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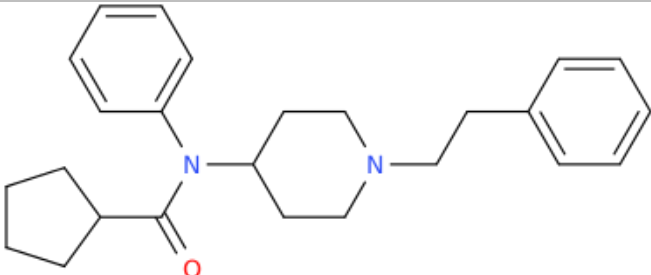
ANALYTICAL REPORT

Cyclopentyl-F (C₂₅H₃₂N₂O)

N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]cyclopentanecarboxamide

Remark – other NPS detected: **none**

Sample ID:	1661-16
Sample description:	powder
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	8/30/2016
Date of entry (M/D/Y) into NFL database:	3/6/2017
Report ¹ 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	N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]cyclopentanecarboxamide
Other names	Cyclopentylfentanyl; Cyclopentyl fentanyl, CPF
Formula (per base form)	C ₂₅ H ₃₂ N ₂ O
M _w (g/mol)	376,54
Salt form/anions detected	oxalate
StdInChIKey (per base form)	PEASFKSPITUZGT-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	

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)
18/05/2017	Empirical formula corrected.

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 7.1 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 (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 °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⁻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

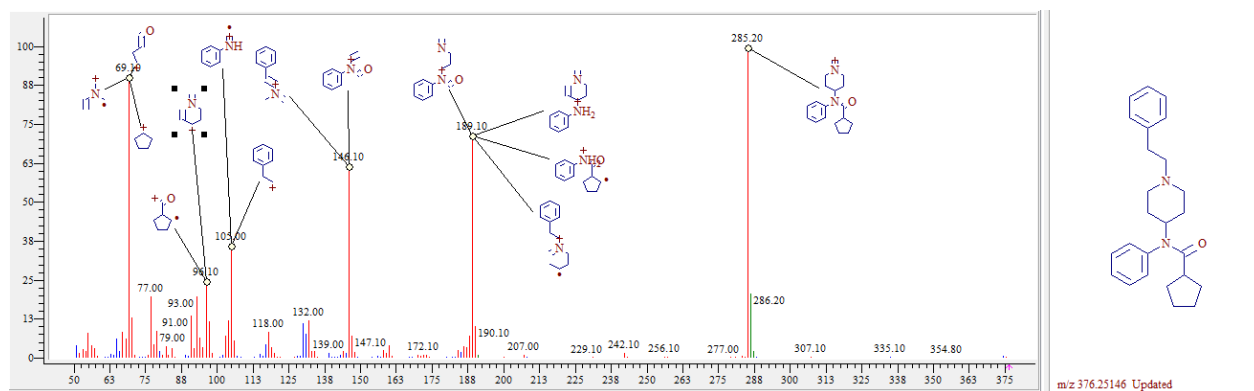
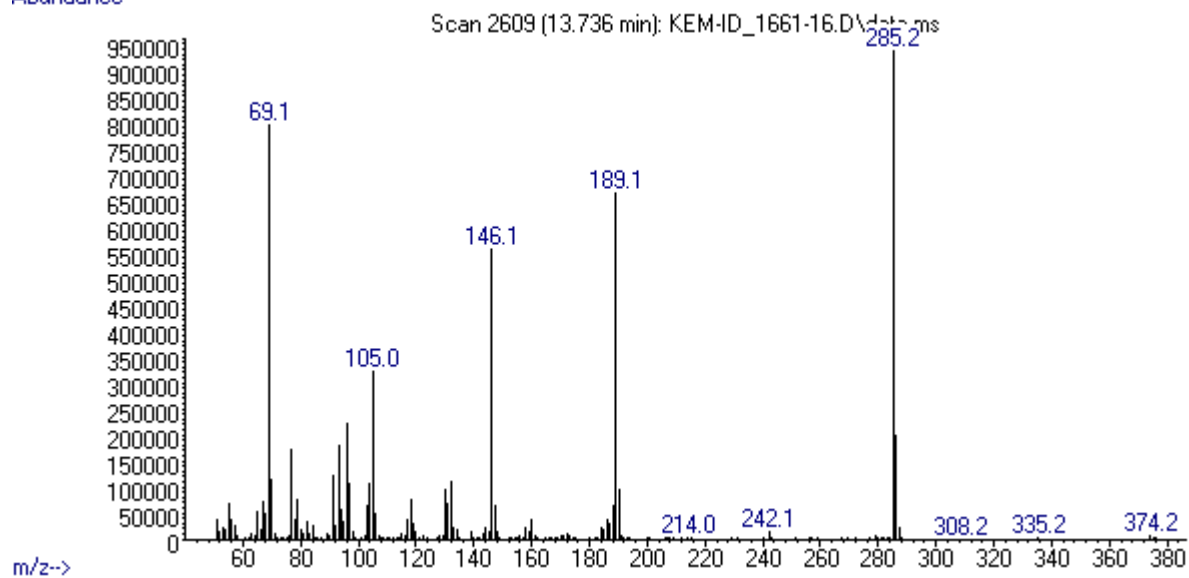
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,74 BP(1): 285; BP(2): 69,BP(3) :189,
HPLC-TOF	+	Exact mass (theoretical): 376,2515; measured value Δppm:-0,56; formula:C ₂₅ H ₃₂ N ₂ O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		MS consistent by the one published in EMCDDA EDND by Sweden
other		

