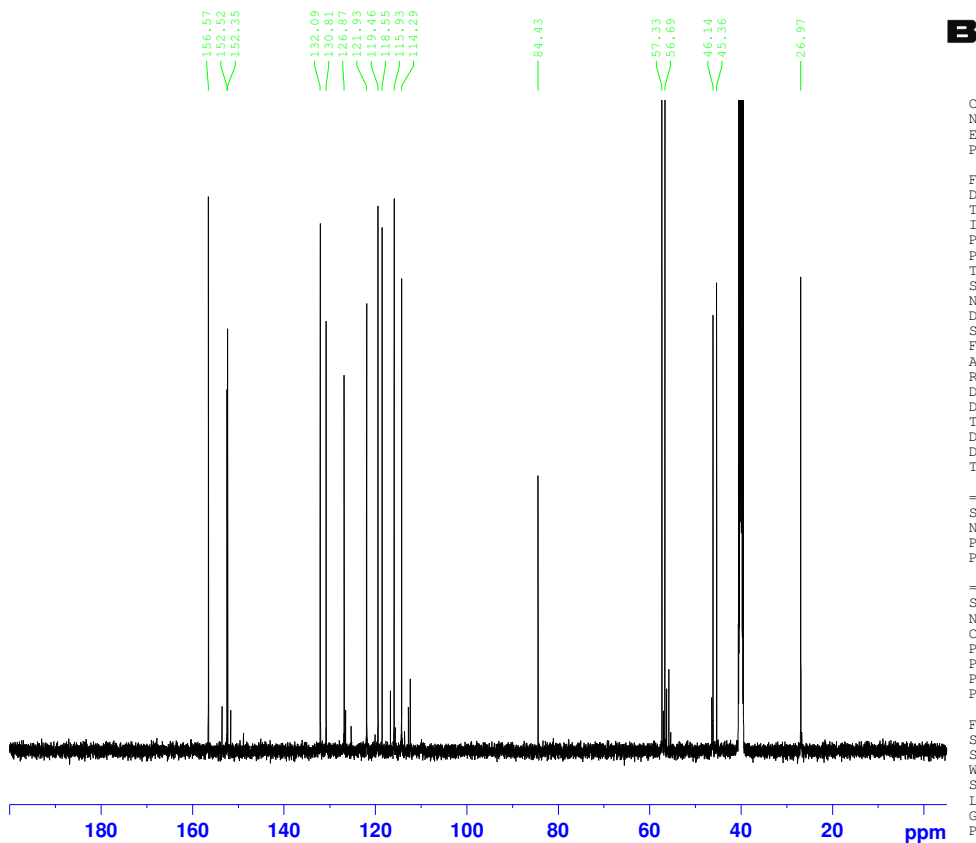
REPORT

Sample ID:	1383-15
Our notebook code:	P-1383-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	N-(2-hydroxybenzyl)-2-(4-iodo-2,5-dimethoxyphenyl)ethan-1-aminium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure according to NMR, there is an evidence of some minor impurities being present.
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	February 17, 2016

P-1383-15



P-1383-15



Current Data Parameters
NAME P-1383-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160217
Time 0.22
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 3.2768500 sec
RG 71.8
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 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME P-1383-15
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160217
Time 1.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
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 1.00000000 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 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40