

FIG. 1A. Psilocybin

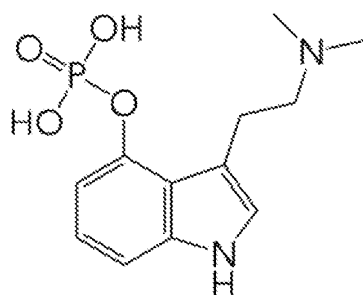


FIG. 1B. Psilocin

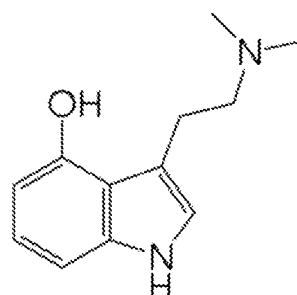


FIG. 1C. Psilocin-O-glucuronide

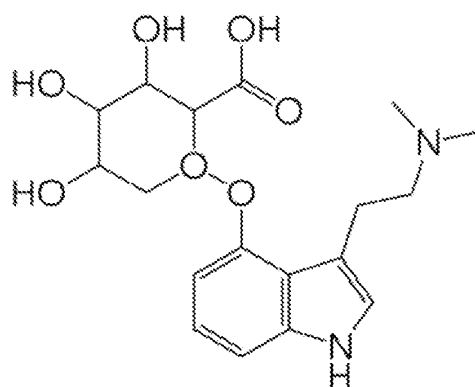


FIG. 1D. 4-HIAA

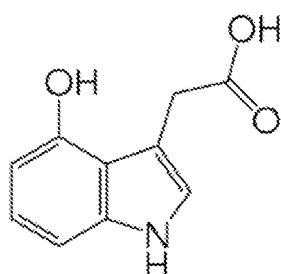


FIG. 1E. 4-HT

