



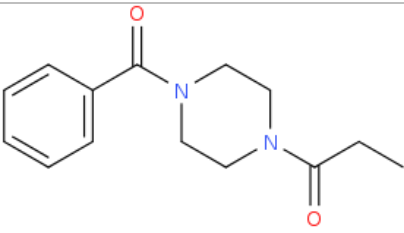
## ANALYTICAL REPORT<sup>1</sup>

### Sunifiram (C<sub>14</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>)

#### 1-(4-benzoylpiperazin-1-yl)propan-1-one

Remark – other NPS detected: **none**

Sample ID:	1537-16
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	2/16/2016
Date of entry (M/D/Y) into NFL database:	8/12/2016
Report updates (if any) will be published here:	<a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a>

Substance identified - structure <sup>2</sup> (base form)	
Systematic name	1-(4-benzoylpiperazin-1-yl)propan-1-one
Other names	DM-235
Formula (per base form)	C <sub>14</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub>
M <sub>w</sub> (g/mol)	246,3
Salt form/anions detected	base
StdInChIKey	DGOWDUFJCINDGI-UHFFFAOYSA-N
Compound Class	Piperazine derivatives
Other NPS detected	none
Add.info (purity..)	pure by HPLC-TOF, GC-MS, NMR

<sup>1</sup> 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.

<sup>2</sup> 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 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 6.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235 °C, source and quadropole temperatures 280 °C and 180 °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 (N<sub>2</sub>) 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**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 °C. Chromatographic separation as above (**1**). Split MS : IR = 1: 9.

MSD source EI = 70 eV. GC-MS transfer line T= 235 °C, source and quadropole temperatures 280 °C and 180 °C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.

IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm<sup>-1</sup>.

**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

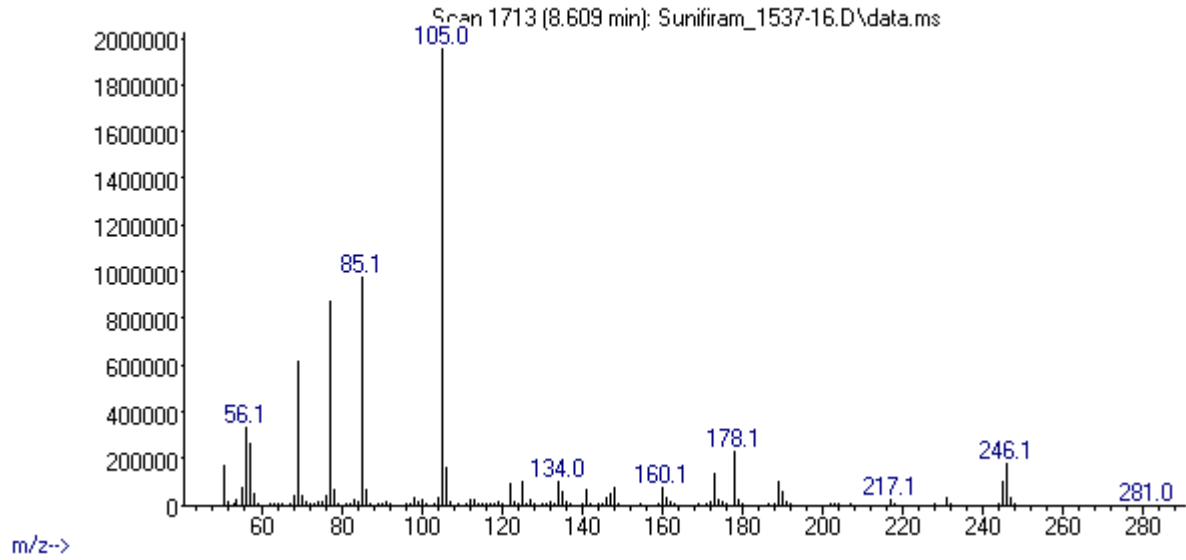
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	soluble
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8,61 BP(1): 105; BP(2): 85,BP(3) :77,
HPLC-TOF	+	Exact mass (theoretical): 246,1368; measured value Δppm:0,55; formula:C14H18N2O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		sold as nootropic

