



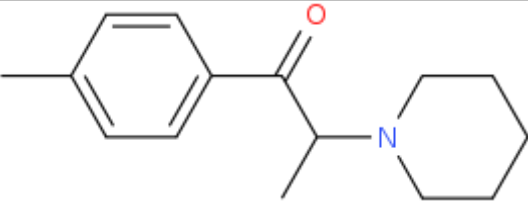
ANALYTICAL REPORT

1-(4-methylphenyl)-2-(piperidin-1-yl)propan-1-one (C₁₅H₂₁NO)

1-(4-methylphenyl)-2-(piperidin-1-yl)propan-1-one

Remark – other NPS detected: **none**

Sample ID:	1753-17
Sample description:	powder - white-off
Sample type:	collected /ENFSI DWG PT 2016 (sample 21)
Date of sample receipt (M/D/Y):	12/17/2016
Date of entry (M/D/Y) into NFL database:	1/10/2017
Report ¹ 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-(4-methylphenyl)-2-(piperidin-1-yl)propan-1-one
Other names	/
Formula (per base form)	C ₁₅ H ₂₁ NO
M _w (g/mol)	231,34
Salt form/anions detected	gallic acid
StdInChIKey (for base form)	DHZOVKWINAURCF-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	Sample is not pure by not pure by GC-MS and TOF. According to the NMR: it contains a large amount of gallic acid (approximate molar ratio of the title compound : gallic acid = 1:1.2), and other impurity

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 7.1 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

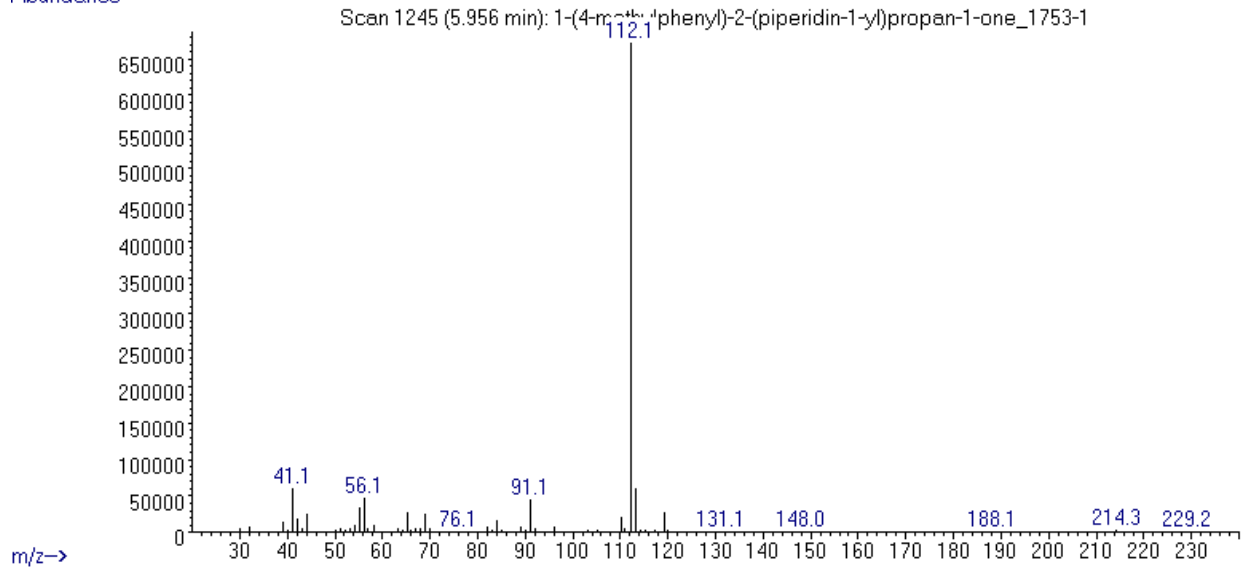
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	soluble

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 5,97 BP(1): 112; BP(2): 113,BP(3) :41,
HPLC-TOF	+	Exact mass (theoretical): 231,1623; measured value Δppm:-0,25; formula:C15H21NO
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

