



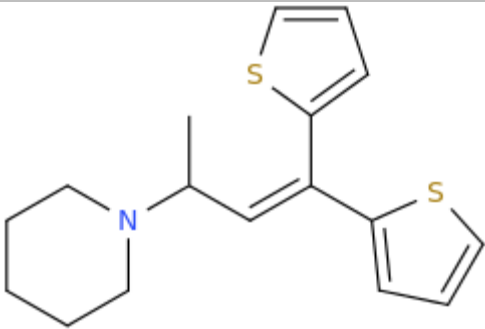
ANALYTICAL REPORT

Piperidylthiambutene (C₁₇H₂₁NS₂)

1-[4,4-bis(thiophen-2-yl)but-3-en-2-yl]piperidine

Remark – other NPS detected: **none**

Sample ID:	2034-18
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of sample receipt (DD/MM/YYYY):	21/11/2018
Date of entry (DD/MM/YYYY) into NFL database:	11/01/2019
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	1-[4,4-bis(thiophen-2-yl)but-3-en-2-yl]piperidine
Other names	1-(3,3-Di-2-thienyl-1-methylallyl)piperidine; 3-Piperidino-1,1-di(2-thienyl)but-1-ene; 1-[4,4-Di(2-thienyl)-3-buten-2-yl]piperidine; Piperidine, 1-(1-methyl-3,3-di-2-thienyl-2-propen-1-yl)-; Piperidinohton
Formula (per base form)	C ₁₇ H ₂₁ NS ₂
M _w (g/mol)	303.48
Salt form/anions detected	HCl
StdInChIKey (per base form)	FQRWJLVMJCKSME-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

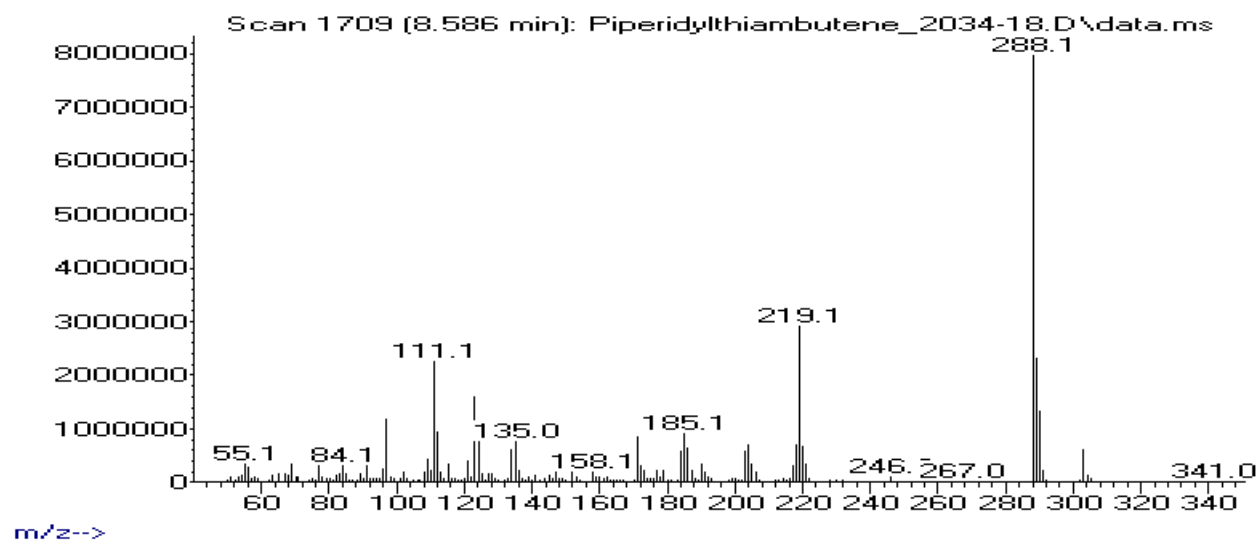
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	low (bad)

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8.59 BP(1): 288; BP(2): 219,BP(3) :289,
HPLC-TOF	+	Exact mass (theoretical): 303.1115; measured value Δppm:-1.28; formula:C17H21NS2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

