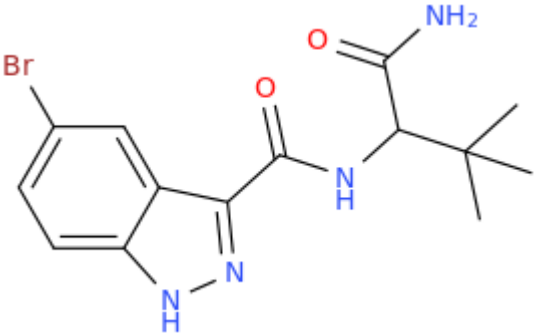


ANALYTICAL REPORT¹ADB-5Br-INACA (C₁₄H₁₇BrN₄O₂)

2-[(5-bromo-1H-indazol-3-yl)formamido]-3,3-dimethylbutanamide

Remark – other NPS detected: none

Sample ID:	3177-22
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	20/10/2022
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	2-[(5-bromo-1H-indazol-3-yl)formamido]-3,3-dimethylbutanamide
Other names	N-(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-5-bromo-1H-indazole-3-carboxamide
Formula (per base form)	C ₁₄ H ₁₇ BrN ₄ O ₂
M _w (g/mol)	353,22
Salt form/anions detected	base
StdInChIKey (per base form)	AJGASUCDTSLMNP-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	70% 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 μ l 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 μ m. 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 quadrupole 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 μ l. 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 solid phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 μ l 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 quadrupole 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 μ l

Supporting information

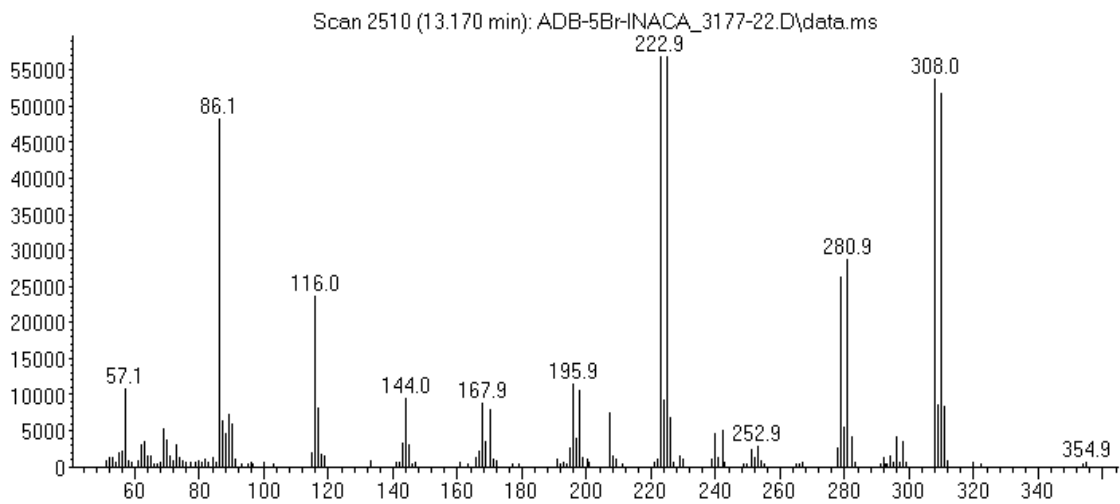
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,17 BP(1): 223; BP(2): 225,BP(3) :308,
HPLC-TOF	+	Exact mass (theoretical): 352,0535; measured value Δppm:0,13; formula:C14H17BrN4O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

