



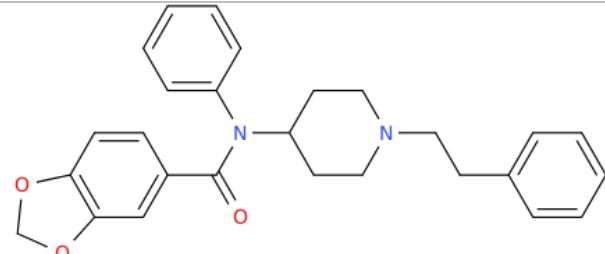
ANALYTICAL REPORT

Benzodioxole-F (C₂₇H₂₈N₂O₃)

N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]-2H-1,3-benzodioxole-5-carboxamide

Remark – other NPS detected: **none**

Sample ID:	1672-16
Sample description:	powder - beige
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	9/8/2016
Date of entry (M/D/Y) into NFL database:	3/5/2017
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	N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]-2H-1,3-benzodioxole-5-carboxamide
Other names	N-(1-phenethylpiperidin-4-yl)-N-phenylbenzo[d][1,3]dioxole-5-carboxamide, Benzodioxole-fentanyl, BZD-F
Formula (per base form)	C ₂₇ H ₂₈ N ₂ O ₃
M _w (g/mol)	428,53
Salt form/anions detected	HCl, traces of sulphate ions
StdInChIKey (for base form)	ZFAAZMIOHJNKGD-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	pure by HPLC-TOF, NMR, by GC-MS traces of N-phenyl-1-(2-phenylethyl)piperidin-4-amine detected (possibly GC degradation product)

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

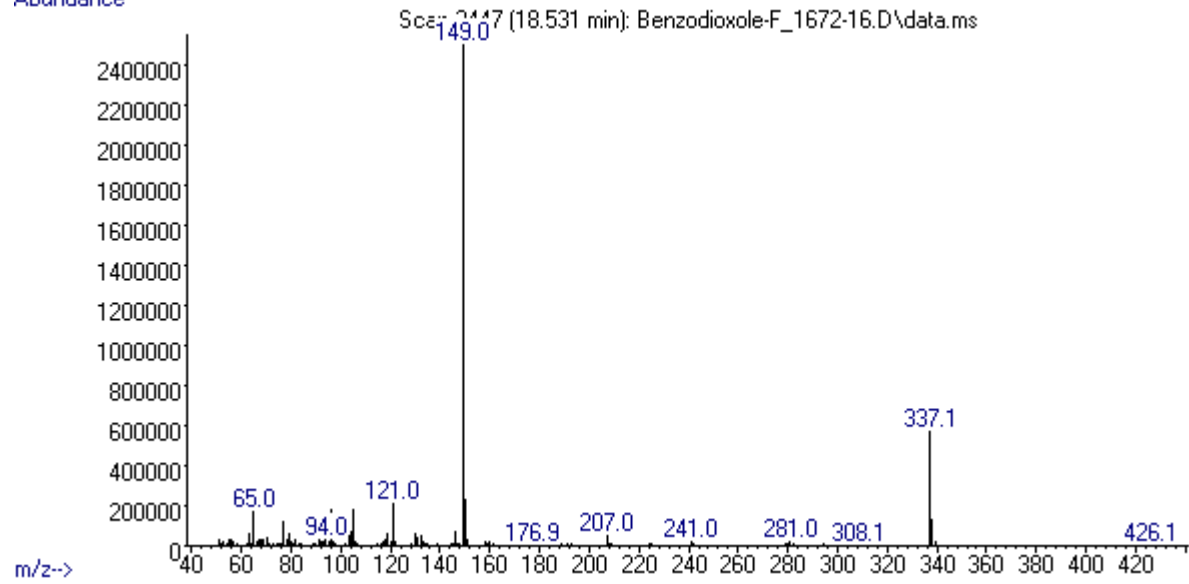
Solubility in	result/remark
CH ₂ Cl ₂	soluble
MeOH	soluble
H ₂ O	partially

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 18,53 BP(1): 149; BP(2): 337,BP(3) :150,
HPLC-TOF	+	Exact mass (theoretical): 428,21; measured value Δppm:-0,02; formula:C27H28N2O3
FTIR-ATR	+	direct measurement (sample as received)
FTIR (condensed phase) always as base form	+	
IC (anions)	+	
NMR (in FKKT)	+	
validation		
other		

