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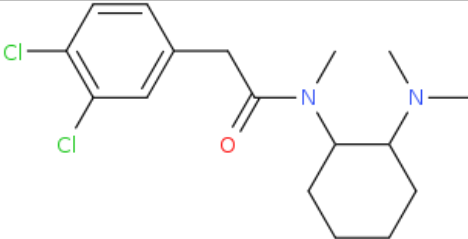
ANALYTICAL REPORT

METHENE-U-47700 (C₁₇H₂₄Cl₂N₂O)

2-(3,4-dichlorophenyl)-N-(2-(dimethylamino)cyclohexyl)-N-methylacetamide

Remark – other NPS detected: **none**

Sample ID:	1729-16
Sample description:	powder - white-off
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	10/17/2016
Date of entry (M/D/Y) into NFL database:	1/13/2017
Report ¹ 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-(3,4-dichlorophenyl)-N-(2-(dimethylamino)cyclohexyl)-N-methylacetamide
Other names	METHENE-U-47700
Formula (per base form)	C ₁₇ H ₂₄ Cl ₂ N ₂ O
M _w (g/mol)	343,29
Salt form/anions detected	HCl
StdInChIKey (for base form)	ISJUYFBACBKBV-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	Sample is not pure by GC-MS, TOF, and 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 (9.258 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 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 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 (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 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

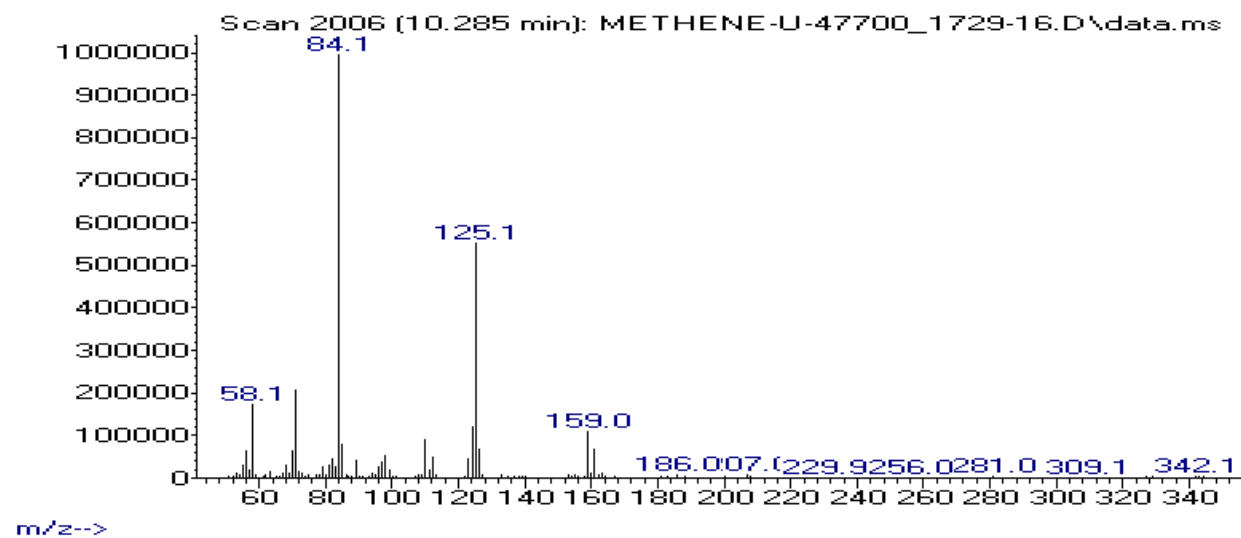
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 10,29 BP(1): 84; BP(2): 125,BP(3) :71,
HPLC-TOF	+	Exact mass (theoretical): 342,1271; measured value Δppm:-1,51; formula:C17H24Cl2N2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