ANALYTICAL RESULTS

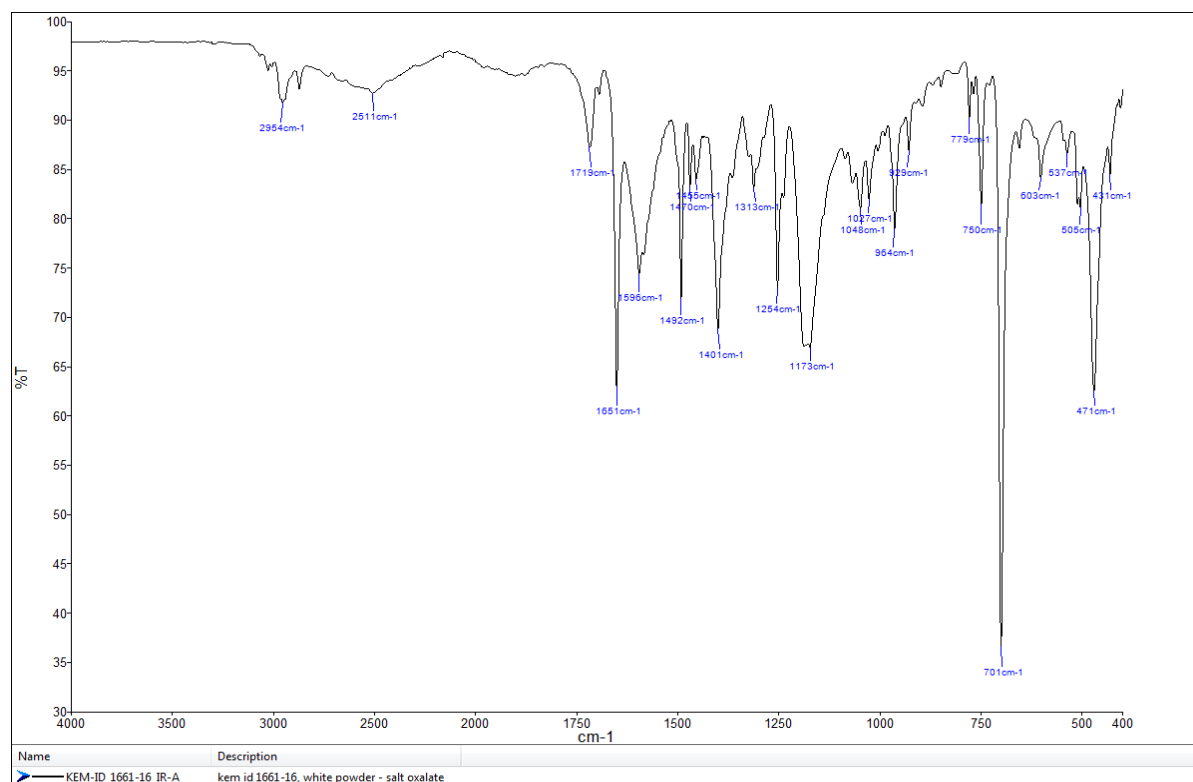
MS (EI)

