EUROPEAN PROJECT RESPONSE to challenges in forensic drugs analyses

Co-funded by the Prevention of and Fight against Crime Programme of the European Union

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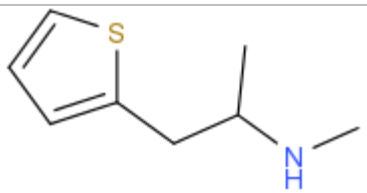
ANALYTICAL REPORT

MPA_Methiopropamine (C8H13NS)

N-Methyl-1-(thiophen-2-yl)propan-2-amine

Remark – other NPS detected: none

Sample ID:	1194-15
Sample description:	powder - white
Sample type:	test purchase (RESPONSE -purchasing)
Comments[1]:	
Date of entry:	9/30/2015

Substance identified-structure[2] (base form)	
Systematic name	N-Methyl-1-(thiophen-2-yl)propan-2-amine
Other names	MPA
Formula (per base form)	C8H13NS
M_w (g/mol)	155,26
Salt form	HCl
StdInChIKey	HPHUWHKFQXTZPS-UHFFFAOYSA-N
Compound Class	Arylalkylamines
Other NPS detected	none
Add.info (purity..)	pure by NMR, GC, HPLC

[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 4 cm^{-1}

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 700, resolution 4 cm^{-1} .

