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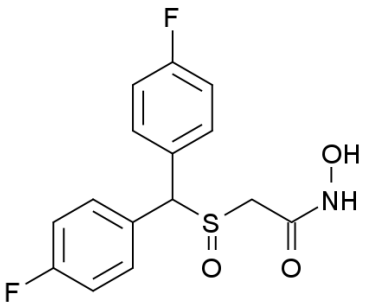
ANALYTICAL REPORT[1]

CRL-40,941 (C15H13F2NO3S)

2-[bis(4-fluorophenyl)methylsulfinyl]-N-hydroxyacetamide

Remark – other NPS detected: none

Sample ID:	1321-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	10/28/2015
Date of entry (M/D/Y) into NFL database:	12/16/2015
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-[bis(4-fluorophenyl)methylsulfinyl]-N-hydroxyacetamide
Other names	
Formula (per base form)	C15H13F2NO3S
M_w (g/mol)	325,33
Salt form	negative
StdInChIKey	VKGUUSVYPXTWMA-UHFFFAOYSA-N
Compound Class	Others
Other NPS detected	none
Add.info (purity..)	inpurities (possibly difluorobenzylhydrol 20% peak area by GC-MS + minor others), vapor phase by SPME:ethylacetate, mathylpyridine, acetic acid)

[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)
21/Feb./2016	Typing error - empirical formula corrected from C15H13FNO3S to C15H13F**2**NO3S.

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 **(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

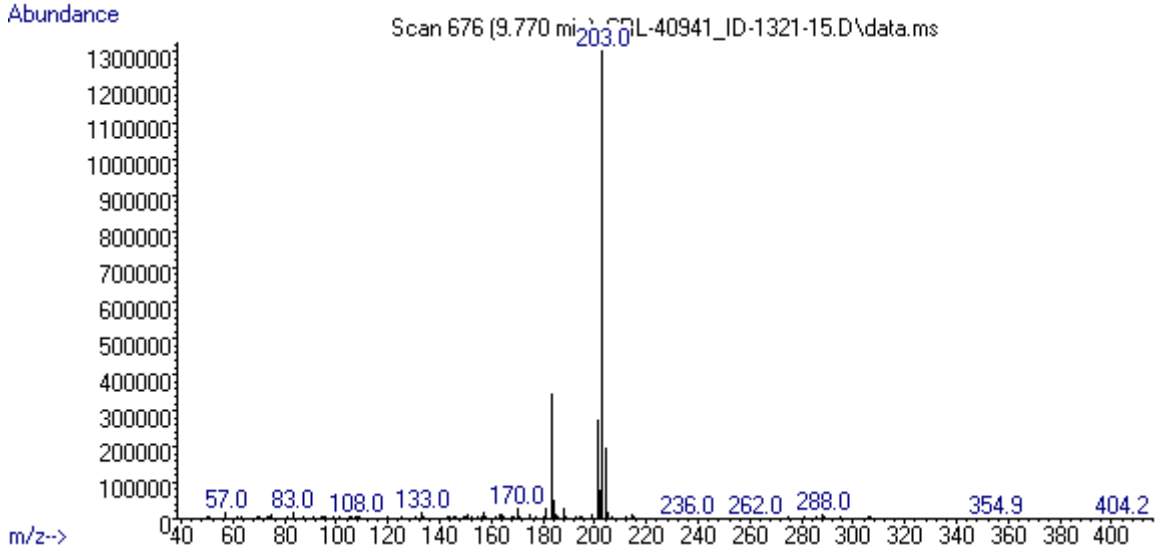
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	partially

