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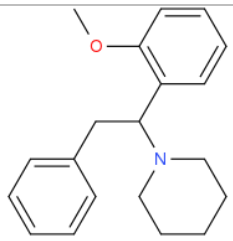
ANALYTICAL REPORT

2-MeO-diphenidine (C20H25NO)

1-(1-(2-methoxyphenyl)-2-phenylethyl)piperidine

Remark – other NPS detected: none

Sample ID:	1195-15
Sample description:	powder - white
Sample type:	test purchase 7/22/20157/22/2015
Comments[1]:	
Date of entry into NFL database: http://www.policija.si/apps/nfl_response_web/seznam.php	10/14/2015

Substance identified-structure[2] (base form)	
Systematic name	1-(1-(2-methoxyphenyl)-2-phenylethyl)piperidine
Other names	
Formula (per base form)	C20H25NO
M_w (g/mol)	295,42
Salt form	HCl
StdInChIKey	QXXCUXIRBHSITD-UHFFFAOYSA-N
Compound Class	Others
Other NPS detected	none
Add.info (purity..)	NMR: sample contains 7% to 8 % MeoH, GC and HPLC pure

[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 ^{0}C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 293 ^{0}C at a rate of 18 ^{0}C/min, hold for 6.1 min, than heating at 50 ^{0}C/min up to 325 ^{0}C and finally 2.8 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 (40) to 550 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 (40) to 550 amu.
IR (condesed phase): IR scan range 4000 to 650, resolution 4 cm^{-1} .

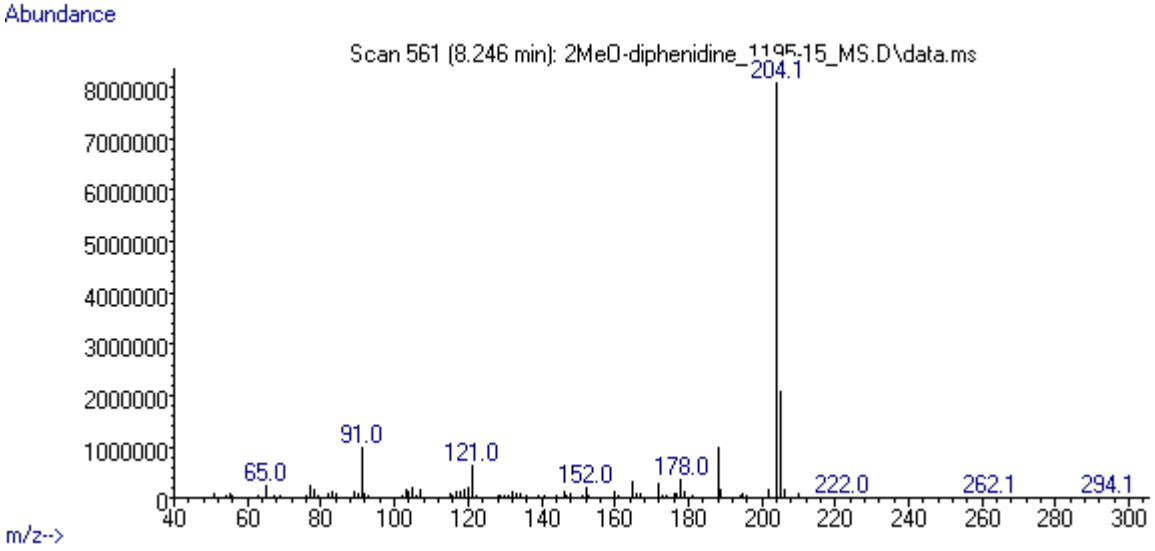
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
other	not tested

