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EUROPEAN PROJECT RESPONSE 2 to challenges in forensic drugs analyses
Co-funded by the Internal Security Found of the European Union

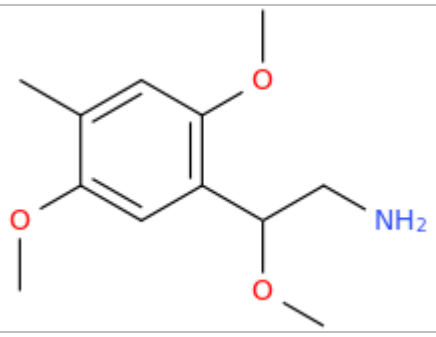
# ANALYTICAL REPORT

## BOD (C12H19NO3)

### 2-(2,5-dimethoxy-4-methylphenyl)-2-methoxyethan-1-amine

Remark – other NPS detected: none

| | |
|---|---|
| Sample ID: | 2067-19 |
| Sample description: | powder |
| Sample type: | test purchase /NFL- purchasing |
| Date of entry (DD/MM/YYYY) into NFL database: | 07/06/2019 |
| Report updates (if any) will be published here: | http://www.policija.si/apps/nfl_response_web/seznam.php |

| | |
|---|---|
| Substance identified - structure[1] (base form) |  |
| Systematic name | 2-(2,5-dimethoxy-4-methylphenyl)-2-methoxyethan-1-amine |
| Other names | 2,5,beta-Trimethoxy-4-methylbenzeneethanamine; 4-Methyl-2,5,beta-trimethoxyphenethylamine; Beta-Methoxy-2c-d |
| Formula (per base form) | C12H19NO3 |
| $M_w$ (g/mol) | 225,29 |
| Salt form/anions detected | fumarate |
| StdInChIKey (per base form) | VTEIFHQUZWABDE-UHFFFAOYSA-N |
| Other NPS detected | none |
| Additional info (purity..) | >95% purity by 1H NMR |

[1] 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 KOH 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