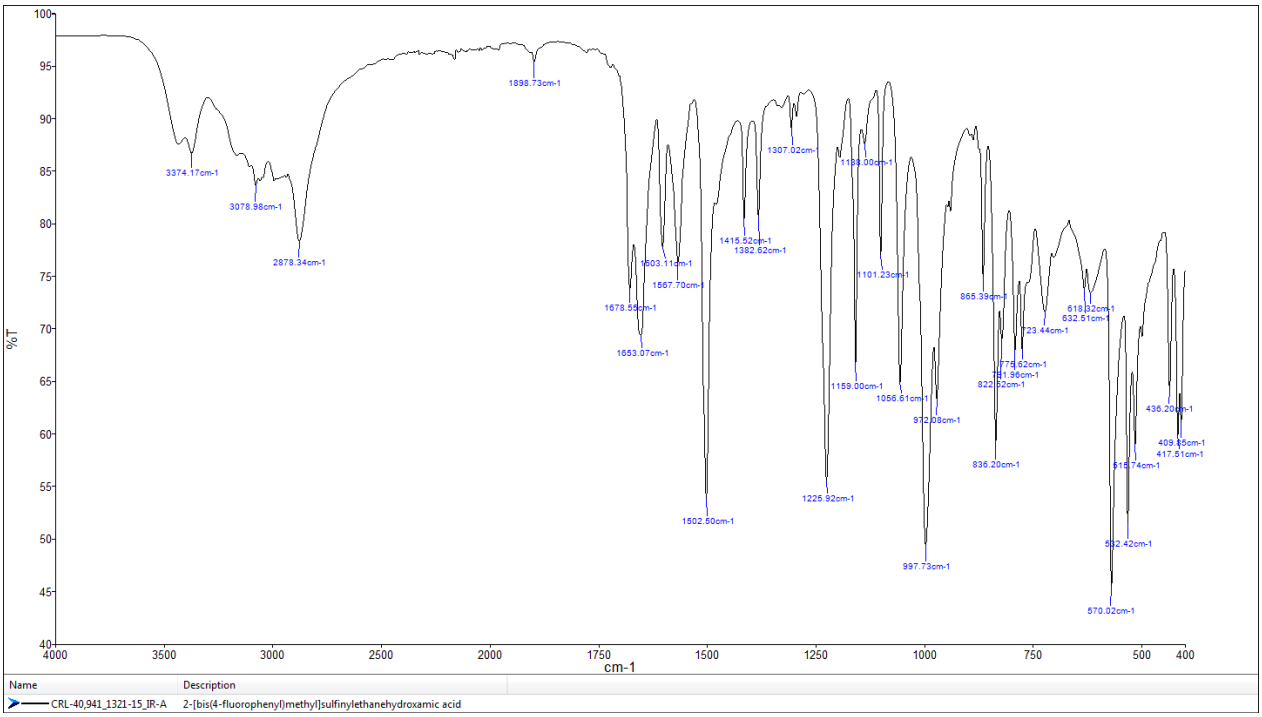
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 9,78 BP(1): 203; BP(2): 183,BP(3) :201,
HPLC-TOF	+	Exact mass (theoretical): 325,0584; measured value Δppm:-4,86; formula:C15H13FNO3S
FTIR-ATR	+	direct measurement (low boiling point solvents evaporated under vacuum)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	(solvents content previously reduced under vacuum)
validation		
other		

