# Innovate Phytoceuticals Cannabis Method Validation Summary

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# 1) SCOPE & PURPOSE

This document is designed to provide a summary of Innovate Phytoceuticals' (INVP) internal validation for cannabis analytical testing methods. All data included herein are examples of validation data obtained by INVP at its analytical laboratory, located at 2-3485 Velocity Avenue, Kelowna BC, V1V 3C2.

The purpose of this document is to provide our clients with confidence that the methods we offer have been validated and found to be effective. It also provides confirmation that our analytical methods are capable of meeting all Health Canada requirements for cannabis testing.

Should you have any questions regarding the contents of this document, please do not hesitate to contact us.

# 2) POTENCY & CANNABINOID TESTING

## 2.1 Method Details

I. Analysis of Cannabinoids using Ultra-High Performance Liquid Chromatography – Diode Array Detection (UHPLC-DAD).
II. This method is a modified United Nations Office on Drugs and Crime (UNODC) method based on the chromatography principles of the United States Pharmacopoeia (USP) General Chapter <621>.
III. Complete method details are outlined in LAB-SOP-ANA-001 Analysis of Cannabinoids from Different Matrices by UHPLC-DAD.
IV. This method is capable of identifying and quantifying THC, THCA, CBD, CBDA, CBDVA, THCV, CBG, CBGA, CBN, CBL, $\Delta^8$-THC, CBDV, THCVA, CBC, and CBCA .

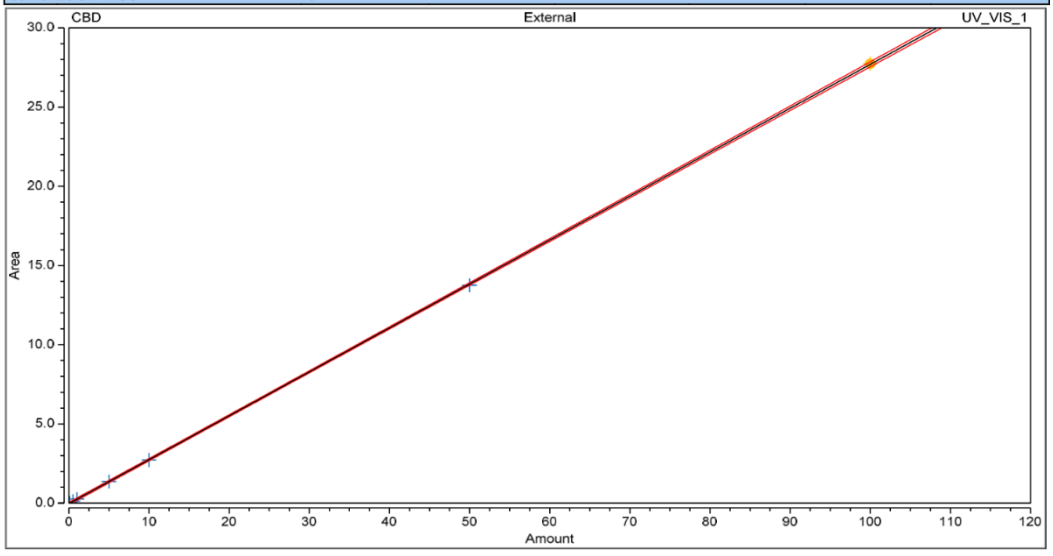
## 2.2 Linearity & Range

Figure 2.2.1 provides a seven-point calibration curve for CBD, as an example:

**Calibration**

| Calibration Details | CBD | | |
|---|---|---|---|
| *Calibration Type* | Lin, WithOffset | *Offset (C0)* | -0.0289 |
| *Evaluation Type* | Area | *Slope (C1)* | 0.2773 |
| *Number of Calibration Points* | 7 | *Curve (C2)* | 0.0000 |
| *Number of disabled Calibration Points* | 0 | *R-Square* | 1.0000 |

**Calibration Plot** **CBD**




I. Calibration curves were completed on a seven-point range (0.1ppm, 0.5ppm, 1ppm, 5ppm, 10ppm, 50ppm & 100ppm). All cannabinoids have a range from 0.1-100ppm.
II. The average $R^2$ value for all cannabinoids was 0.9999.

