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EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

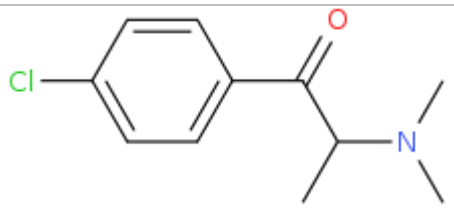
ANALYTICAL REPORT

4-CDC (C11H14ClNO)

1-(4-chlorophenyl)-2-(dimethylamino)propan-1-one

Remark – other NPS detected:

Sample ID:	1908-18 (original sample labele was CTA-5667)
Sample description:	powder
Sample type:	seized /National Romanian Police (sample was provided to NFL for structure elucidation)
Date of sample receipt (M/D/Y):	1/29/2018
Date of entry (M/D/Y) into NFL database:	2/6/2018
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-(4-chlorophenyl)-2-(dimethylamino)propan-1-one
Other names	4-chloro-N,N-dimethylcathinone; 4-CDC; 4-CDMD; 4-chloro-N,N-DMC; 4-chloro-N,N-DMC
Formula (per base form)	C11H14ClNO
M_w (g/mol)	211,69
Salt form/anions detected	HCl
StdInChIKey (per base form)	XMBDJBGDUKIOFM-UHFFFAOYSA-N
Other NPS detected	
Additional info (purity..)	pure by GC-MS, HPLC-TOF, NMR

[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 ml 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 7.1 min, then heating at 50 ^{0}C/min up to 325 ^{0}C and finally 6.1 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 condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 ml 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 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

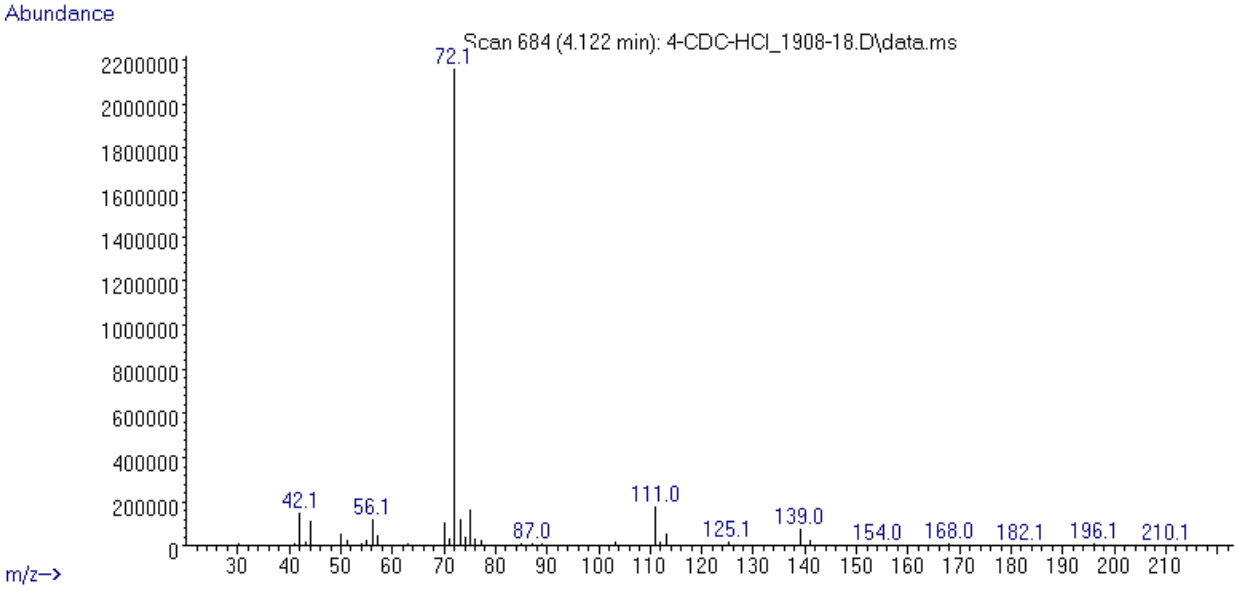
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	soluble

