

## Joint Research Centre

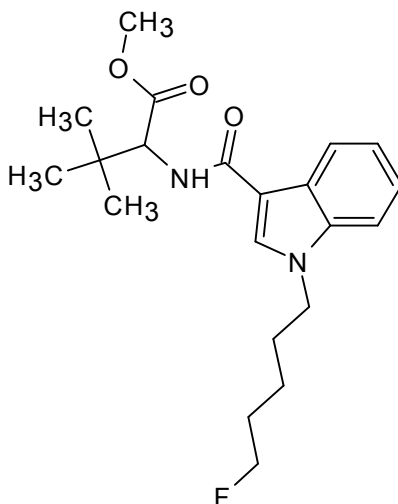
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### ADMINISTRATIVE ARRANGEMENT JRC-Nr 33619-CLEN2SAND-DG TAXUD-Nr TAXUD/2014/DE/315 BETWEEN DG TAXATION AND CUSTOMS UNION (DG TAXUD) AND THE JOINT RESEARCH CENTRE (JRC) for fast recognition of New Psychoactive Substances (NPS) and identification of unknown chemicals

This report was generated on 13/10/2016 based on data from the European Customs laboratories and the Joint Research Centre. This report includes sample and molecular information, spectral data and associated tables and figures. The chemical structure(s) was/were identified by Bio-chemical interactions & metabolomics (BCIM) group chemists on the basis of analytical data available. NMR assignments proposed below were performed by ACD labs tools in agreement with the chemical structure identified by analytical experts. Reported data are related to the sample in the following table:

|                                  |                                                           |                               |                              |
|----------------------------------|-----------------------------------------------------------|-------------------------------|------------------------------|
| <b>Eurodat number</b>            | 16100003                                                  | <b>Received on</b>            | 7 October 2016               |
| <b>Weight [mg]</b>               | 100                                                       | <b>PACKAGING</b>              | 100mg in glass vial          |
| <b>Colour</b>                    | pink-orange                                               | <b>Name of customer</b>       | Belgian Customs laboratory   |
| <b>Customer's identification</b> | 16BD-00507-01A                                            | <b>Customer's information</b> | Confirmation of 5F-MDMB-PICA |
| <b>Customer's comments</b>       | New synthetic cannabinoid - 5F-MDMB-PICA -to be confirmed |                               |                              |

The following structure(s) was/were identified in the sample:



Data of identified compound(s)

|                               |                                                                                                                                                    |
|-------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Chemicalize IUPAC name</b> | methyl 2-[[1-(5-fluoropentyl)-1H-indol-3-yl]formamido]-3,3-dimethylbutanoate                                                                       |
| <b>NAME</b>                   | 5F-MDMB-PICA                                                                                                                                       |
| <b>Formula</b>                | C <sub>21</sub> H <sub>29</sub> FN <sub>2</sub> O <sub>3</sub>                                                                                     |
| <b>FW</b>                     | 376.4650                                                                                                                                           |
| <b>Monoisotopic Mass</b>      | 376.216221                                                                                                                                         |
| <b>InChI</b>                  | InChI=1/C21H29FN2O3/c1-21(2,3)18(20(26)27-4)23-19(25)16-14-24(13-9-5-8-12-22)17-11-7-6-10-15(16)17/h6-7,10-11,14,18H,5,8-9,12-13H2,1-4H3,(H,23,25) |
| <b>SMILES</b>                 | O=C(OC)C(NC(=O)c2cn(CCCCCF)c1ccccc12)C(C)(C)C                                                                                                      |