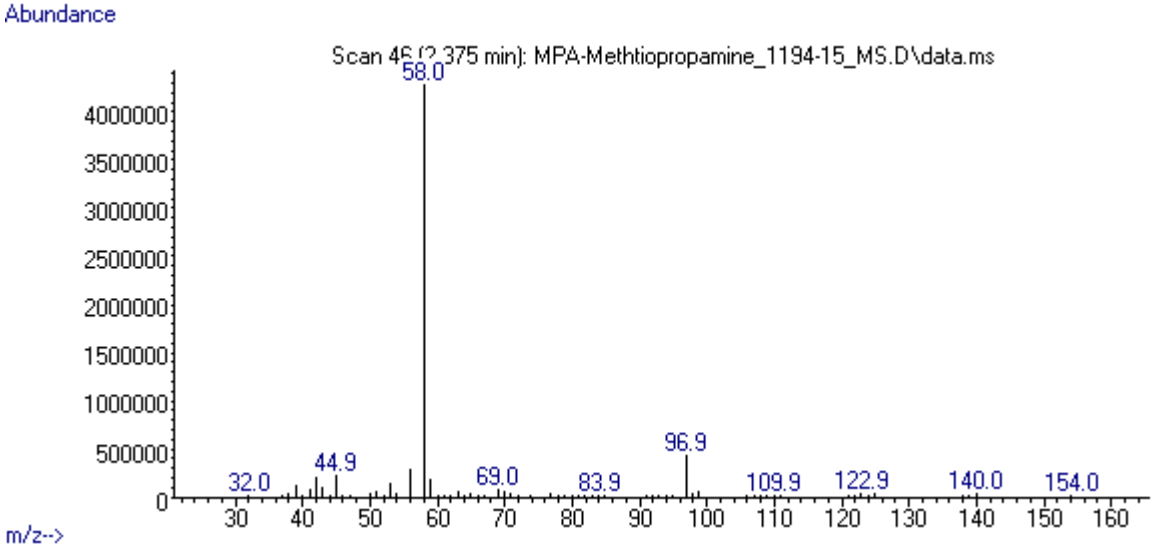
Supporting information

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

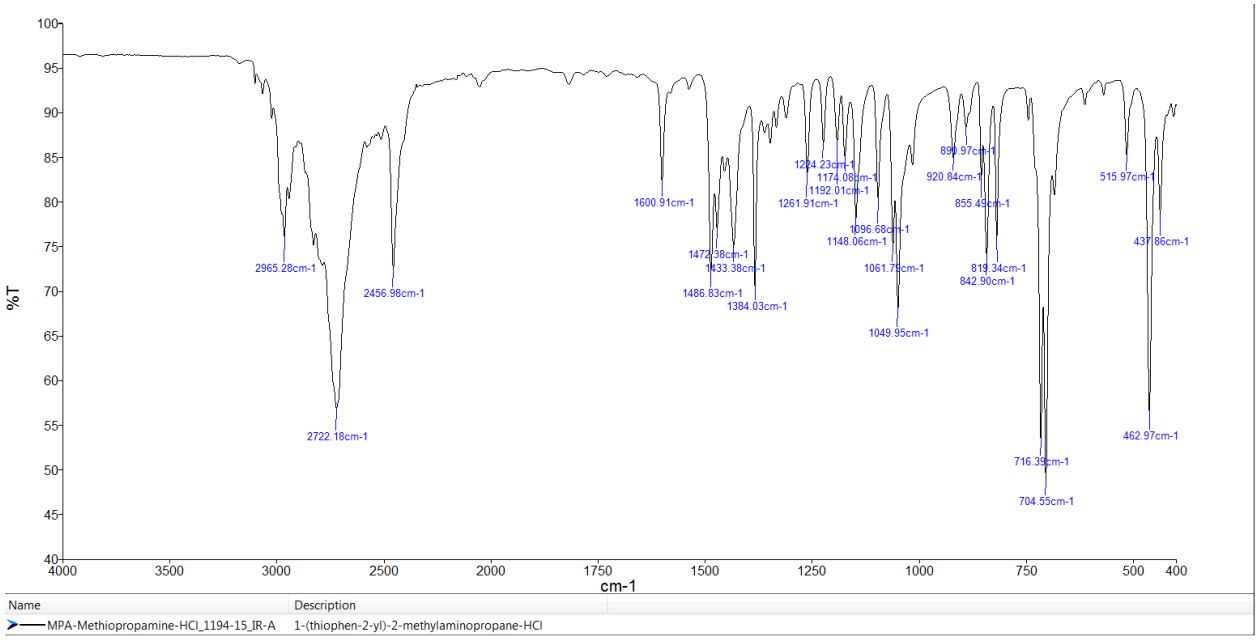
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 2,37 BP(1): 58; BP(2): 97,BP(3) :56,
HPLC-TOF	+	Exact mass (theoretical): 155,0769; measured value Δppm:-2,23; formula:C8H13NS
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
HPLC-TOF		Exact mass (theoretical): 155,0769; measured value Δppm:-2,23; formula:C8H13NS
NMR	+	
validation		IR-ATR consistent by SWGDRUG lib entry (qM>0.99)
other		

ANALYTICAL RESULTS

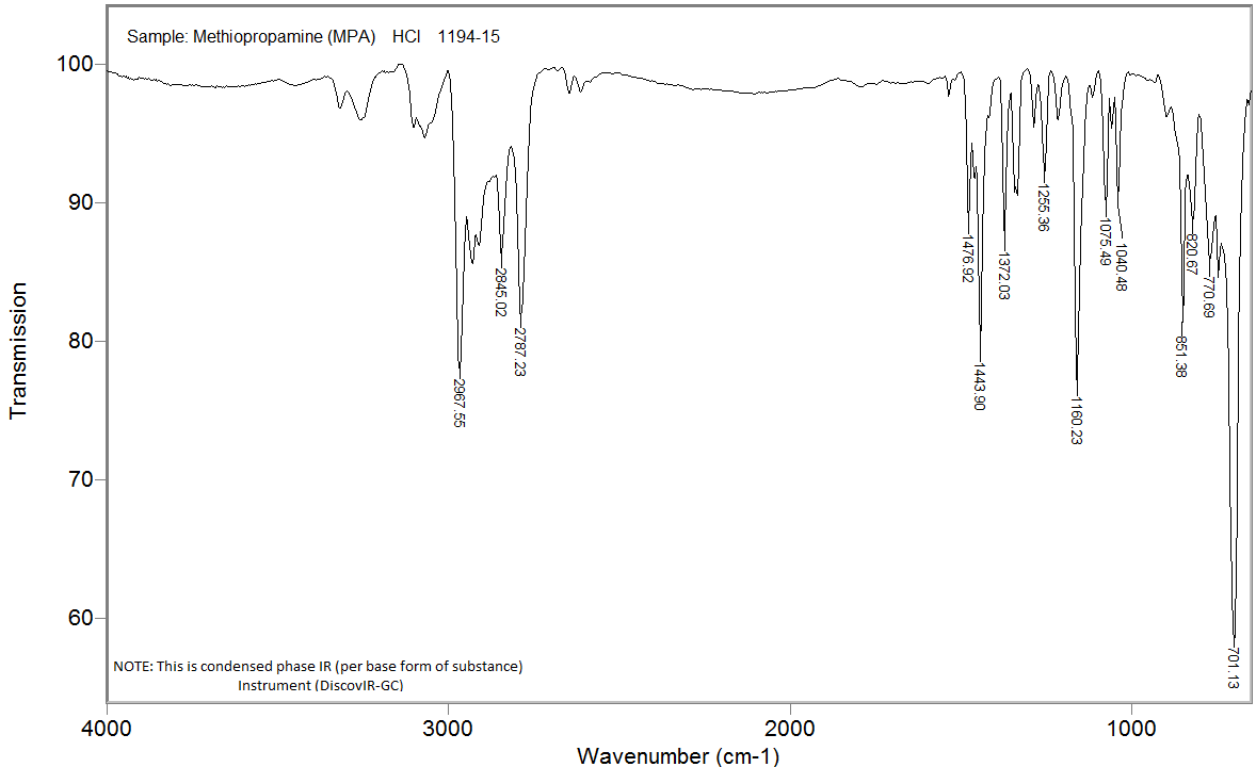
MS (EI)