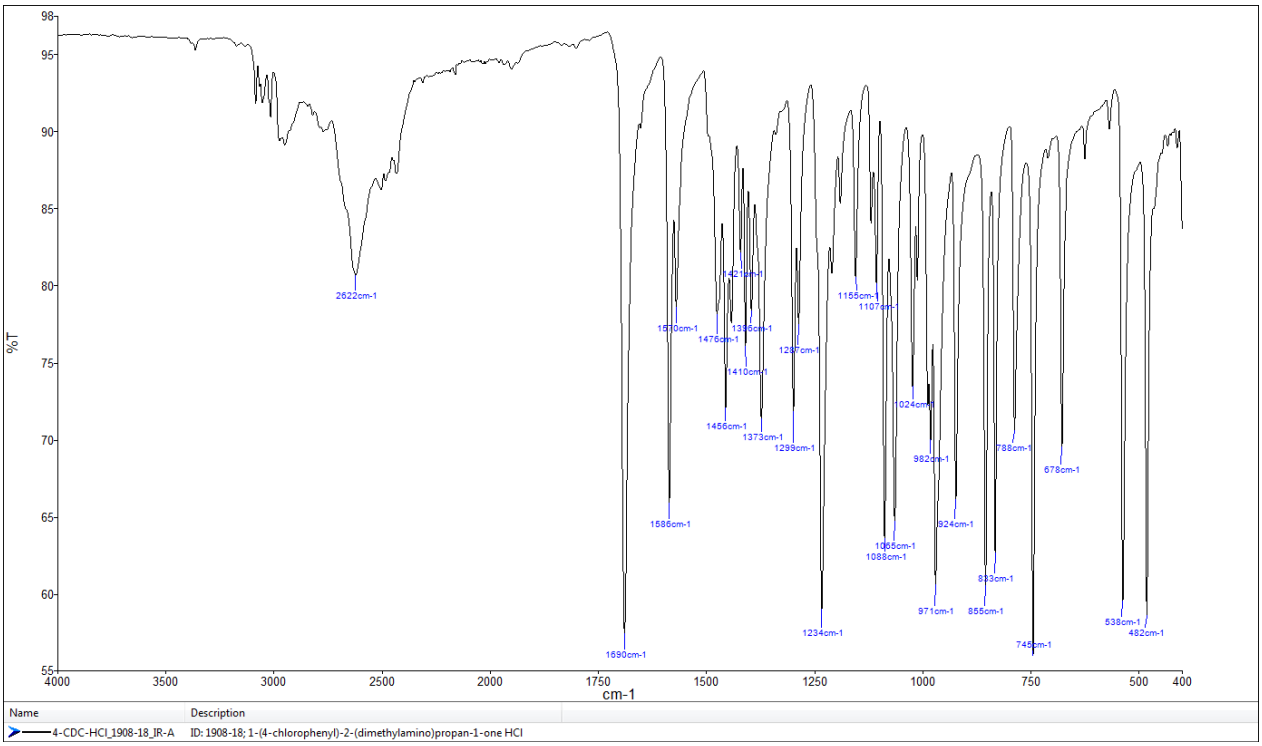
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 4,12 BP(1): 72; BP(2): 111,BP(3) :75,
HPLC-TOF	+	Exact mass (theoretical): 211,0764; measured value Δppm:-2,03; formula:C11H14ClNO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

