EUROPEAN PROJECT RESPONSE to challenges in forensic drugs analyses

Co-funded by the Prevention of and Fight against Crime Programme of the European Union

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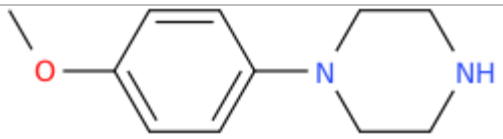
ANALYTICAL REPORT

pMeOPP (C11H16N2O)

1-(4-methoxyphenyl)piperazine

Remark – other active cpd. detected: none

Sample ID:	1785-17
Sample description:	powder - white
Sample type:	RM-reference material
Comments[1]:	CHIRON Batch# 15 100; RESPONSE -purchasing
Date of entry:	4/4/2017

Substance identified-structure[2] (base form)	
Systematic name:	1-(4-methoxyphenyl)piperazine
Other names:	1-(4-Anisyl)piperazine; 4-MeOPP; MeOPP; p-Methoxyphenylpiperazine
Formula (per base form)	C11H16N2O
M_w (g/mol)	192,26
Salt form:	HCl
StdInChIKey (per base form)	MRDGZSKYFPGAKP-UHFFFAOYSA-N
Other active cpd. detected	none
Add.info (purity..)	99,5% (??? - see comment below)
Comments on reference material and vendor`s certificate.	Significant errors were detected on vendor`s certificate! Compound was sold as base form. In fact it was 2XHCl salt. Salt form was identified in National forensic laboratory (NFL) by FTIR-ATR (obtained spectrum is in excellent agreement (QM>0.99) with the one from the Forendex library for the 4-MeOPP*2HCl compound). In NFL the chloride ions were confirmed also by Ion chromatography.

[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)

Supporting information

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 5,5 BP(1): 150; BP(2): 192,BP(3) :120,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form

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. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1}; resolution 4cm^{-1}

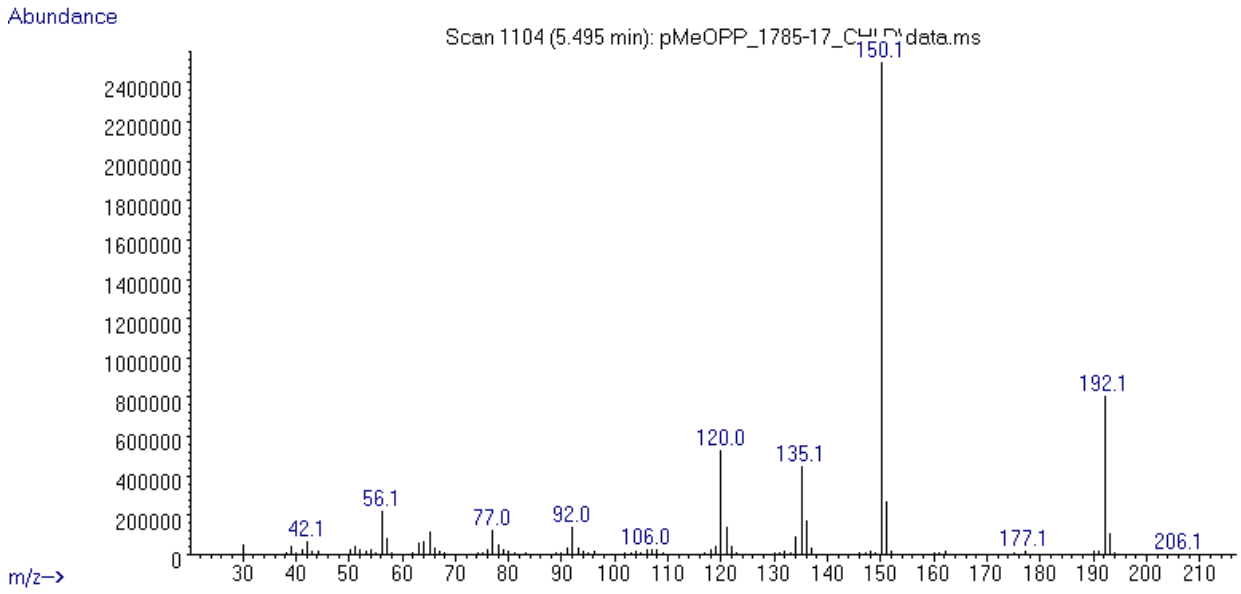
3. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 ^{0}C. Chromatographic separation as above **(1).**
Split MS : IR = 1 : 9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole 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}.

