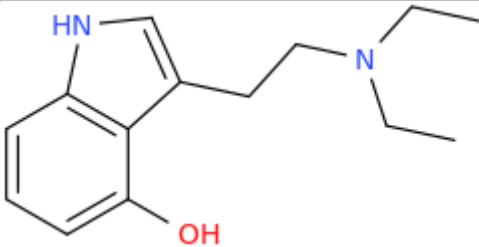


ANALYTICAL REPORT¹4-HO-DET (C₁₄H₂₀N₂O)

3-[2-(diethylamino)ethyl]-1H-indol-4-ol

Remark – other NPS detected:

Sample ID:	3019-21
Sample description:	powder
Sample type:	test purchase/NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	23/05/2022
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified - structure ² (base form)	
Systematic name	3-[2-(diethylamino)ethyl]-1H-indol-4-ol
Other names	4-Hydroxy-N,N-diethyltryptamine; Ethocin
Formula (per base form)	C ₁₄ H ₂₀ N ₂ O
M _w (g/mol)	232,33
Salt form/anions detected	fumarate
StdInChIKey (per base form)	OHHYMKDBKPILO-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	>95% purity of a sample based on 1H NMR spectrum

¹ Approved by: dr. Sonja Klemenc² 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 (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 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⁻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

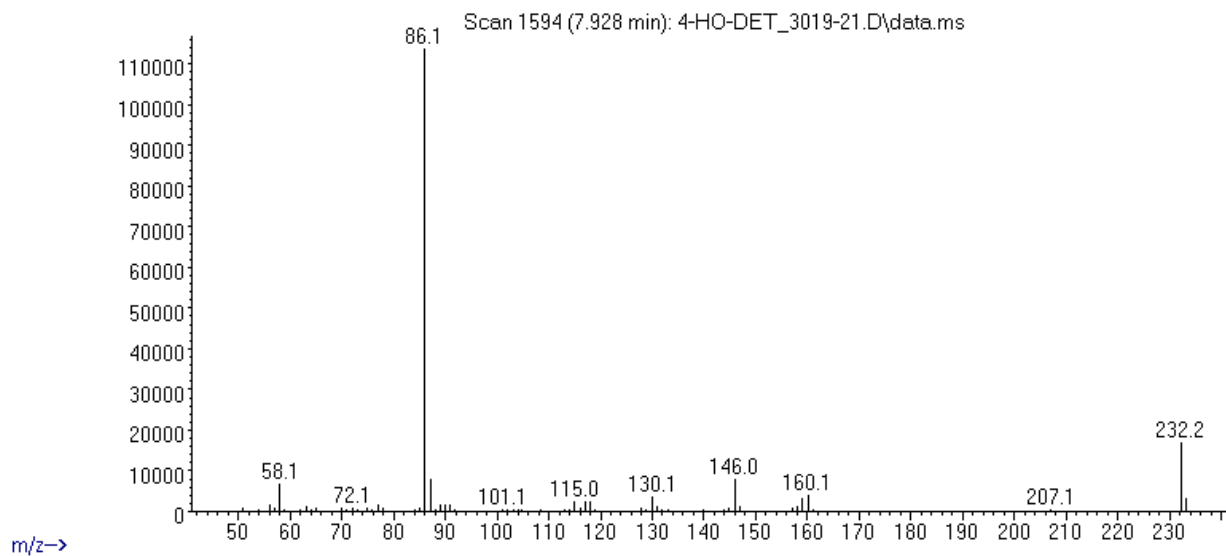
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 7,93 BP(1): 86; BP(2): 232,BP(3) :87,
HPLC-TOF	+	Exact mass (theoretical): 232,1576; measured value Δppm:0,66; formula:C14H20N2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