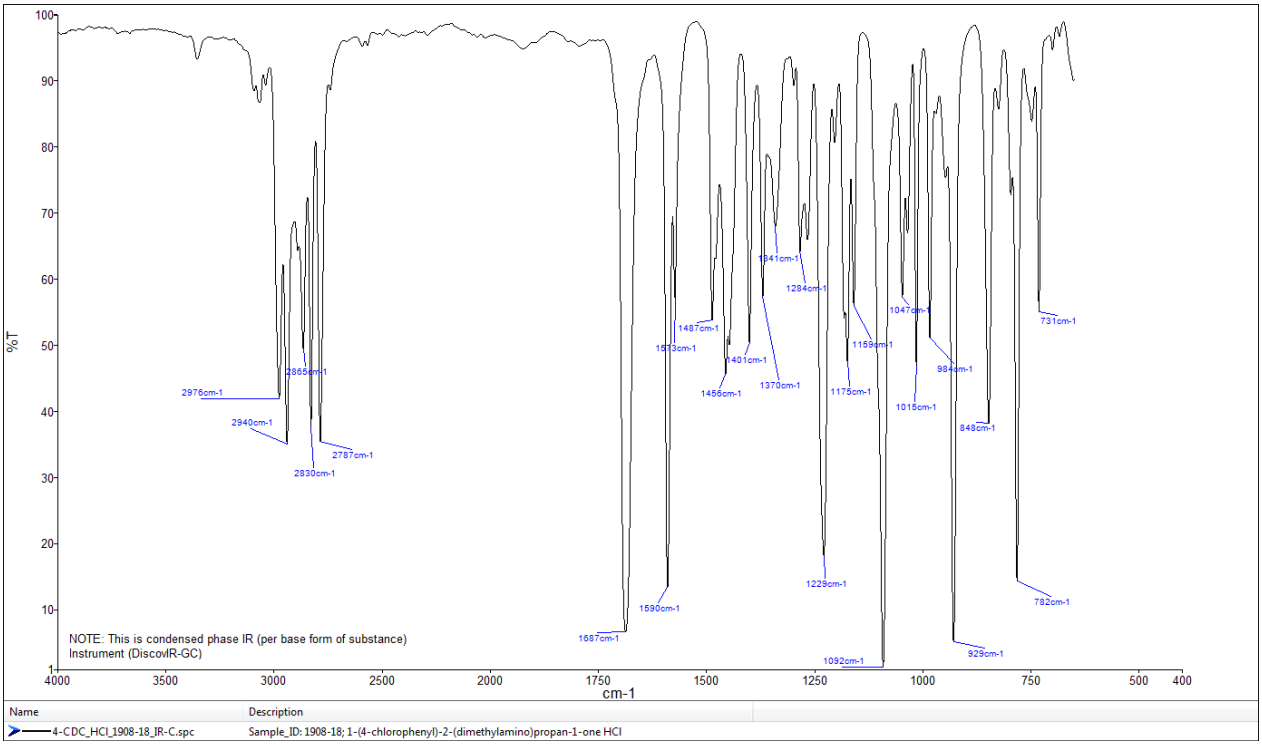
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

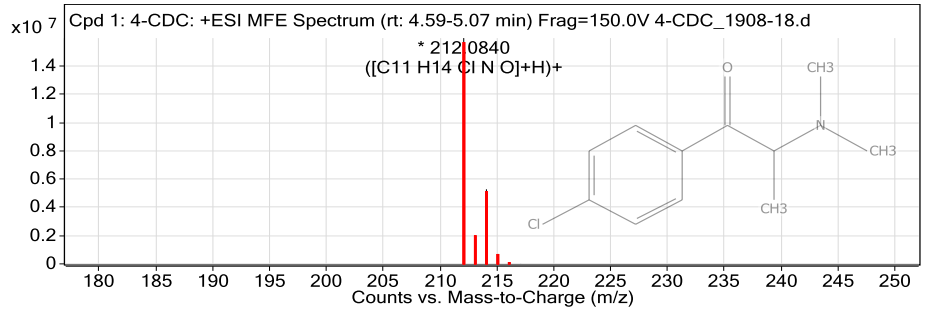
Data File	4-CDC_1908-18.d	**Sample Name**	Romania, unknown, CTA-5667
Sample Type	Sample	**Position**	P2-C3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-04_12_2017-XDB-C18-ESI+.m	**Acquired Time**	1/29/2018 2:06:30 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: 4-CDC	4-CDC	C11 H14 Cl N O	4.67	211.0768

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
4-CDC	212.084	4.67	211.0768	4.67	C11 H14 Cl N O	211.0764	-2.03