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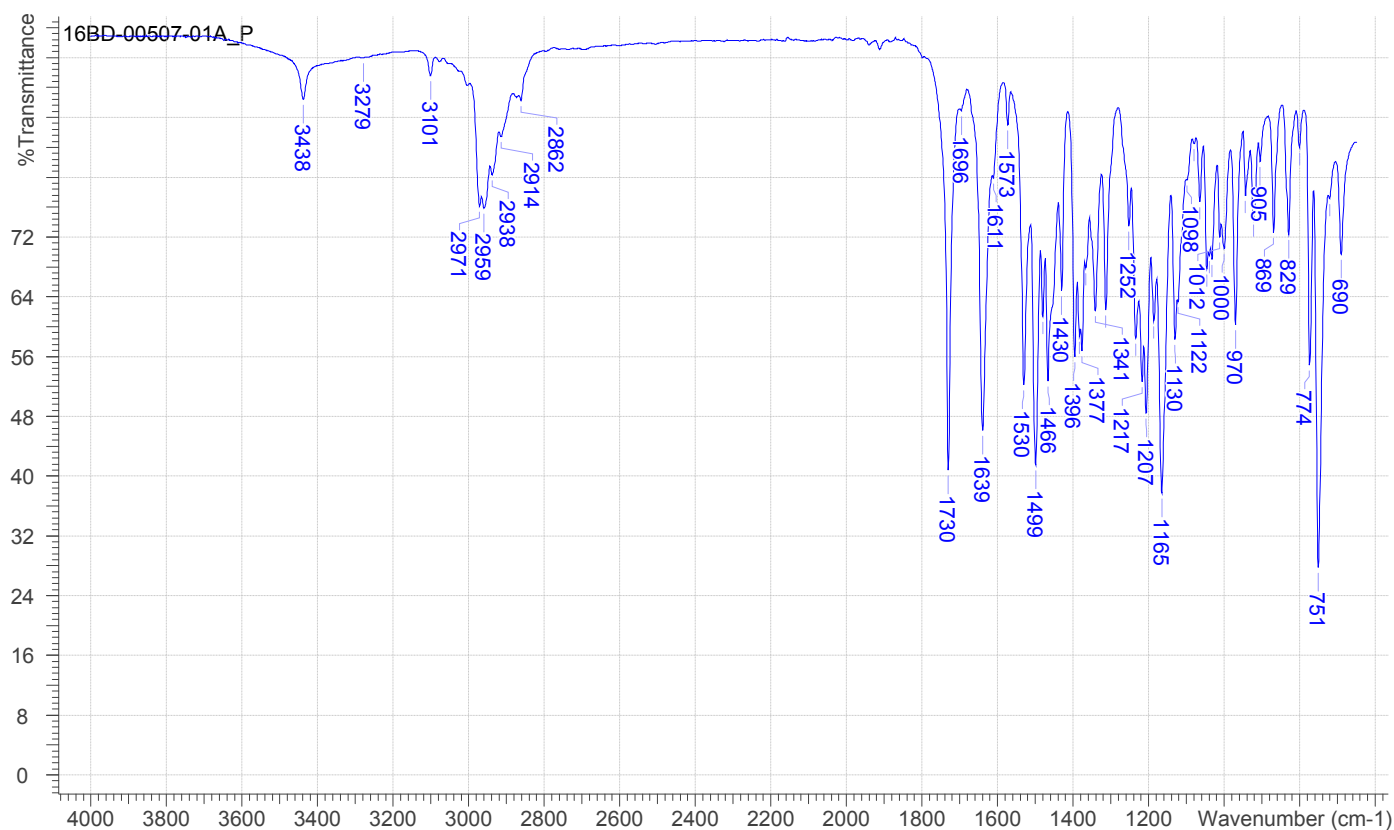
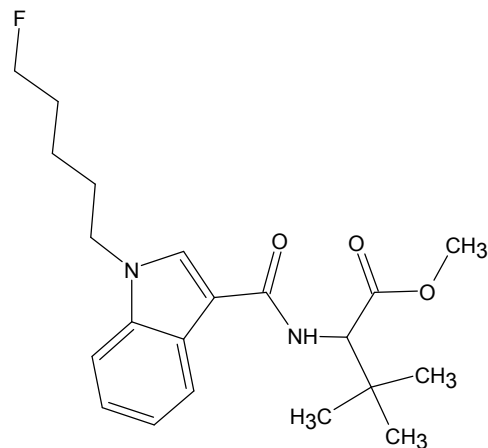
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Processing and interpretation based on data provided by:

Belgian Customs laboratory

|                       |                                                                                                                                                   |                        |                          |
|-----------------------|---------------------------------------------------------------------------------------------------------------------------------------------------|------------------------|--------------------------|
| <b>Comment</b>        | POEDER                                                                                                                                            |                        |                          |
| <b>File Name</b>      | \\139.191.6.82\ihcp\$\I01\BIO_CHEMICAL_INTERACTION_METABONOMICS\CLEN2SAND\PROPOSED-STRUCTURE\CLEN_NAS\16100003_16BD-00507-01A\16BD-00507-01A_P.SP |                        |                          |
| <b>Date Stamp</b>     | wed sep 28 11:07:50 2016                                                                                                                          | <b>Date</b>            | wed sep 28 11:07:50 2016 |
| <b>Technique</b>      | Infrared                                                                                                                                          | <b>Spectral Region</b> | IR                       |
| <b>X Axis</b>         | Wavenumber (cm <sup>-1</sup> )                                                                                                                    | <b>Y Axis</b>          | %Transmittance           |
| <b>Spectrum Range</b> | 650.0000 - 4000.0000                                                                                                                              | <b>Points Count</b>    | 3351                     |
| <b>Data Spacing</b>   | 1.0000                                                                                                                                            |                        |                          |