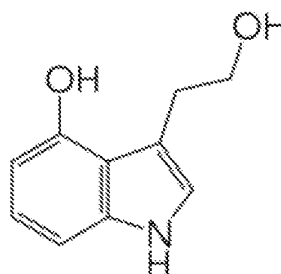


FIG. 2

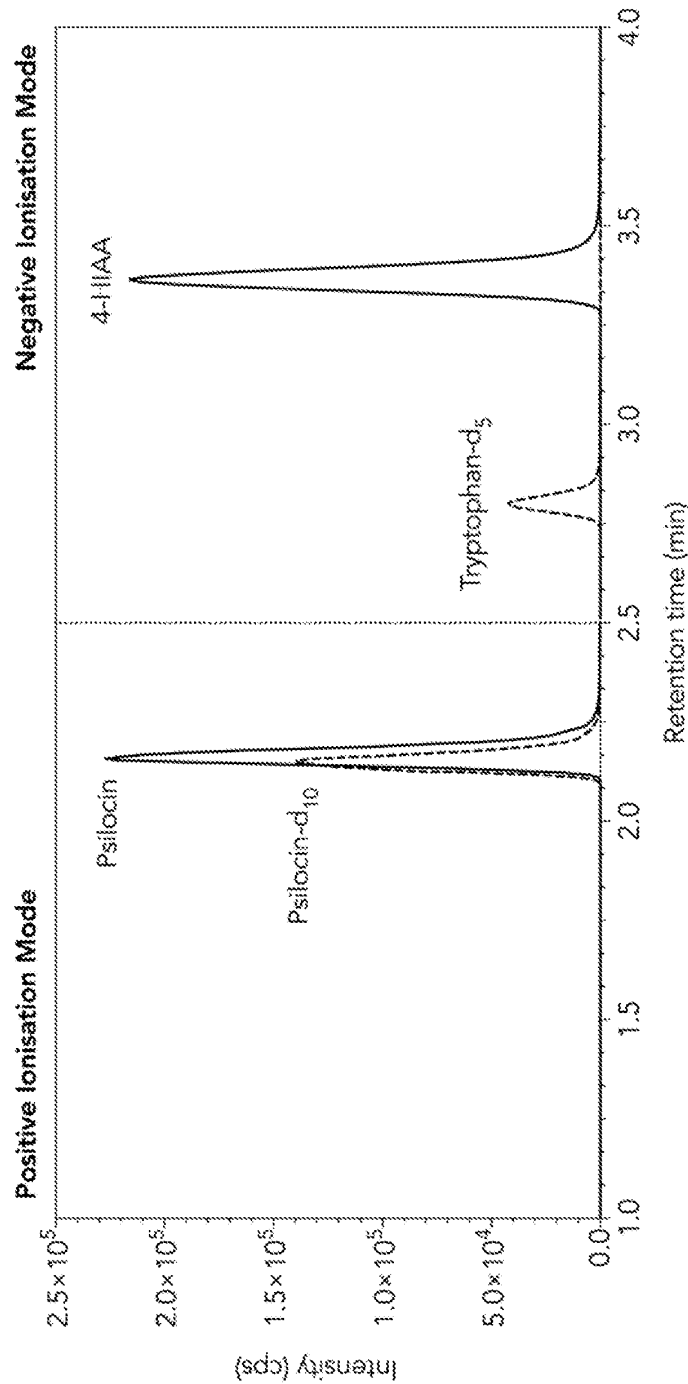


FIG. 3

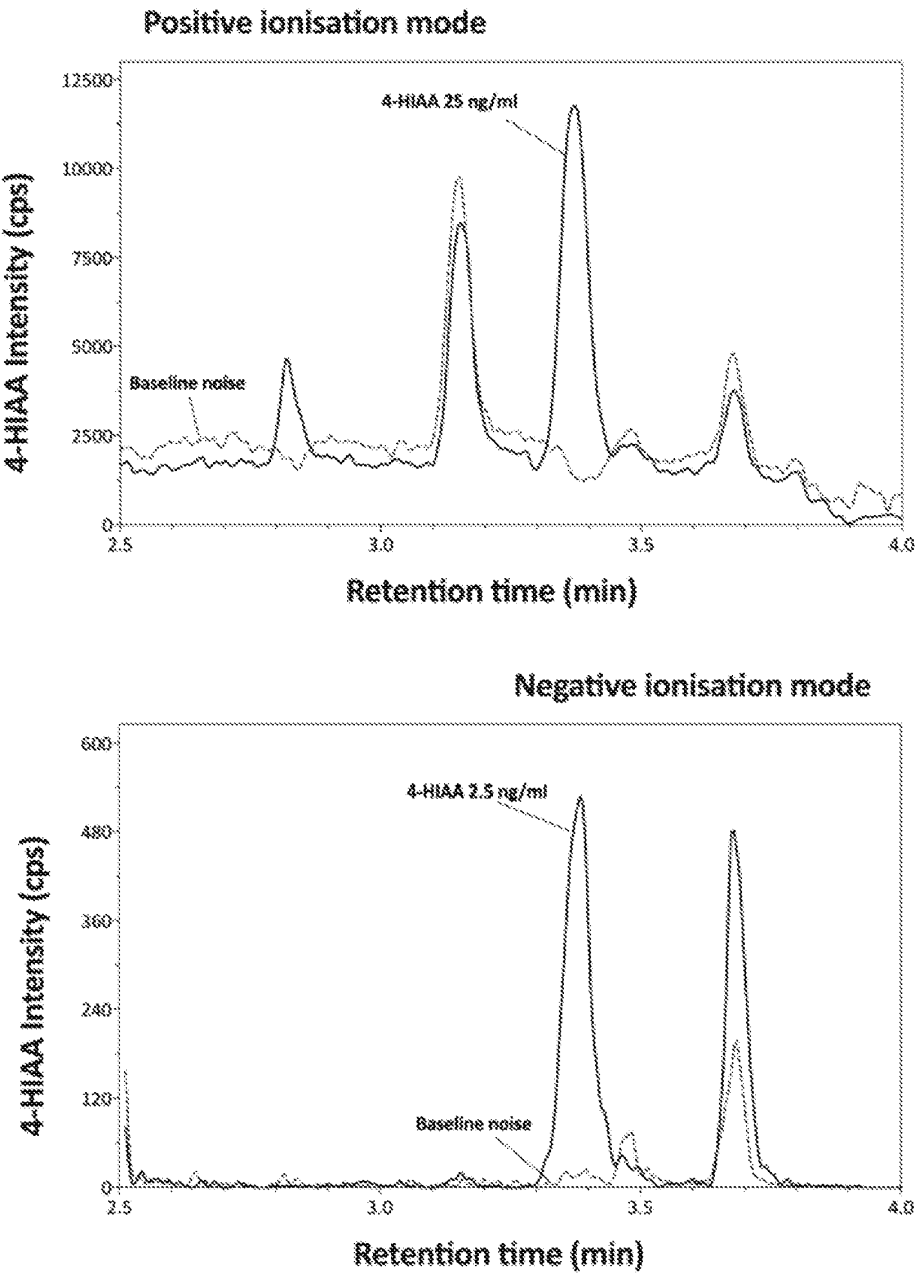


FIG. 4A

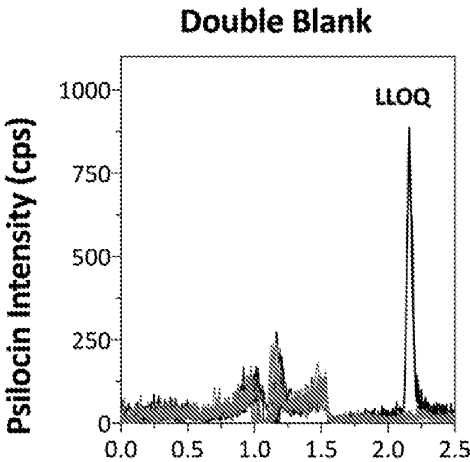


FIG. 4B

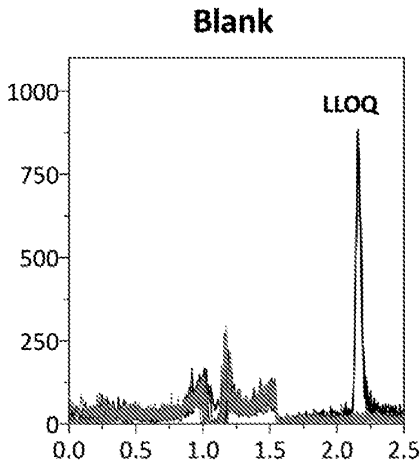


FIG. 4C