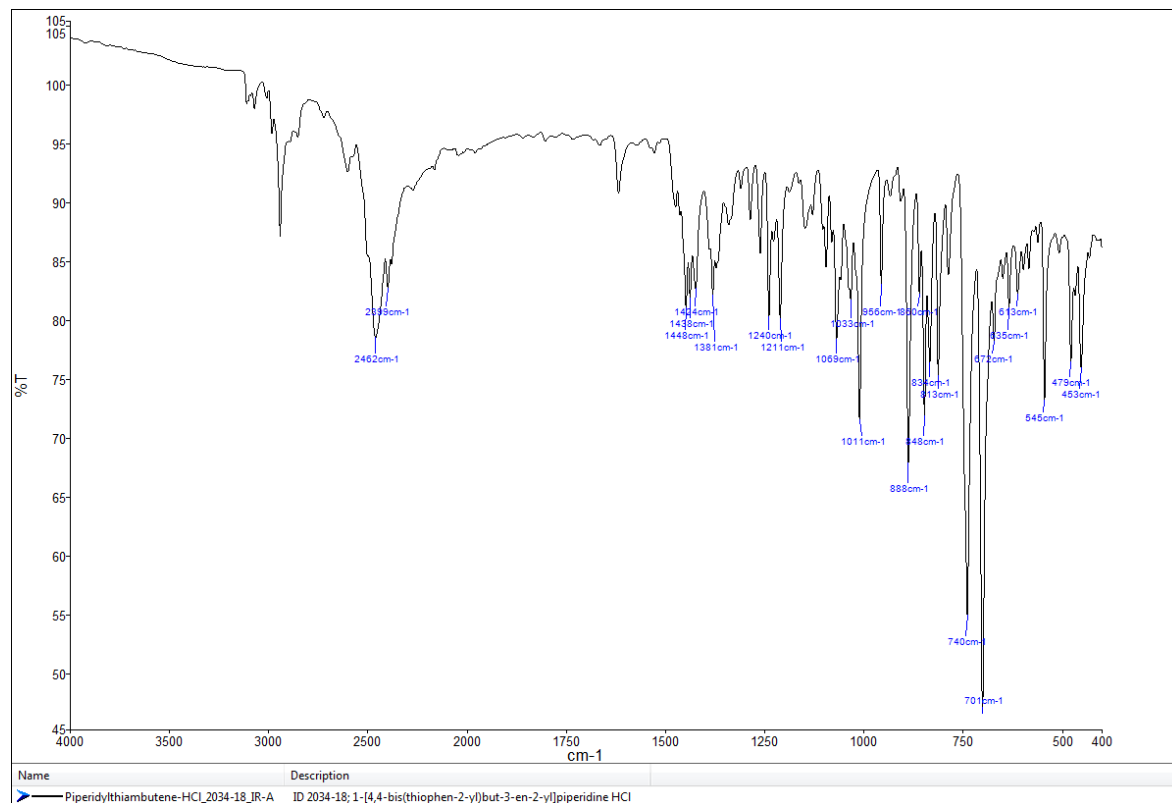
ANALYTICAL RESULTS

MS (EI)

