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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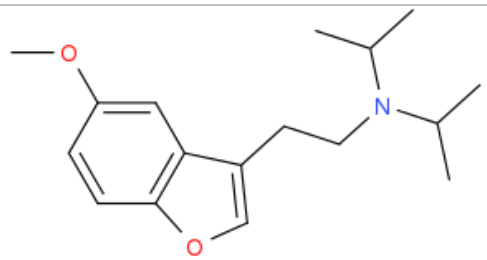
ANALYTICAL REPORT[1]

5-MeO-DiBF (C17H25NO2)

[2-(5-methoxy-1-benzofuran-3-yl)ethyl]bis(propan-2-yl)amine

Remark – other NPS detected: none

Sample ID:	1353-15
Sample description:	crystalinic - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	11/13/2015
Date of entry (M/D/Y) into NFL database:	11/25/2015
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-(5-methoxy-1-benzofuran-3-yl)ethyl]bis(propan-2-yl)amine
Other names	
Formula (per base form)	C17H25NO2
M_w (g/mol)	275.39
Salt form	HCl
StdInChIKey	NBFMSQBTYHYVKP-UHFFFAOYSA-N
Compound Class	Arylalkylamines
Other NPS detected	none
Add.info (purity..)	pure by GC-MS, HPLC-TOFand 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] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Report updates

date	comments (explanation)
19/12/2015	NMR added

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}.

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

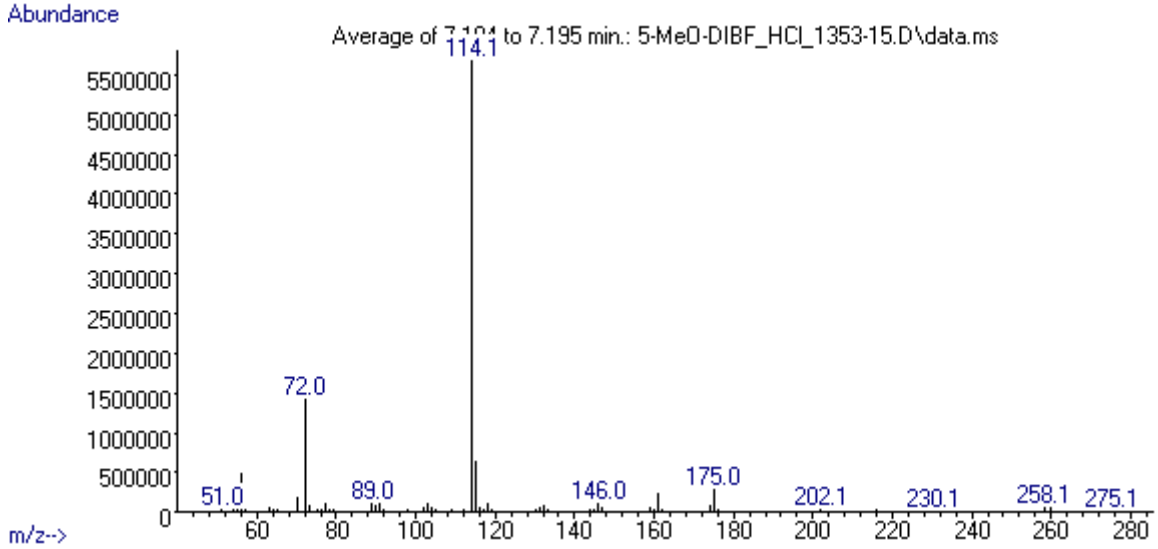
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	soluble

