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EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

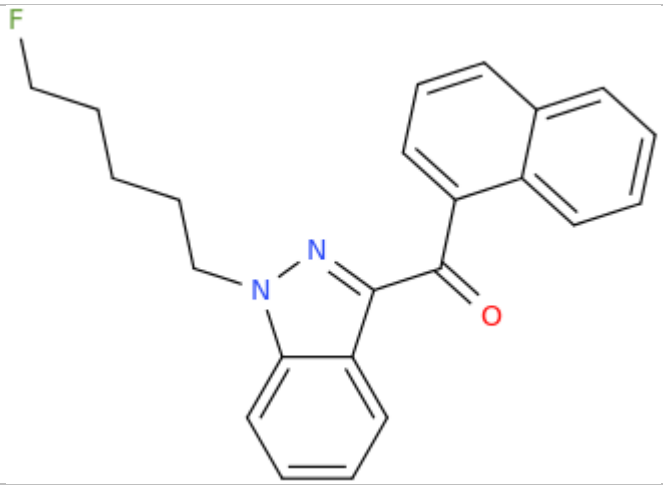
ANALYTICAL REPORT

THJ-2201 (AM-2201 indazole analogue) (C23H21FN2O)

1-(5-fluoropentyl)-3-(naphthalene-1-carbonyl)-1H-indazole

Remark – other NPS detected: none

Sample ID:	1156-15A
Sample description:	powder
Sample type:	seized /LJ
Date of sample receipt (DD/MM/YYYY):	28/10/2014
Date of entry (DD/MM/YYYY) into NFL database:	14/08/2018
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure[1] (base form)	
Systematic name	1-(5-fluoropentyl)-3-(naphthalene-1-carbonyl)-1H-indazole
Other names	AM-2201 indazole analogue; [1-(5-fluoropentyl)-1H-indazol-3-yl](naphthalen-1-yl)methanone; 5F-JWH-018-N; 5F-THJ-018
Formula (per base form)	C23H21FN2O
M_w (g/mol)	360,43
Salt form/anions detected	base
StdInChIKey (per base form)	DULWRYKFTVFPTL-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	pure by GC-MS

[1] 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 (9.258 min). Injection volume 1 ml and split mode (1:50). Injector temperature: 280 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 7.1 min, then 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 until 6 min) 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 (solid) 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 KOH 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

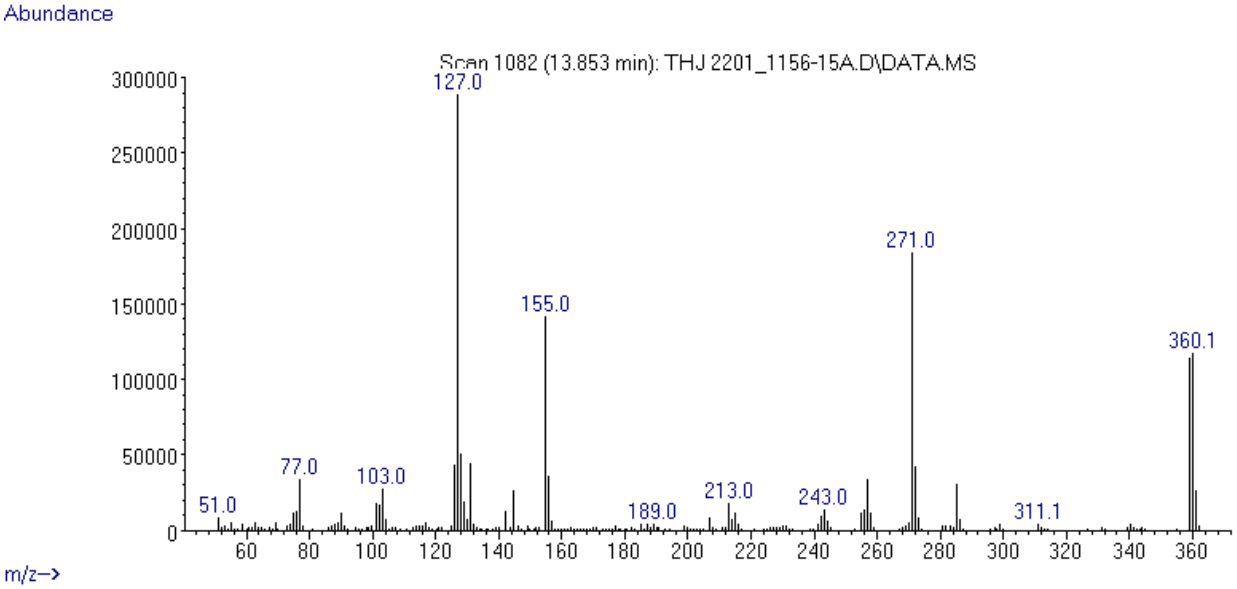
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	partially

