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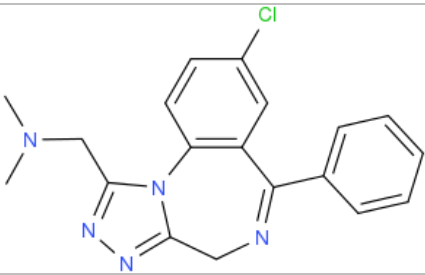
ANALYTICAL REPORT¹

ADINAZOLAM (C₁₉H₁₈ClN₅)

1-(8-chloro-6-phenyl-4H-[1,2,4]triazolo[4,3-a][1,4]benzodiazepin-1-yl)-N,N-dimethylmethanamine

Remark – other NPS detected: **none**

Sample ID:	1277-15
Sample description:	powder - yellowish (gently)
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	9/18/2015
Date of entry (M/D/Y) into NFL database:	10/4/2015
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	1-(8-chloro-6-phenyl-4H-[1,2,4]triazolo[4,3-a][1,4]benzodiazepin-1-yl)-N,N-dimethylmethanamine
Other names	Adinazolamum; Deracyn
Formula (per base form)	C ₁₉ H ₁₈ ClN ₅
M _w (g/mol)	351.83
Salt form	HCl
StdInChIKey	GJSLOMWRLALDCT-UHFFFAOYSA-N
Compound Class	Benzodiazepines
Other NPS detected	none
Add.info (purity..)	pure byGC-MS, HPLC-TOF; small amounts of organic impurities by NMR

¹ This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

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 (RT=9.53 min). Injection volume 1 ml 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 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by 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 2.8 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 (40) to 550 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 ml 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 (40) to 550 amu.

IR (condensed 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 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

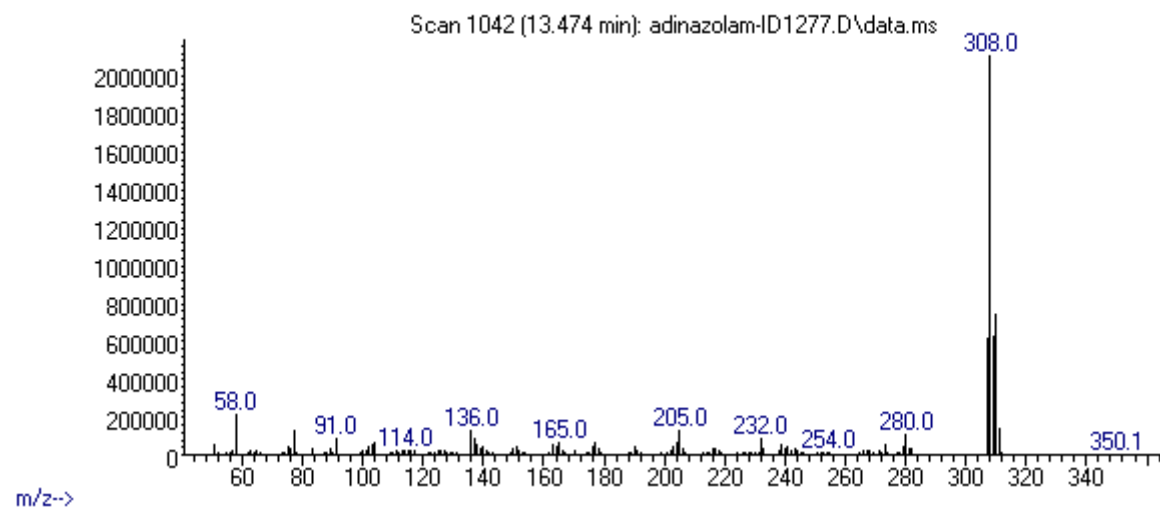
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 13.47 BP(1): 308; BP(2): 310,BP(3) :309,
HPLC-TOF	+	Exact mass (theoretical): 351.1251; measured value Δppm:-0.59; formula:C19H18ClN5
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	after chromatographic separation
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

