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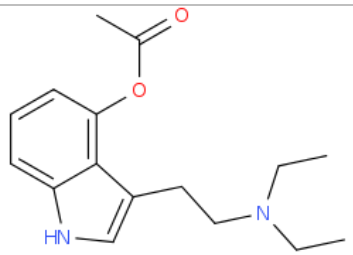
# ANALYTICAL REPORT[1]

## 4-AcO-DET (C16H22N2O2)

### 3-[2-(diethylamino)ethyl]-1H-indol-4-yl acetate

Remark – other NPS detected: none

| | |
|---|---|
| Sample ID: | 1411-16 |
| Sample description: | powder - white-off |
| Sample type: | test purchase /RESPONSE -purchasing |
| Date of sample receipt (M/D/Y): | 1/6/2016 |
| Date of entry (M/D/Y) into NFL database: | 2/12/2016 |
| 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 | 3-[2-(diethylamino)ethyl]-1H-indol-4-yl acetate |
| Other names | 4-Acetoxy-DET |
| Formula (per base form) | C16H22N2O2 |
| $M_w$ (g/mol) | 274,36 |
| Salt form/anions detected | fumarate |
| StdInChIKey | WYEVVQJLTXBMPM-UHFFFAOYSA-N |
| Compound Class | Indolalkylamines (fe tryptamines) |
| Other NPS detected | none |
| Add.info (purity..) | fumaric acid by GC-MS + cca5% of organic 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) |
|---|---|
| 28 May 2018 | Mw typing error corrected. |
| | |
| | |
| | |
| | |

## **Instrumental methods** (if applied) in NFL

**1. GC-MS** (Agilent): GC-method is RT locked to tetracosane (RT=9.53 min). Injection volume 1 ml and split mode (1:50) . Injector temperature: 280 $^{0}$C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 $^{0}$C for 1 min, followed by heating up to 293 $^{0}$C at a rate of 18 $^{0}$C/min, hold for 6.1 min, than 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) 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 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

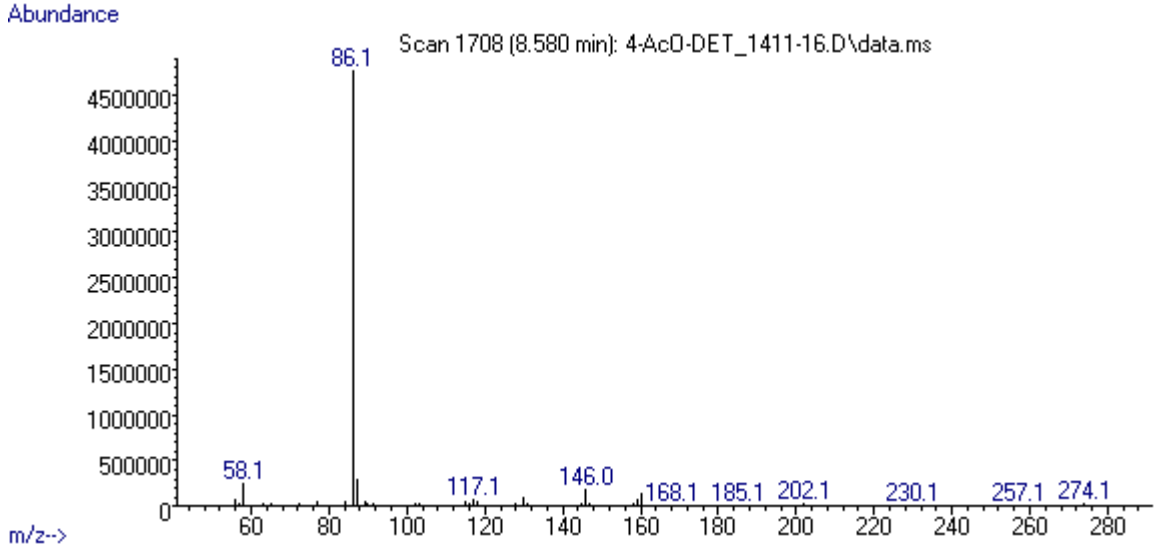
## Supporting information

| Solubility in | result/remark |
|---|---|
| $CH_2Cl_2$ | partially |
| MeOH | soluble |
| $H_2O$ | partially |

