

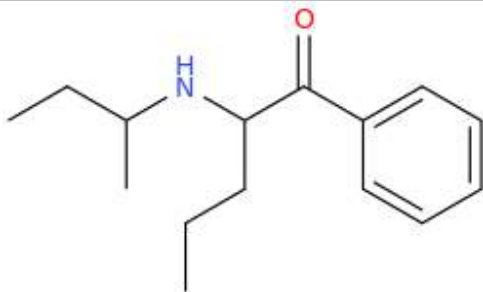
## ANALYTICAL REPORT

N-sec-butyl-pentedrone (C<sub>15</sub>H<sub>23</sub>NO)

## 2-[(butan-2-yl)amino]-1-phenylpentan-1-one

Remark – other NPS detected: none

Sample ID:	3255-23
Sample description:	crystalline
Sample type:	collected /NGO-collected
Date of entry (DD/MM/YYYY) into NFL database:	10/10/2023
Report updates (if any) will be published here:	<a href="http://www.policija.si/apps/nfl_response_web/seznam.php">http://www.policija.si/apps/nfl_response_web/seznam.php</a>

Substance identified - structure <sup>1</sup> (base form)	
Systematic name	2-[(butan-2-yl)amino]-1-phenylpentan-1-one
Other names	2-(sec-butylamino)-1-phenylpentan-1-one
Formula (per base form)	C <sub>15</sub> H <sub>23</sub> NO
M <sub>w</sub> (g/mol)	233,36
Salt form/anions detected	HCl
StdInChIKey (per base form)	WDOQHXCRCRFTHDF-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	sample is pure based on 1H NMR spectrum

<sup>1</sup> 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 µl 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 6.1 min, then heating at 50 °C/min up to 325 °C and finally 9.1 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 (N<sub>2</sub>) 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<sup>-1</sup>; resolution 4cm<sup>-1</sup>

**4. GC- (MS)-IR** solid phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 µl 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<sup>-1</sup>.

**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

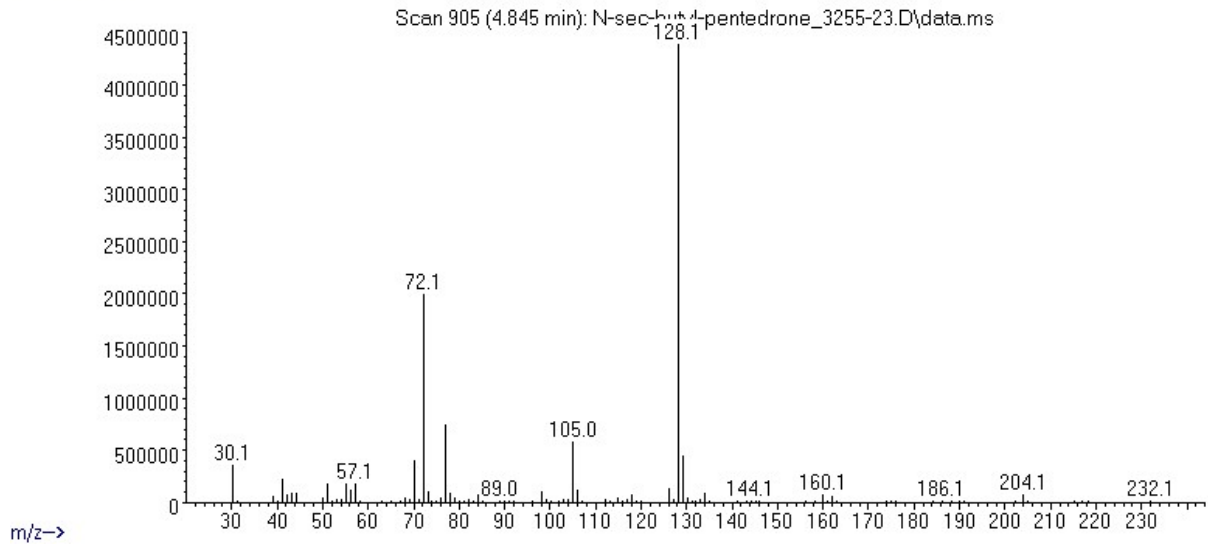
Solubility in	result/remark
CH <sub>2</sub> Cl <sub>2</sub>	/
MeOH	soluble
H <sub>2</sub> O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,85 BP(1): 128; BP(2): 72,BP(3) :77,
HPLC-TOF	+	Exact mass (theoretical): 233,178; measured value Δppm:0,05; formula:C15H23NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