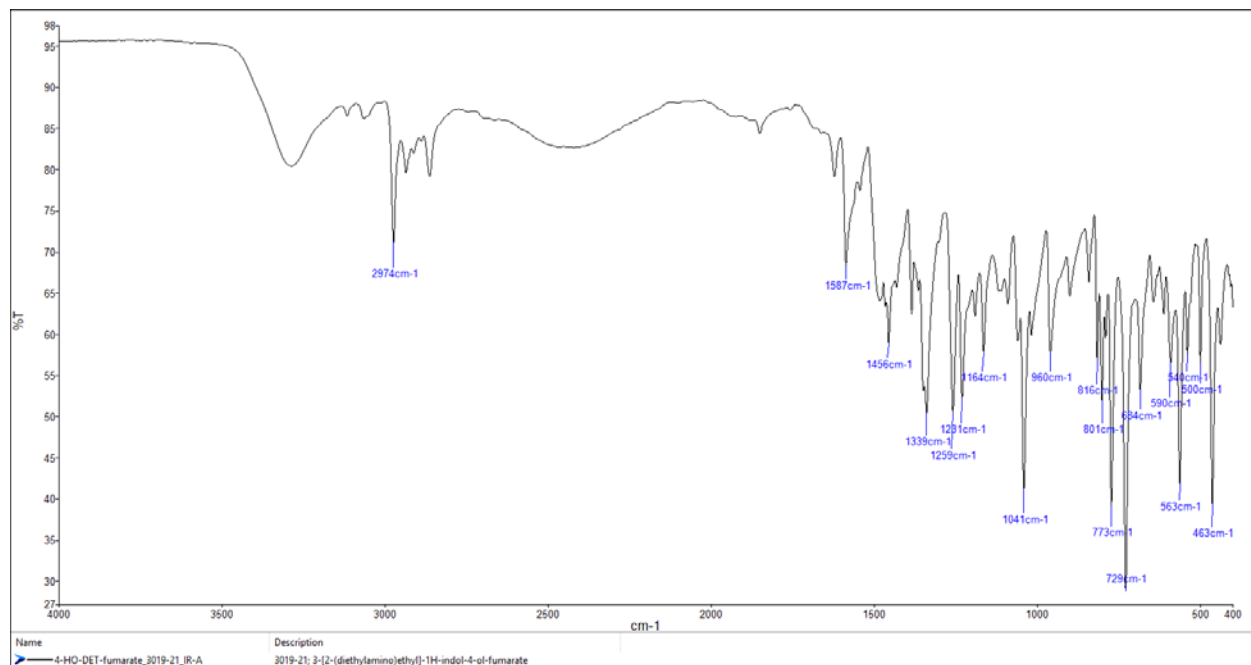
ANALYTICAL RESULTS

MS (EI)

