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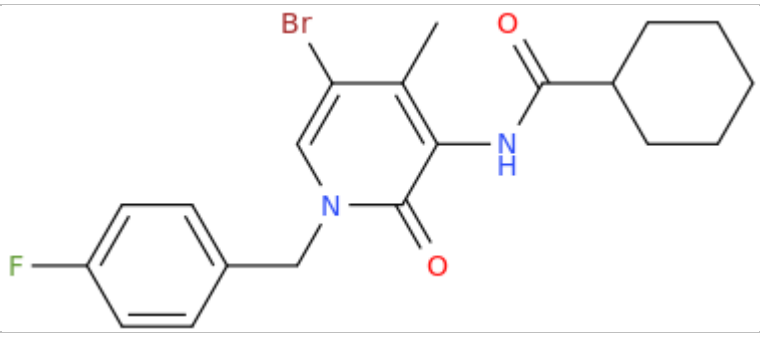
ANALYTICAL REPORT[1]

CH-FUBBMPDORA (C20H22BrFN2O2)

N-{5-bromo-1-[(4-fluorophenyl)methyl]-4-methyl-2-oxo-1,2-dihydropyridin-3-yl}cyclohexanecarboxamide

Remark – other NPS detected: none

Sample ID:	3189-22
Sample description:	powder
Sample type:	test purchase /NFL- purchasing
Date of entry (DD/MM/YYYY) into NFL database:	02/03/2023
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	N-{5-bromo-1-[(4-fluorophenyl)methyl]-4-methyl-2-oxo-1,2-dihydropyridin-3-yl}cyclohexanecarboxamide
Other names	N-(5-bromo-1-(4-fluorobenzyl)-4-methyl-2-oxo-1,2-dihydropyridin-3-yl)cyclohexanecarboxamide
Formula (per base form)	C20H22BrFN2O2
M_w (g/mol)	421,31
Salt form/anions detected	base
StdInChIKey (per base form)	CKYYASUICQFJPH-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	>95% purity of a sample based on 1H NMR spectrum

[1] Approved by: dr. Sonja Klemenc
[2] 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 0C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 µm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 190 ^{0}C at rate 8 ^{0}C/min, then heating up to 293 0C at a rate of 18 ^{0}C/min, hold for 6.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 9.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}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 solid phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 µl and split mode (1:5). Injector temperature 280 ^{0}C. Chromatographic separation as above **(1).** Split MS : IR = 1: 9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.
IR (condesed (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

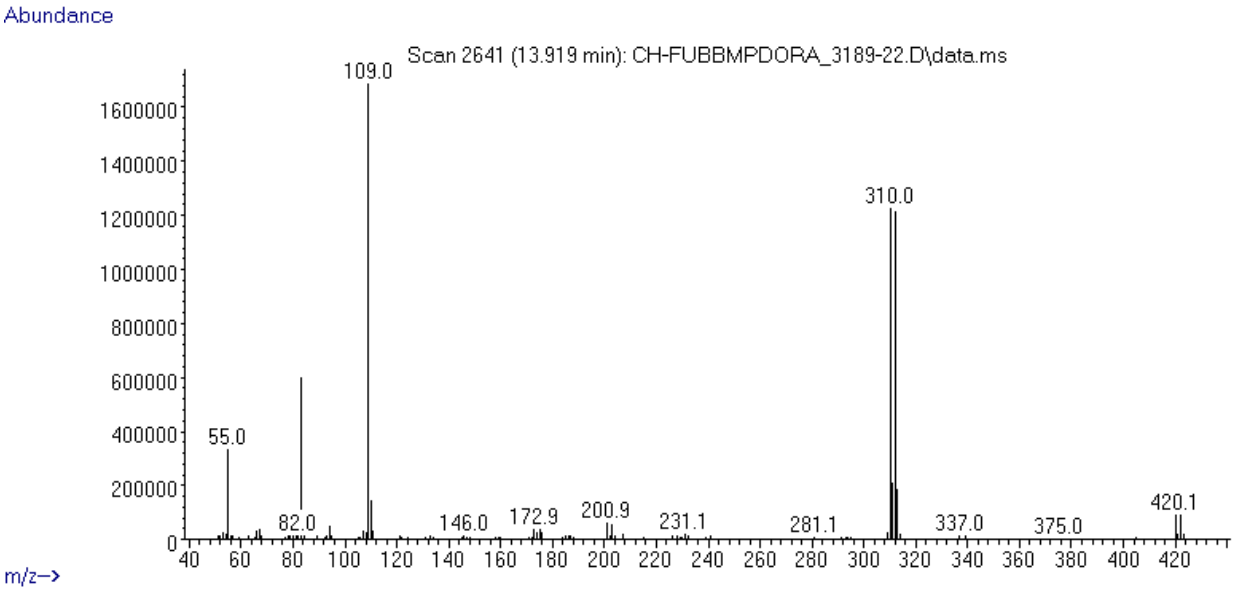
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	partially