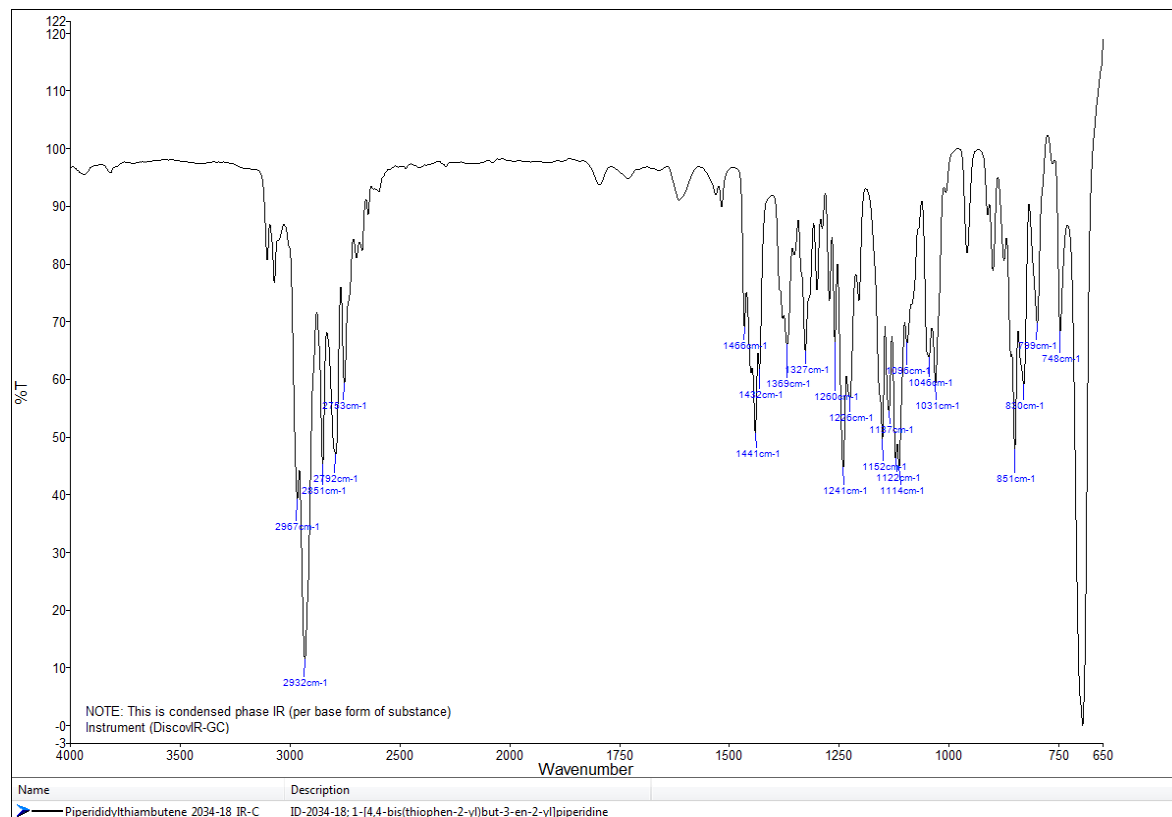
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

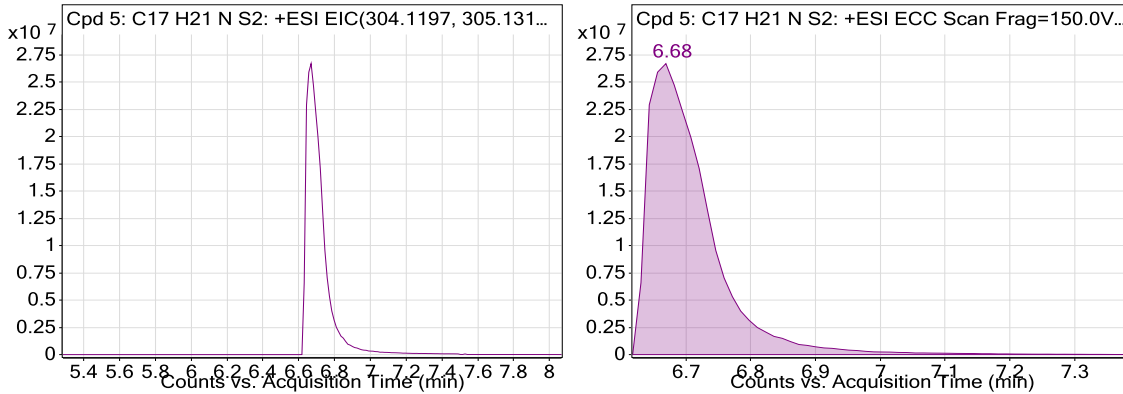
Data File	ID-2034_TOF.d	Sample Name	ID 2034-18
Sample Type	Sample	Position	P1-E5
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-19_10_2018-XDB-C18-ESI+.m	Acquired Time	11/23/2018 10:51:21 AM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