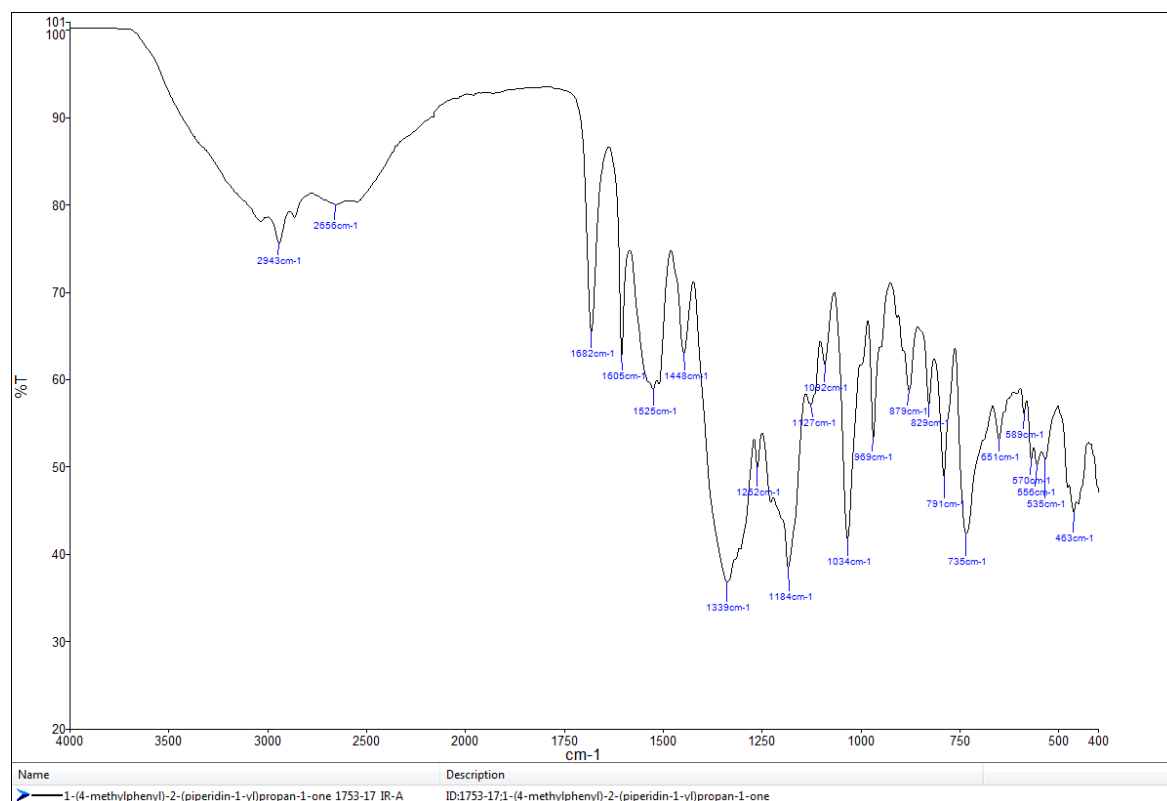
ANALYTICAL RESULTS

MS (EI)

