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

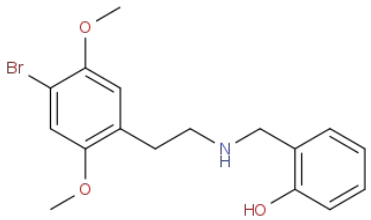
ANALYTICAL REPORT

25B-NBOH (C17H20BrNO3)

2-({[2-(4-bromo-2,5-dimethoxyphenyl)ethyl]amino}methyl)phenol

Remark – other NPS detected: none

Sample ID:	1697-16
Sample description:	powder
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	10/17/2016
Date of entry (M/D/Y) into NFL database:	12/23/2016
Report[1] 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-bromo-2,5-dimethoxyphenyl)ethyl]amino}methyl)phenol
Other names	2C-B-NBOH, NBOH-2C-B
Formula (per base form)	C17H20BrNO3
M_w (g/mol)	366,26
Salt form/anions detected	HCl
StdInChIKey (per base form)	RSUNJYKZRKIBNB-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	pure by HPLC-TOF and NMR; thermal decomposition can occour in GC

[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 (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

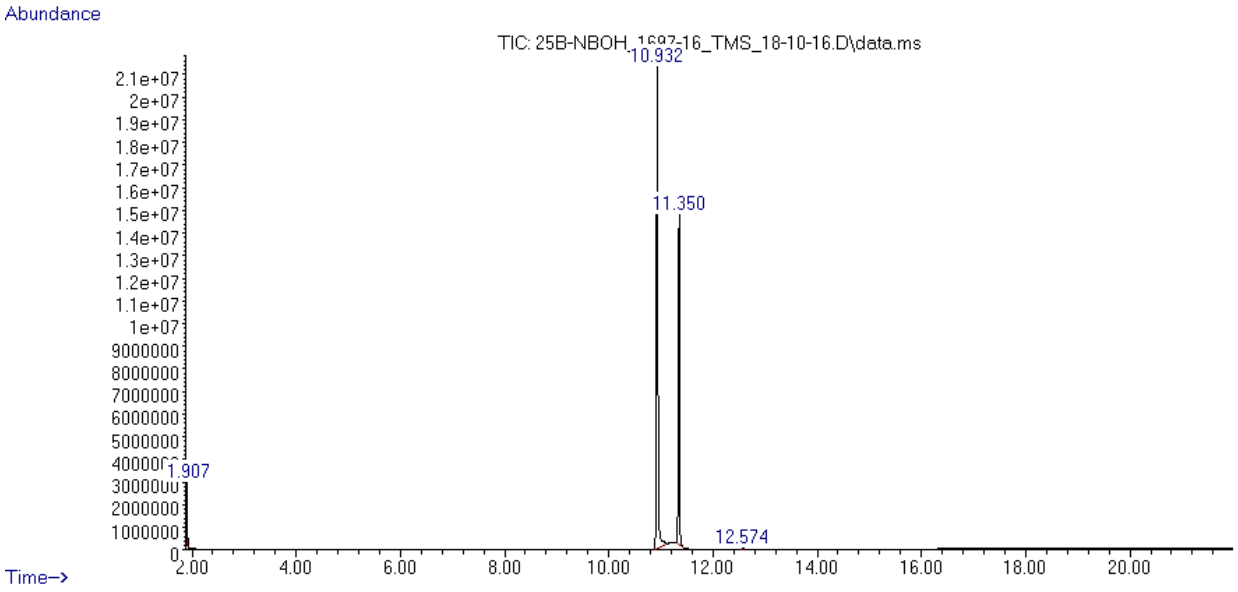
Supporting information

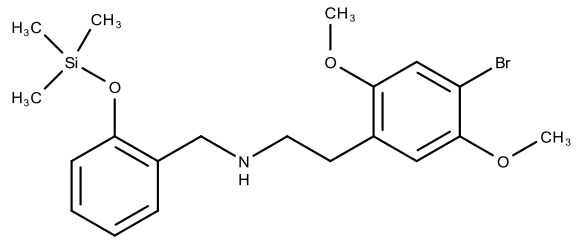
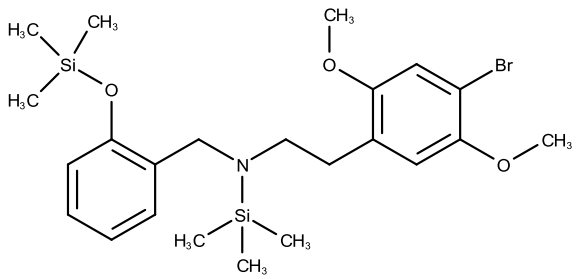
Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 11,35 BP(1): 179; BP(2): 280,BP(3) :73; sample was derivatized by MSTFA GC RT and fragmentation data refer to di-TMS derivative
HPLC-TOF	+	Exact mass (theoretical): 365,063; measured value Δppm:-0,98; formula:C17H20BrNO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	-	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

