



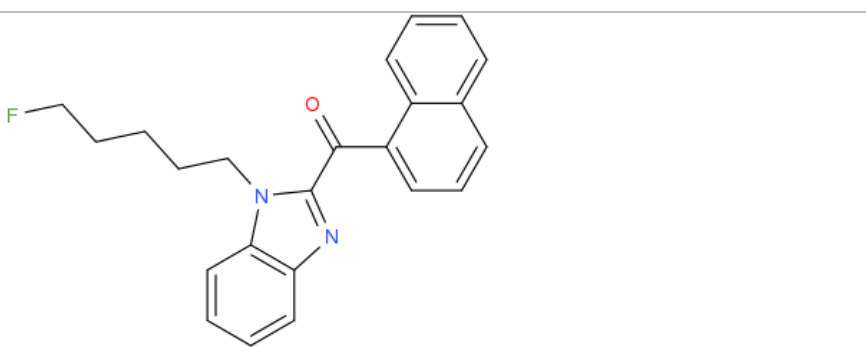
## ANALYTICAL REPORT<sup>1</sup>

### FUBIMINA (C<sub>23</sub>H<sub>21</sub>FN<sub>2</sub>O)

#### (1-(5-fluoropentyl)-1H-benzo[d]imidazol-2-yl)(naphthalen-1-yl)methanone

Remark – other NPS detected: **none**

Sample ID:	1198-15
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	7/22/2015
Date of entry (M/D/Y) into NFL database:	7/22/2015
Report (updates) 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-(5-fluoropentyl)-1H-benzo[d]imidazol-2-yl)(naphthalen-1-yl)methanone
Other names	AM-2201 benzimidazole analog, BIM-2201, BZ-2201, FTHJ
Formula (per base form)	C <sub>23</sub> H <sub>21</sub> FN <sub>2</sub> O
M <sub>w</sub> (g/mol)	360,4
Salt form	base
StdInChIKey	KUESSZMROAFKQJ-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	pure - with the applied methods

<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 (RT=9.53 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 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by 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 2.8 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 (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<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 (40) to 550 amu.

IR (condensed 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

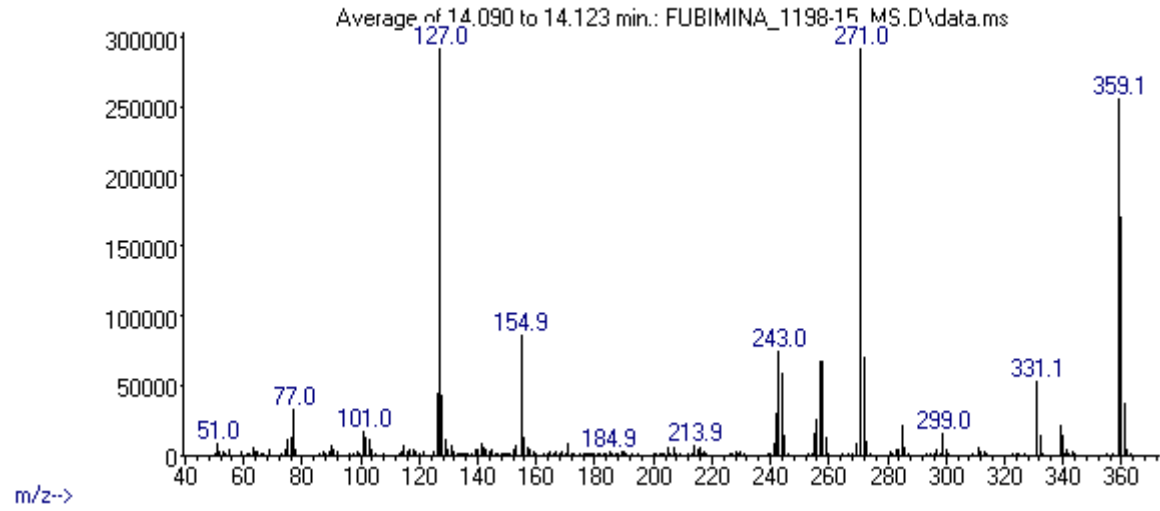
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	soluble
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 14,12 BP(1): 271; BP(2): 127,BP(3) :359,
HPLC-TOF	+	Exact mass (theoretical): 360,1638; measured value Δppm:-0,35; formula:C23H21FN2O
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR	+	
validation		consistent with existing published data
other		