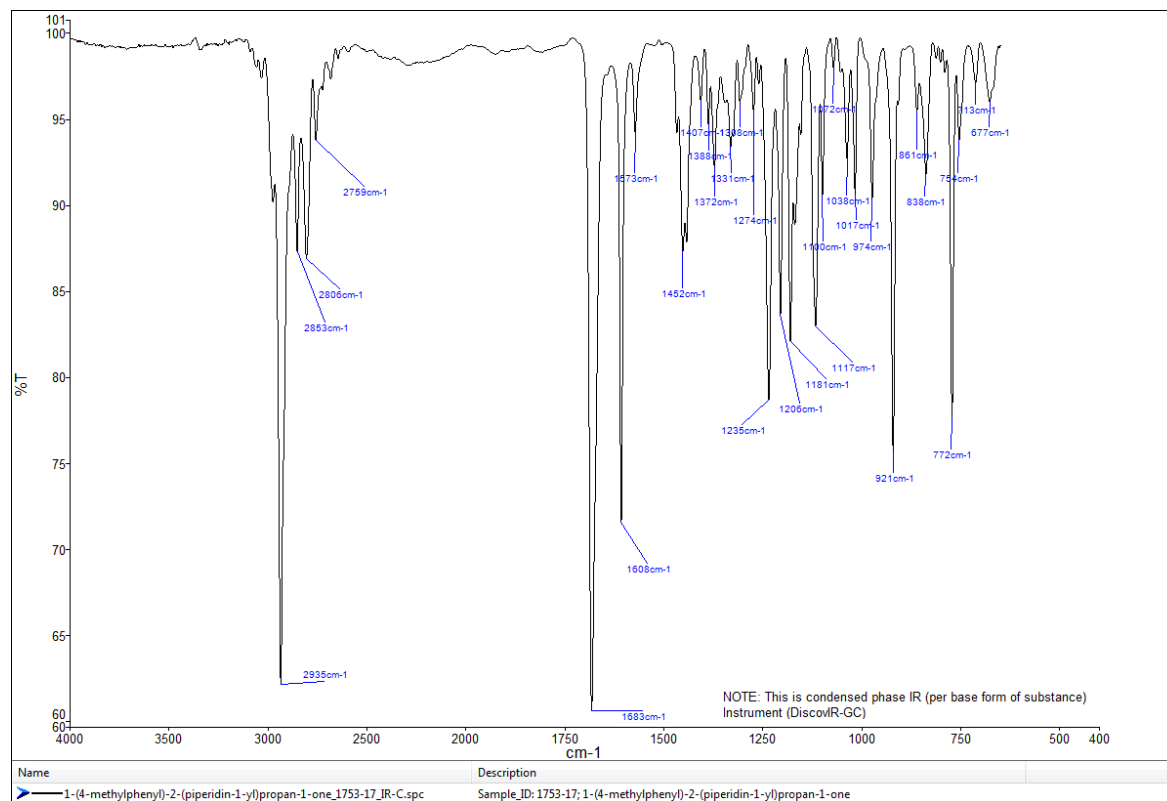
Abundance



FTIR-ATR - direct measurement (*sample as received* substantial amount of gallic acid)



IR (condensed phase – after chromatographic separation)



TOF REPORT

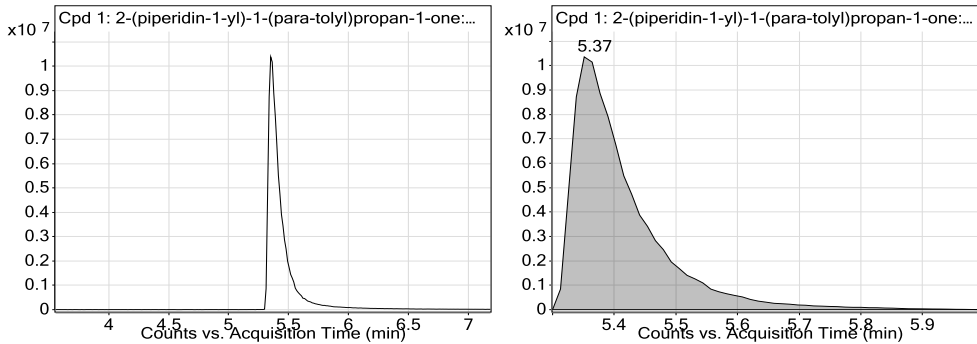
Data File	ENFSI_DWG-PT_2016_vz_21.d	Sample Name	ENFSI-DWG-PT-2016_vz-21
Sample Type	Sample	Position	P1-F8
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-20_12_2016-XDB-C18-ESI-poz-soft.m	Acquired Time	12/21/2016 10:18:09 AM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

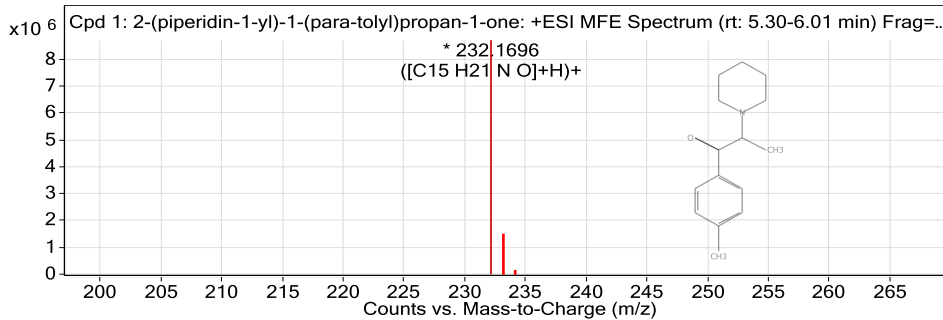
Label	Compound Name	Obs. RT	Obs. Mass
Cpd 1: 2-(piperidin-1-yl)-1-(para-tolyl)propan-1-one	2-(piperidin-1-yl)-1-(para-tolyl)propan-1-one	5.37	231.1624

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
2-(piperidin-1-yl)-1-(para-tolyl)propan-1-one	232.1696	5.37	231.1624	5.37	C15 H21 N O	231.1623	-0.25