4. HPLC-TOF for exact monoisotopic mass and empirical formula control - results are not shown in the report.

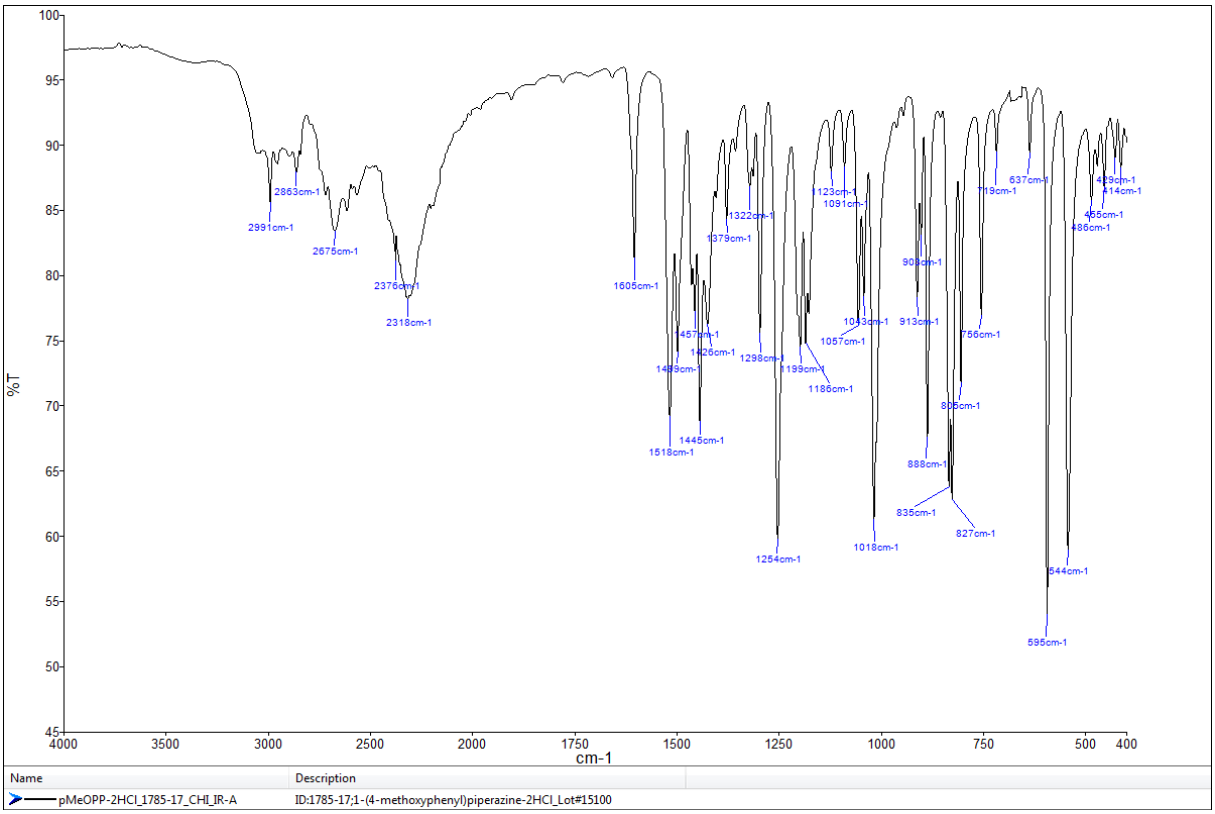
5. **IC** (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30˚C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