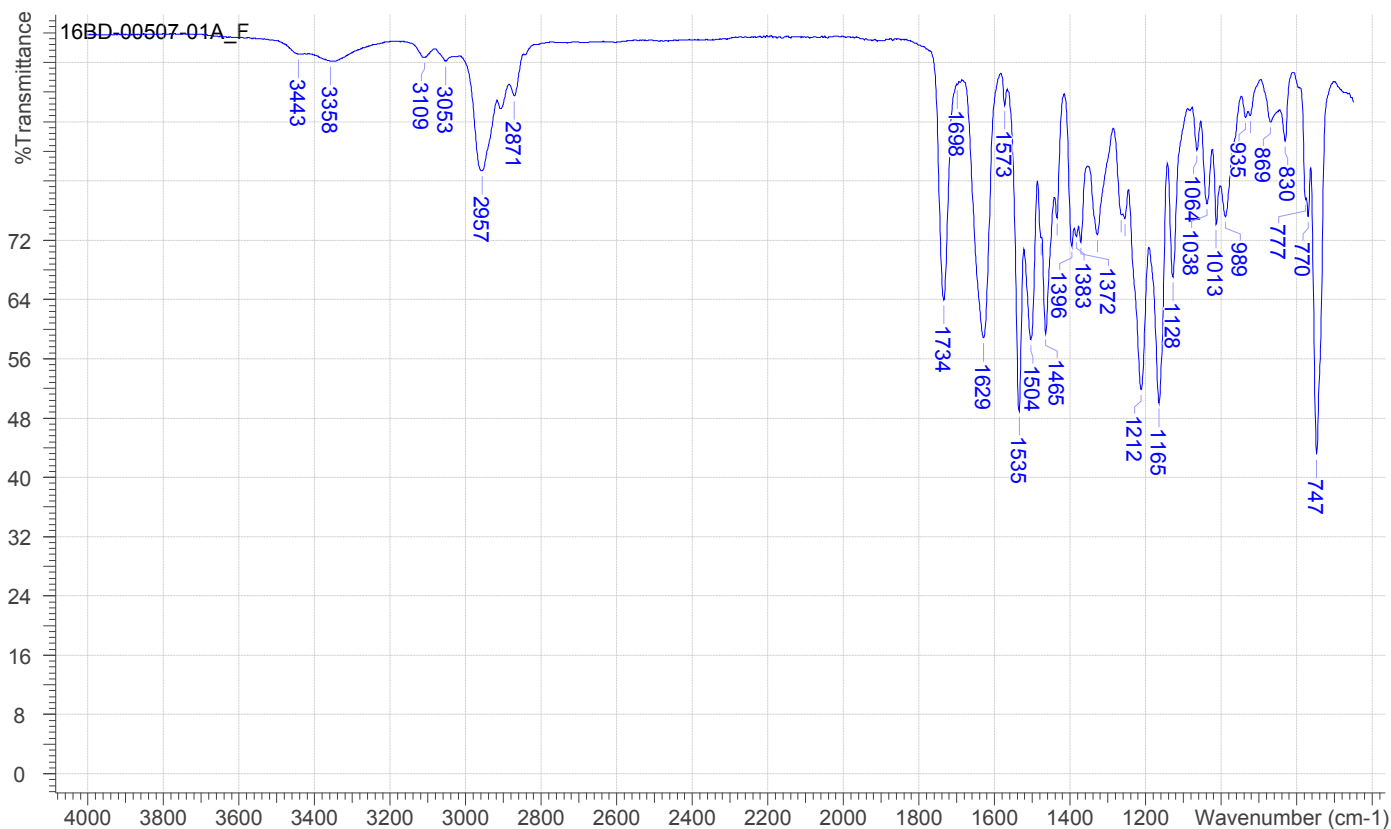
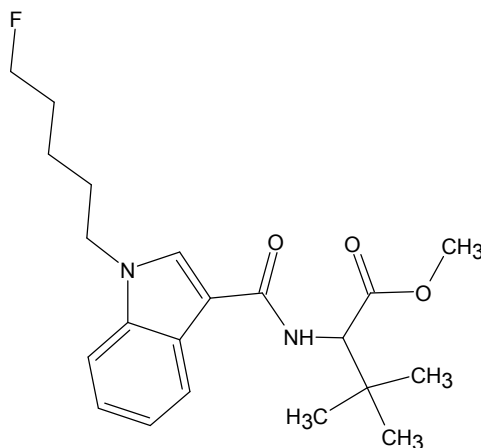
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**Belgian Customs laboratory**

