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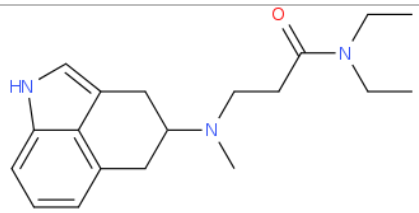
ANALYTICAL REPORT

NDTDI (C19H27N3O)

3-({2-azatricyclo[6.3.1.0^{4,12}]dodeca-1(11),3,8(12),9-tetraen-6-yl}(methyl)amino)-N,N-diethylpropanamide

Remark – other NPS detected: none

Sample ID:	1737-16
Sample description:	powder - grey
Sample type:	test purchase /RESPONSE -purchasing
Date of sample receipt (M/D/Y):	11/28/2016
Date of entry (M/D/Y) into NFL database:	2/6/2017
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	3-({2-azatricyclo[6.3.1.0^{4,12}]dodeca-1(11),3,8(12),9-tetraen-6-yl}(methyl)amino)-N,N-diethylpropanamide
Other names	N,N-diethyl-3-(methyl(1,3,4,5-tetrahydrobenzo[cd]indol-4-yl)amino)propanamide
Formula (per base form)	C19H27N3O
M_w (g/mol)	313,45
Salt form/anions detected	succinate
StdInChIKey (for baze form)	JECGWOMOCPQHDH-UHFFFAOYSA-N
Other NPS detected	none
Additional info (purity..)	Sample is not pure by GC-MS . According to the NMR: it contains amount of succinic acid (approximate molar ratio of the title compound : succinic acid = 1:1), and other impurities

[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 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 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