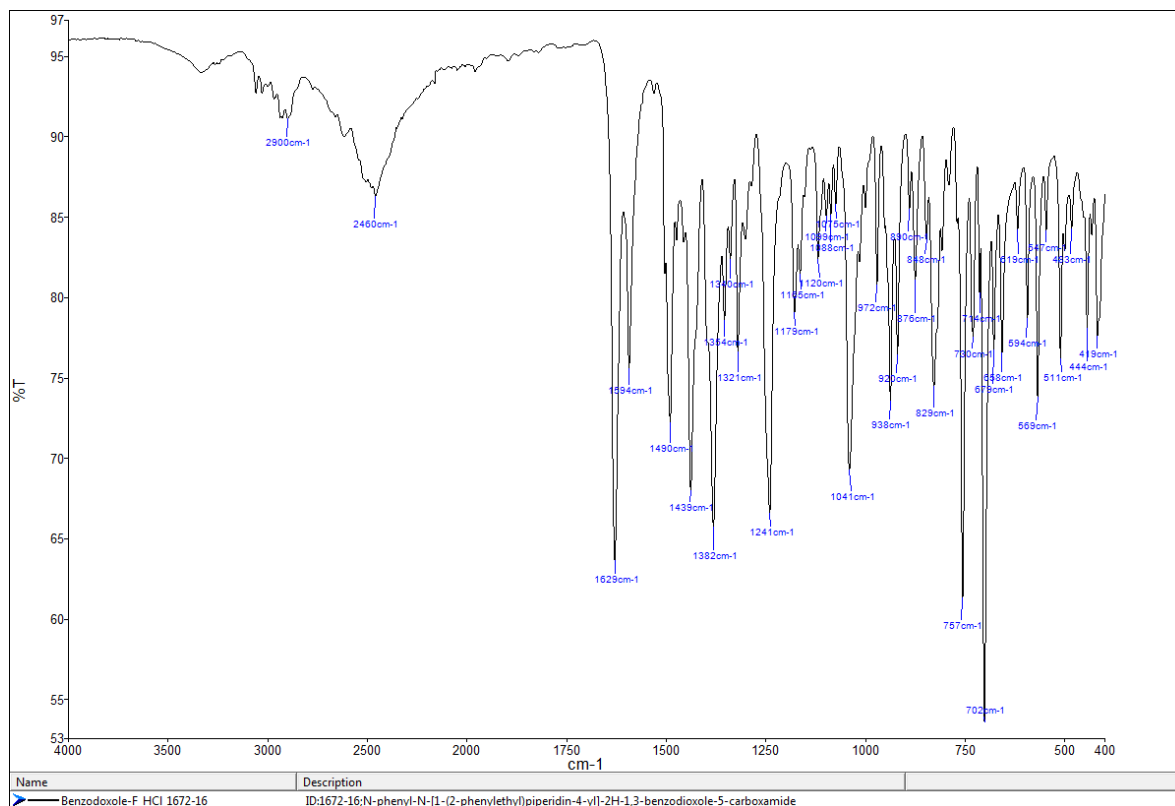
ANALYTICAL RESULTS

MS (EI)

