



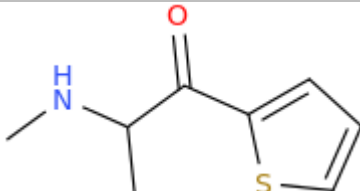
ANALYTICAL REPORT

bk-MPA (C₈H₁₁NOS)

2-(methylamino)-1-(thiophen-2-yl)propan-1-one

Remark – other NPS detected: **none**

Sample ID:	1945-18
Sample description:	powder
Sample type:	seized /KR
Date of sample receipt (DD/MM/YYYY):	28/02/2018
Date of entry (DD/MM/YYYY) into NFL database:	17/05/2018
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-(methylamino)-1-(thiophen-2-yl)propan-1-one
Other names	MTP; thiothinone
Formula (per base form)	C ₈ H ₁₁ NOS
M _w (g/mol)	169,24
Salt form/anions detected	HCl
StdInChIKey (per base form)	DOZQPYDMJMLVKX-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	pure by GC-MS, HPLC-TOF

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 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	soluble

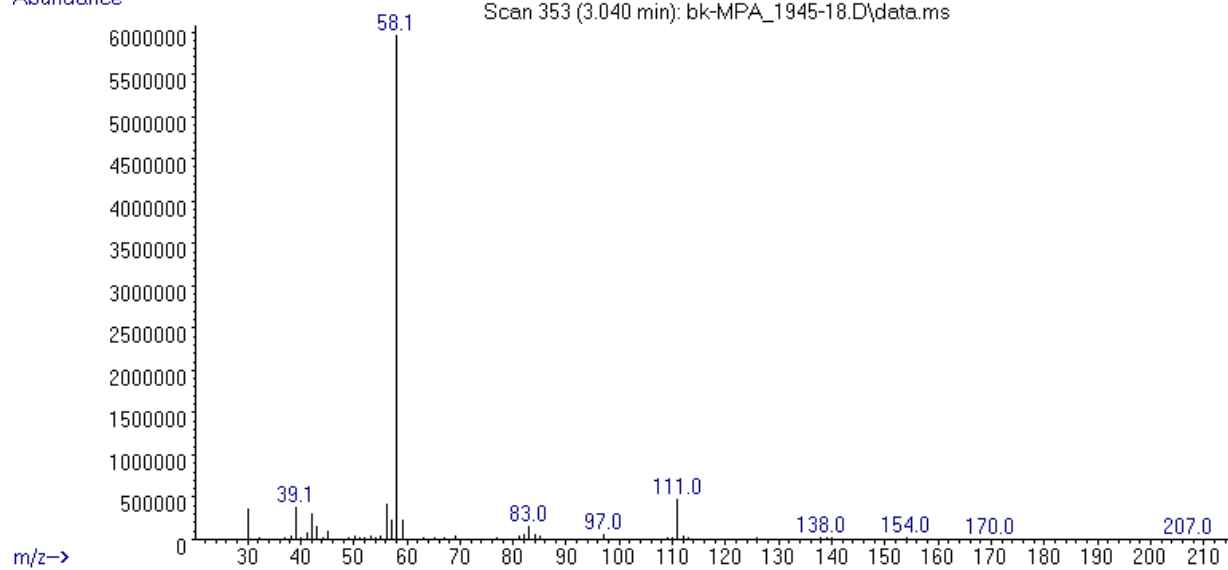
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 3,04 BP(1): 58; BP(2): 111,BP(3) :56,
HPLC-TOF	+	Exact mass (theoretical): 169,0561; measured value Δppm:-0,32; formula:C ₈ H ₁₁ NOS
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