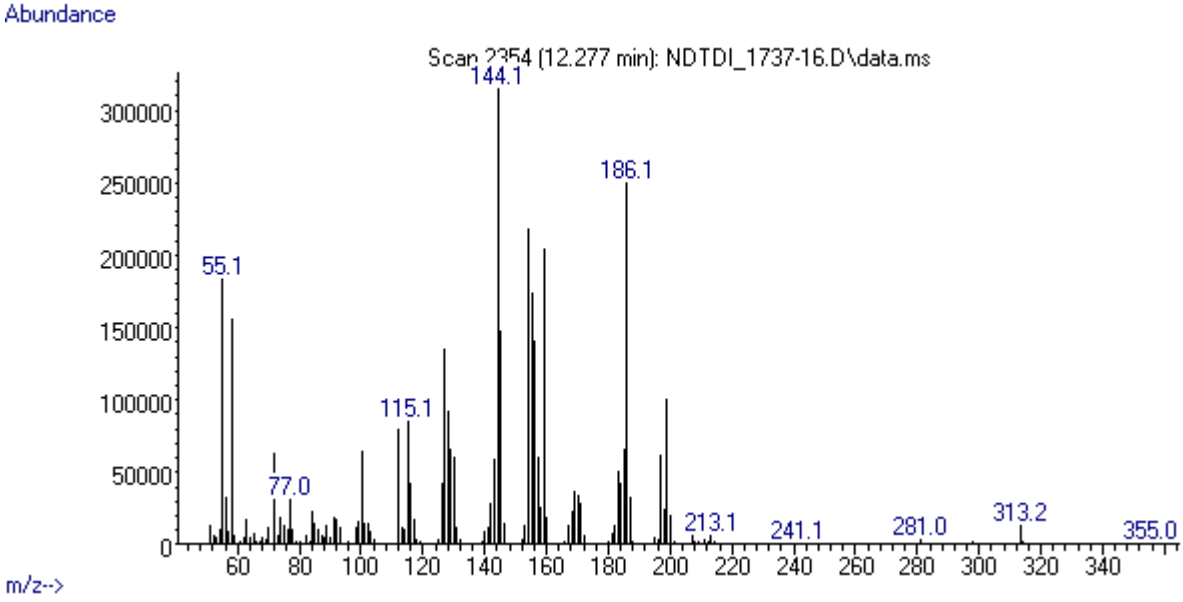
Supporting information

Solubility in	result/remark
CH_2Cl_2	partially
MeOH	soluble
H_2O	soluble

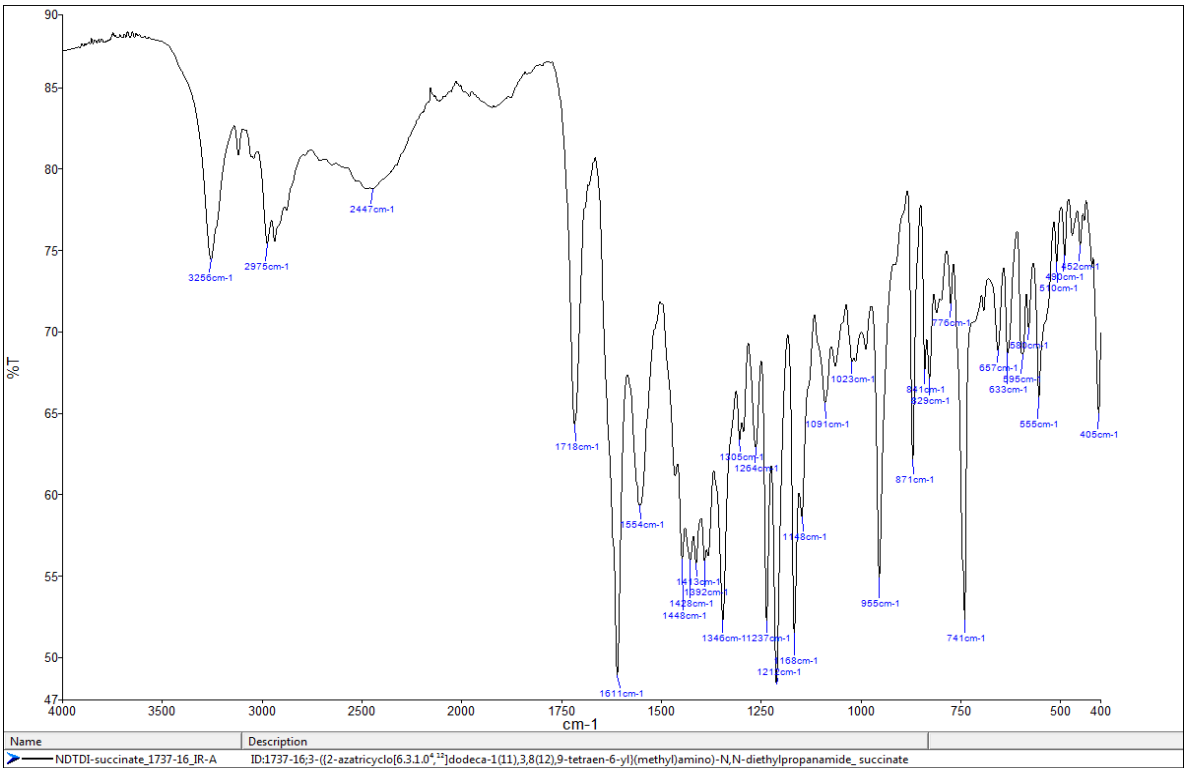
Analytical technique:	applied	remarks
GC-MS (EI ionization)		NFL GC-RT (min): 12,28 BP(1): 144; BP(2): 186,BP(3) :154,
HPLC-TOF		Exact mass (theoretical): 313,2154; measured value Δppm:0,02; formula:C19H27N3O
FTIR-ATR		direct measurement (sample as received)
FTIR (condensed phase) always as base form		
IC (anions)		
NMR (in FKKT)		
validation		
other		