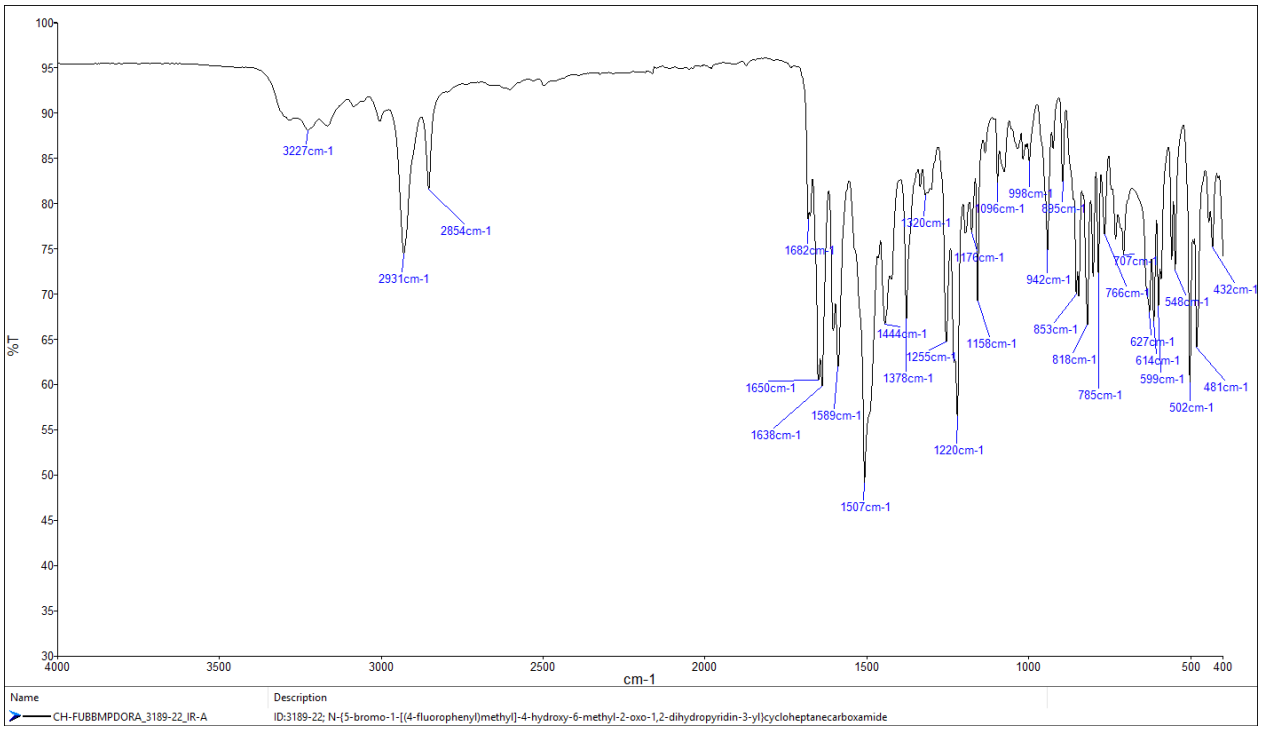
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13,92 BP(1): 109; BP(2): 310,BP(3) :312,
HPLC-TOF	+	Exact mass (theoretical): 420,0849; measured value Δppm:-0,77; formula:C20H22BrFN2O2
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