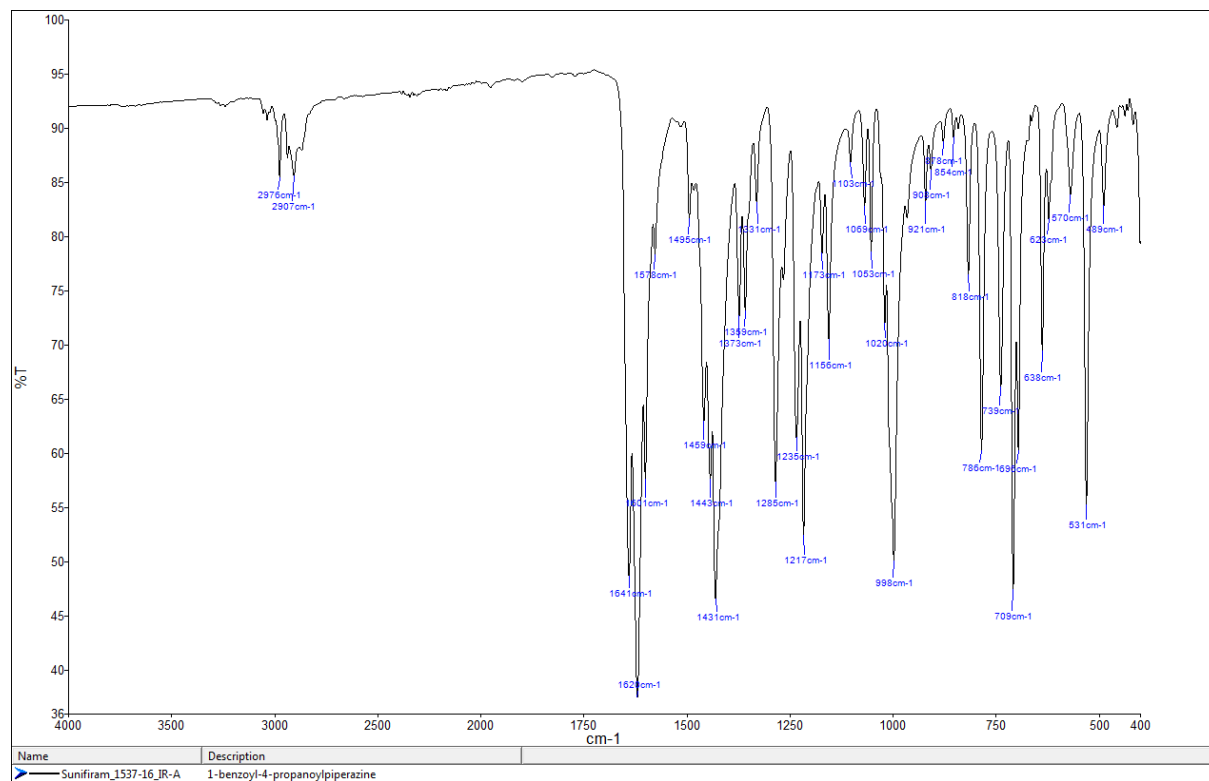
# ANALYTICAL RESULTS

MS (EI)