ANALYTICAL RESULTS

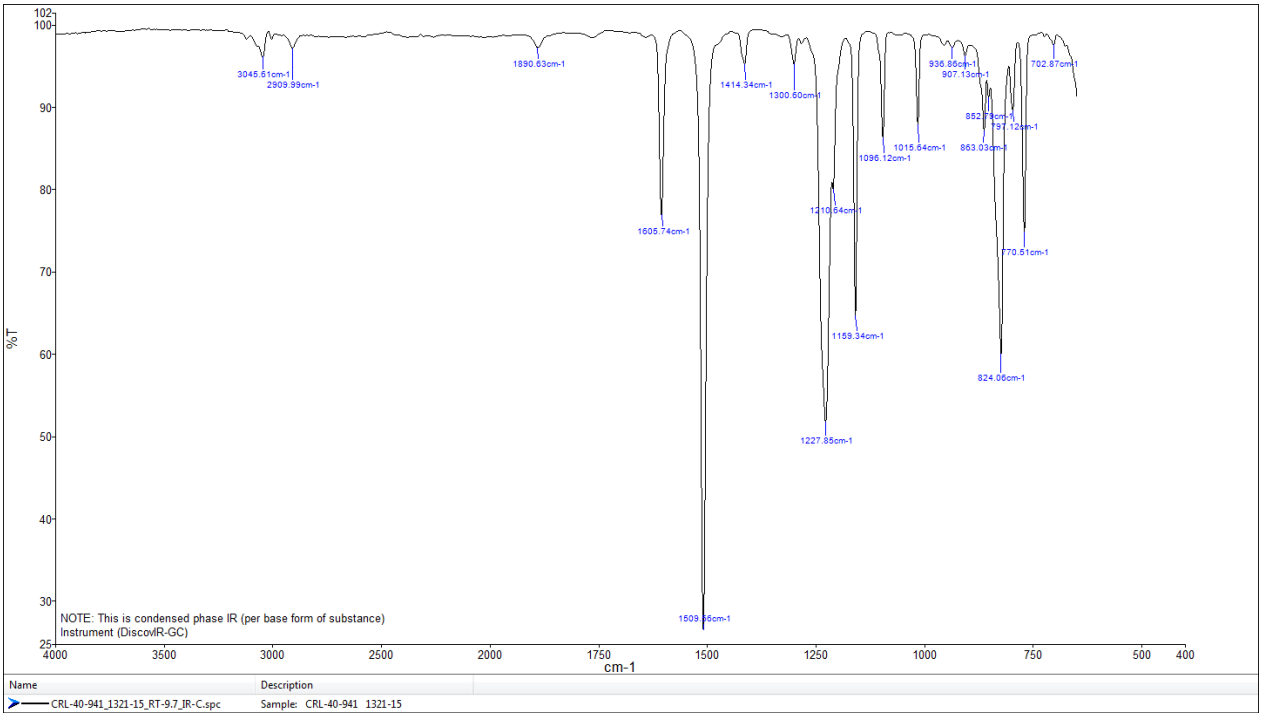
MS (EI)



FTIR-ATR - direct measurement (solvents removed as much as possible)



IR (condensed phase – after chromatographic separation)



TOF REPORT

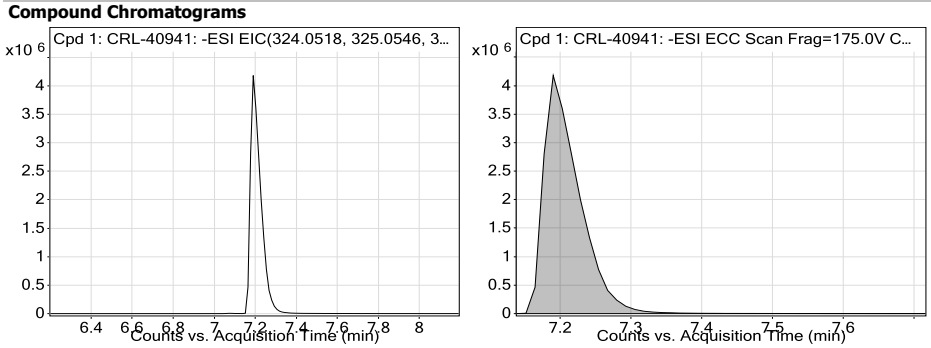
Data File	CRL_40941_1321-15_TOF-po servisu2.d	**Sample Name**	CRL-40941
Sample Type	Sample	**Position**	P1-D4
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-11112015-XDB-C18-ESI-neg.m	**Acquired Time**	11/6/2015 8:10:01 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

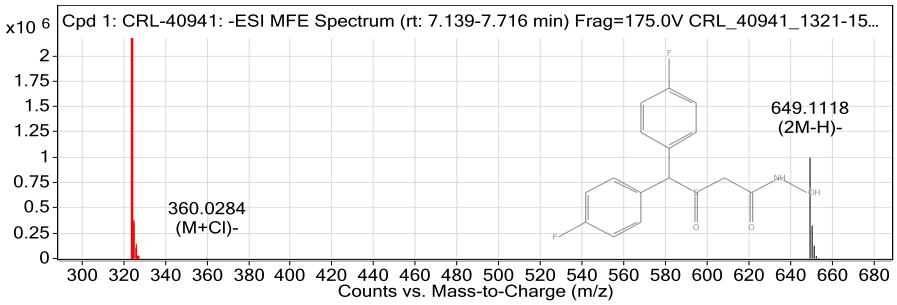
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 1: CRL-40941	CRL-40941	7.199	325.06

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
CRL-40941	324.0527	7.199	325.06	7.199	C15 H13 F2 N O3 S	325.0584	-4.86

