NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY
Vodovodna 95, 1000 LJUBLJANA, SLOVENIA

T: +386 (0)1 428 44 93
E: nfl@policija.si
www.policija.si

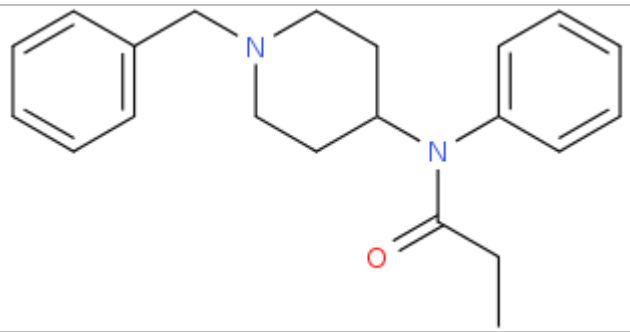
ANALYTICAL REPORT

Benzylfentanyl (C11H26N2O)

N-(1-benzylpiperidin-4-yl)-N-phenylpropanamide

Remark – other NPS detected: none

Sample ID:	1887-17
Sample description:	powder
Sample type:	seized /KP NLF case ref. 233-5217/2017 sample 13
Date of sample receipt (M/D/Y):	10/2/2017
Date of entry (M/D/Y) into NFL database:	11/20/2017
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	N-(1-benzylpiperidin-4-yl)-N-phenylpropanamide
Other names	R-4129 Benzyl Fentanyl N-(1-Benzyl-4-piperidinyl)-N-phenylpropanamide
Formula (per base form)	C21H26N2O
M_w (g/mol)	322,45
Salt form/anions detected	HCl
StdInChIKey (per base form)	POQDXIFVWVZVML-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	sample is a mixture of benzylfentanyl and 1-benzylpiperidin-4-yl propanoate

[1] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Report updates

date	comments (explanation)
25/03/2018	error in empirical formula corected: from C11H26N2O to C21H26N2O

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

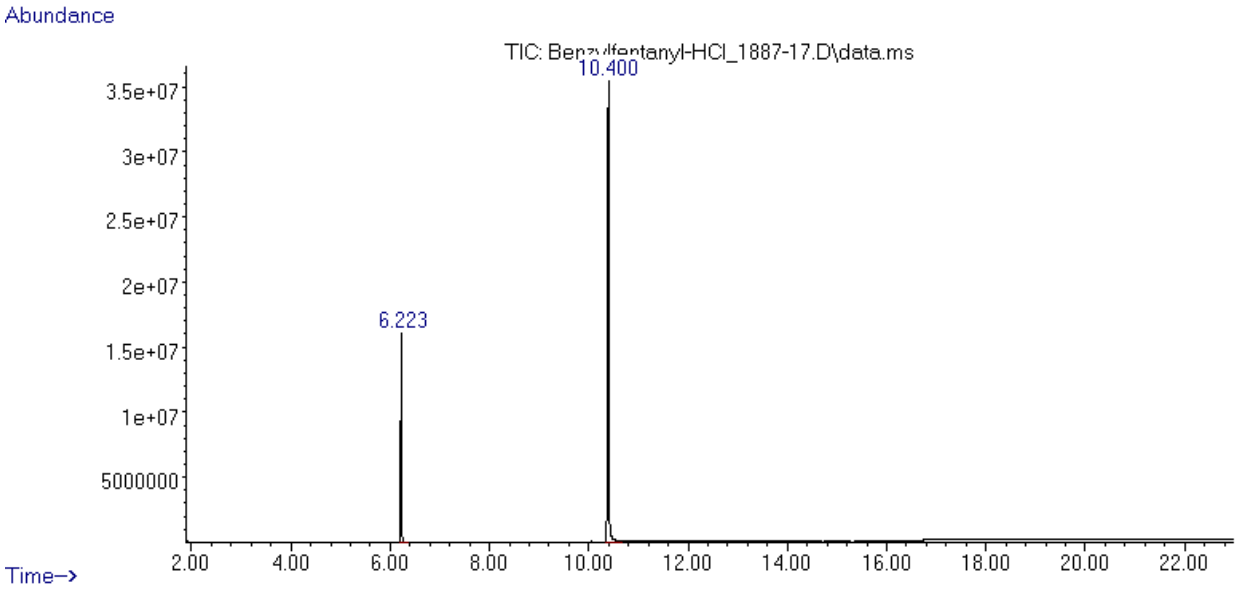
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	soluble