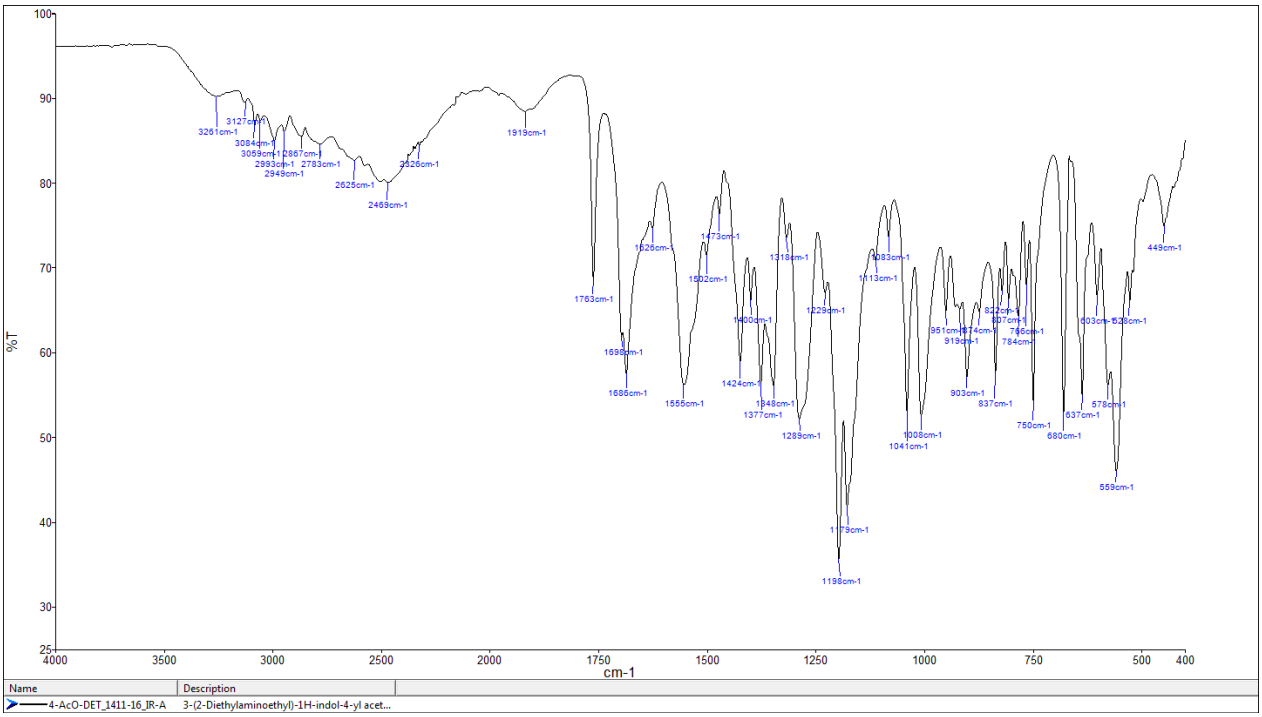
| Analytical technique: | applied | remarks |
|---|---|---|
| GC-MS (EI ionization) | + | NFL GC-RT (min): 8,58<br>BP(1): 86; BP(2): 87,BP(3) :58, |
| HPLC-TOF | + | Exact mass (theoretical): 274,1681;<br>measured value Δppm:0,11;<br>formula:C16H22N2O2 |
| FTIR-ATR | + | direct measurement (sample as received) |
| FTIR (condensed phase) always as base form | + | |
| IC (anions) | + | |
| NMR (in FKKT) | + | |
| validation | | |
| other | | |