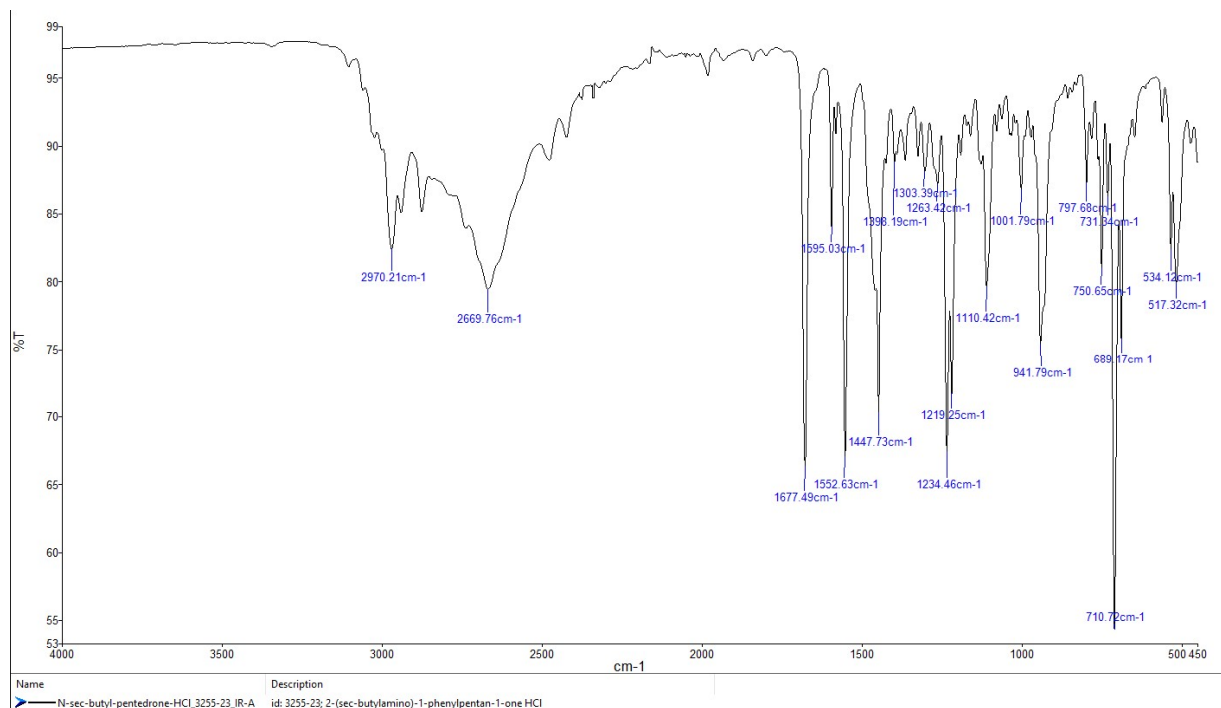
# ANALYTICAL RESULTS

MS (EI)