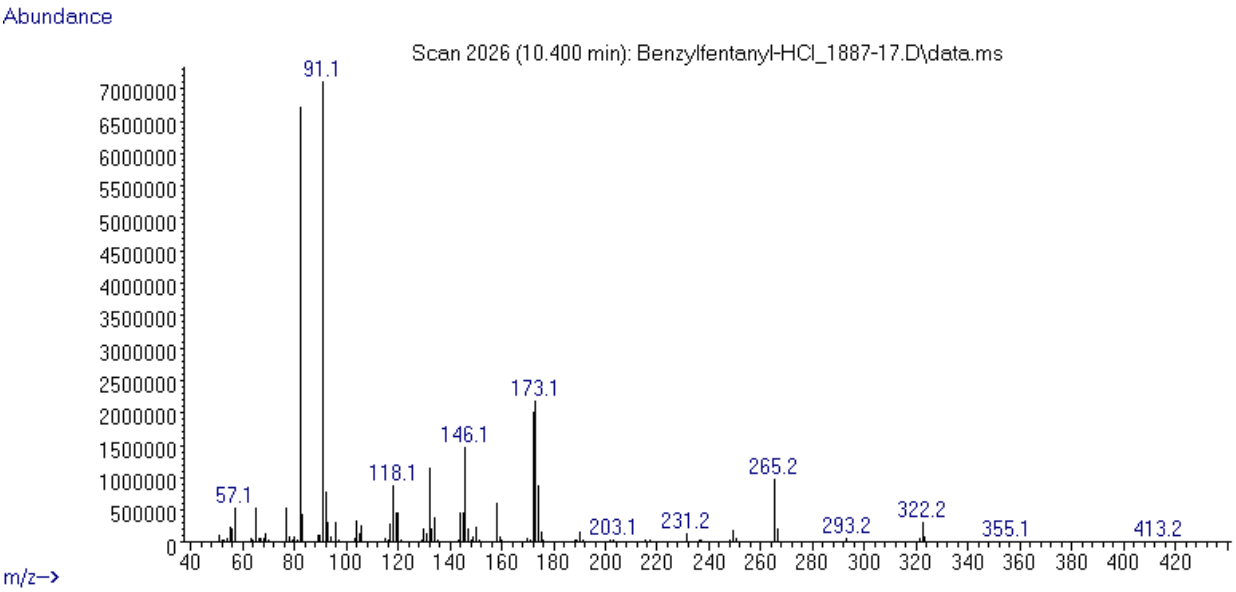
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 10,4 BP(1): 91; BP(2): 82,BP(3) :173,
HPLC-TOF	+	Exact mass (theoretical): 322,2045; measured value Δppm:-0,05; formula:C11H26N2O
FTIR-ATR	+	direct measurement (sample as received)-mixture of two compounds, spectrum is not shown in this report
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	-	
validation	+	validated by the compound CHEM-ID 1856-17 from the RESPONSE project database https://www.policija.si/apps/nfl_response_web/0_Analytical_Reports_final/Benzylfentanyl-ID-1856_17_report.pdf
other		

ANALYTICAL RESULTS

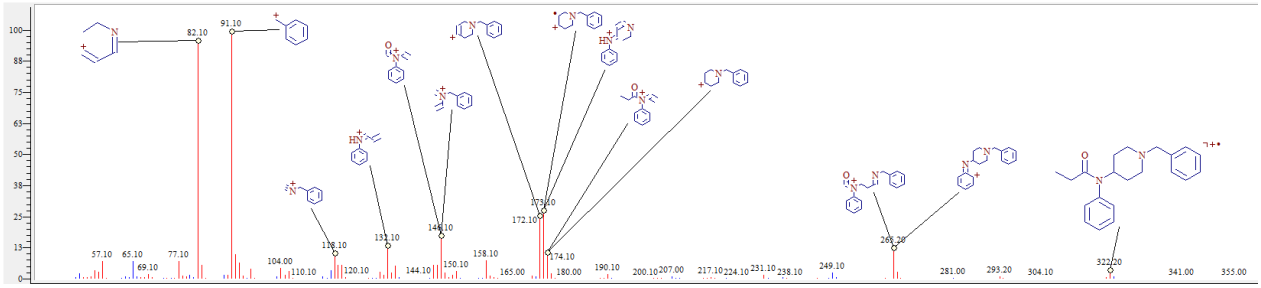
GC chromatogram (compound at RT=6.2 was identified as 1-benzylpiperidin-4-yl propanoate and at RT=10.4 as benzylfentanyl)