ANALYTICAL RESULTS

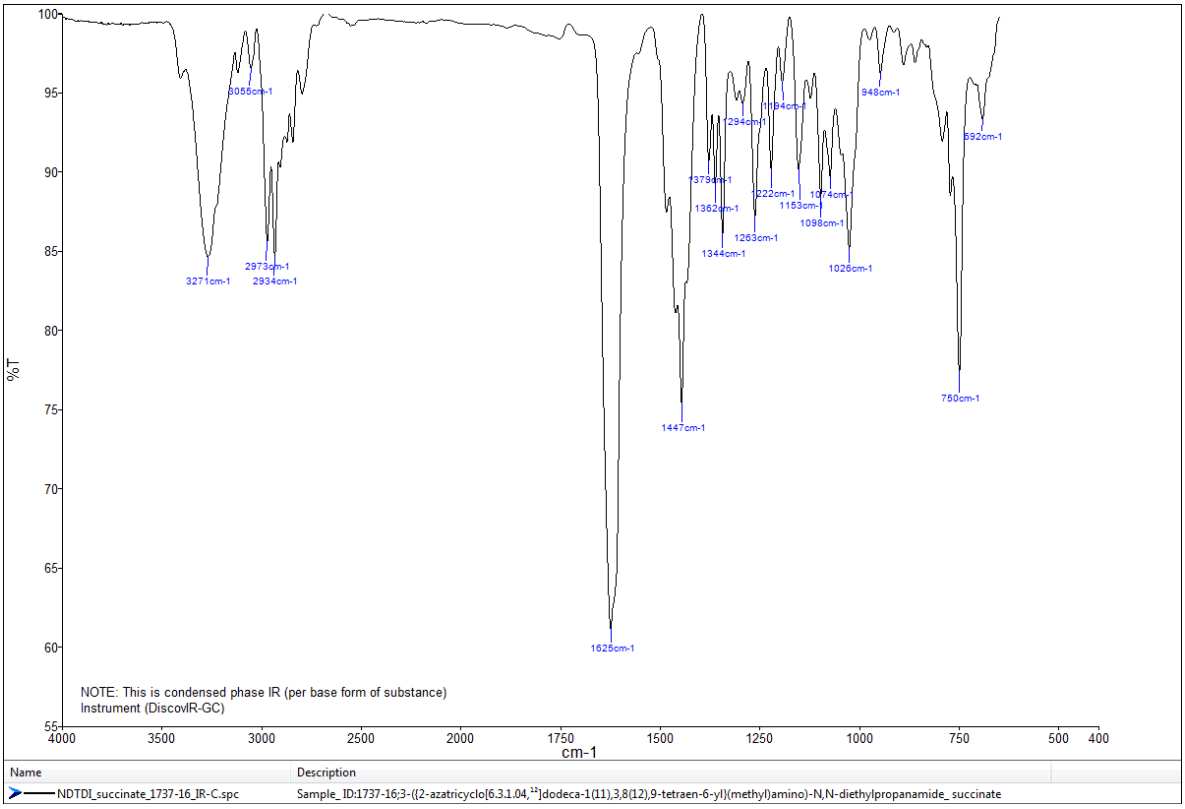
MS (EI)



FTIR-ATR - direct measurement (sample as received)



IR (condensed phase – after chromatographic separation)



TOF REPORT

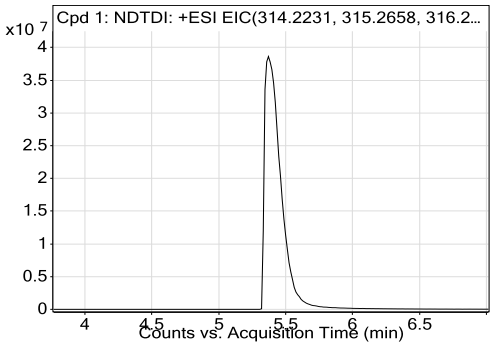
Data File	NDTDI_1737-16.d	**Sample Name**	ID_1737-16
Sample Type	Sample	**Position**	P1-A2
Instrument Name	6230B TOF LC-MS	**User Name**	TG
Acq Method	general-10_10_2016-XDB-C18-ESI-poz-soft.m	**Acquired Time**	12/5/2016 10:27:26 AM
IRM Calibration Status	Success	**DA Method**	Drugs_NFL.m
Comment	extract in MeOH		

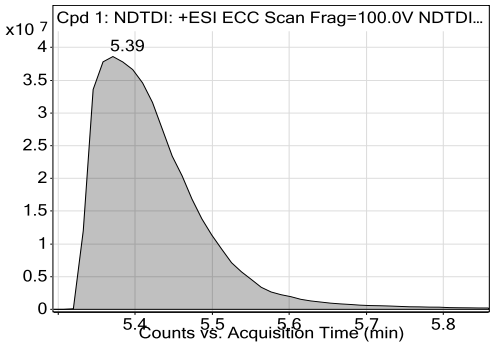
Compound Table

Label	Compound Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: NDTDI	NDTDI	C19 H27 N3 O	5.39	313.2154