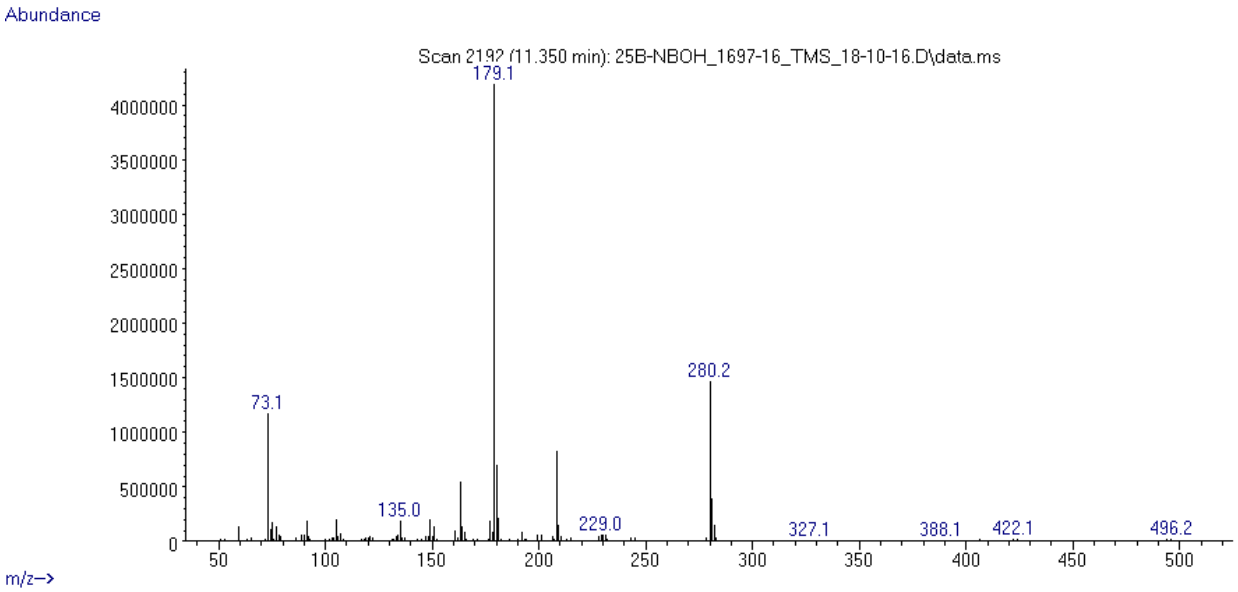
ANALYTICAL RESULTS

GC- chromatogram of derivatized (by MSTFA) compound: RT=0.932 mono TMS derivative and RT=11.35 di-TMS derivative