FTIR-ATR



IR (condensed phase)



Target Compound Screening Report

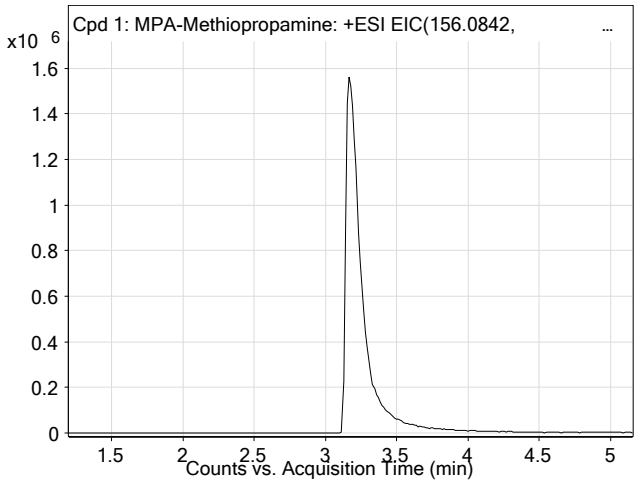
Data File	MPA-Methiopropamine_1194-15_TOF.d	**Sample Name**	MPA-Methiopropamine
Sample Type	Sample	**Position**	P1-D4
Instrument Name	6230B TOF LC-MS	**User Name**	
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	7/27/2015 11:07:58 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

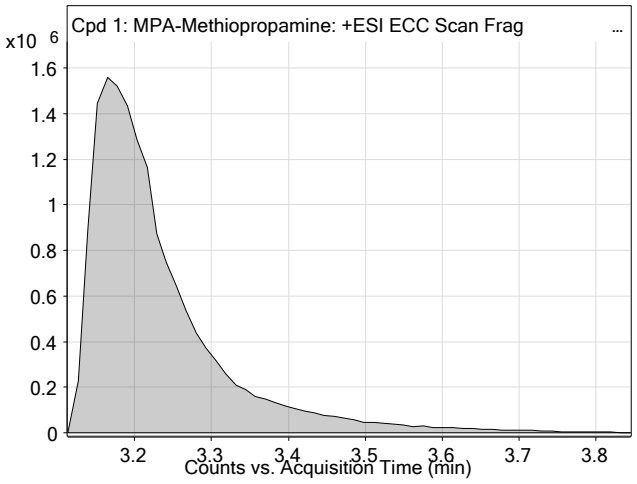
Compound Table

Label	Tgt Name	Obs. RT	Obs. Mass
Cpd 1: MPA-Methiopropamine	MPA-Methiopropamine	3.182	155.0772

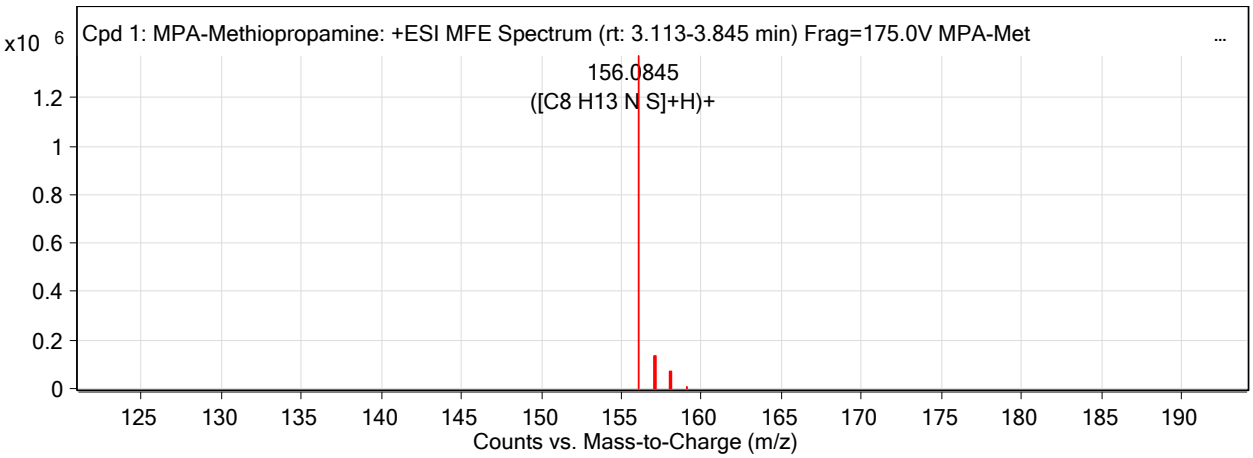
Name	Obs. *m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
MPA-Methiopropamine	156.0845	3.182	155.0772	3.182	C8 H13 N S	155.0769	-2.23	Find by Molecular Feature

