



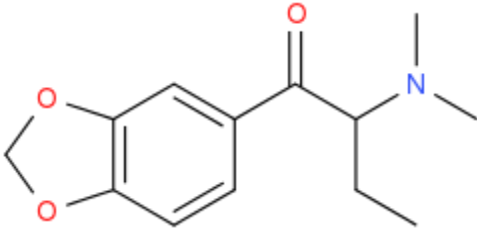
ANALYTICAL REPORT¹

Dibutylone (C₁₃H₁₇N₃O₃)

1-(2H-1,3-benzodioxol-5-yl)-2-(dimethylamino)butan-1-one

Remark – other NPS detected: **none**

Sample ID:	1395-15
Sample description:	powder - granulated - white-off
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	12/12/2015
Date of entry (M/D/Y) into NFL database:	12/18/2015
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	1-(2H-1,3-benzodioxol-5-yl)-2-(dimethylamino)butan-1-one
Other names	methylbutylone, m-butylone, N-methylbutylone, butylone ME, bk-dMBDB
Formula (per base form)	C ₁₃ H ₁₇ N ₃ O ₃
M _w (g/mol)	235,28
Salt form/anions detected	chloride
StdInChIKey	USEBIPUIVPERGC-UHFFFAOYSA-N
Compound Class	Cathinones
Other NPS detected	none
Add.info (purity..)	pure by GC, HPLC-TOF

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

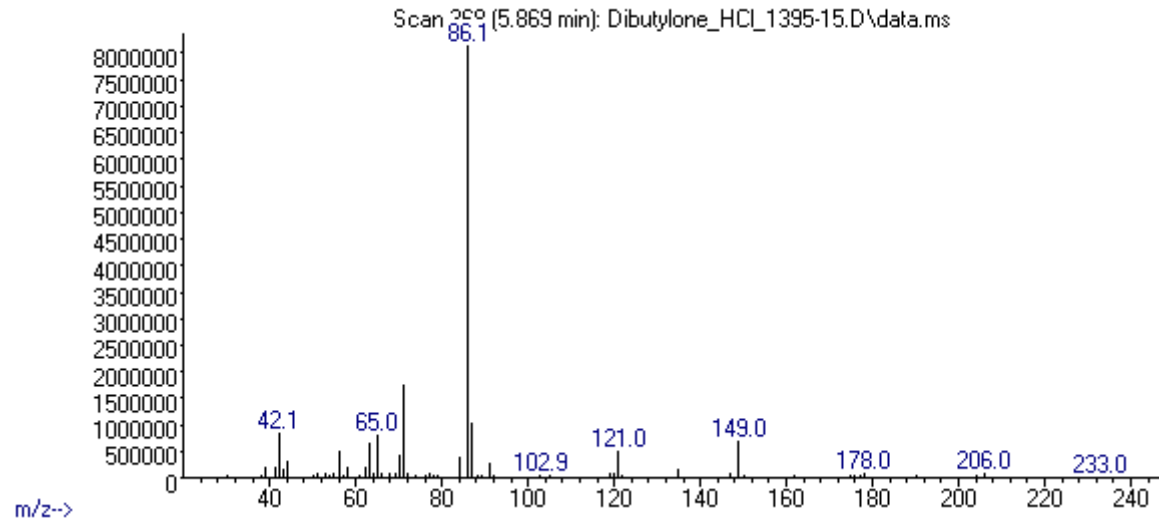
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 5,87 BP(1): 86; BP(2): 71,BP(3) :87,
HPLC-TOF	+	Exact mass (theoretical): 235,1208; measured value Δppm:-0,33; formula: C ₁₃ H ₁₇ NO ₃
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	-	
validation		Ms consistant with publish data, IR-condensed match with the one from Cathinones lib. (DisovIR).
other		

