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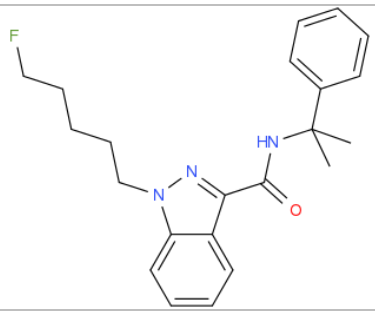
ANALYTICAL REPORT[1]

CUMYL-5F-PINACA (C22H26FN3O)

1-(5-fluoropentyl)-N-(2-phenylpropane-2-yl)-1H-indazole-3-carboxamide

Remark – other NPS detected: none

Sample ID:	1155
Sample description:	liquid – (yellow viscous liquid)
Sample type:	seized /LJ
Date of sample receipt (M/D/Y):	10/28/2014
Date of entry (M/D/Y) into NFL database:	3/27/2015
Report (updates) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified-structure[2] (base form)	
Systematic name	1-(5-fluoropentyl)-N-(2-phenylpropane-2-yl)-1H-indazole-3-carboxamide
Other names	5-fluoro Cumyl-pinaca, SGT 25 5-fluoro Cumyl-pinaca, SGT 25
Formula (per base form)	C22H26FN3O
M_w (g/mol)	367,46
Salt form	base
StdInChIKey	XSHGVIPHMOTDCS-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	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)
04.11.2015	Some typing errors corrected.

