



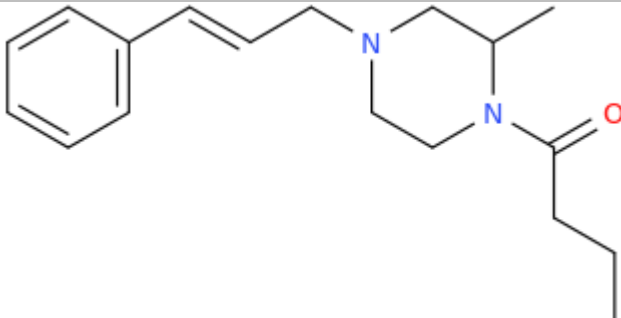
ANALYTICAL REPORT

2-methyl AP-237 (C₁₈H₂₆N₂O)

1-[2-methyl-4-(3-phenylprop-2-en-1-yl)piperazin-1-yl]butan-1-one

Remark – other NPS detected: **none**

Sample ID:	2053-19
Sample description:	powder
Sample type:	test purchase /ISF projekt (NFL-SI)
Date of sample receipt (DD/MM/YYYY):	21/02/2019
Date of entry (DD/MM/YYYY) into NFL database:	29/03/2019
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-[2-methyl-4-(3-phenylprop-2-en-1-yl)piperazin-1-yl]butan-1-one
Other names	1-[2-methyl-4-(3-phenyl-2-propen-1-yl)-1-piperazinyl]-1-butanone; 2-methyl AP-237; 2-methyl-AP-237
Formula (per base form)	C ₁₈ H ₂₆ N ₂ O
M _w (g/mol)	286,42
Salt form/anions detected	HCl
StdInChIKey (per base form)	CRSFXYZFNAFVFC-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>91 % 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

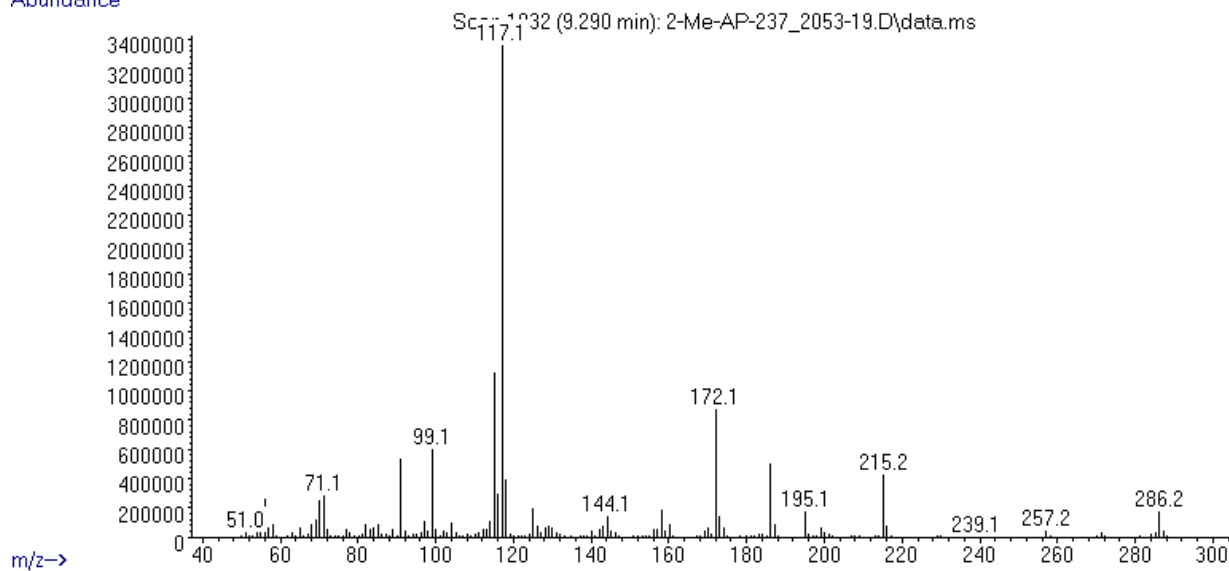
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 9,29 BP(1): 117; BP(2): 115,BP(3) :172,
HPLC-TOF	+	Exact mass (theoretical): 286,2045; measured value Δppm:-4,25; formula:C18H26N2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	-	spot test by AgNO ₃
NMR (in FKKT)	+	
validation		
other		

