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

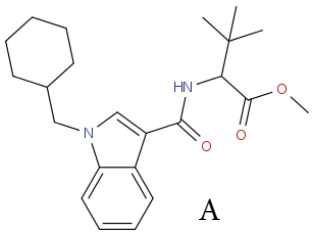
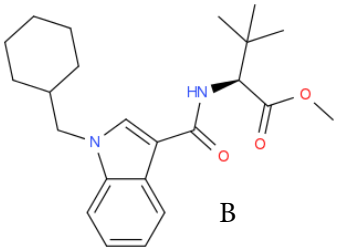
ANALYTICAL REPORT

MDMB-CHMICA (C23H32N2O3)

N-[[1-(cyclohexylmethyl)-1H-indol-3-yl]carbonyl]-3-methyl-valine, methyl ester

Remark – other NPS detected: none

Sample ID:	1190-15
Sample description:	powder - off white
Sample type:	test purchase /RESPONSE -purchasing
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)	 A  B
Systematic name	methyl 2-{[1-(cyclohexylmethyl)-1H-indol-3-yl]formamido}-3,3-dimethylbutanoate (stereochemistry was not confirmed experimentally in the RESPONSE project (structure A)
Other names	N-[[1-(cyclohexylmethyl)-1H-indol-3-yl]carbonyl]-3-methyl-valine, methyl ester; methyl (2S)-2-{[1-(cyclohexylmethyl)-1H-indol-3-yl]formamido}-3,3-dimethylbutanoate (structure B)
Formula (per base form)	C23H32N2O3
M_w (g/mol)	384,5
Salt form	
StdInChIKey	(A) SRJKCVHWIDFUBO-UHFFFAOYSA-N and SRJKCVHWIDFUBO-HXUWFJFHSA-N (B)
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	pure by GC-MS, HPLC-TOF, few % of unidentified organic impurites by 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)
18/05/17	**Empirical formula corrected.**
	Structure (A) where stereochemistry is not defined and corresponding StdInChI key were 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}.

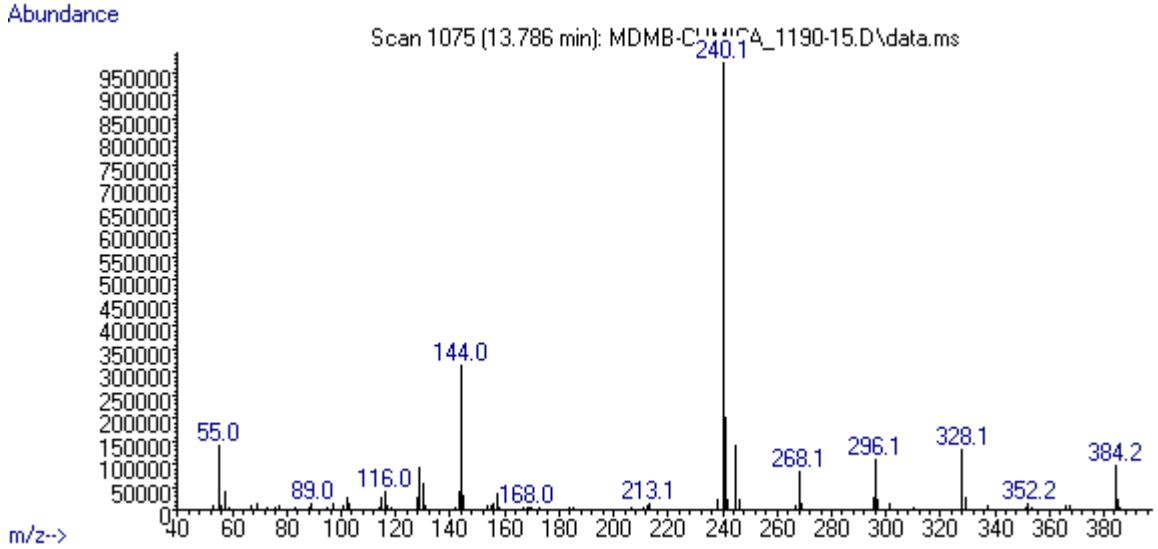
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
water	low