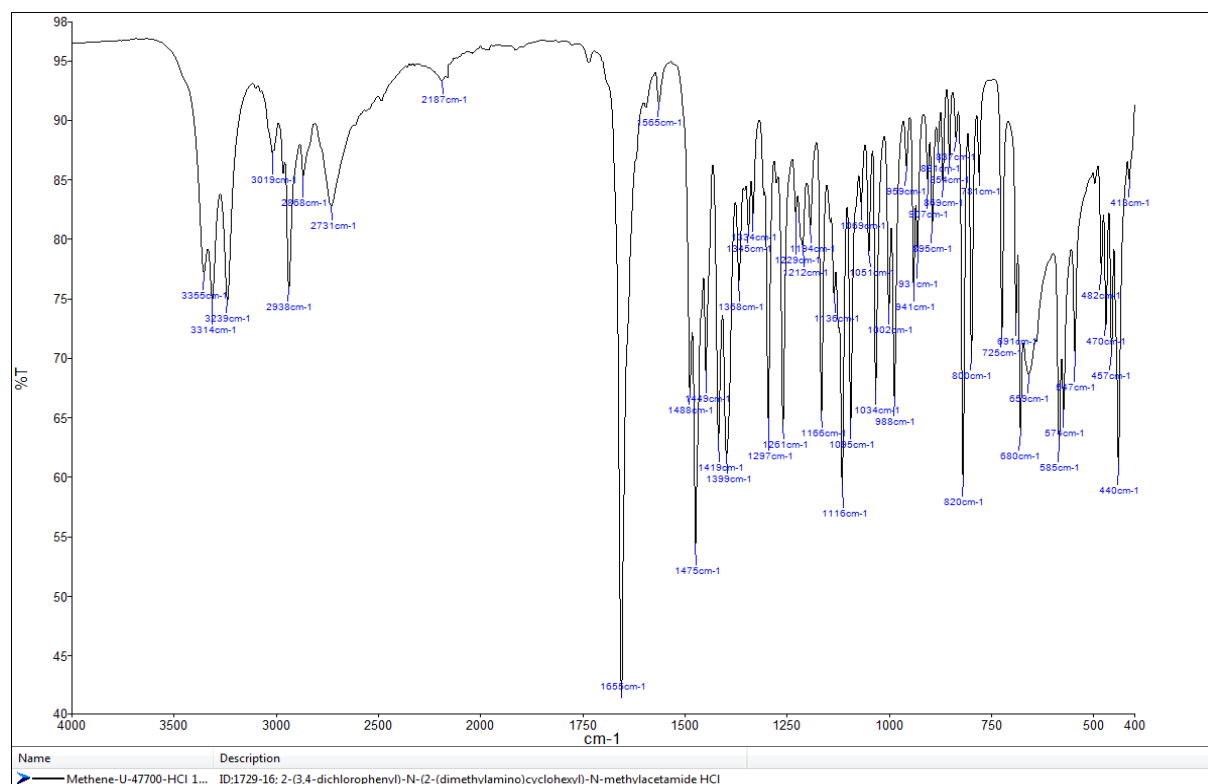
ANALYTICAL RESULTS

MS (EI)

