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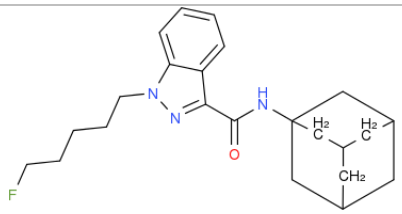
ANALYTICAL REPORT[1]

5F-AKB48 (C23H30FN3O)

N-(1-adamantyl)-1-(5-fluoropentyl)-1H-indazole-3 –carboxamide

Remark – other NPS detected: none

Sample ID:	1179-15
Sample description:	powder - white
Sample type:	seized /Customs
Date of sample receipt (M/D/Y):	5/25/2015
Date of entry (M/D/Y) into NFL database:	6/3/2015
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	N-(1-adamantyl)-1-(5-fluoropentyl)-1H-indazole-3 –carboxamide
Other names	APINACA 5-fluoropentyl analog; 5F-APINACA
Formula (per base form)	C23H30FN3O
M_w (g/mol)	383,51
Salt form/anions detected	base
StdInChIKey	UCMFSGVIEPXYIV-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	residual solvents detected by NMR and confirmed by SPME technique (diisopropylether)

[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 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) 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 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

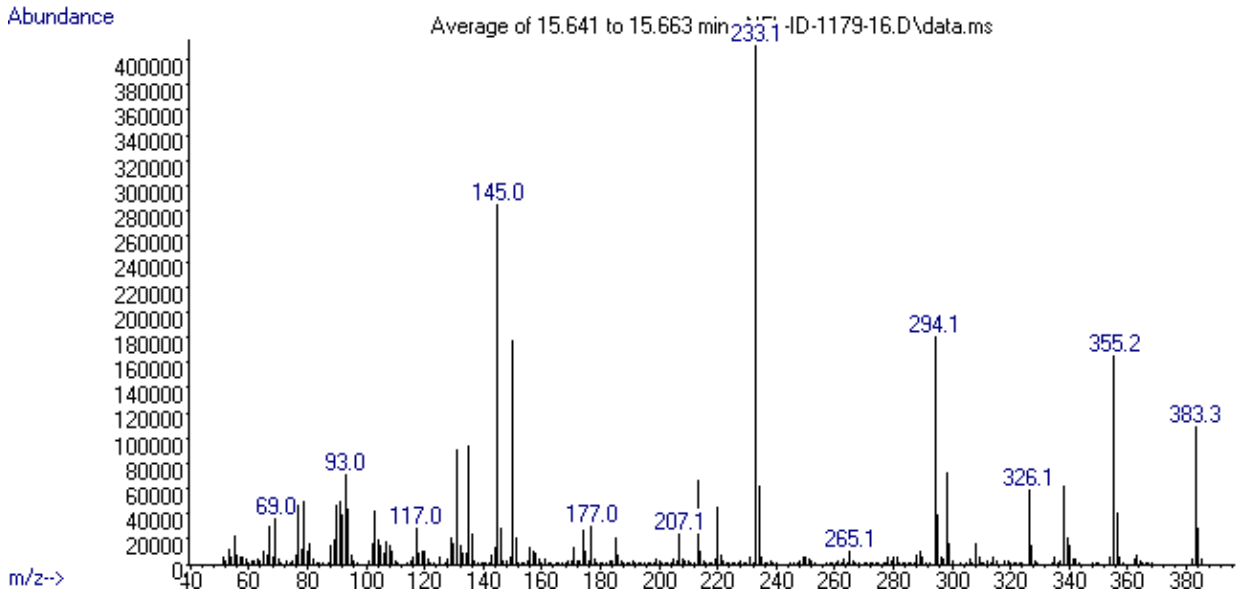
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	