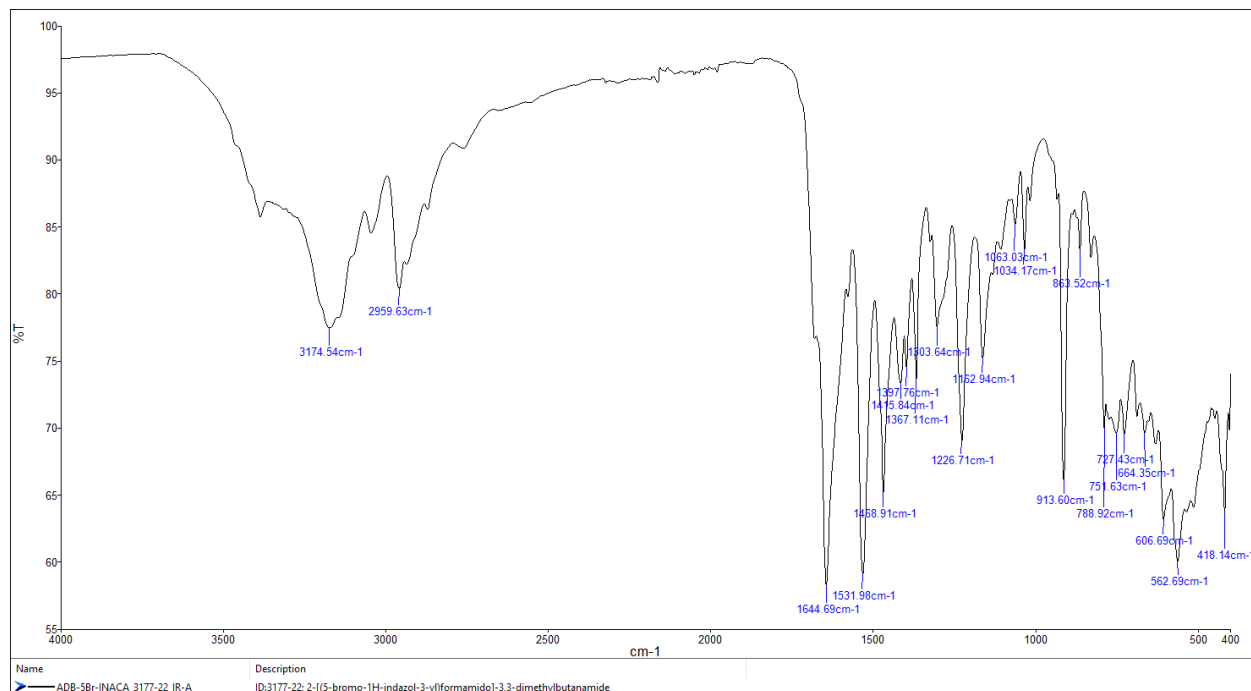
MS (EI)