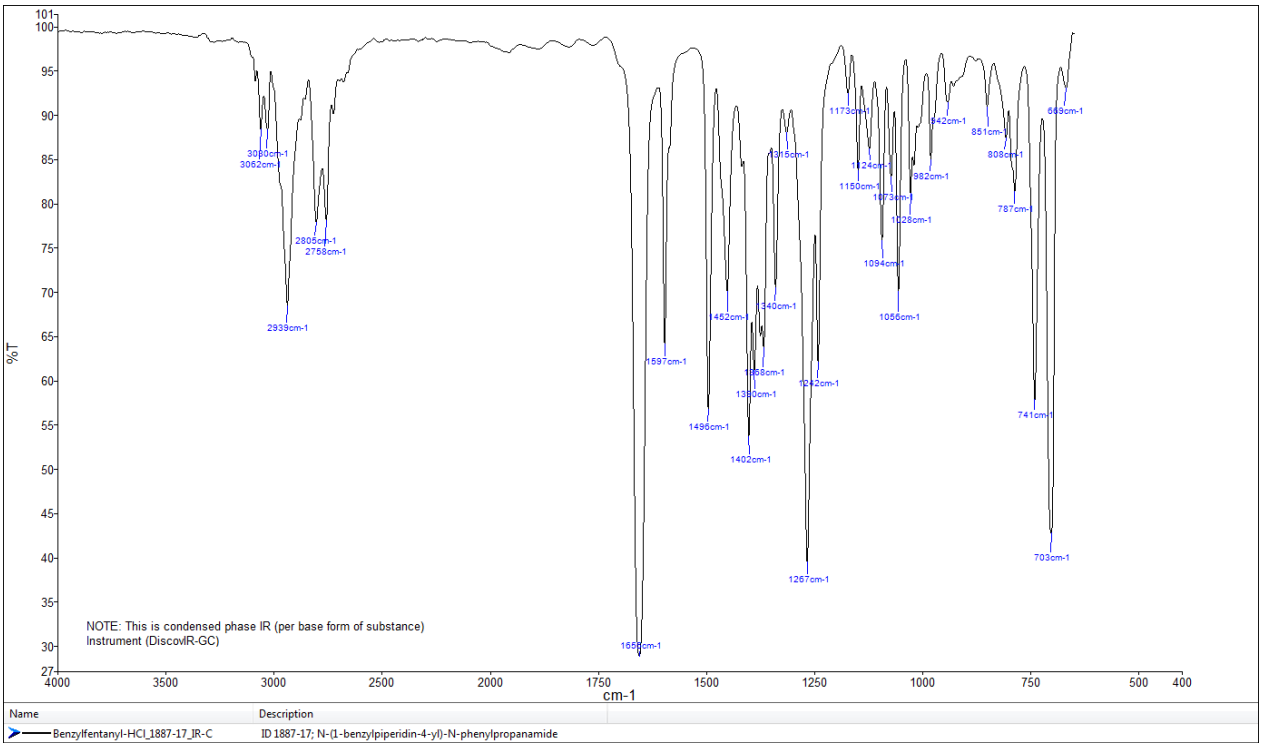
MS spectrum benzylfentanyl



Fragmentation pattern (red labeled peaks explained – possible fragments shown)