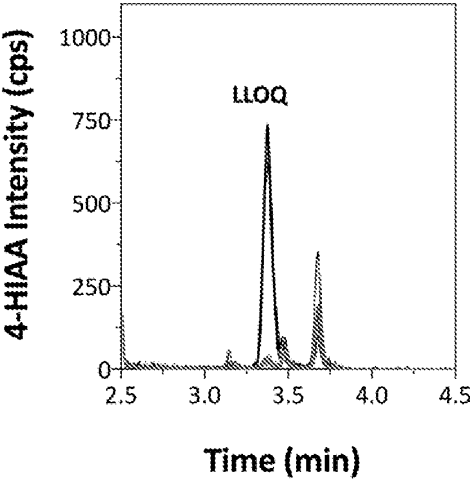
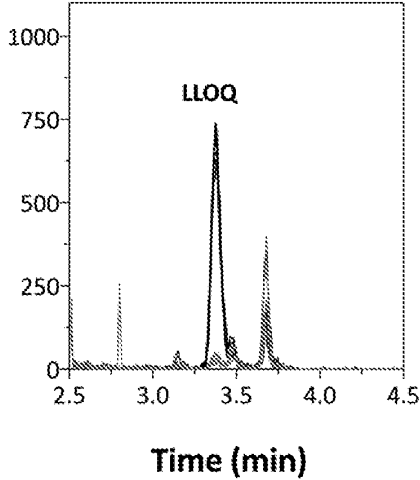


FIG. 4D



## FIG. 5A

Table: The selectivity of psilocin and 4-HIAA in human plasma spiked to LLOQ level (0.25 ng/ml psilocin or 2.5 ng/ml 4-HIAA) compared to signals of double blank and blank.