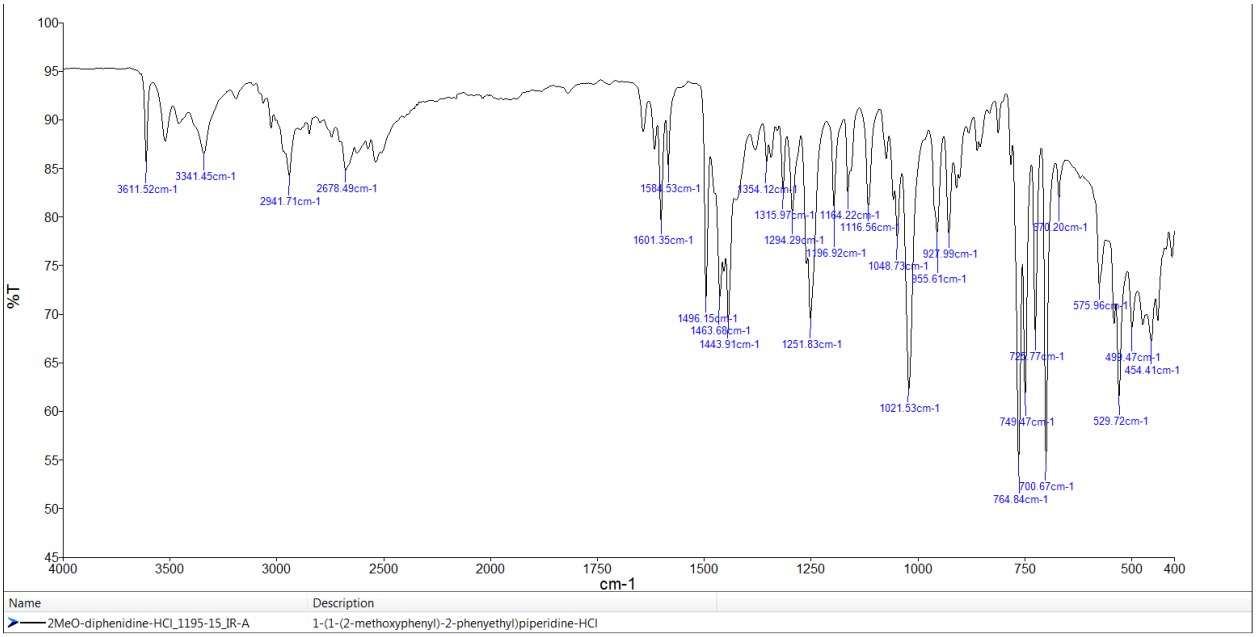
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8,25 BP(1): 204; BP(2): 188,BP(3) :91,
HPLC-TOF	+	Exact mass (theoretical): 295,1936; measured value Δppm:-0,22; formula:C20H25NO
FTIR-ATR	+	direct measurement (note sample contains MeOH)
FTIR (condensed phase) always as base form	+	
NMR	+	
validation		
other		