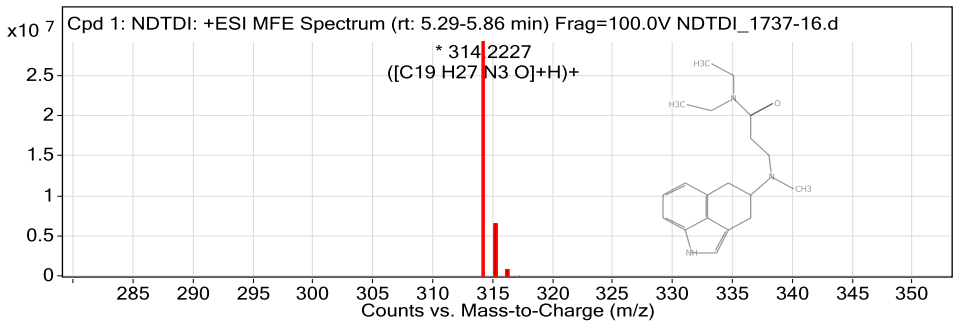
Name	*Obs. m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)
NDTDI	314.2227	5.39	313.2154	5.39	C19 H27 N3 O	313.2154	0.02

Compound Chromatograms

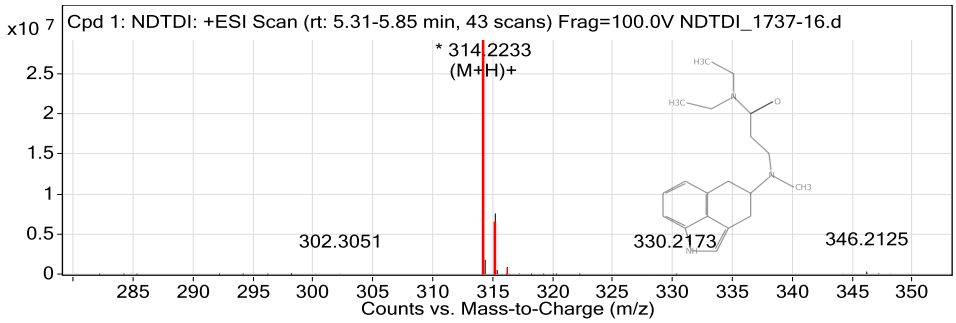




MFE MS Zoomed Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

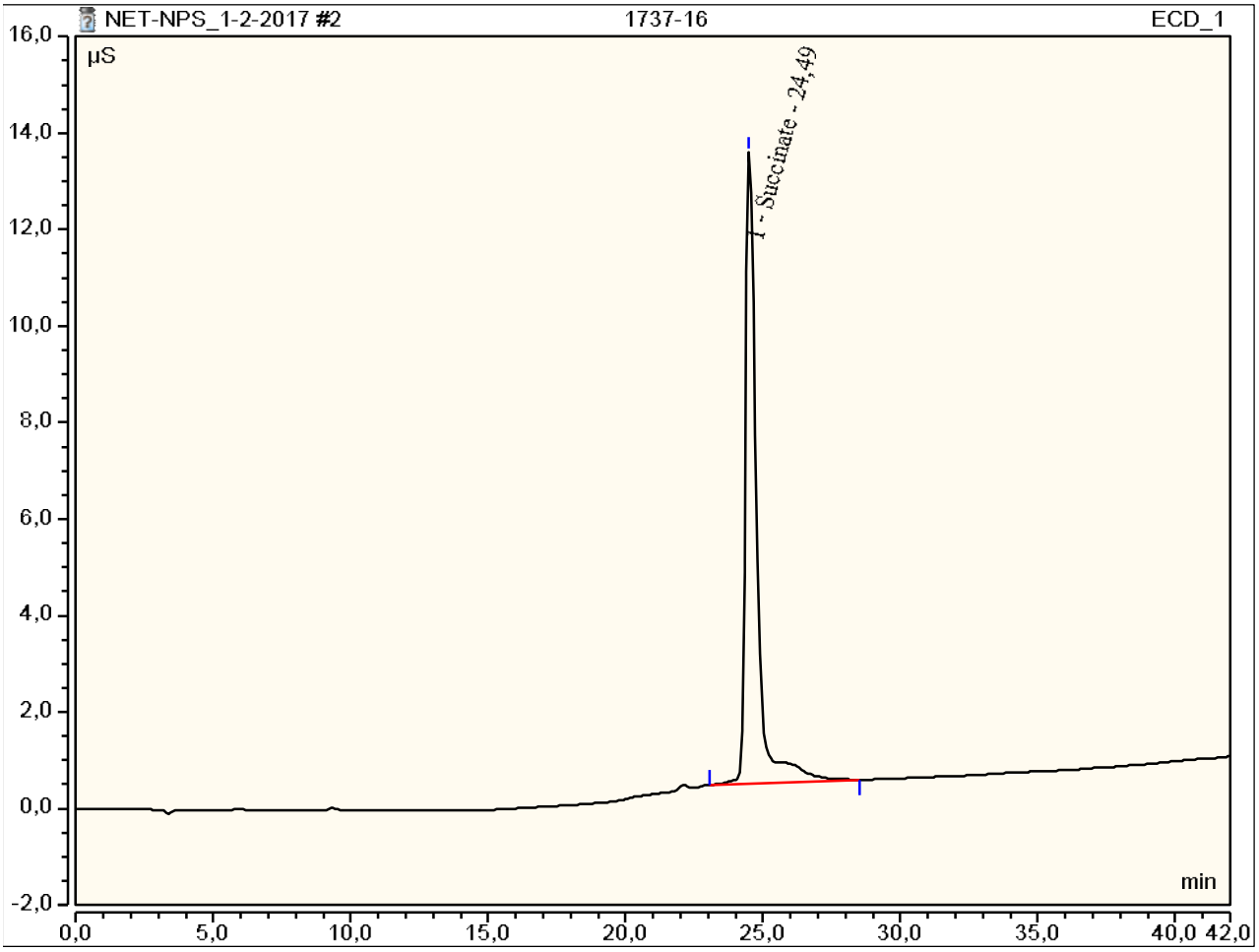
Obs. m/z	Charge	Abund	Formula	Ion/Isotope
314.2227	1	29194660	C19 H27 N3 O	(M+H)+
315.2258	1	6549451	C19 H27 N3 O	(M+H)+
316.2293	1	681251.17	C19 H27 N3 O	(M+H)+
317.2319	1	48438.85	C19 H27 N3 O	(M+H)+
318.2297	1	9278.5	C19 H27 N3 O	(M+H)+