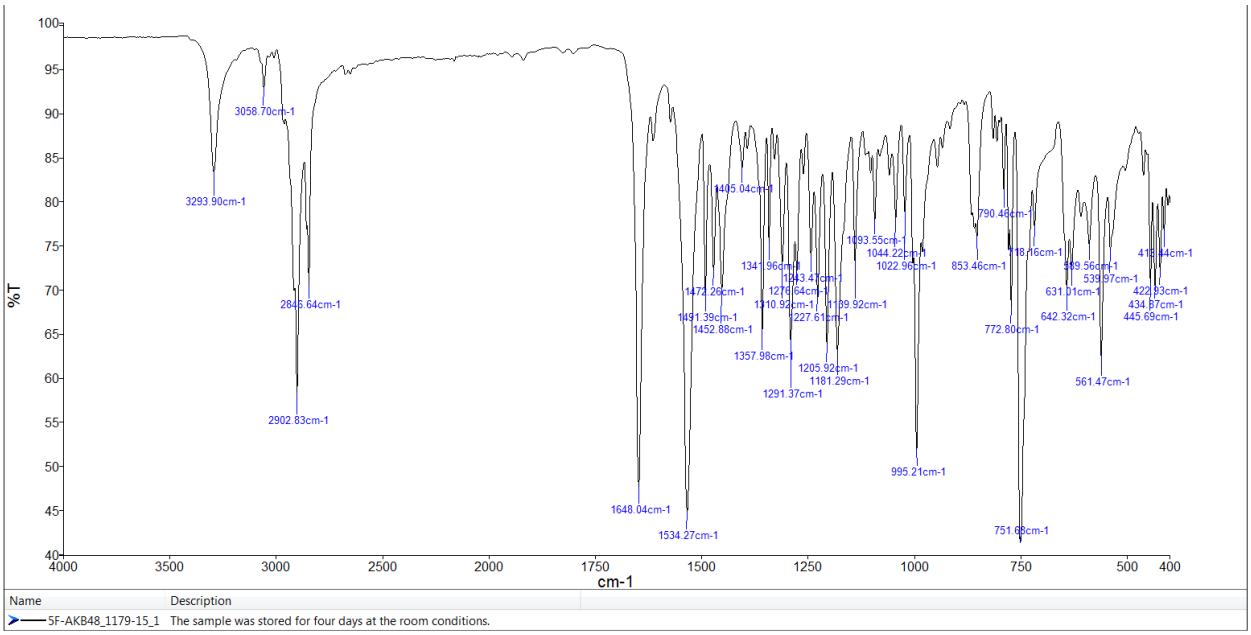
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 15,65 BP(1): 233; BP(2): 145,BP(3) :294,
HPLC-TOF	+	Exact mass (theoretical): 383,2373; measured value Δppm:-0,24; formula:C23H30FN3O
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
IC (anions)	-	spot tests only
NMR (in FKKT)	+	
validation		FTIR-ATR (after solvents evaporation consistent) by SWGDRUG library Lot#N1P53EMG (QU>0.99)
other		