| Condition                                                                    | Subject 1     |               | Subject 2     |               | Subject 3     |               | Subject 4     |               |
|------------------------------------------------------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
|                                                                              | Area (counts) | % of the LLOQ | Area (counts) | % of the LLOQ | Area (counts) | % of the LLOQ | Area (counts) | % of the LLOQ |
| Psilocin                                                                     |               |               |               |               |               |               |               |               |
| LLOQ                                                                         | 2.01E+03      | -             | 2.02E+03      | -             | 2.13E+03      | -             | 2.22E+03      | -             |
| Double Blank                                                                 | 8.17E+01      | 4.1           | 6.52E+01      | 3.2           | 6.41E+01      | 3.0           | 7.12E+01      | 3.2           |
| Blank                                                                        | 7.10E+01      | 3.5           | 5.07E+01      | 2.5           | 5.72E+01      | 2.7           | 7.43E+01      | 3.3           |
| 4-HIAA                                                                       |               |               |               |               |               |               |               |               |
| LLOQ                                                                         | 2.27E+03      | -             | 2.28E+03      | -             | 2.31E+03      | -             | 2.37E+03      | -             |
| Double Blank                                                                 | 7.50E+01      | 3.3           | 7.21E+01      | 3.2           | 9.75E+01      | 4.2           | 9.85E+01      | 4.2           |
| Blank                                                                        | 6.97E+01      | 3.1           | 7.20E+01      | 3.2           | 1.20E+02      | 5.2           | 1.02E+02      | 4.3           |
| Condition                                                                    | Subject 5     |               | Subject 6     |               | Subject 7     |               |               |               |
|                                                                              | Area (counts) | % of the LLOQ | Area (counts) | % of the LLOQ | Area (counts) | % of the LLOQ |               |               |
| Psilocin                                                                     |               |               |               |               |               |               |               |               |
| LLOQ                                                                         | 2.02E+03      | -             | 2.10E+03      | -             | 2.25E+03      | -             |               |               |
| Double Blank                                                                 | 8.11E+01      | 4.0           | 5.65E+01      | 2.7           | 6.27E+01      | 2.8           |               |               |
| Blank                                                                        | 7.33E+01      | 3.6           | 6.44E+01      | 3.1           | 6.12E+01      | 2.7           |               |               |
| 4-HIAA                                                                       |               |               |               |               |               |               |               |               |
| LLOQ                                                                         | 2.31E+03      | -             | 2.30E+03      | -             | 2.35E+03      | -             |               |               |
| Double Blank                                                                 | 8.75E+01      | 3.8           | 1.13E+02      | 4.9           | 6.41E+01      | 2.7           |               |               |
| Blank                                                                        | 1.17E+02      | 5.1           | 1.27E+02      | 5.5           | 6.52E+01      | 2.8           |               |               |
| 4-HIAA, 4-hydroxy-indole-3-acetic acid; LLOQ, lower level of quantification. |               |               |               |               |               |               |               |               |

## FIG. 5B

Table: The selectivity of psilocin and 4-HIAA in human plasma spiked to LLOQ (0.25 ng/ml psilocin or 2.5 ng/ml 4-HIAA).

| Subject                                                                                                   | Psilocin                |            | 4-HIAA                  |            |
|-----------------------------------------------------------------------------------------------------------|-------------------------|------------|-------------------------|------------|
|                                                                                                           | C <sub>calculated</sub> | Accuracy % | C <sub>calculated</sub> | Accuracy % |
| 1                                                                                                         | 0.244                   | 97.5       | 2.176                   | 87.1       |
| 2                                                                                                         | 0.238                   | 95.3       | 2.116                   | 84.7       |
| 3                                                                                                         | 0.249                   | 99.5       | 2.108                   | 84.3       |
| 4                                                                                                         | 0.264                   | 106        | 2.171                   | 86.8       |
| 5                                                                                                         | 0.252                   | 101        | 2.076                   | 83.0       |
| 6                                                                                                         | 0.256                   | 110        | 2.064                   | 82.6       |
| 7                                                                                                         | 0.275                   | 102        | 2.107                   | 84.3       |
| Mean 1-7                                                                                                  | 0.254                   |            | 2.117                   |            |
| Accuracy ± CV%                                                                                            | 102 ± 4.9               |            | 84.7 ± 2.0              |            |
| 4-HIAA, 4-hydroxy-indole-3-acetic acid; LLOQ, lower level of quantification; CV%, Coefficient of variance |                         |            |                         |            |

FIG. 6

Table: The stability of psilocin and 4-HIAA determined following three freeze-thaw cycles (freeze/thaw stability), storage at room temperature for 8 h (bench-top ), and 1 month at -20°C (one month).