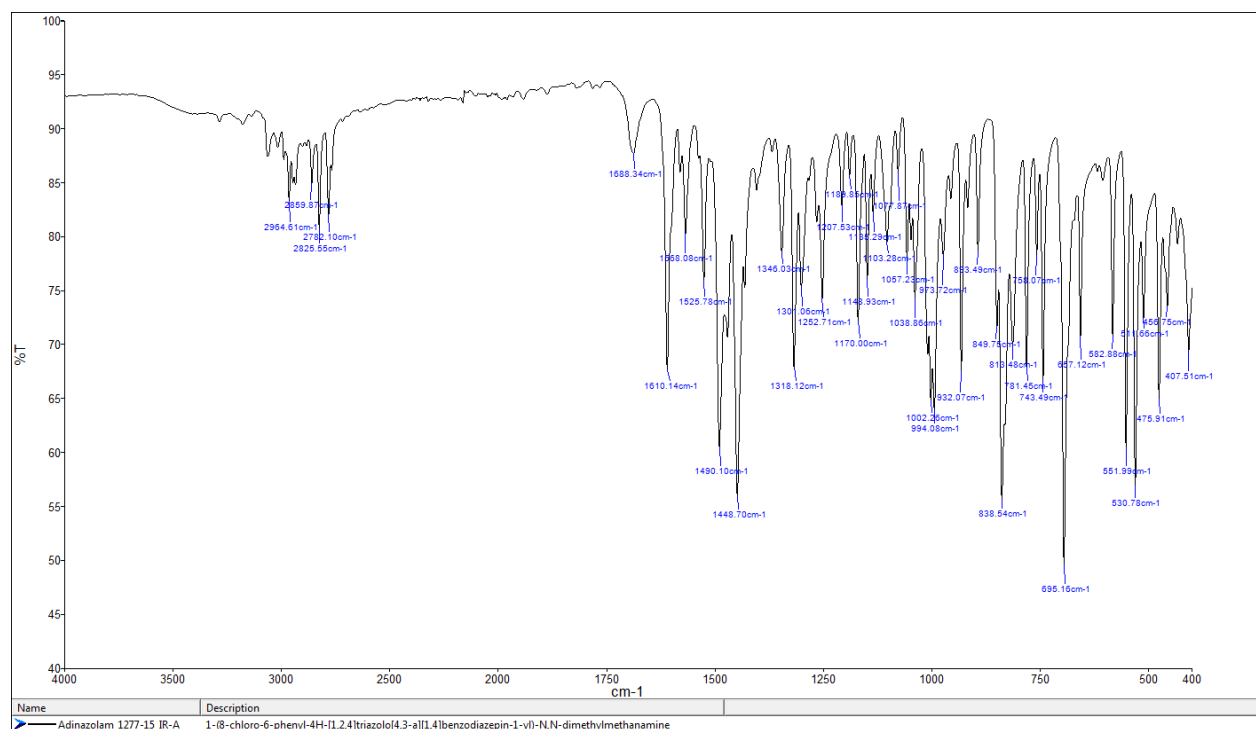
ANALYTICAL RESULTS

MS (EI)