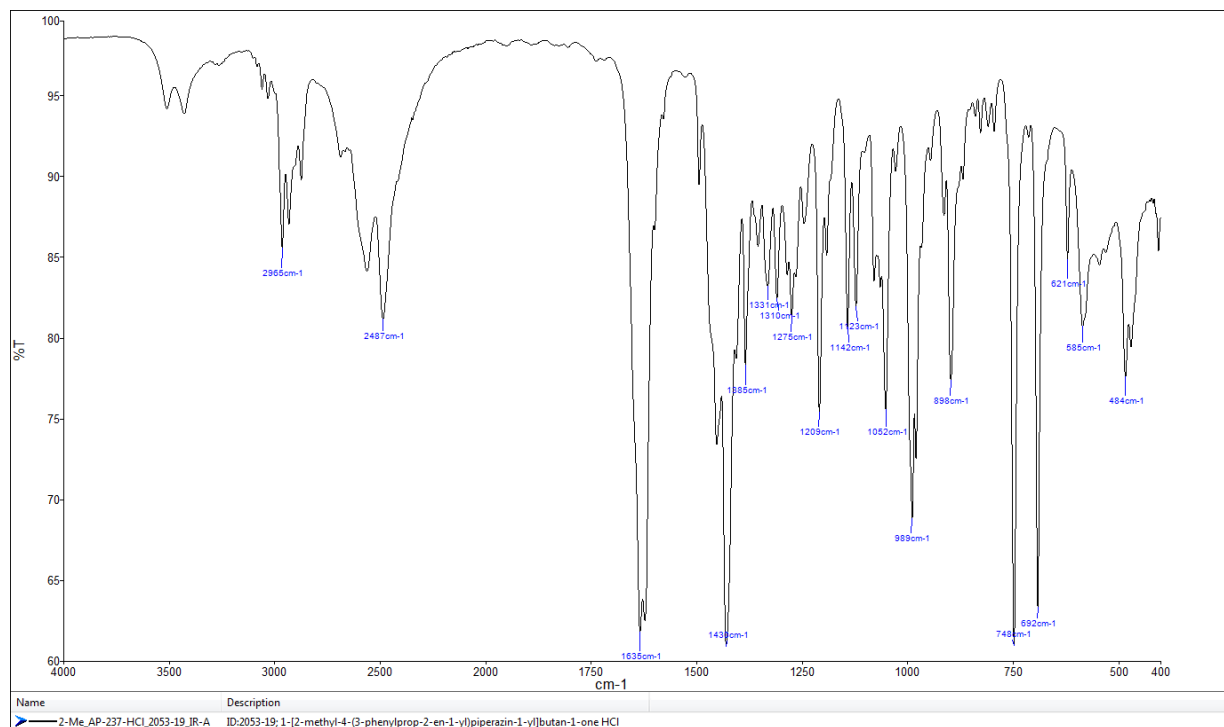
ANALYTICAL RESULTS

MS (EI)