ANALYTICAL RESULTS

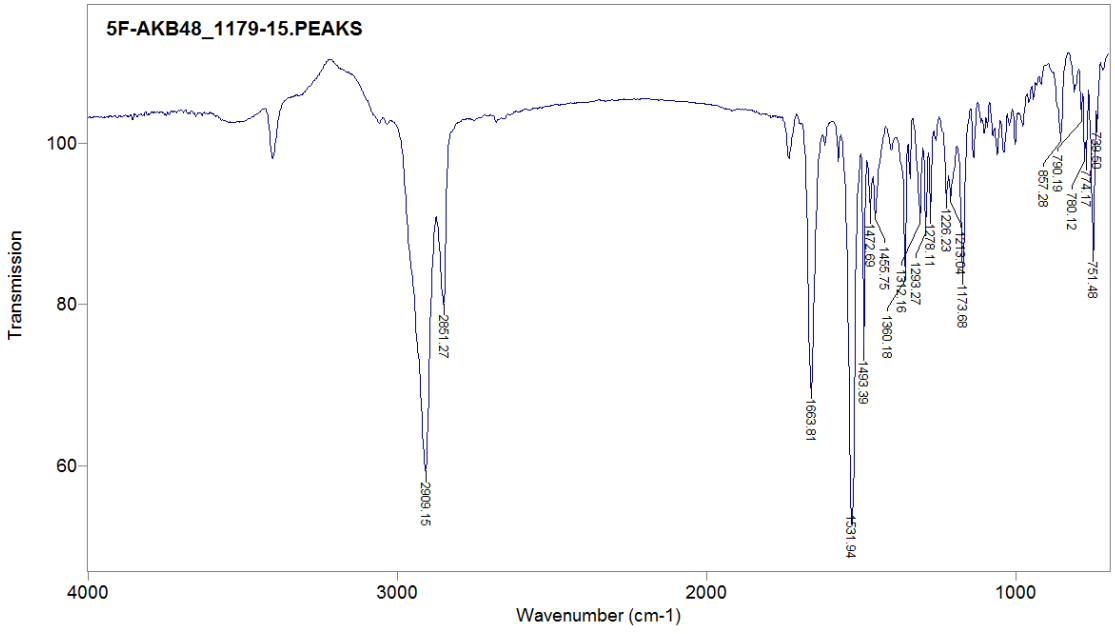
MS (EI)



FTIR-ATR - direct measurement (residual solvents evaporated at room T)



IR (condensed phase – after chromatographic separation)



Target Compound Screening Report

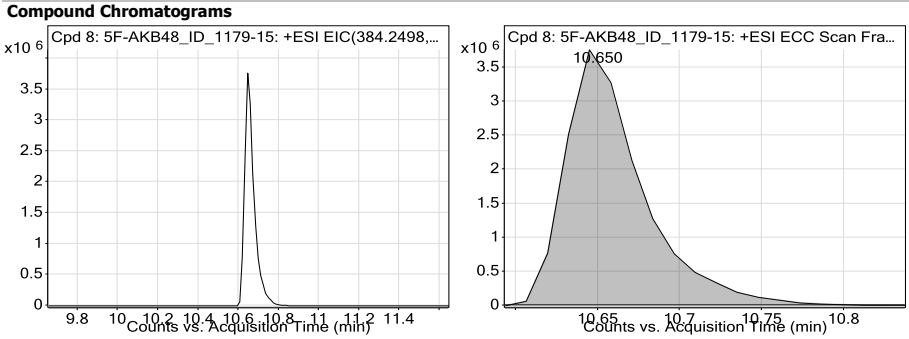
Data File	5F-AKB48_ID 1179_TOF.d	**Sample Name**	5F-AKB48
Sample Type	Sample	**Position**	P1-A2
Instrument Name	SG13170002	**User Name**	
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	5/27/2015 11:53:15 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

Compound Table

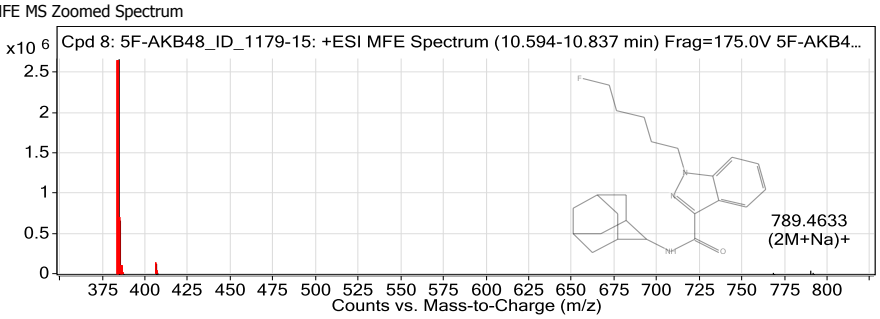
Label	Tgt Name	MFG Formula	Tgt Formula	Obs. RT	Obs. Mass
Cpd 8: 5F-AKB48_ID_1179-15	5F-AKB48_ID_1179-15	C23 H30 F N3 O	C23 H30 F N3 O	10.65	383.2374