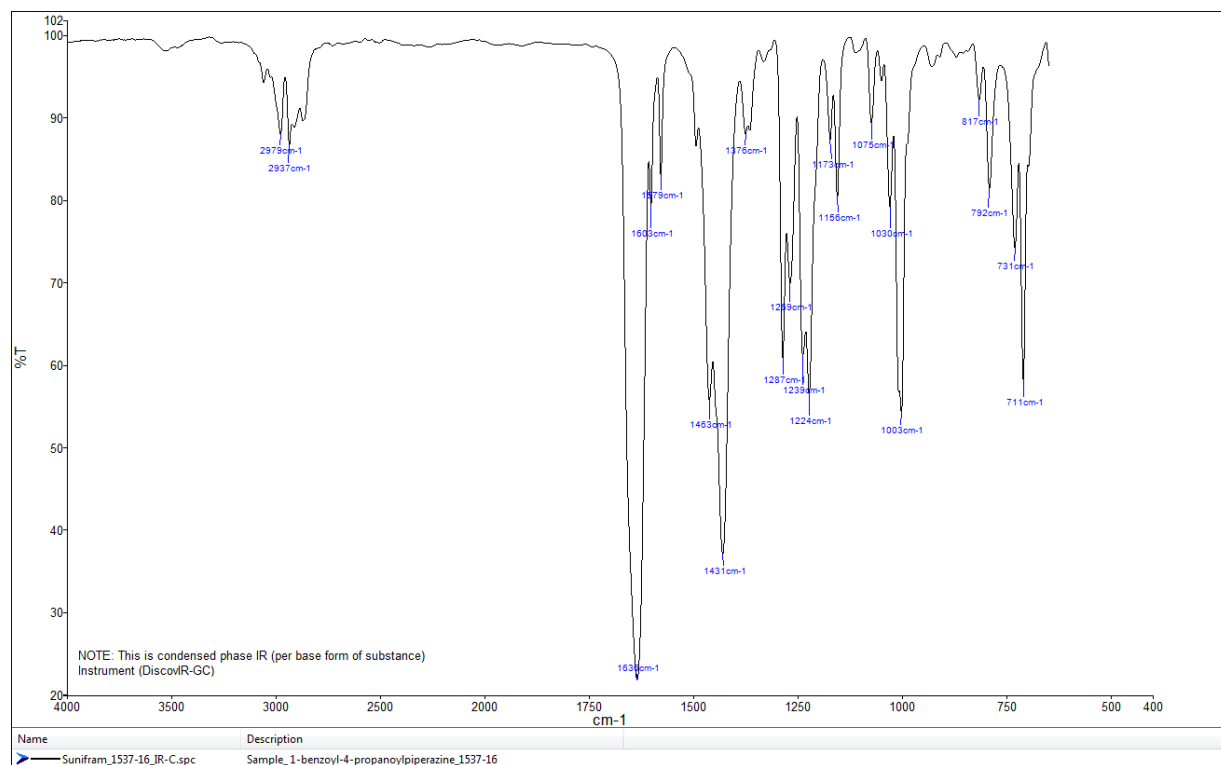
Abundance



## FTIR-ATR - direct measurement (sample as received)



## IR (condensed phase – after chromatographic separation)



# TOF REPORT

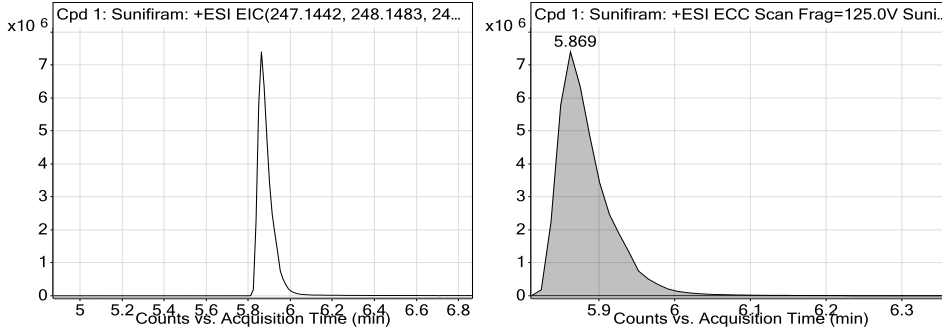
<b>Data File</b>	Sunifiram_1537-16_TOF.d	<b>Sample Name</b>	ID_1537-16
<b>Sample Type</b>	Sample	<b>Position</b>	P1-D5
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	general-1512015-XDB-C18-ESI-poz-pod.m	<b>Acquired Time</b>	2/19/2016 10:55:27 AM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	Drugs_NFL.m
<b>Comment</b>	extract in MeOH		