Abundance

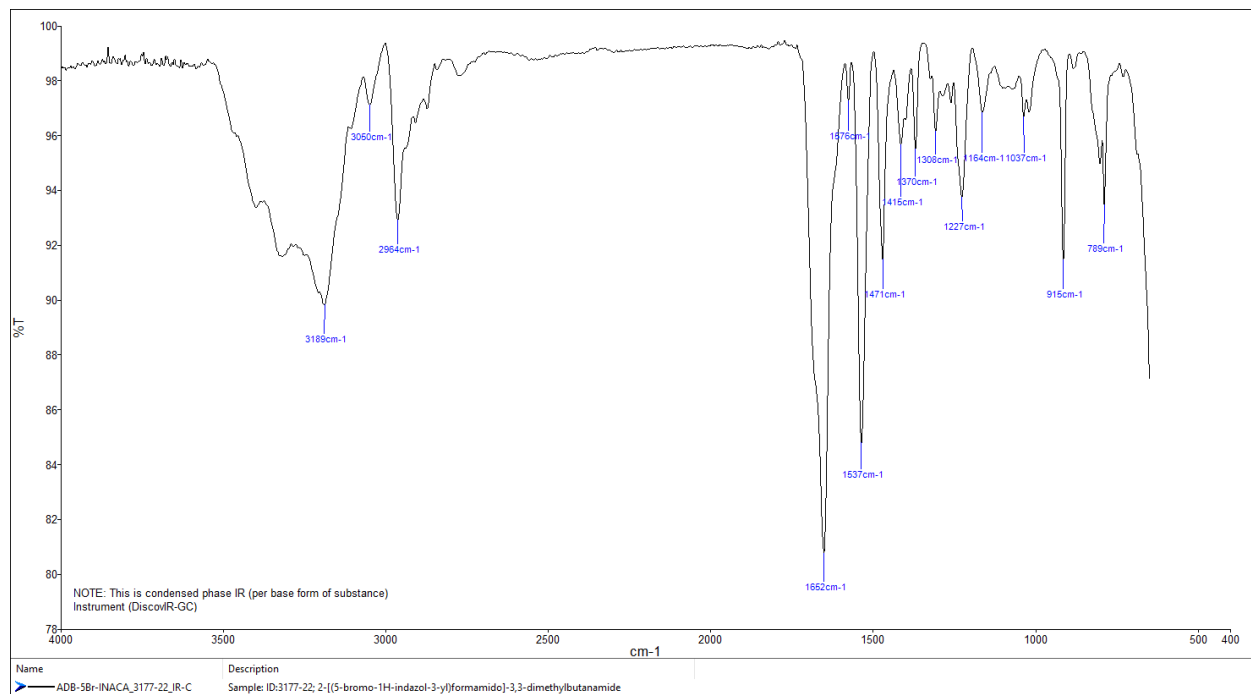


m/z→

FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

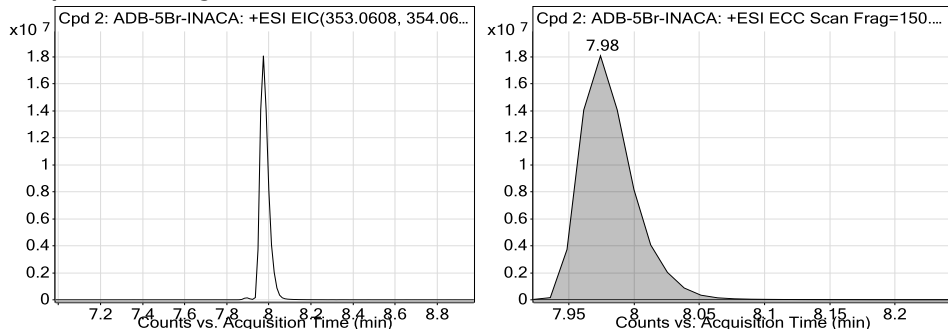
Data File	ADB-5Br-INACA_3177_22.d	Sample Name	ID-3177-22
Sample Type	Sample	Position	P1-B3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	Acquired Time	7/7/2022 7:40:08 AM
IRM Calibration Status	Success	DA Method	0-NPS in sorodne snovi.m
Comment	extract in MeOH		

Compound Table

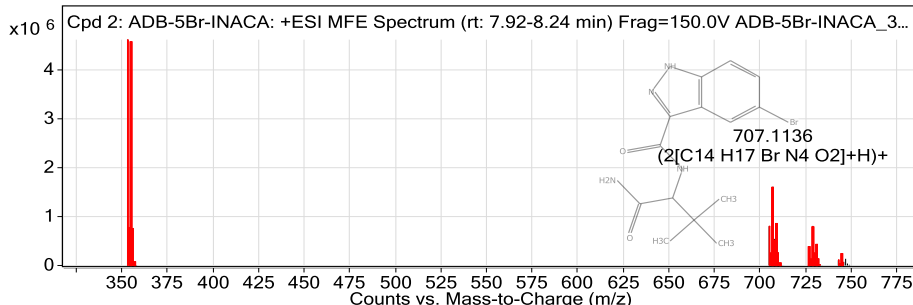
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: ADB-5Br-INACA	ADB-5Br-INACA	C14 H17 Br N4 O2	7.98	352.0534

