



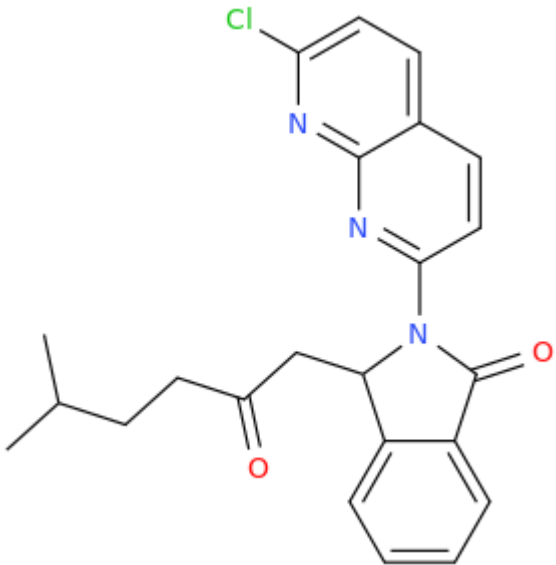
ANALYTICAL REPORT

Pagoclone (C₂₃H₂₂ClN₃O₂)

2-(7-chloro-1,8-naphthyridin-2-yl)-3-(5-methyl-2-oxohexyl)-2,3-dihydro-1H-isoindol-1-one

Remark – other NPS detected: **none**

Sample ID:	2066-19
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	07/06/2019
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	2-(7-chloro-1,8-naphthyridin-2-yl)-3-(5-methyl-2-oxohexyl)-2,3-dihydro-1H-isoindol-1-one
Other names	2-(7-chloro-1,8-naphthyridin-2-yl)-3-(5-methyl-2-oxohexyl)isoindolin-1-one
Formula (per base form)	C ₂₃ H ₂₂ ClN ₃ O ₂
M _w (g/mol)	407,9
Salt form/anions detected	base
StdInChIKey (per base form)	HIUPRQPBWVEQJJ-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>98% purity by ¹ H NMR

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 °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 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

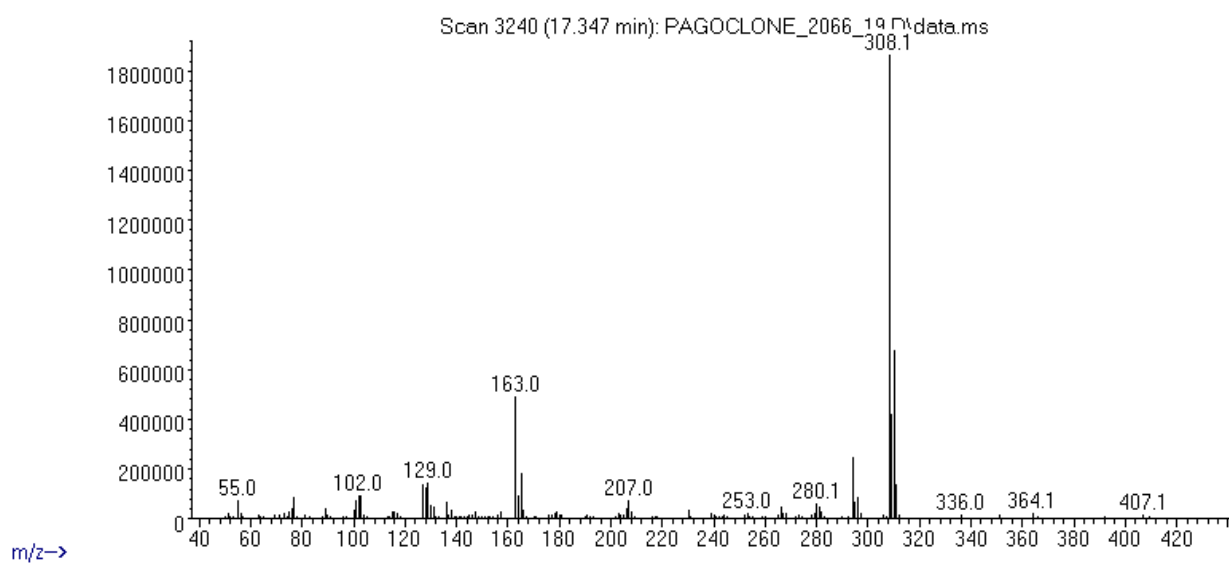
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	partially
H ₂ O	