--- End Of Report ---

Peak Integration Report

Sample Name:	**1737-16**	*Inj. Vol.:*	**25,00**
Injection Type:	**Unknown**	*Dilution Factor:*	**1,0000**
Program:	**ANIONI**	*Operator:*	**kemija**
Inj. Date / Time:	**01-feb-2017 / 08:59**	*Run Time:*	**42,00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount mg/L
1,00	24,49	**Succinate**	BMB	6,26	13,08	n.a.
		TOTAL:		6,26	13,08	0,00



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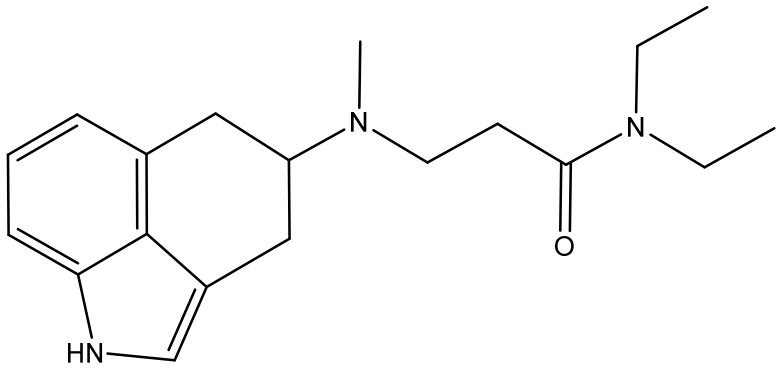
Co-funded by the Prevention of and Fight against Crime Programme of the European Union

REPORT

Sample ID:	**1737-16**
Our notebook code:	P-1737-16
NMR sample preparation:	15 mg dissolved in 0.7 mL DMSO-d_6
NMR experiments:	^{1}H, ^{13}C, ^{1}H–^{1}H *gs*-COSY, ^{1}H–^{13}C *gs*-HSQC, ^{1}H–^{13}C *gs*-HMBC, ^{1}H–^{15}N *gs*-HMBC.
Proposed structure:	
Chemical name:	*N*,*N*-diethyl-3-(methyl(1,3,4,5-tetrahydrobenzo[*cd*]indol-4-yl)amino)propanamide
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Sample is not pure according to the NMR; it contains succinic acid (in approximate molar ratio 1 : 1 with the title compound) as evident by the signal at δ 2.40 ppm in ^{1}H NMR spectrum and signals at δ 29.7 and 174.3 ppm in ^{13}C NMR. There are also some other impurities in the sample as can be observed from the additional minor signals present in NMR spectra.
Supporting information:	Copies of ^{1}H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	February 1, 2017