ANALYTICAL RESULTS

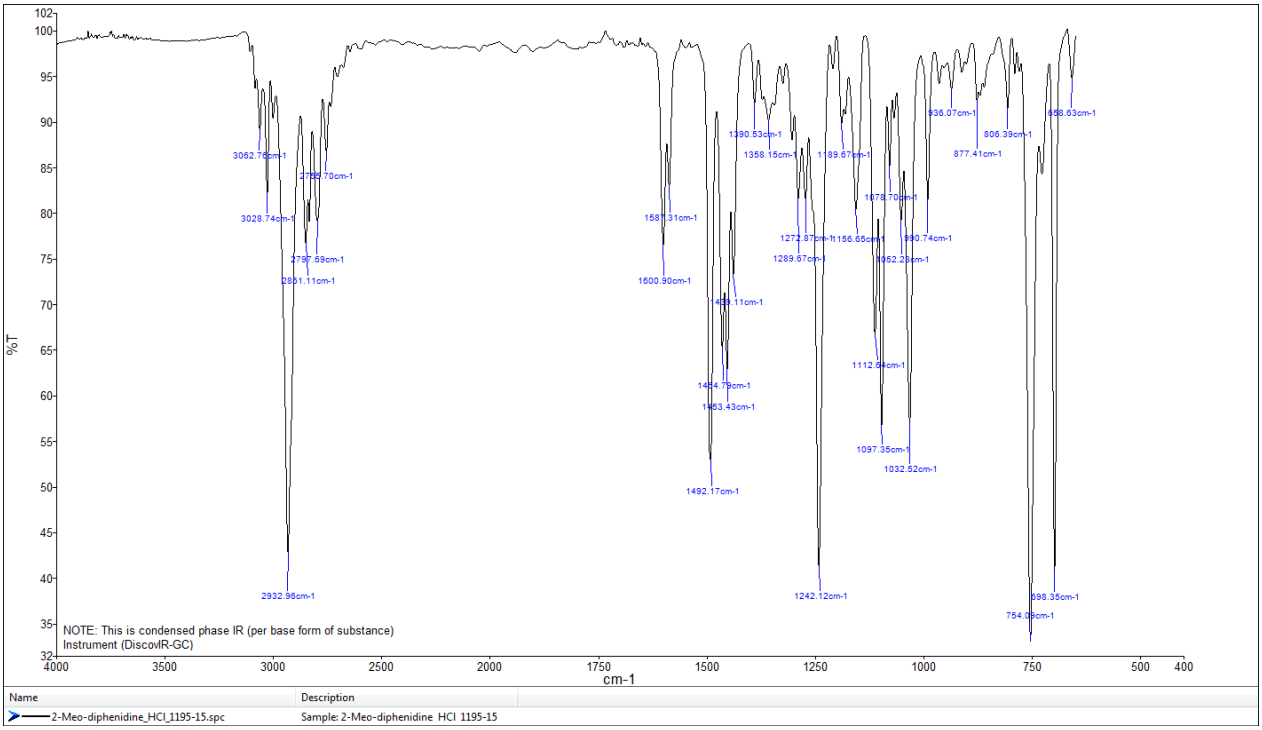
MS (EI)



FTIR-ATR - direct measurement (note! HCl form & sample contains 7% to 8% MeOH)



IR (condensed phase)



Target Compound Screening Report

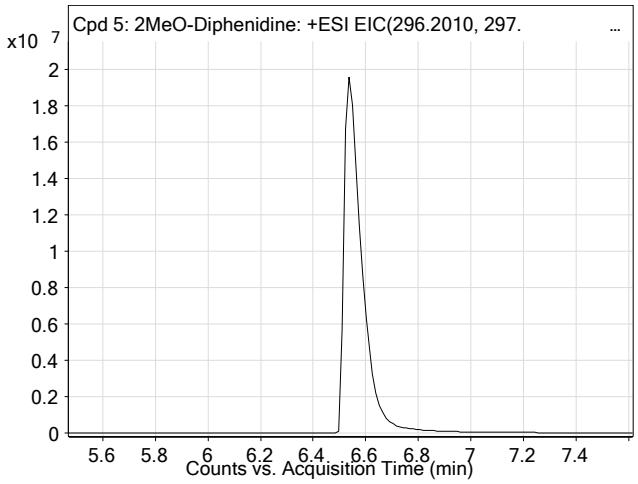
Data File	2MeO-diphenidine_1195-15_TOF.d	**Sample Name**	2MeO-diphenidine
Sample Type	Sample	**Position**	P1-D5
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	7/27/2015 11:23:48 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

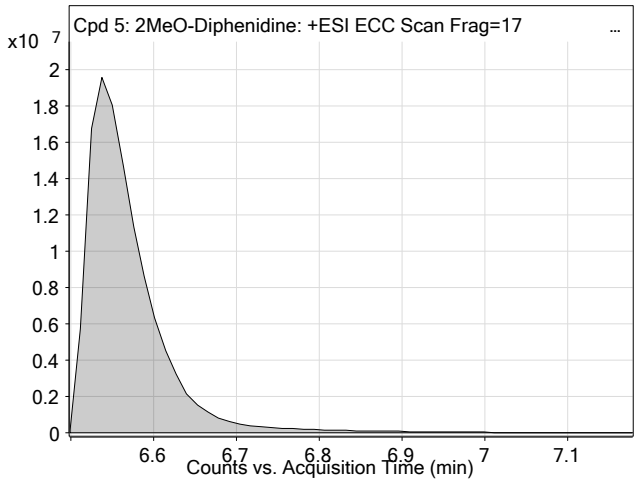
Compound Table

Label	Tgt Name	Obs. RT	Obs. Mass
Cpd 5: 2MeO-Diphenidine	2MeO-Diphenidine	6.545	295.1937