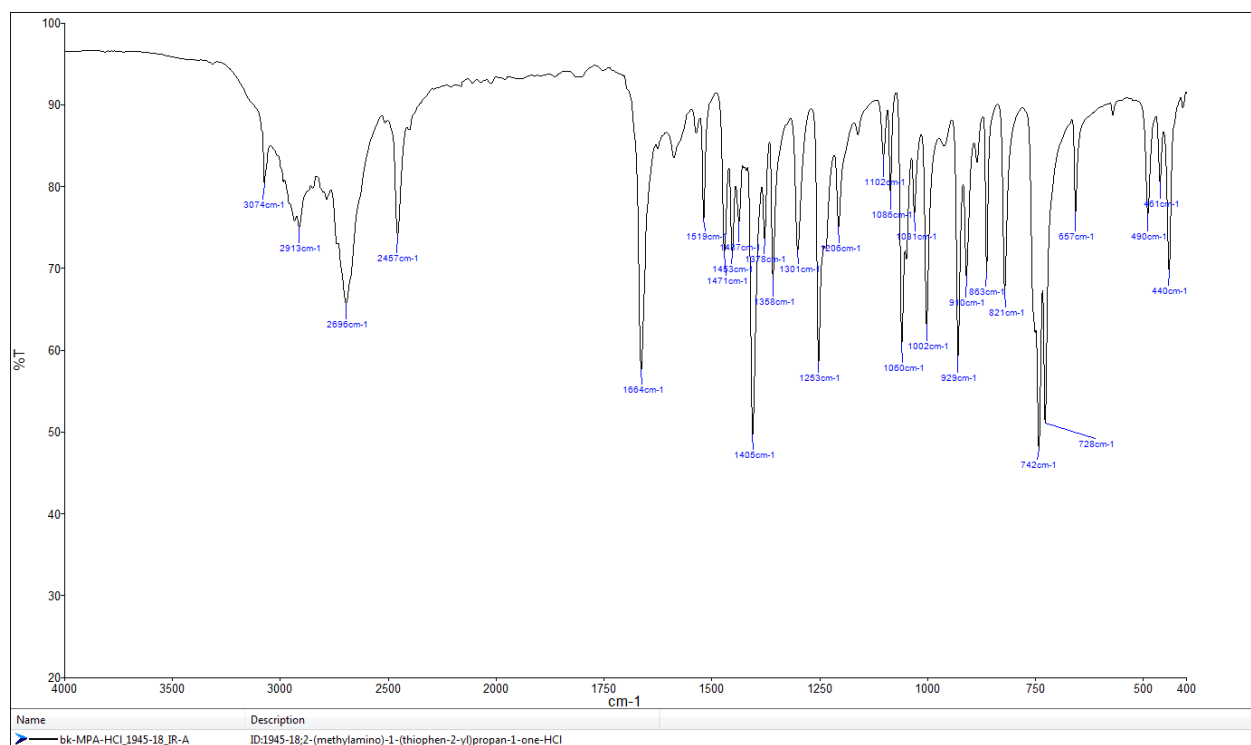
MS (EI)