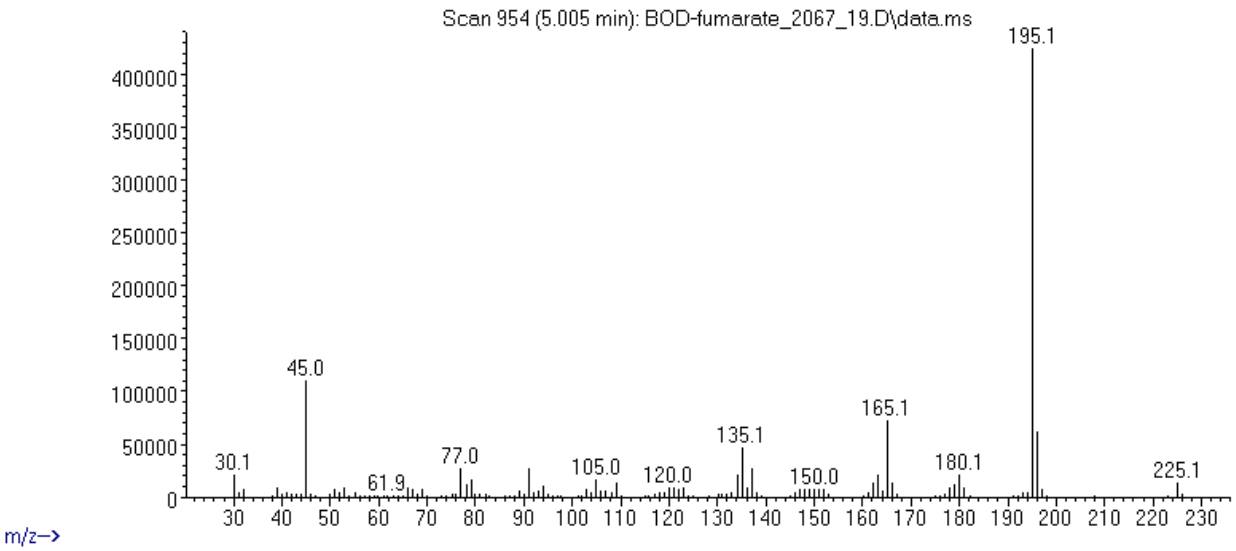
## Supporting information

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

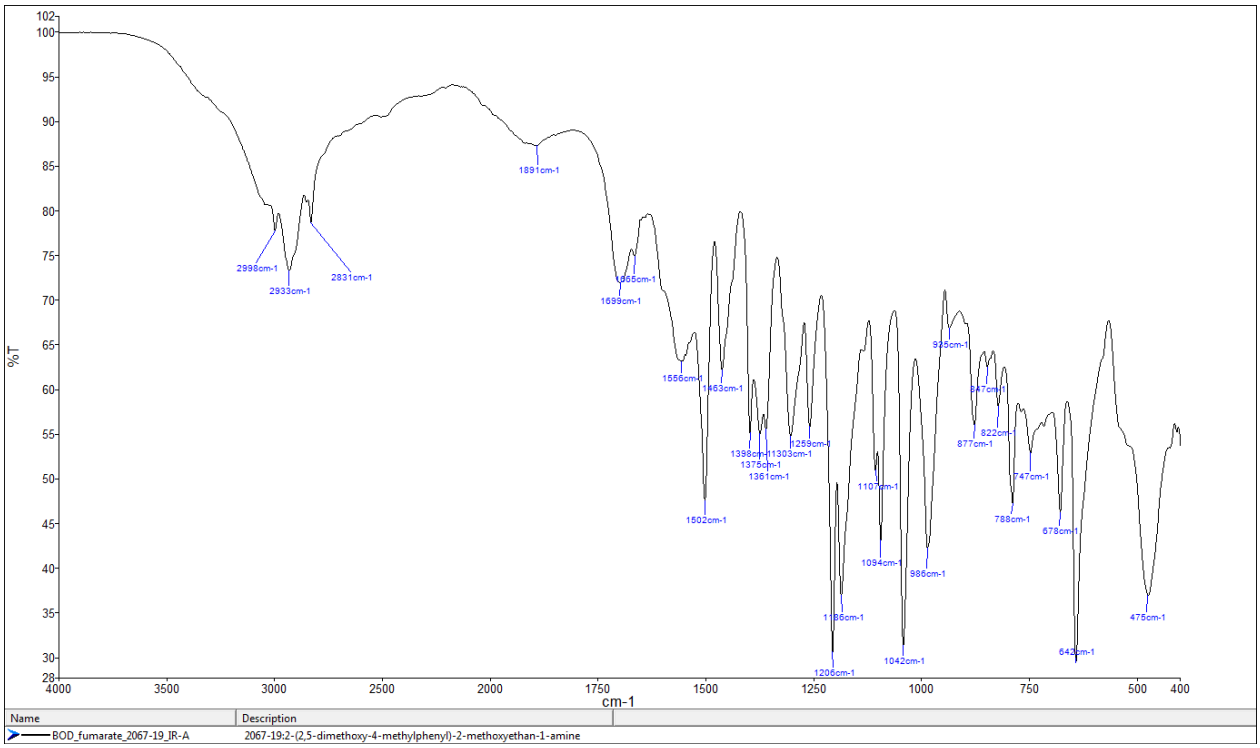
| Analytical technique: | applied | remarks |
|---|---|---|
| GC-MS (EI ionization) | + | NFL GC-RT (min): 5,01<br>BP(1): 195; BP(2): 45,BP(3) :165, |
| HPLC-TOF | + | Exact mass (theoretical): 225,1365;<br>measured value Δppm:2,21;<br>formula:C12H19NO3 |
| FTIR-ATR | + | direct measurement (sample as received) |
| FTIR (solid phase) always as base form | + | |
| IC (anions) | + | |
| NMR (in FKKT) | + | |
| validation | | |
| other | | |