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 17,35 BP(1): 308; BP(2): 310,BP(3) :163,
HPLC-TOF	+	Exact mass (theoretical): 407,1401; measured value Δppm:-0,8; formula:C23H22ClN3O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

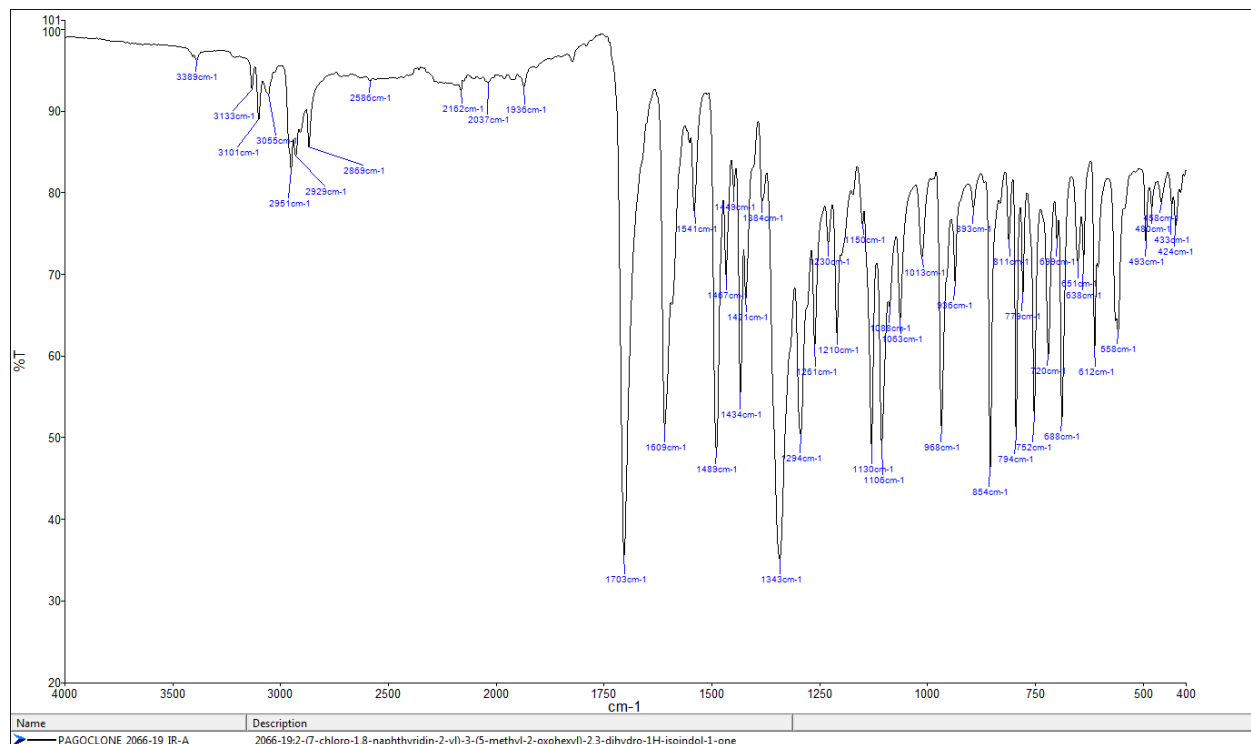
ANALYTICAL RESULTS

MS (EI)