Abundance

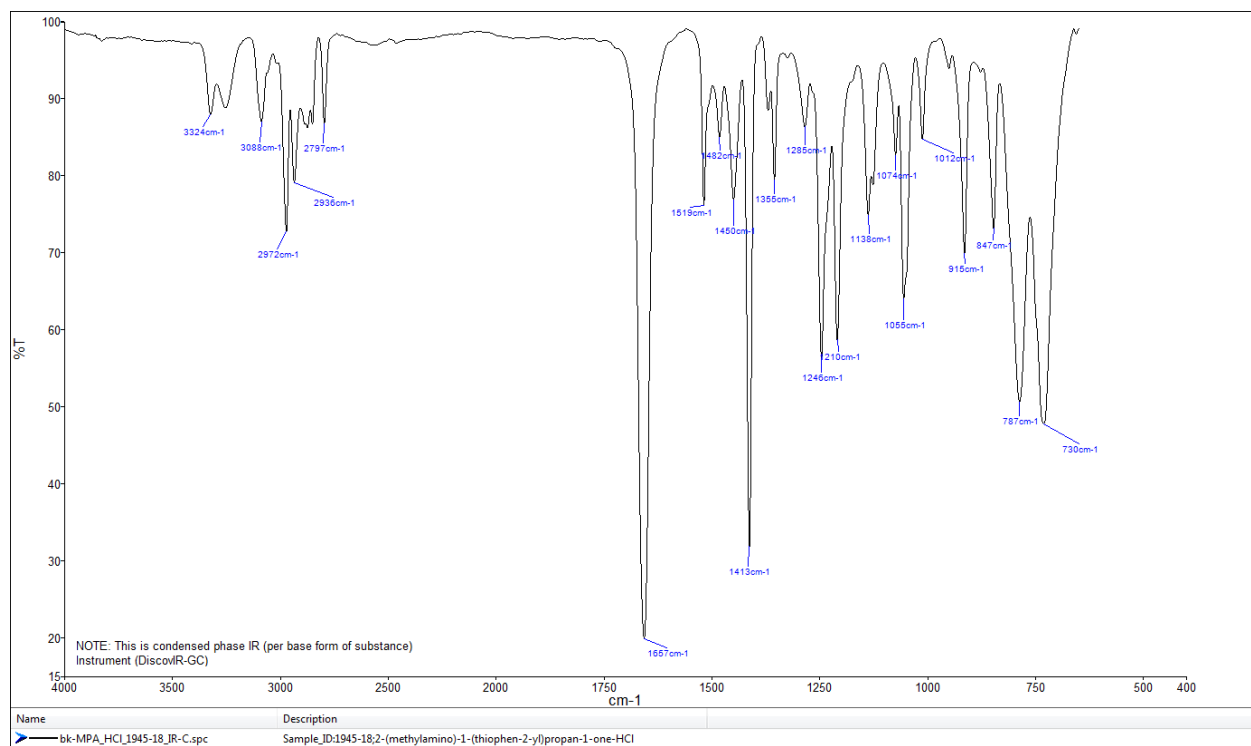
Scan 353 (3.040 min): bk-MPA_1945-18.D\data.ms



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

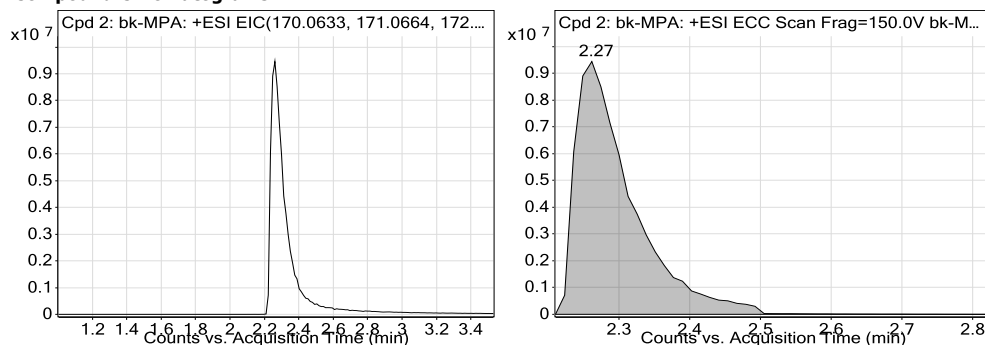
Data File	bk-MPA_1945-18.d	Sample Name	vzorec 12
Sample Type	Sample	Position	P1-A3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-04_12_2017-XDB-C18-ESI+.m	Acquired Time	4/23/2018 2:57:44 PM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

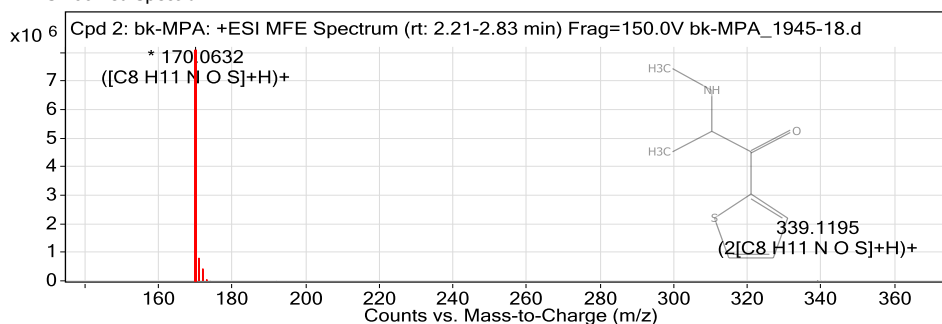
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: bk-MPA	bk-MPA	C8 H11 N O S	2.27	169.0562