III. The average relative standard deviation (RSD) for all cannabinoid calibration curves was 0.9237%.

## 2.3 Accuracy & Precision

Table 2.3.1 shows a dried flower cannabis matrix run 3 times for repeatability of cannabinoids:

| CANNABINOID | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| CBD | 2.130 | 2.071 | 1.973 | 2.058 | 0.079 | 3.83% |
| CBDA | 0.653 | 0.630 | 0.600 | 0.628 | 0.027 | 4.30% |
| THC | 11.162 | 11.109 | 11.160 | 11.144 | 0.030 | 0.27% |
| THCA | 125.52 | 125.42 | 125.40 | 125.45 | 0.064 | 0.05% |
| THCV | 2.547 | 2.461 | 2.449 | 2.486 | 0.053 | 2.13% |
| CBGA | 1.569 | 1.592 | 1.603 | 1.588 | 0.017 | 1.07% |
| CBN | 0.445 | 0.455 | 0.454 | 0.451 | 0.005 | 1.11% |

*All values shown are in ppm, unless otherwise indicated

Table 2.3.2 shows a cannabis dried flower matrix spiked with 10ppm CBDA:

| CANNABINOID | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| CBDA | 9.208 | 9.024 | 9.103 | 9.11 | 0.094 | 1.04% | 91.10% |
| THCA | 124.35 | 124.81 | 125.39 | 124.85 | 0.520 | 0.42% | N/A |
| THC | 9.65 | 9.69 | 9.65 | 9.66 | 0.023 | 0.24% | N/A |

*All values shown are in ppm, unless otherwise indicated

Table 2.3.3 shows results from CBG standard injected 9 times at 50ppm, no matrix:

| CANNABINOID | MAX | MIN | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|
| CBG | 50.711 | 50.050 | 50.392 | 0.232 | 0.46% | 100.78% |

*All values shown are in ppm, unless otherwise indicated

Table 2.3.4 shows cannabis oil matrices with expected values of 20,000ppm of CBD, 20,000ppm CBG & 4,000ppm of THC:

| CANNABINOID | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| CBD | 19686.9 | 19915.7 | 20398.2 | 20000.3 | 363.14 | 1.82% | 100.0% |
| CBG | 22213.9 | 22423.1 | 22567.8 | 22401.6 | 177.93 | 0.79% | 112.0% |
| THC | 3944.6 | 3744.8 | 3954.7 | 3881.4 | 118.4 | 3.05% | 97.04% |

*All values shown are in ppm, unless otherwise indicated

Table 2.3.5 shows cannabis gummy matrices with expected values of 580ppm THC & <70ppm CBD.

| CANNABINOID | RUN 1 | RUN 2 | RUN 3 | RUN 4 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|---|
| THC | 553.74 | 544.55 | 543.96 | 543.05 | 546.33 | 4.982 | 0.91% | 94.19% |
| CBD | 34.45 | 37.81 | 37.59 | 35.89 | 36.43 | 1.58 | 4.33% | N/A |

*All values shown are in ppm, unless otherwise indicated

Table 2.3.6 shows intermediate precision data for several cannabinoids in a dried flower matrix, run by different analysts on different days:

| ANALYST 1 | THC | THCA-A | CBD | CBGA | THCV |
|---|---|---|---|---|---|
| Run 1 (ppm) | 10.0203 | 114.3655 | 2.0731 | 1.4572 | 2.2307 |
| Run 2 (ppm) | 9.8463 | 113.9518 | 1.9676 | 2.0804 | 2.1616 |
| Run 3 (ppm) | 10.3044 | 114.0934 | 1.8514 | 1.4431 | 2.0909 |
| Mean | 10.057 | 114.1369 | 1.9640 | 1.6602 | 2.1611 |
| SD (ppm) | 0.2312 | 0.2102 | 0.1108 | 0.3639 | 0.0699 |
| RSD | 2.3% | 0.18% | 5.65% | 21.92% | 3.23% |
| **ANALYST 2** | **THC** | **THCA-A** | **CBD** | **CBGA** | **THCV** |
| Run 1 (ppm) | 11.1615 | 125.5157 | 2.1299 | 1.5692 | 2.5472 |
| Run 2 (ppm) | 11.1087 | 125.4230 | 2.0708 | 1.5923 | 2.4613 |
| Run 3 (ppm) | 11.1598 | 125.3955 | 1.9727 | 1.6031 | 2.4486 |
| Mean | 11.1433 | 125.4447 | 2.0578 | 1.5882 | 2.4857 |
| SD (ppm) | 0.03 | 0.062 | 0.079 | 0.0173 | 0.0536 |
| RSD | 0.27% | 0.05% | 3.86% | 1.09% | 2.16% |

*All values in ppm, unless otherwise indicated

I. This dataset is an example, and it has been repeated for all cannabinoids across several matrices.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 2.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The LoQ for each cannabinoid in this method is 0.1ppm.
III. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.
IV. The LoD for each cannabinoid in this method is 0.01ppm.

