



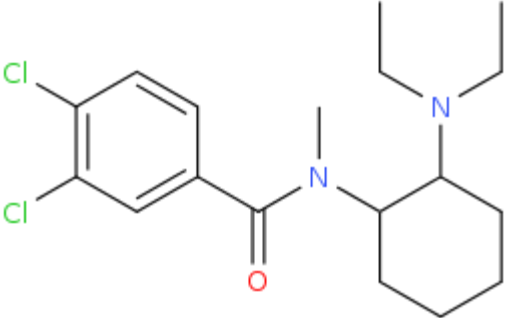
ANALYTICAL REPORT¹

U-49900 (C₁₈H₂₆Cl₂N₂O)

3,4-dichloro-N-[2-(diethylamino)cyclohexyl]-N-methylbenzamide

Remark – other NPS detected: **none**

Sample ID:	1734-16
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	11/14/2016
Date of entry (M/D/Y) into NFL database:	12/7/2016
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	3,4-dichloro-N-[2-(diethylamino)cyclohexyl]-N-methylbenzamide
Other names	U-49900
Formula (per base form)	C ₁₈ H ₂₆ Cl ₂ N ₂ O
M _w (g/mol)	357,32
Salt form/anions detected	HCl
StdInChIKey	AXACJBKFKCCIOR-UHFFFAOYSA-N
Compound Class	Opioids
Other NPS detected	none
Add.info (purity..)	pure by GC-MS, HPLC-TOF, 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 6.1 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

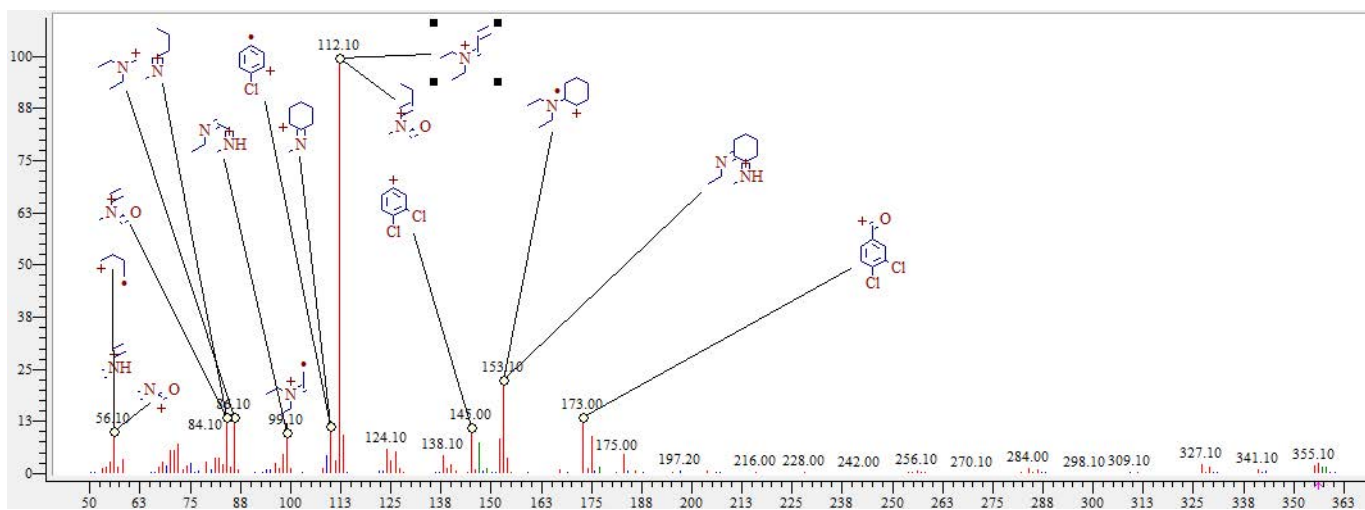
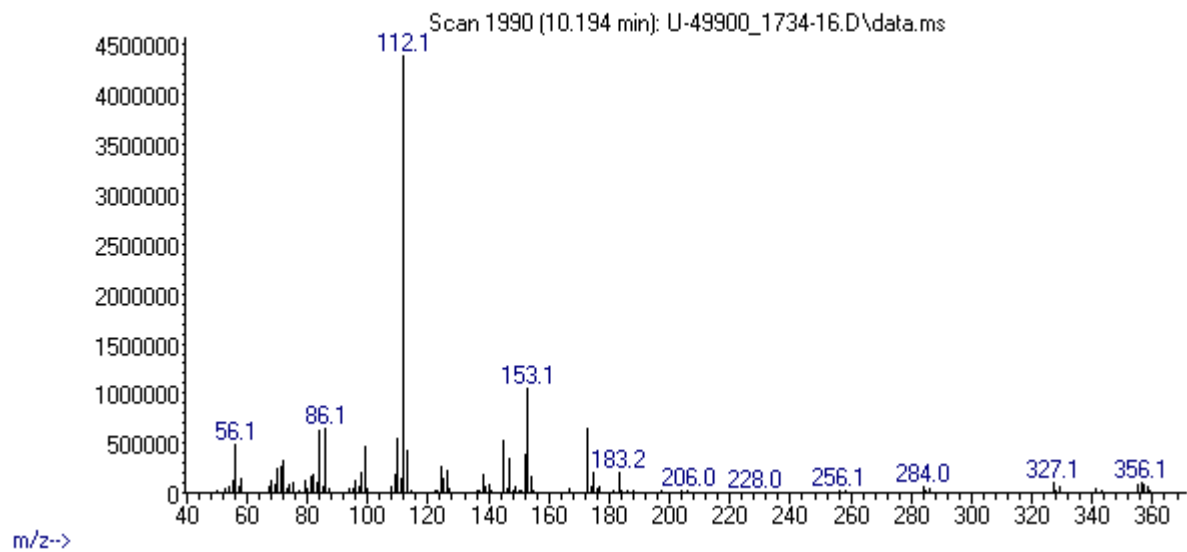
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 10,19 BP(1): 112; BP(2): 153,BP(3) :86,
HPLC-TOF	+	Exact mass (theoretical): 356,1422; measured value Δppm:-1,11; formula:C18H26Cl2N2O
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