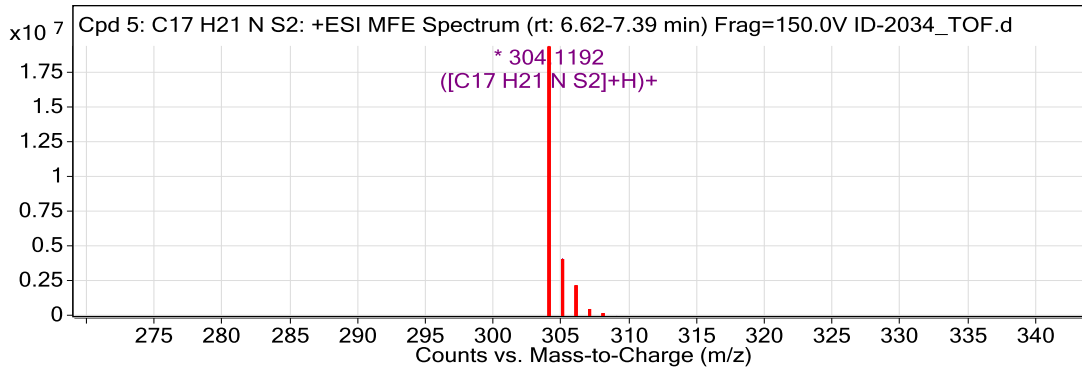
Label	MFG Formula	Obs. RT	Obs. Mass
Cpd 5: C17 H21 N S2	C17 H21 N S2	6.68	303.1119

Obs. m/z	Obs. RT	Obs. Mass
304.1192	6.68	303.1119

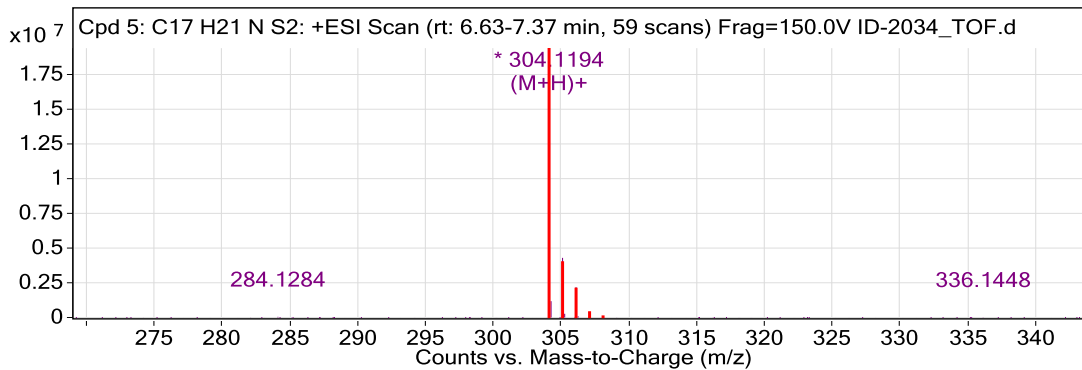
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
304.1192	1	19361586	C17 H21 N S2	(M+H)+
305.1221	1	4096712.53	C17 H21 N S2	(M+H)+
306.1172	1	1895997.83	C17 H21 N S2	(M+H)+