| Storage stability  | $C_{\text{nominal}}$<br>(ng/ml) | Psilocin                  |                          | $C_{\text{nominal}}$<br>(ng/ml) | 4-HIAA                    |                          |
|--------------------|---------------------------------|---------------------------|--------------------------|---------------------------------|---------------------------|--------------------------|
|                    |                                 | $C_{\text{Ø}}$<br>(ng/ml) | Accuracy<br>$\pm$ CV (%) |                                 | $C_{\text{Ø}}$<br>(ng/ml) | Accuracy<br>$\pm$ CV (%) |
| Freeze/thaw        | 0.25                            | 0.269                     | 108 $\pm$ 8.2            | 2.5                             | 2.72                      | 109 $\pm$ 2.8            |
|                    | 0.50                            | 0.533                     | 107 $\pm$ 3.5            | 5.0                             | 5.00                      | 100 $\pm$ 5.4            |
|                    | 10                              | 10.8                      | 108 $\pm$ 4.5            | 100                             | 106                       | 106 $\pm$ 5.3            |
|                    | 50                              | 52.6                      | 105 $\pm$ 3.3            | 500                             | 524                       | 105 $\pm$ 2.1            |
| Bench-top          | 0.25                            | 0.258                     | 103 $\pm$ 7.1            | 2.5                             | 2.45                      | 98.2 $\pm$ 4.9           |
|                    | 0.50                            | 0.534                     | 107 $\pm$ 3.6            | 5.0                             | 4.74                      | 94.8 $\pm$ 4.3           |
|                    | 10                              | 10.7                      | 107 $\pm$ 3.5            | 100                             | 110                       | 110 $\pm$ 3.3            |
|                    | 50                              | 49.0                      | 98.1 $\pm$ 3.6           | 500                             | 481                       | 96.1 $\pm$ 1.8           |
| One month at -20°C | 0.25                            | 0.259                     | 104 $\pm$ 5.9            | 2.5                             | 2.69                      | 108 $\pm$ 6.6            |
|                    | 0.50                            | 0.542                     | 108 $\pm$ 3.9            | 5.0                             | 5.16                      | 103 $\pm$ 5.6            |
|                    | 10                              | 10.6                      | 106 $\pm$ 4.0            | 100                             | 107                       | 107 $\pm$ 3.7            |
|                    | 50                              | 49.4                      | 98.7 $\pm$ 4.2           | 500                             | 487                       | 97.5 $\pm$ 5.6           |

$C_{\text{nominal}}$ , theoretical concentration;  $C_{\text{Ø}}$ , average concentration of seven samples; CV%, Coefficient of variance; 4-HIAA, 4-hydroxy-indole-3-acetic acid.

FIG. 7A

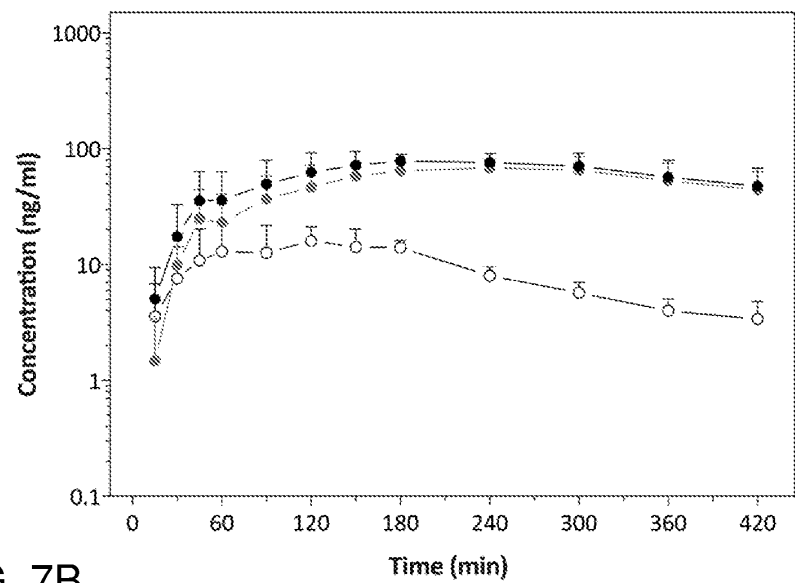


FIG. 7B