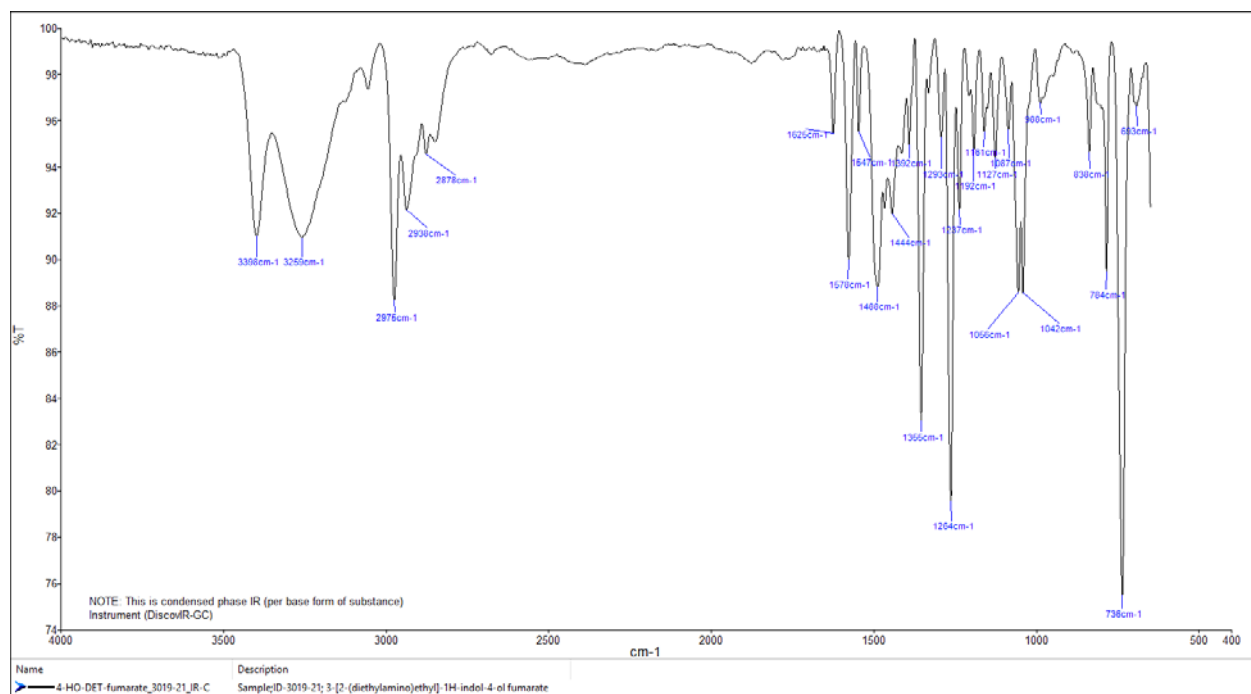
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

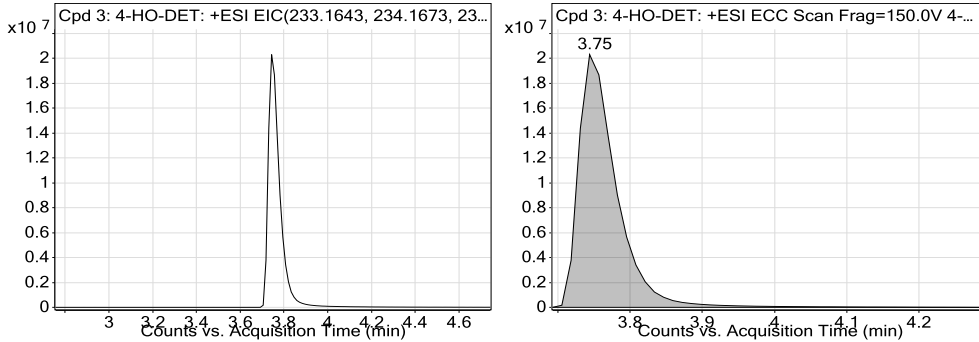
Data File	4-HO-DET_3019_21.d	Sample Name	ID-3019-21
Sample Type	Sample	Position	P1-E3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	Acquired Time	10/27/2021 10:04:52 AM
IRM Calibration Status	Success	DA Method	0-NPS in sorodne snovi.m
Comment	extract in MeOH		

Compound Table

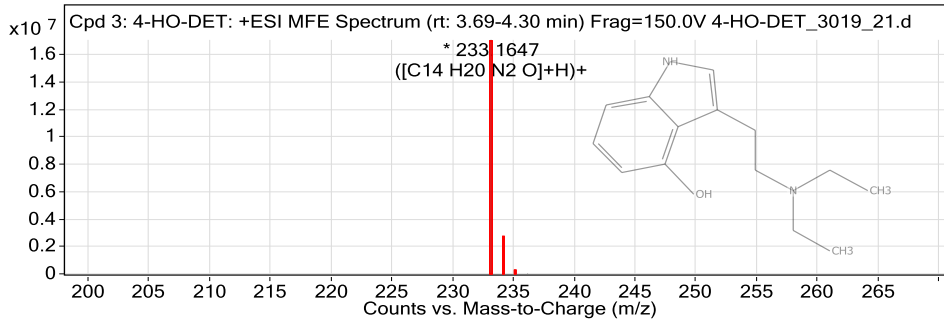
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 3: 4-HO-DET	4-HO-DET	C14 H20 N2 O	3.75	232.1574