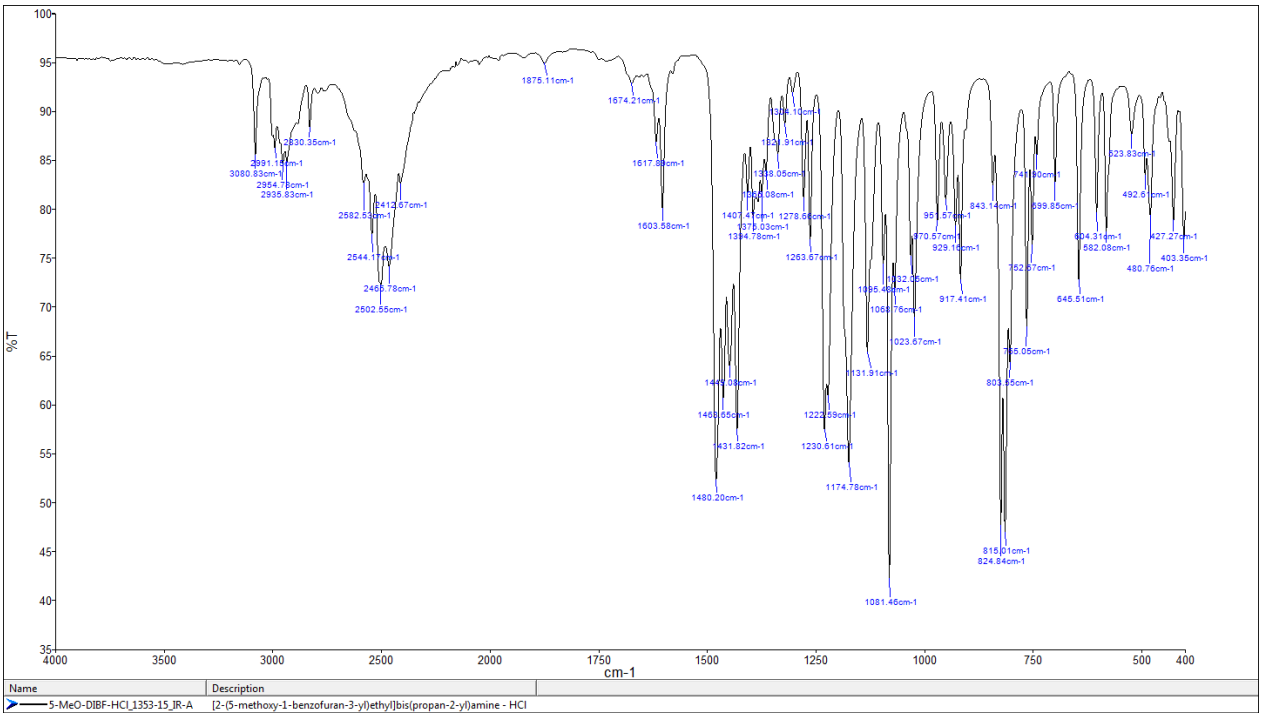
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 7.21 BP(1): 114; BP(2): 72,BP(3) :115,
HPLC-TOF	+	Exact mass (theoretical): 275.1885; measured value Δppm:-0.18; formula: C17H25NO2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form		
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

MS (EI)



FTIR-ATR - direct measurement (sample as received)

