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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

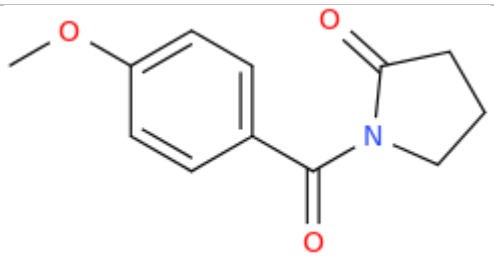
ANALYTICAL REPORT

aniracetam (C12H13NO3)

1-(4-methoxybenzoyl)pyrrolidin-2-one

Remark – other NPS detected: none

Sample ID:	1962-18
Sample description:	powder
Sample type:	collected /Customs, Slovenia
Date of sample receipt (DD/MM/YYYY):	02/08/2018
Date of entry (DD/MM/YYYY) into NFL database:	05/09/2018
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-methoxybenzoyl)pyrrolidin-2-one
Other names	Ro-13-3057;Draganon; Sarpul;Ampamet;Memodrin; Referan;1-(4-methoxyphenyl)carbonylpyrrolidin-2-one;1-(4-methoxybenzoyl)-2-pyrrolidone
Formula (per base form)	C12H13NO3
M_w (g/mol)	219.24
Salt form/anions detected	base
StdInChIKey (per base form)	ZXNRTKGTQJPIJK-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	pure by GC-MS

[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 KOH 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

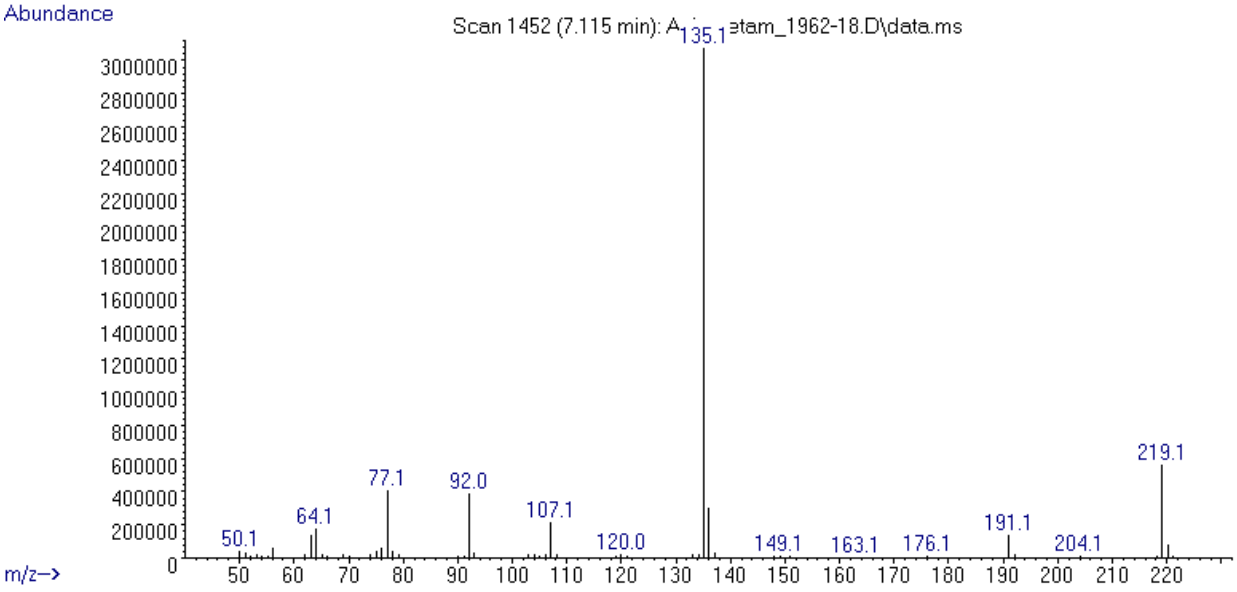
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
H_2O	not soluble