Abundance

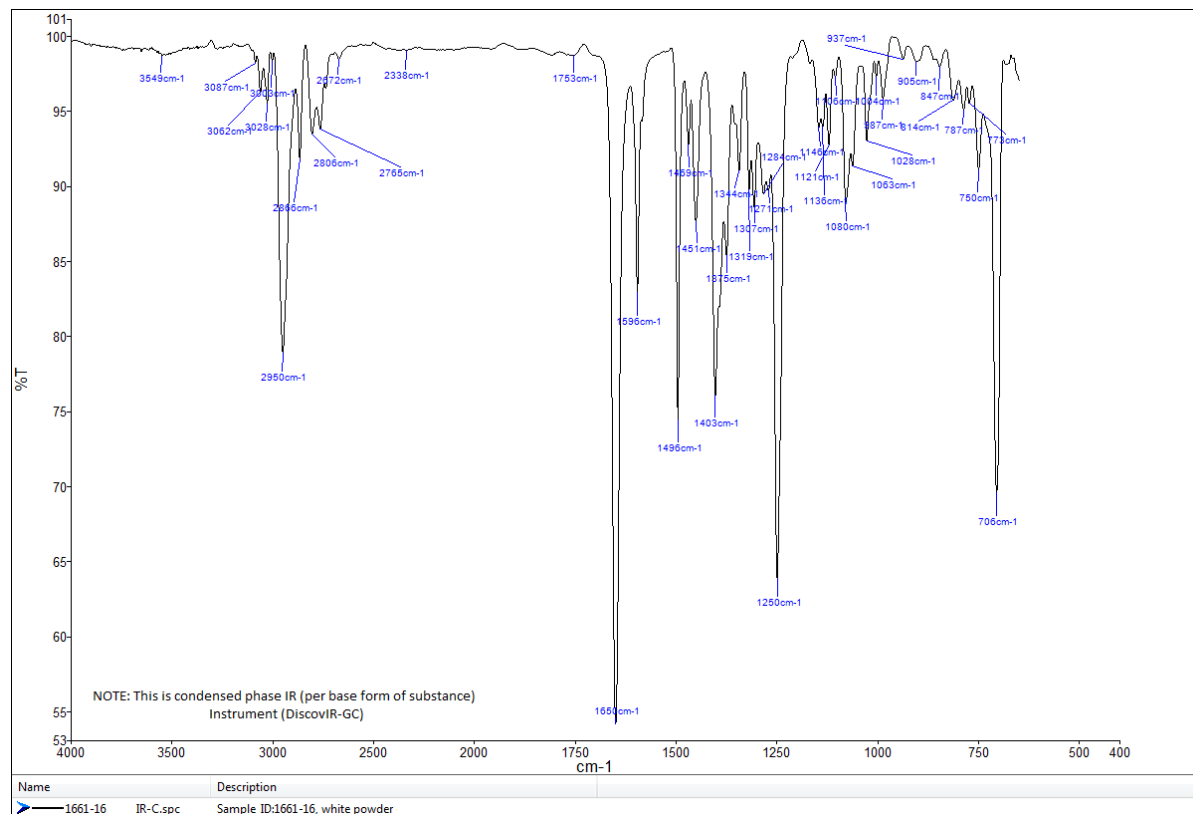


MS-explained (red and green labeled peaks)

FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

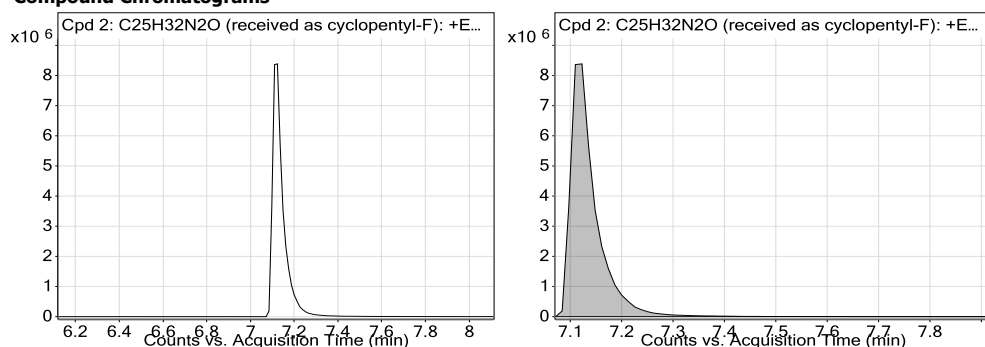
Data File	1661-16_TOF.d	Sample Name	ID_1661-16
Sample Type	Sample	Position	P1-A4
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-24_08_2016-XDB-C18-ESI-poz-soft.m	Acquired Time	9/1/2016 8:14:33 AM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