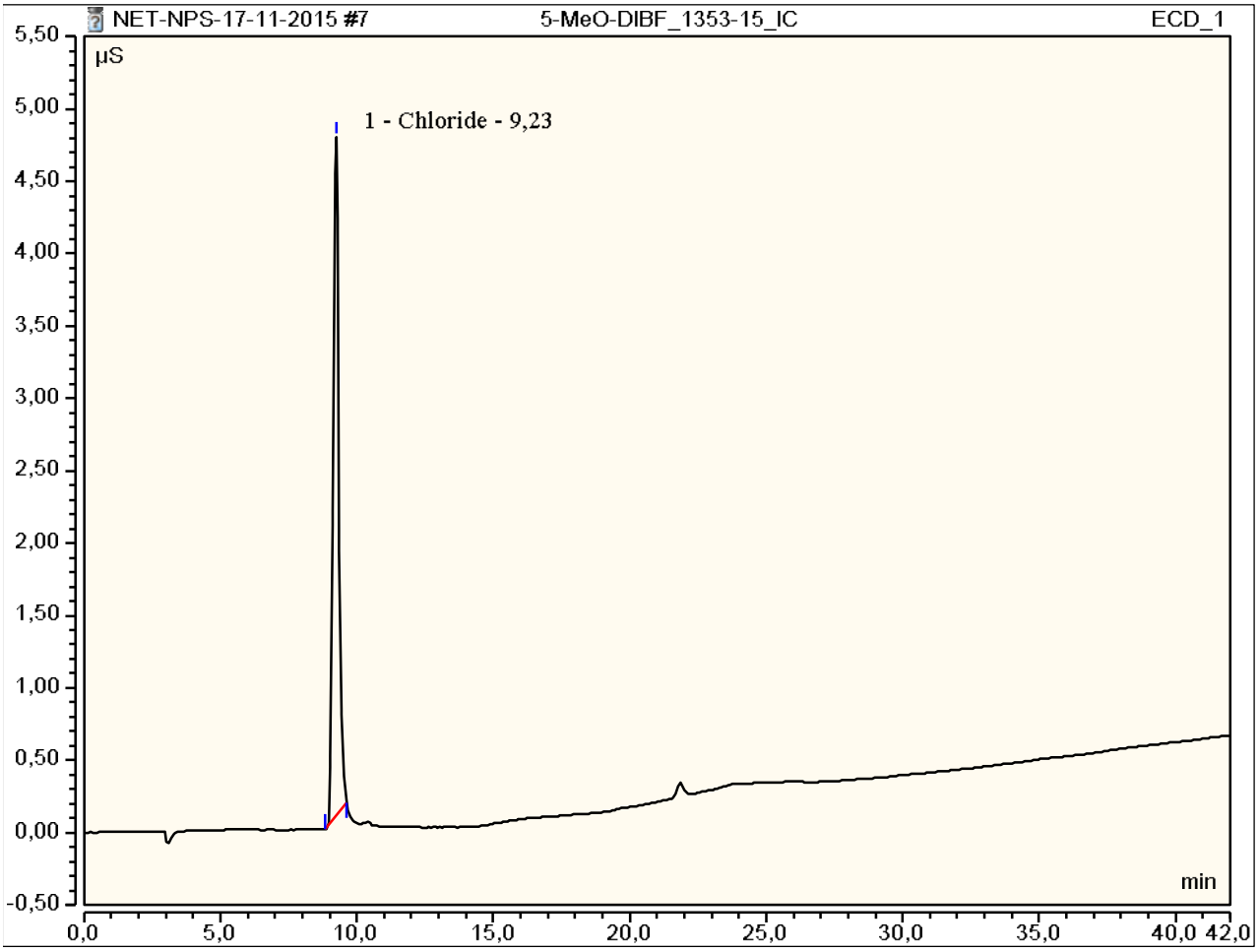


IR (condensed phase – after chromatographic separation)

Peak Integration Report

Sample Name:	**5-MeO-DIBF_1353-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**17-nov-2015 / 14:39**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,23	**Chloride**	BMB	1,11	4,69	n.a.
		TOTAL:		1,11	4,69	0,00



TOF REPORT

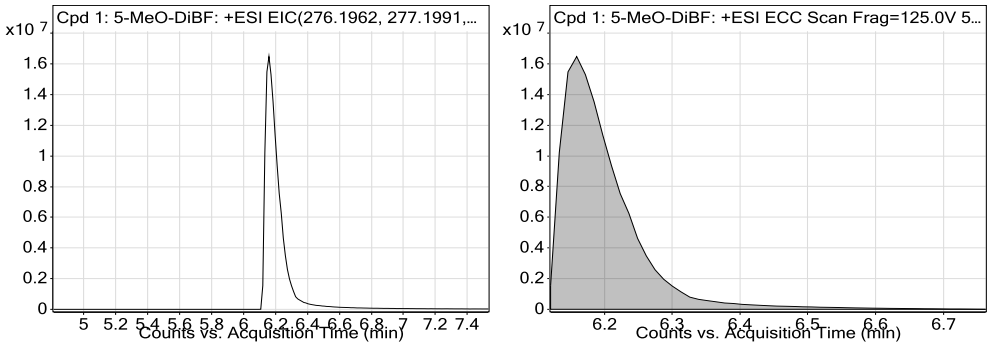
Data File	5-MeO-DIBF_1353-15_TOF.d	**Sample Name**	ID_1353-15
Sample Type	Sample	**Position**	P1-A8
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-17112015-XDB-C18-ESI-poz.m	**Acquired Time**	11/17/2015 10:43:30 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