## ANALYTICAL RESULTS

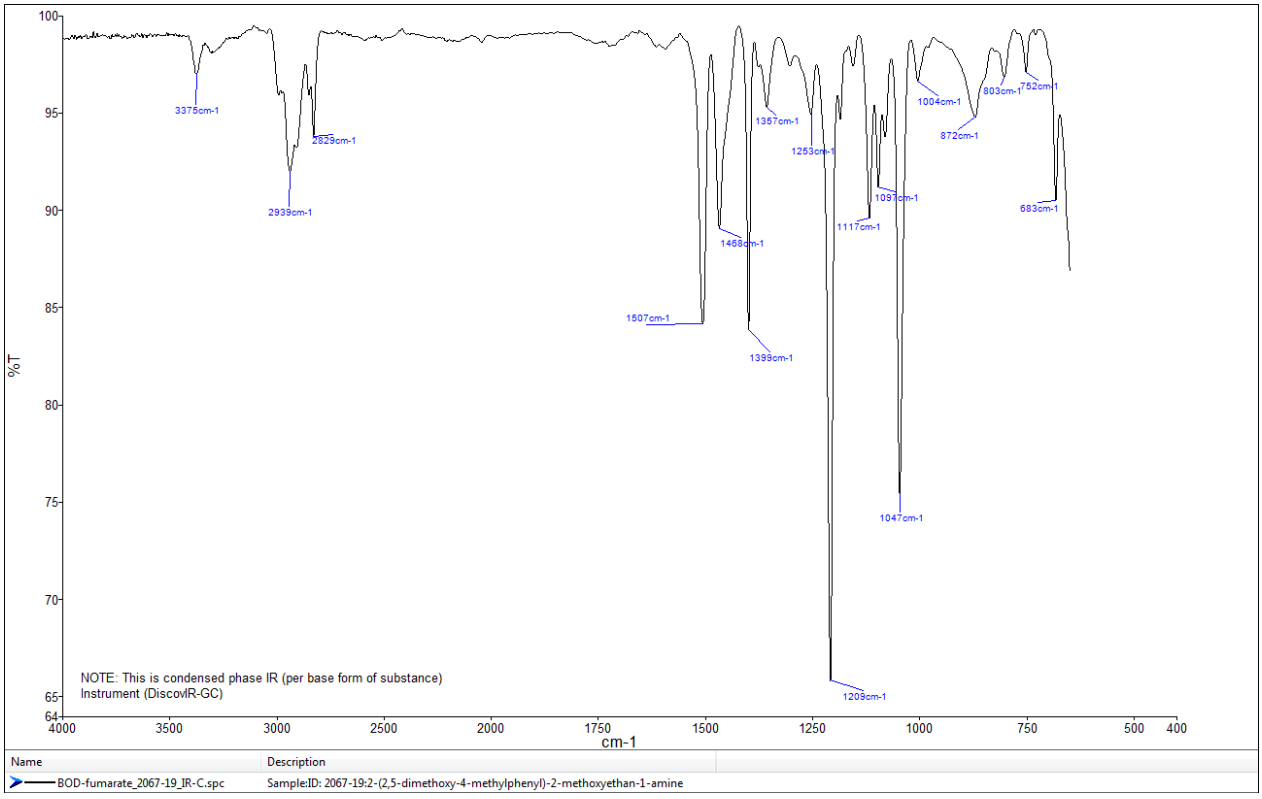
MS (EI)



FTIR-ATR - direct measurement (sample as received – fumarate salt)