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error	Tgt Formula	Find Cpds Algorith
5F-AKB48_ID_1179-15	384.2445	10.65	383.2374	10.65	C23 H30 F N3 O	383.2373	-0.24	C23 H30 F N3 O	Find by Molecular Feature

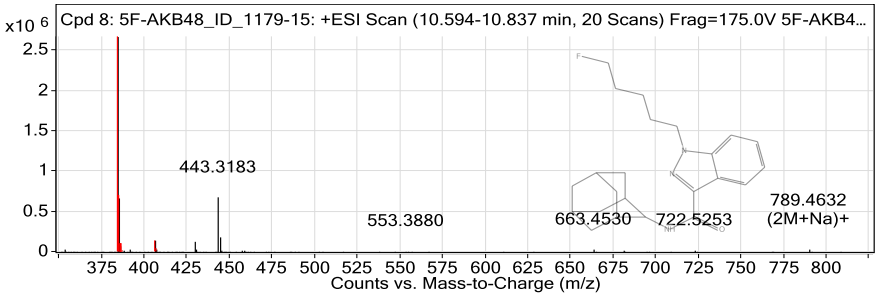
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
384.2445	1	2667331	C23 H30 F N3 O	(M+H)+
385.2482	1	676387.85	C23 H30 F N3 O	(M+H)+
386.2509	1	83303.44	C23 H30 F N3 O	(M+H)+
387.255	1	9001.77	C23 H30 F N3 O	(M+H)+
406.2264	1	137104.8	C23 H30 F N3 O	(M+Na)+
407.2297	1	33450.91	C23 H30 F N3 O	(M+Na)+
767.481	1	21695.41		(2M+H)+
768.4844	1	11873.9		(2M+H)+
789.4633	1	56882.85		(2M+Na)+
790.466	1	29157.46		(2M+Na)+