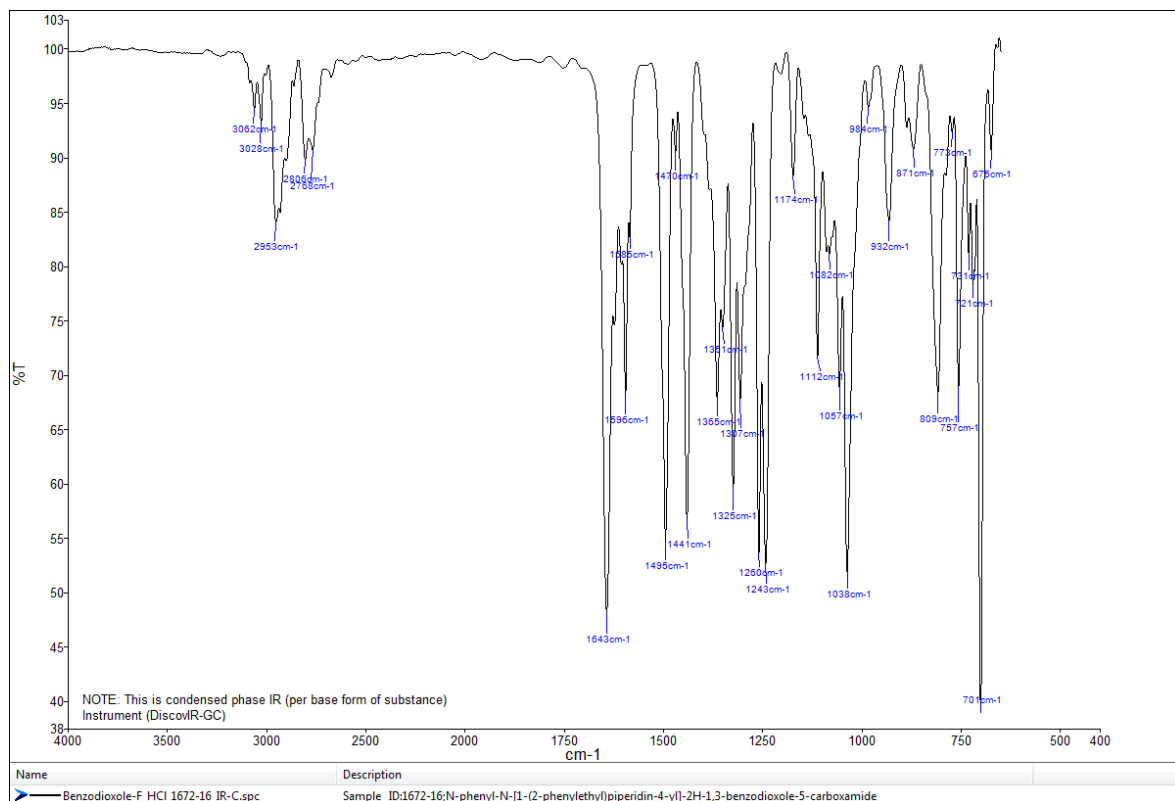
Abundance



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

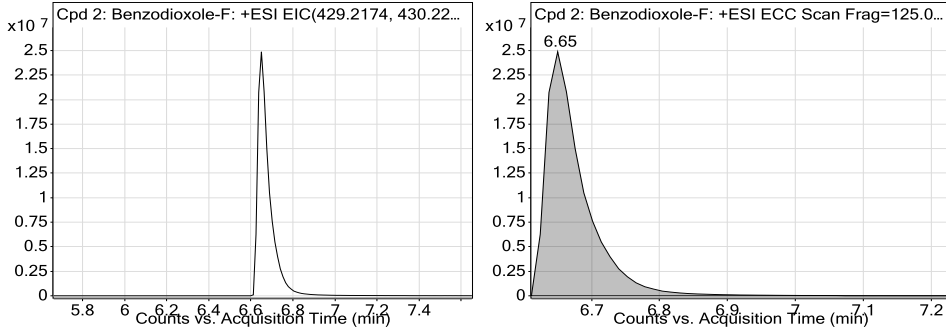
Data File	Benzodioxole_F_1672-16_TOF.d	Sample Name	ID_1672-16
Sample Type	Sample	Position	P1-C2
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	general-24_08_2016-XDB-C18-ESI-poz-soft.m	Acquired Time	9/14/2016 1:41:54 PM
IRM Calibration Status	Success	DA Method	Drugs_NFL.m
Comment	extract in MeOH		

Compound Table

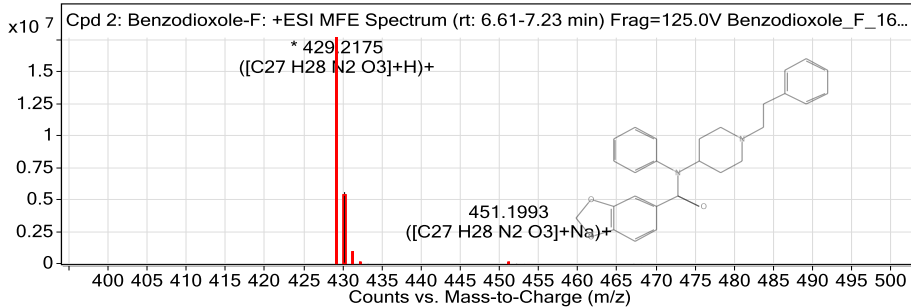
Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 2: Benzodioxole-F	Benzodioxole-F	C27 H28 N2 O3	6.65	428.21

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
Benzodioxole-F	429.2175	6.65	428.21	6.65	C27 H28 N2 O3	428.21	-0.02