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
ADB-5Br-INACA	353.0614	7.98	352.0534	7.98	C14 H17 Br N4 O2	352.0535	0.13

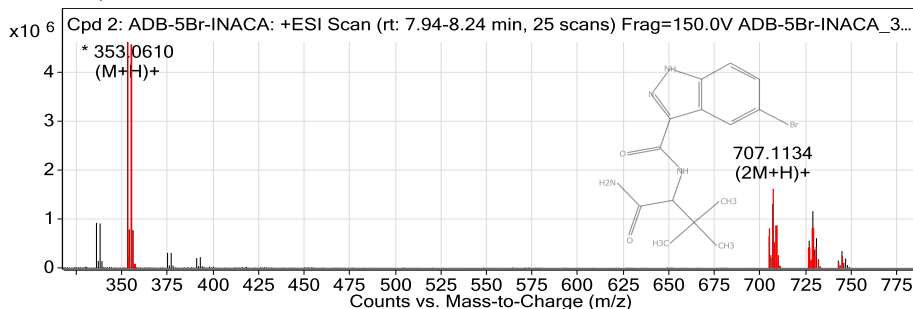
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

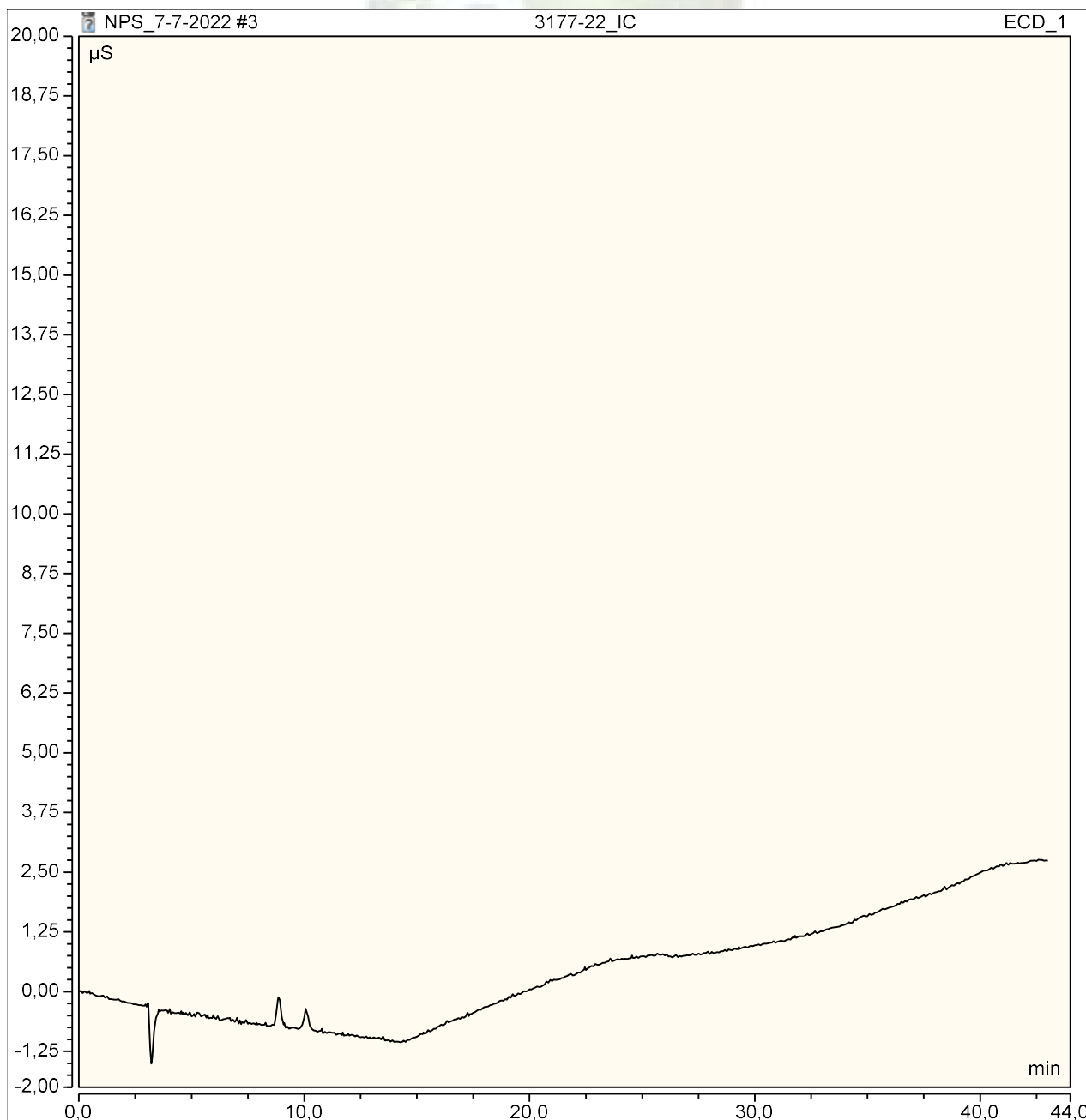
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
353.0614	1	4618522	C14 H17 Br N4 O2	(M+H)+
354.0646	1	773770.9	C14 H17 Br N4 O2	(M+H)+
355.0594	1	4568078.26	C14 H17 Br N4 O2	(M+H)+
356.0626	1	767676.87	C14 H17 Br N4 O2	(M+H)+
705.1152	1	801874.38	C14 H17 Br N4 O2	(2M+H)+
707.1136	1	1610342.38	C14 H17 Br N4 O2	(2M+H)+
708.1161	1	544307.92	C14 H17 Br N4 O2	(2M+H)+
709.1123	1	853369.21	C14 H17 Br N4 O2	(2M+H)+
729.0955	1	809609.06	C14 H17 Br N4 O2	(2M+Na)+
731.0938	1	423423.69	C14 H17 Br N4 O2	(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	3177-22_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Instrument Method:	ANIONI	Operator:	Admin
Inj. Date / Time:	07-Jul-2022 / 10:16	Run Time:	43,00