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

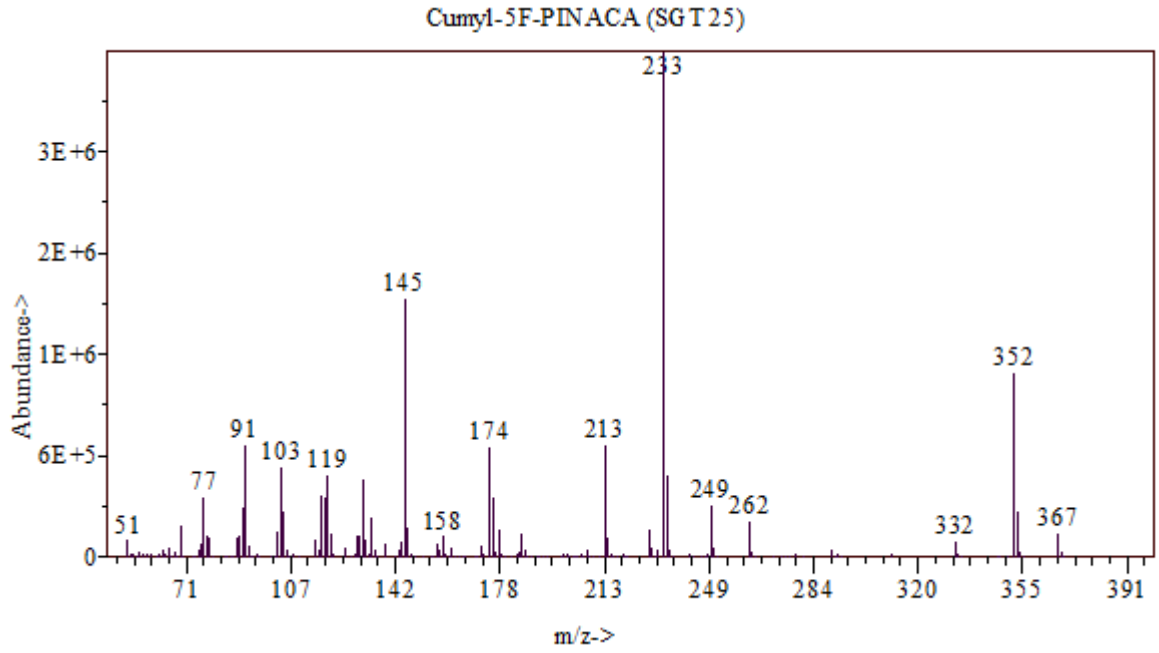
Supporting information

Solubility in	result/remark
CH_2Cl_2	+
MeOH	+
H_2O	Not tested

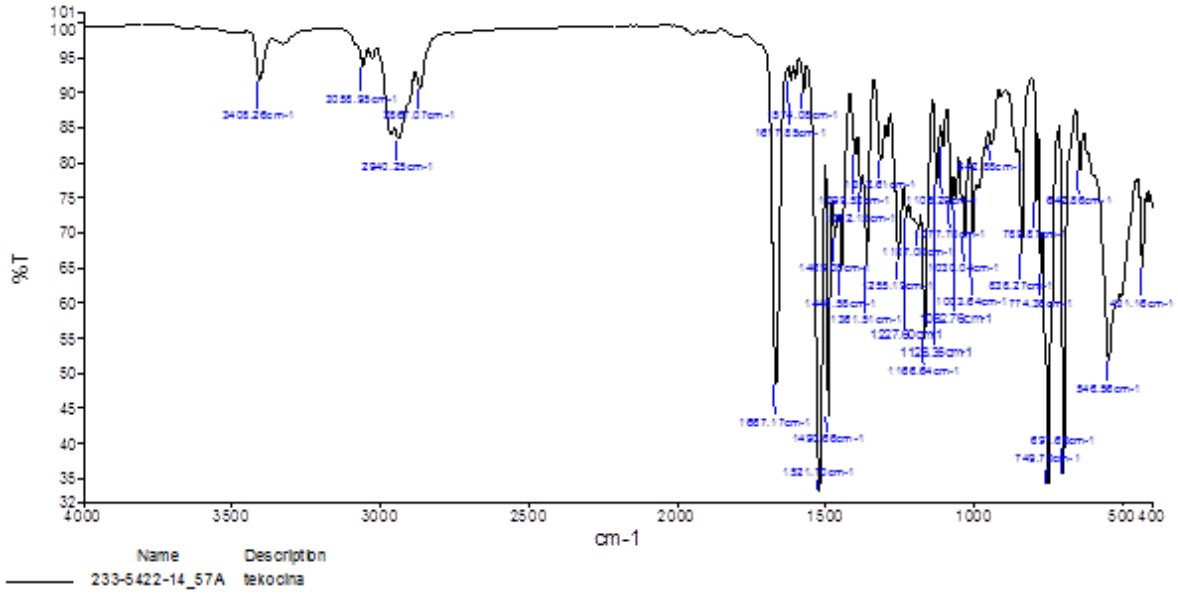
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 12,05 BP(1): 233; BP(2): 145,BP(3) :352,
HPLC-TOF	+	Formula confirmed:C22H26FN3O
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form		
IC (anions)		
NMR		
validation		
other		

ANALYTICAL RESULTS

MS (EI)



FTIR-ATR - direct measurement



ID 1155

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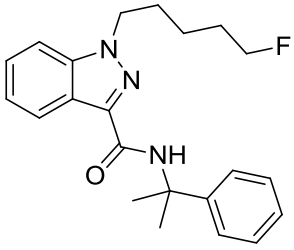
Dr. Janez Košmrlj
Professor of Organic Chemistry

January 17, 2015

Dr. Sonja Klemenc
Head of Chemistry Department
Vodovodna 95
1000 Ljubljana
Slovenija