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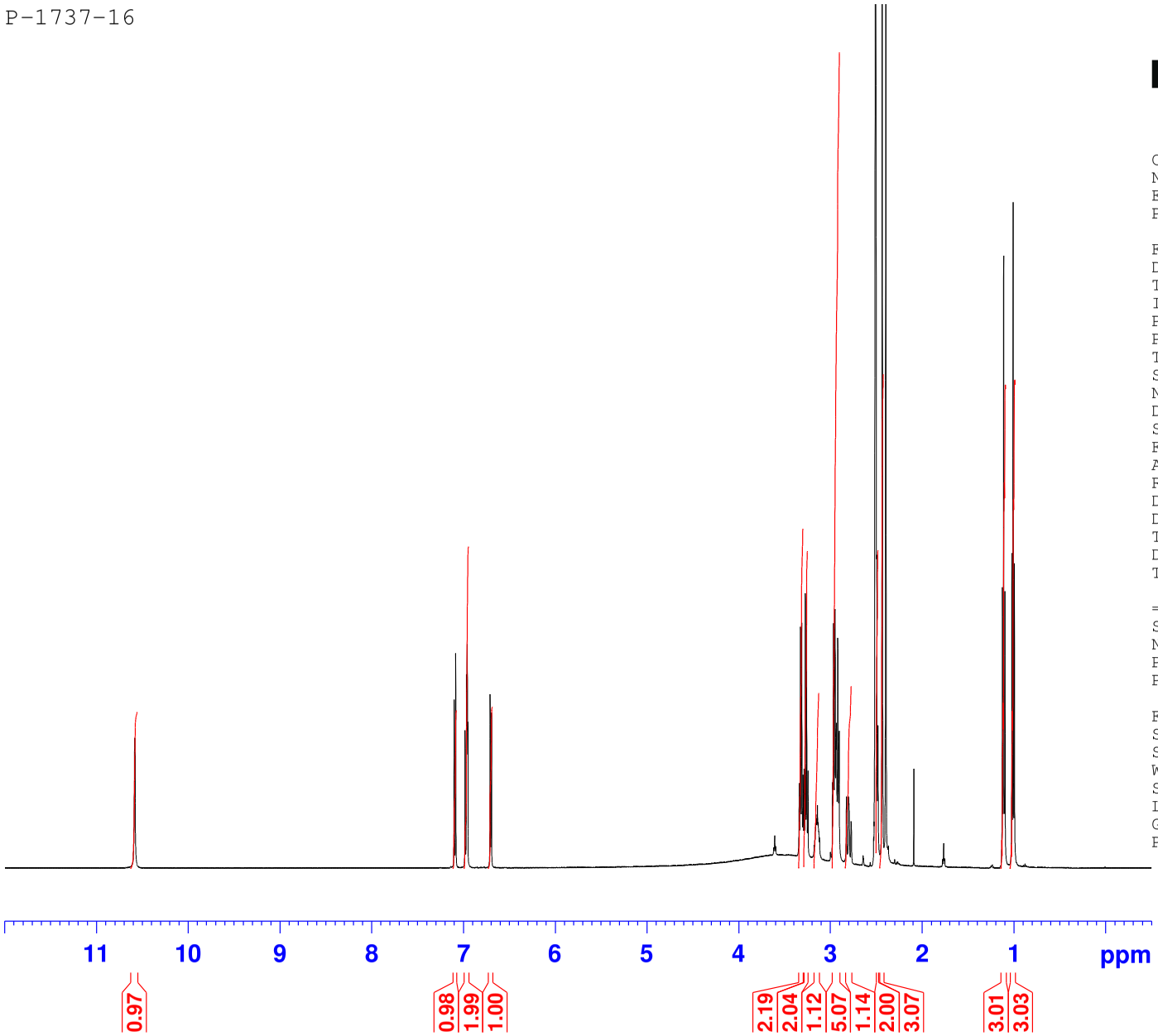
P-1737-16