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4-HO-DET	233.1647	3.75	232.1574	3.74	C14 H20 N2 O	232.1576	0.66

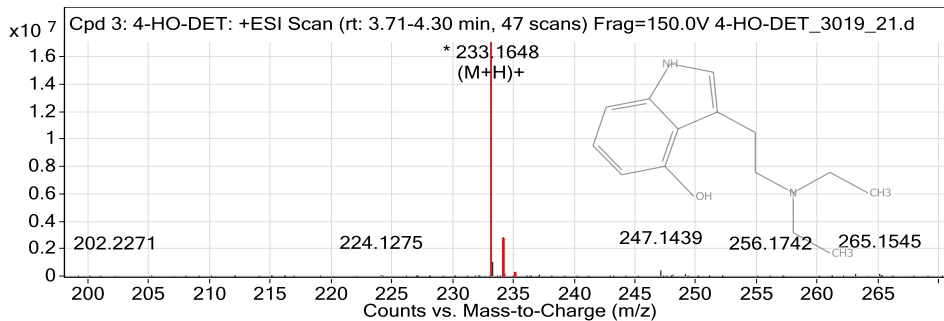
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

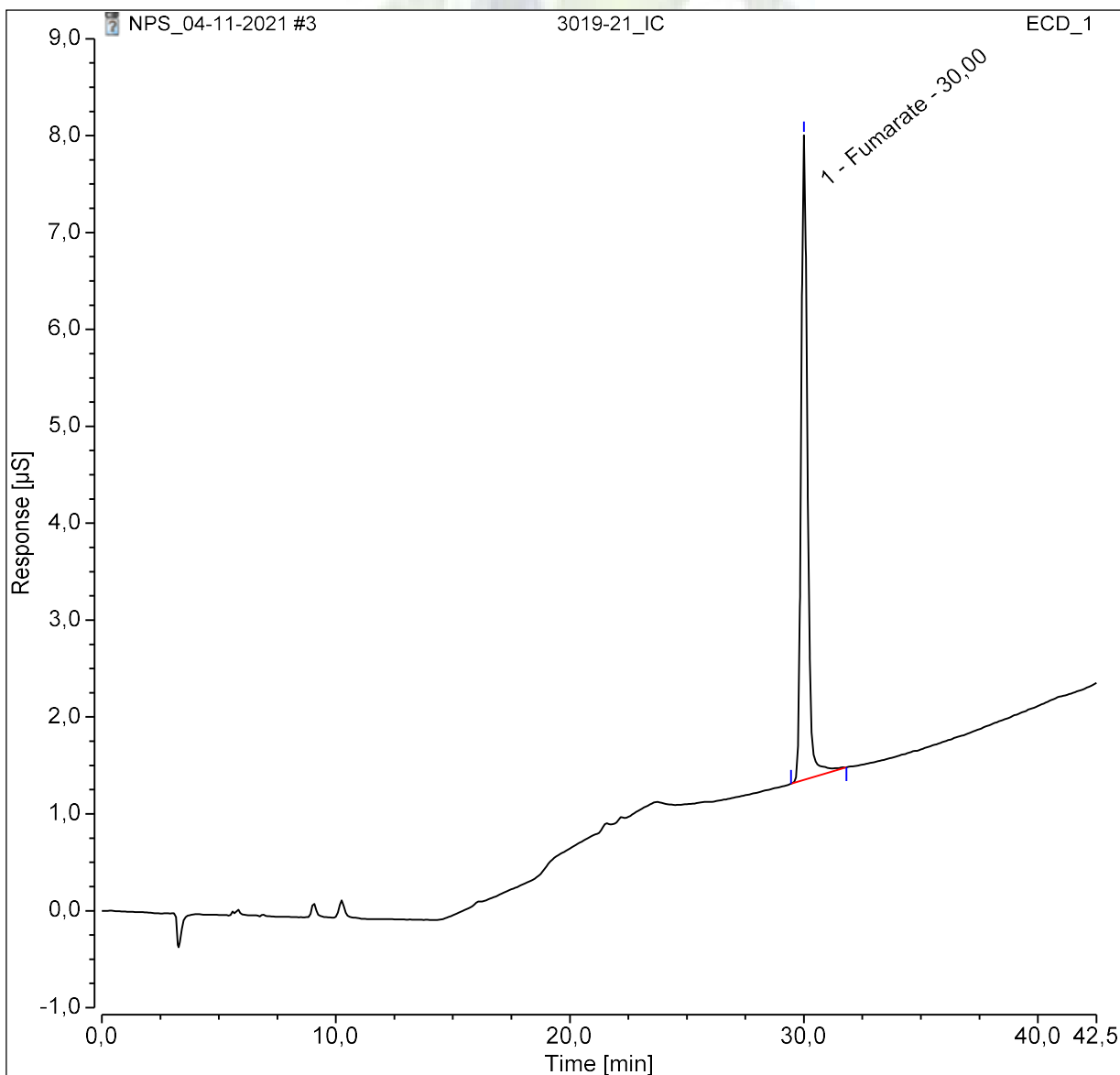
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
233.1647	1	17067760	C14 H20 N2 O	(M+H)+
234.1678	1	2729321.26	C14 H20 N2 O	(M+H)+
235.1708	1	219126.74	C14 H20 N2 O	(M+H)+
236.1725	1	13698.69	C14 H20 N2 O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	3019-21_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	Admin
Inj. Date / Time:	04-Nov-2021 / 14:36	Run Time:	43,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1	30,00	Fumarate	BMB	2,100	6,655	n.a.
TOTAL:				2,10	6,65	0,0

