

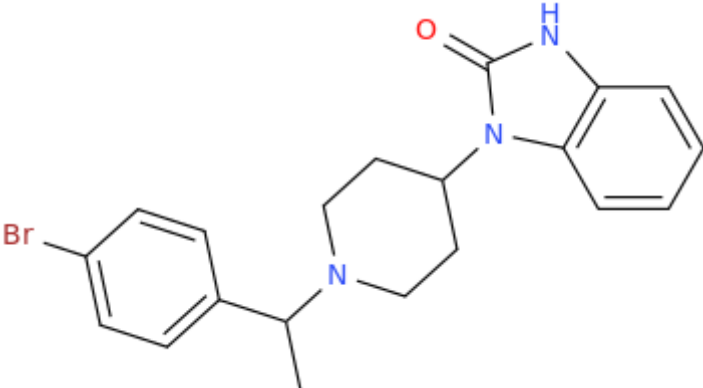
ANALYTICAL REPORT

Brorphine (C₂₀H₂₂BrN₃O)

1-{1-[1-(4-bromophenyl)ethyl]piperidin-4-yl}-2,3-dihydro-1H-1,3-benzodiazol-2-one

Remark – other NPS detected: none

Sample ID:	2178-20
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	09/07/2020
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-{1-[1-(4-bromophenyl)ethyl]piperidin-4-yl}-2,3-dihydro-1H-1,3-benzodiazol-2-one
Other names	1-(1-(1-(4-bromophenyl)ethyl)piperidin-4-yl)-1,3-dihydro-2H-benzo[d]imidazol-2-one
Formula (per base form)	C ₂₀ H ₂₂ BrN ₃ O
M _w (g/mol)	400,32
Salt form/anions detected	base
StdInChIKey (per base form)	CNOFBGYRMCBVLO-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>97% pure based on 1H NMR spectrum

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 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 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 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

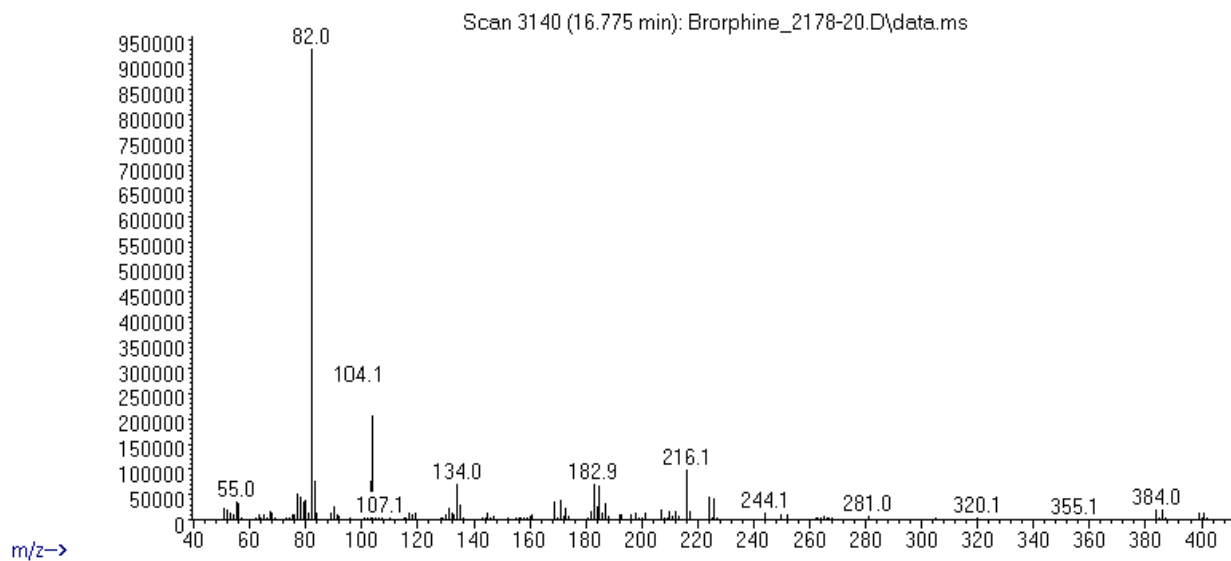
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	partially
H ₂ O	low (bad)