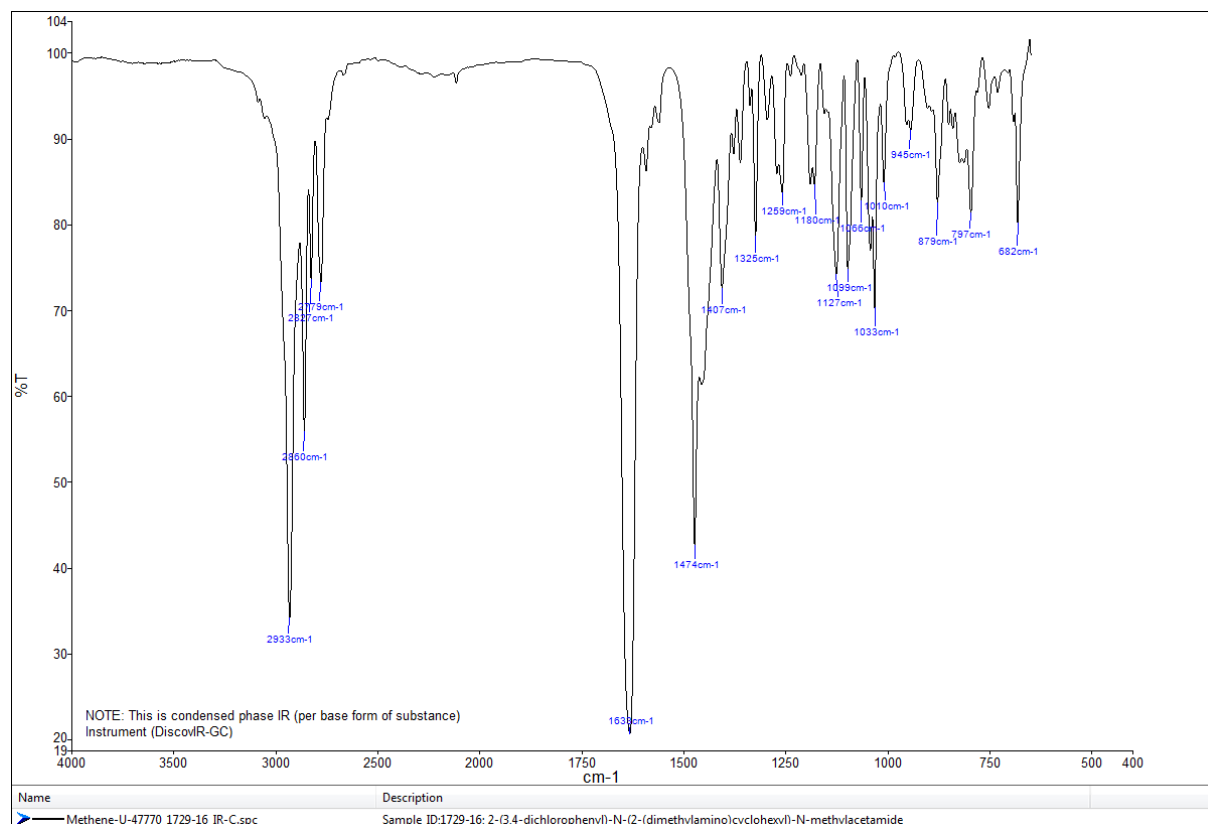
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

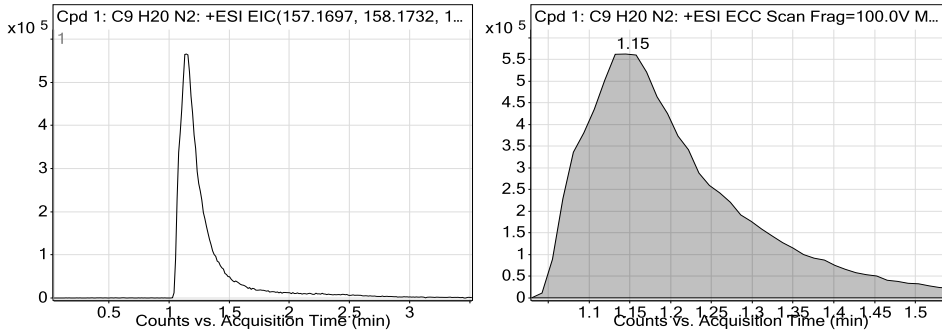
Data File	Methene-U-47700_1729-16.d	Sample Name	ID_1729-16
Sample Type	Sample	Position	P1-E1
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-10_10_2016-XDB-C18-ESI-poz-soft.m	Acquired Time	10/18/2016 12:05:38 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