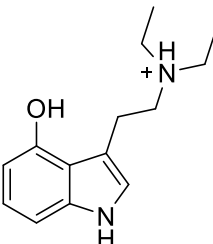


University
of Ljubljana

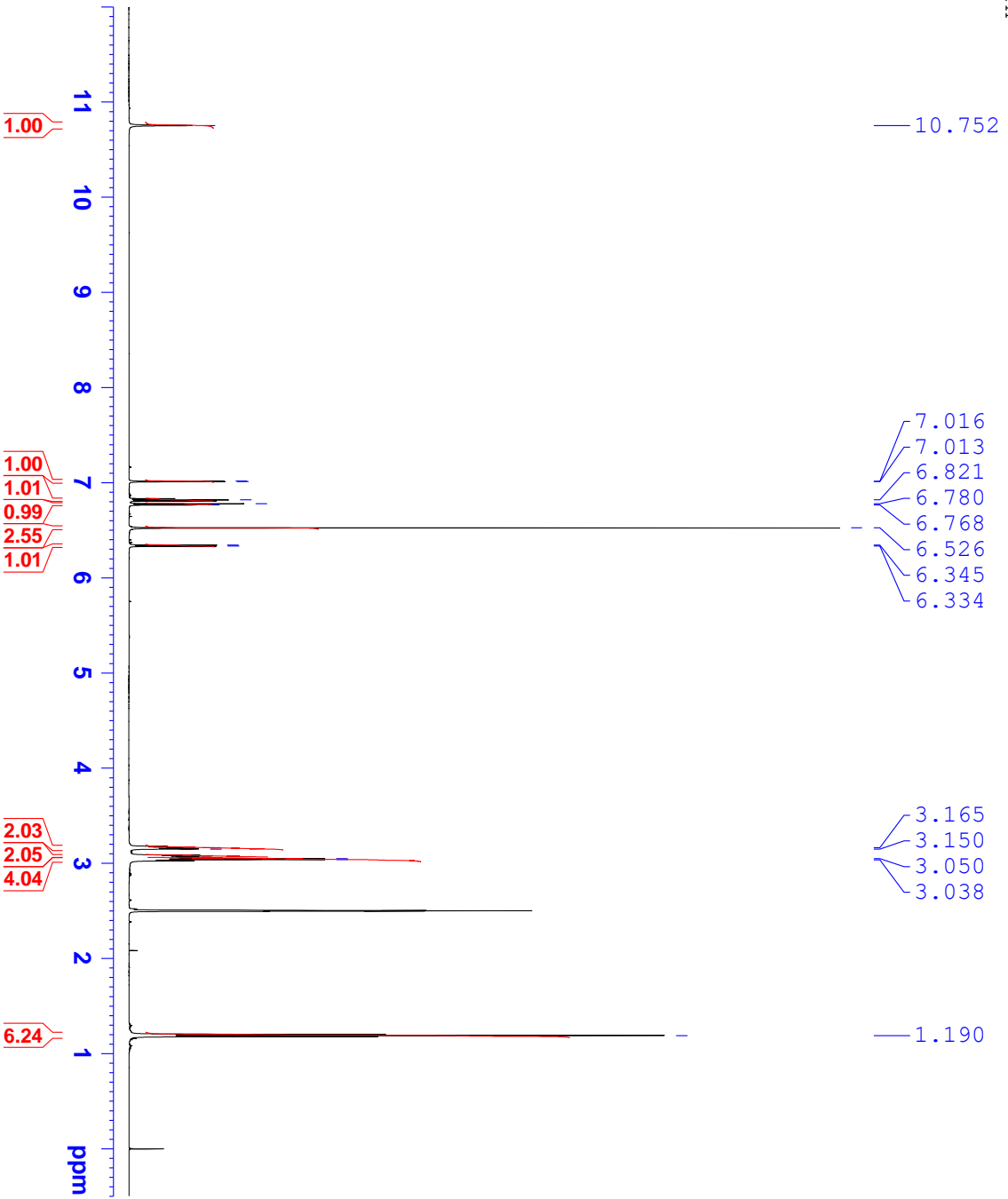
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-21-460153 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	3019-21
Received date:	March 14, 2022
Our notebook code:	NFL-3019-21
NMR sample preparation:	21.1 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:	 <p>Chemical Formula: C₁₄H₂₁N₂O⁺ Exact Mass: 233,1648 Molecular Weight: 233,3345</p>
Chemical name:	<i>N</i> -protonated 3-(2-(diethylamino)ethyl)-1 <i>H</i> -indol-4-ol
Comments:	<ul style="list-style-type: none">- Structure elucidation based on 1D and 2D NMR spectra and HRMS.- The sample consist of herein identified compound and fumarate ion in a molar ratio of 1 : 1.3, based on ¹H NMR spectrum.->95% 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:	April 3, 2022