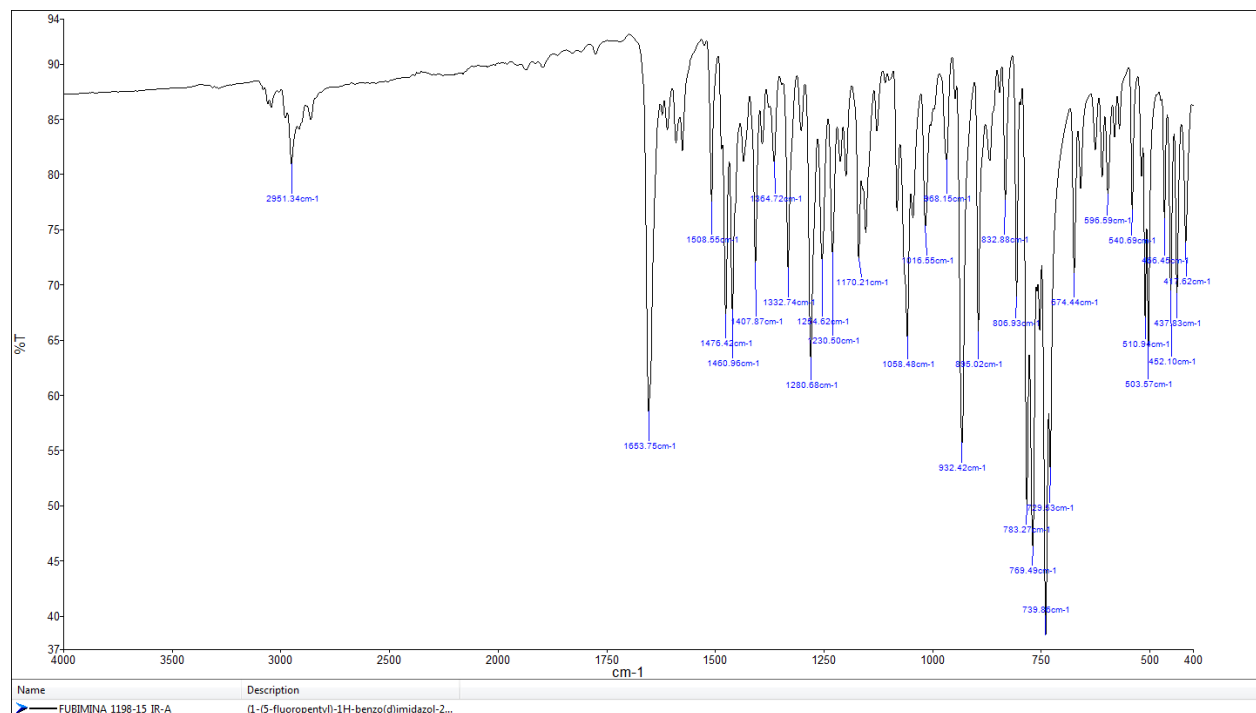
# ANALYTICAL RESULTS

MS (EI)