|                       |                                                                                                                                                   |                        |                          |
|-----------------------|---------------------------------------------------------------------------------------------------------------------------------------------------|------------------------|--------------------------|
| <b>Comment</b>        | EVAPORATED SOLUTION IN DCM/MTOH                                                                                                                   |                        |                          |
| <b>File Name</b>      | \\139.191.6.82\ihcp\$\I01\BIO_CHEMICAL_INTERACTION_METABONOMICS\CLEN2SAND\PROPOSED-STRUCTURE\CLEN_NAS\16100003_16BD-00507-01A\16BD-00507-01A_F.SP |                        |                          |
| <b>Date Stamp</b>     | wed oct 05 09:14:05 2016                                                                                                                          | <b>Date</b>            | wed oct 05 09:14:05 2016 |
| <b>Technique</b>      | Infrared                                                                                                                                          | <b>Spectral Region</b> | IR                       |
| <b>X Axis</b>         | Wavenumber (cm <sup>-1</sup> )                                                                                                                    | <b>Y Axis</b>          | %Transmittance           |
| <b>Spectrum Range</b> | 650.0000 - 4000.0000                                                                                                                              | <b>Points Count</b>    | 3351                     |
| <b>Data Spacing</b>   | 1.0000                                                                                                                                            |                        |                          |



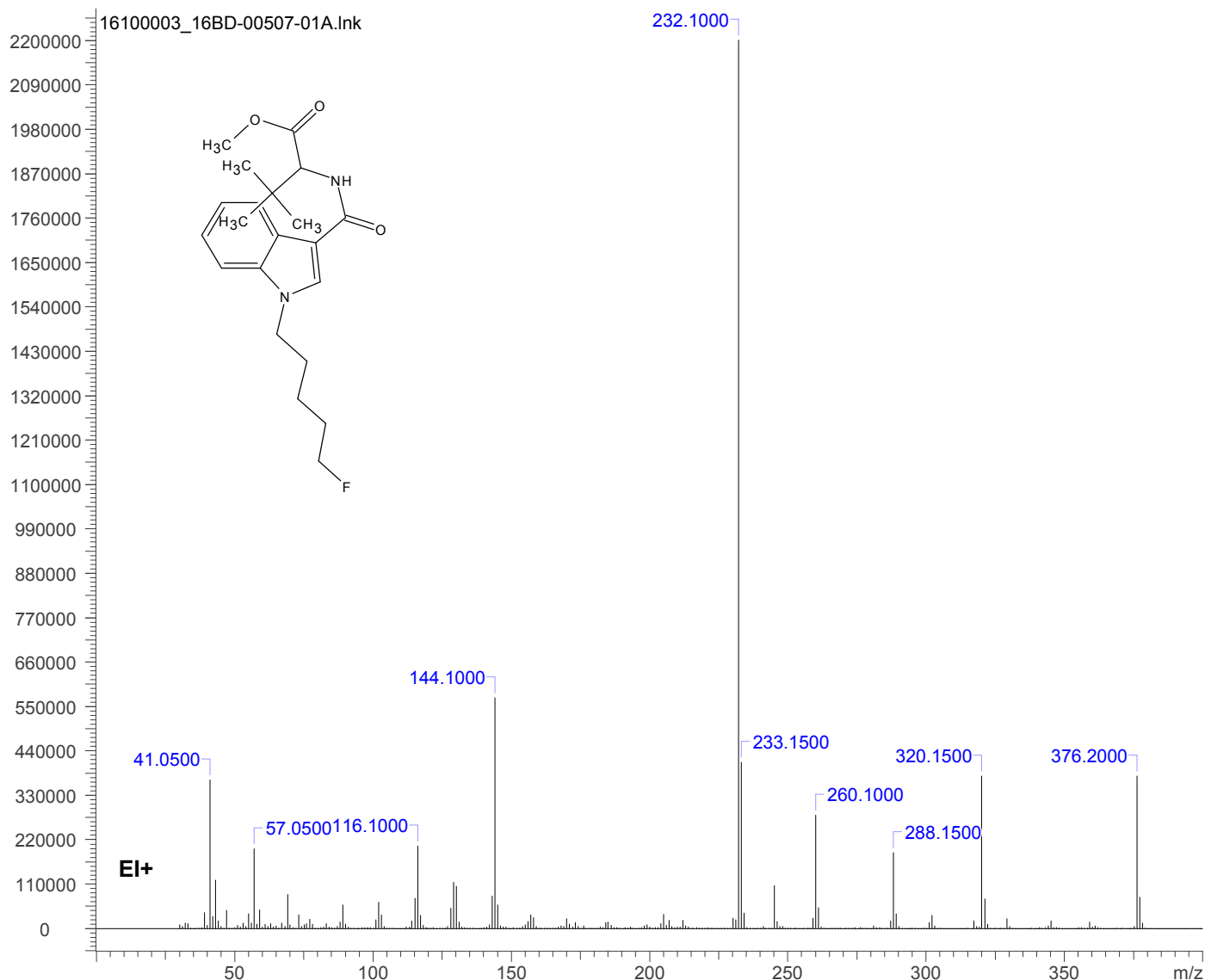
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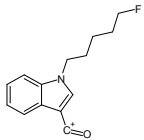
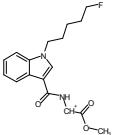
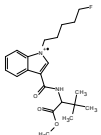
|                                |                      |                        |                                                          |                  |                          |
|--------------------------------|----------------------|------------------------|----------------------------------------------------------|------------------|--------------------------|
| <b>Comment</b>                 | DCM/MeOH             | <b>Count</b>           | 471                                                      | <b>Data Type</b> | Centroided Mass Spectrum |
| <b>Date</b>                    | 12 Oct 2016 17:04:24 | <b>Date Stamp</b>      | 28 Sep 2016 16:14:48                                     |                  |                          |
| <b>Estimated Molecular Ion</b> | 376.2                | <b>File Name</b>       | 16BD-00507-01A 28092016 4 1 Centroided Mass Spectrum EI+ |                  |                          |
| <b>Inlet Model</b>             | Other Probe          | <b>Ion Mode</b>        | EI+                                                      | <b>Operator</b>  | Admin                    |
| <b>Plot Type</b>               | Stick                | <b>Retention Time</b>  | 15.030                                                   | <b>Sample</b>    | 16BD-00507-01A           |
| <b>Scan</b>                    | 4573                 | <b>Separation Type</b> | Gas-Solid Chromatography                                 |                  |                          |
| <b>Spectrum Title</b>          | DCM/MeOH             | <b>Spectrum Type</b>   | MS                                                       | <b>TIC</b>       | 361.92                   |
| <b>Total Signal</b>            | 7964221              |                        |                                                          |                  |                          |