## ANALYTICAL RESULTS

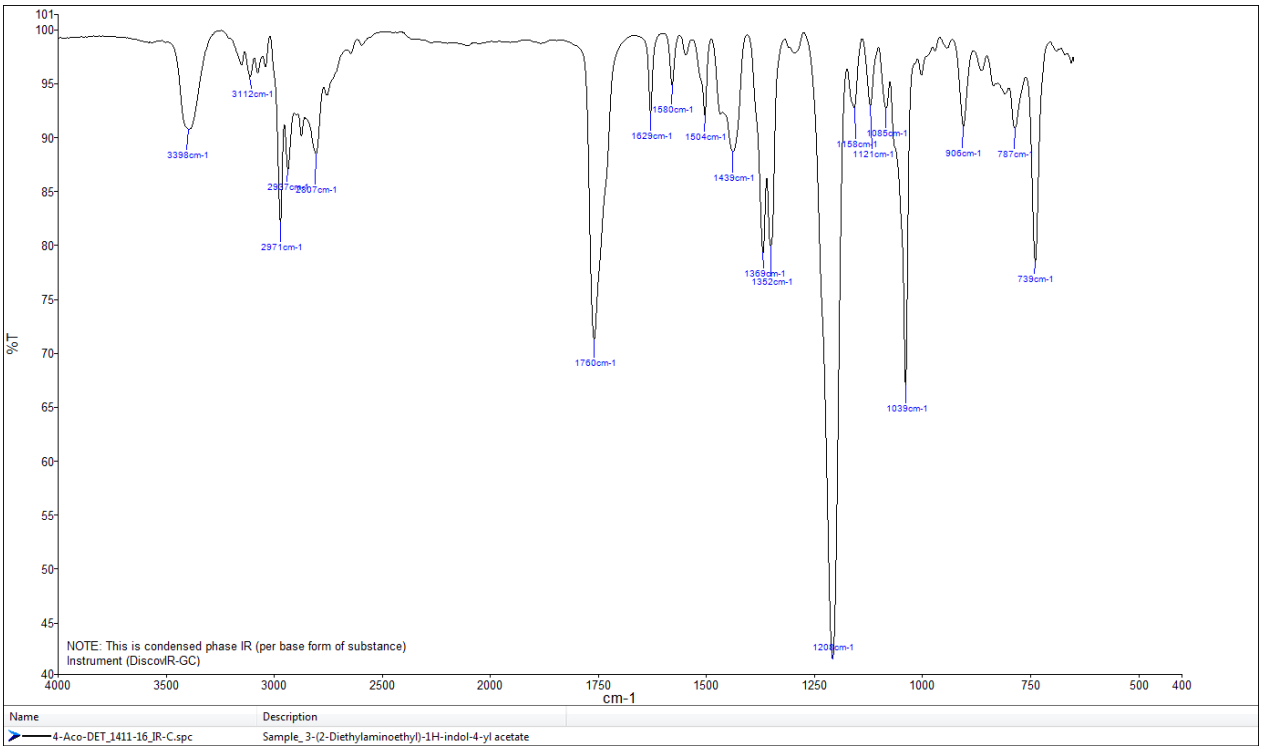
MS (EI)



FTIR-ATR - direct measurement (sample as received)




IR (condensed phase – after chromatographic separation)