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
bk-MPA	170.0632	2.27	169.0562	2.27	C8 H11 N O S	169.0561	-0.32

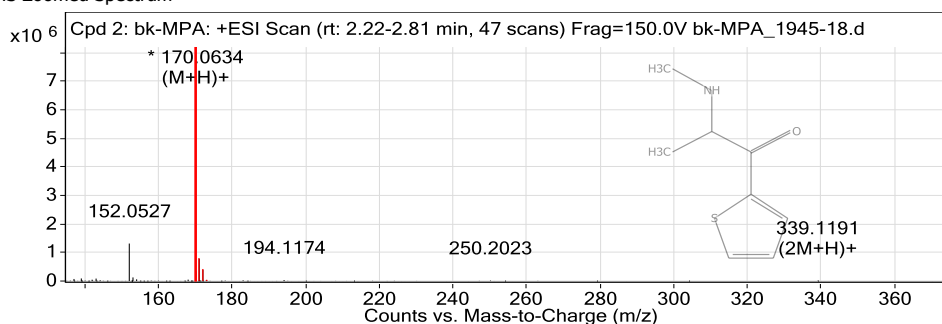
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

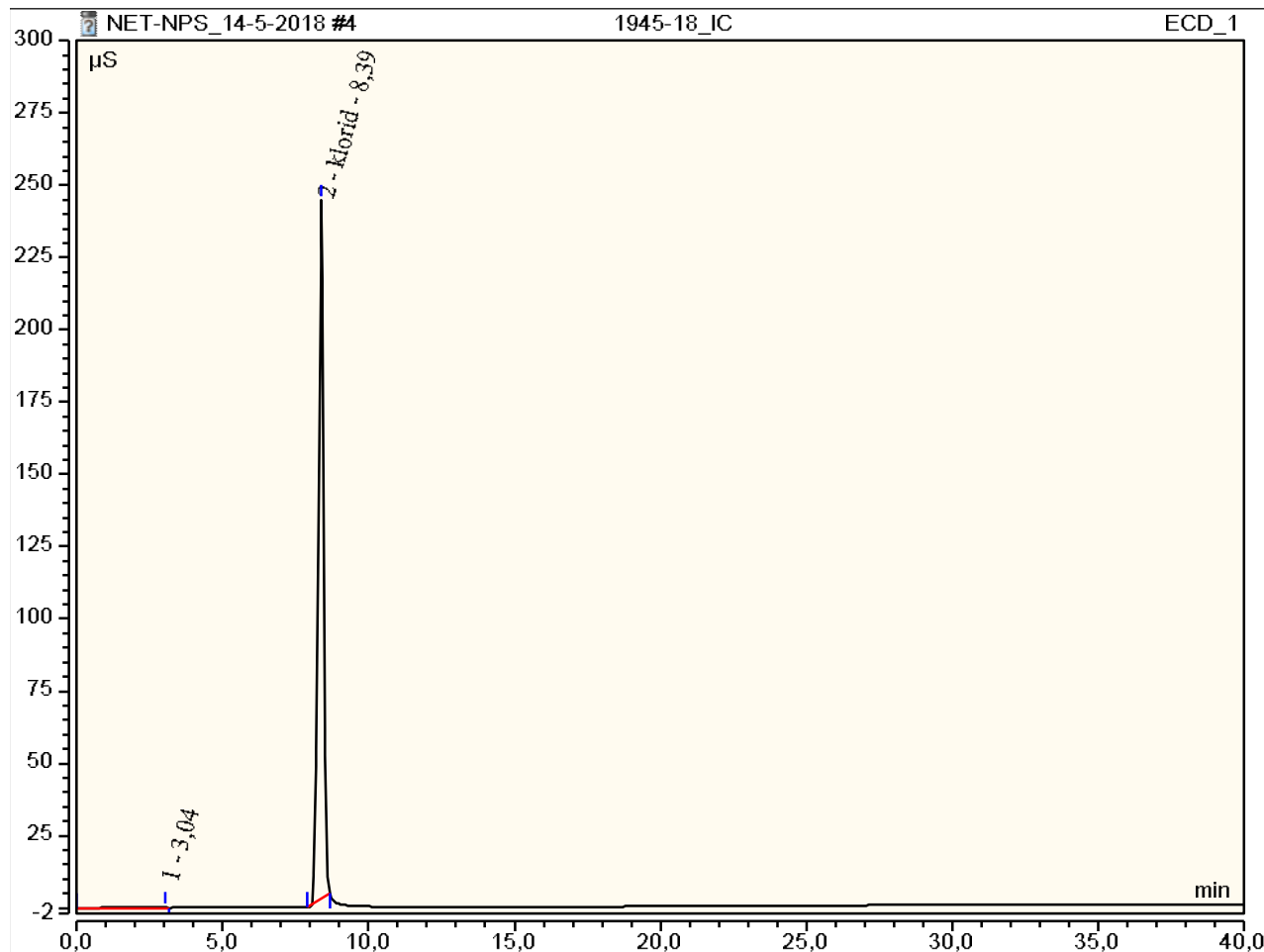
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
170.0632	1	8203604.5	C8 H11 N O S	(M+H)+
171.067	1	729454.53	C8 H11 N O S	(M+H)+
172.061	1	367574.84	C8 H11 N O S	(M+H)+
173.0633	1	36949.79	C8 H11 N O S	(M+H)+
174.0627	1	3399	C8 H11 N O S	(M+H)+
192.0457	1	1651.32	C8 H11 N O S	(M+Na)+
339.1195	1	14183.45	C8 H11 N O S	(2M+H)+
340.1226	1	2765.64	C8 H11 N O S	(2M+H)+
341.118	1	1663.43	C8 H11 N O S	(2M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	1945-18_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	14-maj-2018 / 13:40	Run Time:	42,00