ANALYTICAL RESULTS

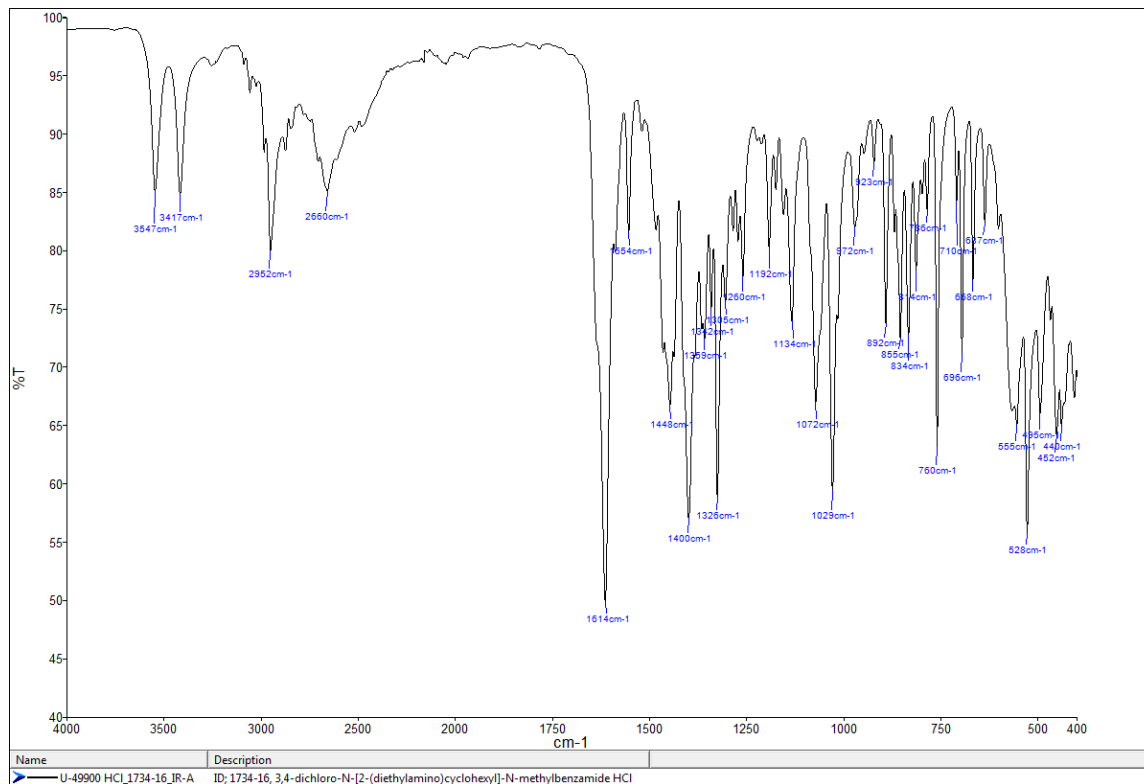
MS (EI)