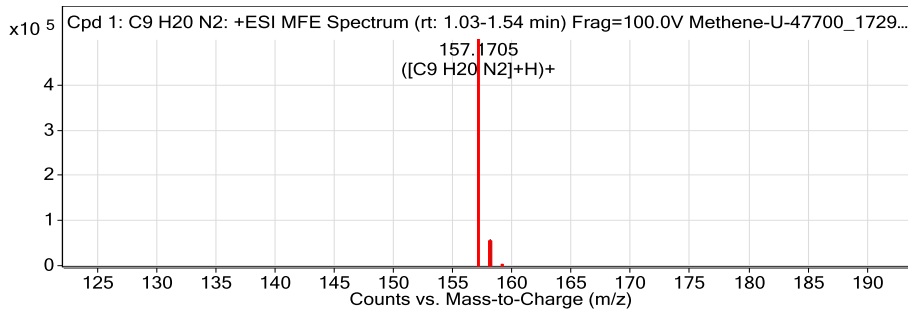
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: C9 H20 N2		C9 H20 N2	1.15	156.1632
Cpd 2: Methene-U-47700	Methene-U-47700	C17 H24 Cl2 N2 O	6.95	342.1271

Obs. m/z	Obs. RT	Obs. Mass
157.1705	1.15	156.1632

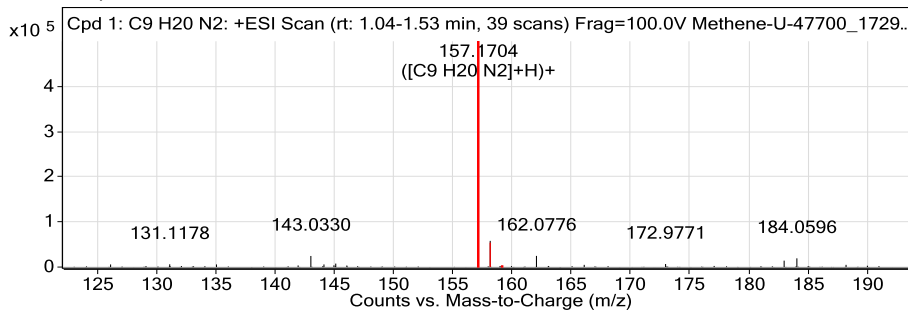
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



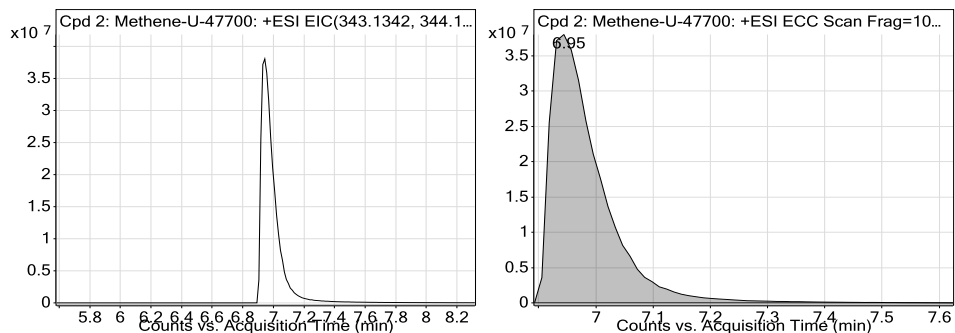
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
157.1705	1	499979.16	C9 H20 N2	(M+H)+
158.1732	1	58725.75	C9 H20 N2	(M+H)+
159.1762	1	2935.53	C9 H20 N2	(M+H)+