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### Table of fragments

| No. | Fragment   | Structure                                                                           | Formula                                                            | Label                                               | m/z Calc. | TIC Calc. (%) | m/z Exp. | RI Exp. (%) | TIC Exp. (%) |
|-----|------------|-------------------------------------------------------------------------------------|--------------------------------------------------------------------|-----------------------------------------------------|-----------|---------------|----------|-------------|--------------|
| 1   | 1-7,15(+H) |                                                                                     | C <sub>6</sub> H <sub>11</sub> FN(+)                               | M - C <sub>15</sub> H <sub>18</sub> NO <sub>3</sub> | 116.087   | 2.752         | 116.100  | 9.275       | 2.752        |
| 2   | 18-27      |                                                                                     | C <sub>7</sub> H <sub>14</sub> NO <sub>2</sub> (+)                 | M - C <sub>14</sub> H <sub>15</sub> FNO             | 144.102   | 7.834         | 144.100  | 25.984      | 7.834        |
| 3   | 1-17       |    | C <sub>14</sub> H <sub>15</sub> FNO(+)                             | M - C <sub>7</sub> H <sub>14</sub> NO <sub>2</sub>  | 232.113   | 32.498        | 232.100  | 100.000     | 32.498       |
| 4   | 7-27(+H)   |                                                                                     | C <sub>16</sub> H <sub>20</sub> N <sub>2</sub> O <sub>3</sub> (+)  | M - C <sub>5</sub> H <sub>9</sub> F                 | 288.147   | 2.858         | 288.150  | 8.525       | 2.858        |
| 5   | 1-23(+H)   |  | C <sub>17</sub> H <sub>21</sub> FN <sub>2</sub> O <sub>3</sub> (+) | M - C <sub>4</sub> H <sub>8</sub>                   | 320.153   | 5.813         | 320.150  | 17.149      | 5.813        |
| 6   | M          |  | C <sub>21</sub> H <sub>29</sub> FN <sub>2</sub> O <sub>3</sub> (+) | M                                                   | 376.216   | 6.094         | 376.200  | 17.184      | 5.950        |