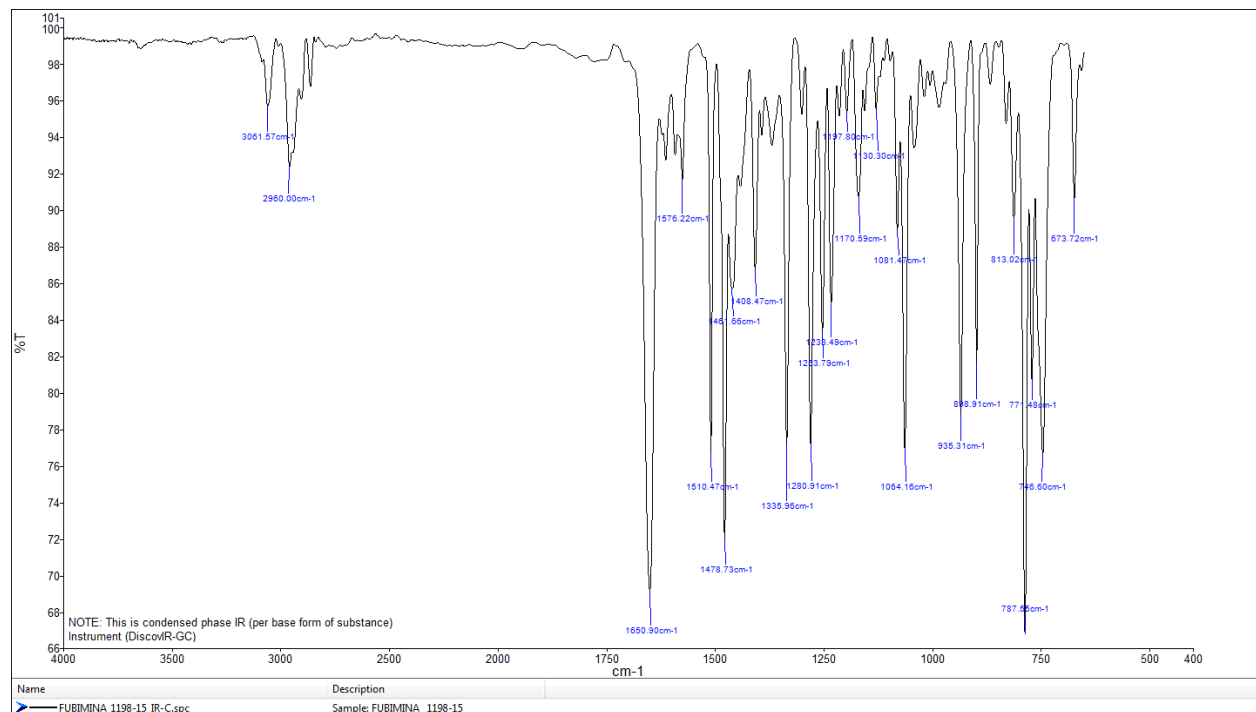
Abundance



## FTIR-ATR - direct measurement



## IR (condensed phase)



# TOF REPORT

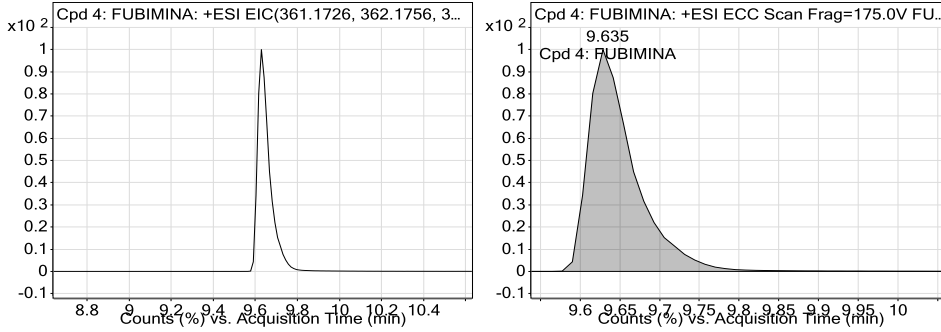
<b>Data File</b>	FUBIMINA_1198-15_TOF.d	<b>Sample Name</b>	FUBIMINA
<b>Sample Type</b>	Sample	<b>Position</b>	P1-D8
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	droge general-13-5-2015-XDB-C18-ESI-poz.m	<b>Acquired Time</b>	7/27/2015 12:07:27 PM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	Drugs_NFL.m
<b>Comment</b>	extract in MeOH		