## Compound Table

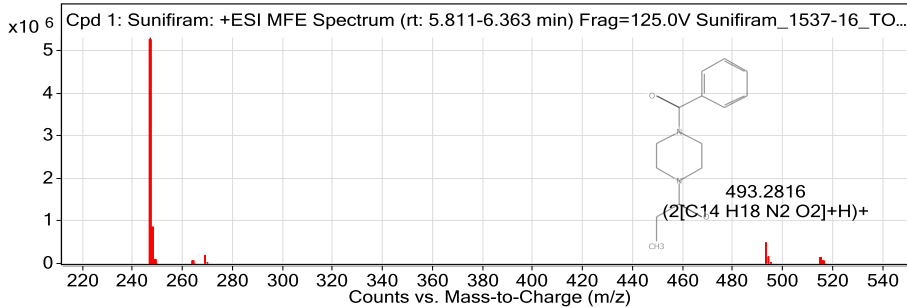
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Sunifiram	Sunifiram	C14 H18 N2 O2	5.869	246.1367

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Sunifiram	247.1442	5.869	246.1367	5.87	C14 H18 N2 O2	246.1368	0.55

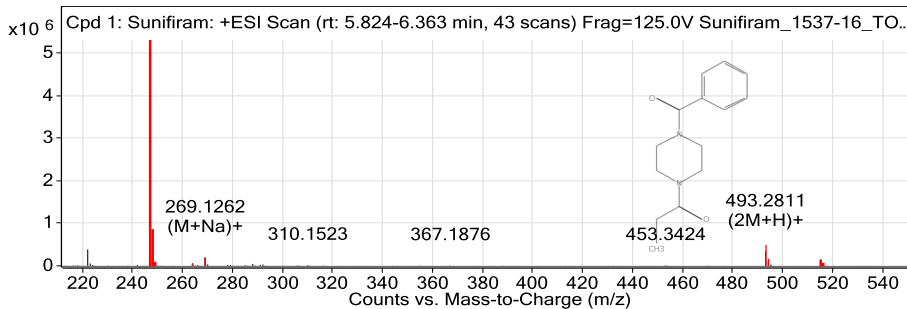
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