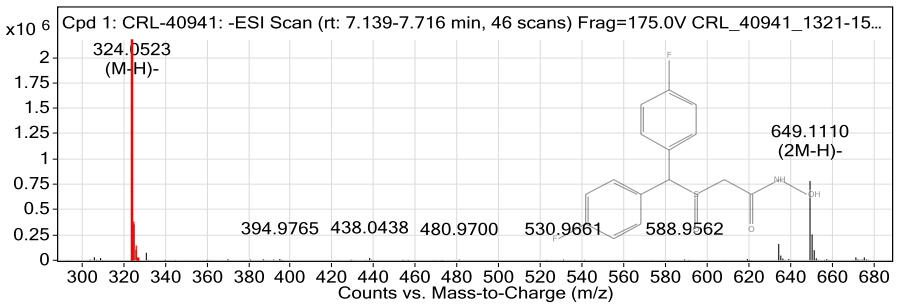
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

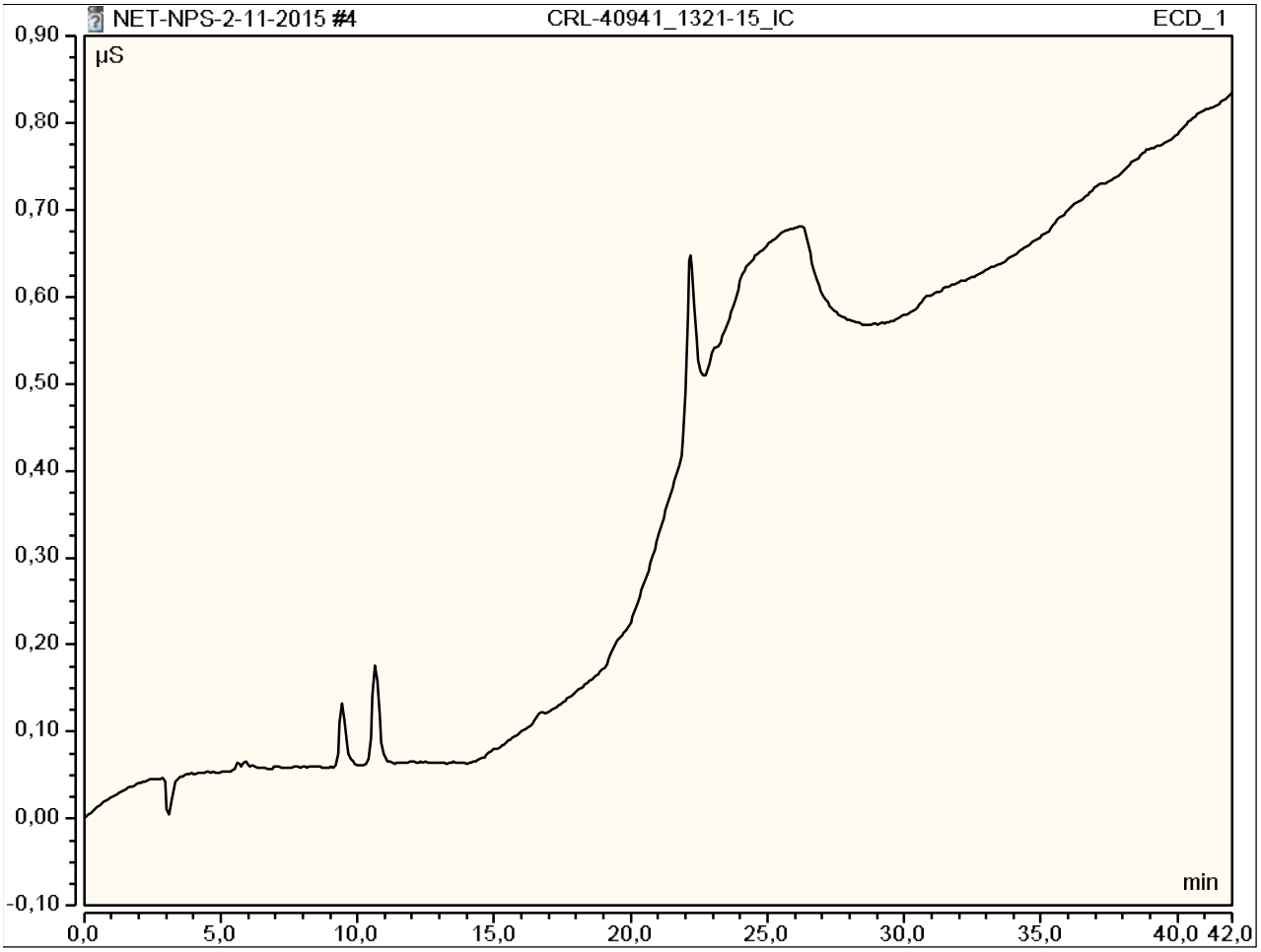
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
324.0527	-1	2174794	C15 H13 F2 N O3 S	(M-H)-
325.056	-1	356232.11	C15 H13 F2 N O3 S	(M-H)-
326.0515	-1	105087.96	C15 H13 F2 N O3 S	(M-H)-
327.0529	-1	11484.37	C15 H13 F2 N O3 S	(M-H)-
360.0284	-1	4463.79		(M+Cl)-
649.1118	-1	995539.69		(2M-H)-
650.1145	-1	324923.93		(2M-H)-
651.1115	-1	127247.96		(2M-H)-
652.1116	-1	24069.59		(2M-H)-
653.1106	-1	4307.22		(2M-H)-