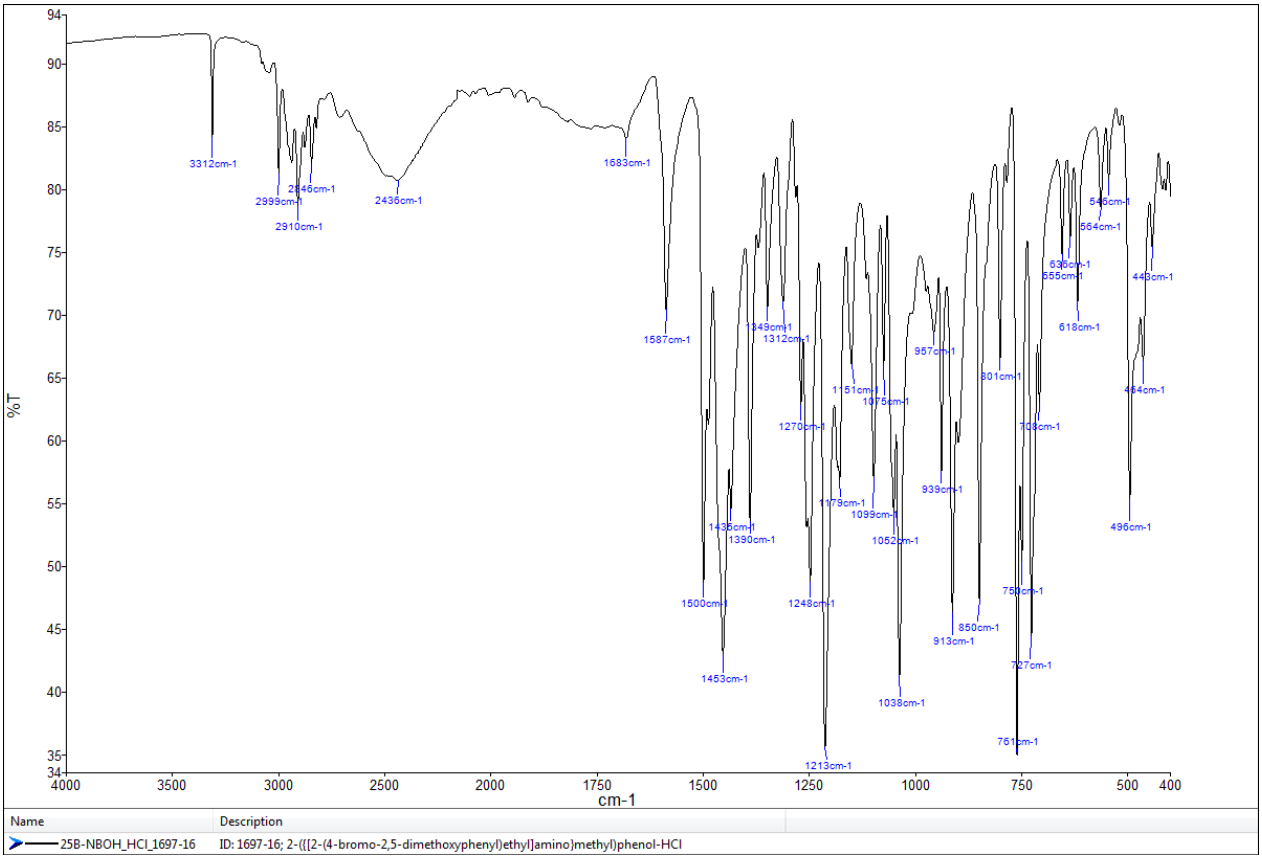


O-TMS derivative	N, O- di-TMS derivative
	

MS spectrum of di-TMS derivative



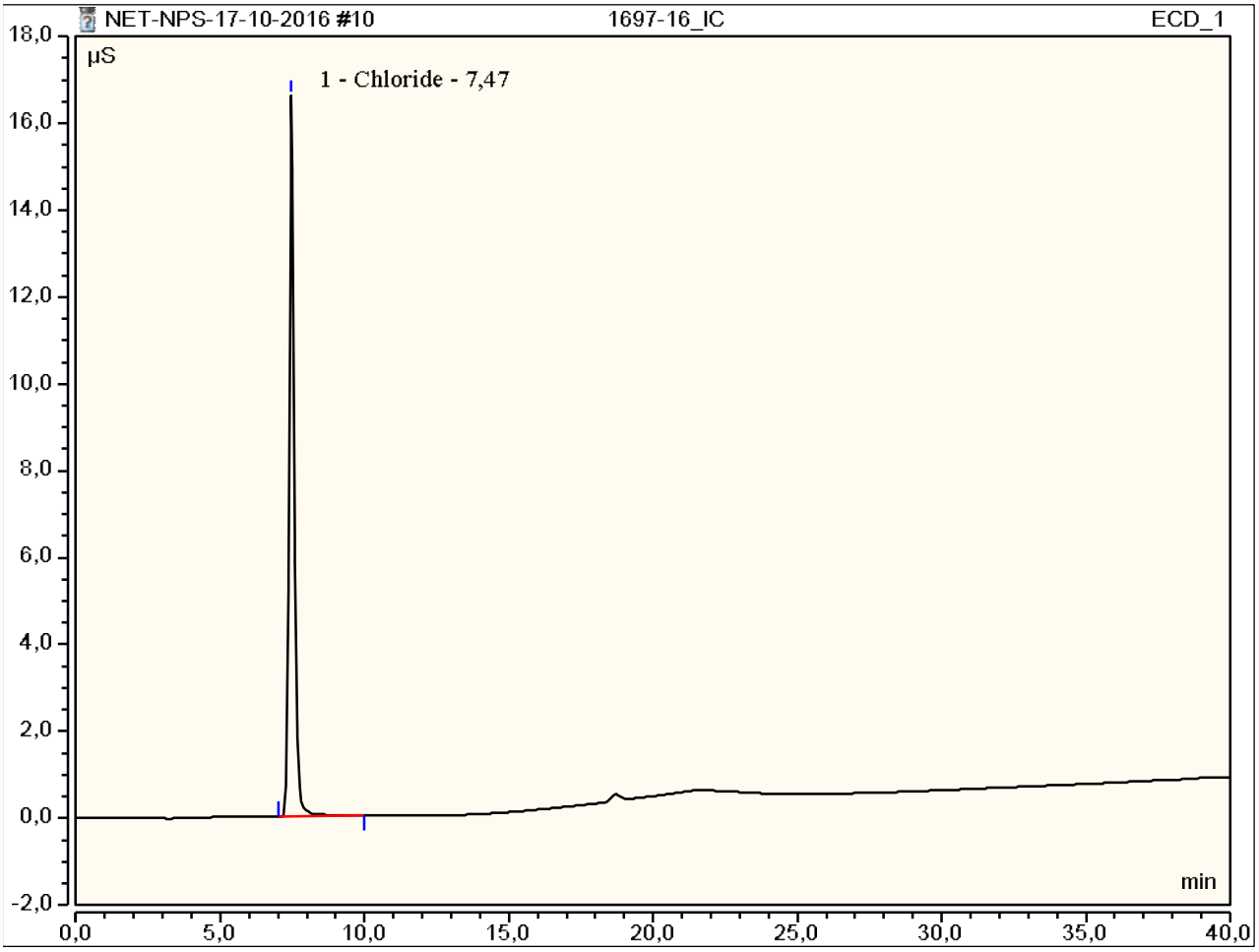
FTIR-ATR - direct measurement (sample as received)