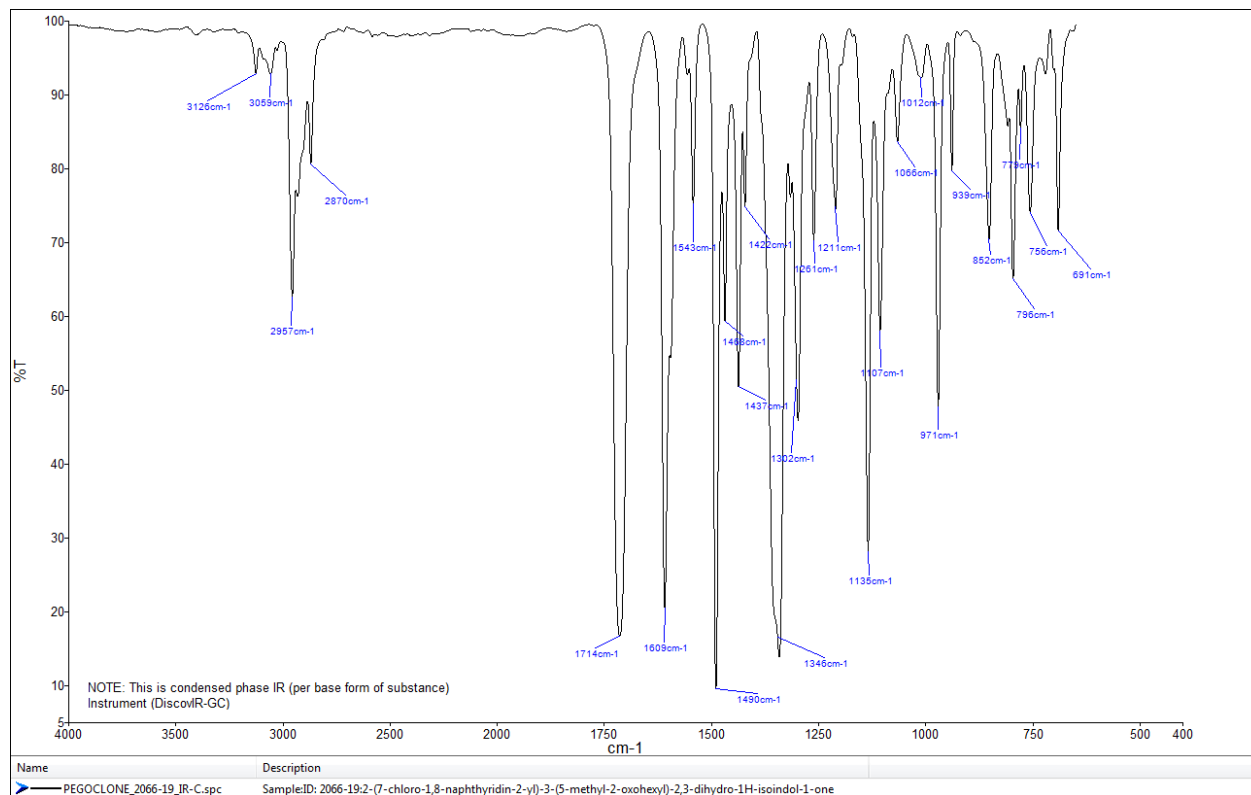
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

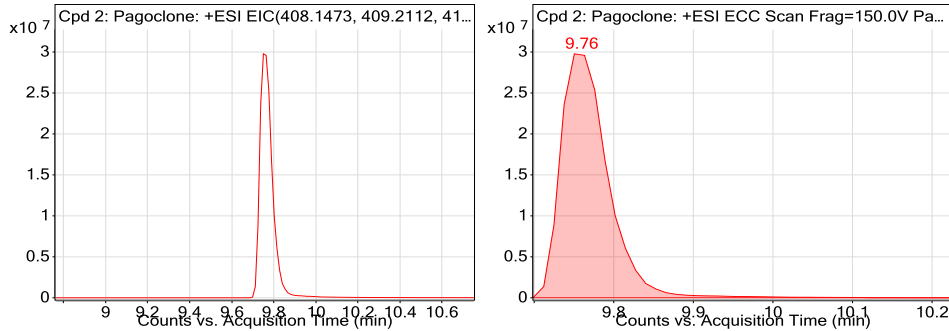
Data File	Pagoclone_2066-19.d	Sample Name	ID-2066-19
Sample Type	Sample	Position	P1-A5
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	Acquired Time	5/13/2019 1:33:10 PM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