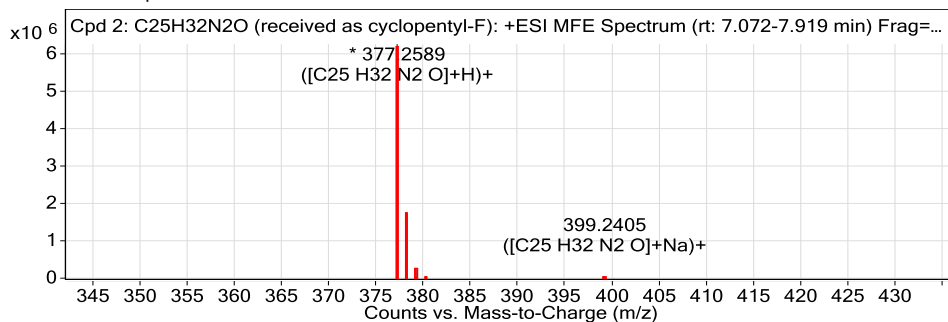
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: C25H32N2O (received as cyclopentyl-F)	C25H32N2O (received as cyclopentyl-F)	C25 H32 N2 O	7.12	376.2517

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
C25H32N2O (received as cyclopentyl-F)	377.2589	7.12	376.2517	7.12	C25 H32 N2 O	376.2515	-0.56

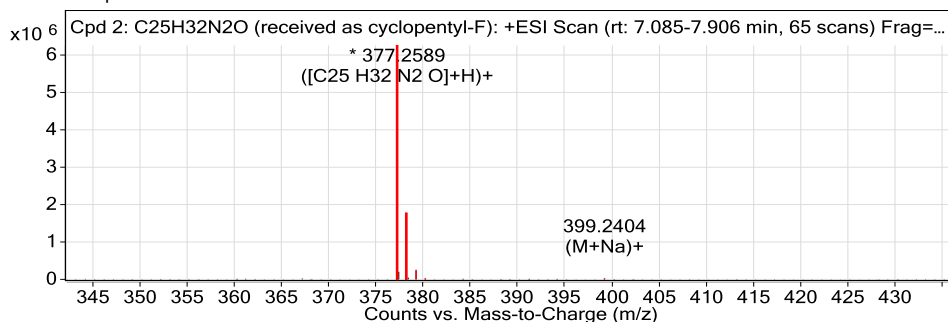
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