MFE MS Zoomed Spectrum



MS Spectrum Peak List

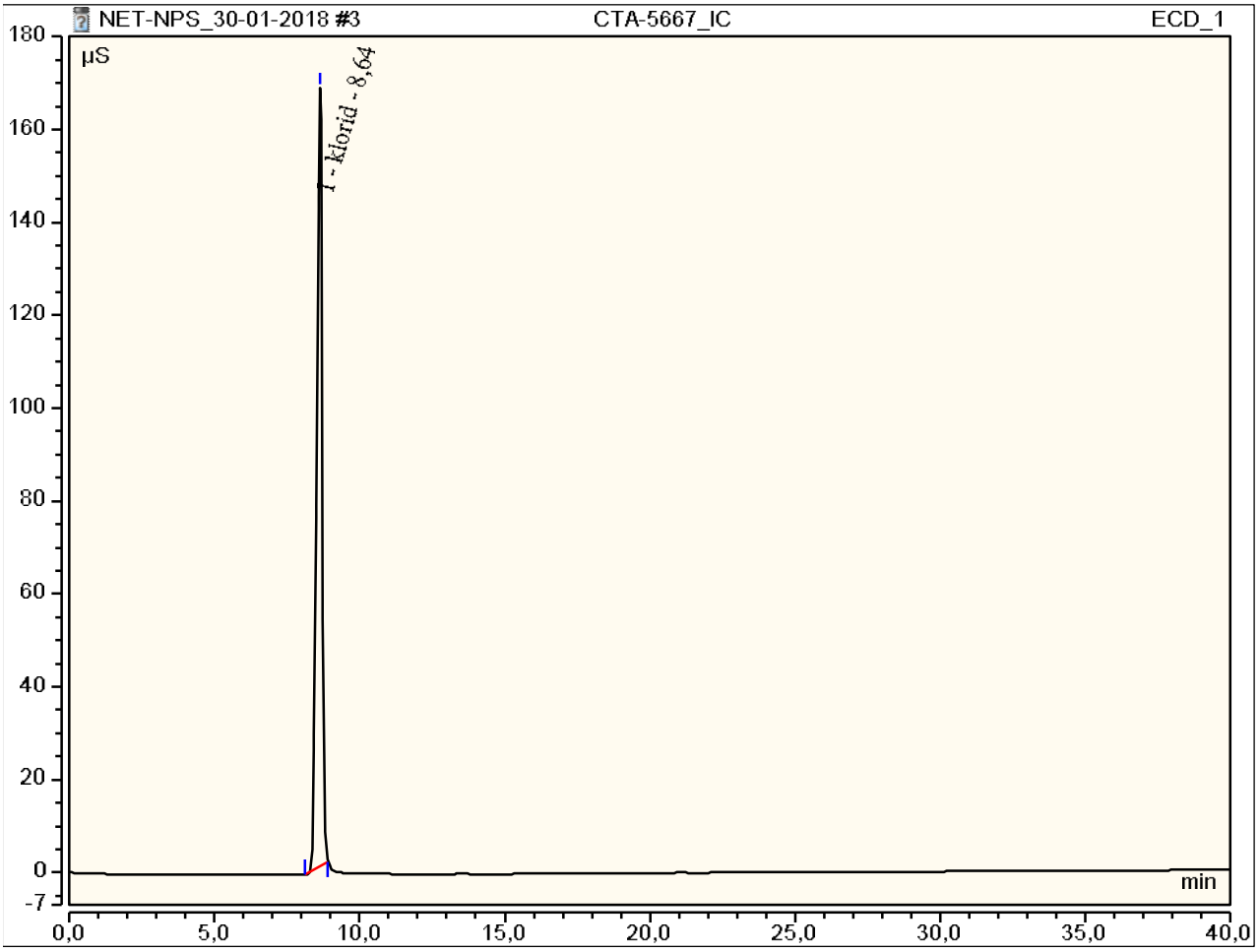
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
212.084	1	15998369	C11 H14 Cl N O	(M+H)+
213.0875	1	1862803.83	C11 H14 Cl N O	(M+H)+
214.0815	1	5283859.43	C11 H14 Cl N O	(M+H)+
215.085	1	256714.51	C11 H14 Cl N O	(M+H)+
216.0874	1	36885.63	C11 H14 Cl N O	(M+H)+
217.0945	1	4018.99	C11 H14 Cl N O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**CTA-5667_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**30-jan-2018 / 11:21**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	8,64	**klorid**	BMB	36,58	167,68	n.a.
		TOTAL:		36,58	167,68	0,00