Peak Integration Report

Sample Name:	**1697-16_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**17-okt-2016 / 21:17**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	7,47	**Chloride**	BMB	3,51	16,61	n.a.
		TOTAL:		3,51	16,61	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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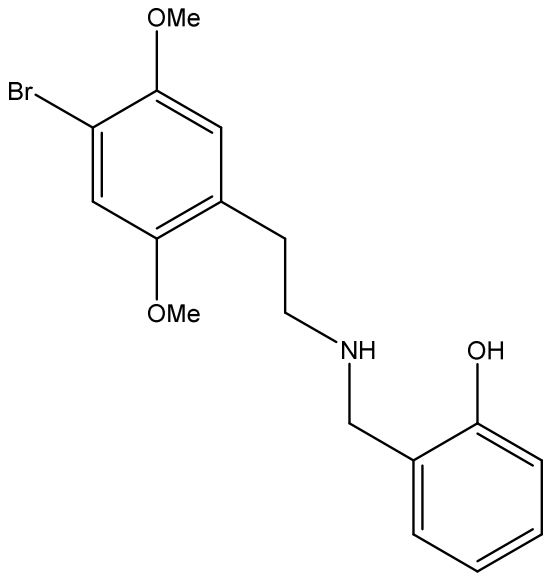
REPORT

Sample ID:	**1697-16**
Our notebook code:	P-1697-16
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:	2-(((4-bromo-2,5-dimethoxyphenethyl)amino)methyl)phenol
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure as evident by NMR.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 22, 2016