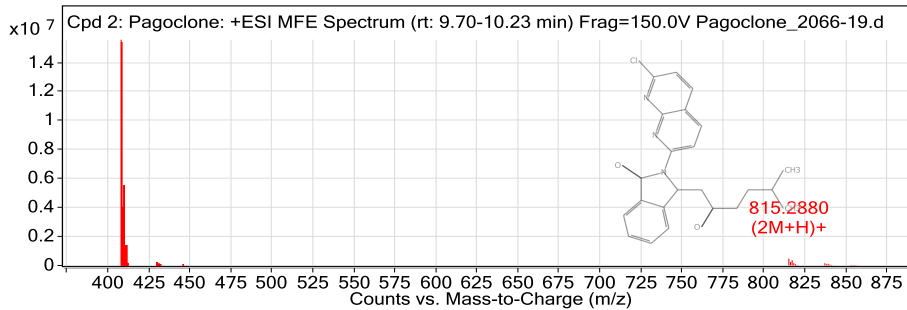
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: Pagoclone	Pagoclone	C23 H22 Cl N3 O2	9.76	407.1404

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Pagoclone	408.1476	9.76	407.1404	9.76	C23 H22 Cl N3 O2	407.1401	-0.8

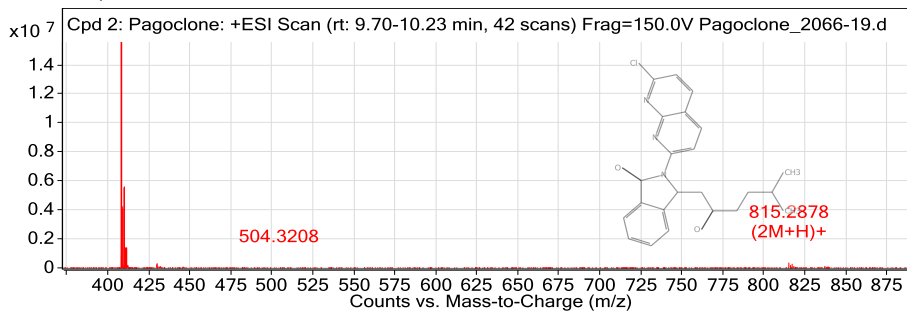
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