Abundance

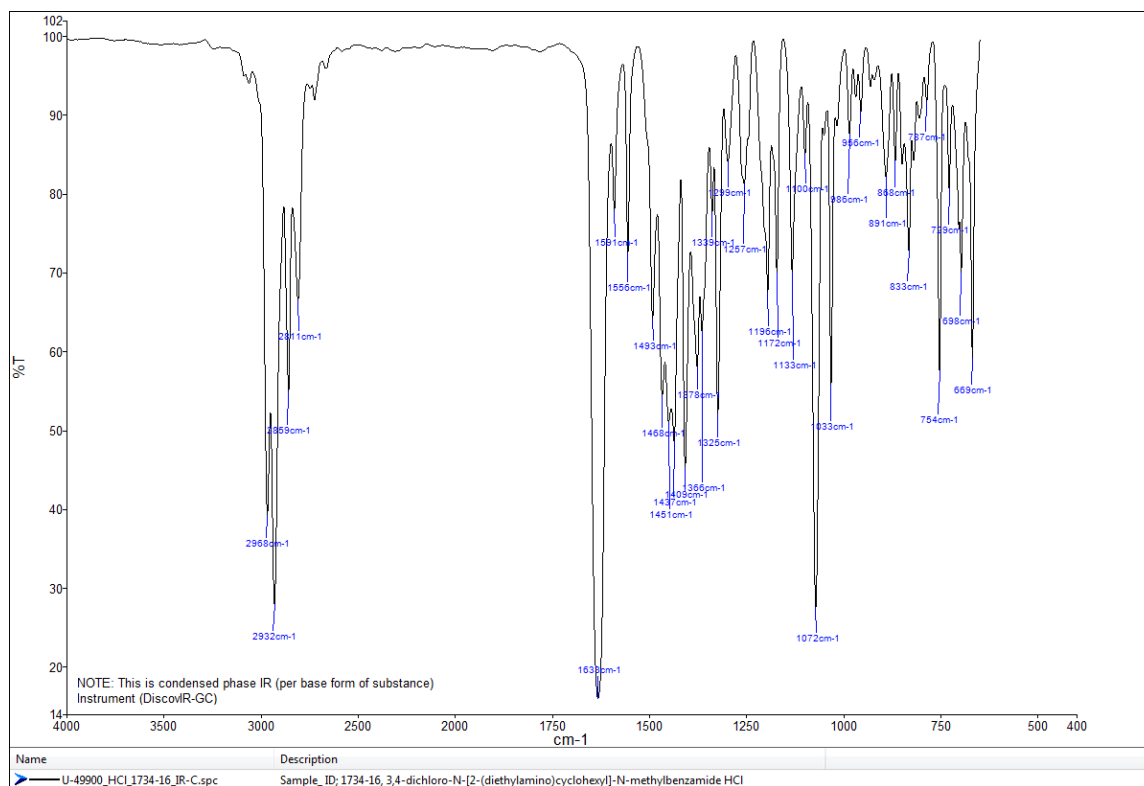


MS-fragmentation (red and green labeled peaks explained)

FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

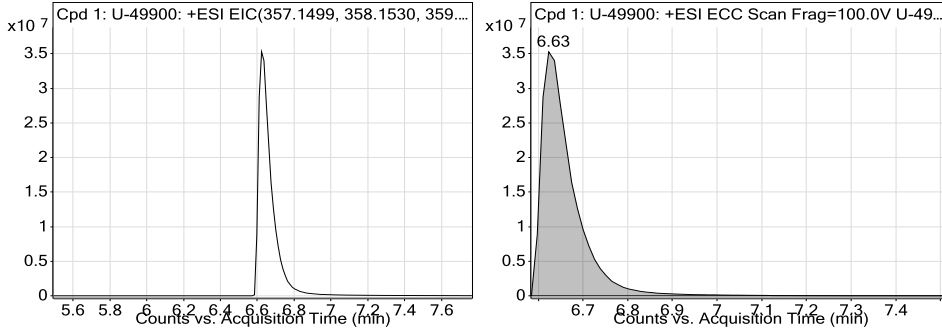
Data File	U-49900_1734-16.d	Sample Name	ID_1734-16
Sample Type	Sample	Position	P1-C5
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-10_10_2016-XDB-C18-ESI-poz-soft.m	Acquired Time	11/16/2016 1:18:06 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

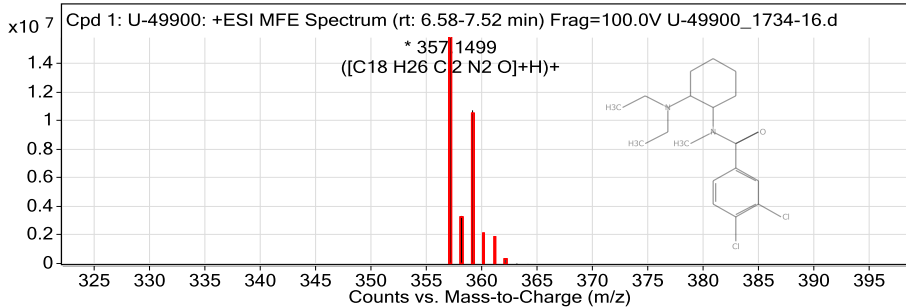
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: U-49900	U-49900	C18 H26 Cl2 N2 O	6.63	356.1426

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
U-49900	357.1499	6.63	356.1426	6.63	C18 H26 Cl2 N2 O	356.1422	-1.11

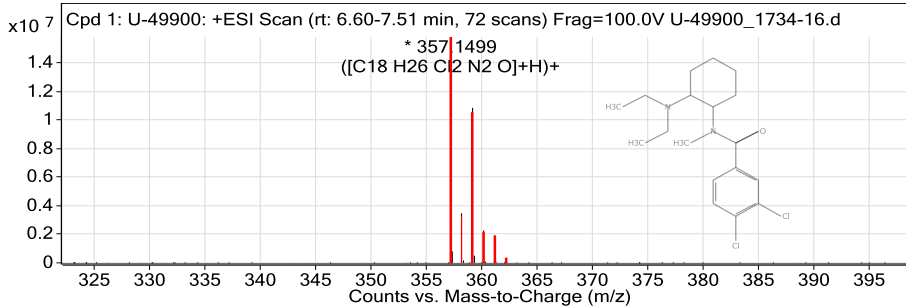
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