TOF REPORT

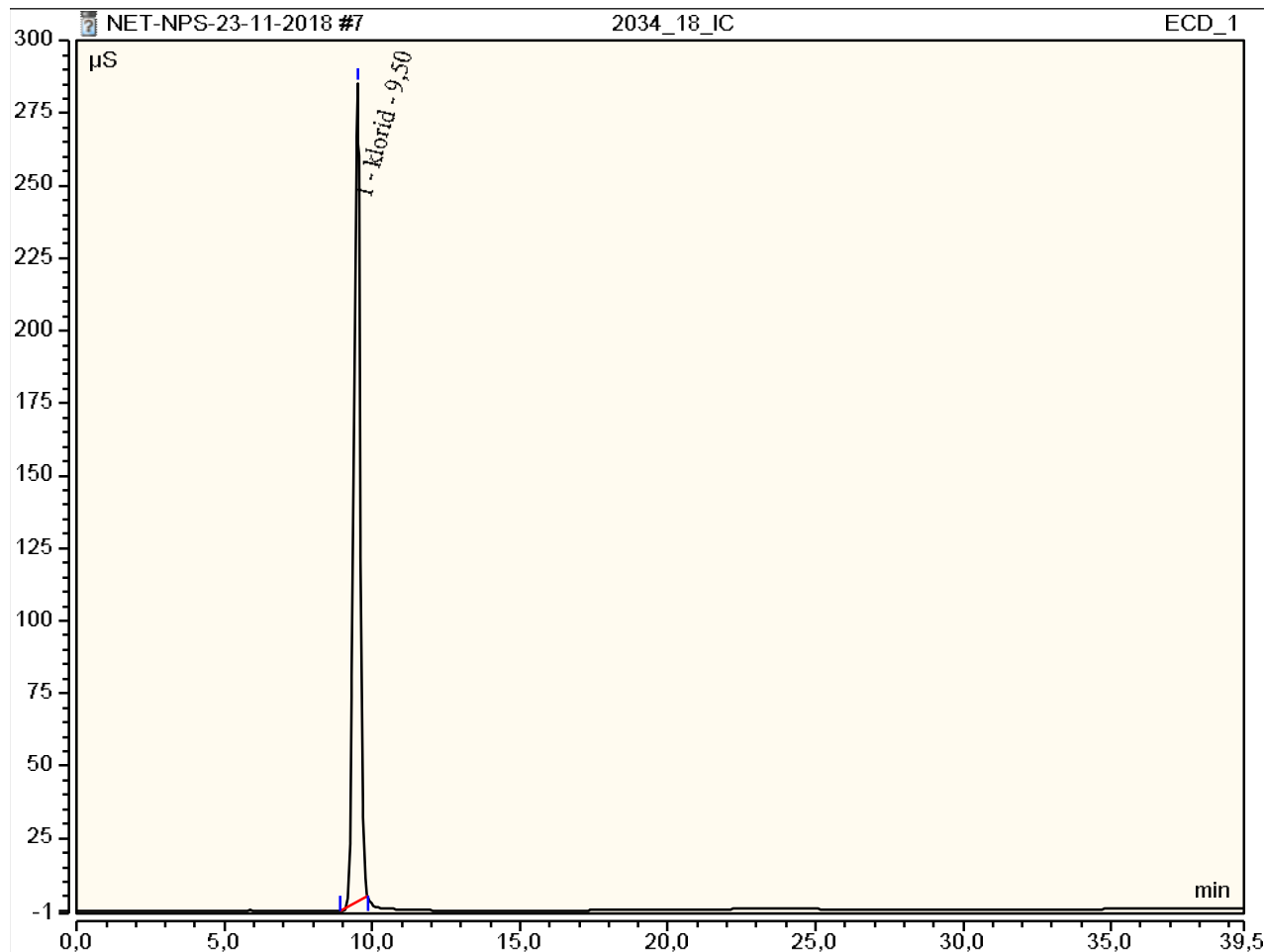
307.1193	1	314906.75	C17 H21 N S2	(M+H)+
308.1163	1	59442.11	C17 H21 N S2	(M+H)+
309.1168	1	7225.48	C17 H21 N S2	(M+H)+

--- End Of Report ---

Peak Integration Report

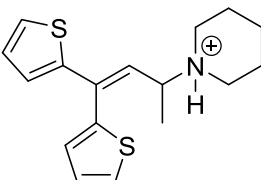
Sample Name:	2034_18_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	23-nov-2018 / 12:55	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	9,50	klorid	BMB	69,52	281,72	n.a.
		TOTAL:		69,52	281,72	0,00