--- End Of Report ---

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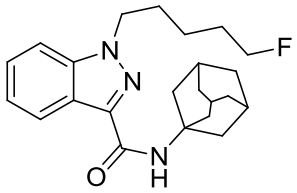
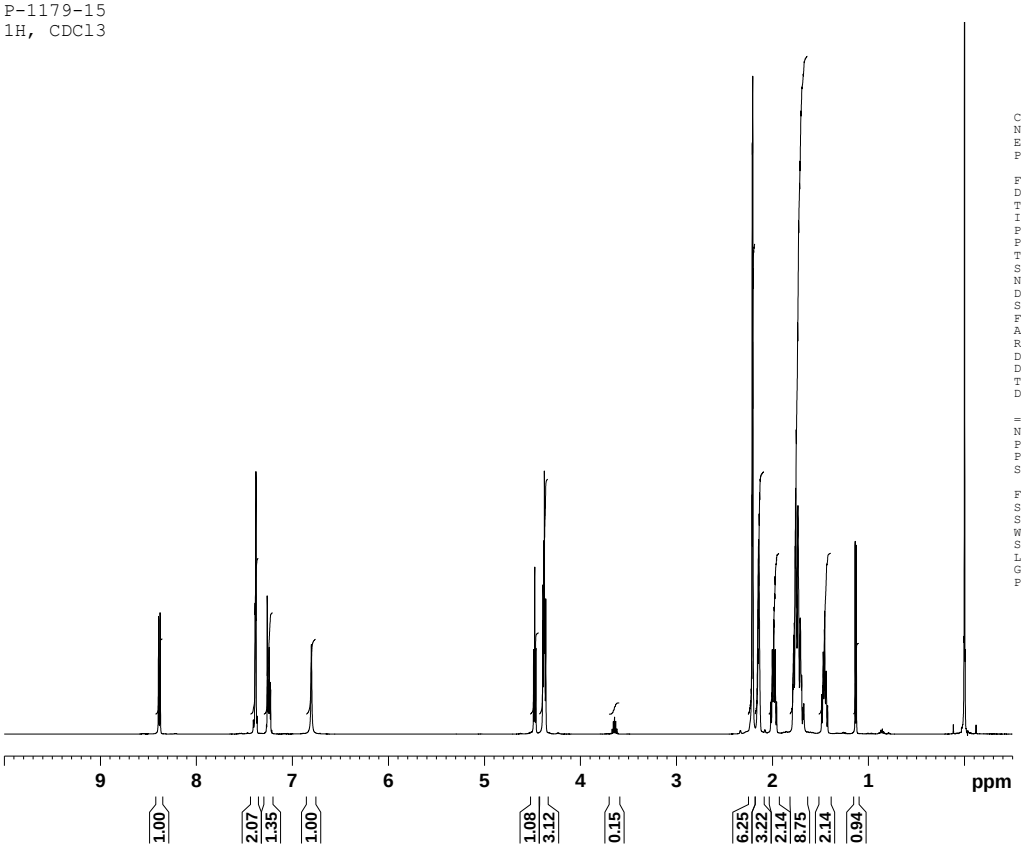
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1179-15**
Our notebook code:	P-1179-15
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$ 15 mg dissolved in 0.7 mL $DMSO_6$
NMR experiments:	^{1}H, ^{13}C, ^{19}F, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC, ^{1}H–^{15}N *gs*-HMBC. All experiments in both indicated solvents.
Proposed structure with chemical name:	 **A** *N*-(adamantan-1-yl)-1-(5-fluoropentyl)-1*H*-indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - An impurity is detected in ^{1}H and ^{13}C NMR spectra. Based on the comparison with the SDBS database, the impurity is most likely diisopropyl ether (**B**) in the ratio **A** : **B** of ≈ 1 : 0.07. Less likely the impurity is 2-propanol (**C**) in the ration **A** : **C** of ≈ 1 : 0.14.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra in $CDCl_3$ and $DMSO_6$
Author:	Prof. Dr. Janez Košmrlj
Date of report:	June 2, 2015

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 publication are the sole responsibility of the Author and can in no way be taken to reflect the views of the European Commission.