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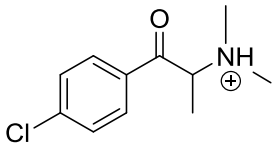
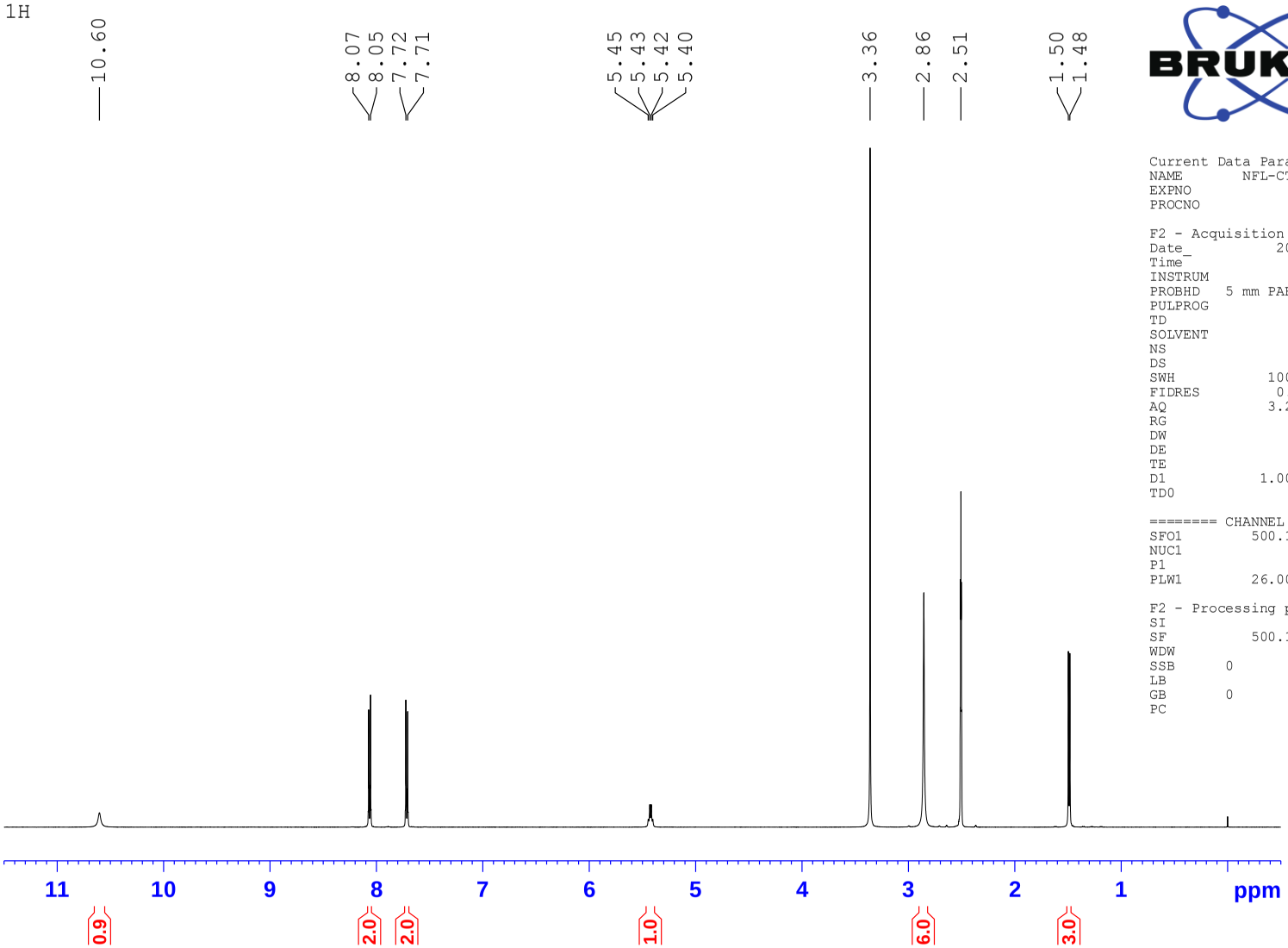
R E P O R T

Contract No.	C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE)
Sample ID:	**CTA-5667**
Received date:	January 31, 2018
Our notebook code:	NFL-CTA-5667
NMR sample preparation:	22.2 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
Proposed structure with formula, exact mass, molecular weight:	 Chemical Formula: $C_{11}H_{15}ClNO^+$ Exact Mass: 212,0837 Molecular Weight: 212,6965
Chemical name:	*N*-protonated 1-(4-chlorophenyl)-2-(dimethylamino)propan-1-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra and HRMS. - Compound is pure by NMR.
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:	February 2, 2018