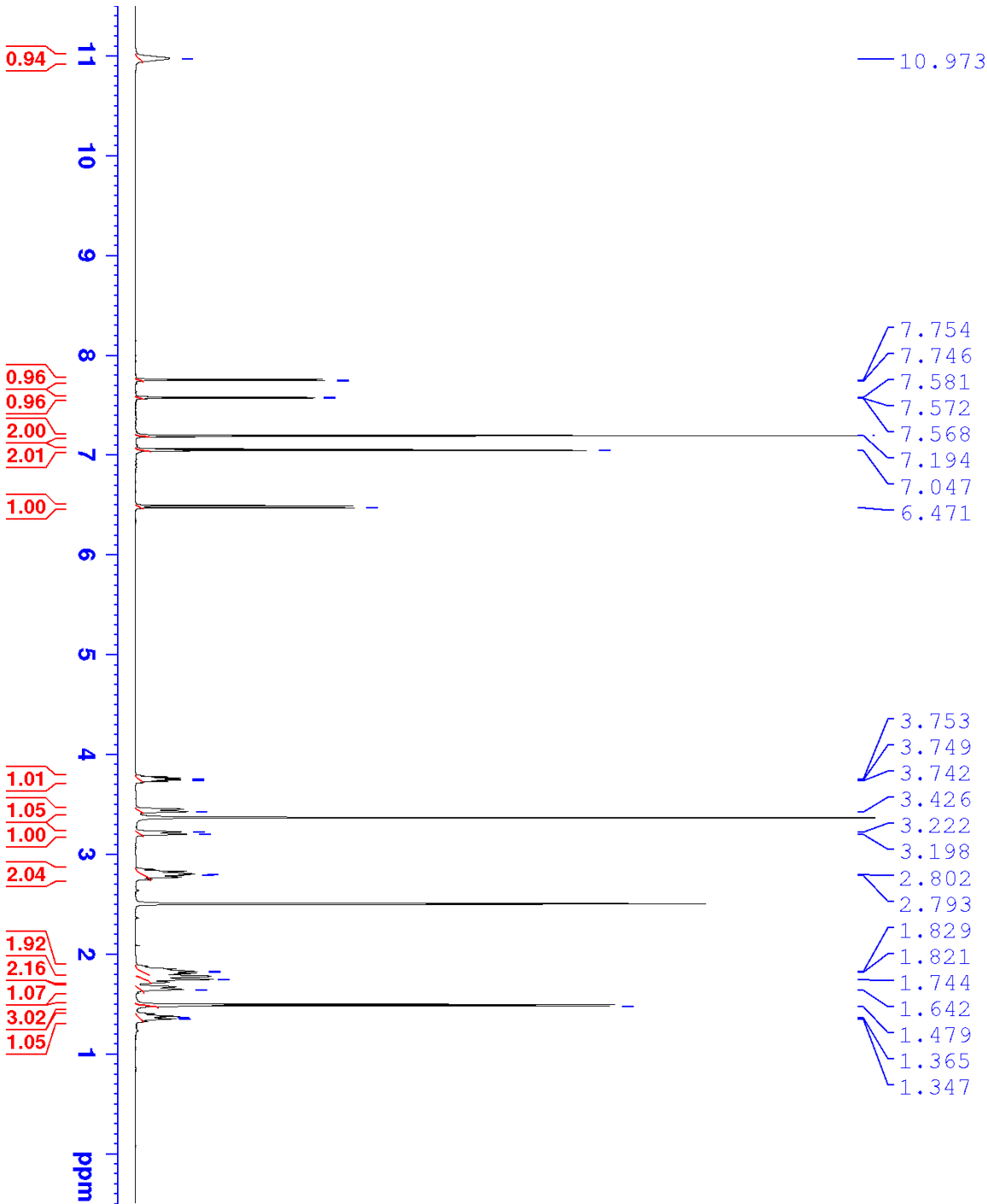




R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2034-18
Received date:	January 9, 2019
Our notebook code:	NFL-2034-18
NMR sample preparation:	19.5 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC,
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $\text{C}_{17}\text{H}_{22}\text{NS}_2^+$ Exact Mass: 304,1188 Molecular Weight: 304,4895
Chemical name:	<i>N</i> -protonated 1-(4,4-di(thiophen-2-yl)but-3-en-2-yl)piperidine
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 ^1H and ^{13}C NMR spectra, ^1H and ^{13}C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	January 17, 2019