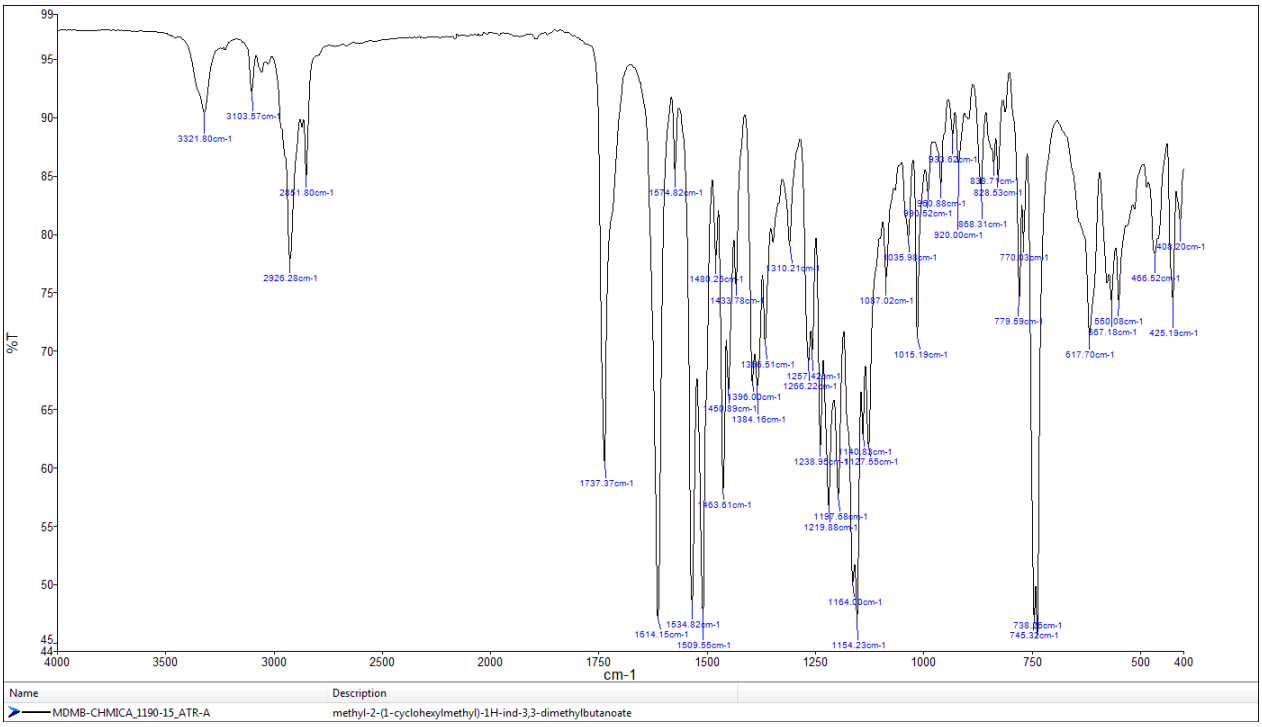
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,79 BP(1): 240; BP(2): 144,BP(3) :241,
HPLC-TOF	+	Exact mass (theoretical): 384,2413; measured value Δppm:0,08; formula:C23H32N2O3
FTIR-ATR	+	direct measurement (note sample contains MeOH)
FTIR (condensed phase) always as base form	+	
NMR	+	
validation		
other		

