NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY

Vodovodna 95
1000 Ljubljana
SLOVENIJA

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

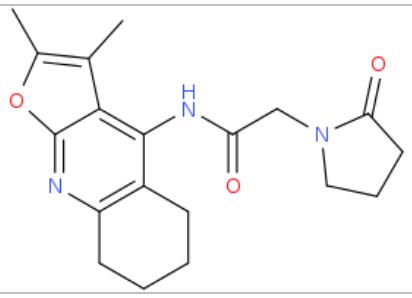
ANALYTICAL REPORT[1]

Coluracetam (C19H23N3O3)

N-{2,3-dimethyl-5H,6H,7H,8H-furo[2,3-b]quinolin-4-yl}-2-(2-oxopyrrolidin-1-yl)acetamide

Remark – other NPS detected: none

Sample ID:	1677-16
Sample description:	powder - white
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	9/16/2016
Date of entry (M/D/Y) into NFL database:	10/3/2016
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	N-{2,3-dimethyl-5H,6H,7H,8H-furo[2,3-b]quinolin-4-yl}-2-(2-oxopyrrolidin-1-yl)acetamide
Other names	BCI-540, MKC-231
Formula (per base form)	C19H23N3O3
M_w (g/mol)	341,4
Salt form/anions detected	base
StdInChIKey	PSPGQHXMUKWNDI-UHFFFAOYSA-N
Compound Class	Piperidines & pyrrolidines
Other NPS detected	none
Add.info (purity..)	

[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 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 6.1 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

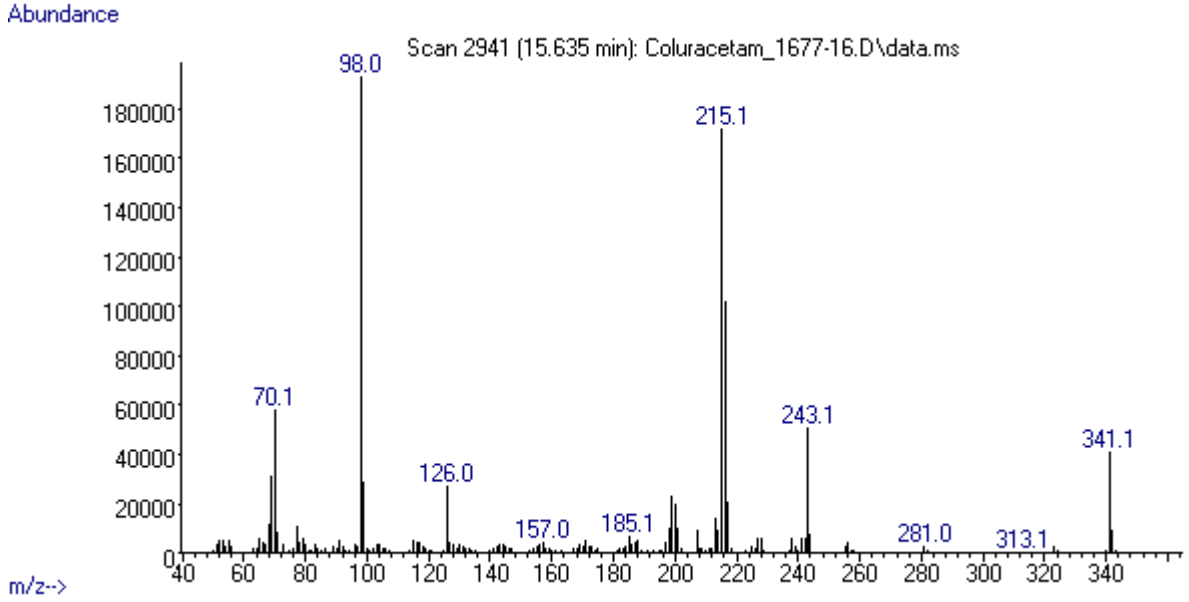
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	solubility
H_2O	soluble