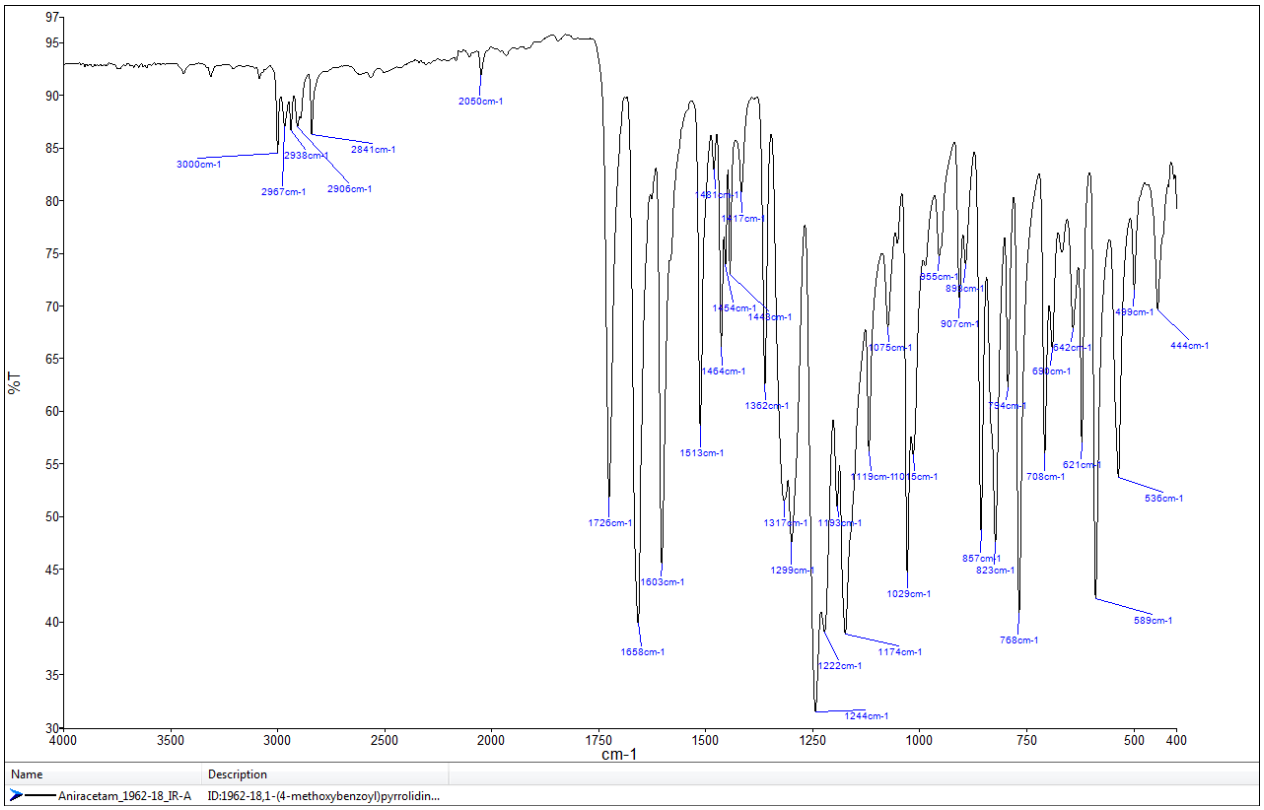
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 7,12 BP(1): 135; BP(2): 219,BP(3) :77,
HPLC-TOF	+	Exact mass (theoretical): 219,0895; measured value Δppm:0,05; formula:C12H13NO3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (solid phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	-	
validation		MS spectrum consistent by those published in NIST2014.L and DD2018.L
other		