Dear Dr. Sonja Klemenc,

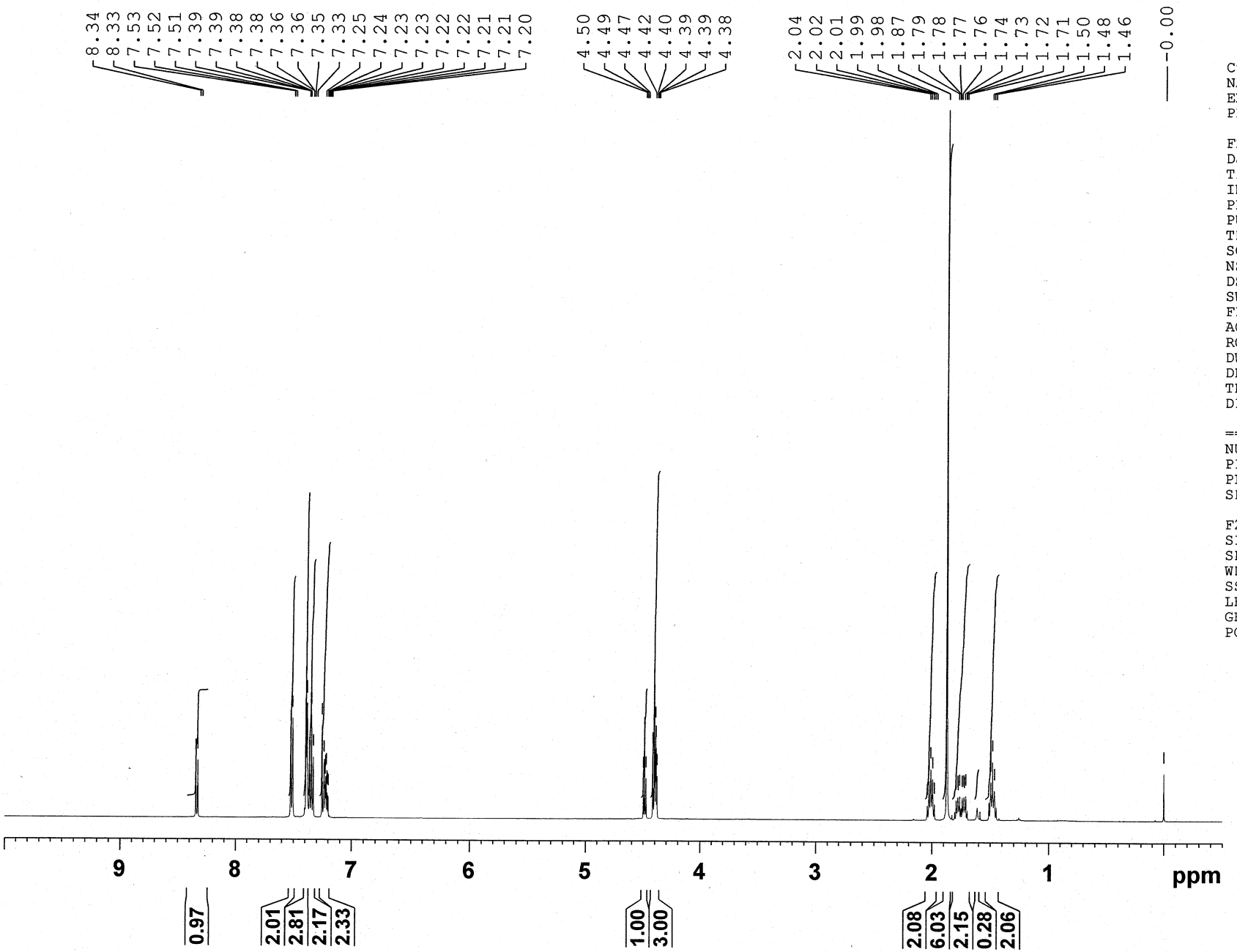
Please find enclosed the results of the structure elucidation for the sample:

Sample ID:	**233-5422-14-57b**
Received date:	November, 2014
Our notebook code:	P-233-5422-14-57b
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
NMR experiments:	^{1}H NMR, ^{13}C NMR
Proposed structure with atom numbering scheme, formula, exact mass, molecular weight:	 Chemical Formula: $C_{22}H_{26}FN_3O$ Exact Mass: 367.2060 Molecular Weight: 367.4597
Chemical name:	1-(5-Fluoropentyl)-*N*-(2-phenylpropan-2-yl)-1*H*-indazole-3-carboxamide
Comments:	- The analysis of ^{1}H NMR and ^{13}C NMR spectra confirm the structure proposed by MS.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra (pp 2-3)

Sincerely,

Janez Košmrlj

P-233-5422-14-57b



Current Data Parameters

NAME	P-233-5422-14-57b
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20141230
Time	16.39
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	80.6
DW	48.400 usec
DE	6.50 usec
TE	296.1 K
D1	1.00000000 sec