NFL-2034-18
1H



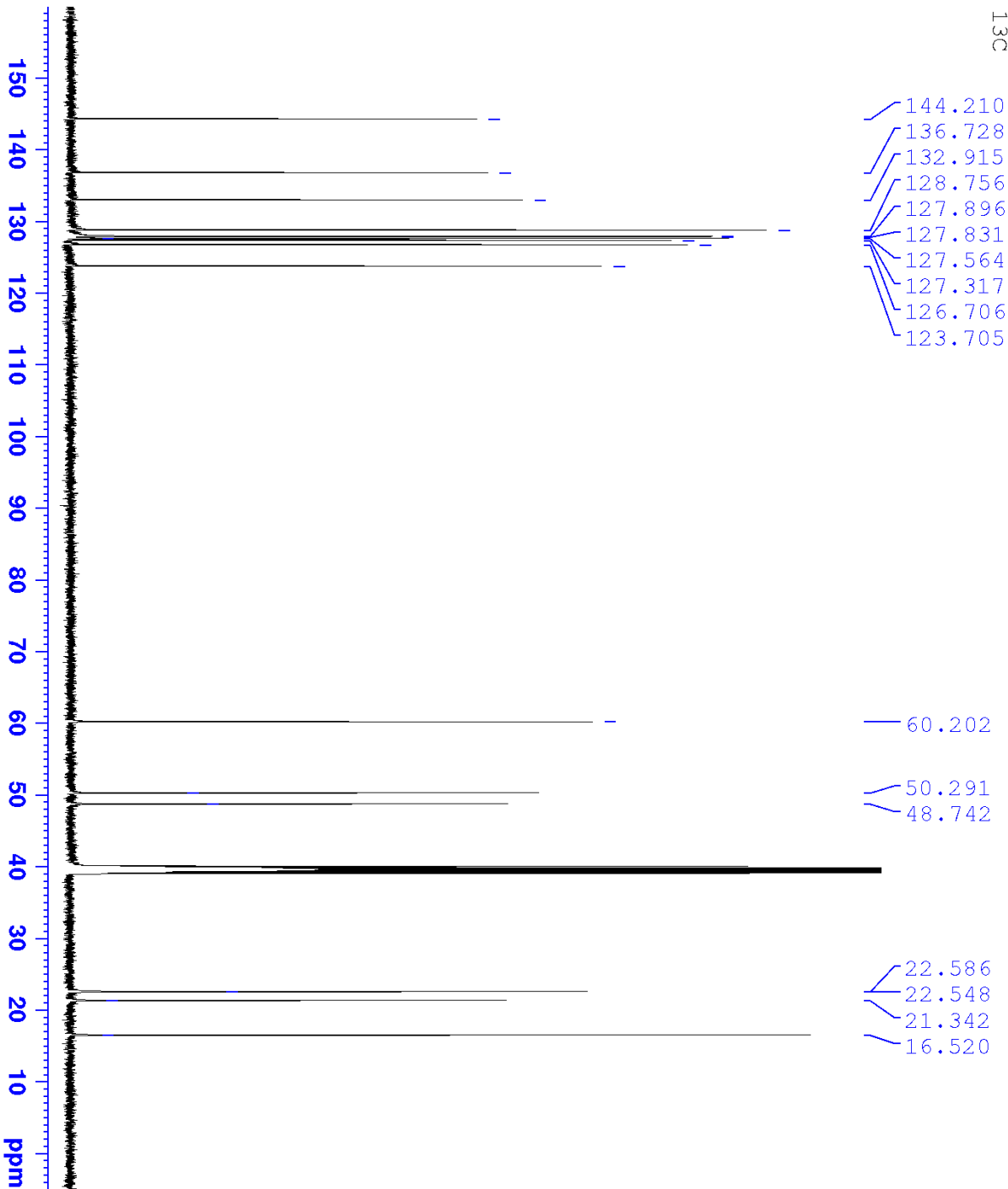
Current Data Parameters
NAME NFL-2034-18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20181221
Time 18.37
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 3.2767999 sec
RG 71.8
DE 50.000 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 8.70 usec
PLM1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-2034-18
13C



Current Data Parameters
NAME NFL-2034-18
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20181221
Time 19.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3072
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 8.70 usec
PLW1 122.00000000 W

CHANNEL f2
SFO2 500.1320005 MHz
NUC2 1H
CEPRG12 waltz16
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.30046001 W
PLW13 0.15113001 W

F2 - Processing parameters
SI 32768
SF 125.7578471 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40