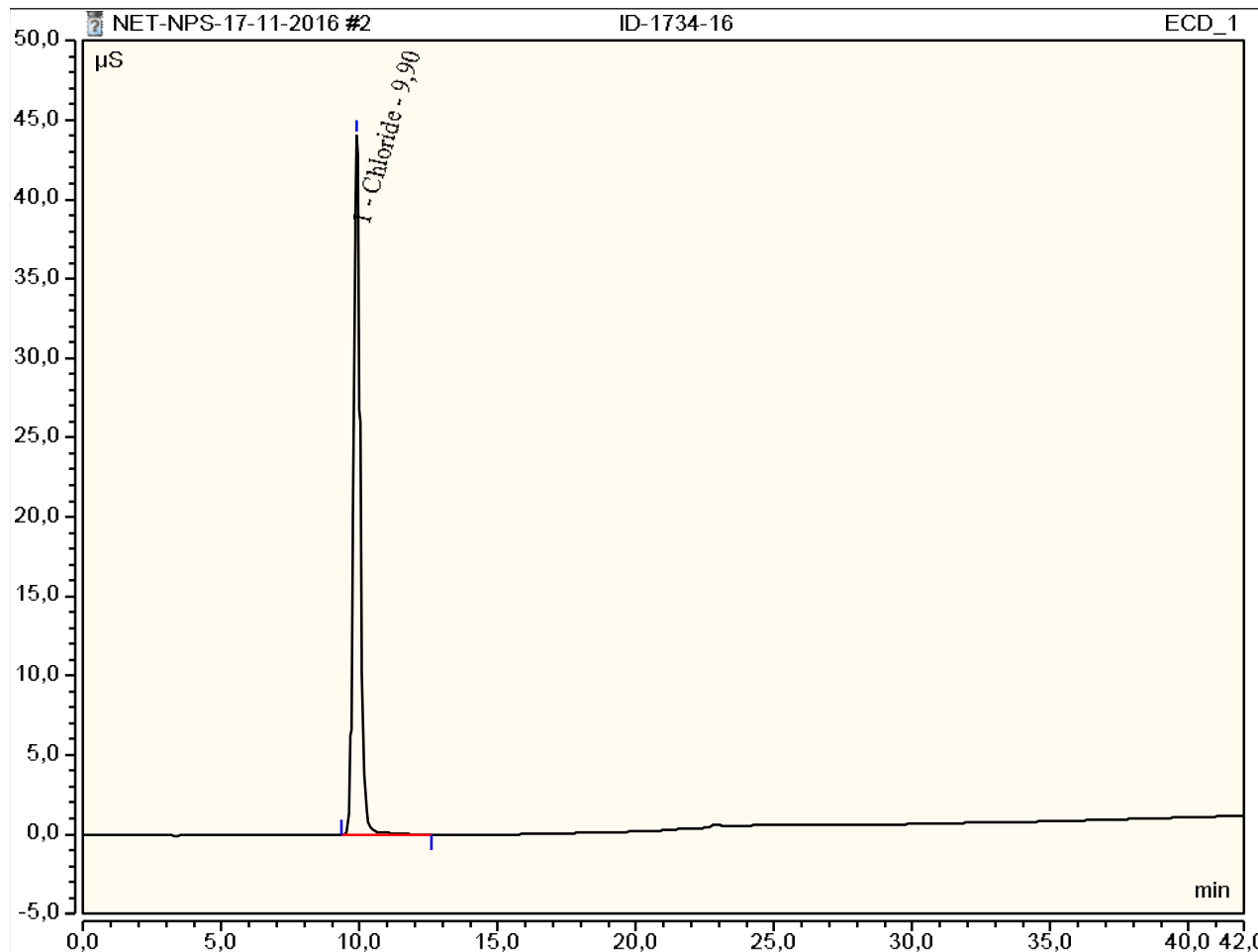
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
357.1499	1	15790953	C18 H26 Cl2 N2 O	(M+H)+
358.153	1	3324710.63	C18 H26 Cl2 N2 O	(M+H)+
359.1472	1	10709592.66	C18 H26 Cl2 N2 O	(M+H)+
360.1505	1	2074224.84	C18 H26 Cl2 N2 O	(M+H)+
361.1453	1	1762713.96	C18 H26 Cl2 N2 O	(M+H)+
362.1481	1	306293.16	C18 H26 Cl2 N2 O	(M+H)+
363.1506	1	34318.9	C18 H26 Cl2 N2 O	(M+H)+
364.1531	1	2898.18	C18 H26 Cl2 N2 O	(M+H)+