IR (condensed phase – after chromatographic separation): benzylfentanyl



TOF REPORT

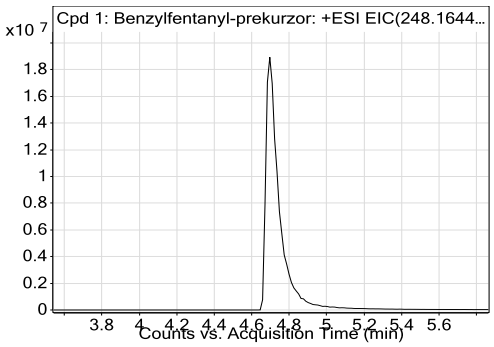
Data File	233-5217-2017_13.d	**Sample Name**	eks. in MeOH
Sample Type	Sample	**Position**	P1-D3
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-19_07_2017-XDB-C18-ESI-final.m	**Acquired Time**	11/10/2017 1:31:28 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	MeOH		

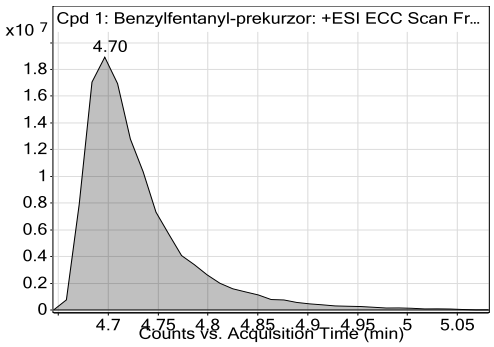
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Benzylfentanyl-prekurzor	Benzylfentanyl-prekurzor	C15 H21 N O2	4.7	247.1572
Cpd 2: Benzylfentanyl	Benzylfentanyl	C21 H26 N2 O	6.18	322.2045

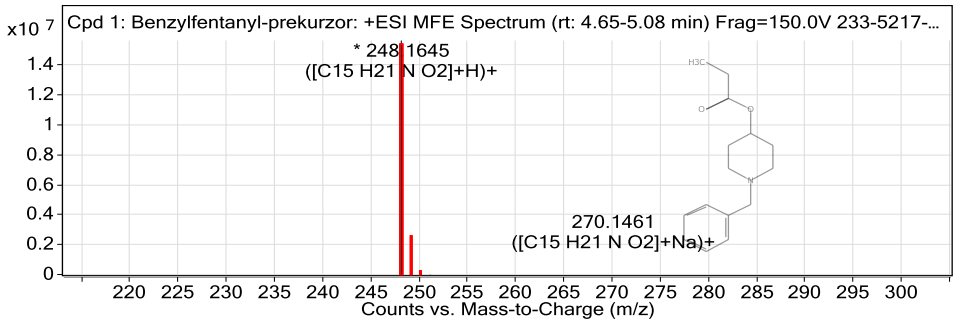
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Benzylfentanyl-prekurzor	248.1645	4.7	247.1572	4.7	C15 H21 N O2	247.1572	0.03