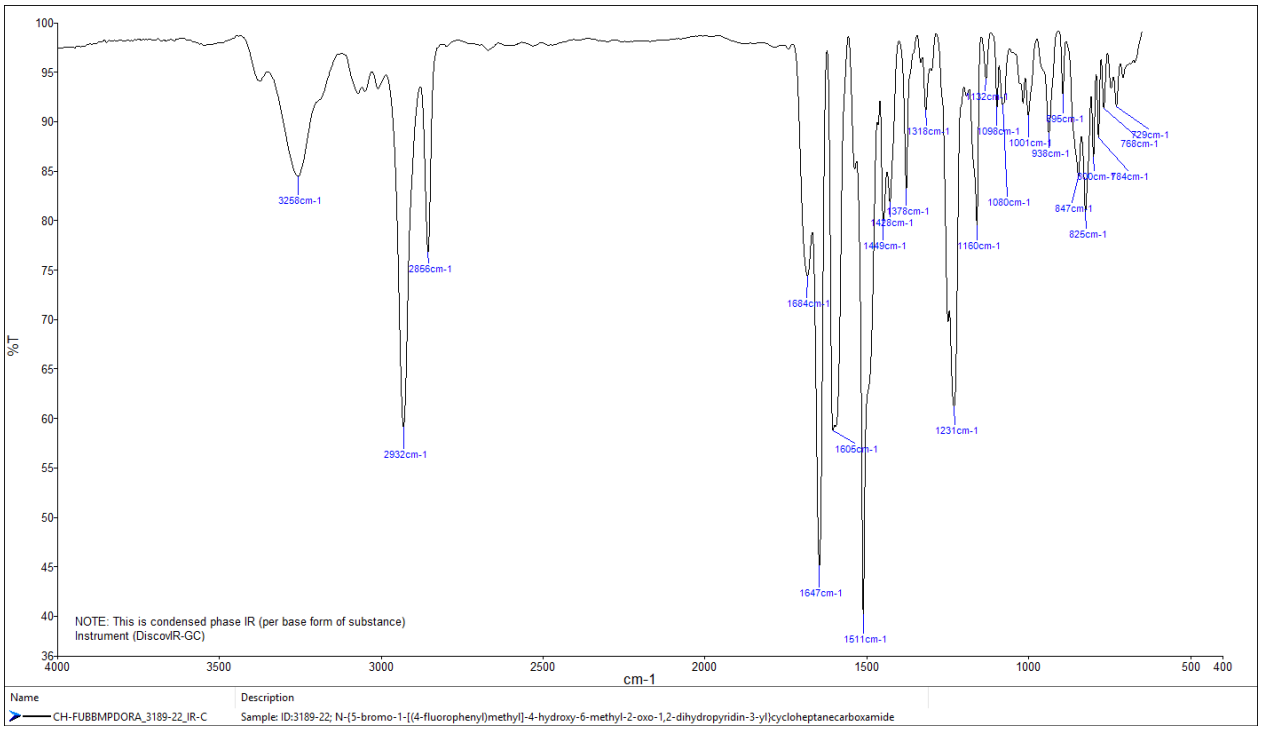
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

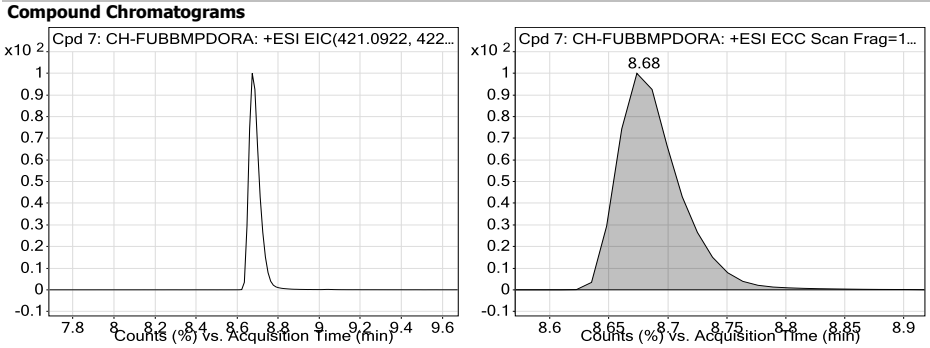
Data File	CH-FUBBMPDORA_3189-22.d	**Sample Name**	ID-3189-22
Sample Type	Sample	**Position**	P1-A5
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-15_01_2020-XDB-C18-ESI+.m	**Acquired Time**	8/5/2022 8:44:51 AM
IRM Calibration Status	Success	**DA Method**	0-NPS in sorodne snovi.m
Comment	extract in MeOH		