--- End Of Report ---

Peak Integration Report

Sample Name:	**CRL-40941_1321-15_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**02-nov-2015 / 15:22**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount n.a.
		TOTAL:		0,00	0,00	0,00



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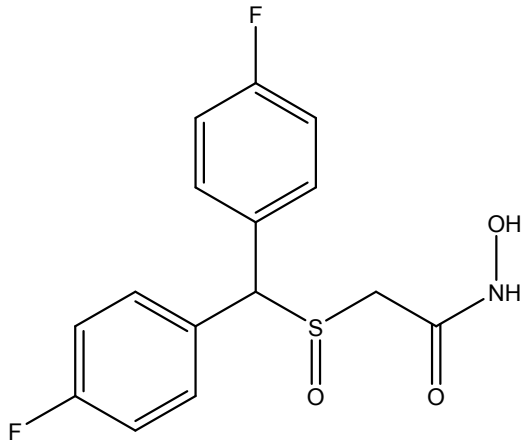
REPORT

Sample ID:	**1321-15**
Our notebook code:	P-1321-15
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:	2-((bis(4-fluorophenyl)methyl)sulfinyl)-N-hydroxyacetamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure by NMR, it contains some other compound(s), as evident from the redundant peaks observed in 1H NMR (at 10.4, 9.4, 3.5 ppm) and 13C NMR (at 168.0, 53.9 ppm).
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 24, 2015

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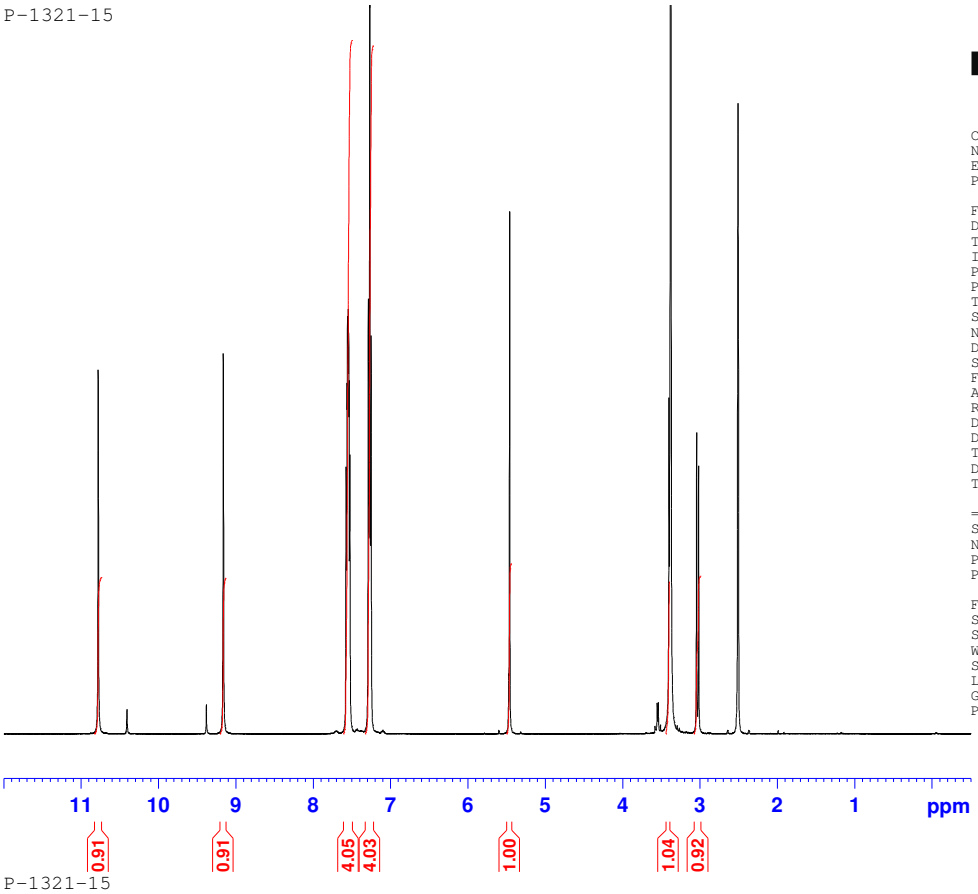
P-1321-15