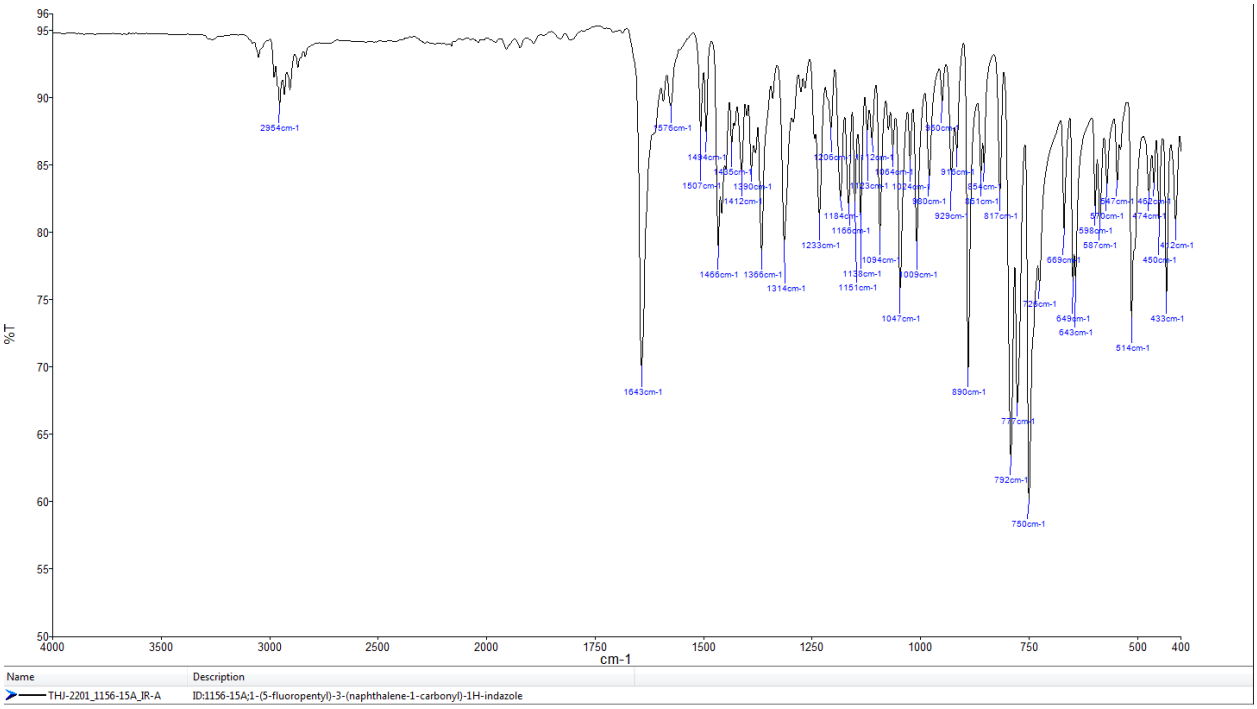
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,85 BP(1): 127; BP(2): 271,BP(3) :155,
HPLC-TOF	-	Exact mass (theoretical): ; measured value Δppm:; formula:
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	-	(by spot tests only)
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