Compound Table

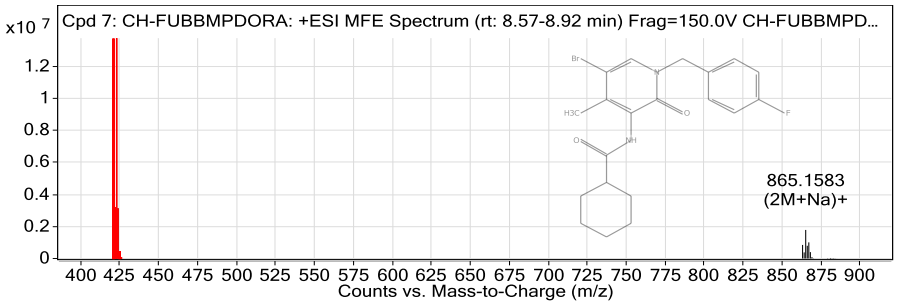
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 7: CH-FUBBMPDORA	CH-FUBBMPDORA	C20 H22 Br F N2 O2	8.68	420.0852

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
CH-FUBBMPDORA	421.0924	8.68	420.0852	8.67	C20 H22 Br F N2 O2	420.0849	-0.77

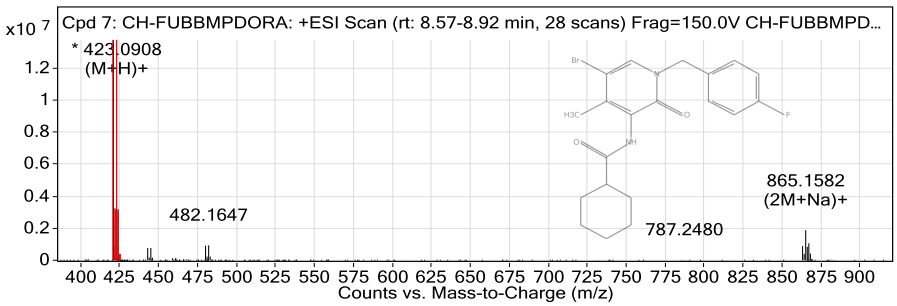
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
421.0924	1	13732656.35	C20 H22 Br F N2 O2	(M+H)+
422.0958	1	3176759.19	C20 H22 Br F N2 O2	(M+H)+
423.0906	1	13766980	C20 H22 Br F N2 O2	(M+H)+
424.0938	1	3138240.15	C20 H22 Br F N2 O2	(M+H)+
863.1603	1	849185.63		(2M+Na)+
864.1631	1	381518.84		(2M+Na)+
865.1583	1	1785534.63		(2M+Na)+
866.1617	1	796665.83		(2M+Na)+
867.1579	1	1008385		(2M+Na)+
868.1597	1	399563.46		(2M+Na)+