P-1179-15
1H, CDCl3



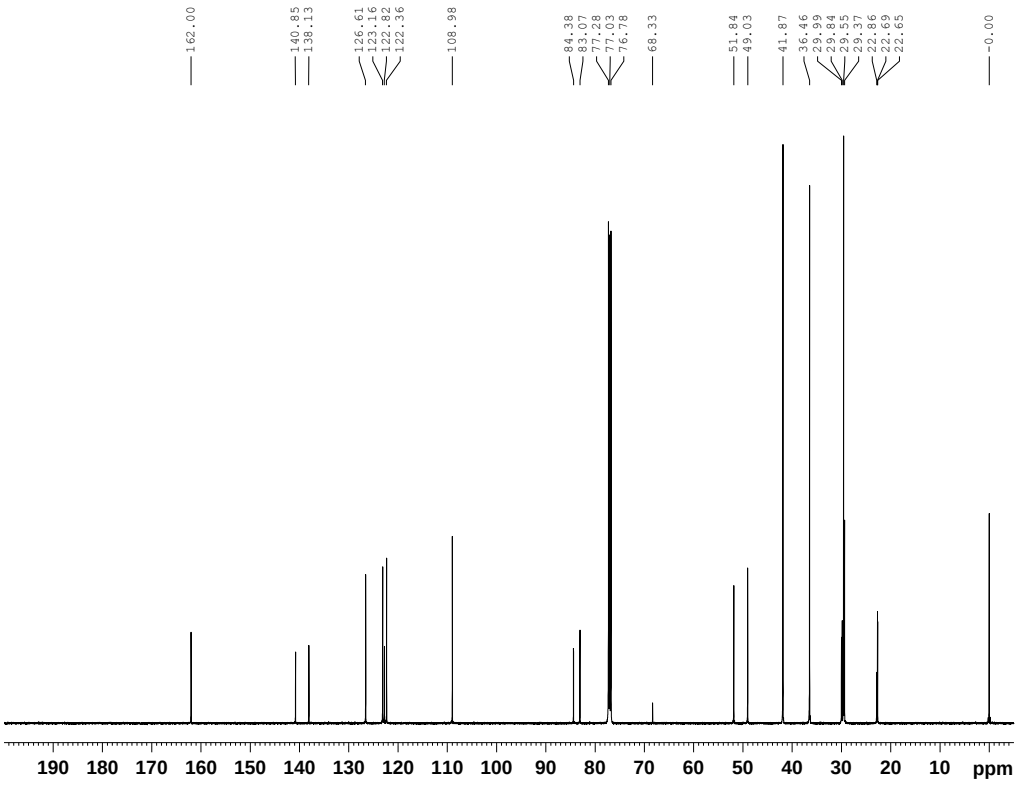

```
Current Data Parameters
NAME                  P-1179-15
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150530
Time                      14.05
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                          64
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                500.1300123 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```

P-1179-15
13C, CDCl3

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Current Data Parameters
NAME                  P-1179-15
EXPNO                         7
PROCNO                        1

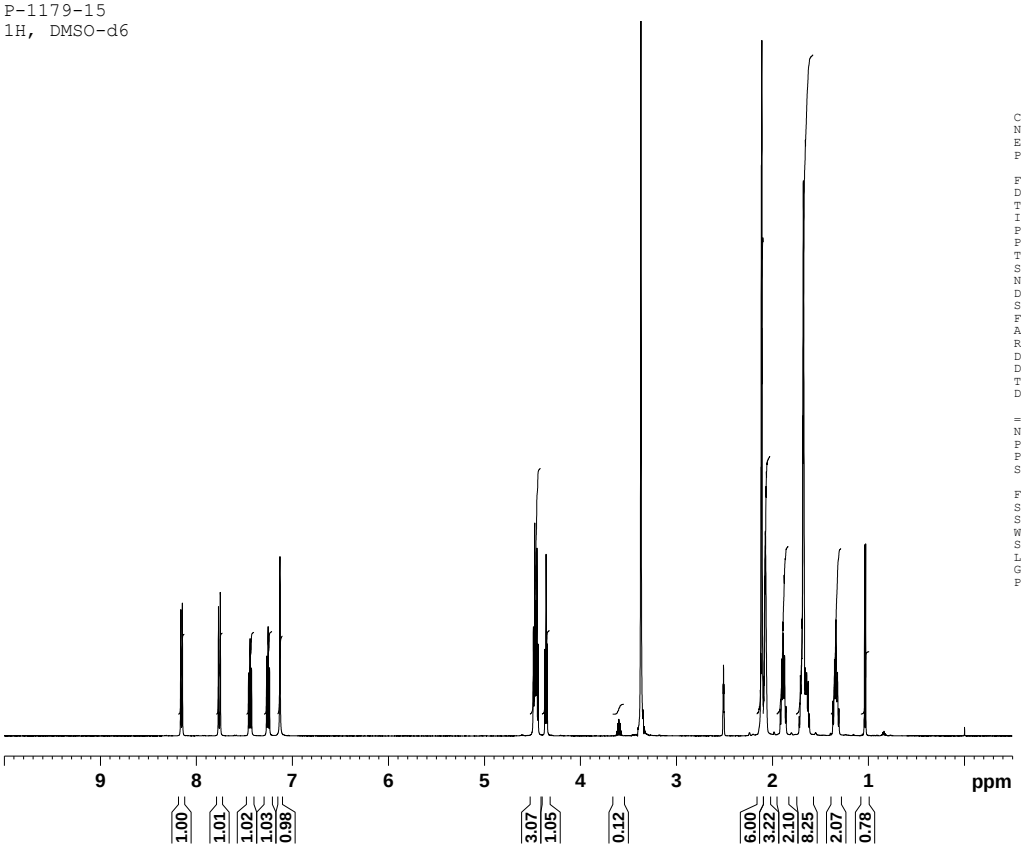
F2 - Acquisition Parameters
Date_                  20150531
Time                      13.19
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                       10240
DS                           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                125.7577902 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

P-1179-15
1H, DMSO-d6

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EXPNO                        20
PROCNO                        1