## 2.5 Summary

Table 2.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|

| | |
|---|---|
| Average $R^2$ | 0.9999 |
| Calibration Range | 0.1-100ppm |
| LoQ | 0.1ppm |
| LoD | 0.01ppm |
| Specificity | Tested in various matrices, shown to be specific. Only CRMs used for calibrations. |
| Accuracy | ±2%, with an RSD <1% |
| Repeatability | RSD <1% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times across several matrices. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Method tested with different UHPLC/HPLC systems, different columns, different detectors, liquid phases, etc. Additional experiments are currently in progress. |

## 2.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

# 3) HEAVY METAL TESTING

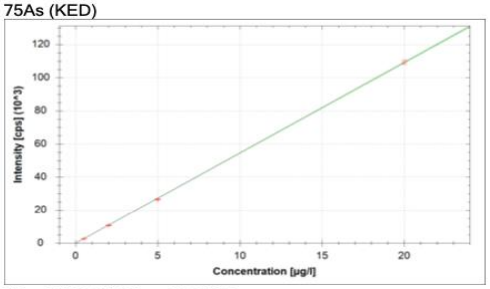
## 3.1 Method Details

I. Analysis of heavy metals using Inductively Coupled Plasma Mass Spectrometry (ICP-MS).
II. This method is derived from the United States Pharmacopoeia (USP) General Chapters <232> and <233>.
III. Complete method details are outlined in LAB-SOP-ANA-007 Analysis of Heavy Metals in Cannabis Samples by ICP-MS.

## 3.2 Linearity & Range

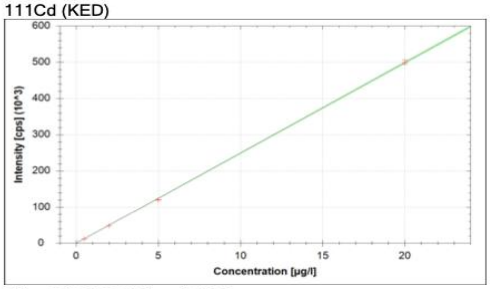
Figure 3.2.1 below shows calibration curves for Arsenic (As), Cadmium (Cd), Mercury (Hg) and Lead (Pb)

75As (KED)



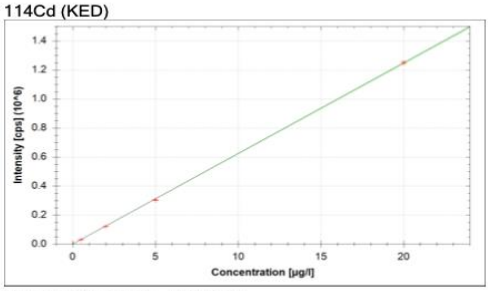

f(x) = 5456.3179*x + 23.3195
R² = 0.9999
BEC = 0.004 µg/l
LoD = 0.0021 µg/l

111Cd (KED)



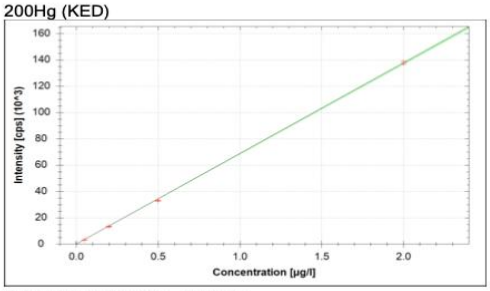

f(x) = 24937.0682*x + 4.0025
R² = 0.9998
BEC = 0.000 µg/l
LoD = 0.0002 µg/l

114Cd (KED)



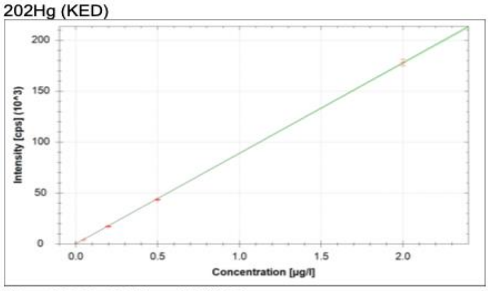

f(x) = 62401.8652*x + 40.0138
R² = 0.9999
BEC = 0.001 µg/l
LoD = 0.0004 µg/l

200Hg (KED)