Compound Chromatograms

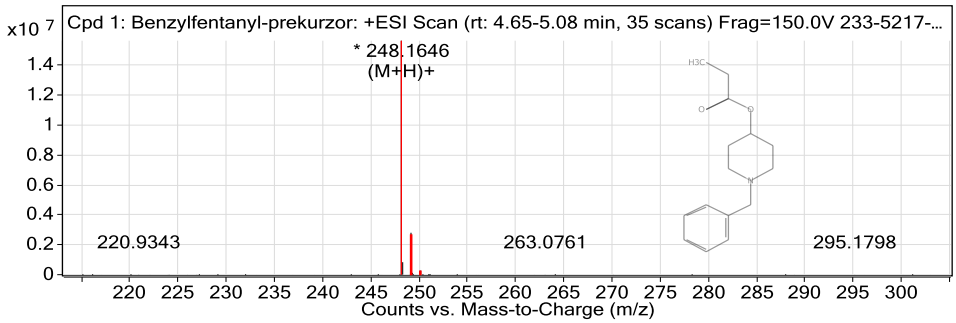




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



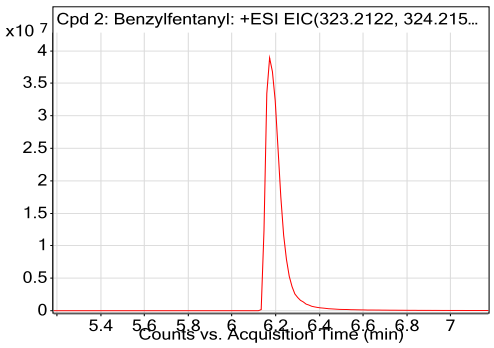
MS Spectrum Peak List

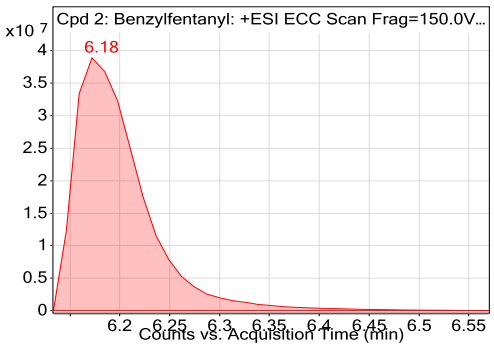
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
248.1645	1	15645148	C15 H21 N O2	(M+H)+
249.1679	1	2432736.37	C15 H21 N O2	(M+H)+
250.1709	1	243166.04	C15 H21 N O2	(M+H)+
251.1732	1	18571.44	C15 H21 N O2	(M+H)+
270.1461	1	2693.4	C15 H21 N O2	(M+Na)+

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Benzylfentanyl	323.2117	6.18	322.2045	6.18	C21 H26 N2 O	322.2045	-0.05

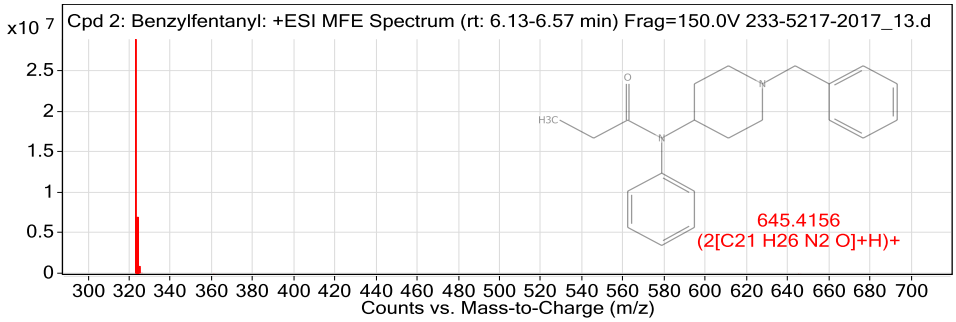
Compound Chromatograms

TOF REPORT

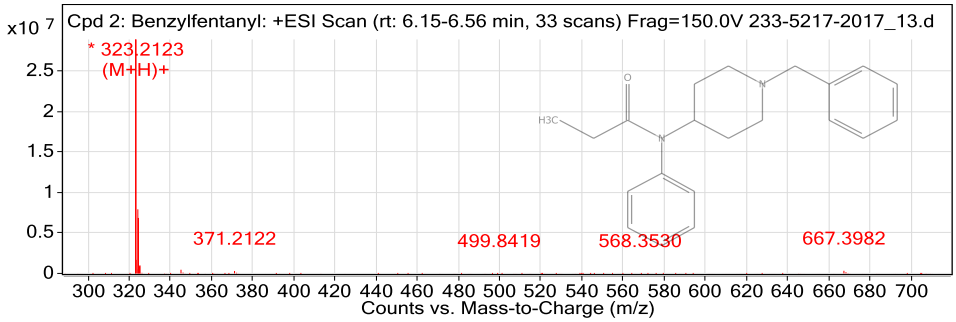




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
323.2117	1	28869686	C21 H26 N2 O	(M+H)+
324.2153	1	6977164.59	C21 H26 N2 O	(M+H)+
325.2184	1	732147.11	C21 H26 N2 O	(M+H)+
326.2215	1	59266.35	C21 H26 N2 O	(M+H)+
361.1674	1	26014.57	C21 H26 N2 O	(M+K)+
362.1708	1	6275.72	C21 H26 N2 O	(M+K)+
645.4156	1	21329.1	C21 H26 N2 O	(2M+H)+
646.419	1	10553.87	C21 H26 N2 O	(2M+H)+
683.3719	1	11354.52	C21 H26 N2 O	(2M+K)+
684.3762	1	5377.08	C21 H26 N2 O	(2M+K)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**233-5217-17_13**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**15-nov-2017 / 12:06**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
2,00	9,11	**Chloride**	BMB	5,79	27,87	n.a.
		TOTAL:		5,79	27,87	0,00