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 16,78 BP(1): 82; BP(2): 104,BP(3) :216,
HPLC-TOF	+	Exact mass (theoretical): 399,0946; measured value Δppm:-0,65; formula:C20H22BrN3O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

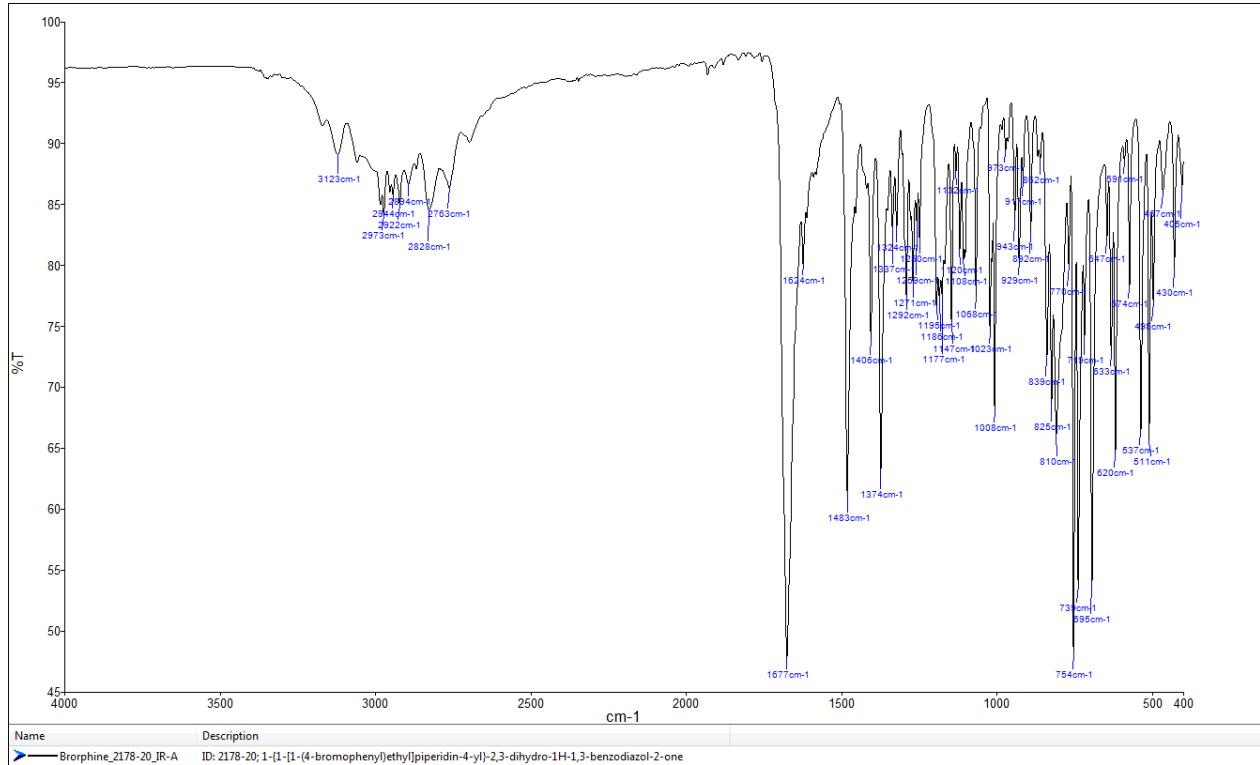
ANALYTICAL RESULTS

MS (EI)