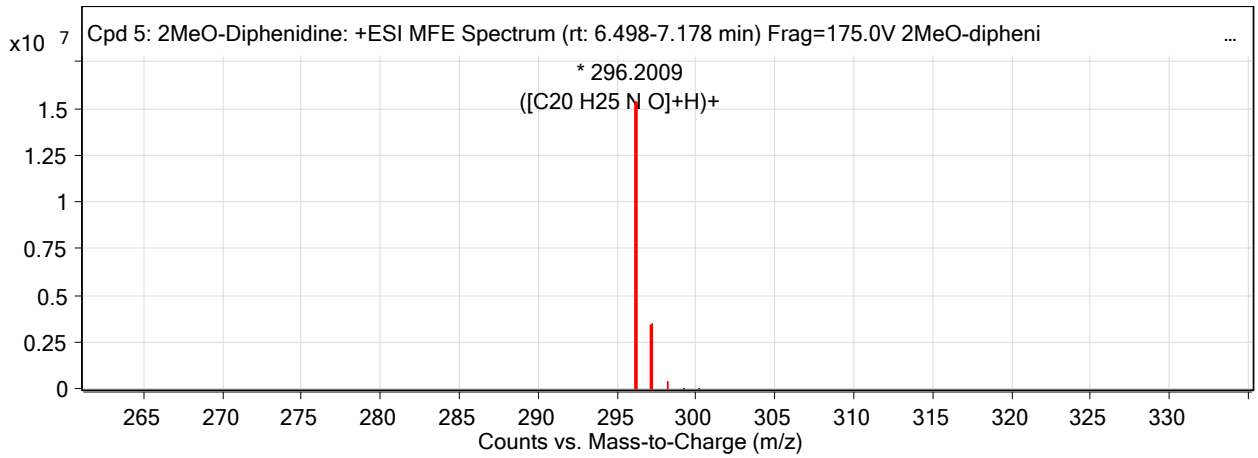
Name	Obs. *m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
2MeO-Diphenidine	296.2009	6.545	295.1937	6.545	C20 H25 N O	295.1936	-0.22	Find by Molecular Feature