NFL-3019-21
1H

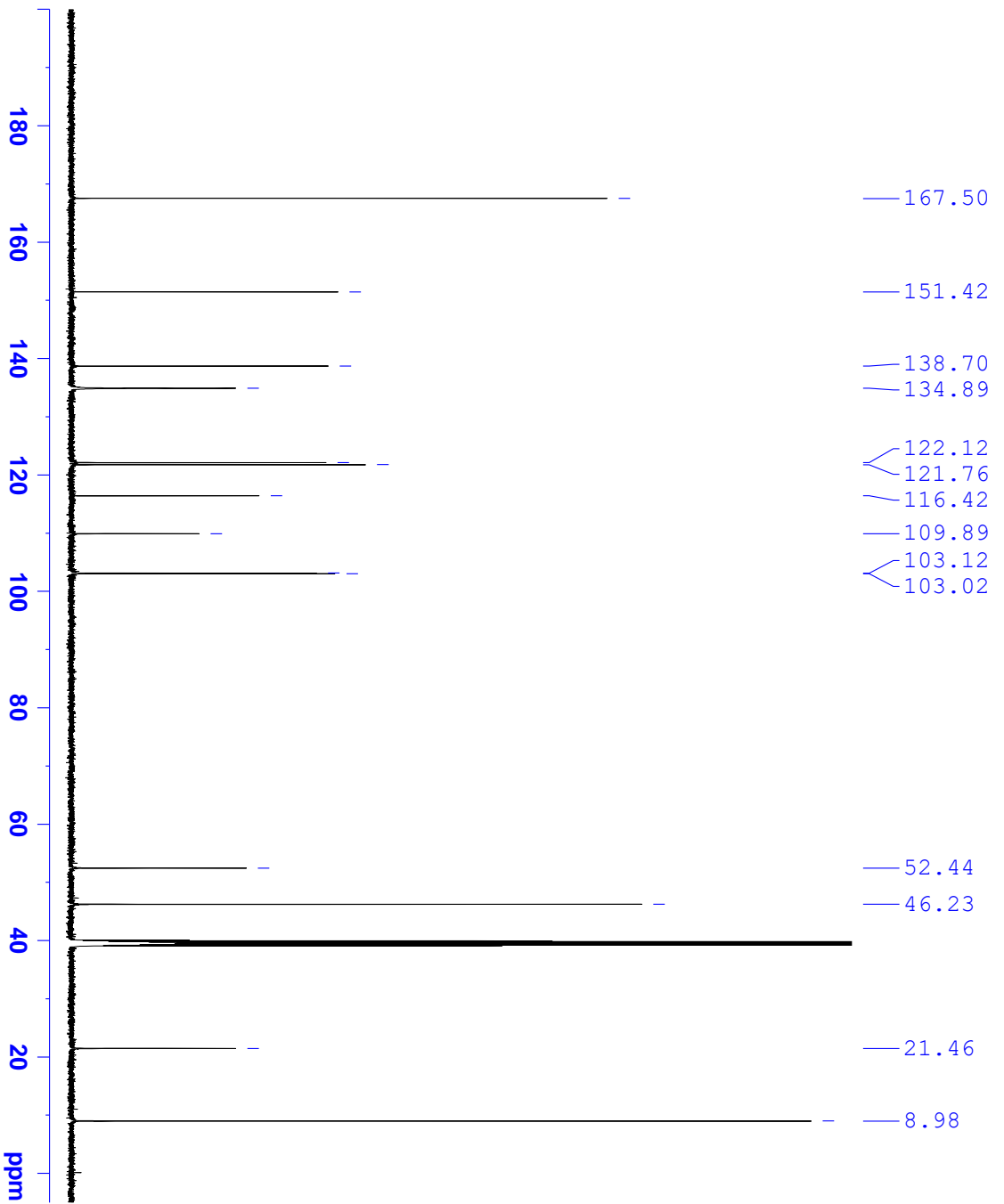


Current Data Parameters
NAME NFL-3019-21
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220314
Time_ 16:22 h
INSTRUM AV600 NEO
PROBHD z176567_0002 (PULPROG zg30)
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 11904.762 Hz
FIDRES 0.363304 Hz
AQ 2.7525120 sec
RG 54.7368
DW 42.000 usec
DE 8.79 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1
SFO1 600.1337058 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLM1 16.51399994 W

F2 - Processing parameters
SI 65536
SF 600.1300048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NFL-3019-21
13C



Current Data Parameters
 NAME NFL-3019-21
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20220314
 Time 18.04 h
 INSTRUM AV600 NEO
 PROBHD Z176567_0002 (

PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 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 298.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec

TD0 1
 SF01 150.9178988 MHz
 NUC1 13C
 P0 4.00 usec
 P1 12.00 usec

PLW1 101.26000214 W
 SFO2 600.1324005 MHz
 NUC2 1H
 NUC3 13C

CPDPRG12 waltz65
 PCPD2 70.00 usec
 PLW2 16.51399994 W
 PLW12 0.33702001 W
 PLW13 0.16952001 W

F2 - Processing parameters
 SI 32768
 SF 150.9028772 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40