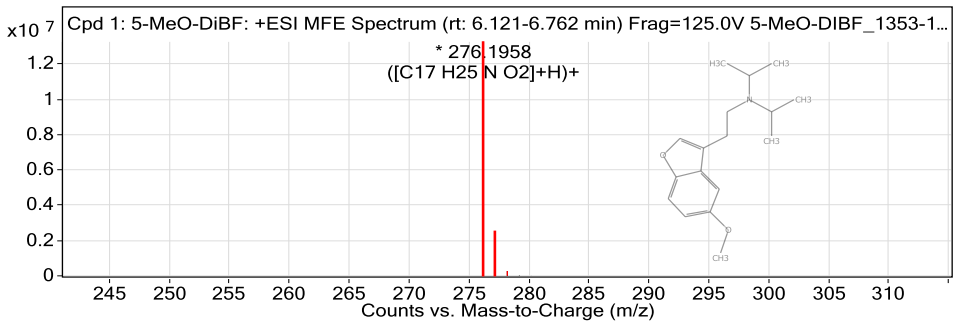
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 1: 5-MeO-DiBF	5-MeO-DiBF	6.169	275.1886

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
5-MeO-DiBF	276.1958	6.169	275.1886	6.17	C17 H25 N O2	275.1885	-0.18

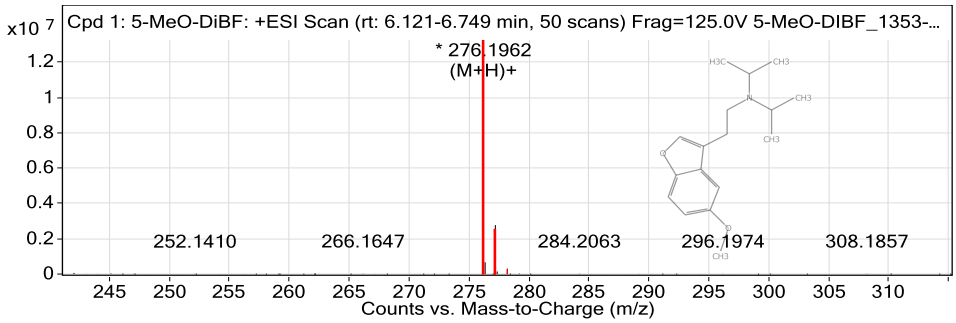
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
276.1958	1	13245692	C17 H25 N O2	(M+H)+
277.1992	1	2434221.07	C17 H25 N O2	(M+H)+
278.2026	1	274276.11	C17 H25 N O2	(M+H)+
279.2047	1	23204.8	C17 H25 N O2	(M+H)+
280.1967	1	3633.13	C17 H25 N O2	(M+H)+

--- End Of Report ---

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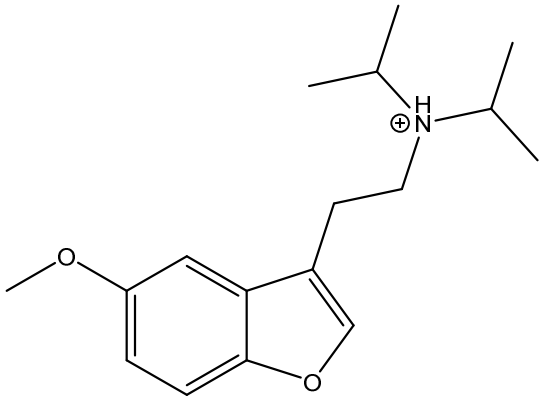
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1353-15**
Our notebook code:	P-1353-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO-d_6
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:	
Chemical name:	N-isopropyl-N-(2-(5-methoxybenzofuran-3-yl)ethyl)propan-2-aminium
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample 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:	December 18, 2015