# TOF REPORT

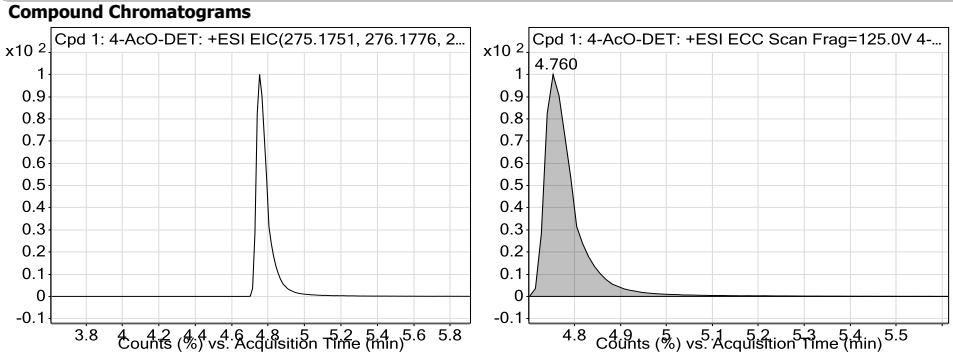
| | | | |
|---|---|---|---|
| **Data File** | 4-AcO-DET_1411-15_TOF.d | **Sample Name** | ID_1411-15 |
| **Sample Type** | Sample | **Position** | P1-D9 |
| **Instrument Name** | 6230B TOF LC-MS | **User Name** | TG |
| **Acq Method** | general-1512015-XDB-C18-ESI-poz.m | **Acquired Time** | 1/11/2016 1:00:23 PM |
| **IRM Calibration Status** | Success | **DA Method** | Drugs_NFL.m |
| **Comment** | extract in MeOH | | |

**Compound Table**

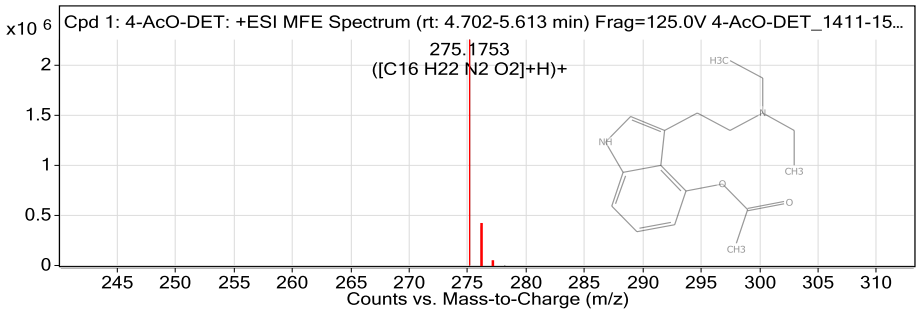
| Label | Compound Name | Obs. RT | Obs. Mass |
|---|---|---|---|
| Cpd 1: 4-AcO-DET | 4-AcO-DET | 4.76 | 274.1681 |

| Name | *Obs. m/z* | Obs. RT | Obs. Mass | DB RT | DB Formula | DB Mass | DB Mass Error (ppm) |
|---|---|---|---|---|---|---|---|
| **4-AcO-DET** | 275.1753 | 4.76 | 274.1681 | 4.76 | C16 H22 N2 O2 | 274.1681 | 0.11 |