ANALYTICAL RESULTS

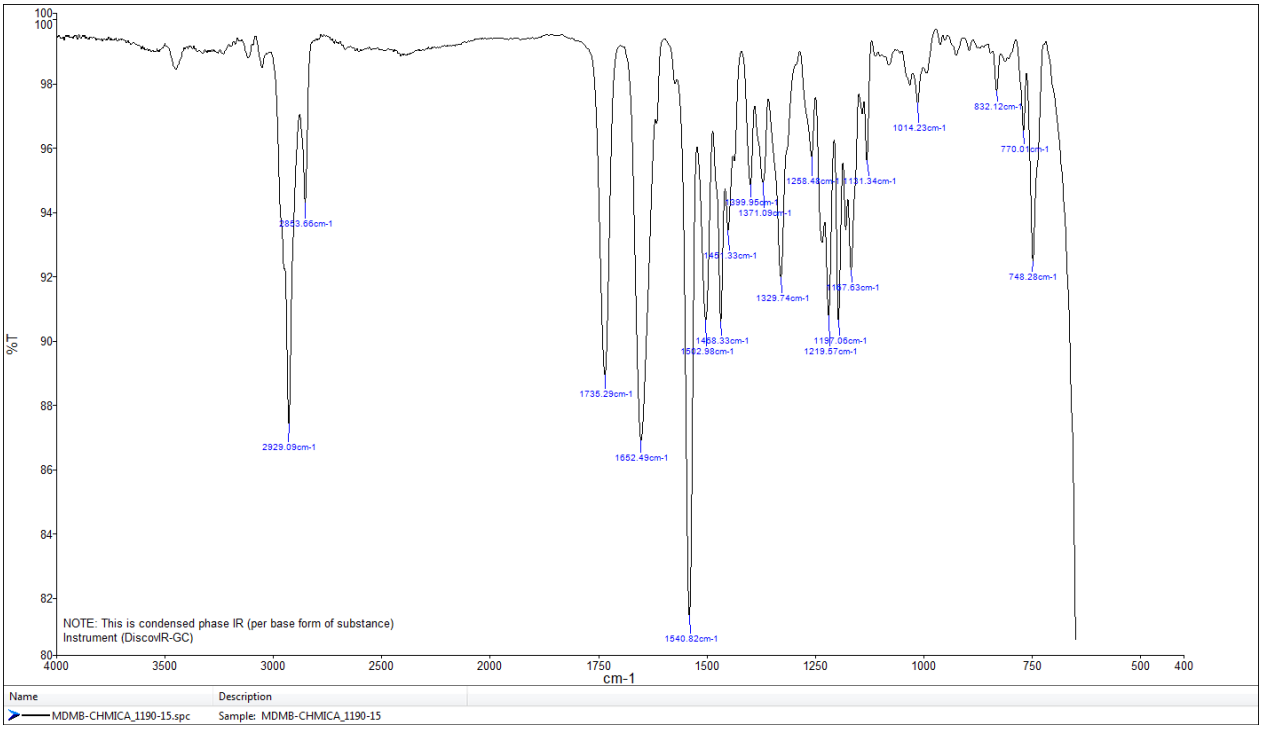
MS (EI)



FTIR-ATR - direct measurement (note: sample contains few % of organic impurities)



IR (condensed phase)



Target Compound Screening Report

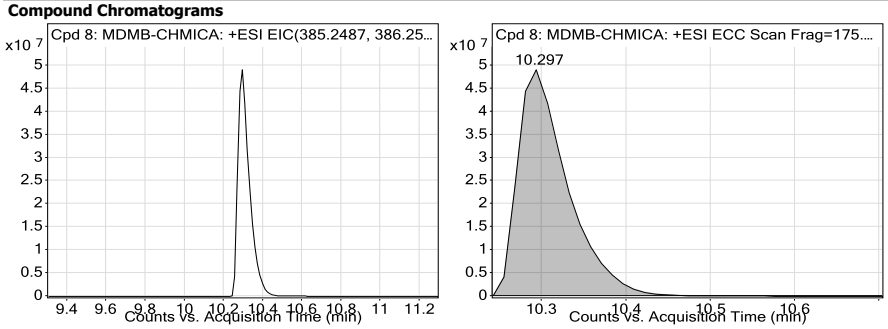
Data File	MDMB-CHMICA_1190-15_TOF.d	**Sample Name**	MDMB-CHMICA
Sample Type	Sample	**Position**	P1-C4
Instrument Name	SG13170002	**User Name**	
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	7/8/2015 11:08:06 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

Compound Table

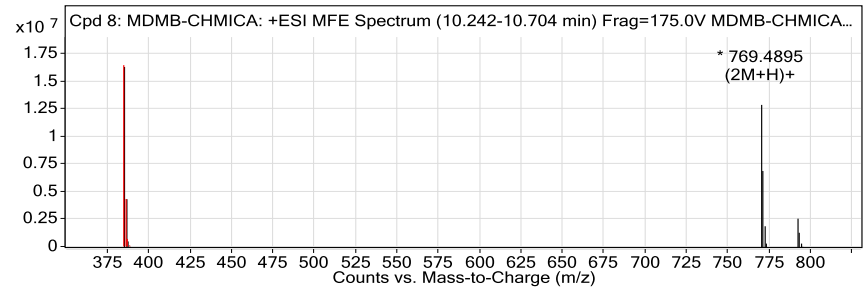
Label	Tgt Name	MFG Formula	Tgt Formula	Obs. RT	Obs. Mass
Cpd 8: MDMB-CHMICA	MDMB-CHMICA	C23 H32 N2 O3	C23 H32 N2 O3	10.297	384.2413