IR (solid phase – after chromatographic separation) – base form of substance



# TOF REPORT

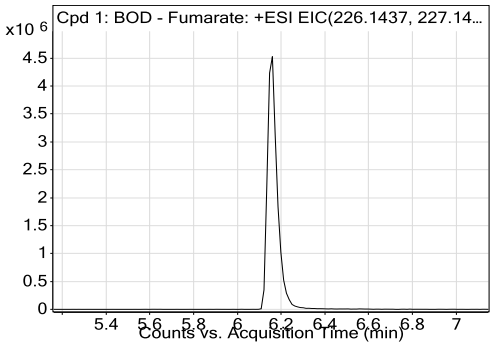
| | | | |
|---|---|---|---|
| **Data File** | BOD-Fumarate_2067-19.d | **Sample Name** | ID-2067-19 |
| **Sample Type** | Sample | **Position** | P1-A6 |
| **Instrument Name** | 6230B TOF LC-MS | **User Name** | TG |
| **Acq Method** | general-8_1_2019-XDB-C18-ESI+.m | **Acquired Time** | 5/13/2019 1:49:45 PM |
| **IRM Calibration Status** | Success | **DA Method** | a-Drugs_NFL.m |
| **Comment** | MeOH | | |

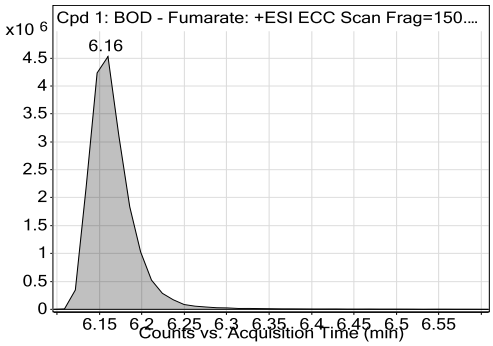
**Compound Table**

| Label | Compound Name | MFG Formula | Obs. RT | Obs. Mass |
|---|---|---|---|---|
| Cpd 1: BOD - Fumarate | BOD - Fumarate | C12 H19 N O3 | 6.16 | 225.136 |

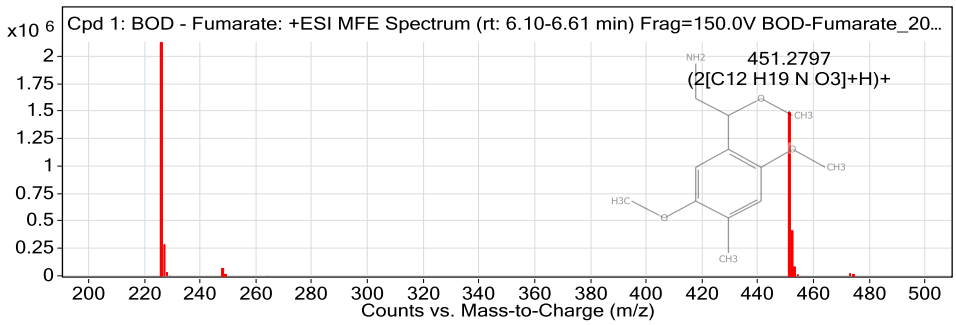
| Name | *Obs. m/z* | Obs. RT | Obs. Mass | DB RT | DB Formula | DB Mass | DB Mass Error (ppm) |
|---|---|---|---|---|---|---|---|
| **BOD - Fumarate** | 226.1427 | 6.16 | 225.136 | 6.16 | C12 H19 N O3 | 225.1365 | 2.21 |