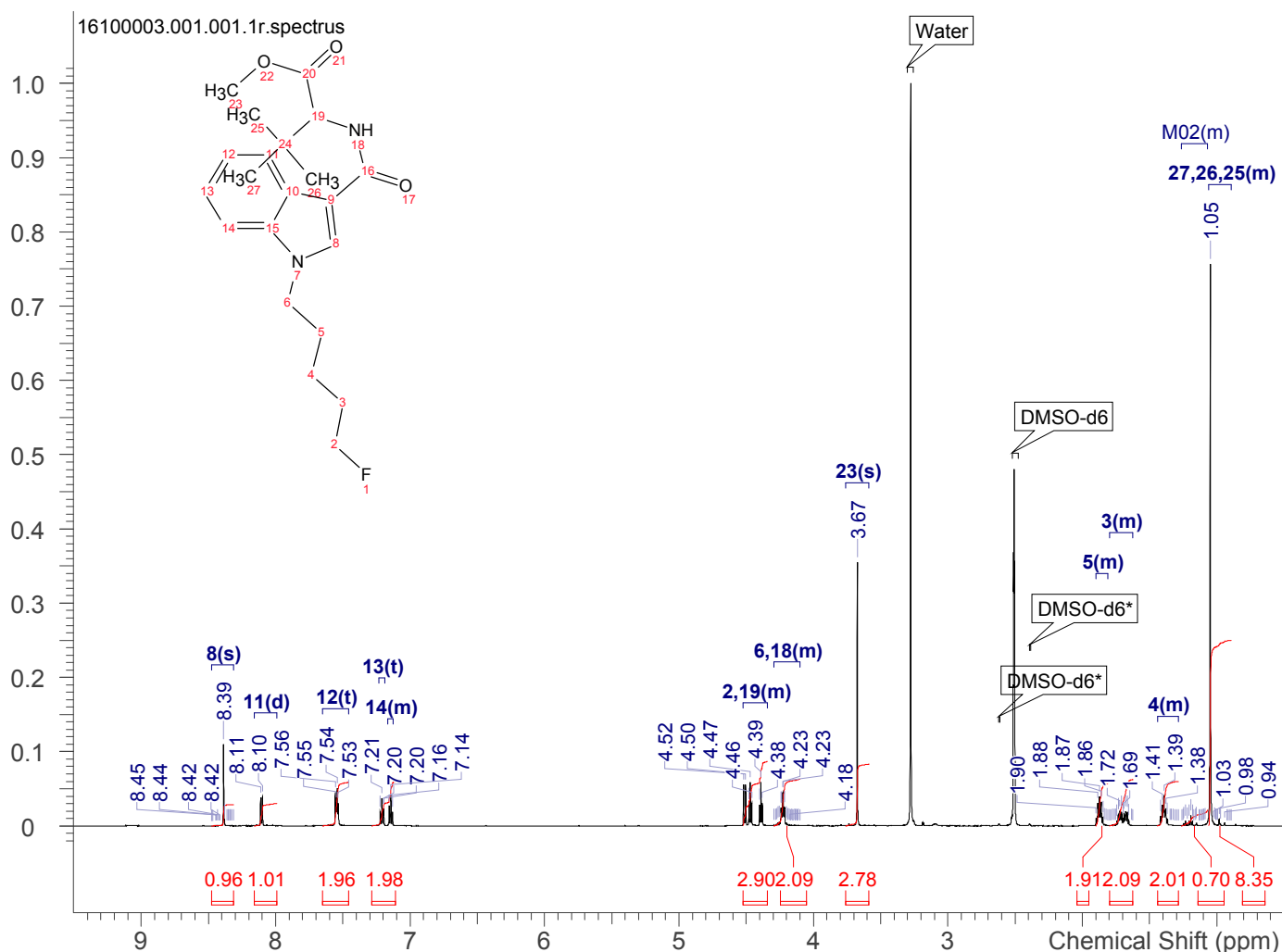
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### Data from BCIM group at JRC, IHCP Ispra

#### NMR spectra

|                        |                                                                                                                                      |                        |                      |
|------------------------|--------------------------------------------------------------------------------------------------------------------------------------|------------------------|----------------------|
| Acquisition Time (sec) | 2.7263                                                                                                                               | Date                   | 07 Oct 2016 11:57:52 |
| Date Stamp             | 07 Oct 2016 11:57:52                                                                                                                 |                        |                      |
| File Name              | \\139.191.6.82\ihcp\$\I01\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\proposed-structure\BCIM_NAS\BCIM_600\16100003\1\PDATA\1\1r |                        |                      |
| Frequency (MHz)        | 600.1300                                                                                                                             | Nucleus                | <sup>1</sup> H       |
| Original Points Count  | 32768                                                                                                                                | Owner                  | nmrsu                |
| Pulse Sequence         | zg30                                                                                                                                 | Receiver Gain          | 23.55                |
| Solvent                | DMSO-d <sub>6</sub>                                                                                                                  | Spectrum Offset (Hz)   | 3706.0515            |
| Sweep Width (Hz)       | 12019.05                                                                                                                             | Temperature (degree C) | 37.001               |
|                        |                                                                                                                                      | Number of Transients   | 64                   |
|                        |                                                                                                                                      | Points Count           | 65536                |
|                        |                                                                                                                                      | SW(cyclical) (Hz)      | 12019.23             |
|                        |                                                                                                                                      | Spectrum Type          | standard             |