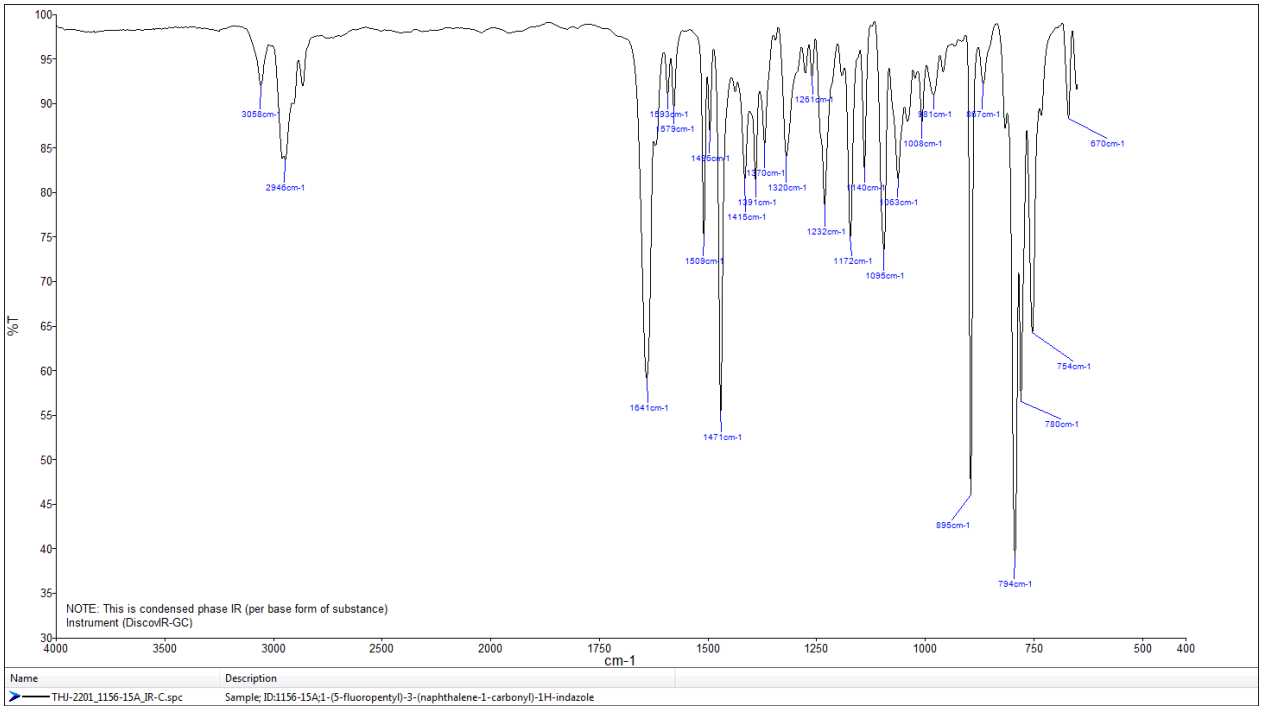
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



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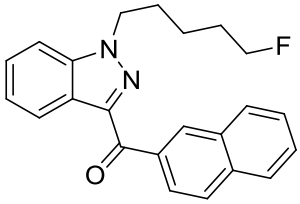
Dr. Janez Košmrlj
Professor of Organic Chemistry