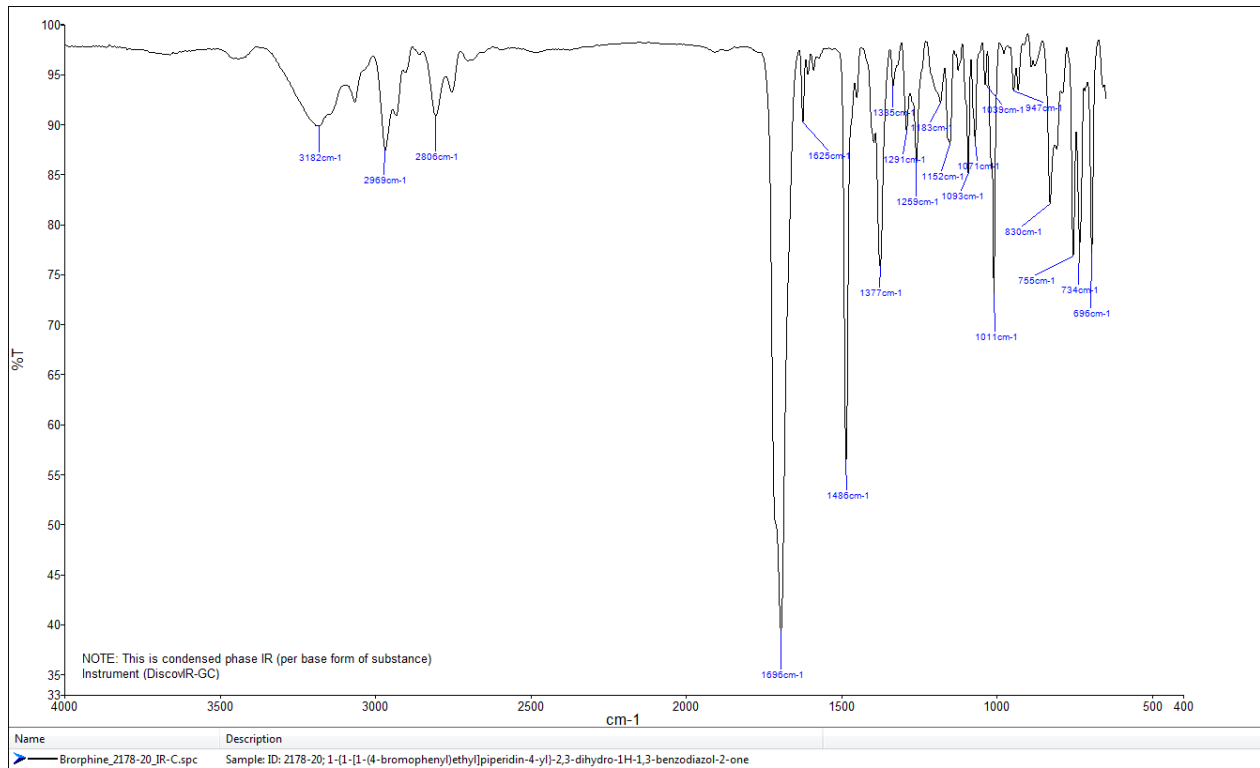
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

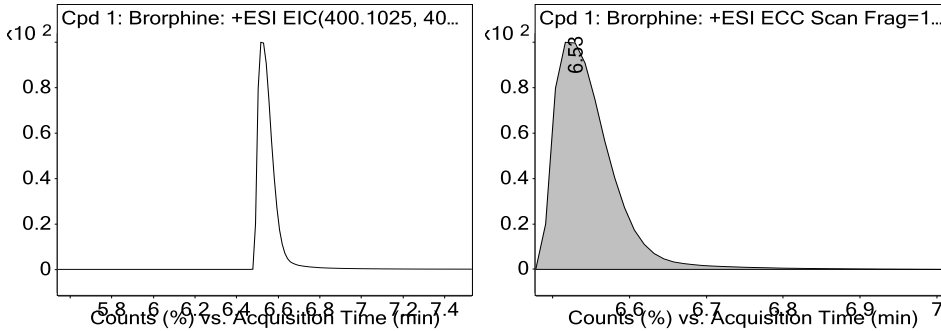
Data File	Brorphine_2178_20.d	Sample Name	ID-2178-20
Sample Type	Sample	Position	P1-A7
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	Acquired Time	6/5/2020 1:23:26 PM
IRM Calibration Status	Success	DA Method	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