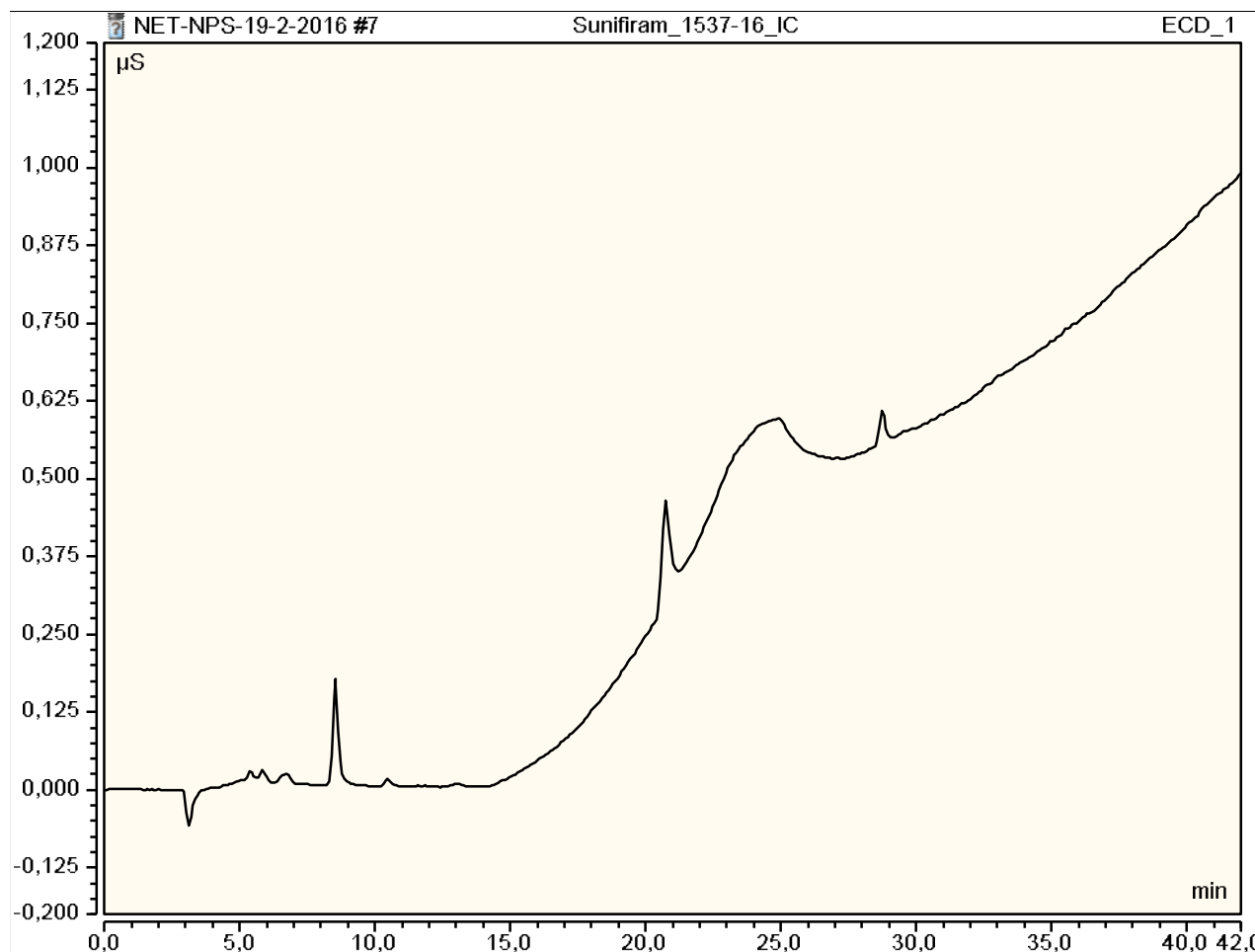
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
247.1442	1	5301197	C14 H18 N2 O2	(M+H)+
248.1483	1	793535.25	C14 H18 N2 O2	(M+H)+
249.1502	1	88213.26	C14 H18 N2 O2	(M+H)+
264.1708	1	63995.17	C14 H18 N2 O2	(M+NH4)+
269.1265	1	203465.7	C14 H18 N2 O2	(M+Na)+
270.1294	1	32625.64	C14 H18 N2 O2	(M+Na)+
493.2816	1	493641.88	C14 H18 N2 O2	(2M+H)+
494.2842	1	155728	C14 H18 N2 O2	(2M+H)+
515.2629	1	136883.61	C14 H18 N2 O2	(2M+Na)+
516.2656	1	41687.09	C14 H18 N2 O2	(2M+Na)+

--- End Of Report ---

### Peak Integration Report

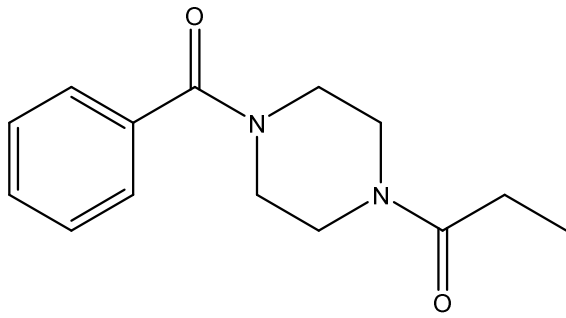
Sample Name:	Sunifiram_1537-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	22-feb-2016 / 15:06	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height $\mu\text{S}$	Amount n.a.
		TOTAL:		0,00	0,00	0,00