**Compound Table**

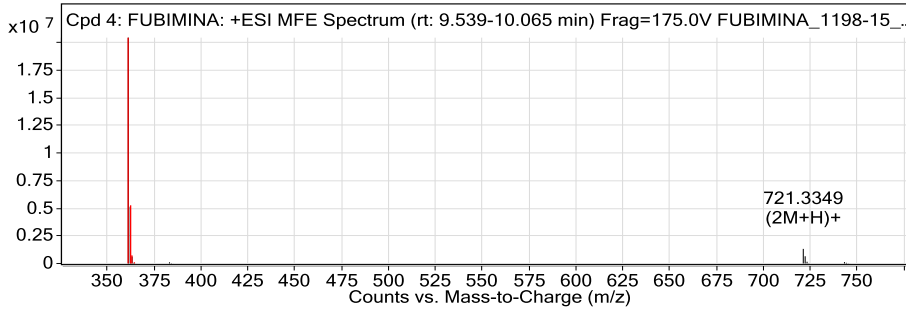
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 4: FUBIMINA	FUBIMINA	9.635	360.1642

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
FUBIMINA	361.1715	9.635	360.1642	9.635	C23 H21 F N2 O	360.1638	-1.07

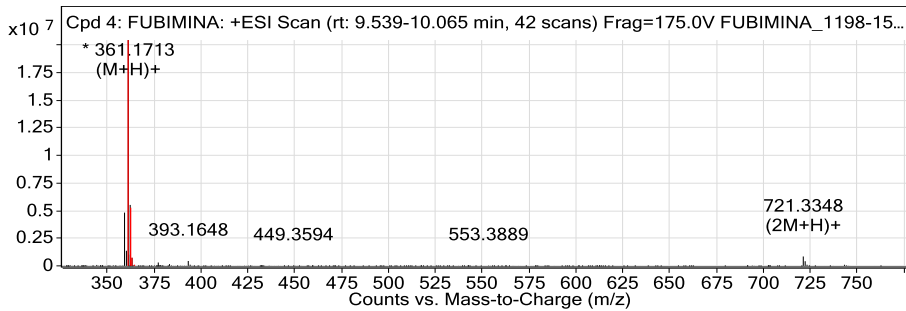
**Compound Chromatograms**



**MFE MS Zoomed Spectrum**



**MS Zoomed Spectrum**



**MS Spectrum Peak List**

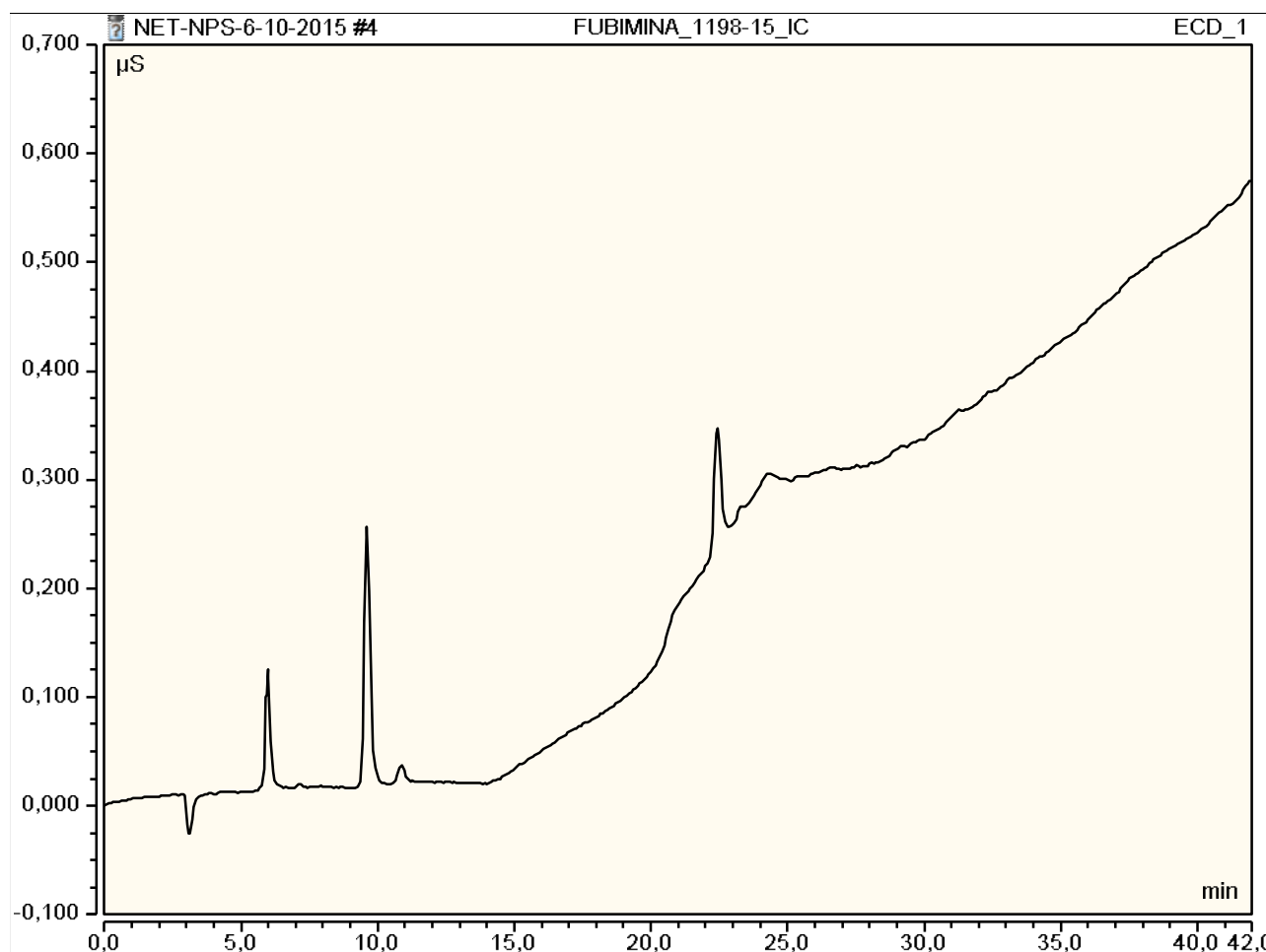
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