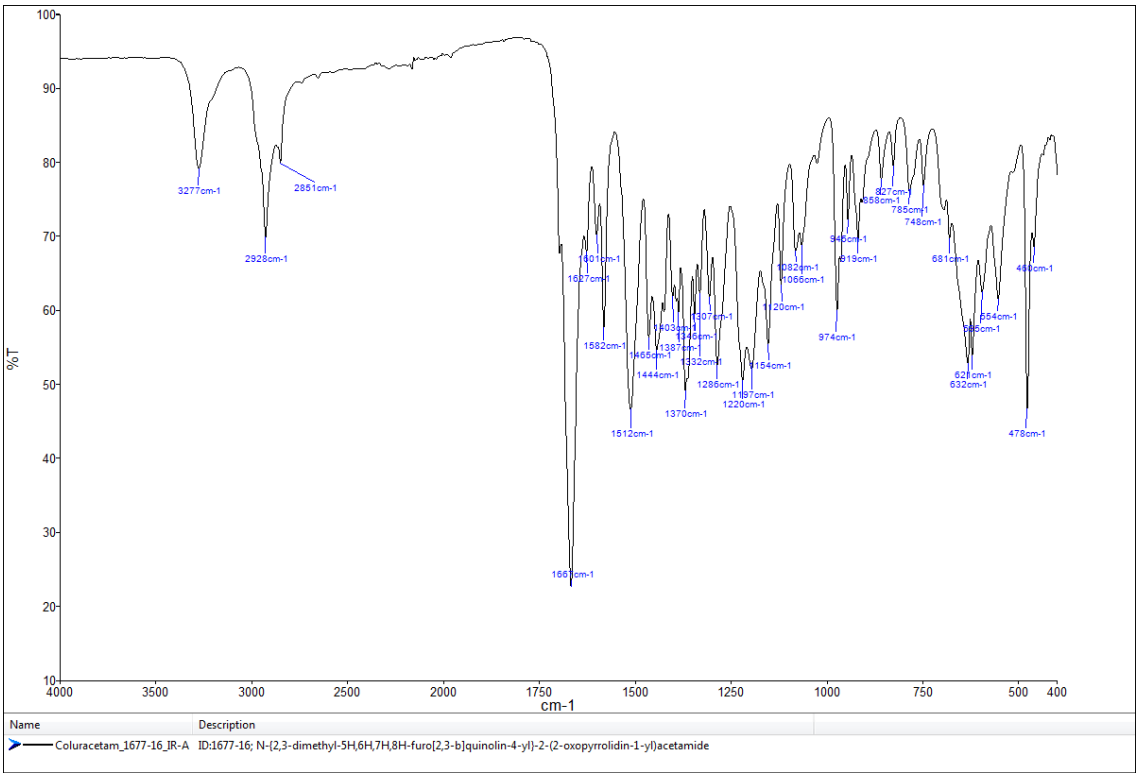
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 15,63 BP(1): 98; BP(2): 215,BP(3) :70,
HPLC-TOF	+	Exact mass (theoretical): 341,1739; measured value Δppm:0,06; formula:C19H23N3O3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	-	
validation		MS spectrum consistent by ENFSI2016.L andS WGDRUG.L
other		

ANALYTICAL RESULTS

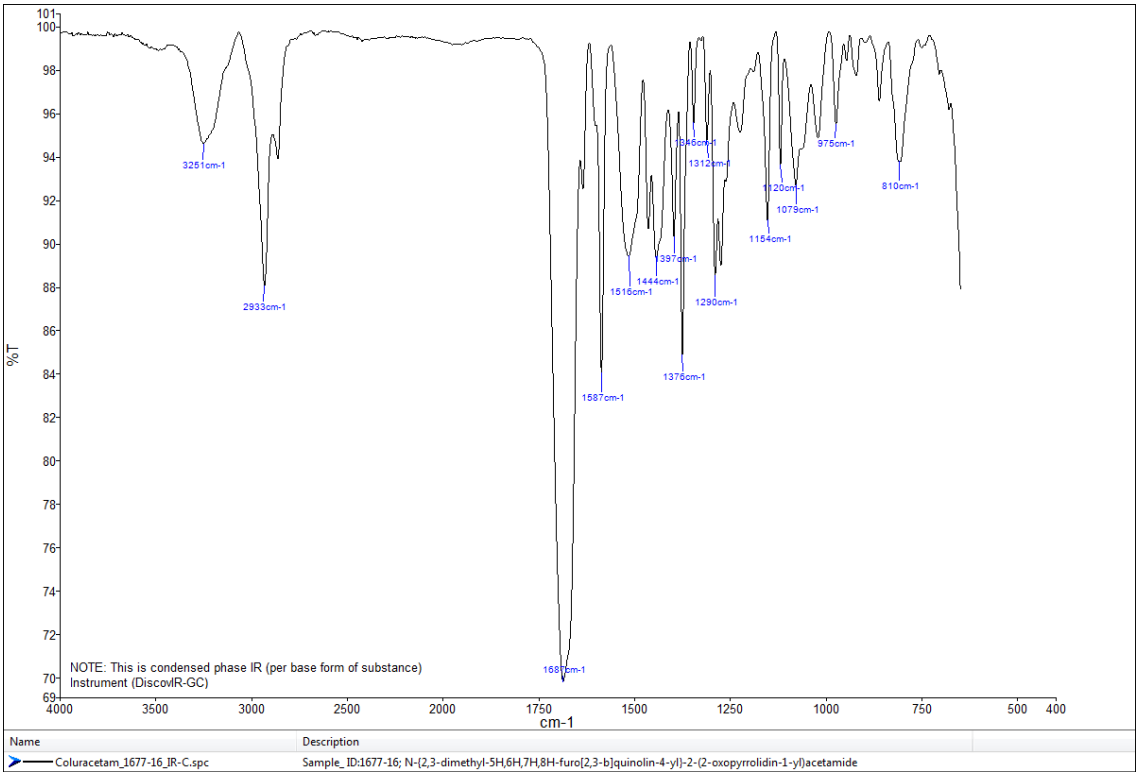
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