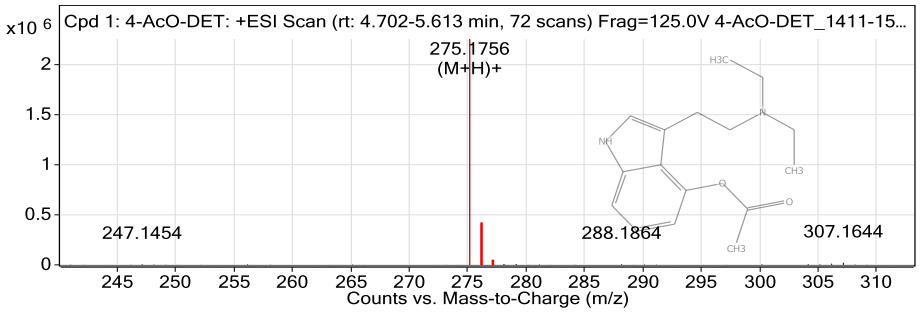
**Compound Chromatograms**




MFE MS Zoomed Spectrum




MS Zoomed Spectrum




**MS Spectrum Peak List**

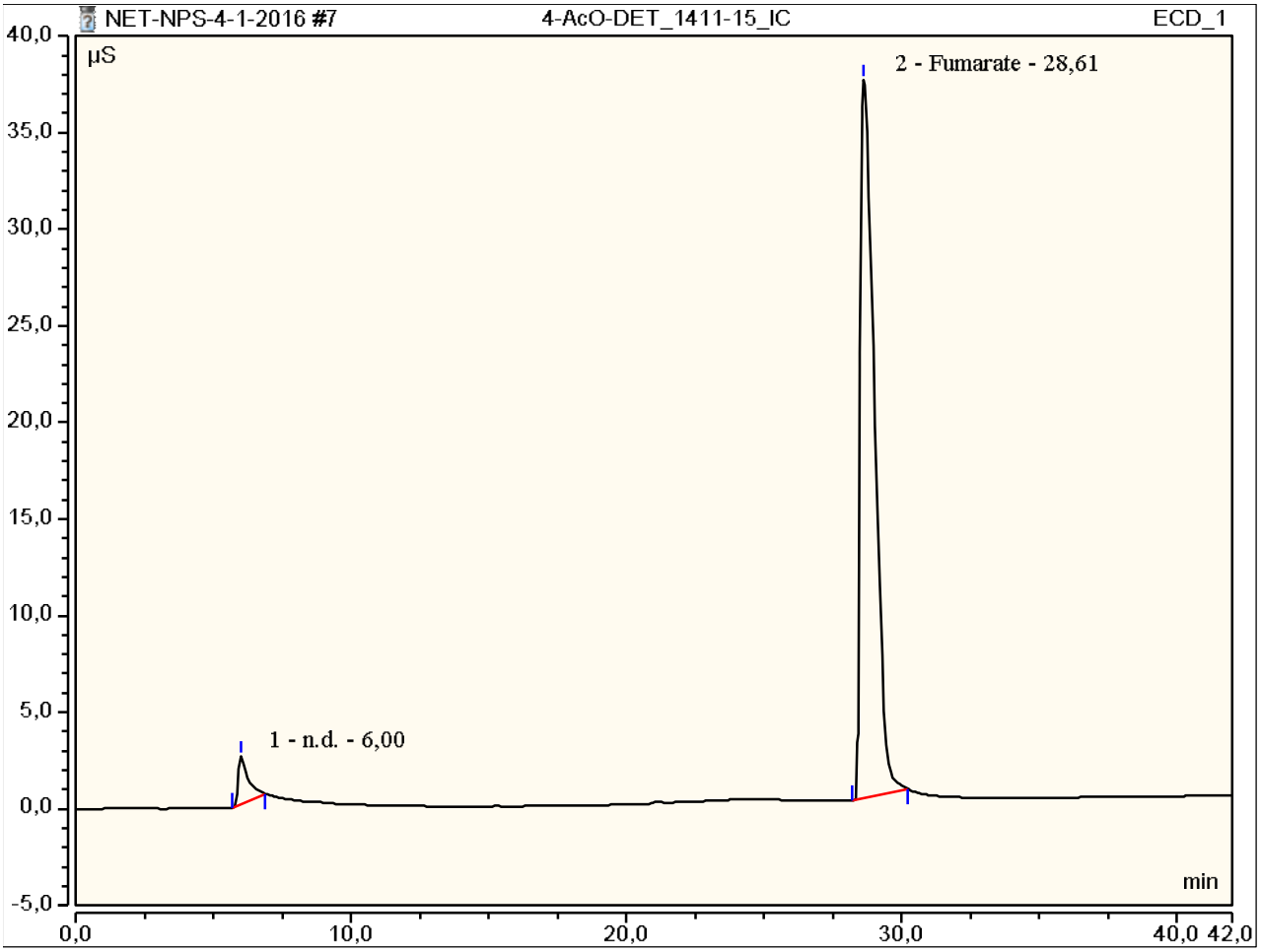
| *Obs. m/z* | Charge | Abund | Formula | Ion/Isotope |
|---|---|---|---|---|
| 275.1753 | 1 | 2261063.75 | C16 H22 N2 O2 | (M+H)+ |
| 276.1793 | 1 | 391863.09 | C16 H22 N2 O2 | (M+H)+ |
| 277.1813 | 1 | 44915.31 | C16 H22 N2 O2 | (M+H)+ |
| 278.1836 | 1 | 3831.46 | C16 H22 N2 O2 | (M+H)+ |