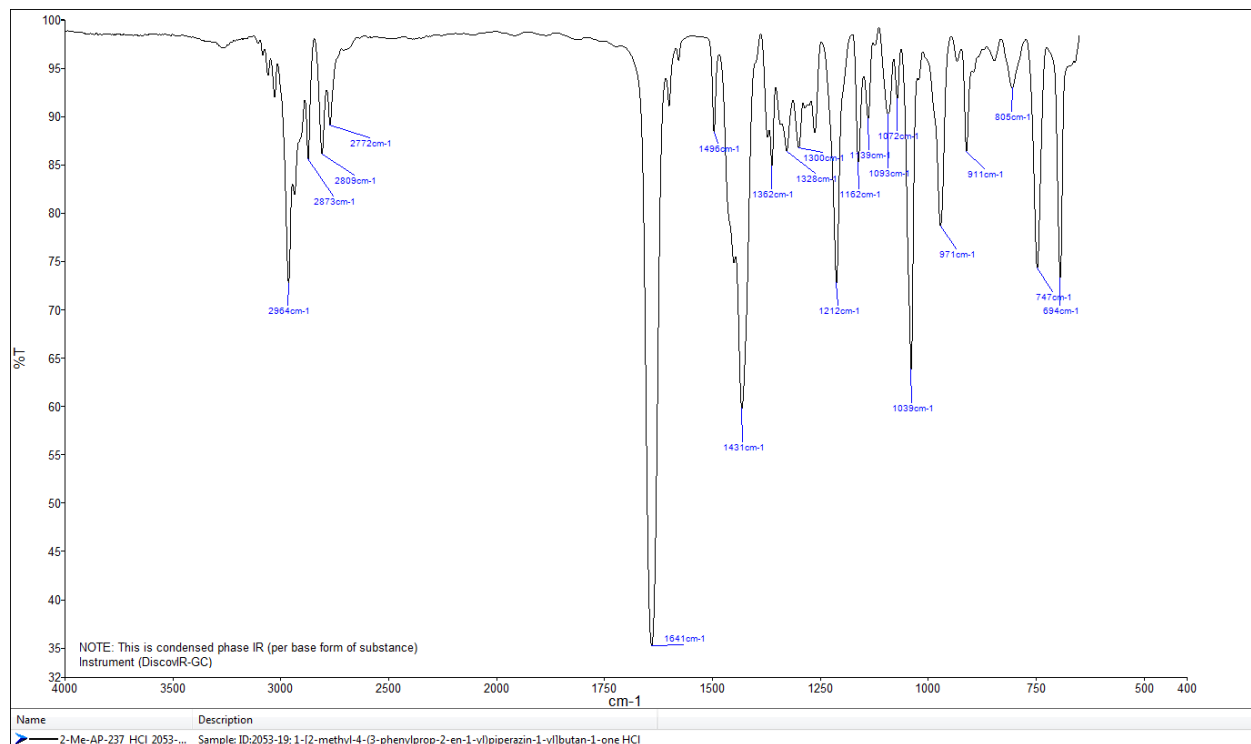
Abundance



FTIR-ATR - direct measurement (sample as received) IR (solid phase – after chromatographic separation)



IR (solid phase) – after chromatographic separation



TOF REPORT

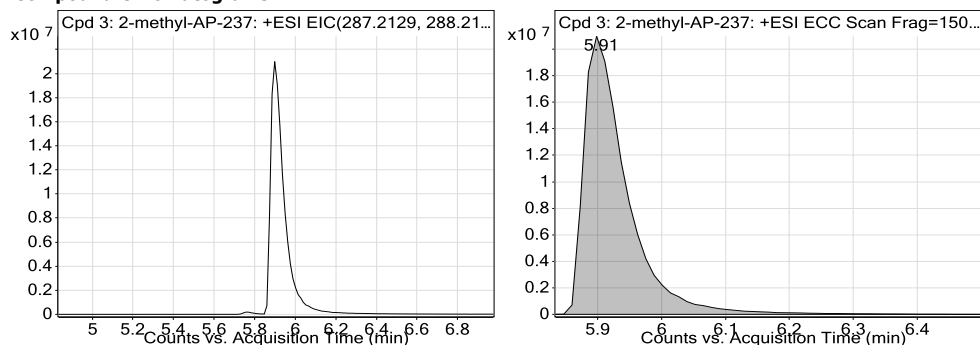
Data File	2-Methyl-AP-237_2053_19.d	Sample Name	ID_2053-19
Sample Type	Sample	Position	P1-F3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-8_1_2019-XDB-C18-ESI+.m	Acquired Time	2/21/2019 3:25:44 PM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