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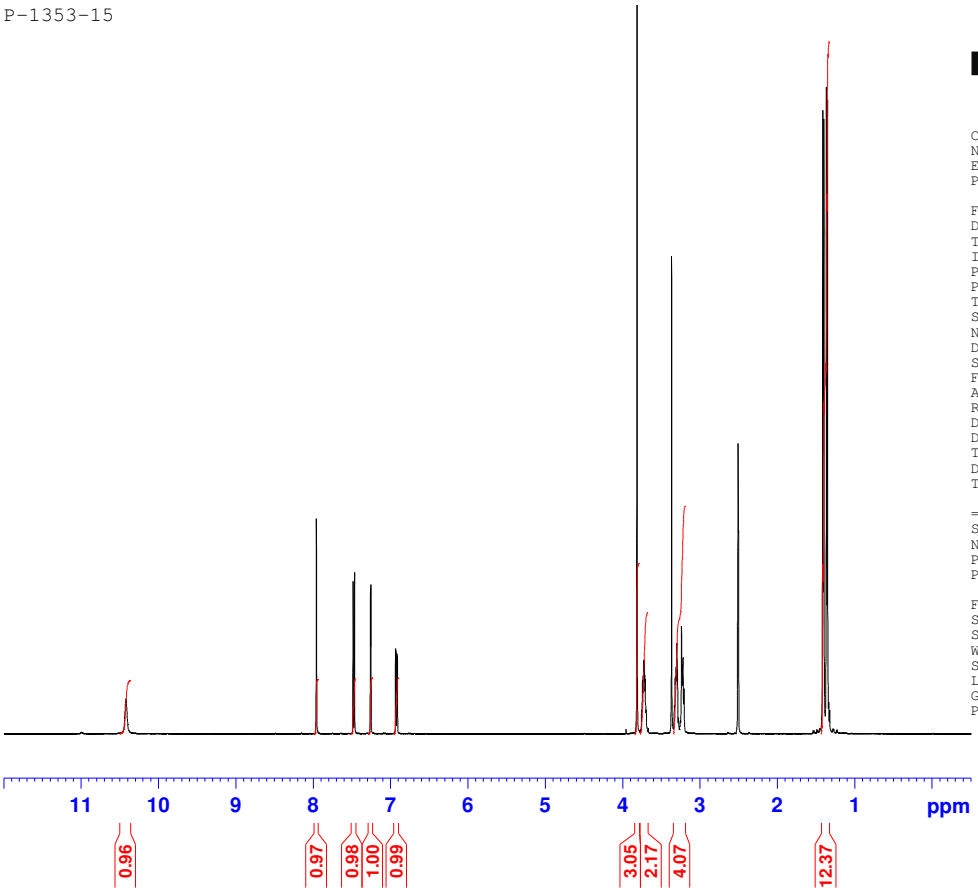
P-1353-15



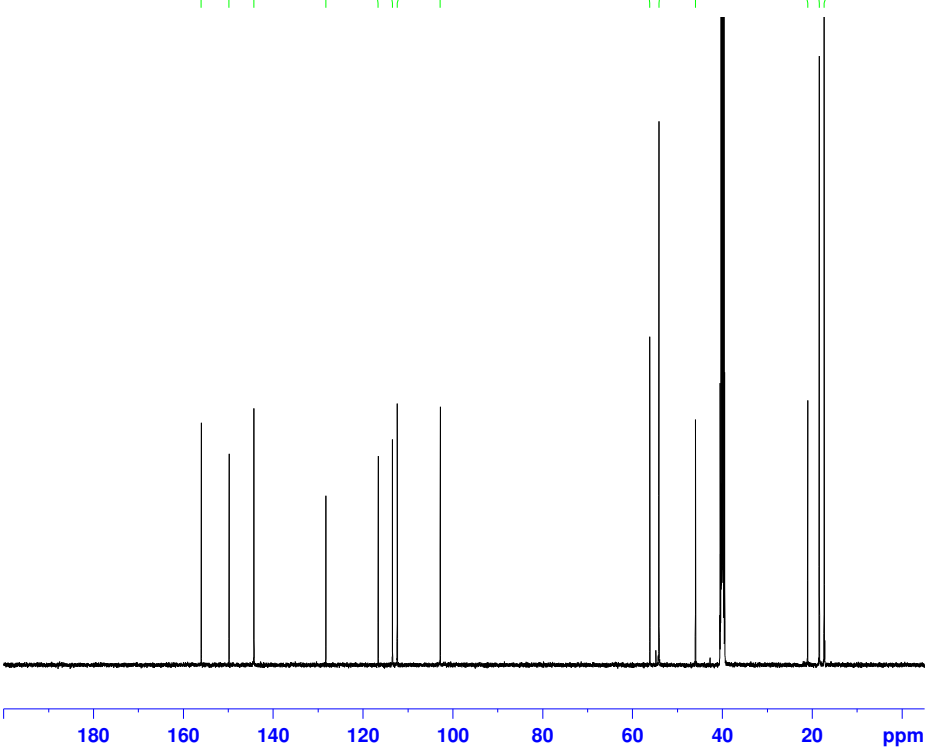

```
Current Data Parameters
NAME          P-1353-15
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20151217
Time              22.53
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                   57
DW               50.000 usec
DE                6.50 usec
TE                296.0 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
```

P-1353-15




```
Current Data Parameters
NAME          P-1353-15
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20151218
Time              0.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
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                296.0 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
```