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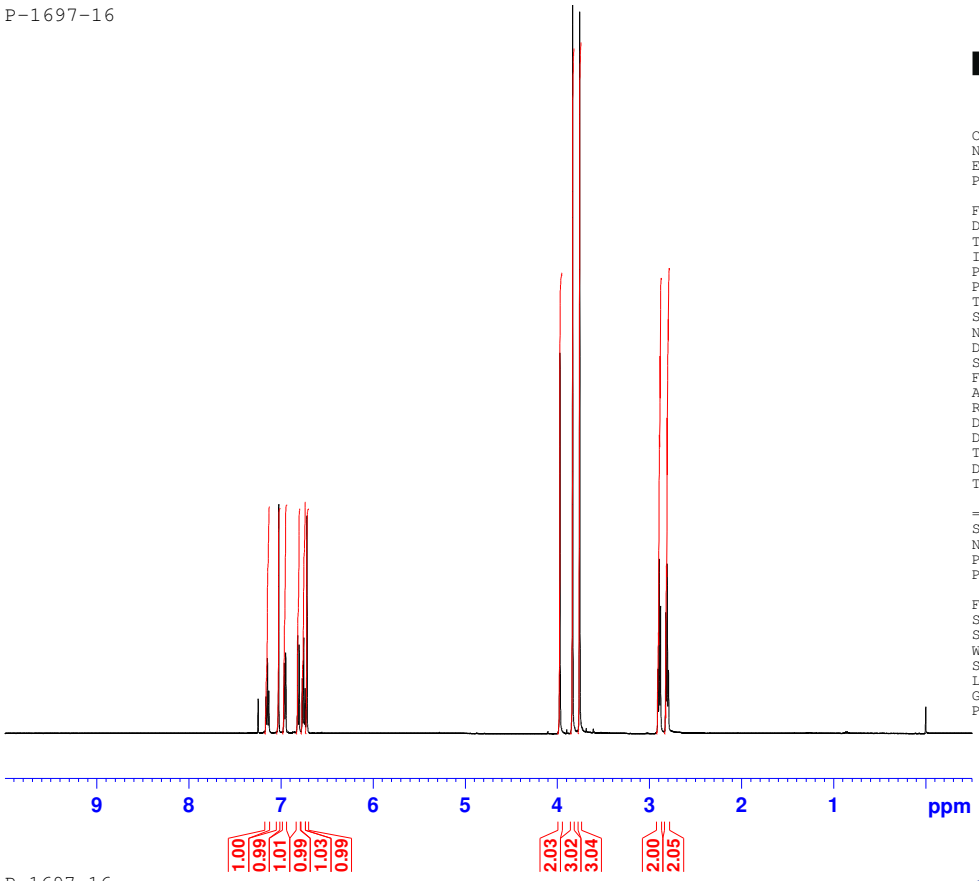


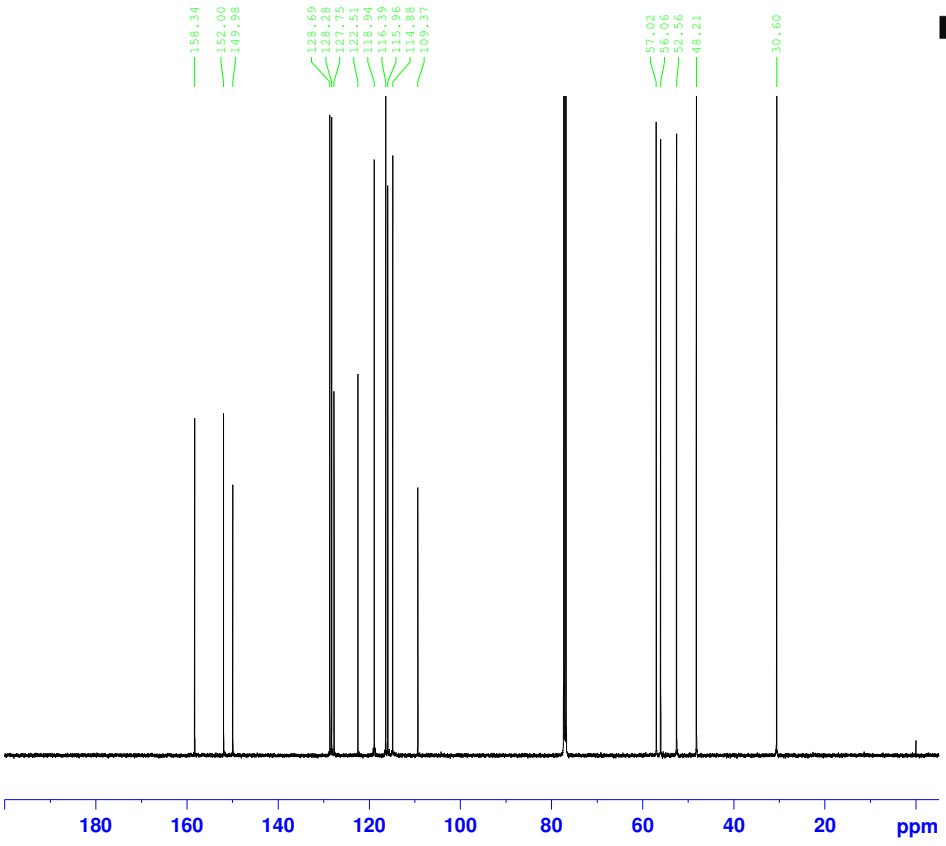

```
Current Data Parameters
NAME          P-1697-16
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20161212
Time              20.54
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDC13
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                298.4 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                 1H
P1                8.60 usec
PLW1        26.00000000 W

F2 - Processing parameters
SI                65536
SF           500.1300175 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

P-1697-16

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Current Data Parameters
NAME          P-1697-16
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20161212
Time              23.44
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 3072
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                299.3 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           125.7577885 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```