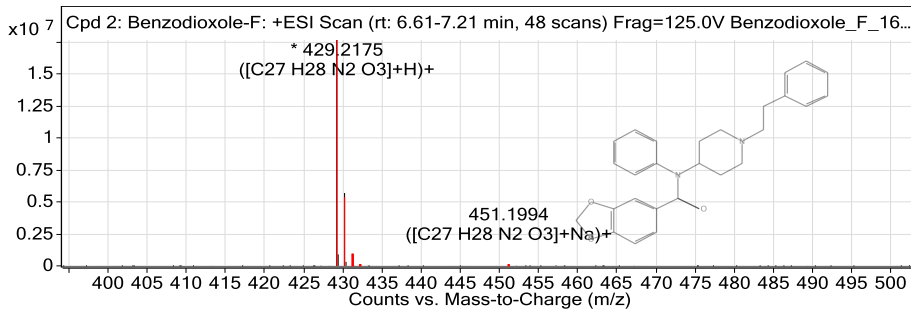
Compound Chromatograms



MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

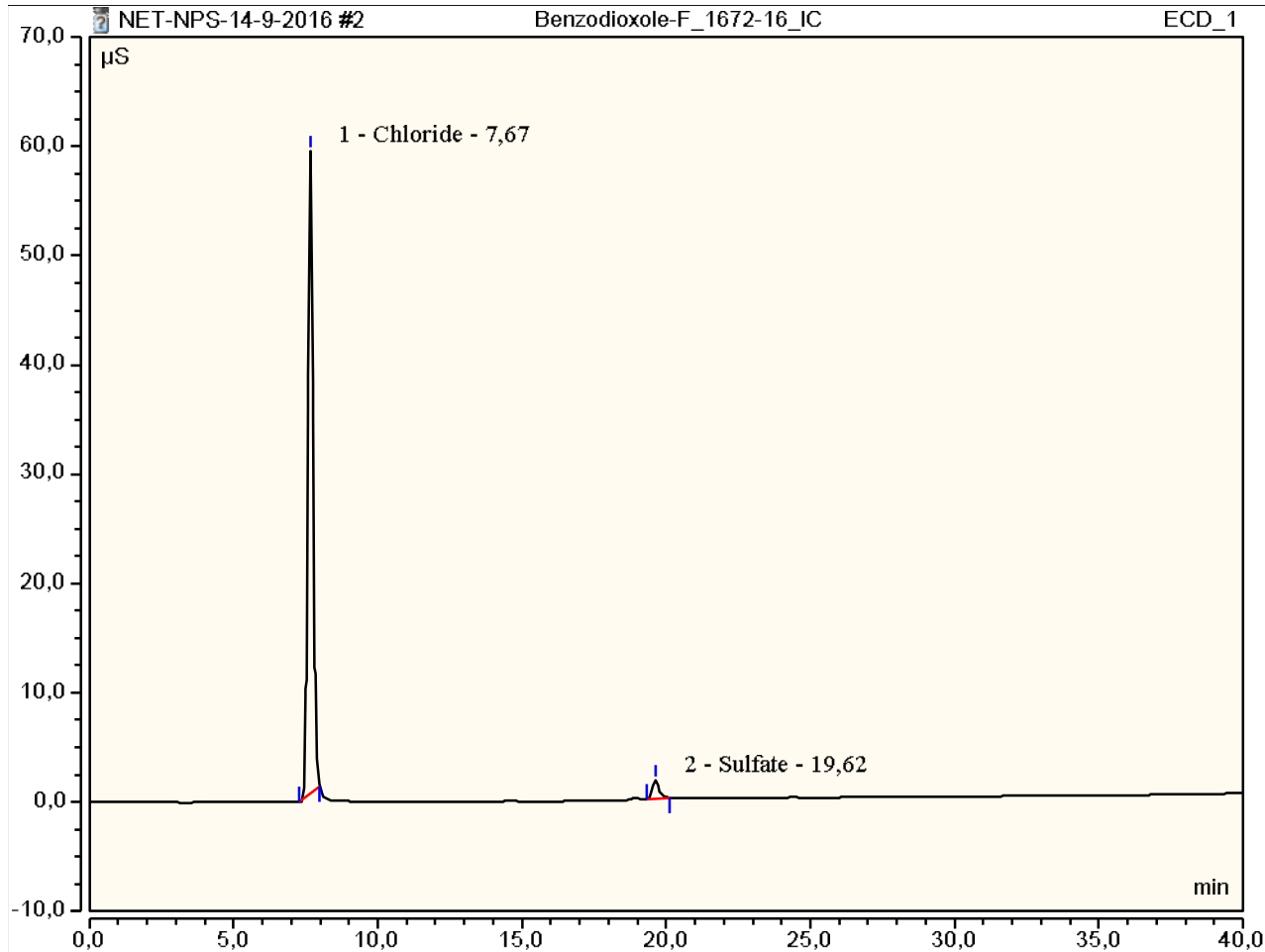
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
429.2175	1	17657226	C27 H28 N2 O3	(M+H)+
430.221	1	5581697.28	C27 H28 N2 O3	(M+H)+
431.2244	1	844798.19	C27 H28 N2 O3	(M+H)+
432.2268	1	95323.18	C27 H28 N2 O3	(M+H)+
433.2293	1	8688.93	C27 H28 N2 O3	(M+H)+
451.1993	1	47422.28	C27 H28 N2 O3	(M+Na)+
452.2023	1	14179.09	C27 H28 N2 O3	(M+Na)+
453.205	1	2470.5	C27 H28 N2 O3	(M+Na)+
467.173	1	8055.45	C27 H28 N2 O3	(M+K)+
468.176	1	2664.88	C27 H28 N2 O3	(M+K)+