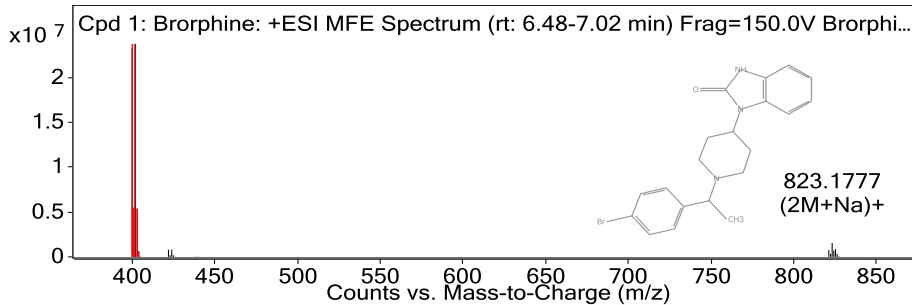
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 1: Brorphine	Brorphine	6.53	399.0949

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Brorphine	400.102	6.53	399.0949	6.53	C20 H22 Br N3 O	399.0946	-0.65

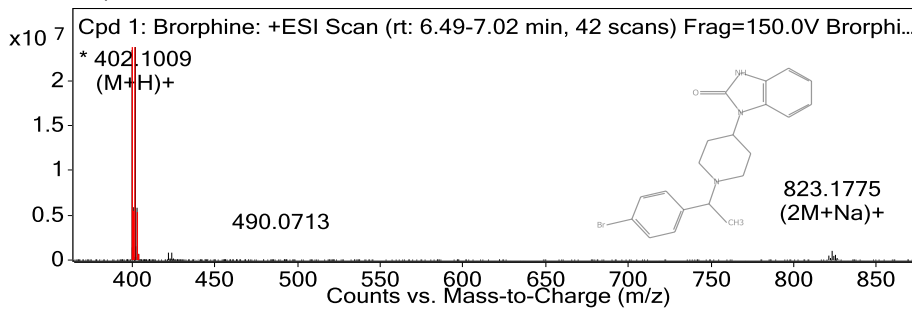
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