**Compound Chromatograms**

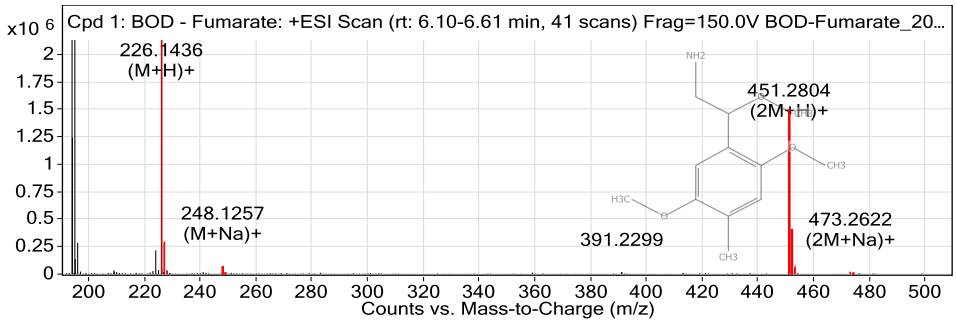



MFE MS Zoomed Spectrum




MS Zoomed Spectrum




**MS Spectrum Peak List**

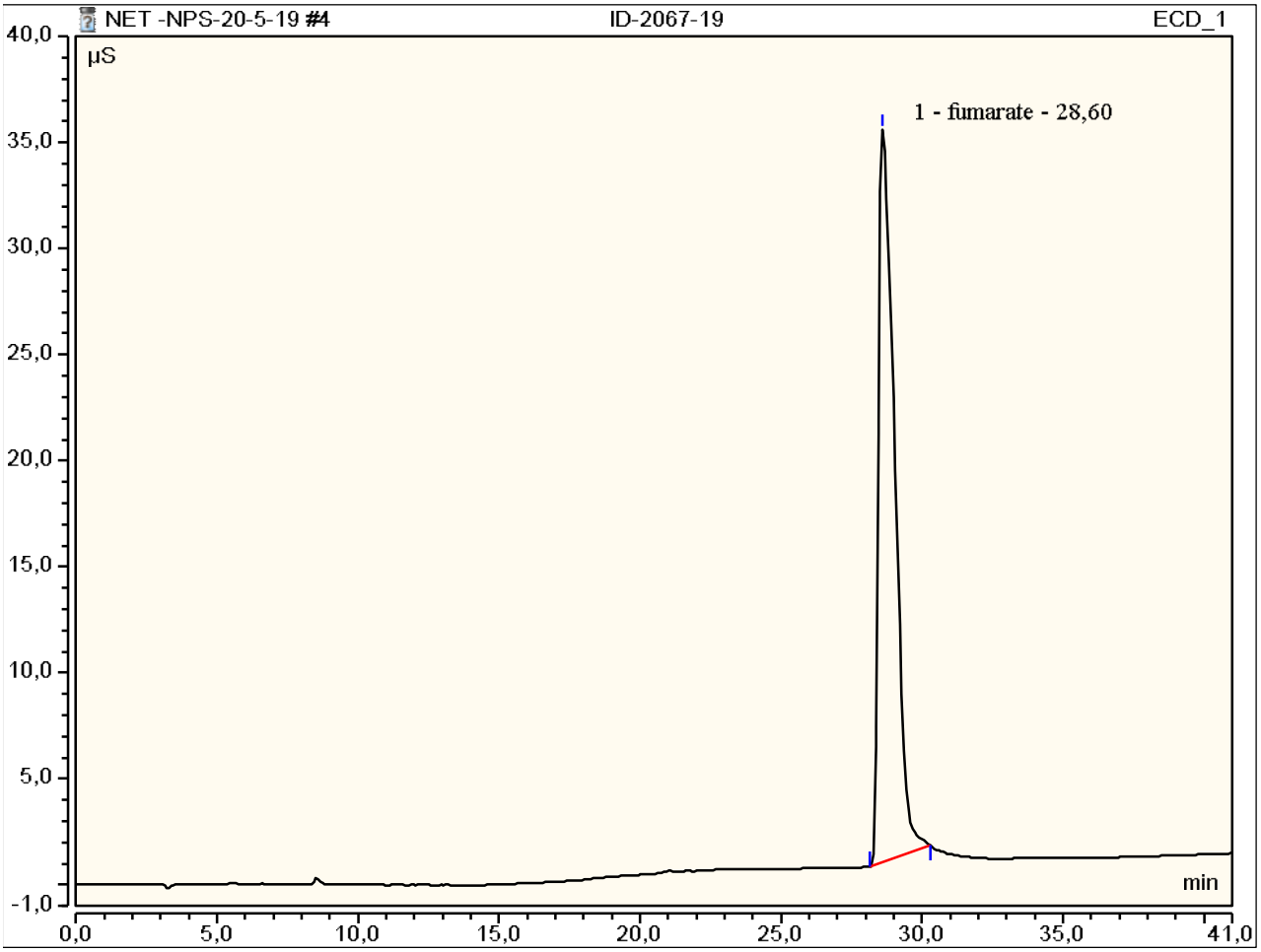
| *Obs. m/z* | Charge | Abund | Formula | Ion/Isotope |
|---|---|---|---|---|
| 226.1427 | 1 | 2130266.5 | C12 H19 N O3 | (M+H)+ |
| 227.1461 | 1 | 283185.17 | C12 H19 N O3 | (M+H)+ |
| 228.1483 | 1 | 30876.68 | C12 H19 N O3 | (M+H)+ |
| 248.1248 | 1 | 69869.52 | C12 H19 N O3 | (M+Na)+ |
| 249.1284 | 1 | 9982.77 | C12 H19 N O3 | (M+Na)+ |
| 451.2797 | 1 | 1498378.75 | C12 H19 N O3 | (2M+H)+ |
| 452.2831 | 1 | 399256.91 | C12 H19 N O3 | (2M+H)+ |
| 453.2855 | 1 | 67988.38 | C12 H19 N O3 | (2M+H)+ |
| 454.2873 | 1 | 9780.56 | C12 H19 N O3 | (2M+H)+ |
| 473.2616 | 1 | 20484.72 | C12 H19 N O3 | (2M+Na)+ |