--- End Of Report ---

Peak Integration Report

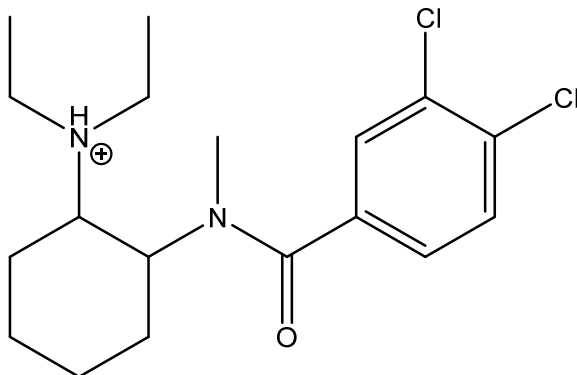
Sample Name:	ID-1734-16	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	17-nov-2016 / 13:09	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S} \cdot \text{min}$	Height μS	Amount mg/L
1,00	9,90	Chloride	BMB	12,42	44,09	n.a.
TOTAL:				12,42	44,09	0,00





REPORT

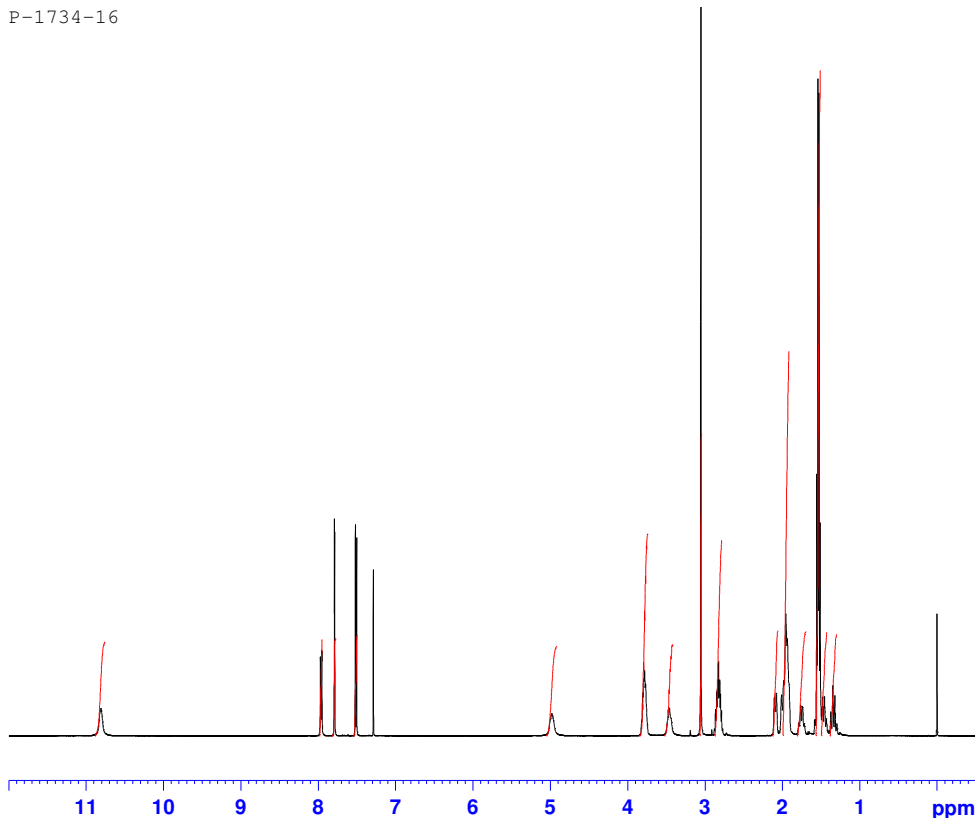
Sample ID:	1734-16
Our notebook code:	P-1734-16
NMR sample preparation:	15 mg dissolved in 0.7 mL CDCl ₃
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC, ¹ H- ¹⁵ N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	2-(3,4-dichloro- <i>N</i> -methylbenzamido)- <i>N,N</i> -diethylcyclohexan-1-aminium cation
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure according to the NMR.
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	December 7, 2016