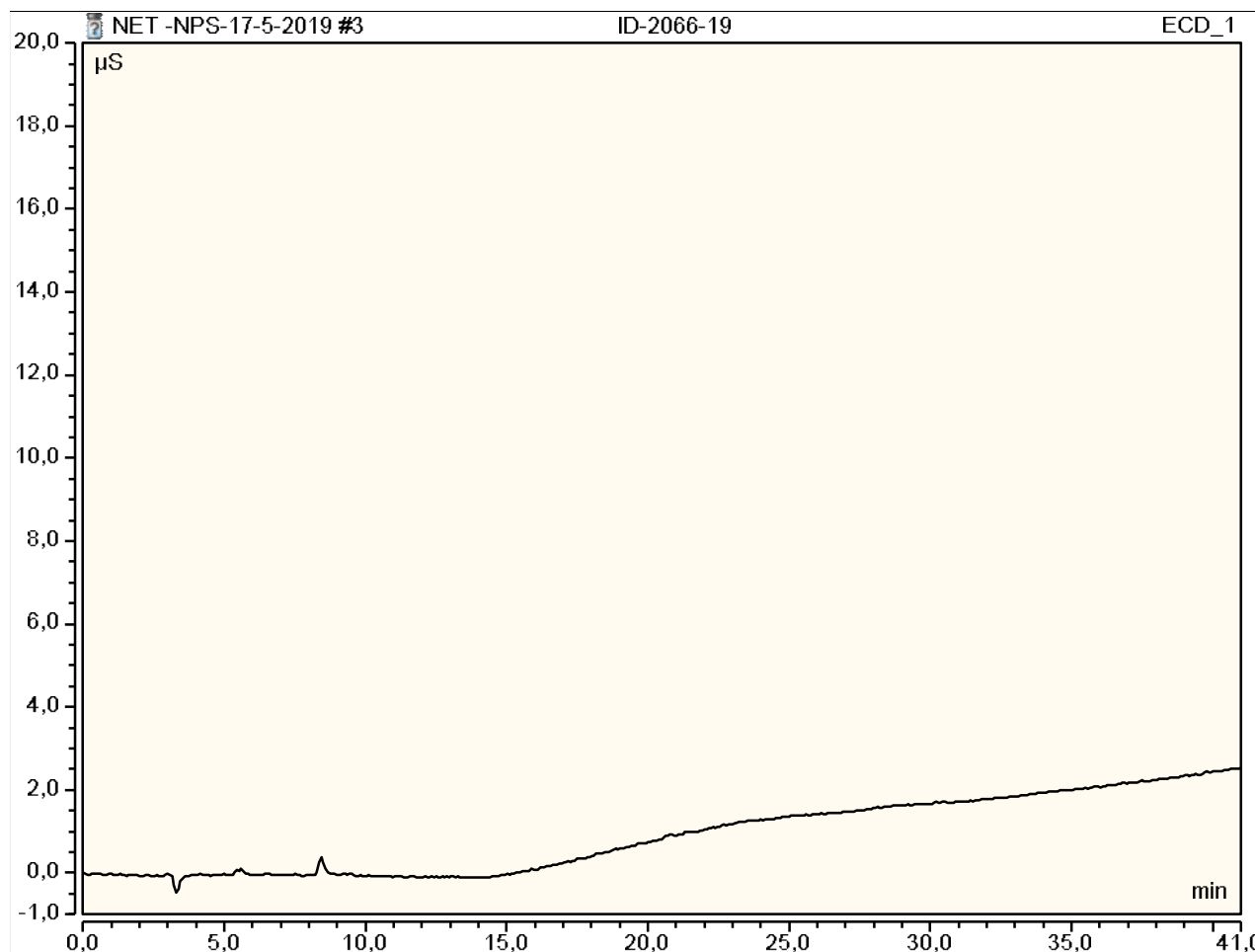
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
408.1476	1	15569336	C23 H22 Cl N3 O2	(M+H)+
409.1508	1	4071168.39	C23 H22 Cl N3 O2	(M+H)+
410.1456	1	5344855.87	C23 H22 Cl N3 O2	(M+H)+
411.1486	1	1303544.09	C23 H22 Cl N3 O2	(M+H)+
412.1513	1	185225.74	C23 H22 Cl N3 O2	(M+H)+
430.1296	1	223818	C23 H22 Cl N3 O2	(M+Na)+
815.288	1	468341.56		(2M+H)+
816.2909	1	243408.25		(2M+H)+
817.2868	1	367804.5		(2M+H)+
818.2885	1	165413.15		(2M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	ID-2066-19	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	17-maj-2019 / 11:50	Run Time:	42,00

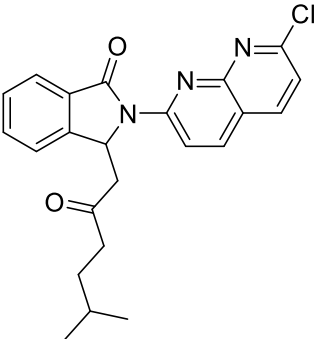
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height μS	Amount n.a.
		TOTAL:		0,00	0,00	0,00