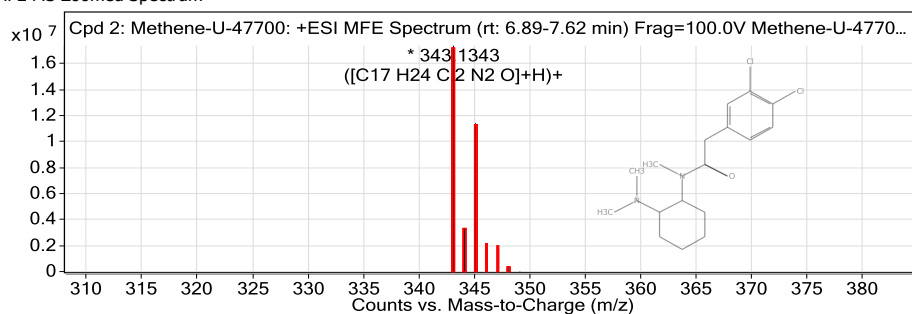
Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Methene-U-47700	343.1343	6.95	342.1271	6.95	C17 H24 Cl2 N2 O	342.1266	-1.51

Compound Chromatograms

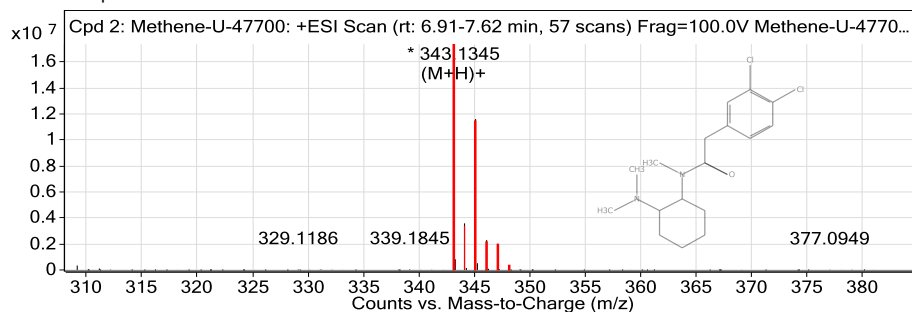
TOF REPORT



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

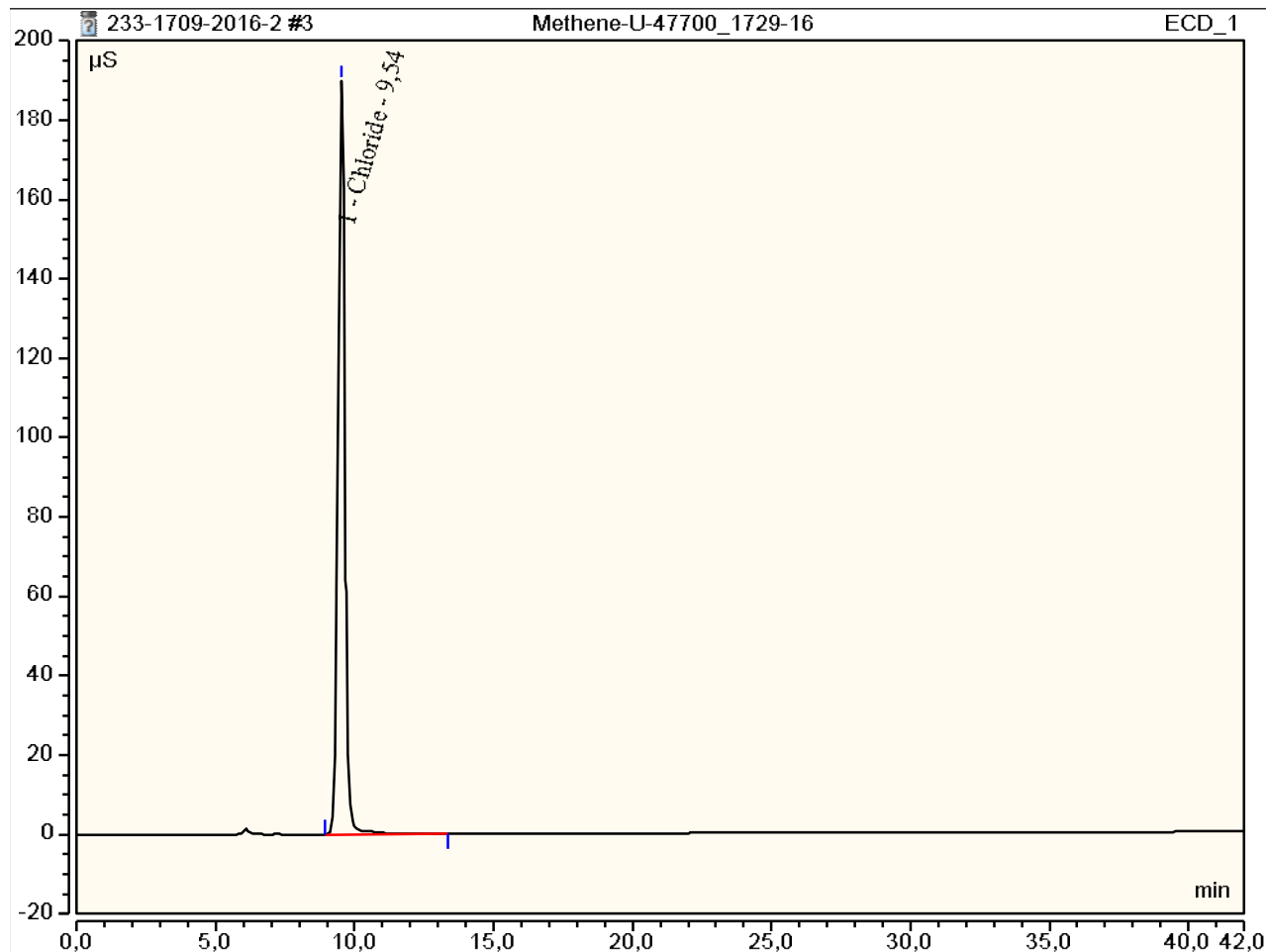
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
343.1343	1	17389178	C17 H24 Cl2 N2 O	(M+H)+
344.1378	1	3346386.38	C17 H24 Cl2 N2 O	(M+H)+
345.1316	1	11423149.2	C17 H24 Cl2 N2 O	(M+H)+
346.135	1	2083871.91	C17 H24 Cl2 N2 O	(M+H)+
347.1298	1	1804125.56	C17 H24 Cl2 N2 O	(M+H)+
348.1326	1	299175.74	C17 H24 Cl2 N2 O	(M+H)+
349.1351	1	30803.13	C17 H24 Cl2 N2 O	(M+H)+
350.1371	1	1711.68	C17 H24 Cl2 N2 O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	Methene-U-47700_1729-16	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	13-jan-2017 / 09:54	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	9,54	Chloride	BMB	53,35	189,89	n.a.
		TOTAL:		53,35	189,89	0,00