--- End Of Report ---

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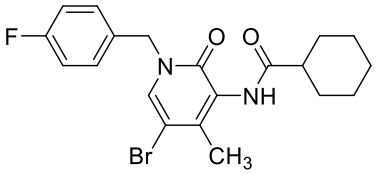
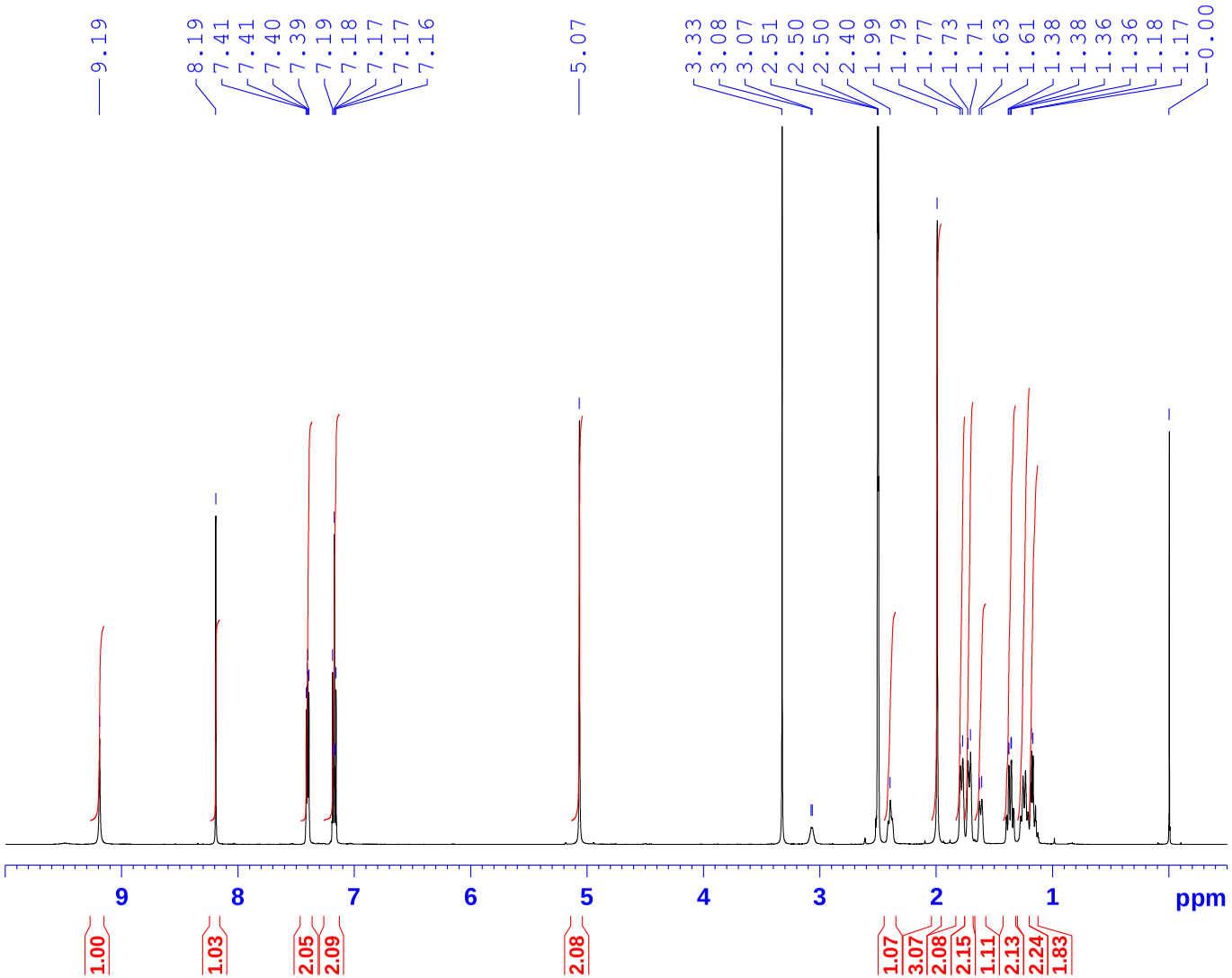
University
of Ljubljana

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:	**3189-22**
Received date:	September 29, 2022
Our notebook code:	NFL-3189-22
NMR sample preparation:	16.0 mg dissolved in 0.7 mL DMSO-d_6
NMR experiments:	^{1}H, ^{13}C, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC, ^{1}H–^{15}N *gs*-HMBC, ^{1}H–^{1}H NOESY.
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{20}H_{22}BrFN_2O_2$ Exact Mass: 420,0849 Molecular Weight: 421,3104
Chemical name:	N-(5-bromo-1-(4-fluorobenzyl)-4-methyl-2-oxo-1,2-dihydropyridin-3-yl)cyclohexanecarboxamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra. - >95% purity of a sample based on ^{1}H NMR spectrum. - The structure is confirmed by X-Ray structural analysis of a monocrystal.
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 13, 2022