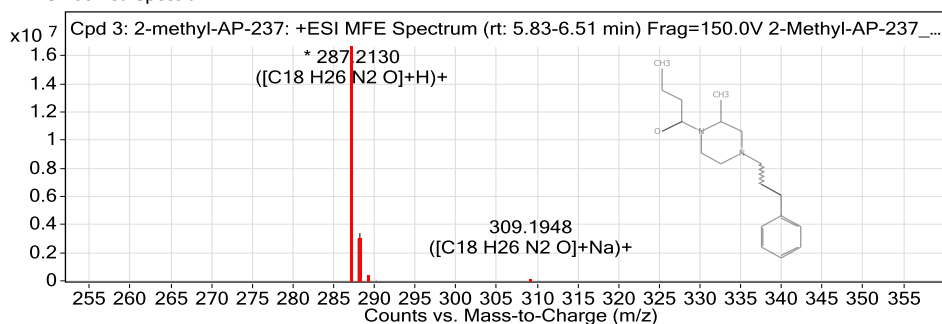
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 3: 2-methyl-AP-237	2-methyl-AP-237	C18 H26 N2 O	5.91	286.2057

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
2-methyl-AP-237	287.213	5.91	286.2057	5.9	C18 H26 N2 O	286.2045	-4.25

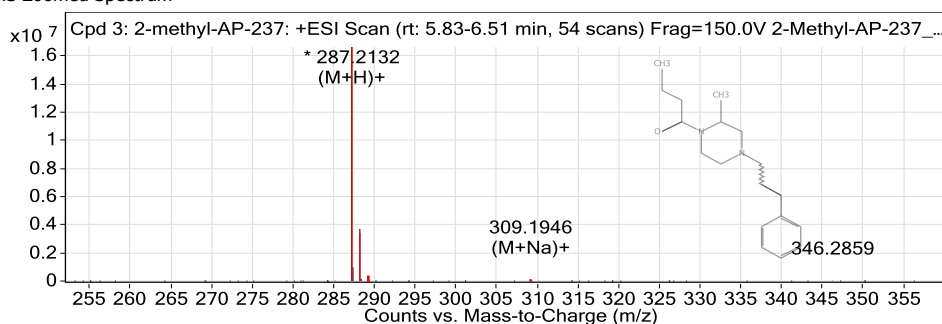
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
287.213	1	16624994	C18 H26 N2 O	(M+H)+
288.2164	1	3392389.45	C18 H26 N2 O	(M+H)+
289.2196	1	309555.48	C18 H26 N2 O	(M+H)+
290.2221	1	24610.56	C18 H26 N2 O	(M+H)+
291.2253	1	756.18	C18 H26 N2 O	(M+H)+
309.1948	1	78577.22	C18 H26 N2 O	(M+Na)+
310.1977	1	15987.4	C18 H26 N2 O	(M+Na)+
325.1686	1	7734.48	C18 H26 N2 O	(M+K)+
326.1722	1	1751.62	C18 H26 N2 O	(M+K)+