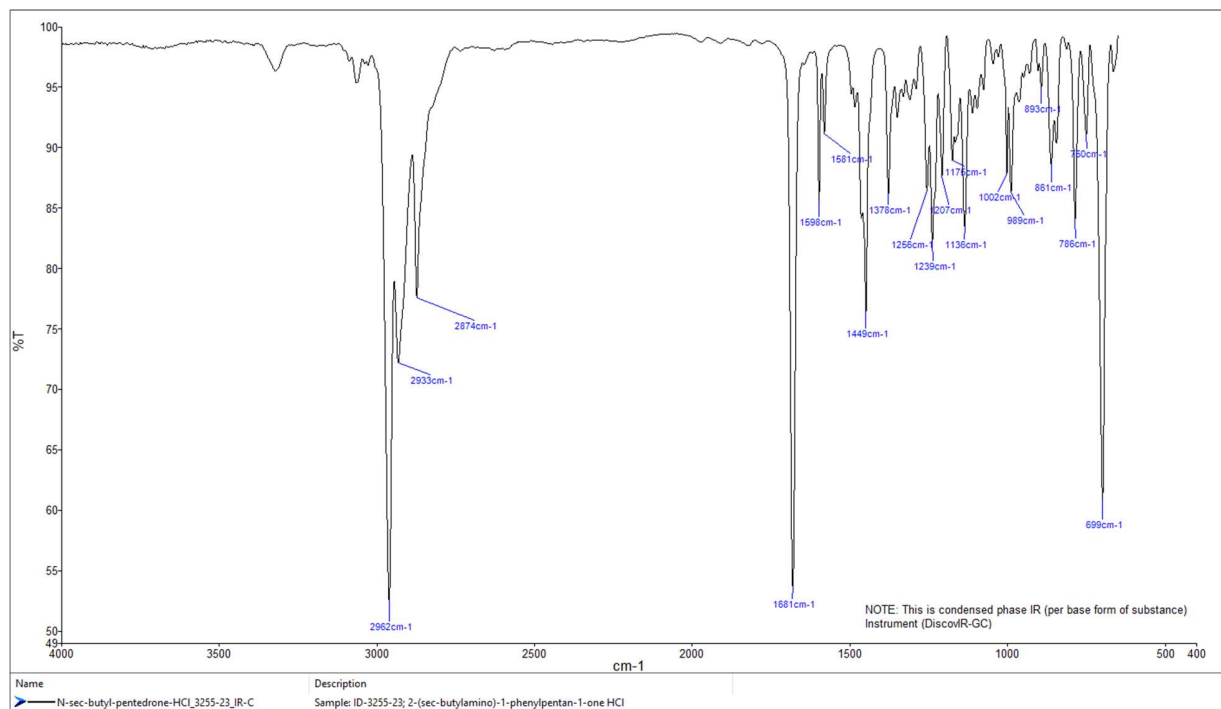
Abundance



### FTIR-ATR - direct measurement (sample as received)



### IR (solid phase – after chromatographic separation)



# TOF REPORT

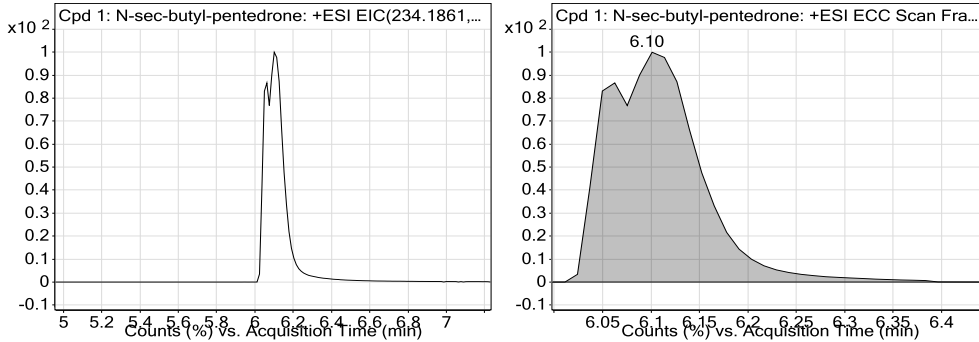
<b>Data File</b>	N-sec-butyl-pentredone.d	<b>Sample Name</b>	SOC85
<b>Sample Type</b>	Sample	<b>Position</b>	P1-A3
<b>Instrument Name</b>	6230B TOF LC-MS	<b>User Name</b>	TG
<b>Acq Method</b>	general-13_03_2023-XDB-C18-ESI+.m	<b>Acquired Time</b>	6/15/2023 11:12:13 AM
<b>IRM Calibration Status</b>	Success	<b>DA Method</b>	0-NPS in sorodne snovi.m
<b>Comment</b>	extract in MeOH		

## Compound Table

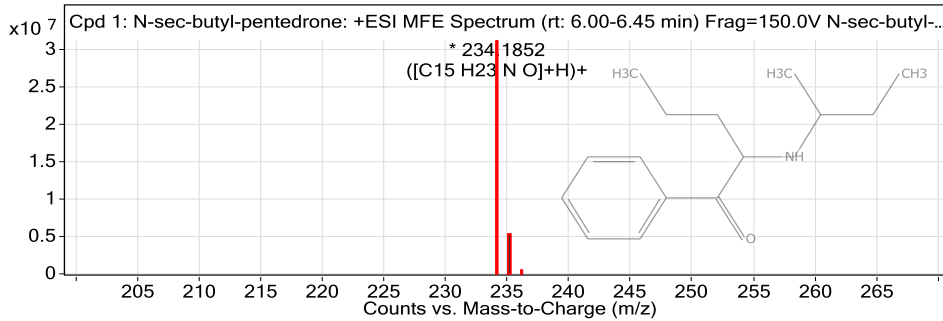
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: N-sec-butyl-pentredone	N-sec-butyl-pentredone	C15 H23 N O	6.1	233.1779