ANALYTICAL RESULTS

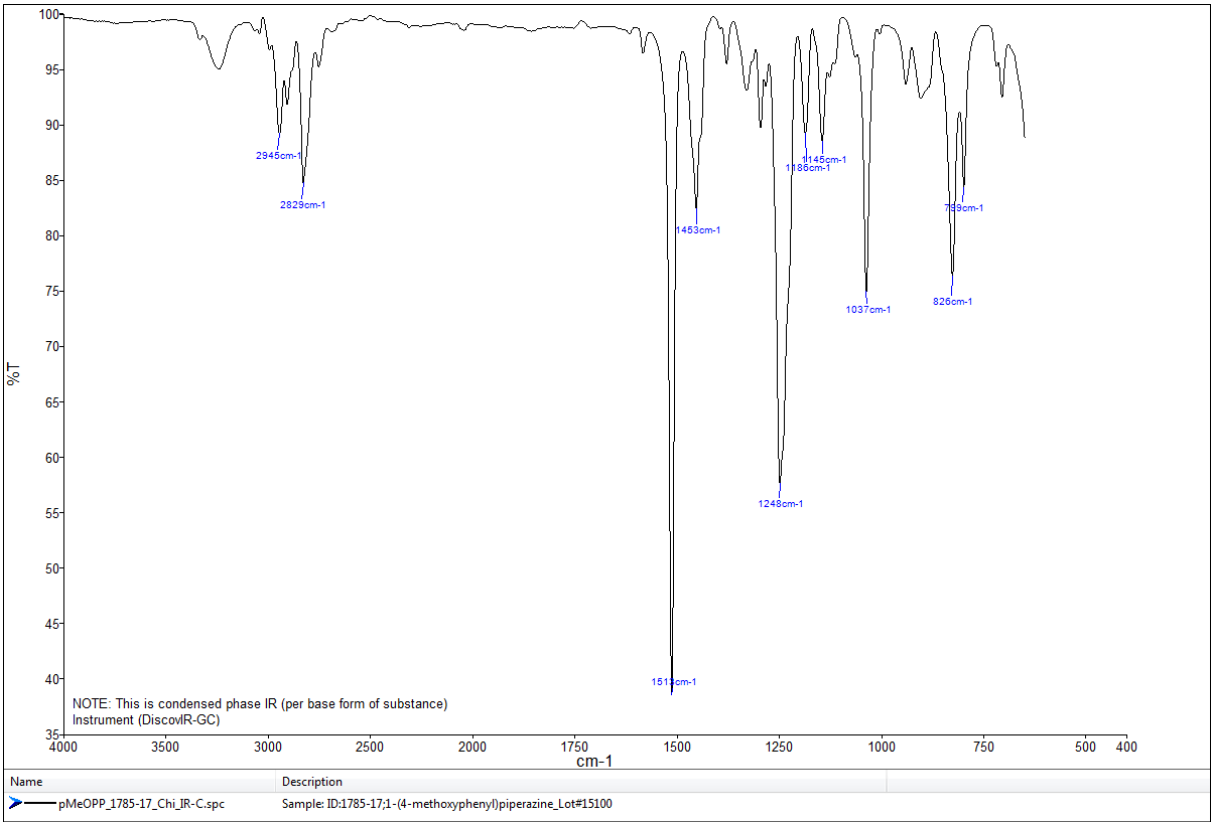
MS (EI)