NFL-CTA-5667
1H



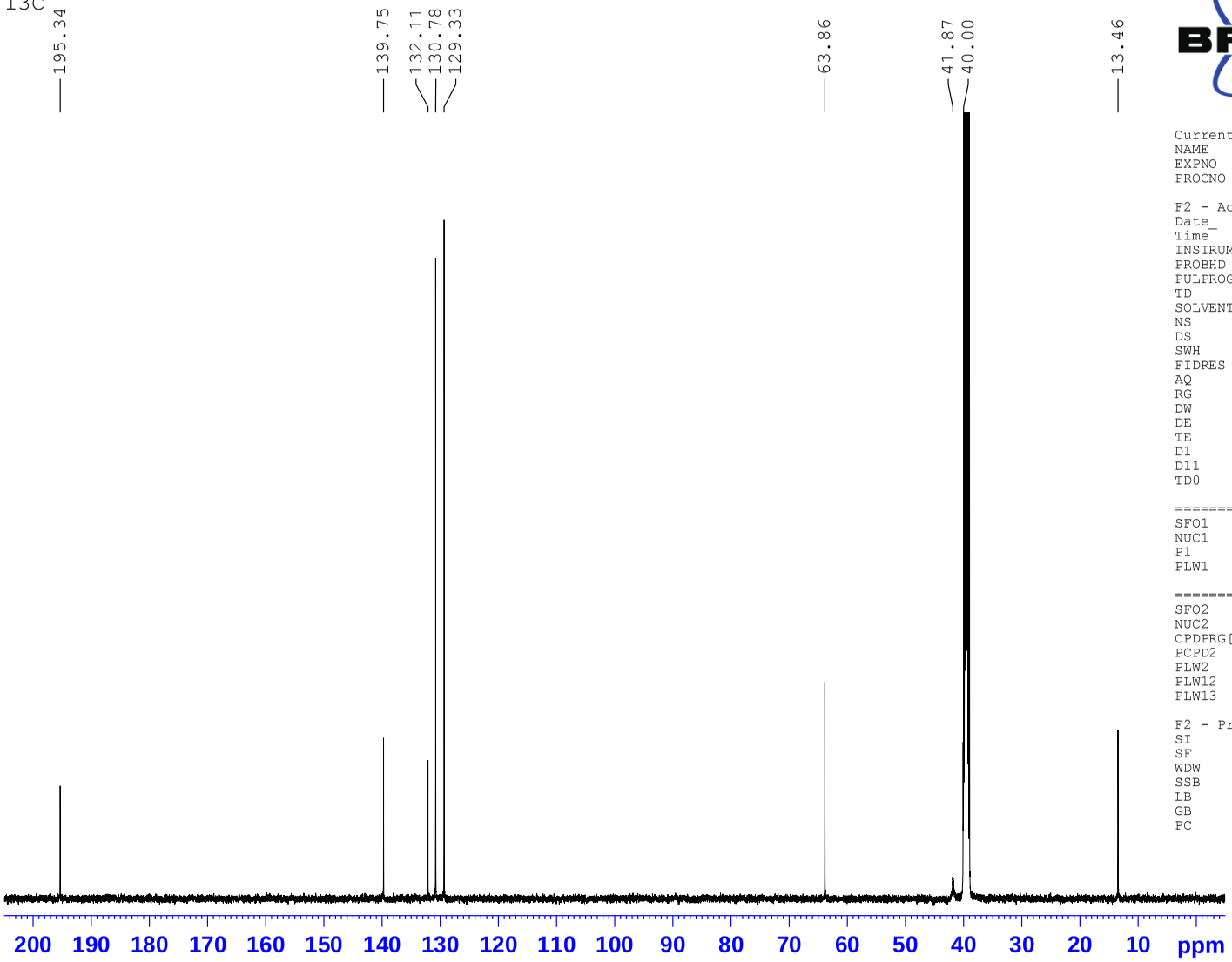

```
Current Data Parameters
NAME        NFL-CTA-5667
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180201
Time              19.27
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                  114
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                8.70 usec
PLW1         26.00000000 W

F2 - Processing parameters
SI                65536
SF           500.1300013 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

NFL-CTA-5667
13C



BRUKER

Current Data Parameters
NAME NFL-CTA-5667
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180202
Time 0.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 5120
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 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 8.70 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 0.30046001 W
PLW13 0.15113001 W

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