ANALYTICAL RESULTS

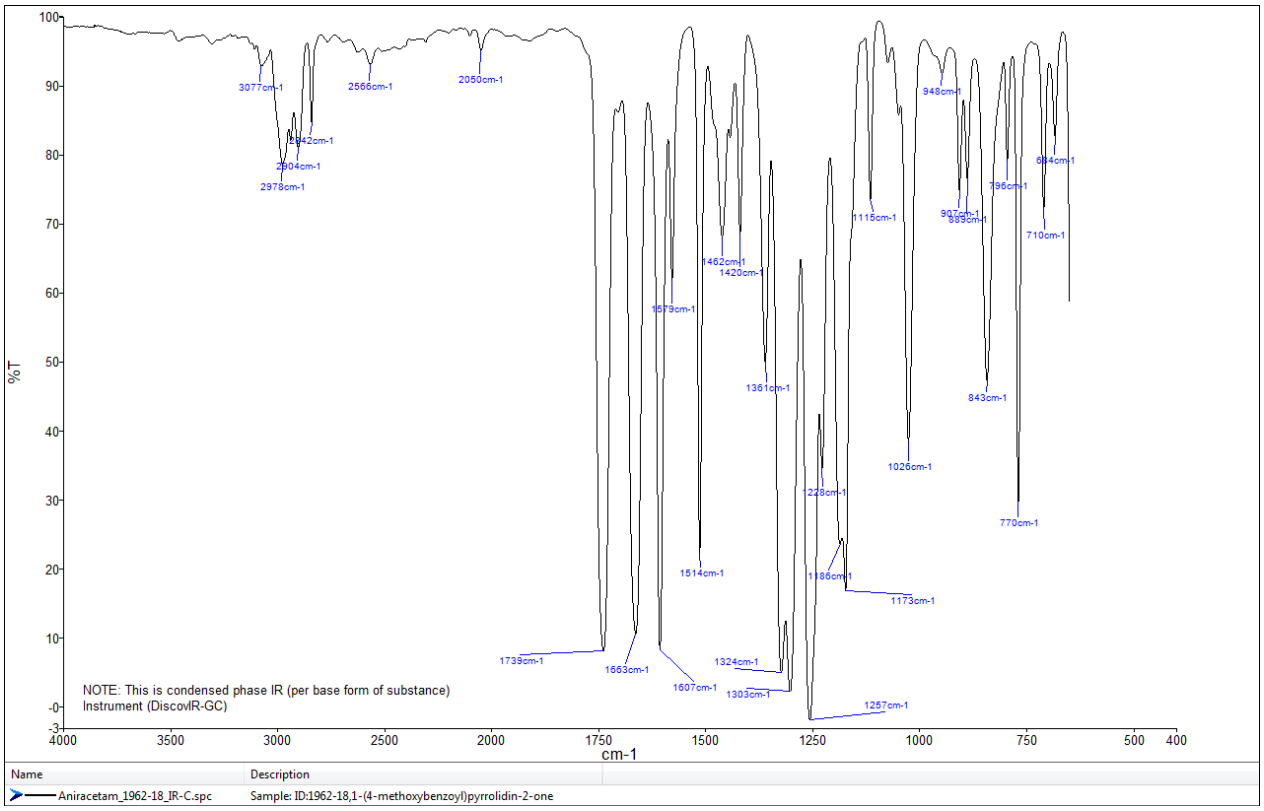
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (solid phase – after chromatographic separation)



TOF REPORT

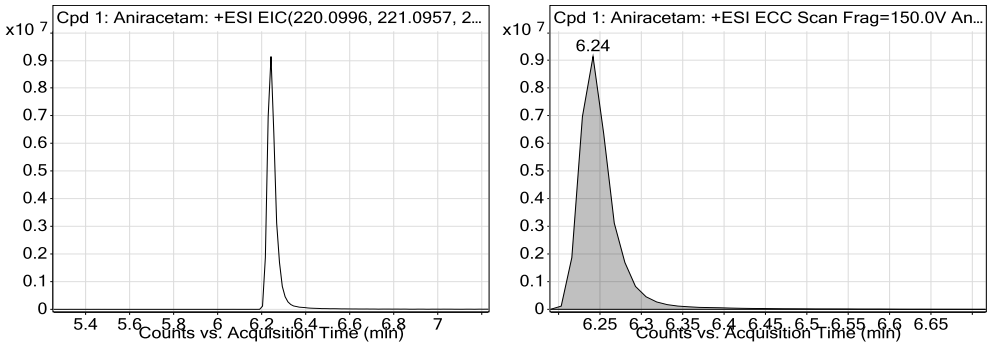
Data File	Aniracetam.d	**Sample Name**	ID 1962-18
Sample Type	Sample	**Position**	P1-C8
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-04_12_2017-XDB-C18-ESI+.m	**Acquired Time**	8/22/2018 4:40:28 PM
IRM Calibration Status	Success	**DA Method**	a-Drugs_NFL.m
Comment	MeOH		