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
N-sec-butyl-pentredone	234.1852	6.1	233.1779	6.1	C15 H23 N O	233.178	0.05

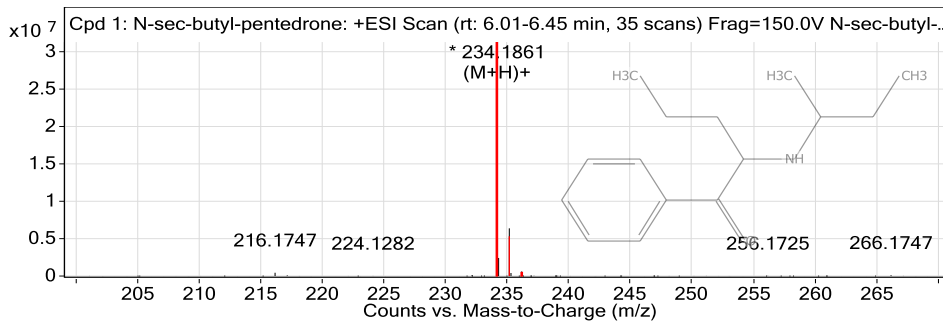
## Compound Chromatograms



## MFE MS Zoomed Spectrum



## MS Zoomed Spectrum



## MS Spectrum Peak List

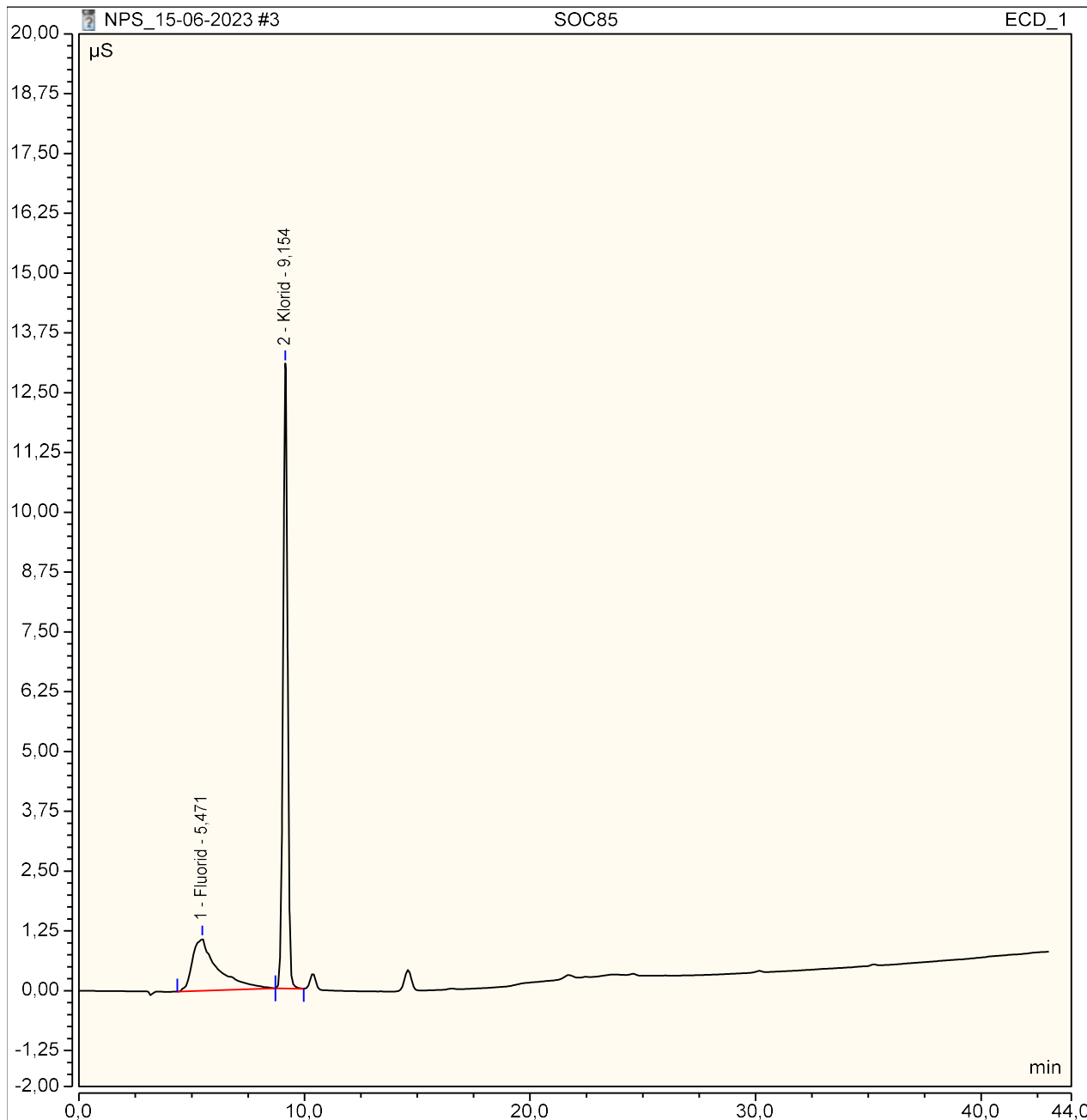
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
234.1852	1	31234644	C15 H23 N O	(M+H)+
235.1884	1	5223566.24	C15 H23 N O	(M+H)+
236.1919	1	431590.37	C15 H23 N O	(M+H)+