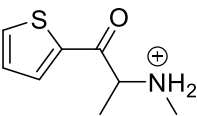
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
2,00	8,39	klorid	BMB	52,33	241,77	n.a.
		TOTAL:		52,33	241,77	0,00



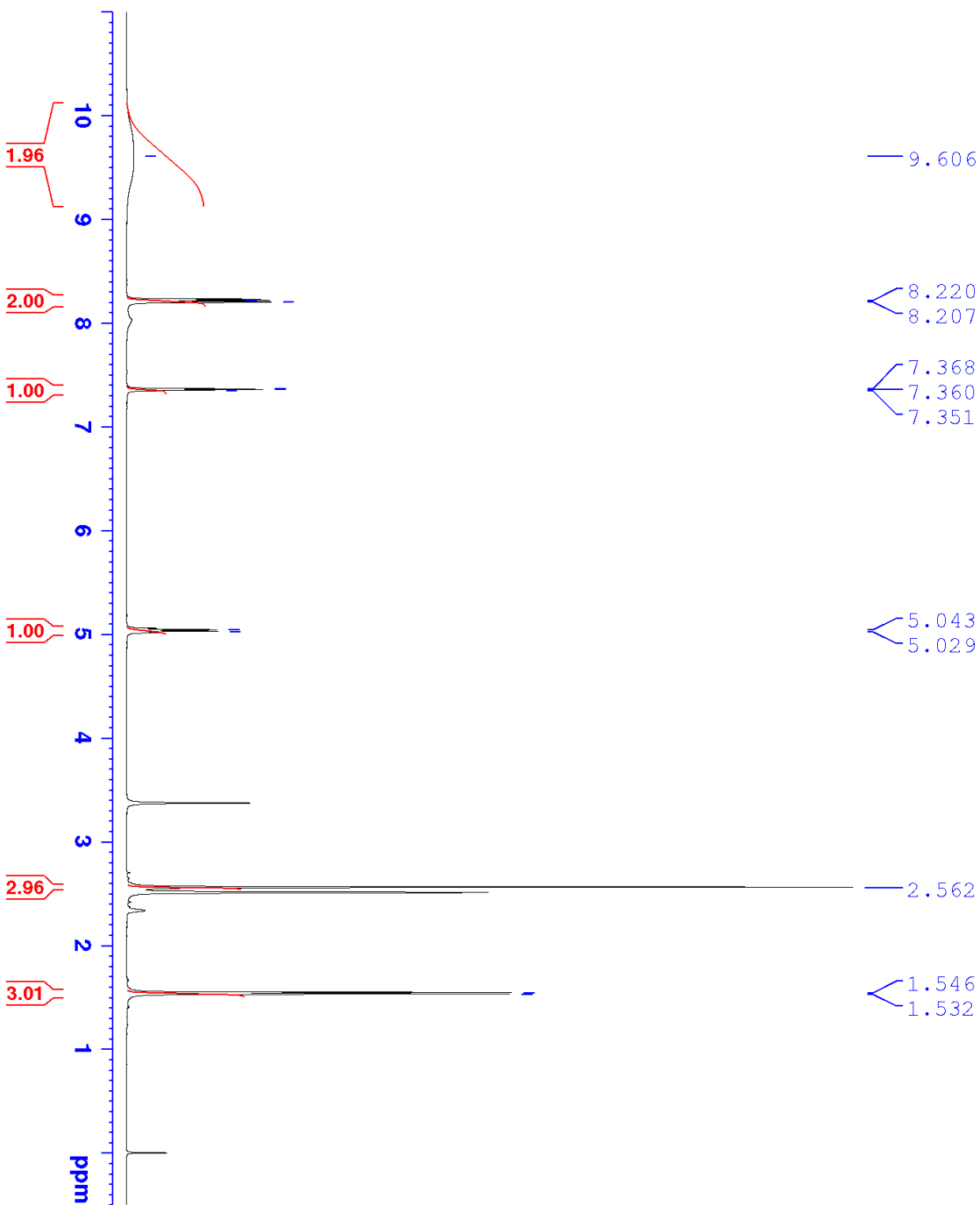
University
of Ljubljana
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	1945-18
Received date:	June 4, 2018
Our notebook code:	NFL-1945-18
NMR sample preparation:	15.3 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 with formula, exact mass, molecular weight:	 Chemical Formula: C₈H₁₂NOS⁺ Exact Mass: 170,0634 Molecular Weight: 170,2495
Chemical name:	<i>N</i> -protonated 2-(methilamino)-1-(thiophen-2-yl)propan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >95% purity of a sample based on ¹H NMR spectrum. - Spectroscopic data are in agreement with the literature report (Bouso, E. D.; Gardner, E. A.; O'Brien, J. E.; Talbot, B.; Kavanagh, P. V. Characterization of the pyrolysis products of methiopropamine. <i>Drug Test. Analysis</i> 2014, 6, 676–683).
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:	June 21, 2018

NFL-1945-18



```

Current Data Parameters
NAME      NFL-1945-18
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20180111
Time      22.21
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          3.276799 sec
RG          101
DW          50.000 usec
DE          6.50 usec
TE         296.0 K
D1          1.00000000 sec
TD0         1

===== CHANNEL f1 =====
SFO1      500.130885 MHz
NUC1      1H
P1         8.70 usec
PLM1      26.00000000 W

F2 - Processing parameters
SI         65536
SF         500.129992 MHz
WDW        0
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

NFL-1945-18

Current Data Parameters
NAME NFL-1945-18
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20180612
Time 1.27
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 296.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TDO 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
CPDPRG12 waltz16
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.30046001 W
PLW13 0.15113001 W

F2 - Processing parameters

SI 32768
SF 125.7578466 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