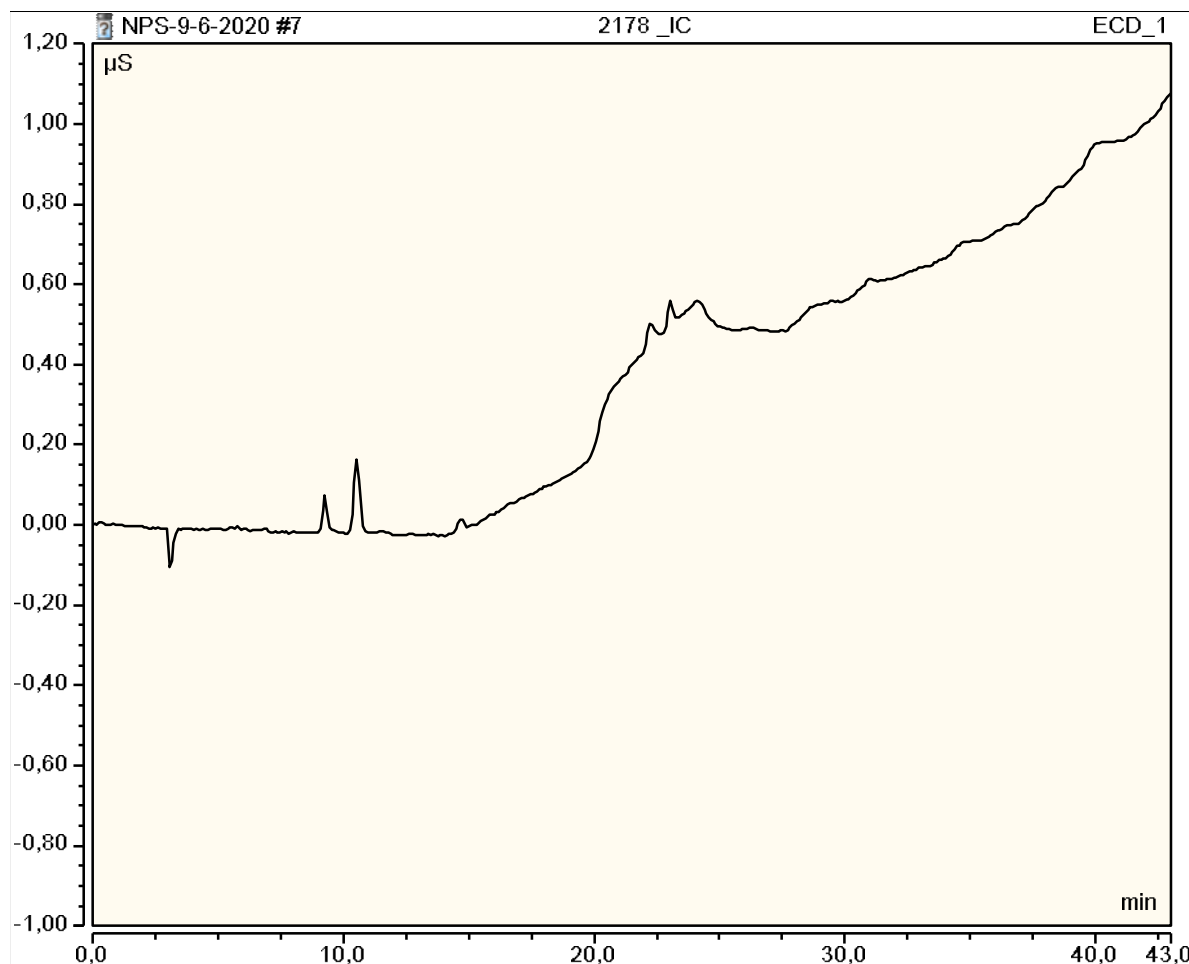
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
400.102	1	23293763.59	C20 H22 Br N3 O	(M+H)+
401.1055	1	5457005.4	C20 H22 Br N3 O	(M+H)+
402.1003	1	23732032	C20 H22 Br N3 O	(M+H)+
403.1035	1	5393805.38	C20 H22 Br N3 O	(M+H)+
422.0845	1	796946.62		(M+Na)+
424.0827	1	797818.75		(M+Na)+
821.1795	1	748955.05		(2M+Na)+
823.1777	1	1545961.75		(2M+Na)+
824.1808	1	698029.63		(2M+Na)+
825.1772	1	873745.11		(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	2178_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	09-jun-2020 / 14:07	Run Time:	43,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height μS	Amount n.a.
TOTAL:				0,00	0,00	0,0