REPORT

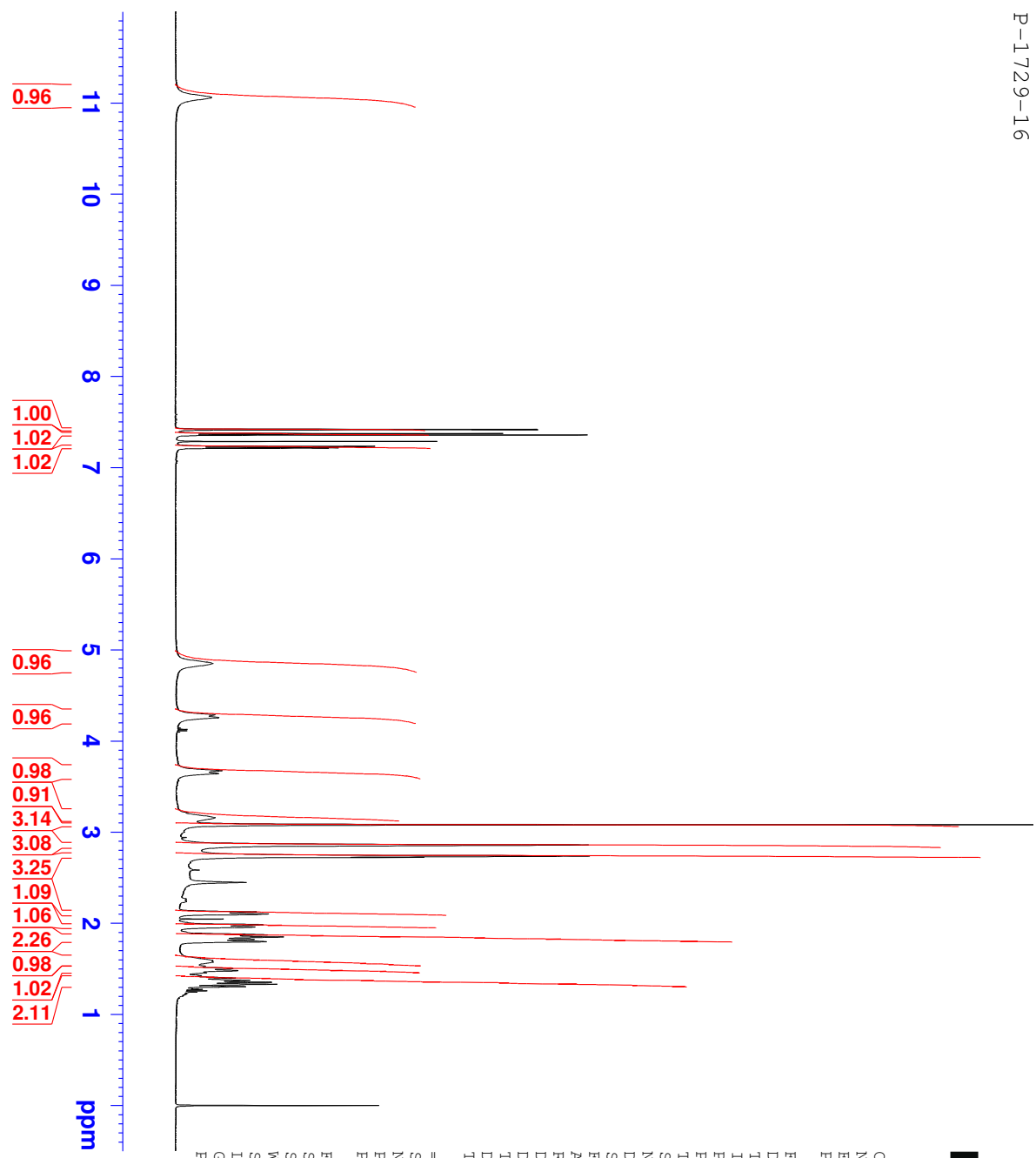
Sample ID:	1729-16
Our notebook code:	P-1729-16
NMR sample preparation:	15 mg dissolved in 0.7 mL CDCl ₃
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	2-(2-(3,4-dichlorophenyl)-N-methylacetamido)-N,N-dimethylcyclohexan-1-aminium cation
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure as evident by NMR (redundant signals in ¹H NMR at δ 2.25, 2.45 and 2.72 as well as in ¹³C NMR at δ 23.9, 26.4 and 30.2).
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 12, 2017



Current Data Parameters
 NAME P-1729-16
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161208
 Time 3.27
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 71.8
 DW 50.000 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 500.130885 MHz
 NUC1 1H
 P1 8.60 usec
 PLW1 26.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 500.1299990 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
NAME P-1729-16
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161208
Time 5.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 298.0 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
CPDPRG12 waltz16
PCPD2 80.00 usec
PLW2 26.00000000 W
PLW12 0.30046001 W
PLW13 0.15113001 W

F2 - Processing parameters
SI 32768
SF 125.77879 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