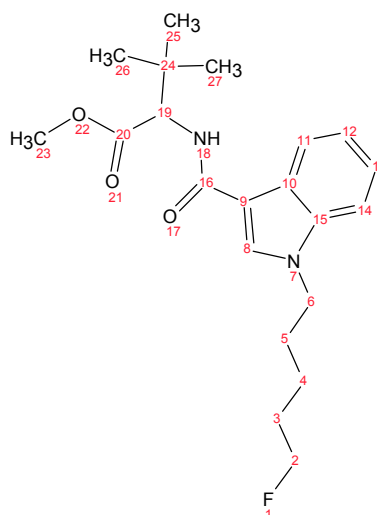
<sup>1</sup>H NMR (600 MHz, DMSO-d<sub>6</sub>) δ ppm 0.89 - 1.06 (m, 8 H) 1.07 - 1.26 (m, 1 H) 1.29 - 1.44 (m, 2 H) 1.62 - 1.80 (m, 2 H) 1.81 - 1.90 (m, 2 H) 3.67 (s, 3 H) 4.10 - 4.29 (m, 2 H) 4.34 - 4.52 (m, 3 H) 7.13 - 7.16 (m, 1 H) 7.21 (t, *J*=7.85 Hz, 1 H) 7.55 (t, *J*=6.70 Hz, 2 H) 8.11 (d, *J*=7.89 Hz, 1 H) 8.39 (s, 1 H)

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Table of assignments and zoomed parts:

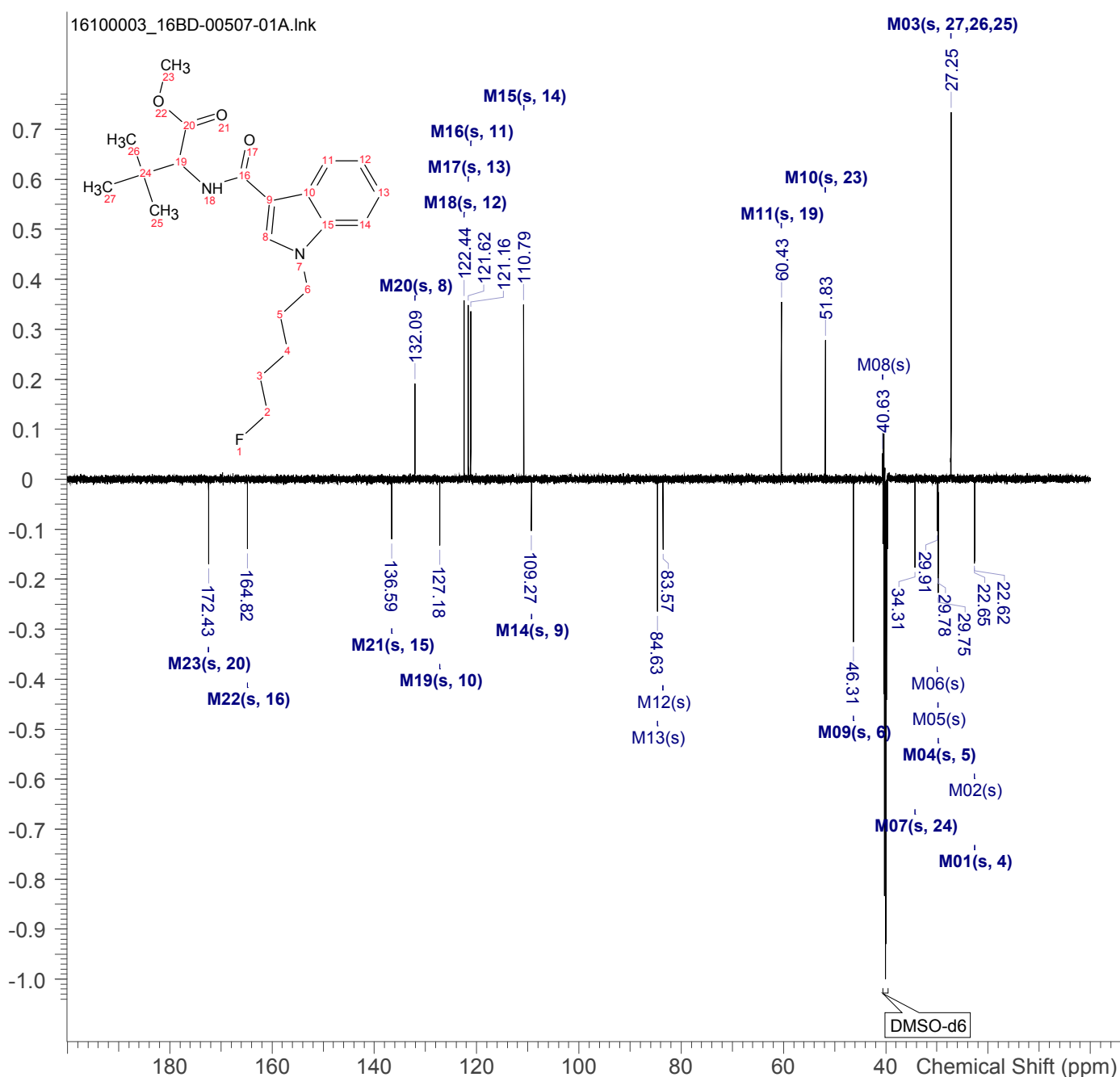
| No. | Atom | Exp. Shift (ppm) | Multiplet |
|-----|------|------------------|-----------|
| 1   | 27   | 1.05             | M01       |
| 2   | 26   | 1.05             | M01       |
| 3   | 25   | 1.05             | M01       |
| 4   | 4    | 1.39             | M03       |
| 5   | 3    | 1.70             | M04       |
| 6   | 5    | 1.87             | M05       |
| 7   | 23   | 3.67             | M06       |
| 8   | 6    | 4.22             | M07       |
| 9   | 18   | 4.22             | M07       |
| 10  | 2    | 4.46             | M08       |
| 11  | 19   | 4.46             | M08       |
| 12  | 14   | 7.14             | M09       |
| 13  | 13   | 7.21             | M13       |
| 14  | 12   | 7.55             | M10       |
| 15  | 11   | 8.11             | M11       |
| 16  | 8    | 8.39             | M12       |