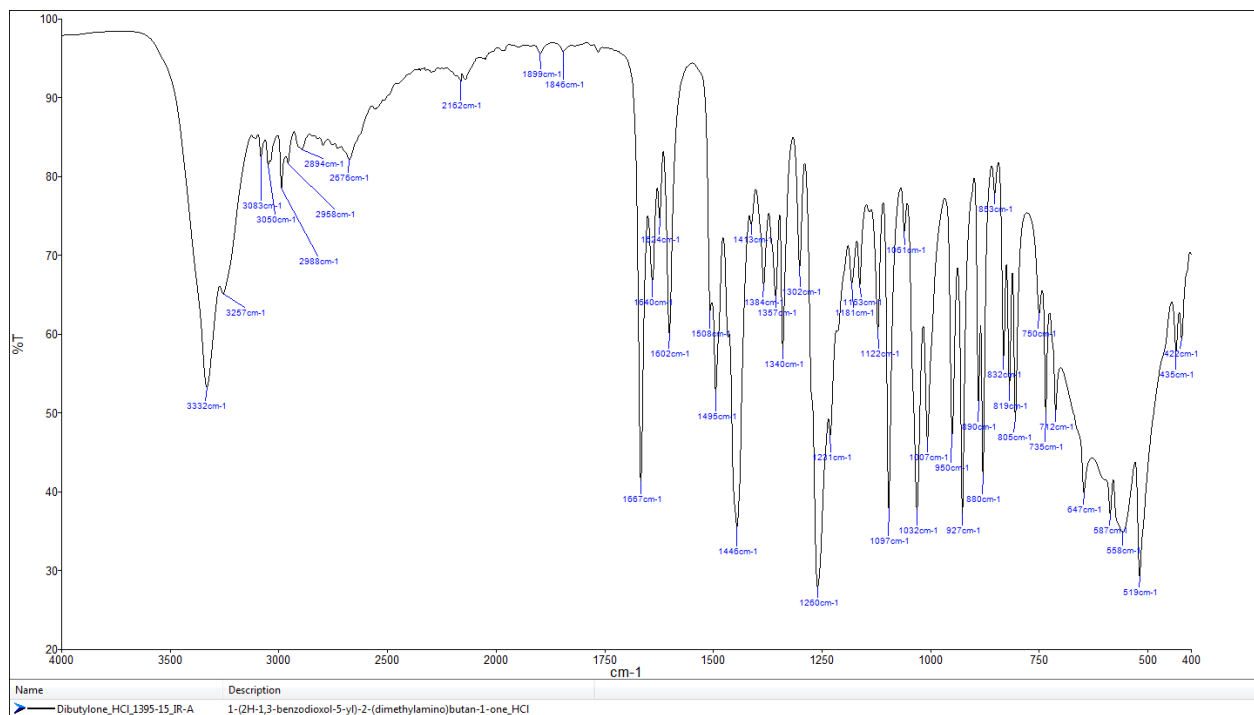
ANALYTICAL RESULTS

MS (EI)

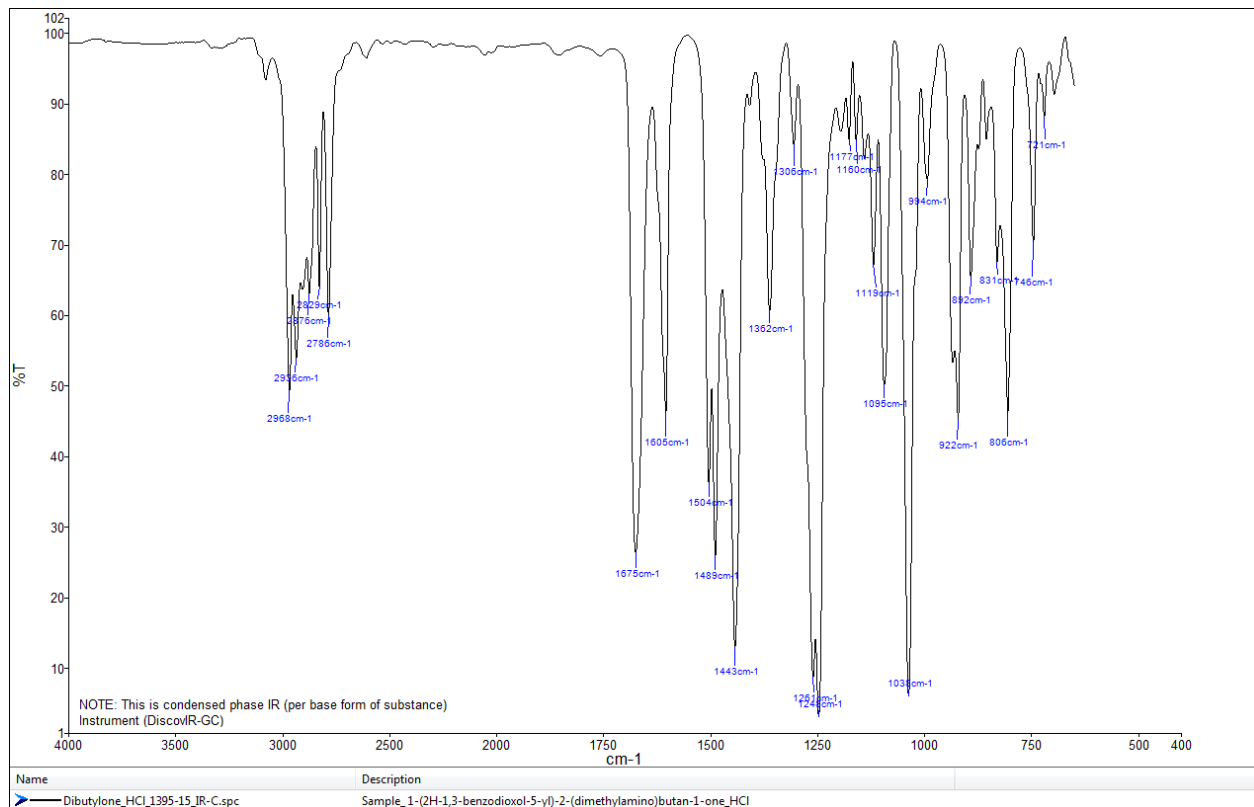
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase - after chromatographic separation)