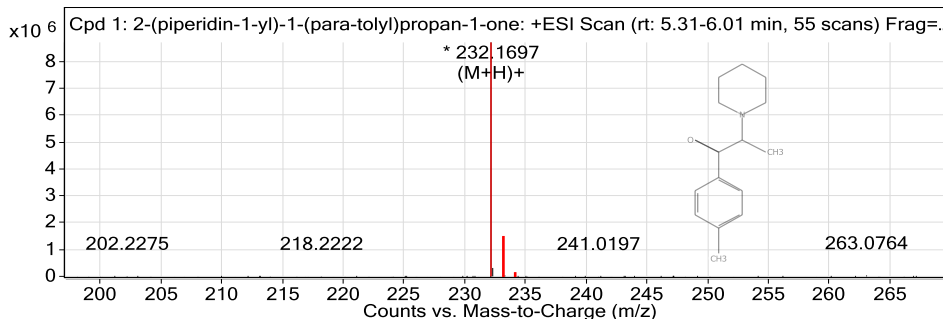
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



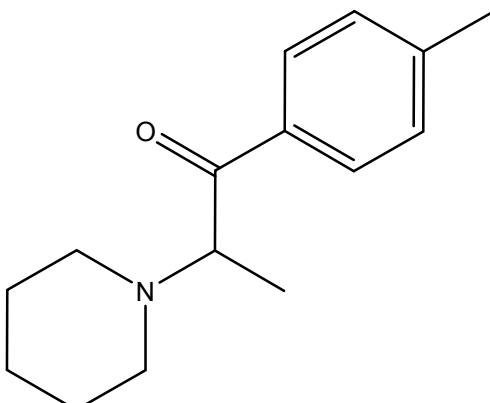
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
232.1696	1	8711914	C15 H21 N O	(M+H)+
233.1732	1	1402794	C15 H21 N O	(M+H)+
234.1761	1	118778.85	C15 H21 N O	(M+H)+
235.1791	1	7416.48	C15 H21 N O	(M+H)+

--- End Of Report ---



REPORT

Sample ID:	DWG-PT2016 (vz21)
Our notebook code:	DWG-PT2016 (vz21)
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- <i>d</i> ₆
NMR experiments:	¹ H, ¹³ C, ¹ H- ¹ H <i>gs</i> -COSY, ¹ H- ¹³ C <i>gs</i> -HSQC, ¹ H- ¹³ C <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	2-(piperidin-1-yl)-1-(<i>para</i> -tolyl)propan-1-one
Comments:	<p>- Structure elucidation based on 1D and 2D NMR spectra</p> <p>- Sample is not pure according to the NMR: it contains a large amount of gallic acid (approximate molar ratio of the title compound : gallic acid = 1:1.2 determined by the ¹H NMR signal at δ 6.92 ppm; signals for gallic acid observed in ¹³C NMR are at δ 168.2, 145.9, 138.4, 121.2 and 109.2). The sample contains also at least one other impurity (minor signals observed in ¹H and ¹³C NMR).</p> <p>NB. Integrals and peak picking in the attached NMR spectra is shown for the title compound only.</p>
Supporting information:	Copies of ¹ H and ¹³ C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	January 5, 2017

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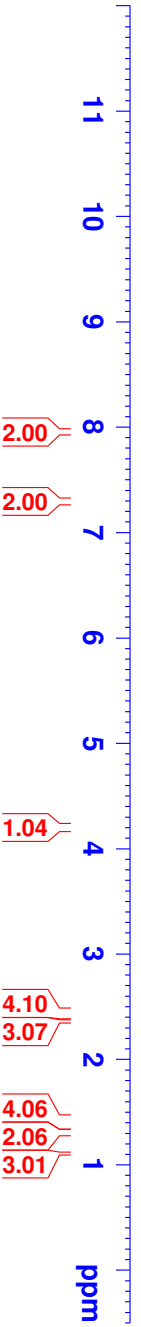


Current Data Parameters
 NAME DMG2016vz21
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161229
 Time 8.39
 INSTRUM spect
 PROBHD 5 mm PABBO B-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 71.8
 DW 50.000 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SF01 500.1330885 MHz
 NUC1 1H
 P1 8.60 usec
 PLW1 26.00000000 W

F2 - Processing parameters
 SI 65536
 SF 500.1300007 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



DMG2016vz21



Current Data Parameters
 NAME DMG2016vz21
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161229
 Time 9.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1299
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 298.3 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 8.70 usec
 PLW1 122.00000000 W

==== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRGf2 waltz16
 PCPD2 80.00 usec
 PLW2 26.00000000 W
 PLW12 0.30046001 W
 PLW13 0.15113001 W

F2 - Processing parameters
 SI 32768
 SF 125.7577885 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40