Compound Chromatograms





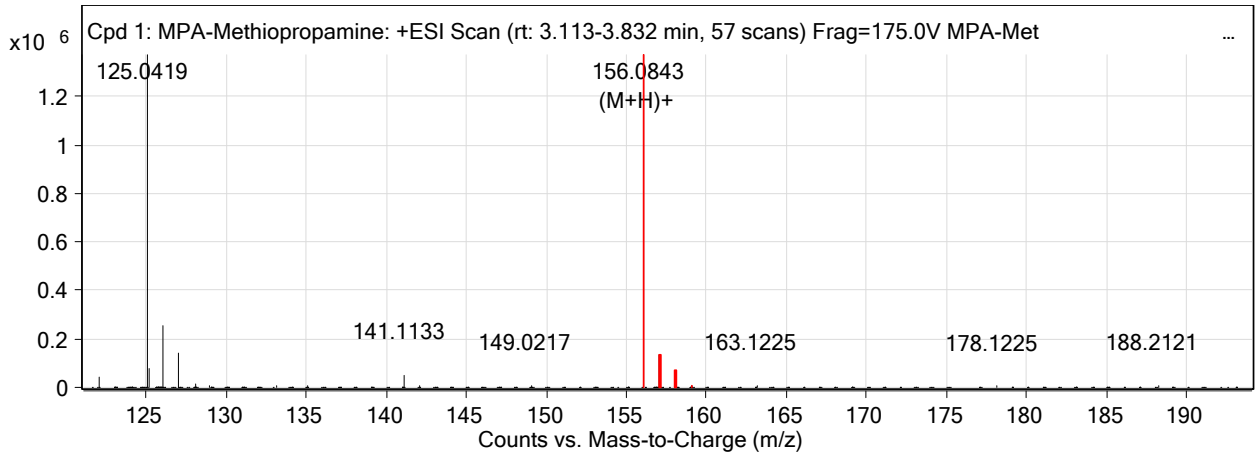
MFE MS Zoomed Spectrum



MS Spectrum Peak List

Obs. *m/z*	Charge	Abund	Formula	Ion/Isotope
156.0845	1	1372734.88	C8 H13 N S	(M+H)+
157.0873	1	131786.03	C8 H13 N S	(M+H)+
158.0808	1	61929.11	C8 H13 N S	(M+H)+
159.0834	1	5460.93	C8 H13 N S	(M+H)+

MS Zoomed Spectrum



--- End Of Report ---

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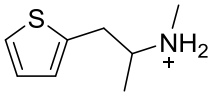
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1194-15**
Our notebook code:	P-1194-15
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:	 *N*-methyl-1-(thiophen-2-yl)propan-2-aminium ion
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	September 28, 2015