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error	Tgt Formula	Find Cpds Algorith
MDMB-CHMICA	385.2485	10.297	384.2413	10.294	C23 H32 N2 O3	384.2413	0.08	C23 H32 N2 O3	Find by Molecular Feature

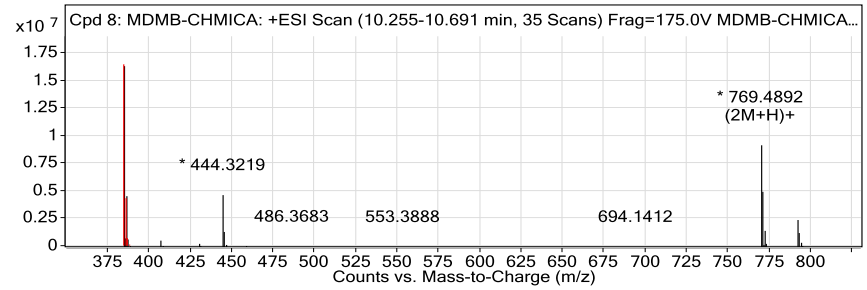
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
385.2485	1	16352723	C23 H32 N2 O3	(M+H)+
386.2518	1	4397796.08	C23 H32 N2 O3	(M+H)+
387.2552	1	590485.43	C23 H32 N2 O3	(M+H)+
769.4895	1	12944640		(2M+H)+
770.4928	1	6915544.8		(2M+H)+
771.496	1	1947154.22		(2M+H)+
772.4996	1	358547.83		(2M+H)+
791.4715	1	2652121.5		(2M+Na)+
792.4754	1	1389992.18		(2M+Na)+
793.4784	1	372498.4		(2M+Na)+

--- End Of Report ---

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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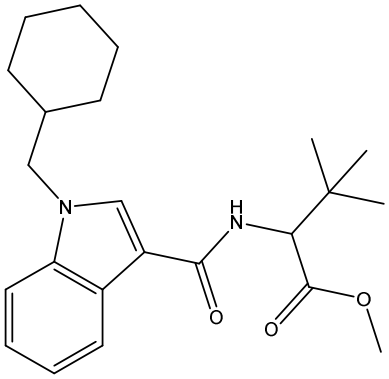
REPORT

Sample ID:	**1190-15**
Our notebook code:	P-1190-15
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:	 methyl 2-(1-(cyclohexylmethyl)-1*H*-indole-3-carboxamido)-3,3-dimethylbutanoate
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR, containing a small amount (a few %) of unidentified organic compound(s).
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	October 6, 2015

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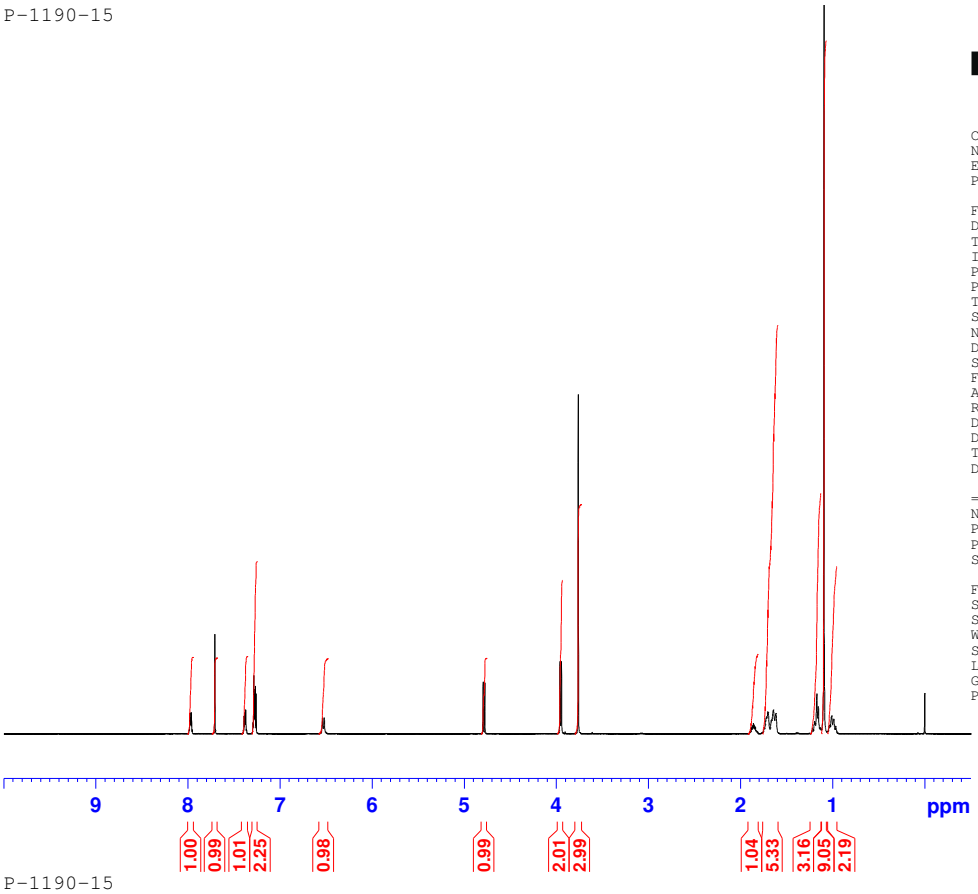
P-1190-15