Compound Chromatograms





MFE MS Zoomed Spectrum

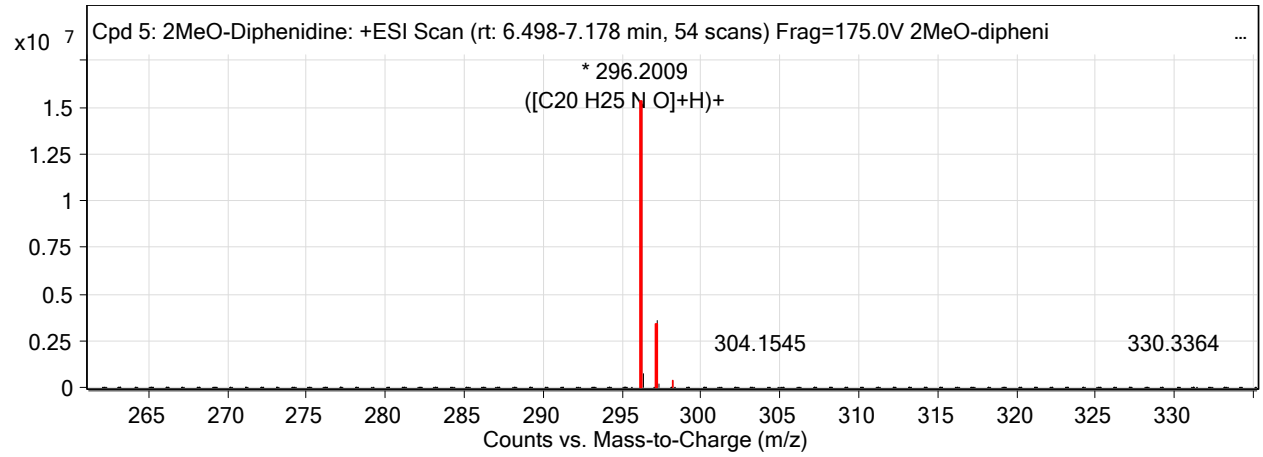


MS Spectrum Peak List

Obs. *m/z*	Charge	Abund	Formula	Ion/Isotope
296.2009	1	15338817	C20 H25 N O	(M+H)+
297.2044	1	3492598.58	C20 H25 N O	(M+H)+
298.2077	1	364591.07	C20 H25 N O	(M+H)+
299.2103	1	29507.91	C20 H25 N O	(M+H)+
300.2126	1	2991.47	C20 H25 N O	(M+H)+

MS Zoomed Spectrum

Agilent Technologies



--- End Of Report ---

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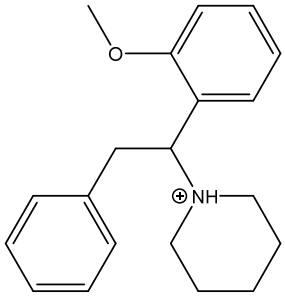
REPORT

Sample ID:	**1195-15**
Our notebook code:	P-1195-15
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 with chemical name:	 1-(1-(2-methoxyphenyl)-2-phenylethyl)piperidin-1-ium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is not pure by NMR, containing approx. 7–8% of methanol (signals in ^{1}H NMR at 3.49 and 1.30 and in ^{13}C NMR at 50.77), degrading the quality of spectra.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	October 12, 2015

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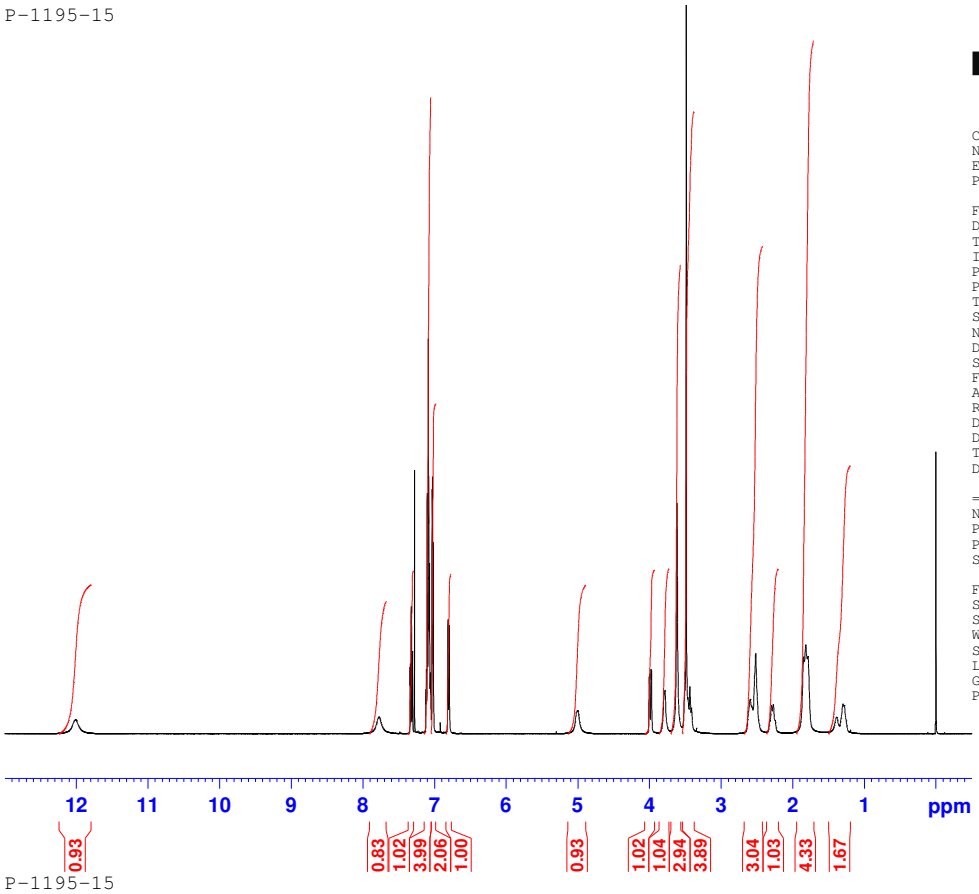
P-1195-15