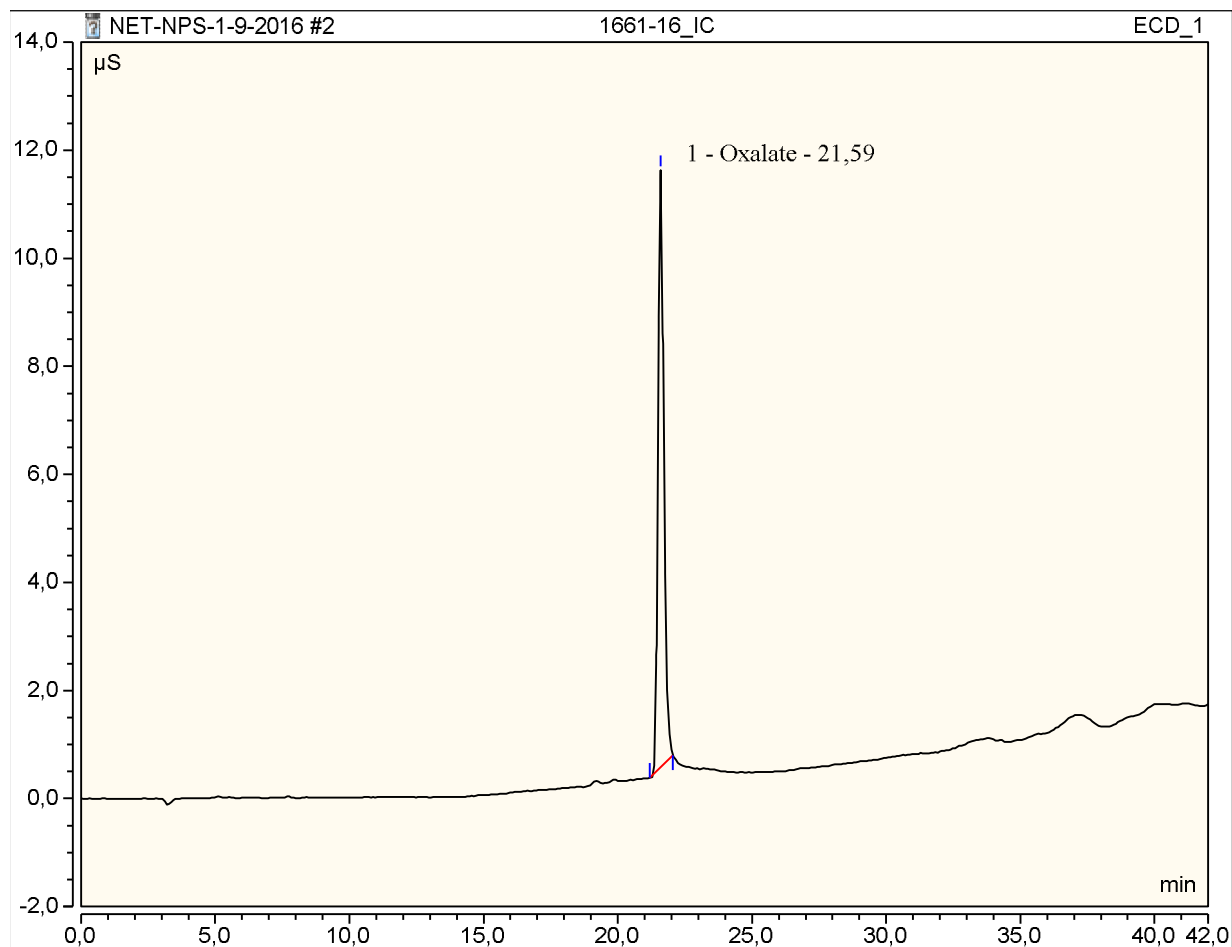
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
377.2589	1	6255873	C25 H32 N2 O	(M+H)+
378.2622	1	1715430.4	C25 H32 N2 O	(M+H)+
379.2655	1	226304.78	C25 H32 N2 O	(M+H)+
380.2683	1	21549.87	C25 H32 N2 O	(M+H)+
381.2667	1	1914.08	C25 H32 N2 O	(M+H)+
399.2405	1	15893.89	C25 H32 N2 O	(M+Na)+
400.244	1	4571.63	C25 H32 N2 O	(M+Na)+
401.244	1	987.13	C25 H32 N2 O	(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	1661-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	01-sep-2016 / 10:31	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	21,59	Oxalate	BMB	2,78	11,05	n.a.
		TOTAL:		2,78	11,05	0,00





REPORT

Sample ID:	1661-16
Our notebook code:	P-1661-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	<i>N</i> -(1-phenethylpiperidin-4-yl)- <i>N</i> -phenylcyclopentanecarboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure as evident by NMR; it contains oxalate (signals in ¹H NMR around 5 and in ¹³C NMR at 164.7 ppm) and also some other minor impurities.
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	April 9, 2017