--- End Of Report ---

Peak Integration Report

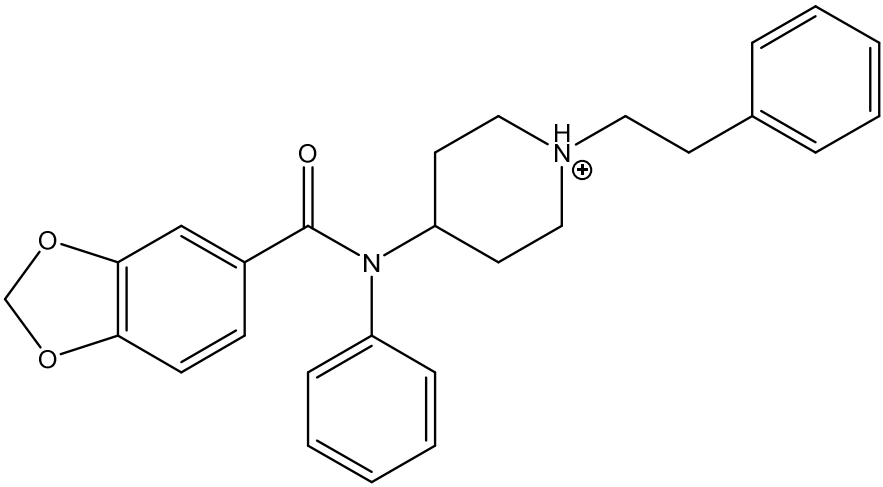
Sample Name:	Benzodioxole-F_1672-16_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	14-sep-2016 / 13:44	Run Time:	40,69

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}\cdot\text{min}$	Height μS	Amount mg/L
1,00	7,67	Chloride	BMB	12,03	58,81	n.a.
2,00	19,62	Sulfate	BMB	0,44	1,67	n.a.
TOTAL:				12,48	60,48	0,00





REPORT

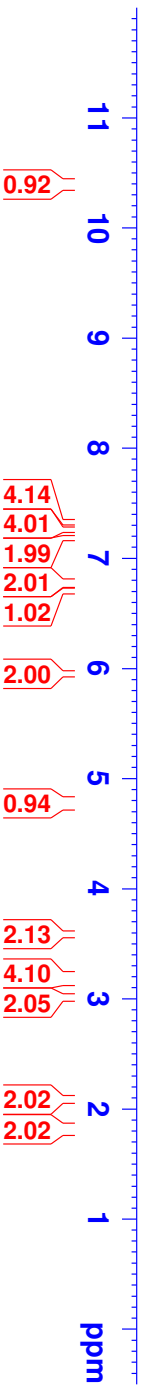
Sample ID:	1672-16
Our notebook code:	P-1672-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO- d_6
NMR experiments:	^1H , ^{13}C , ^1H - ^1H <i>gs</i> -COSY, ^1H - ^{13}C <i>gs</i> -HSQC, ^1H - ^{13}C <i>gs</i> -HMBC, ^1H - ^{15}N <i>gs</i> -HMBC.
Proposed structure:	
Chemical name:	1-phenethyl-4-(<i>N</i> -phenylbenzo[<i>d</i>][1,3]dioxole-5-carboxamido)piperidin-1-ium cation
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is pure according to the NMR.
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	February 22, 2017



Current Data Parameters
 NAME P-1672-16
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170210
 Time 9.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 114
 DW 50.000 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 500.1330885 MHz
 NUC1 1H
 P1 8.60 usec
 PLW1 26.00000000 W
 F2 - Processing parameters
 SI 65536
 SF 500.1300028 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME P-1672-16
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170210
 Time 11.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 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 298.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 125.7703637 MHz
 NUC1 13C
 P1 8.70 usec
 P1M1 122.00000000 W

==== CHANNEL f2 =====
 SFO2 500.1320005 MHz
 NUC2 1H
 CPDPRG12 waltz16
 PCPD2 80.00 usec
 P1M2 26.00000000 W
 P1M12 0.30046001 W
 P1M13 0.151113001 W

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