FTIR-ATR - sample as received



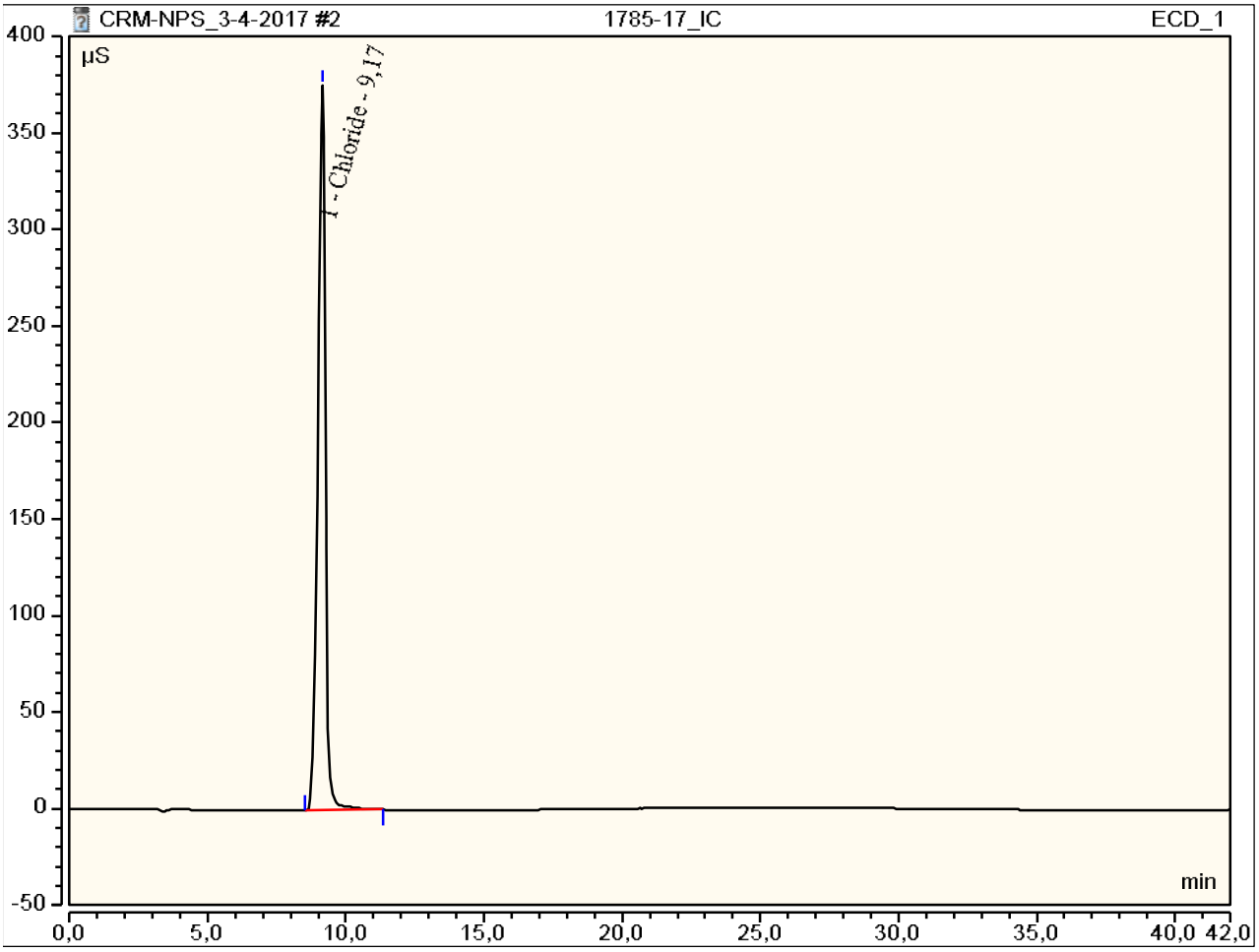
IR (condensed phase – after chromatographic separation)



Peak Integration Report

Sample Name:	**1785-17_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**03-apr-2017 / 10:14**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	9,17	**Chloride**	BMB	117,17	375,74	n.a.
		TOTAL:		117,17	375,74	0,00