January 17, 2015

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

Dear Dr. Sonja Klemenc,

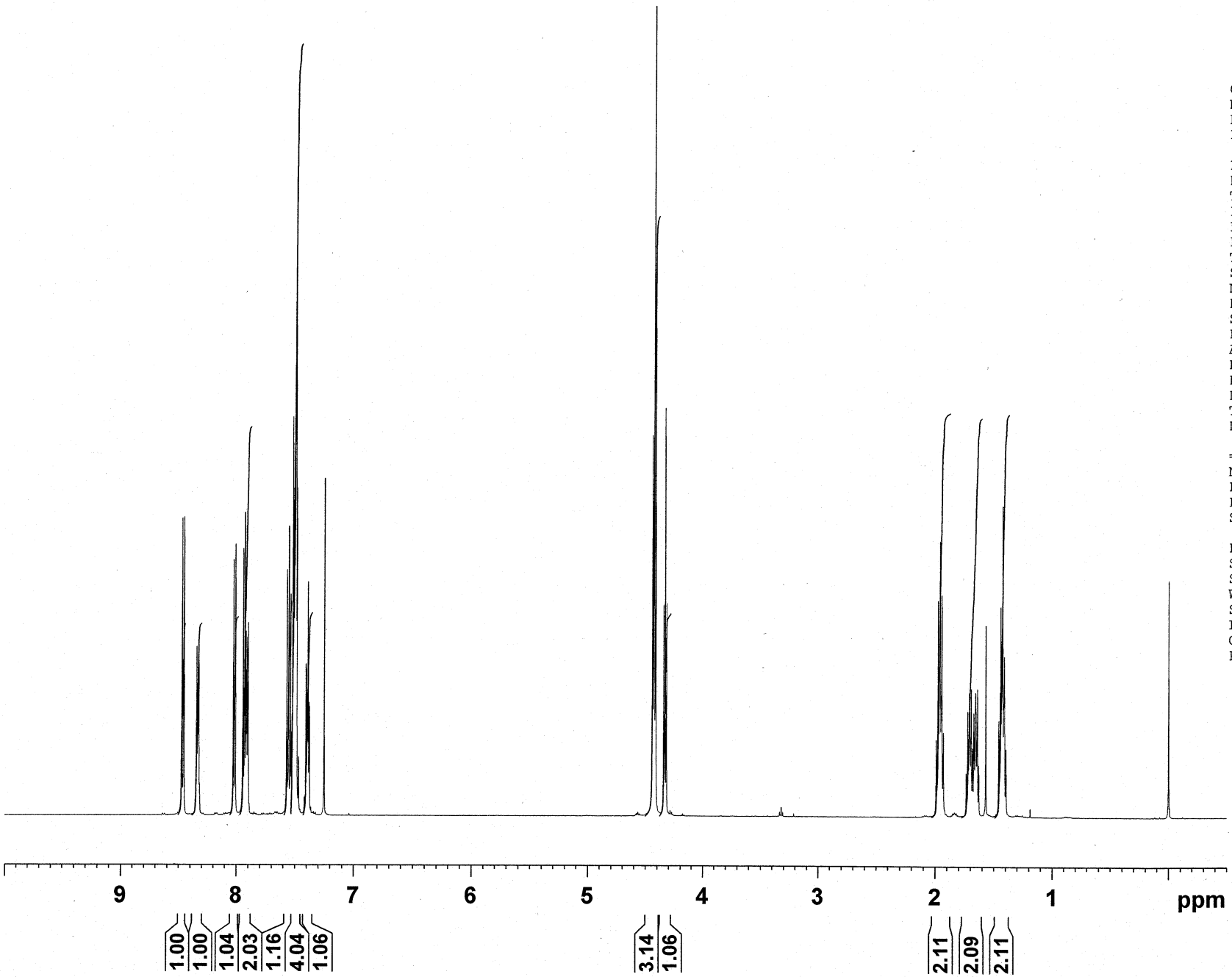
Please find enclosed the results of the structure elucidation for the sample:

Sample ID:	**233-5422-14-64b**
Received date:	November, 2014
Our notebook code:	P-233-5422-14-64b
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: $C_{23}H_{21}FN_2O$ Exact Mass: 360.1638 Molecular Weight: 360.4240
Chemical name:	(1-(5-Fluoropentyl)-1*H*-indazol-3-yl)(naphthalen-2-yl)methanone
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-233-5422-14-64b