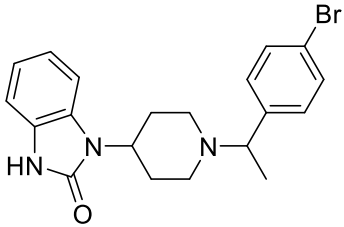


University
of Ljubljana

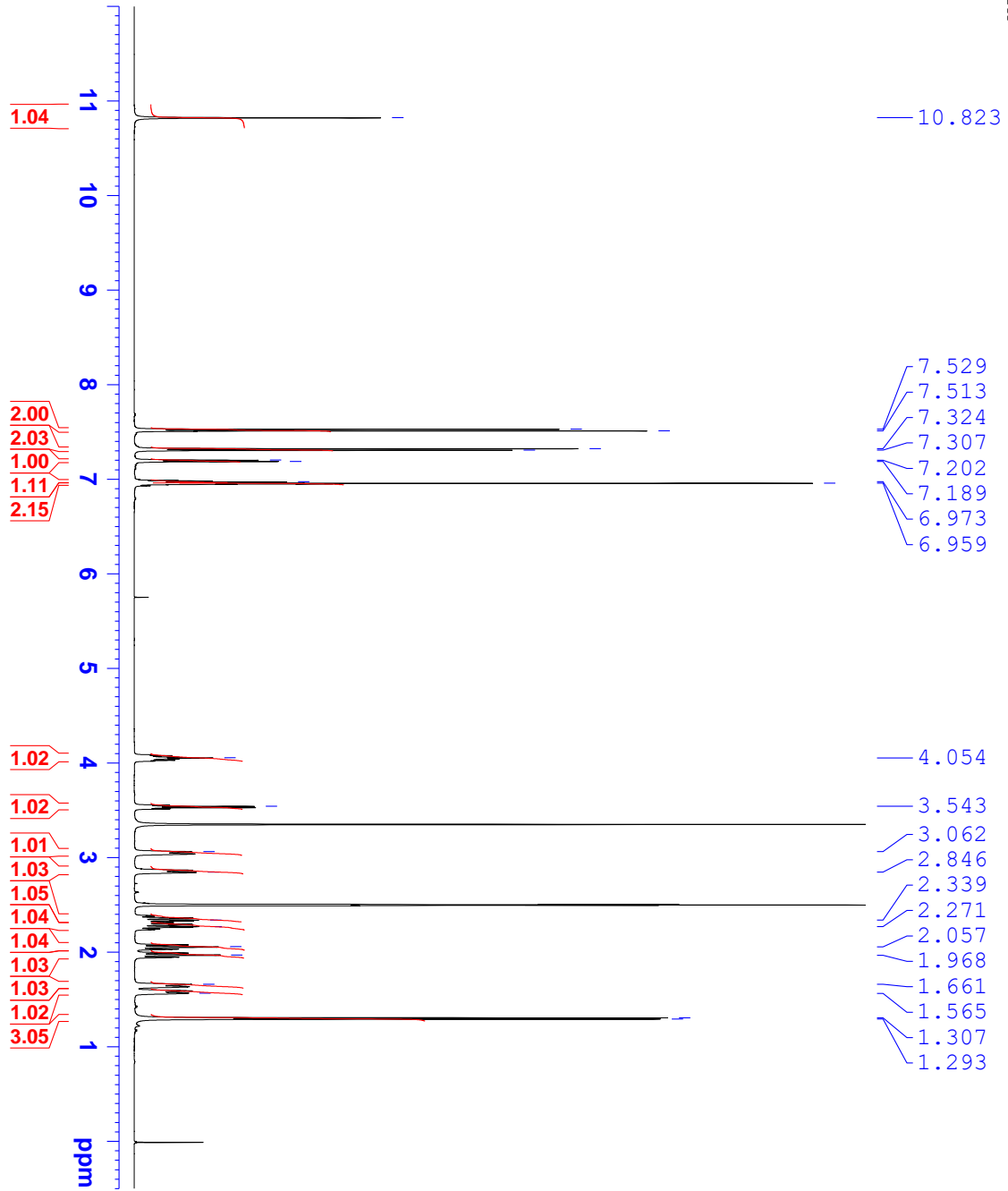
Faculty of Chemistry
and Chemical Technology



R E P O R T

Contract No.	C1714-19-460155 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	2178-20
Received date:	June 15, 2020
Our notebook code:	NFL-2178-20
NMR sample preparation:	21.5 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: C₂₀H₂₂BrN₃O Exact Mass: 399,0946 Molecular Weight: 400,3200</p>
Chemical name:	1-(1-(1-(4-bromophenyl)ethyl)piperidin-4-yl)-1,3-dihydro-2H-benzo[<i>d</i>]imidazol-2-one
Comments:	<ul style="list-style-type: none">- Structure elucidation based on 1D and 2D NMR spectra and HRMS.- >97% purity of a sample based on ¹H NMR spectrum.- Spectroscopic data are in agreement with the literature report (Kennedy, N. M.; et. al. <i>J. Med. Chem.</i> 2018, <i>61</i>, 8895–8907).
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:	July 1, 2020