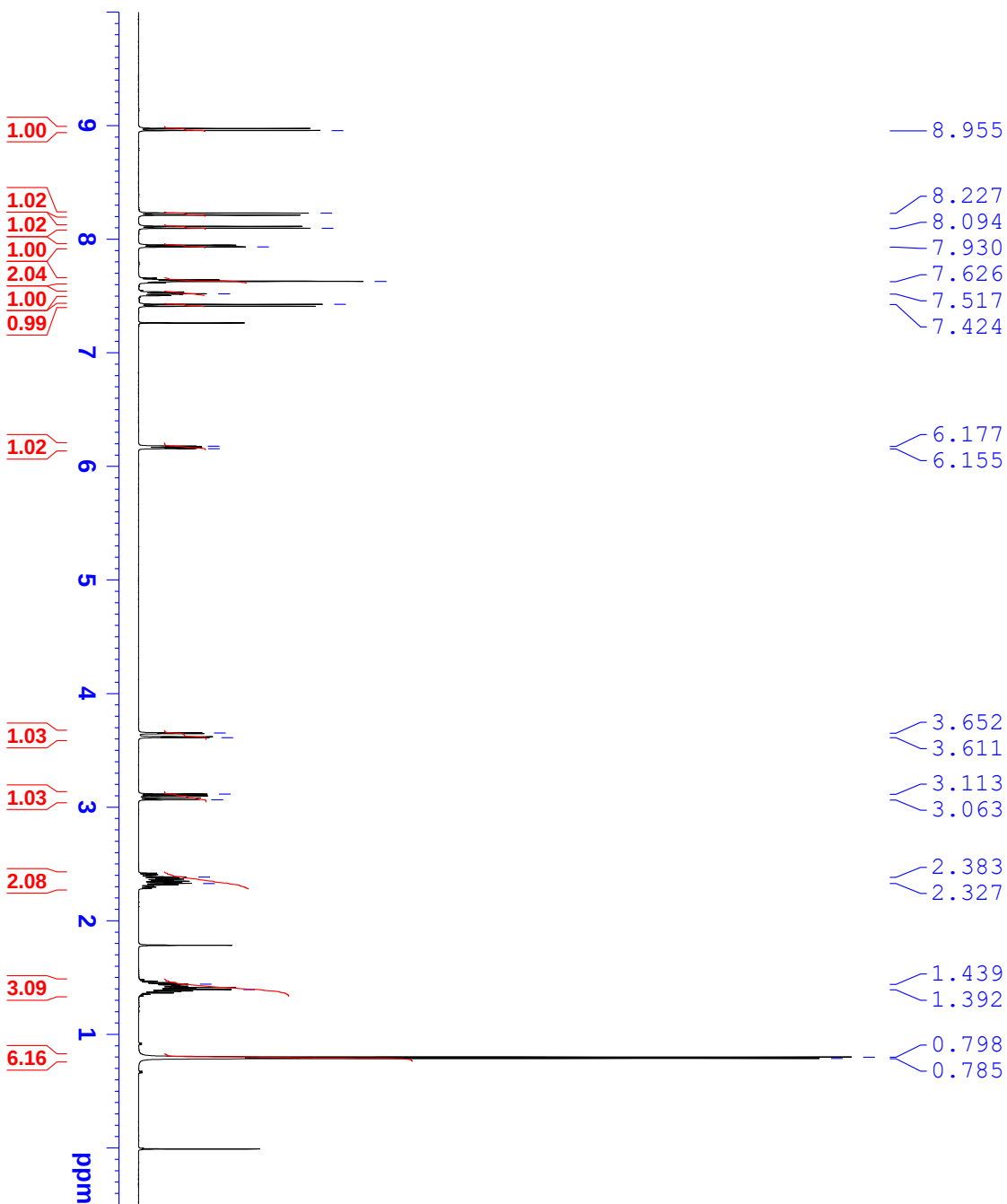
University
of Ljubljana
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2066-19
Received date:	May 20, 2019
Our notebook code:	NFL-2066-19
NMR sample preparation:	19.7 mg dissolved in 0.7 mL CDCl ₃
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 with formula, exact mass, molecular weight:	 Chemical Formula: C₂₃H₂₂ClN₃O₂ Exact Mass: 407,1401 Molecular Weight: 407,8980
Chemical name:	2-(7-chloro-1,8-naphthyridin-2-yl)-3-(5-methyl-2-oxohexyl)isoindolin-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >98% purity of a sample based on ¹H NMR spectrum.
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra, ¹ H and ¹³ C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	May 28, 2019