--- End Of Report ---

### Peak Integration Report

Sample Name:	3255-23	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Instrument Method:	ANIONI	Operator:	Admin
Inj. Date / Time:	15-Jun-2023 / 13:44	Run Time:	43,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height $\mu\text{S}$	Amount mg/L
1	5,47	Fluorid	BMB	1,412	1,094	n.a.
2	9,15	Klorid	bMB	3,005	13,064	n.a.
TOTAL:				4,42	14,16	0,00



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University  
of Ljubljana  
Faculty of Chemistry  
and Chemical Technology



## R E P O R T

Contract No.	C1714-21-460153 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	<b>3255-23 (NLZOH_SOC85)</b>
Received date:	July 14, 2023
Our notebook code:	NFL-NLZOH_SOC85
NMR sample preparation:	17.3 mg dissolved in 0.8 mL DMSO- <i>d</i> <sub>6</sub>
NMR experiments:	<sup>1</sup> H, <sup>13</sup> C, <sup>1</sup> H- <sup>1</sup> H <i>gs</i> -COSY, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HSQC, <sup>1</sup> H- <sup>13</sup> C <i>gs</i> -HMBC, <sup>13</sup> C DEPT-135, <sup>13</sup> C DEPT-90, <sup>13</sup> C DEPT-45, <sup>1</sup> H- <sup>15</sup> N <i>gs</i> -HMBC
Proposed structure with formula, exact mass, molecular weight:	
Chemical name:	<i>N</i> -protonated 2-( <i>sec</i> -butylamino)-1-phenylpentan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - Sample is pure based on <sup>1</sup> H NMR spectrum.
Supporting information:	Copies of <sup>1</sup> H and <sup>13</sup> C NMR spectra, <sup>1</sup> H and <sup>13</sup> C FIDs.
Principal investigator:	Prof. Dr. Janez Košmrlj
Date of report:	July 19, 2023