361.1715	1	20464124	C23 H21 F N2 O	(M+H)+
362.1746	1	5149985.32	C23 H21 F N2 O	(M+H)+
363.1782	1	625994.28	C23 H21 F N2 O	(M+H)+
364.1809	1	55725.41	C23 H21 F N2 O	(M+H)+
383.1532	1	106589.42		(M+Na)+
721.3349	1	1322248.5		(2M+H)+
722.3384	1	653663.69		(2M+H)+
723.3411	1	157609.96		(2M+H)+
743.3163	1	124902.77		(2M+Na)+
744.3193	1	60406.82		(2M+Na)+

--- End Of Report ---

### Peak Integration Report

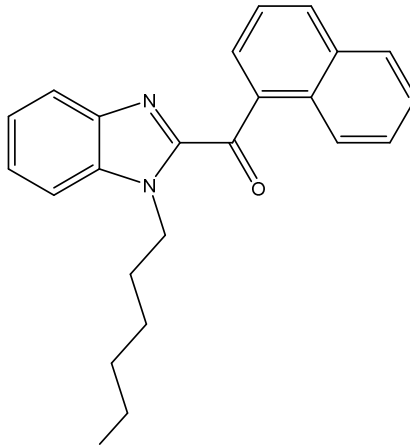
Sample Name:	FUBIMINA_1198-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	06-okt-2015 / 10:32	Run Time:	41,99