NFL-2066-19
¹H



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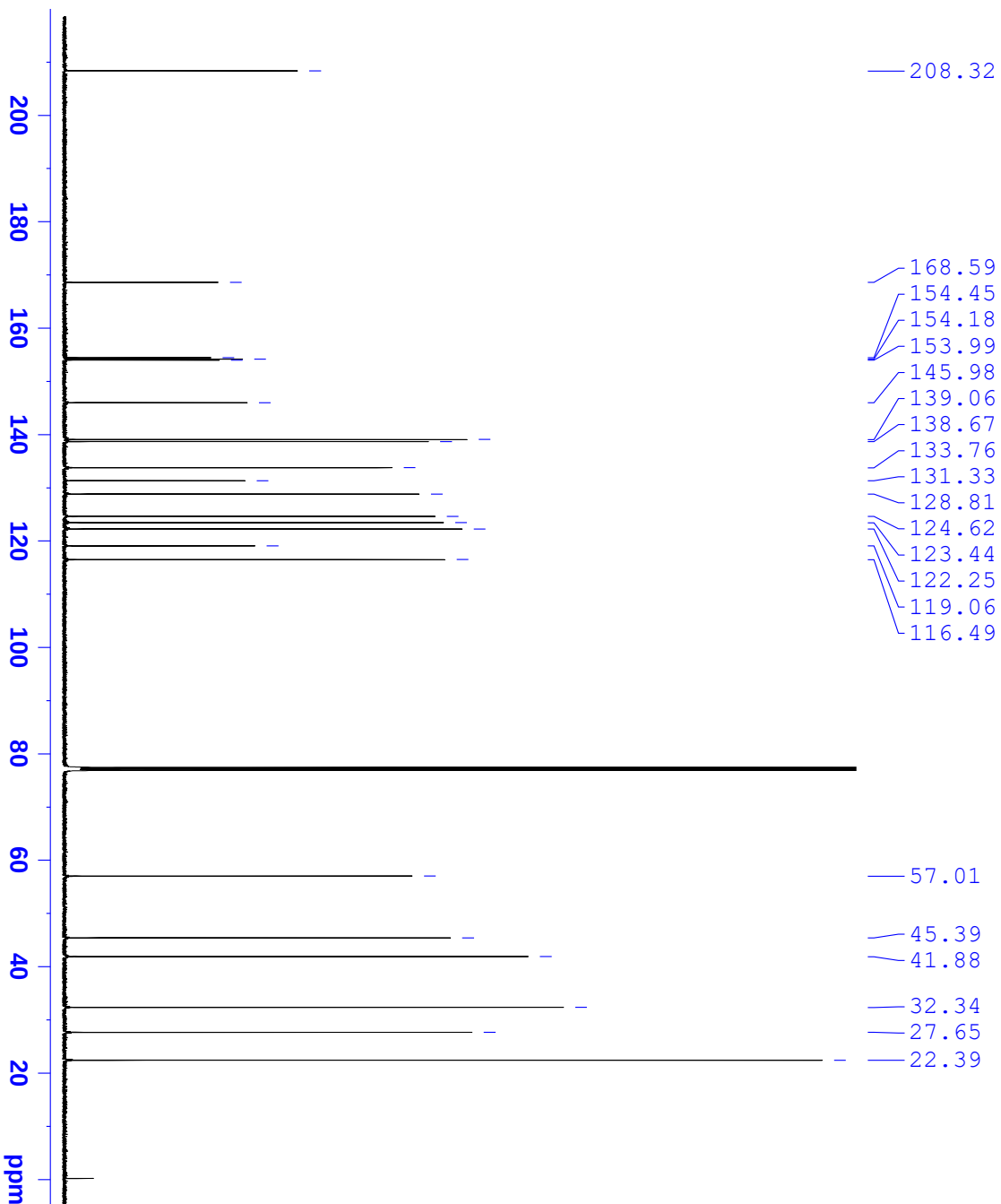
Current Data Parameters
NAME      NFL-2066-19
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20190524
Time      18.33
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         32
DS         2
SWH        10000.000 Hz
FIDRES     0.152588 Hz
AQ         3.2767999 sec
RG         90.5
DW         50.000 usec
DE         6.50 usec
TE         296.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
SFO1      500.1330885 MHz
NUC1      1H
P1        8.70 usec
PLW1      26.00000000 W

F2 - Processing parameters
SI        65536
SF        500.1300126 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

NFL-2066-19
13C



Current Data Parameters
NAME NFL-2066-19
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190524
Time 21.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 1.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 125.7703637 MHz
NUC1 13C
P1 8.70 usec
PLW1 122.0000000 W

===== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.30046001 W
PLW13 0.15113001 W

F2 - Processing parameters
SI 32768
SF 125.757751 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40