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|                               |                                                                                                                                      |                             |                                                |
|-------------------------------|--------------------------------------------------------------------------------------------------------------------------------------|-----------------------------|------------------------------------------------|
| <b>Acquisition Time (sec)</b> | 0.9044                                                                                                                               | <b>Comment</b>              | 5 mm CPQCI 1H/19F-13C/15N/D Z-GRD Z114073/0012 |
| <b>Date</b>                   | 07 Oct 2016 12:13:20                                                                                                                 | <b>Date Stamp</b>           | 07 Oct 2016 12:13:20                           |
| <b>File Name</b>              | \\139.191.6.82\ihcp\$\101\Bio_Chemical_Interaction_Metabonomics\CLEN2SAND\proposed-structure\BCIM_NAS\BCIM_600\16100003\2\PDATA\1\1r |                             |                                                |
| <b>Frequency (MHz)</b>        | 150.9028                                                                                                                             | <b>Nucleus</b>              | 13C                                            |
| <b>Number of Transients</b>   | 256                                                                                                                                  | <b>Pulse Sequence</b>       | jmod                                           |
| <b>Receiver Gain</b>          | 183.05                                                                                                                               | <b>SW(cyclical) (Hz)</b>    | 36231.88                                       |
| <b>Solvent</b>                | DMSO-d6                                                                                                                              | <b>Spectrum Offset (Hz)</b> | 18108.3359                                     |
| <b>Spectrum Type</b>          | APT                                                                                                                                  | <b>Sweep Width (Hz)</b>     | 36230.78                                       |
| <b>Temperature (degree C)</b> | 37.001                                                                                                                               |                             |                                                |





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Table of assignments and zoomed parts:

| No. | Atom | Exp. Shift (ppm) | Multiplet |
|-----|------|------------------|-----------|
| 1   | 4    | 22.62            | M01       |
| 2   | 27   | 27.25            | M03       |
| 3   | 26   | 27.25            | M03       |
| 4   | 25   | 27.25            | M03       |
| 5   | 5    | 29.75            | M04       |
| 6   | 24   | 34.31            | M07       |
| 7   | 6    | 46.31            | M09       |
| 8   | 23   | 51.83            | M10       |
| 9   | 19   | 60.43            | M11       |
| 10  | 9    | 109.27           | M14       |
| 11  | 14   | 110.79           | M15       |
| 12  | 11   | 121.16           | M16       |
| 13  | 13   | 121.62           | M17       |
| 14  | 12   | 122.44           | M18       |
| 15  | 10   | 127.18           | M19       |
| 16  | 8    | 132.09           | M20       |
| 17  | 15   | 136.59           | M21       |
| 18  | 16   | 164.82           | M22       |
| 19  | 20   | 172.43           | M23       |