NFL-NLZOH\_SOC85  
1H

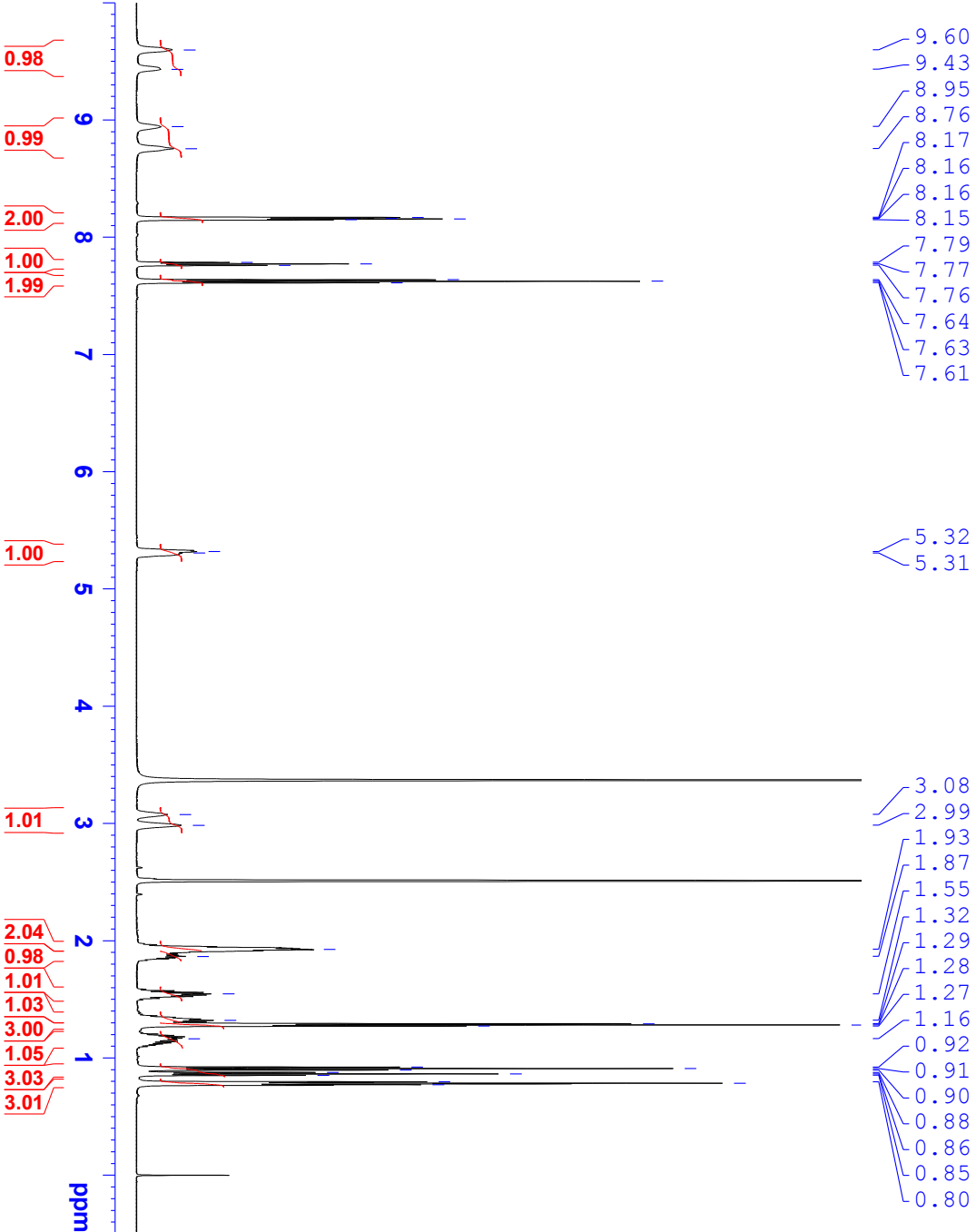


Current Data Parameters  
NAME NFL-NLZOH\_SOC85  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20230715  
Time 19.35 h  
INSTRUM AV600 NEO  
PROBHD z176567\_0002 ( PULPROG zg30  
TD 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 11904.762 Hz  
FIDRES 0.363304 Hz  
AQ 2.7525120 sec  
RG 57.7778  
DW 42.000 usec  
DE 8.79 usec  
TE 298.6 K  
D1 1.00000000 sec  
TD0 1  
SFO1 600.137058 MHz  
NUC1 1H  
P0 3.33 usec  
P1 10.00 usec  
PLWI 16.51399994 W

F2 - Processing parameters  
SI 65536  
SF 600.1299977 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



NFL-NLZOH\_S0C85  
13C



Current Data Parameters  
NAME NFL-NLZOH\_S0C85  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20230715  
Time\_ 22.11 h  
INSTRUM AV600 NEO  
PROBHD Z176567\_0002 (PULPROG zgpg30  
TD 65336  
SOLVENT DMSO  
NS 4096  
DS 4  
SWH 35714.285 Hz  
FIDRES 1.089913 Hz  
AQ 0.9175040 sec  
RG 101  
DW 14.000 usec  
DE 6.50 usec  
TE 300.1 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1  
SFO1 150.9178988 MHz  
NUC1 13C  
P0 4.00 usec  
P1 12.00 usec  
PLW1 101.26000214 W  
SFO2 600.1324005 MHz  
NUC2 1H  
CPDPRG12 waltz65  
PCPD2 70.00 usec  
PLW2 16.51399994 W  
PLM12 0.33702001 W  
PLM13 0.16952001 W

F2 - Processing parameters  
SI 32768  
SF 150.9028939 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