--- End Of Report ---

# Peak Integration Report

| | | | |
|---|---|---|---|
| *Sample Name:* | **ID-2067-19** | *Inj. Vol.:* | **25,00** |
| *Injection Type:* | **Unknown** | *Dilution Factor:* | **1,0000** |
| *Program:* | **ANIONI** | *Operator:* | **kemija** |
| *Inj. Date / Time:* | **20-maj-2019 / 11:17** | *Run Time:* | **42,00** |

| No. | Time min | Peak Name | Peak Type | Area µS*min | Height µS | Amount mg/L |
|---|---|---|---|---|---|---|
| 1,00 | 28,60 | **fumarate** | BMB | 23,01 | 34,57 | n.a. |
| | | **TOTAL:** | | 23,01 | 34,57 | 0,00 |



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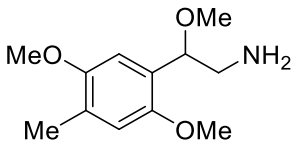
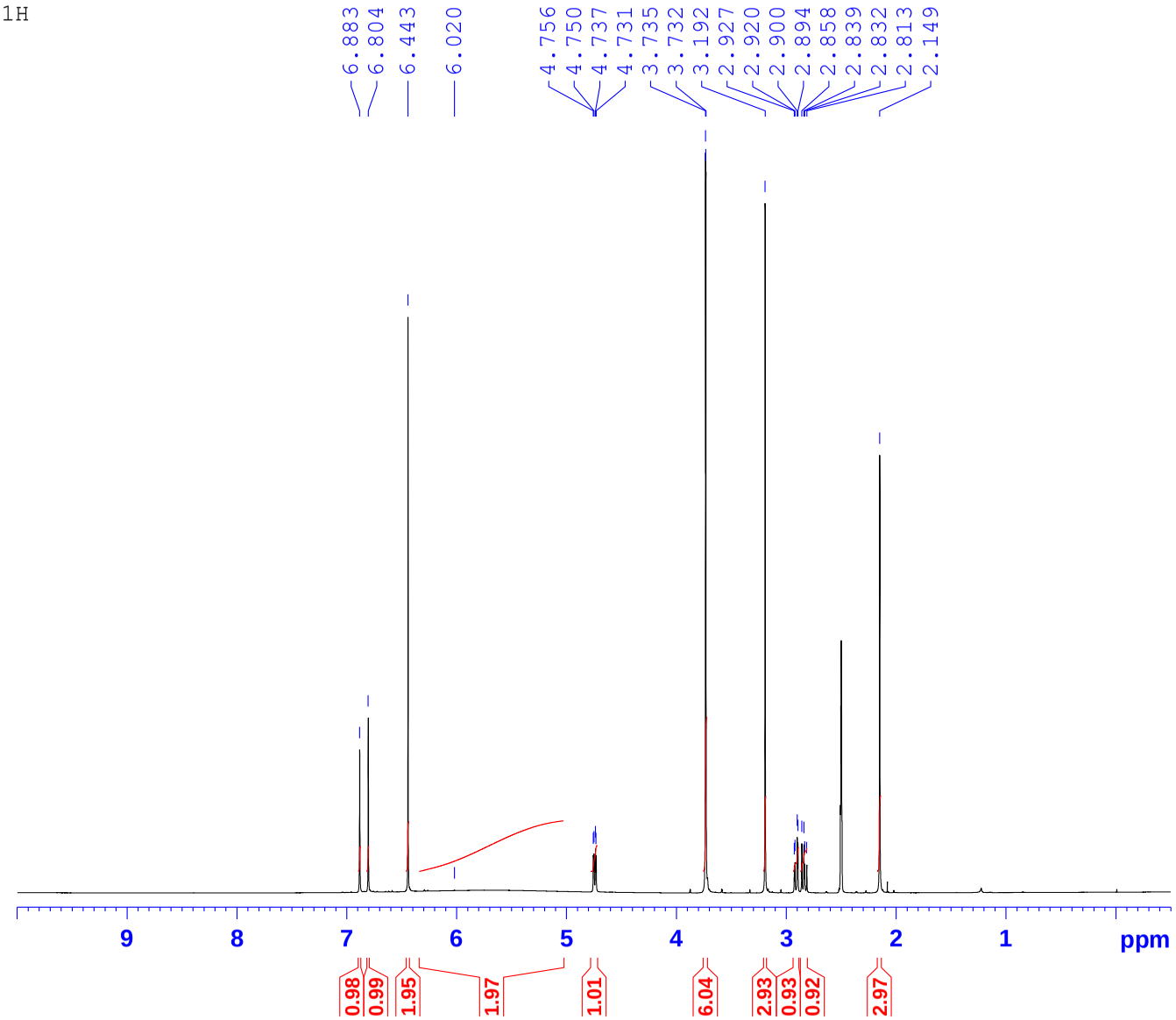
University
*of Ljubljana*

Faculty *of Chemistry*
*and Chemical Technology*

# R E P O R T

| | |
|---|---|
| Contract No. | C1714-17-460078 (Republic of Slovenia, Ministry of the Interior, POLICE) |
| Sample ID: | **2067-19** |
| Received date: | May 20, 2019 |
| Our notebook code: | NFL-2067-19 |
| NMR sample preparation: | 20.3 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 with formula, exact mass, molecular weight: |  Chemical Formula: $C_{12}H_{19}NO_3$<br>Exact Mass: 225,1365<br>Molecular Weight: 225,2880 |
| Chemical name: | 2-(2,5-dimethoxy-4-methylphenyl)-2-methoxyethan-1-amine |
| Comments: | - Structure elucidation based on 1D and 2D NMR spectra and HRMS.<br>- The sample consists of herein identified compound as a salt with fumaric acid.<br>- >95% purity of a sample, based on $^1$H NMR spectrum. |
| Supporting information: | Copies of $^1$H and $^{13}$C NMR spectra, $^1$H and $^{13}$C FIDs. |
| Principal investigator: | Prof. Dr. Janez Košmrlj |
| Date of report: | May 28, 2019 |