--- End Of Report ---

# Peak Integration Report

| | | | |
|---|---|---|---|
| *Sample Name:* | **4-AcO-DET_1411-15_IC** | *Inj. Vol.:* | **25,00** |
| *Injection Type:* | **Unknown** | *Dilution Factor:* | **1,0000** |
| *Program:* | **ANIONI** | *Operator:* | **kemija** |
| *Inj. Date / Time:* | **11-jan-2016 / 19:02** | *Run Time:* | **42,00** |

| No. | Time min | Peak Name | Peak Type | Area µS*min | Height µS | Amount mg/L |
|---|---|---|---|---|---|---|
| 1,00 | 6,00 | **n.d.** | BMB | 1,06 | 2,46 | n.a. |
| 2,00 | 28,61 | **Fumarate** | BMB | 22,23 | 37,14 | n.a. |
| | | **TOTAL:** | | 23,29 | 39,60 | 0,00 |



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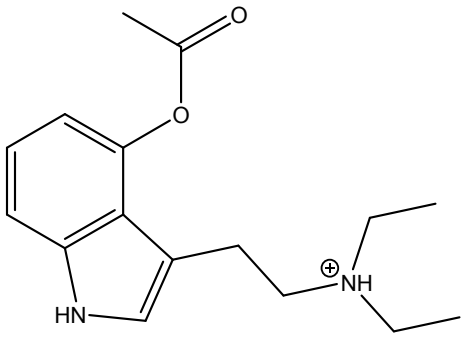
# REPORT

| | |
|---|---|
| Sample ID: | **1411-16** |
| Our notebook code: | P-1411-16 |
| 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: | 2-(4-acetoxy-1H-indol-3-yl)-N,N-diethylethan-1-aminium |
| Comments: | - Structure elucidation based on 1D and 2D NMR spectra<br>- Sample is not pure according to NMR, it contains fumaric acid or its anion ($^1$H NMR signal at 6.59, $^{13}$C NMR at 167.3 and 134.9) and some other minor impurities (evident in $^1$H and $^{13}$C NMR). NB. Signals belonging to fumaric fragment are neither peak-picked nor integrated. |
| Supporting information: | Copies of $^1$H and $^{13}$C NMR spectra |
| Author: | Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc |
| Date of report: | February 18, 2016 |

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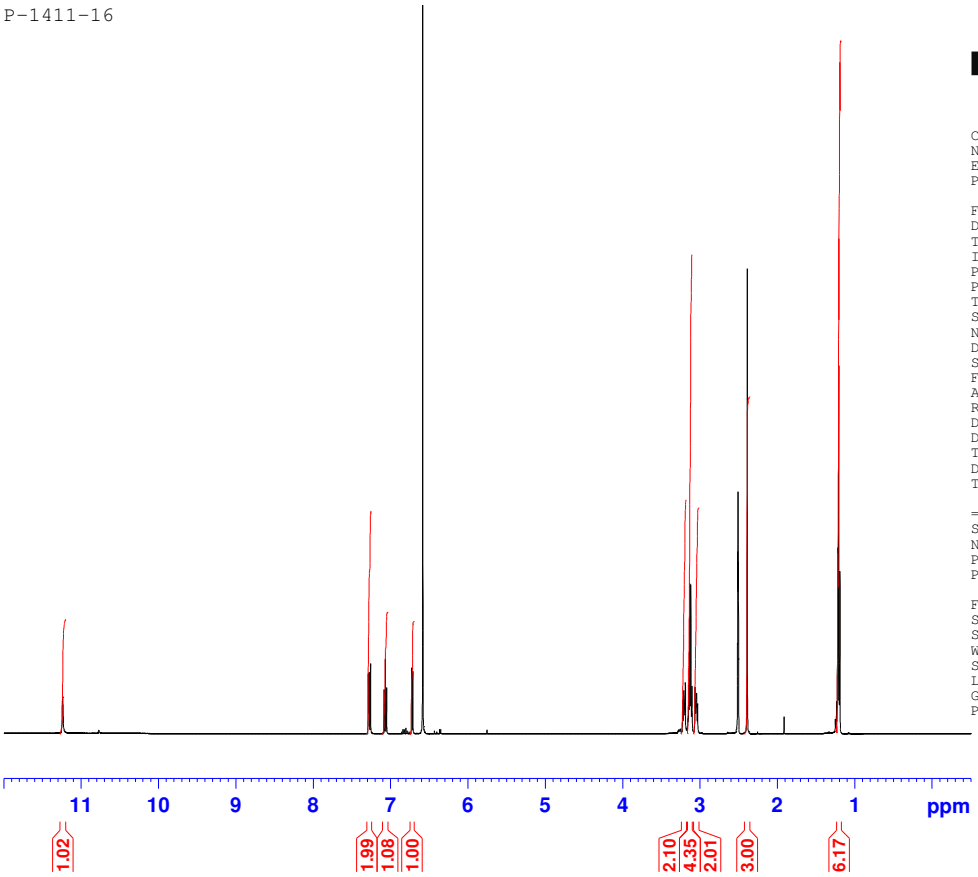
P-1411-16