P-1661-16

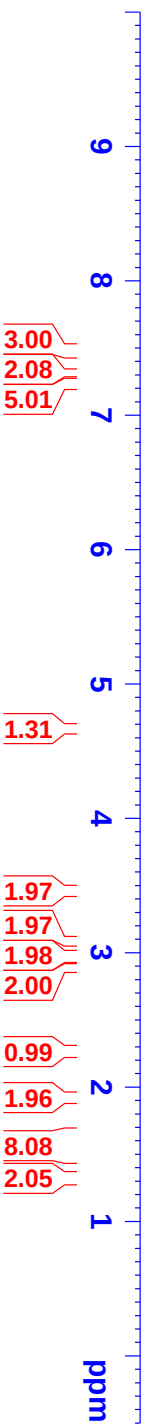


Current Data Parameters
NAME p-1661-16
EXPNO 1
PROCNO 1

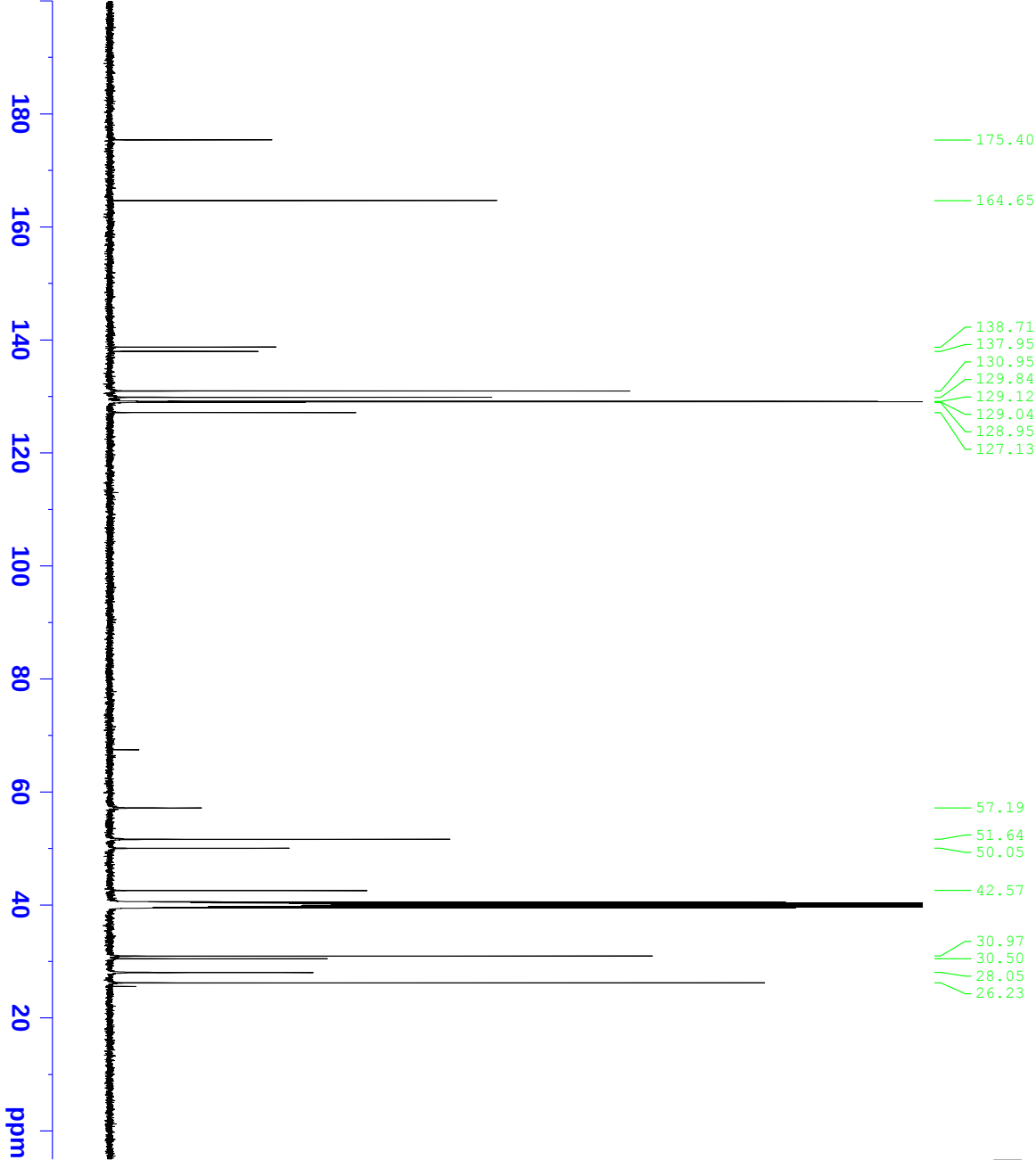
F2 - Acquisition Parameters
Date_ 20160912
Time_ 18.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 71.8
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 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-1661-16



Current Data Parameters
NAME P-1661-16
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160912
Time 22.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 4096
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 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 125.7703637 MHz
NUC1 13C
P1 9.00 usec
PIW1 122.00000000 W

===== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 80.00 usec
PIW2 26.00000000 W
PIW12 0.32179001 W
PIW13 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