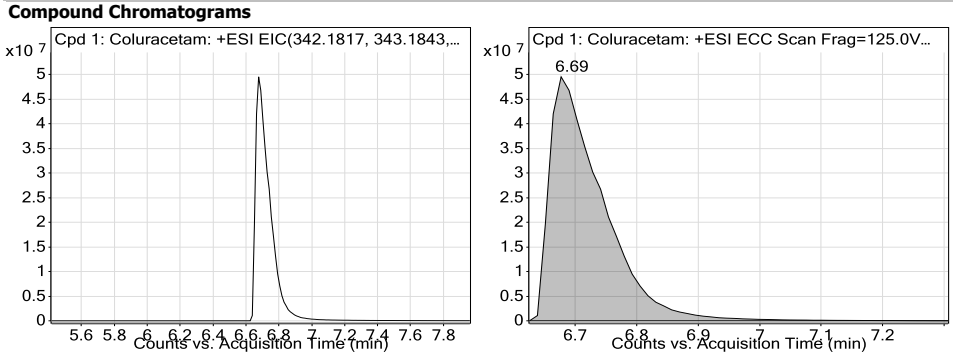
Data File	Coluracetam_1677-16_TOF.d	**Sample Name**	ID_1677-16
Sample Type	Sample	**Position**	P1-D6
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-24_08_2016-XDB-C18-ESI-poz-soft.m	**Acquired Time**	9/26/2016 12:45:34 PM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

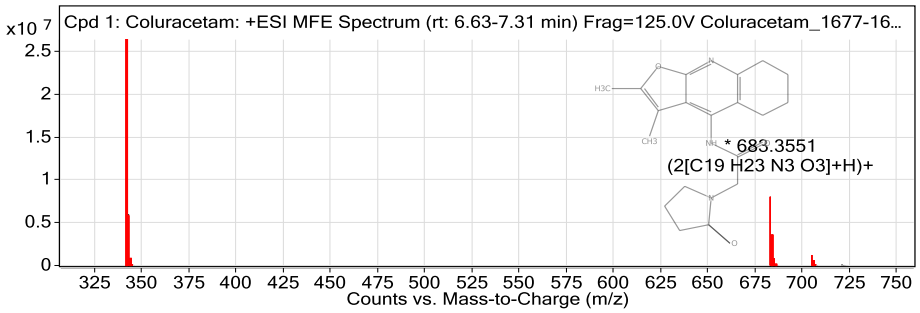
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Coluracetam	Coluracetam	C19 H23 N3 O3	6.69	341.1739

Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Coluracetam	342.1812	6.69	341.1739	6.69	C19 H23 N3 O3	341.1739	0.06

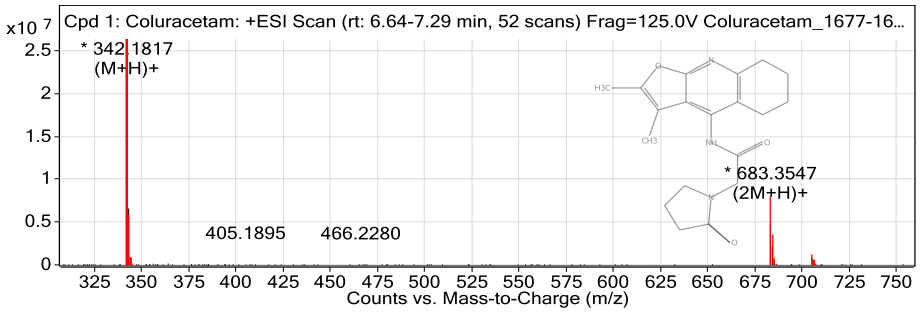
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
342.1812	1	26346106	C19 H23 N3 O3	(M+H)+
343.1843	1	5989017.02	C19 H23 N3 O3	(M+H)+
344.1878	1	700892.38	C19 H23 N3 O3	(M+H)+
683.3551	1	8020158	C19 H23 N3 O3	(2M+H)+
684.3582	1	3616107.43	C19 H23 N3 O3	(2M+H)+
685.3616	1	845033.44	C19 H23 N3 O3	(2M+H)+
686.3641	1	139610.64	C19 H23 N3 O3	(2M+H)+
705.3371	1	1196085.88	C19 H23 N3 O3	(2M+Na)+
706.3406	1	494433.79	C19 H23 N3 O3	(2M+Na)+
721.3122	1	145730.83	C19 H23 N3 O3	(2M+K)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**Coluracetam_1677-16_IC**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**26-sep-2016 / 14:14**	*Run Time:*	**42,00**

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