--- End Of Report ---

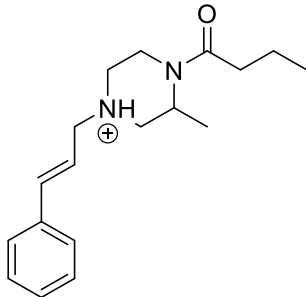
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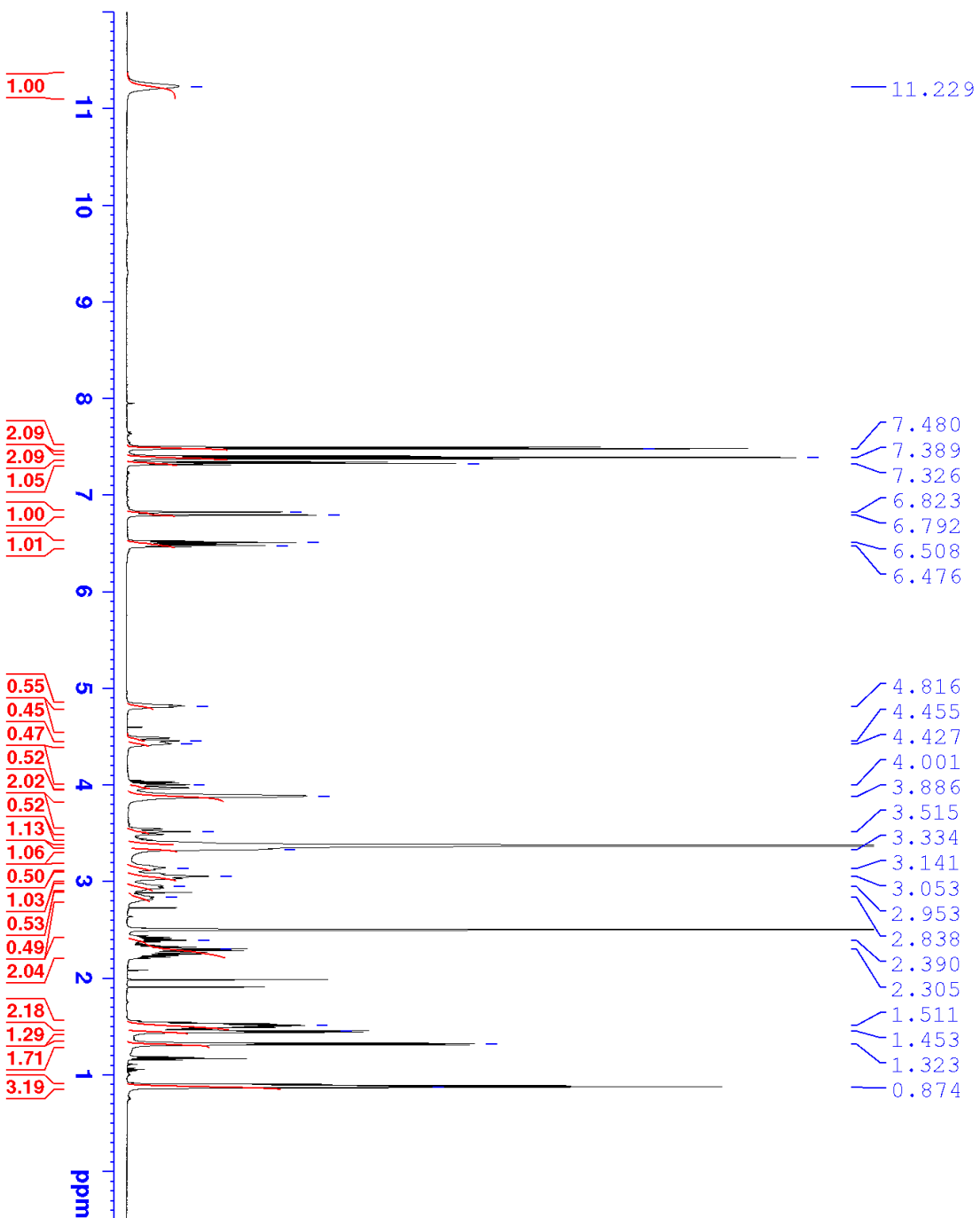
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2053-19
Received date:	February 28, 2019
Our notebook code:	NFL-2053-19
NMR sample preparation:	21.6 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: $\text{C}_{18}\text{H}_{27}\text{N}_2\text{O}^+$ Exact Mass: 287,2118 Molecular Weight: 287,4265
Chemical name:	<i>N</i> -protonated 1-(4-cinnamyl-2-methylpiperazin-1-yl)butan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - >91% 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:	March 19, 2019