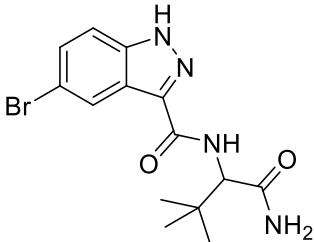
No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height μS	Amount n.a.
TOTAL:				0,00	0,00	0,00



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:	3177-22
Received date:	September 29, 2022
Our notebook code:	NFL-3177-22
NMR sample preparation:	15.1 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: C₁₄H₁₇BrN₄O₂ Exact Mass: 352,0535 Molecular Weight: 353,2200</p>
Chemical name:	<i>N</i> -(1-amino-3,3-dimethyl-1-oxobutan-2-yl)-5-bromo-1 <i>H</i> -indazole-3-carboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra. - ~70% purity of a sample based on ¹ H 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:	October 5, 2022

NFL-3177-22
 1H

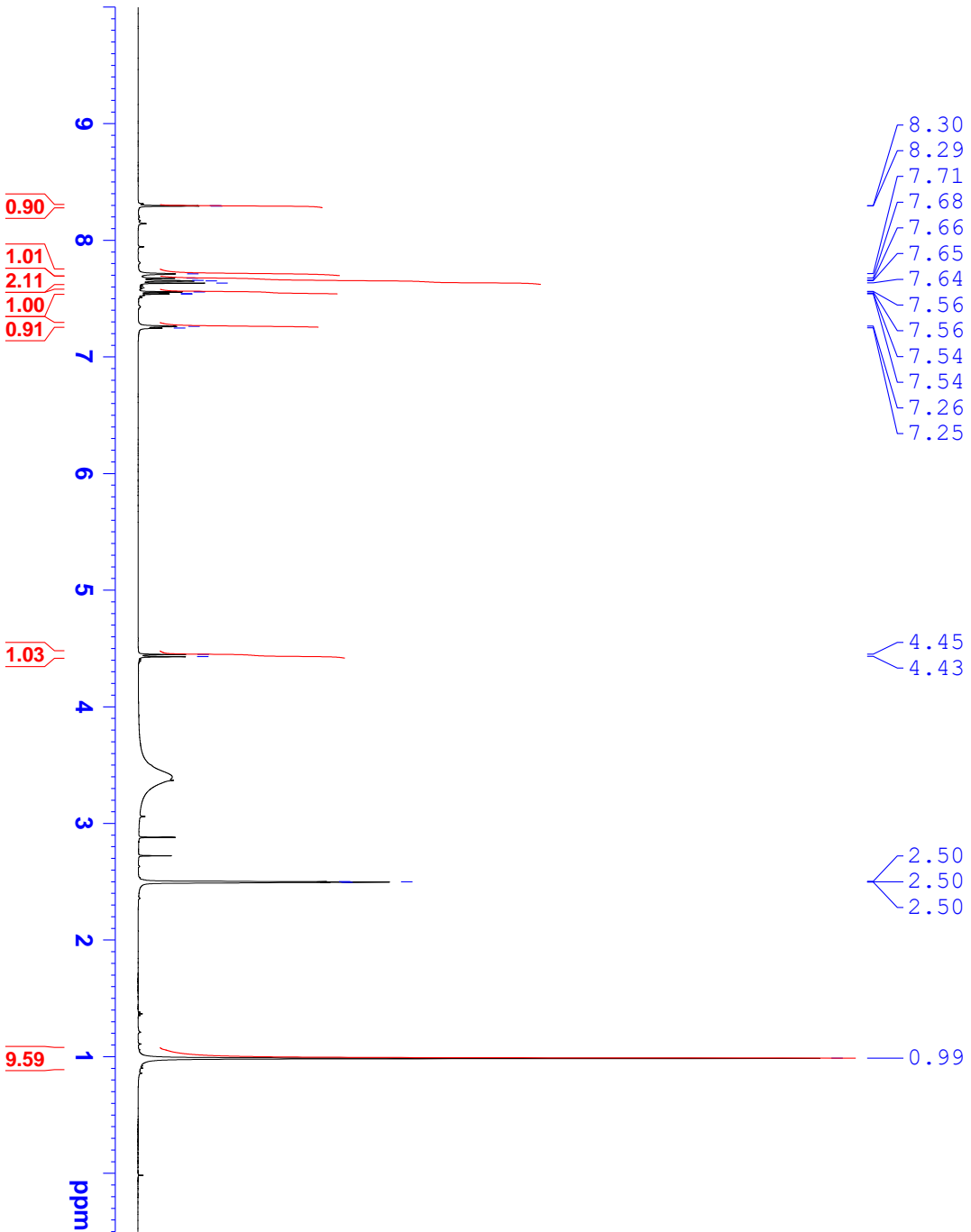


Current Data Parameters
 NAME NFL-3177-22
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20220719
 Time 17.58
 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 3.2767999 sec
 RG 101
 DW 50.000 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 8.70 usec
 PLW1 26.00000000 W