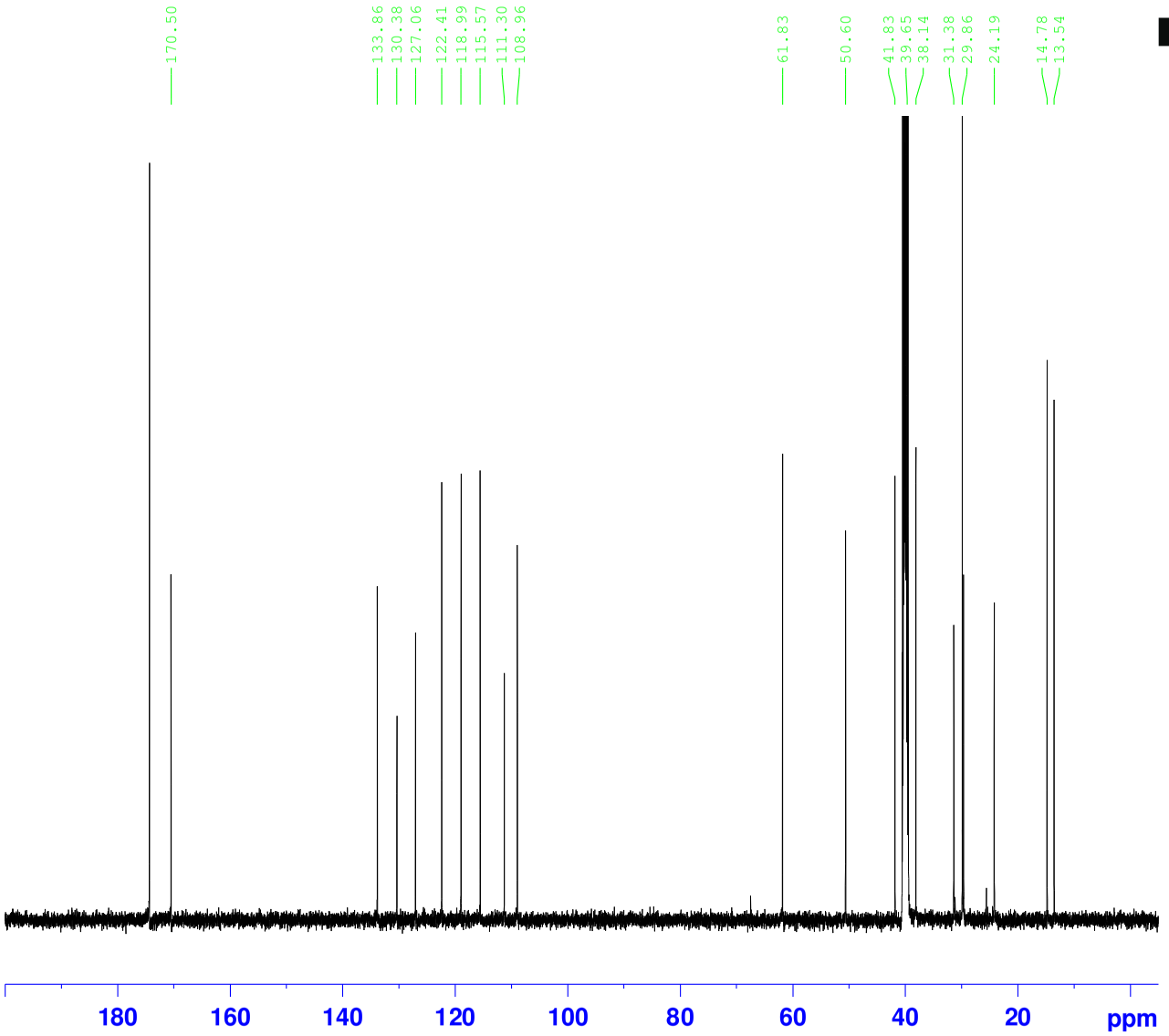
Current Data Parameters
NAME P-1737-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161231
Time 10.10
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 80.6
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 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

P-1737-16



BRUKER

```
Current Data Parameters
NAME                  P-1737-16
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20161231
Time                      12.45
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                  zgpg30
TD                        65536
SOLVENT                    DMSO
NS                         4096
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
PLW1               122.00000000 W

======== CHANNEL f2 ========
SFO2                500.1320005 MHz
NUC2                        1H
CPDPRG[2                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
```