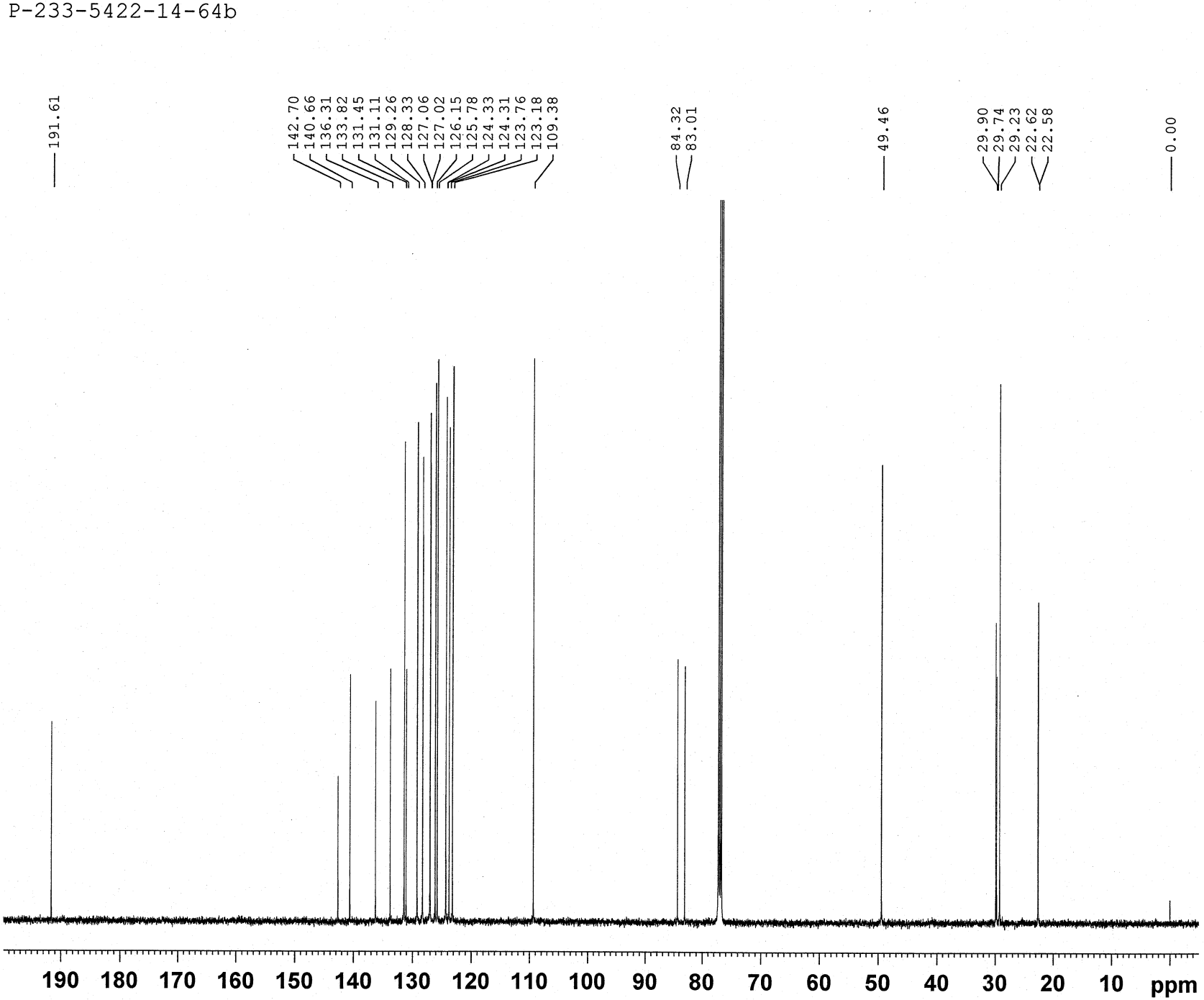
Current Data Parameters
NAME P-233-5422-14-64b
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141231
Time 2.49
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 296.1 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 500.1300185 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-233-5422-14-64b



Current Data Parameters

NAME	P-233-5422-14-64b
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20141231
Time	5.18
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	125.7577920 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40