Compound Table

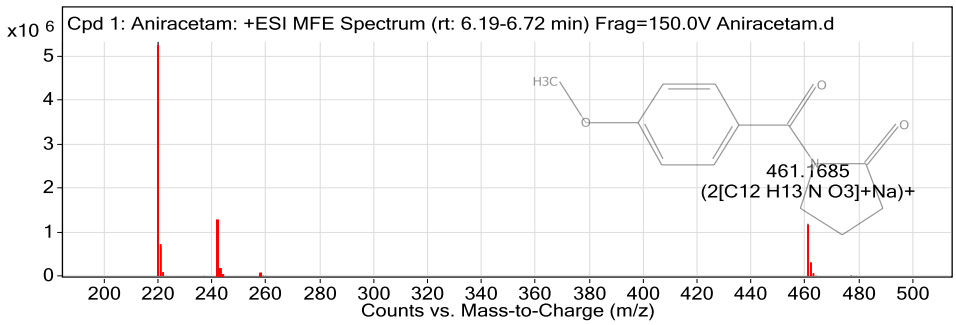
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Aniracetam	Aniracetam	C12 H13 N O3	6.24	219.0895

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Aniracetam	220.0966	6.24	219.0895	6.24	C12 H13 N O3	219.0895	0.05

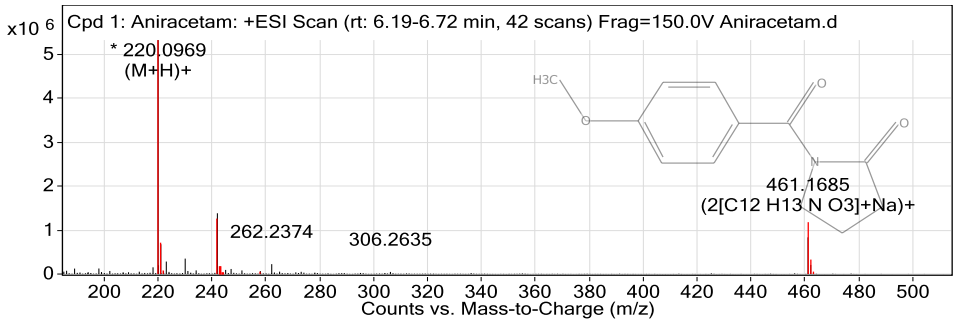
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
220.0966	1	5317944	C12 H13 N O3	(M+H)+
221.1006	1	646318.55	C12 H13 N O3	(M+H)+
222.1027	1	65479.34	C12 H13 N O3	(M+H)+
242.0787	1	1268084.13	C12 H13 N O3	(M+Na)+
243.082	1	153196.37	C12 H13 N O3	(M+Na)+
244.0844	1	16156.22	C12 H13 N O3	(M+Na)+
258.0524	1	58653.91	C12 H13 N O3	(M+K)+
461.1685	1	1184331.88	C12 H13 N O3	(2M+Na)+
462.1718	1	292635.01	C12 H13 N O3	(2M+Na)+
463.1741	1	46470.31	C12 H13 N O3	(2M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**1962_18_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI - julij 2018**	*Operator:*	**kemija**
Inj. Date / Time:	**23-avg-2018 / 09:55**	*Run Time:*	**47,10**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount n.a.
		TOTAL:		0,00	0,00	0,00