NFL-2067-19
1H

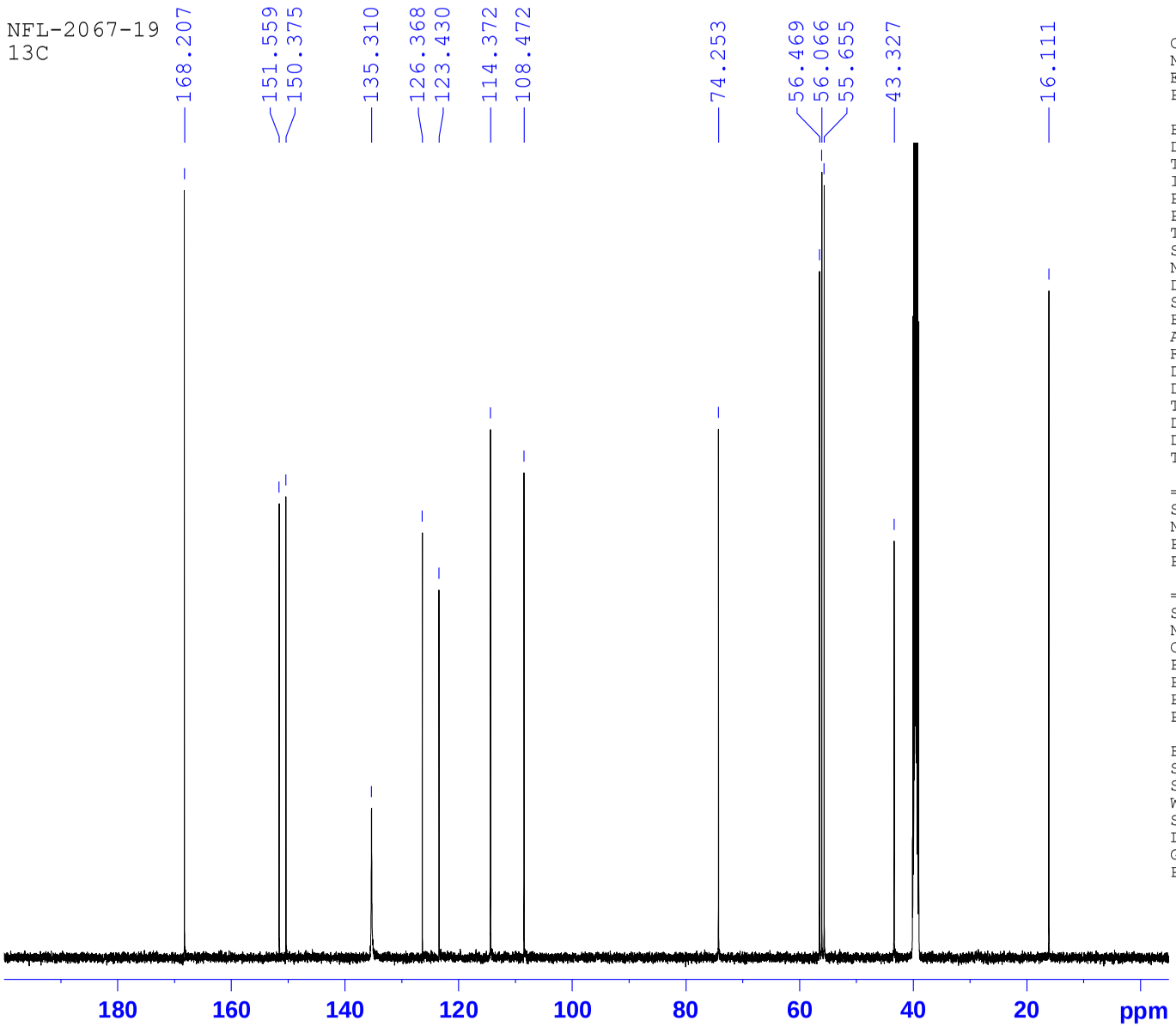



```
Current Data Parameters
NAME         NFL-2067-19
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190521
Time                2.25
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT              DMSO
NS                     32
DS                      2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2767999 sec
RG                  71.8
DW                50.000 usec
DE                 6.50 usec
TE                296.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                  8.70 usec
PLW1         26.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300042 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME         NFL-2067-19
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190521
Time                4.17
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                296.0 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           125.7578421 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```