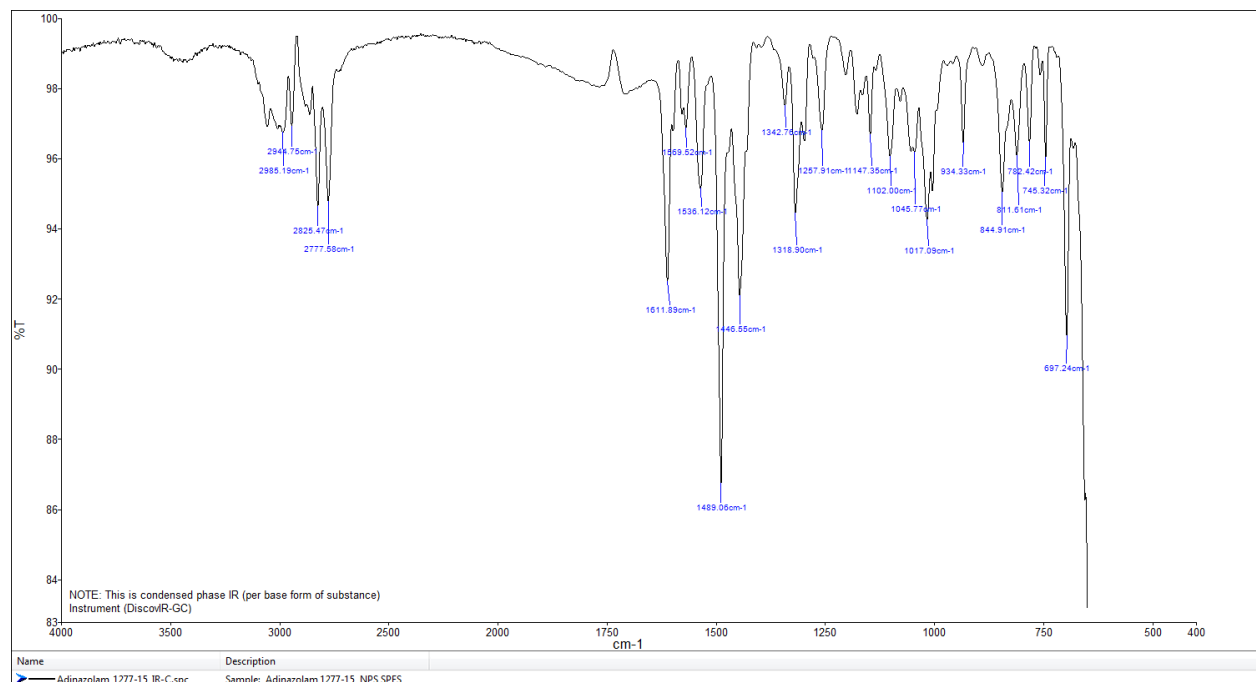
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

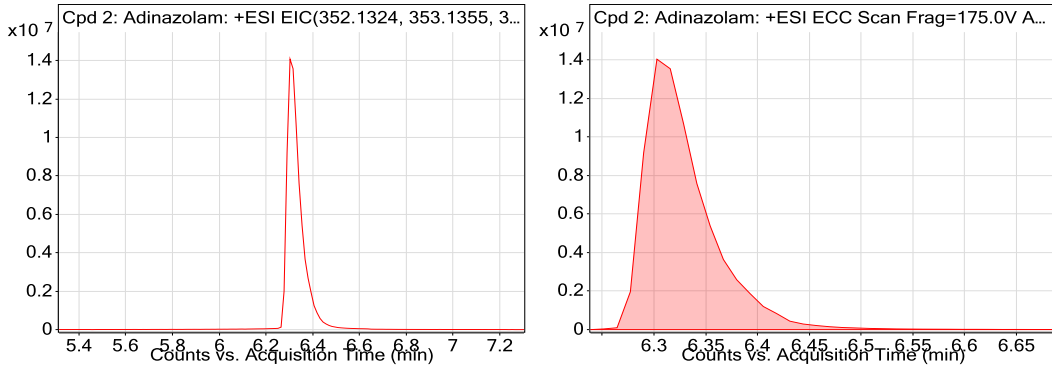
Data File	ADINAZOLAM_1277-15_TOF.d	Sample Name	ADINAZOLAM
Sample Type	Sample	Position	P1-E2
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	Acquired Time	9/21/2015 9:12:23 AM
IRM Calibration Status	Success	DA Method	Droge_Default.m
Comment	extract in MeOH		

Compound Table

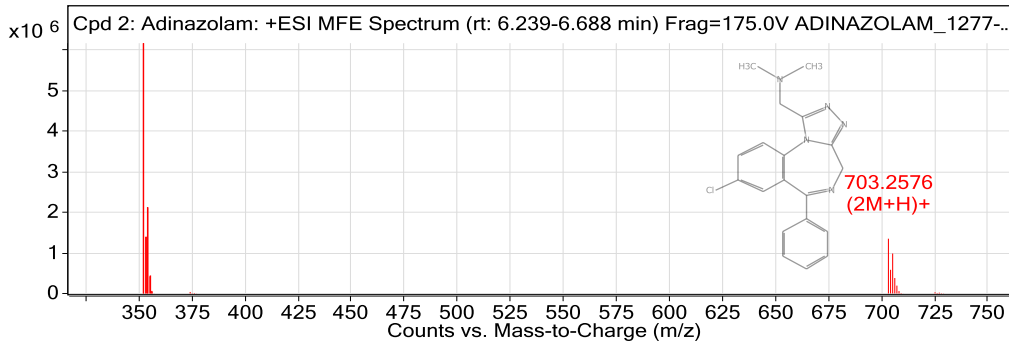
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 2: Adinazolam	Adinazolam	6.314	351.1253