NFL-2053-19
1H



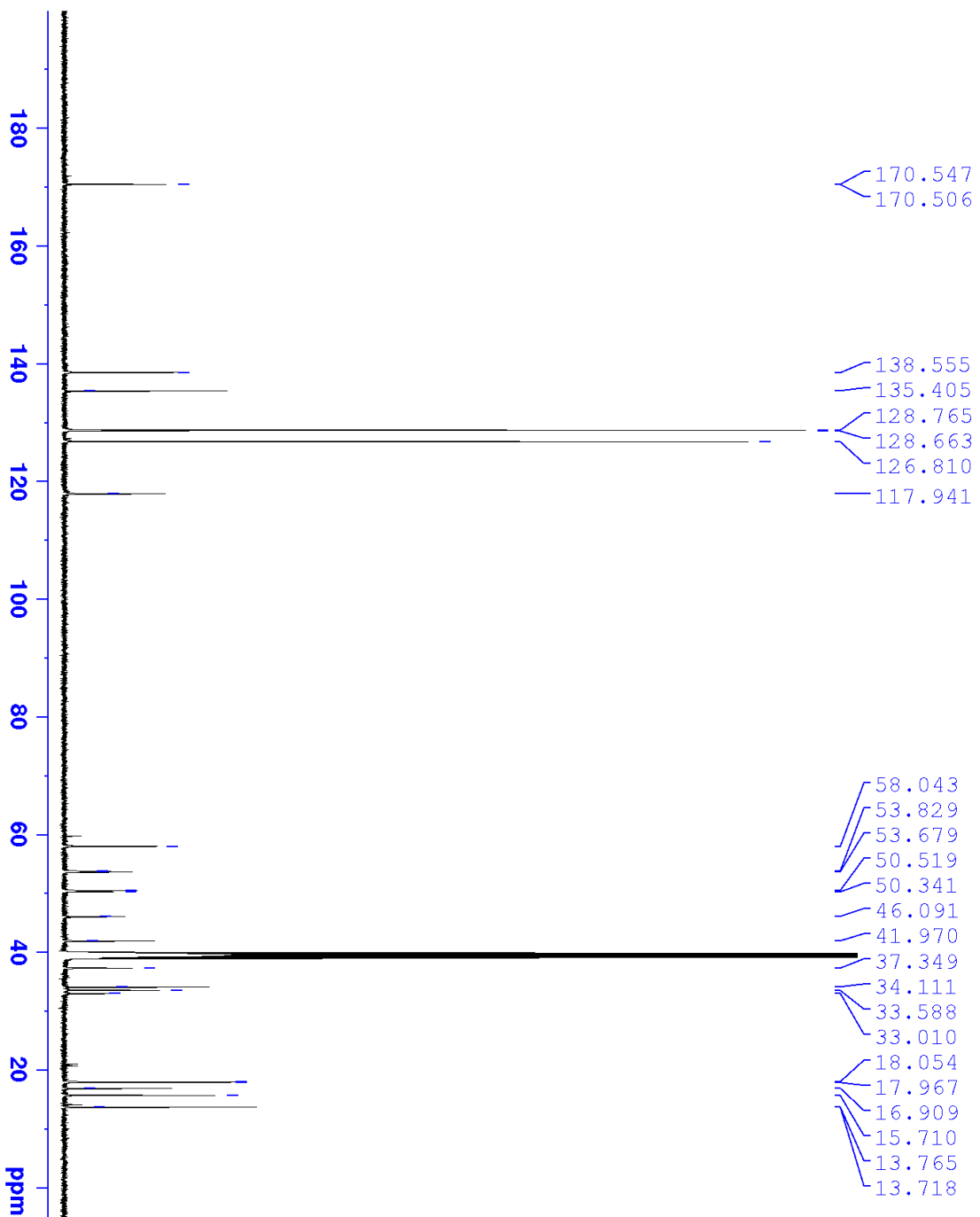
Current Data Parameters
NAME NFL-2053-19
EXPNO 111
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190306
Time 6.19
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 3.276799 sec
RG 37
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.130885 MHz
NUC1 1H
P1 8.70 usec
PL1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-2053-19
13C



Current Data Parameters
NAME NFL-2053-19
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190303
Time 18.16
INSTRUM spect
PROBHD 5 mm PABO 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 298.0 K
D1 2.0000000 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 26.0000000 W
PLW12 0.30046001 W
PLW13 0.15113001 W

F2 - Processing parameters
SI 32768
SF 125.7578474 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40