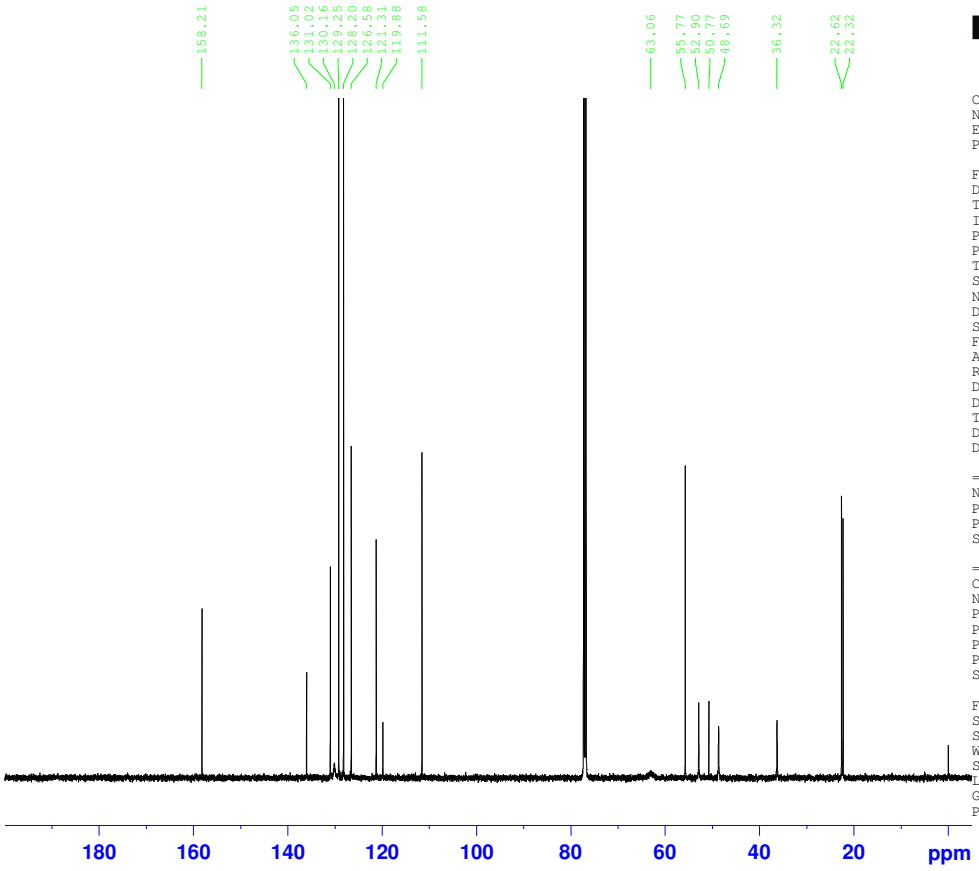
Current Data Parameters
NAME p-1195-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150823
Time 12.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 90.5
DW 48.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec

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

F2 - Processing parameters
SI 65536
SF 500.1300034 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1195-15



Current Data Parameters
NAME p-1195-15
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150823
Time 14.02
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 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 122.00000000 W
SFO1 125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.32179001 W
PLW13 0.20595001 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40