## REPORT

Sample ID:	<b>1537-16</b>
Our notebook code:	P-1537-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- <i>d</i> <sub>6</sub>
NMR experiments:	<sup>1</sup> H, <sup>13</sup> C, <sup>1</sup> H- <sup>1</sup> H <i>gs</i> -COSY, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HSQC, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HMBC, <sup>1</sup> H- <sup>15</sup> N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	1-(4-benzoylpiperazin-1-yl)propan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure according to NMR.
Supporting information:	Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	May 9, 2016



P-1537-16

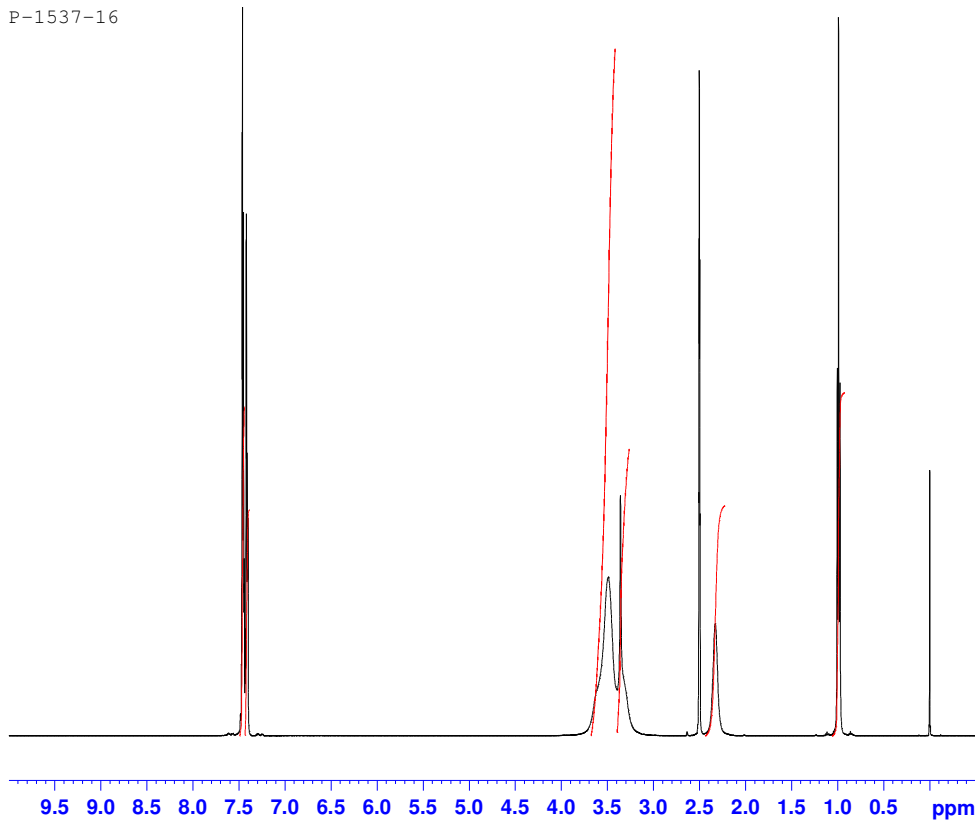


Current Data Parameters  
 NAME P-1537-16  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160418  
 Time 22.37  
 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 90.5  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 300.0 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 500.1300039 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



P-1537-16



Current Data Parameters  
 NAME P-1537-16  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20160419  
 Time 1.49  
 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 300.0 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  
 FCPD2 80.00 usec  
 PLW2 26.00000000 W  
 PLW12 0.32179001 W  
 PLW13 0.16186000 W

F2 - Processing parameters  
 SI 32768  
 SF 125.7579241 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