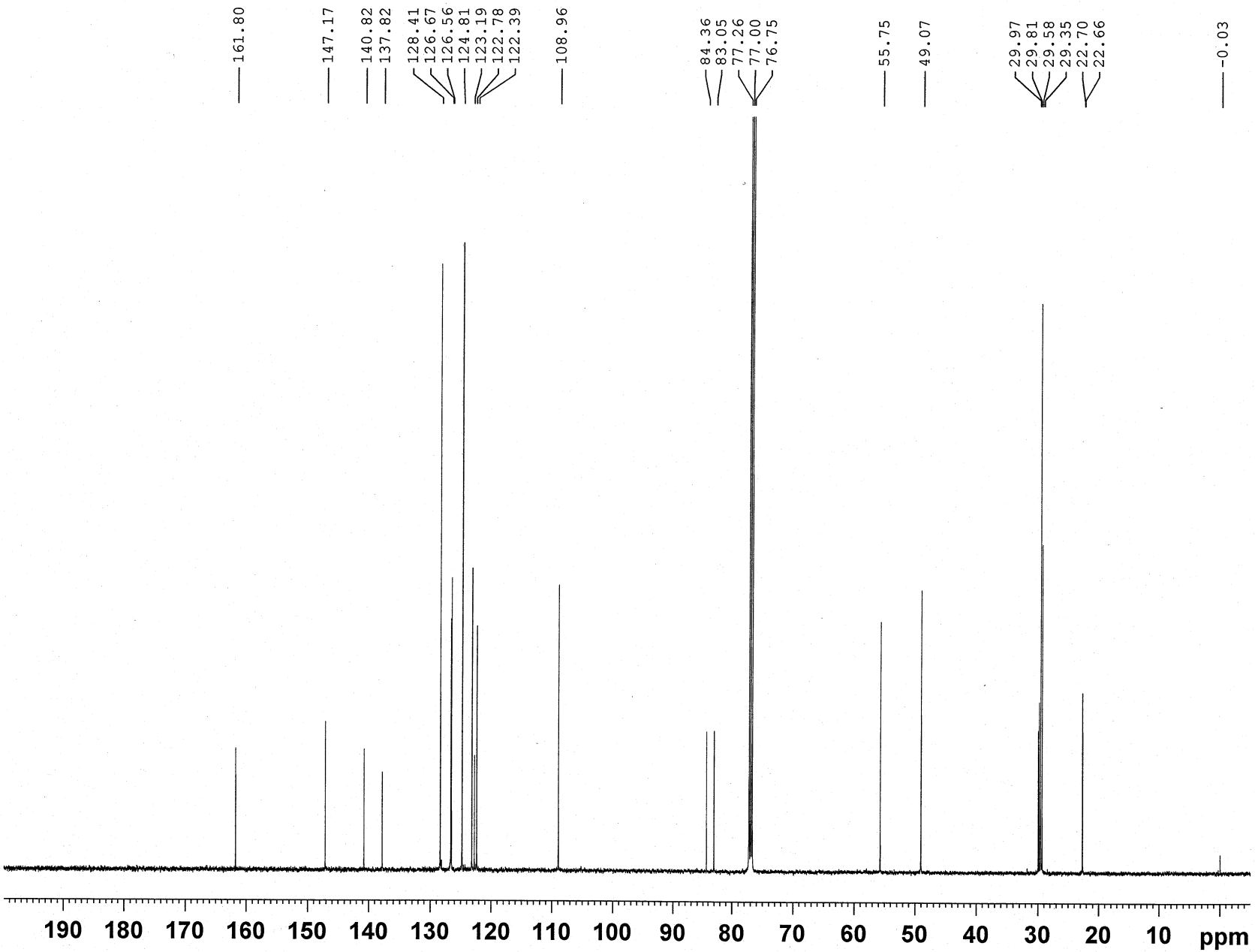
======== CHANNEL f1 ========

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

F2 - Processing parameters

SI	65536
SF	500.1300172 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

P-233-5422-14-57b



BRUKER

Current Data Parameters
NAME P-233-5422-14-57b
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141230
Time 19.07
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 125.7577953 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40