Name	Obs. m/z	Obs. RT	Obs. Mass	DB Formula	DB Mass	DB Mass Error (ppm)
Adinazolam	352.1324	6.314	351.1253	C19 H18 Cl N5	351.1251	-0.59

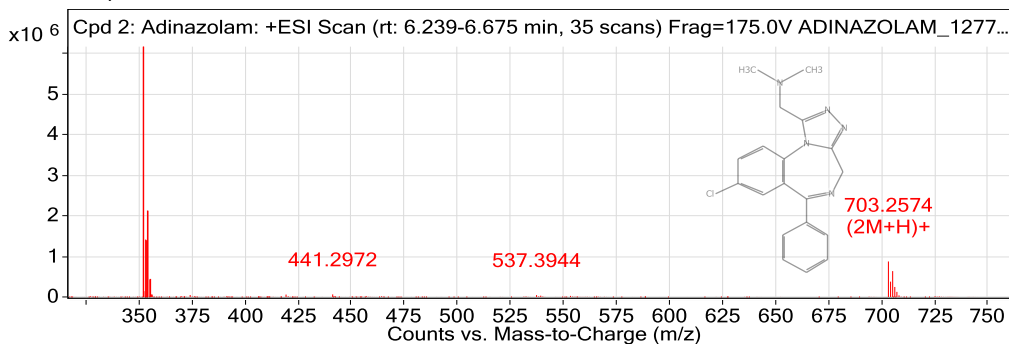
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
352.1324	1	6170093.5	C19 H18 Cl N5	(M+H)+
353.1358	1	1396431.77	C19 H18 Cl N5	(M+H)+
354.1302	1	2113472.59	C19 H18 Cl N5	(M+H)+
355.133	1	424584.94	C19 H18 Cl N5	(M+H)+
703.2576	1	1350420.5		(2M+H)+
704.2608	1	583857.52		(2M+H)+
705.2561	1	985032.02		(2M+H)+