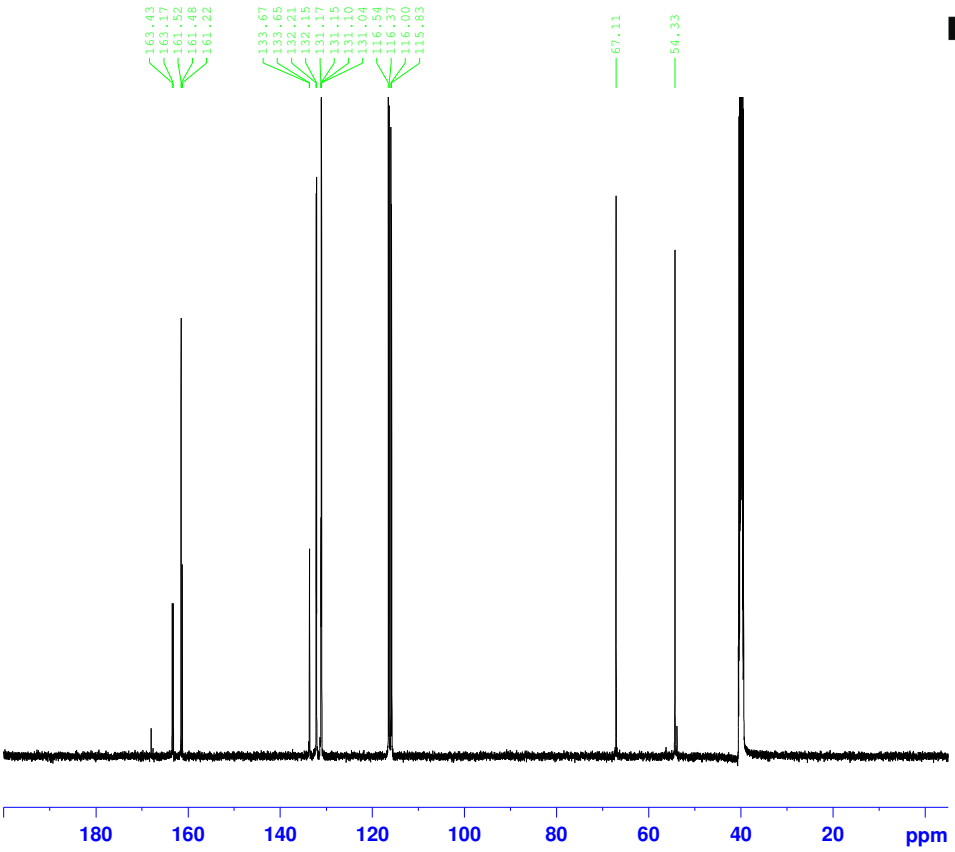
Current Data Parameters
NAME P-1321-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151222
Time 17.26
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 71.8
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-1321-15



Current Data Parameters
NAME P-1321-15
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151222
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 5120
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