F2 - Acquisition Parameters
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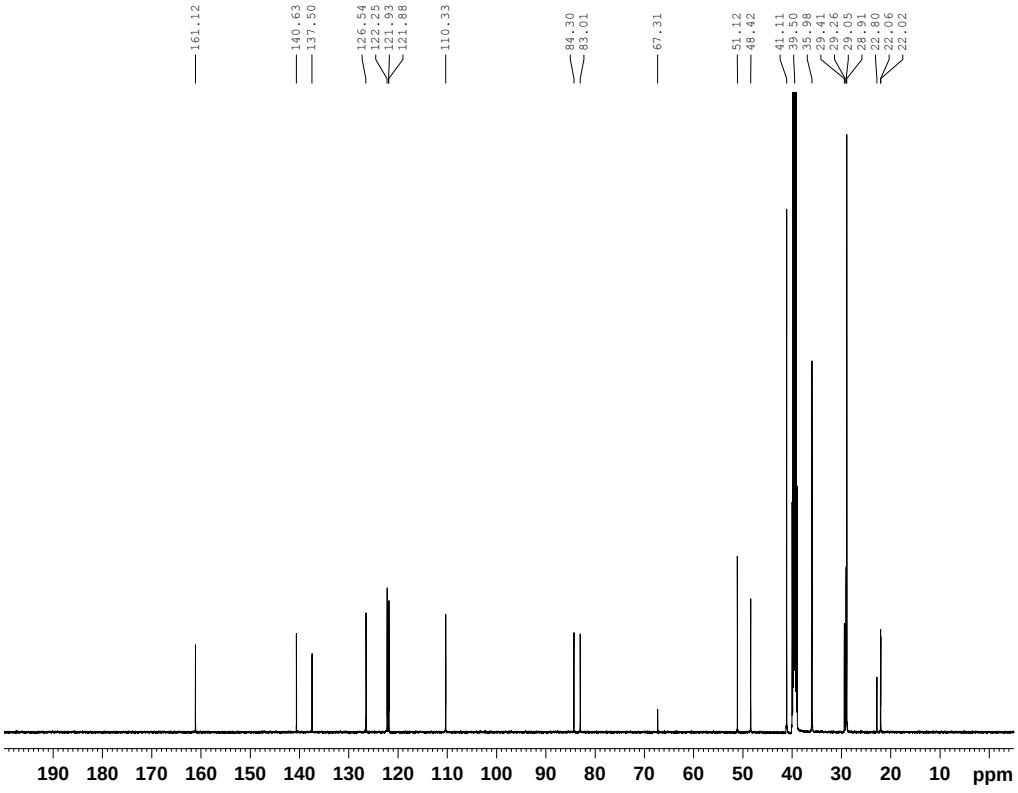
Time                     14.11
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                          16
DS                           2
SWH                10330.578 Hz
FIDRES              0.157632 Hz
AQ                  3.1719923 sec
RG                          64
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                500.1300012 MHz
WDW                         EM
SSB          0
LB                        0.30 Hz
GB          0
PC                        1.00
```

P-1179-15
13C, DMSO-d6

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EXPNO                        26
PROCNO                        1

F2 - Acquisition Parameters
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Time                     1.47
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                  DMSO
NS                       10240
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                125.7578464 MHz
WDW                         EM
SSB          0
LB                        1.00 Hz
GB          0
PC                        1.40
```