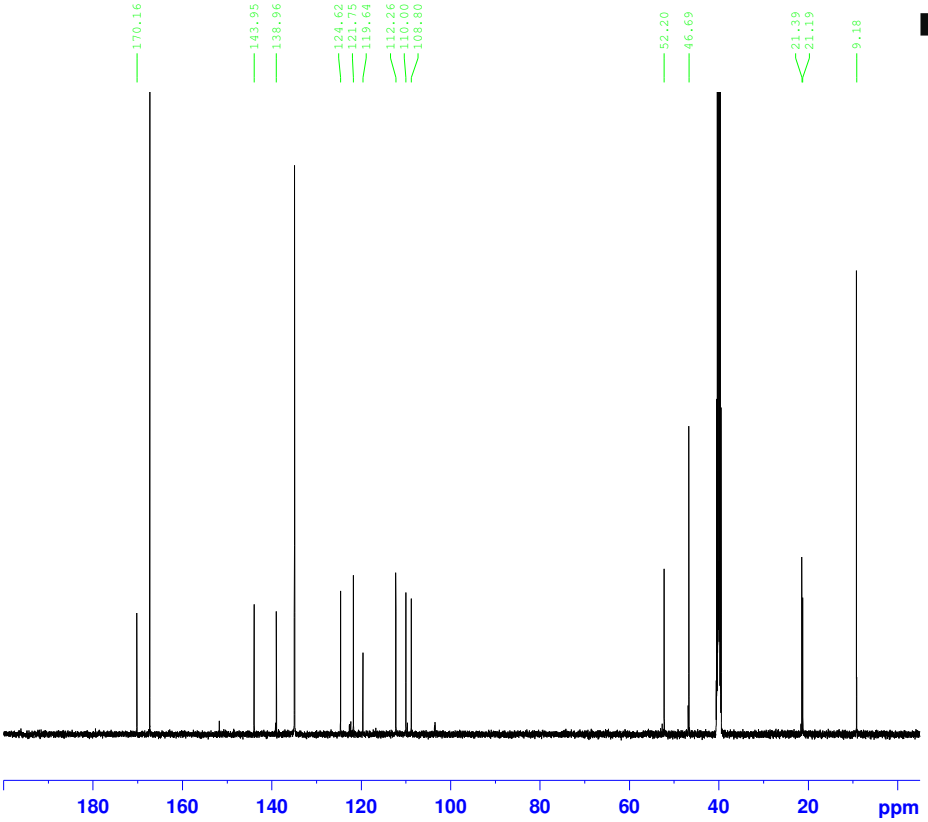

Current Data Parameters
NAME P-1411-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160216
Time 21.06
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 71.8
DW 50.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 8.90 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-1411-16




Current Data Parameters
NAME P-1411-16
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160216
Time 22.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
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 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.00 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 0.32179001 W
PLW13 0.16186000 W

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