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>1198-15</b>
Our notebook code:	P-1198-15
NMR sample preparation:	15 mg dissolved in 0.7 mL CDCl <sub>3</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 with chemical name:	 <p>(1-(5-fluoropentyl)-1H-benzo[d]imidazol-2-yl)(naphthalen-1-yl)methanone</p>
Comments:	<ul style="list-style-type: none"><li>- Structure elucidation based on 1D and 2D NMR spectra</li><li>- Compound is pure by NMR, possibly containing a few % of unknown organic impurities (signals in <sup>1</sup>H NMR at 2.17 and 1.25).</li></ul>
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:	October 13, 2015



P-1198-15

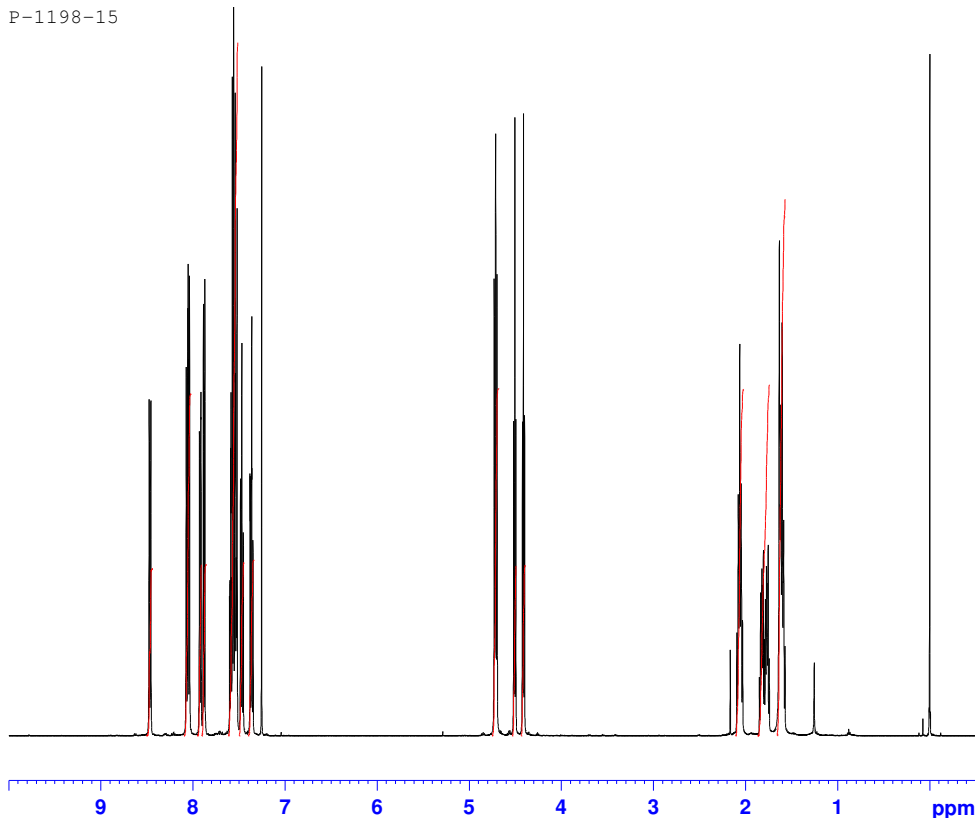


Current Data Parameters  
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 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20150821  
 Time 19.29  
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 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 101  
 DW 48.400 usec  
 DE 6.50 usec  
 TE 296.0 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 500.1300163 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



P-1198-15



Current Data Parameters  
 NAME P-1198-15  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20150821  
 Time 22.41  
 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 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 125.7577925 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