TOF REPORT

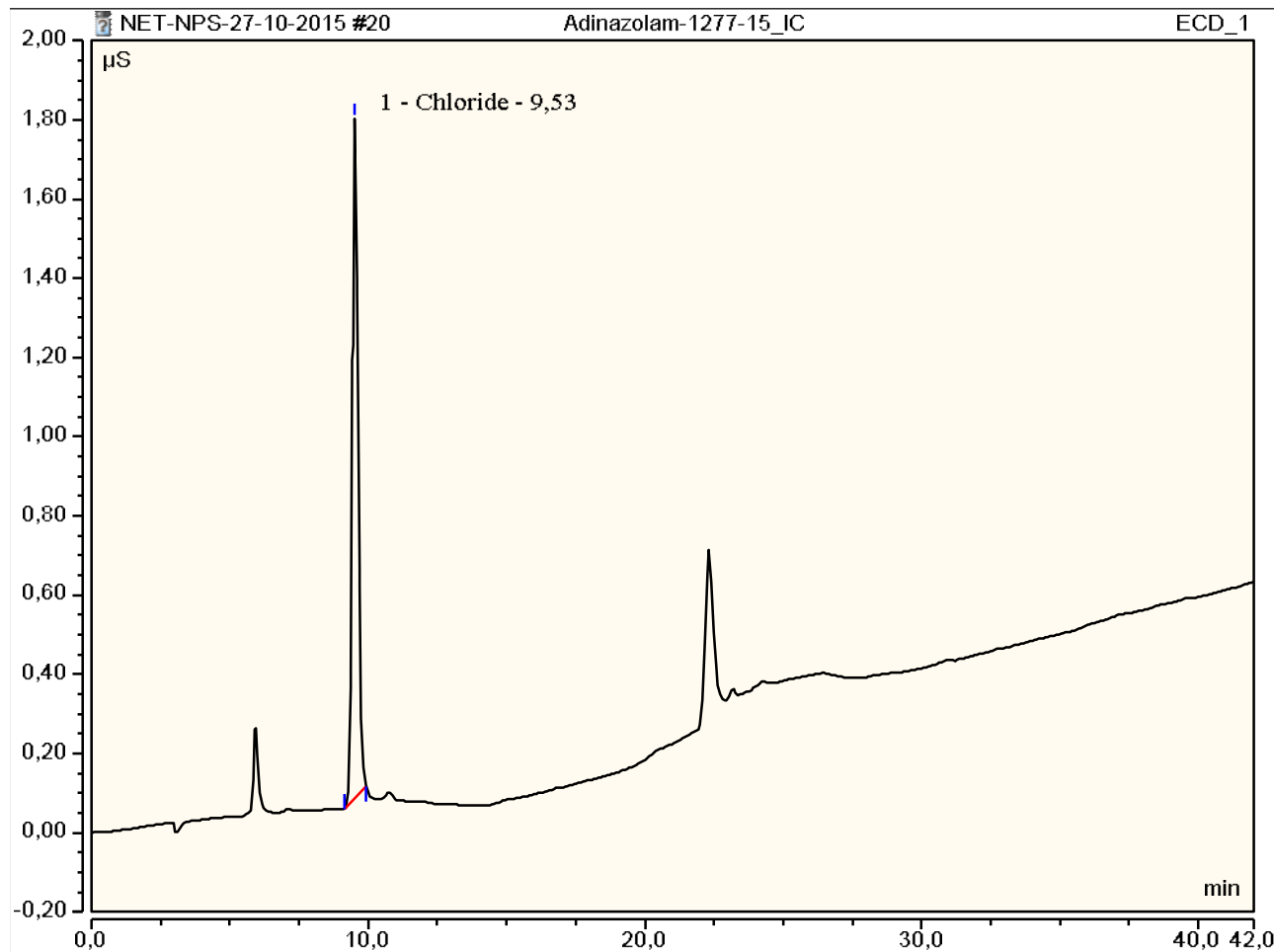
706.2582	1	380864.52	(2M+H)+
707.2551	1	193831.27	(2M+H)+
708.2555	1	59386.62	(2M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	Adinazolam-1277-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	28-okt-2015 / 03:08	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	9,53	Chloride	BMB	0,42	1,71	n.a.
		TOTAL:		0,42	1,71	0,00