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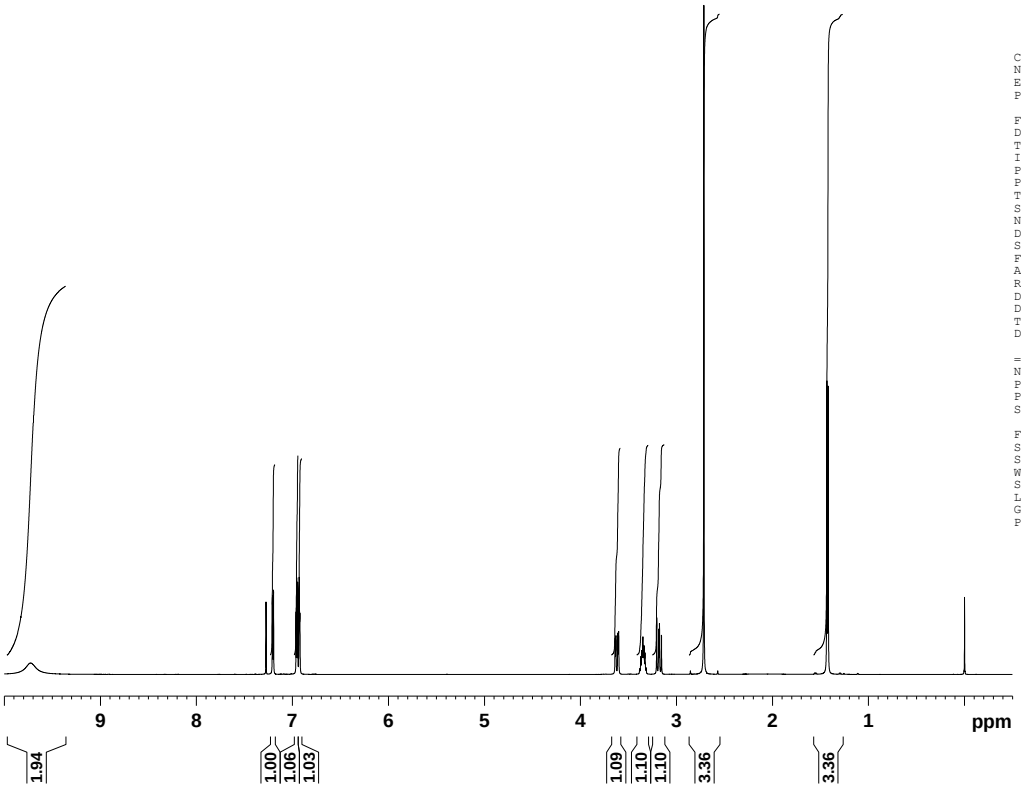
P-1194-15



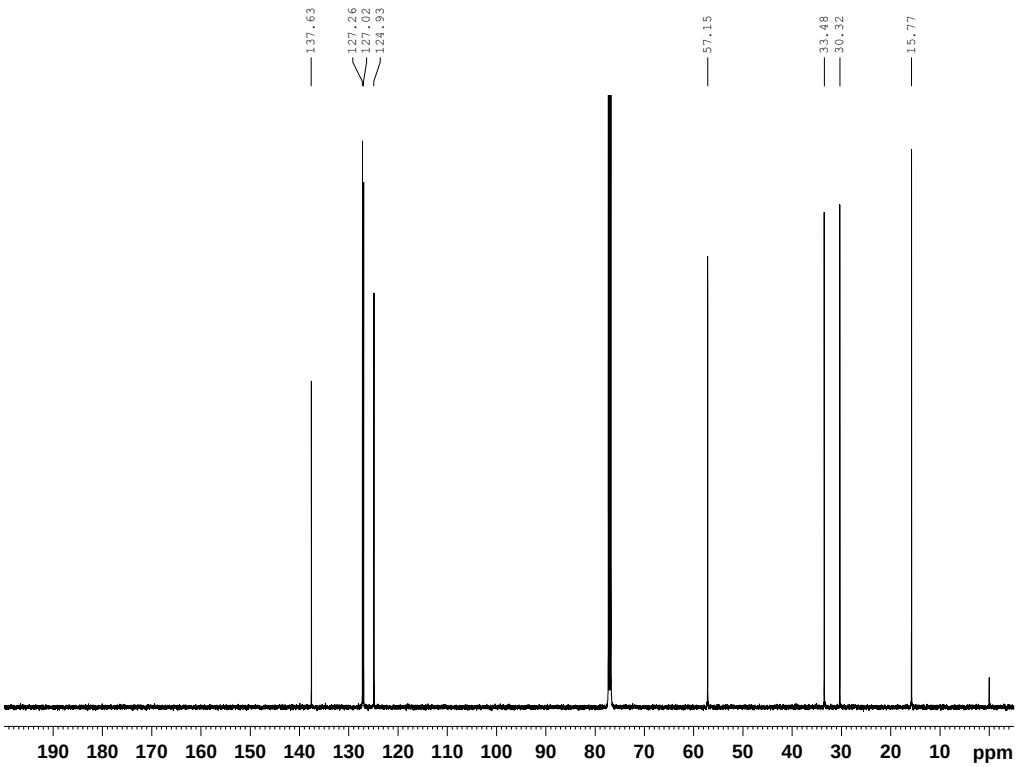

```
Current Data Parameters
NAME                  P-1194-15
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150822
Time                       1.24
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
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                 500.1300059 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```

P-1194-15

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Current Data Parameters
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EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150822
Time                       3.59
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        4096
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                 125.7577906 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```