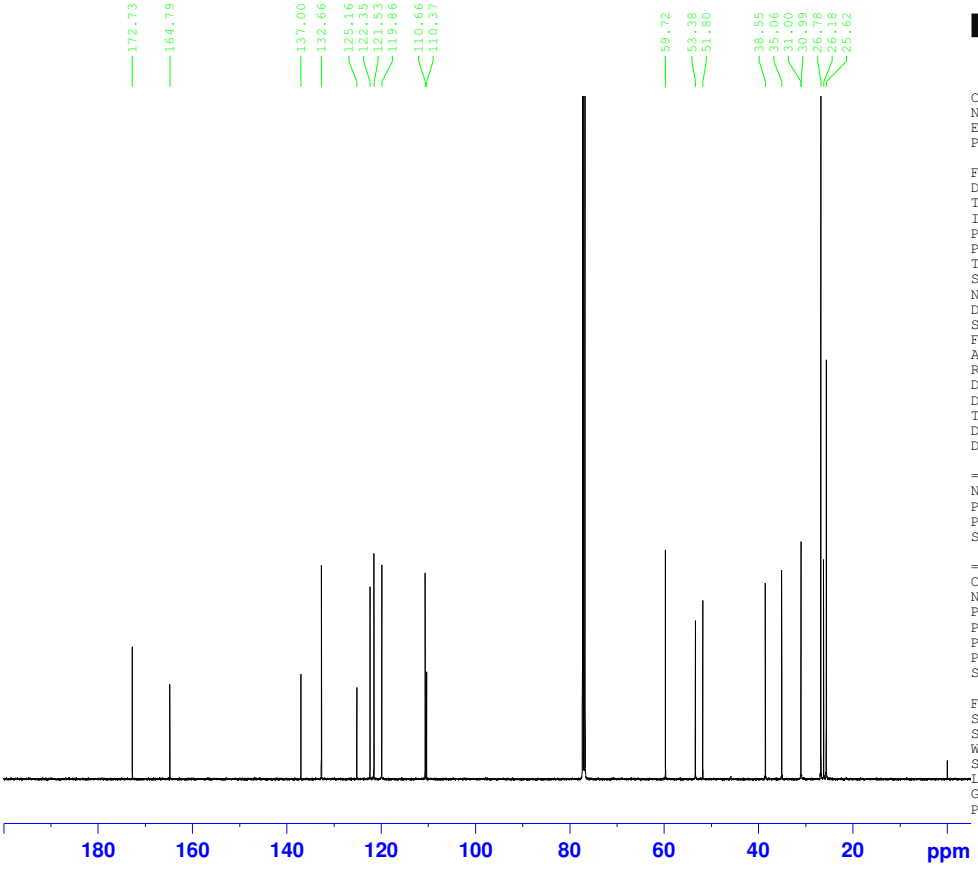
Current Data Parameters
NAME P-1190-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150811
Time 0.40
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 64
DW 48.400 usec
DE 6.50 usec
TE 298.5 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 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1190-15



Current Data Parameters
NAME P-1190-15
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150811
Time 4.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 5120
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 299.1 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 125.7577897 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40