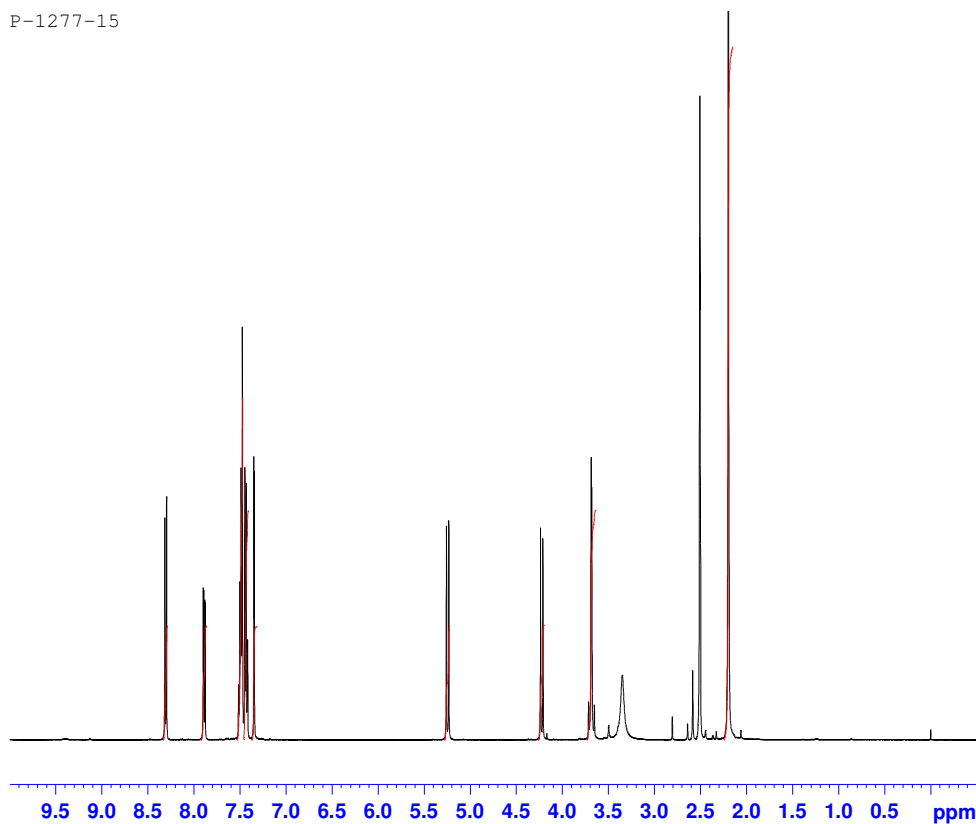




REPORT

Sample ID:	1277-15
Our notebook code:	P-1277-15
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C .
Proposed structure:	
Chemical name:	1-(8-chloro-6-phenyl-4H-benzo[f][1,2,4]triazolo[4,3-a][1,4]diazepin-1-yl)-N,N-dimethylmethanamine
Comments:	- Structure elucidation based on 1D NMR spectra - Compound is not pure by NMR, contains a minor amount of organic impurities.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 9, 2015

P-1277-15



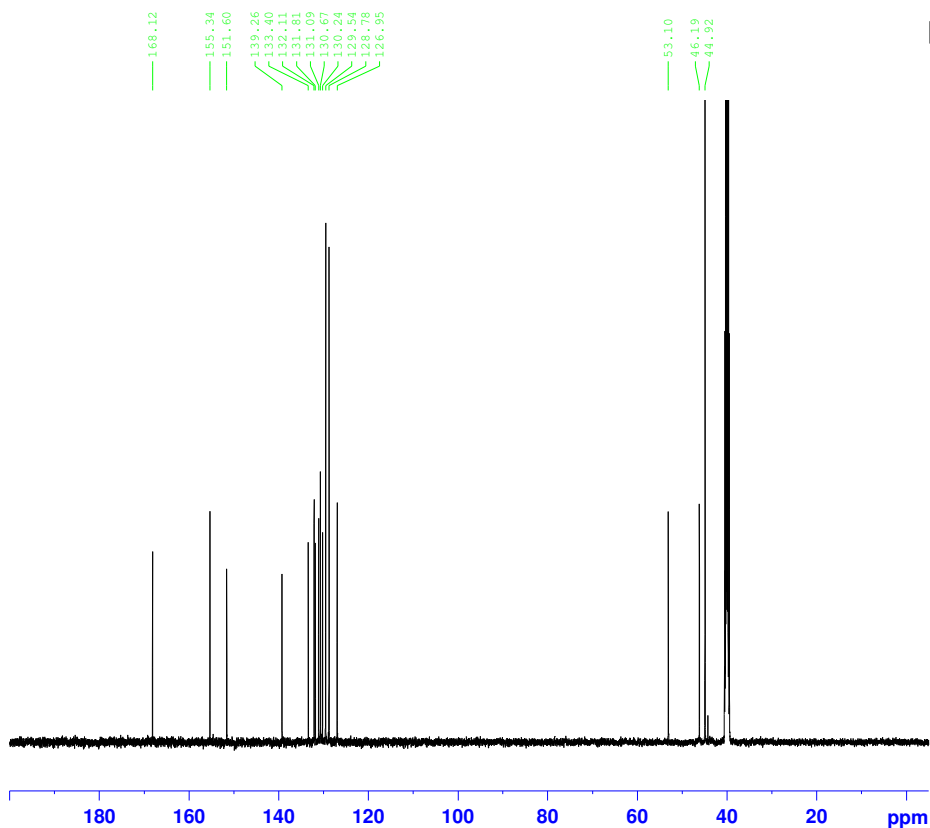
Current Data Parameters
NAME P-1277-15
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151129
Time 9.59
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 3.2768500 sec
RG 80.6
DW 50.000 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1330885 MHz
NUC1 1H
P1 8.90 usec
PLW1 26.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1277-15



Current Data Parameters
NAME P-1277-15
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151129
Time 11.51
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 9.00 usec
PLW1 122.00000000 W

===== CHANNEL f2 =====
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.32179001 W
PLW13 0.16186000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40