NFL-2178-20
1H



Current Data Parameters
NAME NFL-2178-20
EXPNO 1
PROCNO 1

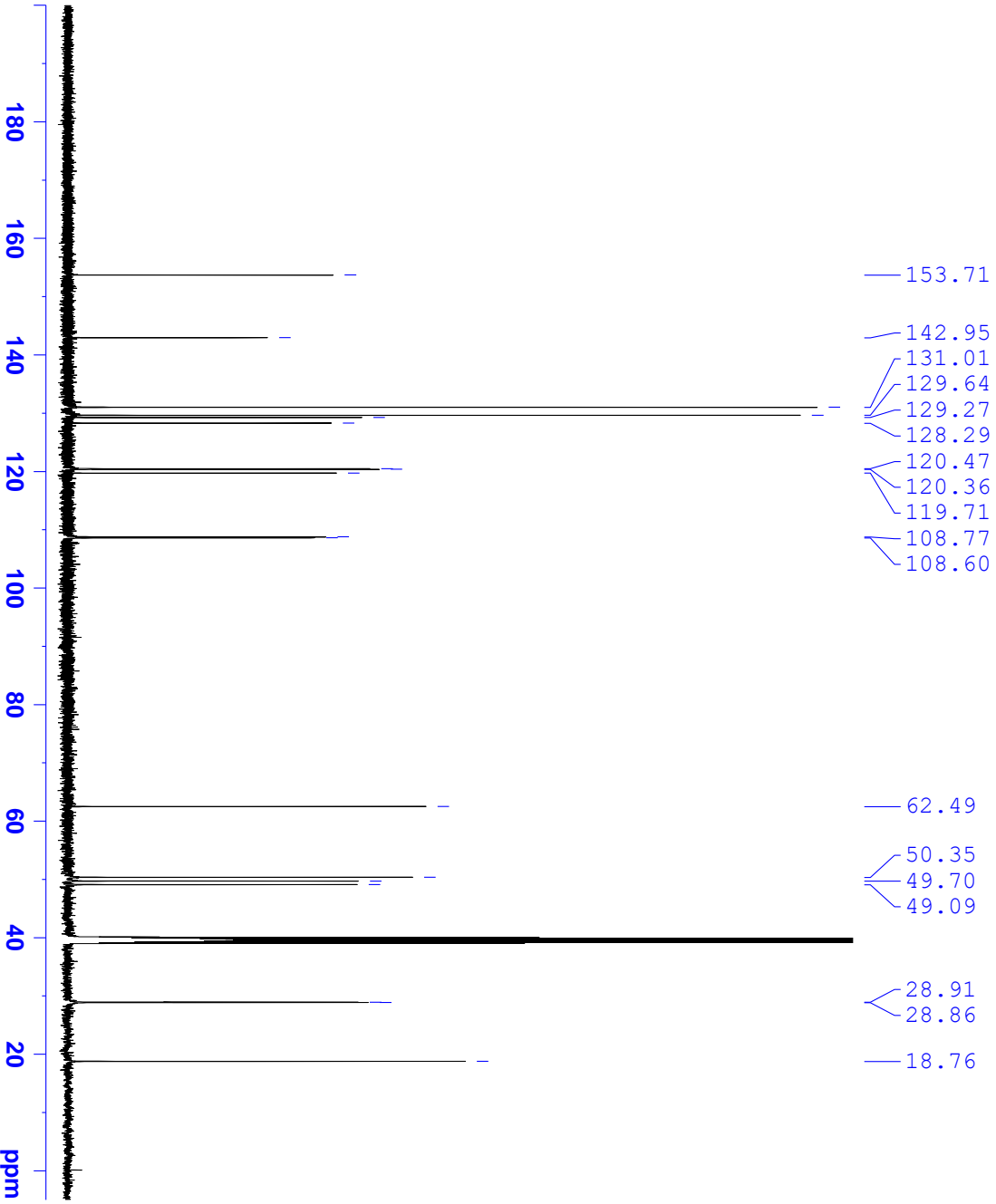
F2 - Acquisition Parameters
Date_ 20200617
Time 22.01

INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2

SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 80.6
DM 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
PLW1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME NFL-2178-20
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200617
Time 23.54

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
PLM1 122.0000000 W

==== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 1H
CPPPRG12 waltz16
PCPD2 80.00 usec
PLM2 26.00000000 W
PLM12 0.30046001 W
PLM13 0.15113001 W

F2 - Processing parameters
SI 32768
SF 125.7578465 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40