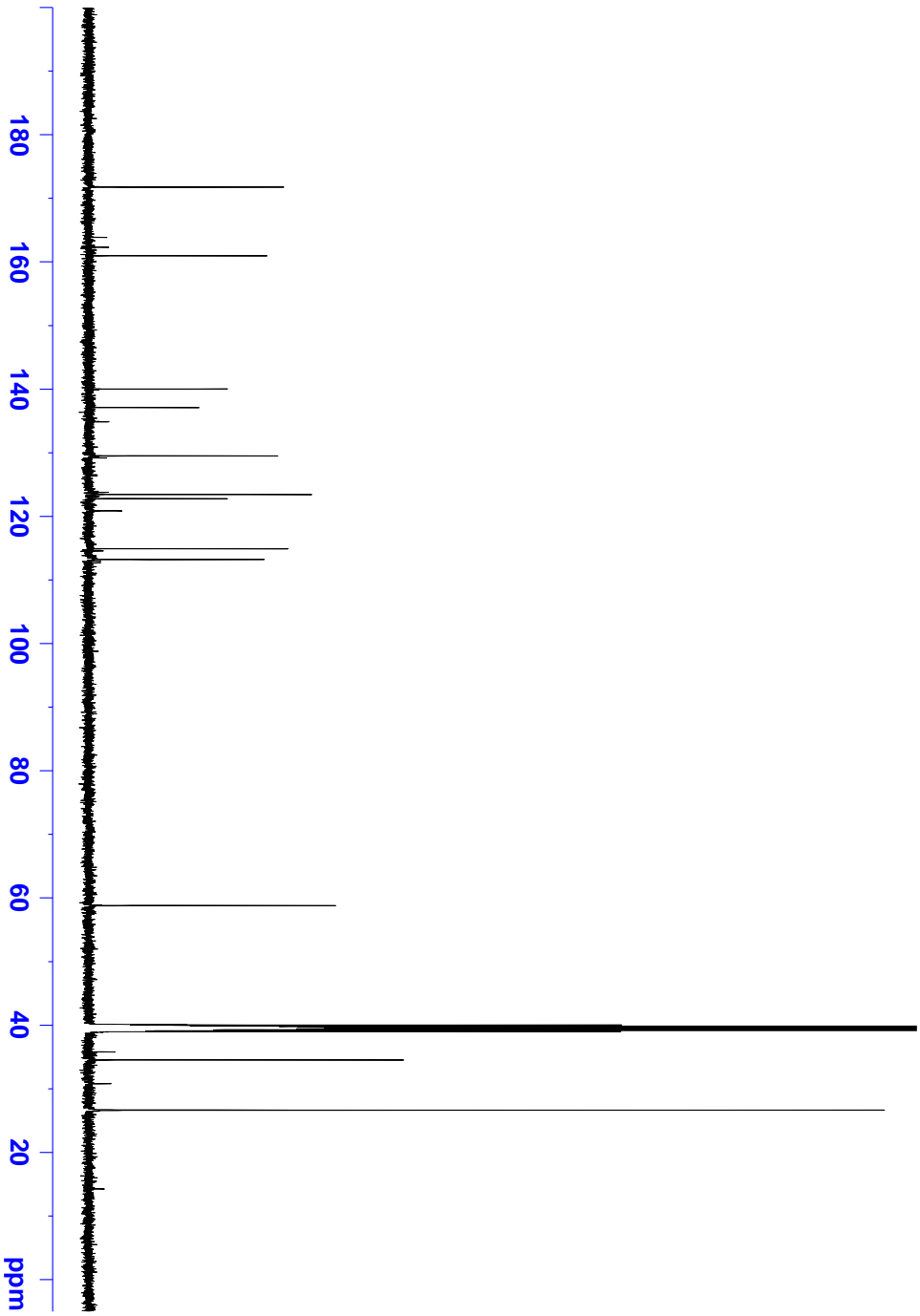
F2 - Processing parameters
 SI 65536
 SF 500.1300037 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



NFL-3177-22
1H



171.74
160.97
140.03
137.11
129.52
123.45
122.78
114.91
113.23
58.80
34.52
26.64



Current Data Parameters
NAME NFL-3177-22
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20220720
Time_ 0.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1843
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 298.1 K
D1 1.0000000 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
CDDPRG12 waltz16
PCPD2 80.00 usec
PLW2 26.0000000 W
PLW12 0.30046001 W
PLW13 0.15113001 W

F2 - Processing parameters

SI 32768
SF 125.7578455 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40