TOF REPORT

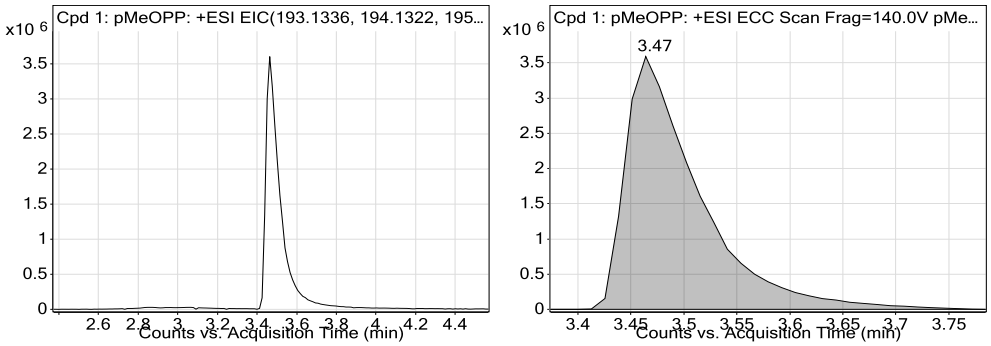
Data File	pMeOPP_1785-17.d	**Sample Name**	ID_1785-17
Sample Type	Sample	**Position**	P1-C5
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-17_03_2017-XDB-C18-ESI-final.m	**Acquired Time**	3/29/2017 9:31:02 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

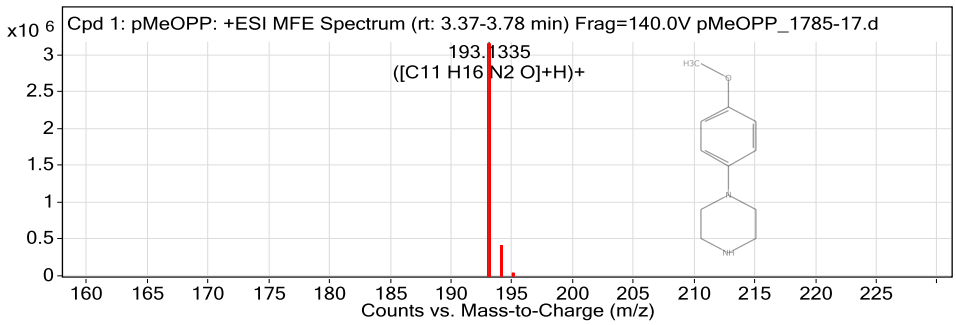
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: pMeOPP	pMeOPP	C11 H16 N2 O	3.47	192.1263

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
pMeOPP	193.1335	3.47	192.1263	3.47	C11 H16 N2 O	192.1263	-0.04

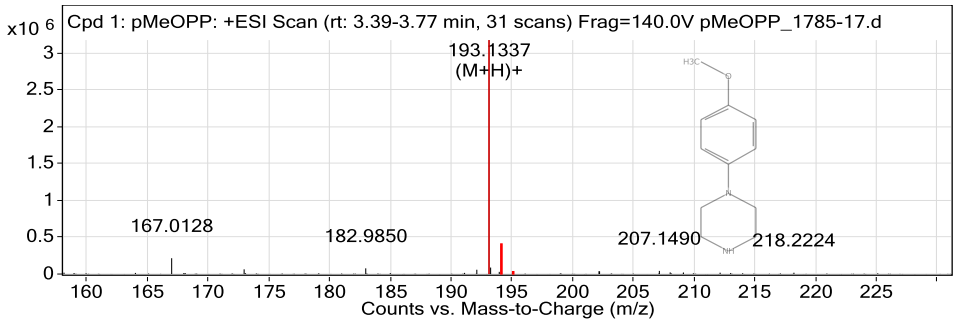
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
193.1335	1	3175292.75	C11 H16 N2 O	(M+H)+
194.1369	1	380259.06	C11 H16 N2 O	(M+H)+
195.1374	1	21230.13	C11 H16 N2 O	(M+H)+
196.1368	1	842.41	C11 H16 N2 O	(M+H)+

--- End Of Report ---