NFL-3189-22
1H



Current Data Parameters

NAME	NFL-3189-22
EXPNO	1
PROCNO	1

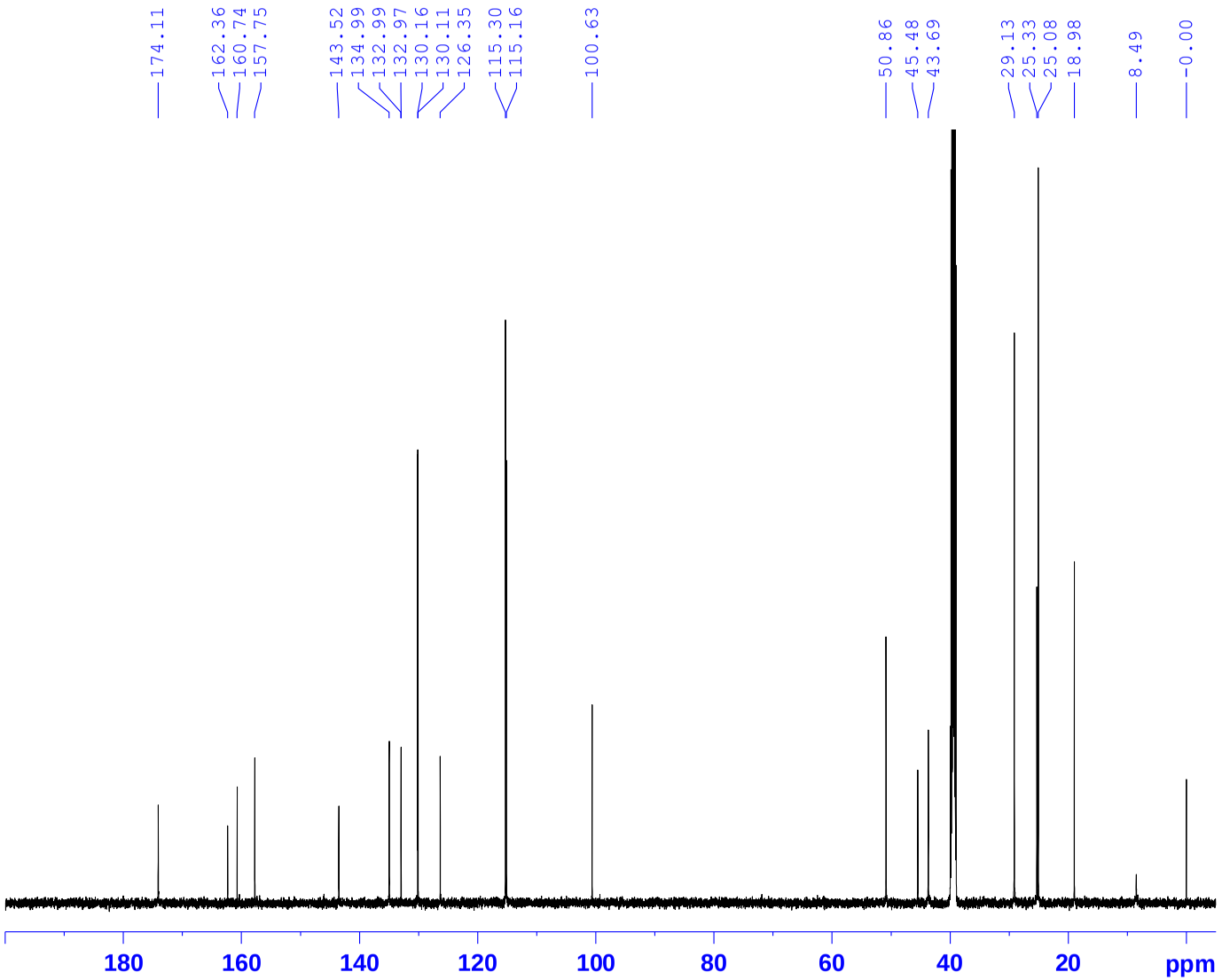
F2 - Acquisition Parameters

Date_	20220930
Time	17.38 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	93.8889
DW	42.000 usec
DE	8.79 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
SFO1	600.1337058 MHz
NUC1	1H
P0	3.33 usec
P1	10.00 usec
PLW1	16.51399994 W

F2 - Processing parameters

SI	65536
SF	600.1300031 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

NFL-3189-22
13C



Current Data Parameters

NAME	NFL-3189-22
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20220930
Time	19.21 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	298.1 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
CPDPRG[2	waltz65
PCPD2	70.00 usec
PLW2	16.51399994 W
PLW12	0.33702001 W
PLW13	0.16952001 W

F2 - Processing parameters

SI	32768
SF	150.9028950 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40