TOF REPORT

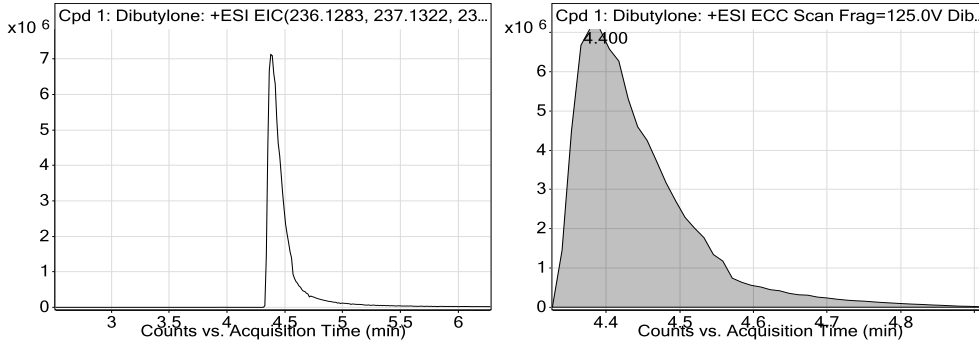
Data File	Dibutylone_1395-15_TOF.d	Sample Name	ID_1395-15
Sample Type	Sample	Position	P1-C3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-17112015-XDB-C18-ESI-poz.m	Acquired Time	12/14/2015 2:10:48 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

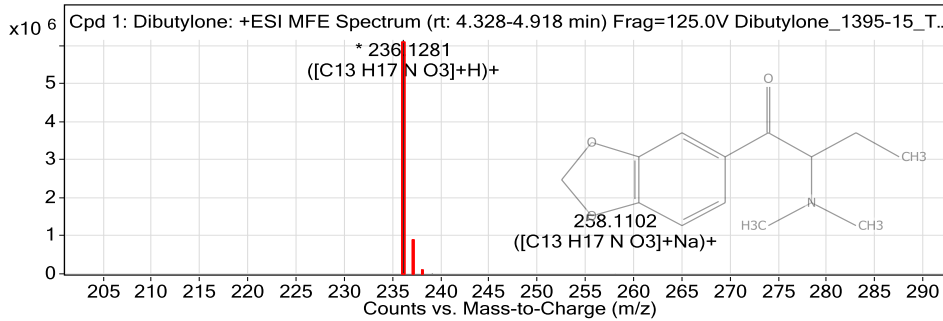
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Dibutylone	Dibutylone	C13 H17 N O3	4.4	235.1209

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Dibutylone	236.1281	4.4	235.1209	4.4	C13 H17 N O3	235.1208	-0.33

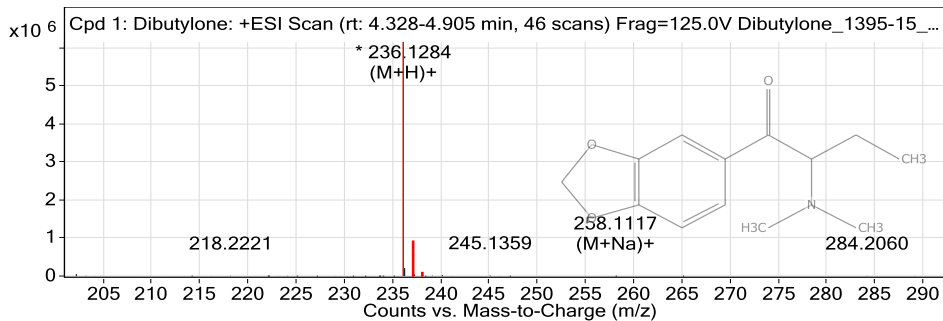
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
236.1281	1	6145532.5	C13 H17 N O3	(M+H)+
237.1323	1	813680.95	C13 H17 N O3	(M+H)+
238.134	1	104177.57	C13 H17 N O3	(M+H)+
239.1368	1	8572.98	C13 H17 N O3	(M+H)+
258.1102	1	1422.2	C13 H17 N O3	(M+Na)+

--- End Of Report ---

Peak Integration Report

Sample Name:	Dibutylone_1395-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	14-dec-2015 / 16:29	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	9,01	Chloride	BMB	38,23	163,86	n.a.
TOTAL:				38,23	163,86	0,00