P-1734-16



Current Data Parameters
 NAME P-1734-16
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161126
 Time 23.45
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 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 298.0 K
 D1 1.00000000 sec
 TD0 1



===== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 8.60 usec
 PLW1 26.00000000 W

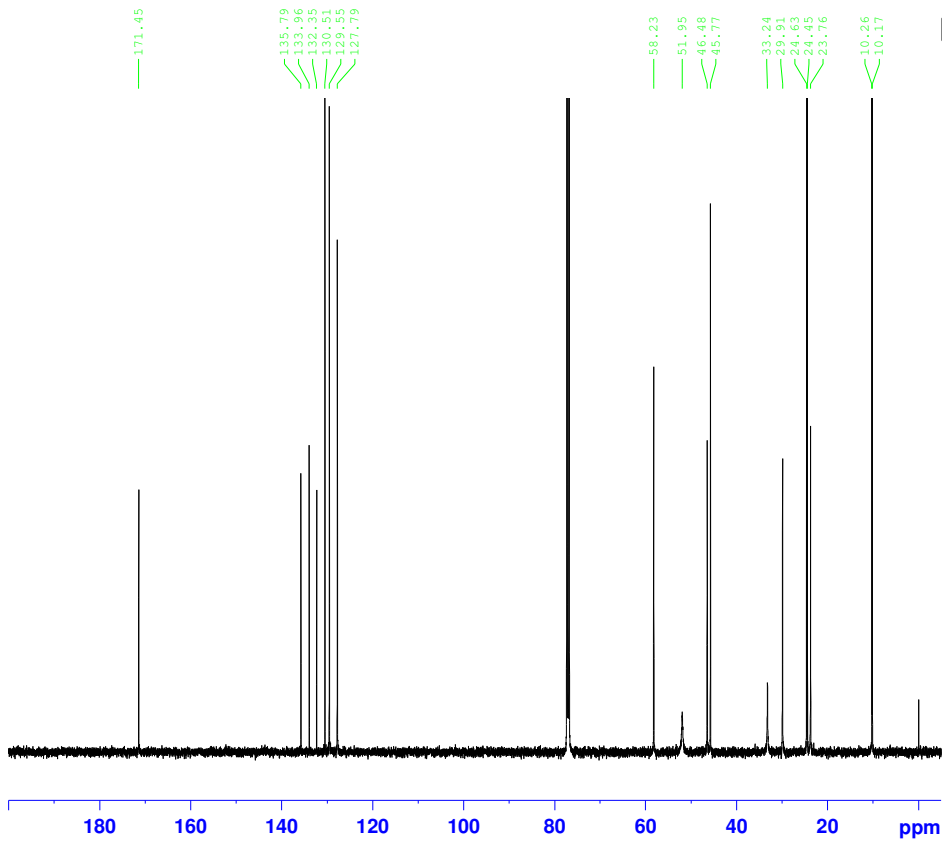
F2 - Processing parameters
 SI 65536
 SF 500.1299989 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

P-1734-16



Current Data Parameters
 NAME P-1734-16
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161127
 Time 2.20
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 4096
 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 1.00000000 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
 CPDPRG2 waltz16
 PCPD2 80.00 usec
 PLW2 26.00000000 W
 PLW12 0.30046001 W
 PLW13 0.15113001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577879 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40