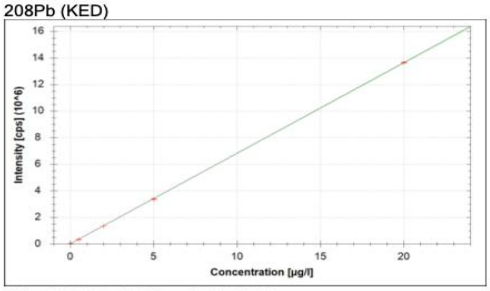

f(x) = 68757.2907*x + 27.6302
R² = 0.9998
BEC = 0.000 µg/l
LoD = 0.0002 µg/l

202Hg (KED)




f(x) = 88828.9354*x + 40.4125
R² = 0.9999
BEC = 0.000 µg/l
LoD = 0.0004 µg/l

208Pb (KED)




f(x) = 681290.1868*x + 9403.1486
R² = 1.0000
BEC = 0.014 µg/l
LoD = 0.0017 µg/l

I. The average $R^2$ value for all heavy metals was 0.9997 across a range of 5-50ppb.

## 3.3 Accuracy & Precision

Table 3.3.1 below shows repeatability data for Arsenic from a dried cannabis flower matrix injected 3 times with 3 repeats per injection:

| HEAVY METAL | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| $^{75}$As | 72.00 | 70.57 | 68.59 | 70.39 | 1.71 | 2.43% |
| $^{75}$As | 68.62 | 71.09 | 68.33 | 69.35 | 1.52 | 2.19% |
| $^{75}$As | 65.10 | 64.01 | 69.35 | 66.15 | 2.82 | 4.26% |
| Average | | | | 68.63 | 2.21 | 3.22% |

*All values in ppb, unless otherwise indicated

Table 3.3.2 below shows repeatability data for Cadmium from a dried cannabis flower matrix injected 3 times with 3 repeats per injection:

| HEAVY METAL | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| $^{111}$Cd | 173.01 | 181.19 | 170.82 | 175.01 | 5.46 | 3.12% |
| $^{111}$Cd | 169.56 | 177.86 | 181.87 | 176.43 | 6.28 | 3.56% |
| $^{111}$Cd | 184.23 | 174.68 | 178.34 | 179.08 | 4.82 | 2.69% |
| Average | | | | 176.84 | 2.07 | 1.17% |

*All values in ppb, unless otherwise indicated

Table 3.3.3 below shows repeatability data for Mercury from a dried cannabis flower matrix injected 3 times with 3 repeats per injection:

| HEAVY METAL | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| $^{202}$Hg | 2.65 | 2.77 | 2.85 | 2.76 | 0.10 | 3.62% |
| $^{202}$Hg | 2.78 | 2.74 | 2.74 | 2.75 | 0.02 | 0.73% |
| $^{202}$Hg | 2.79 | 2.80 | 2.62 | 2.74 | 0.10 | 3.65% |
| Average | | | | 2.75 | 0.01 | 0.36% |

*All values in ppb, unless otherwise indicated

Table 3.3.4 below shows repeatability data for Lead from a dried cannabis flower matrix injected 3 times with 3 repeats per injection:

| HEAVY METAL | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| $^{208}$Pb | 26.54 | 26.36 | 27.30 | 26.73 | 0.49 | 1.83% |
| $^{208}$Pb | 27.53 | 27.92 | 27.87 | 27.78 | 0.21 | 0.76% |
| $^{208}$Pb | 28.51 | 28.47 | 28.04 | 28.34 | 0.26 | 0.92% |
| Average | | | | 27.62 | 0.82 | 2.97% |

*All values in ppb, unless otherwise indicated

Table 3.3.5 shows intermediate precision data for a sample of dried flower cannabis, run by different analysts on different days:

| ANALYST 1 | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| $^{75}$As | 56.76 | 55.46 | 56.59 | 56.27 | 0.71 | 1.26% |
| $^{111}$Cd | 107.69 | 113.49 | 110.23 | 110.47 | 2.91 | 2.63% |
| $^{202}$Hg | 3.19 | 3.19 | 3.63 | 3.34 | 0.25 | 7.49% |
| $^{208}$Pb | 27.79 | 27.84 | 27.64 | 27.77 | 0.10 | 0.36% |
| **ANALYST 2** | **RUN 1** | **RUN 2** | **RUN 3** | **MEAN** | **SD** | **RSD** |
| $^{75}$As | 48.15 | 51.11 | 44.59 | 47.95 | 3.26 | 6.79% |
| $^{111}$Cd | 130.77 | 132.86 | 129.58 | 131.07 | 1.66 | 1.27% |
| $^{202}$Hg | 2.70 | 2.69 | 2.91 | 2.77 | 0.12 | 4.33% |

| | | | | | | |
|---|---|---|---|---|---|---|
| $^{208}Pb$ | 38.42 | 37.07 | 38.21 | 37.90 | 0.73 | 1.93% |

*All values in ppb, unless otherwise indicated

I. These experiments have been repeated across multiple cannabis matrices, including oils, extracts, and edibles.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

### 3.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The LoQ for each heavy metal in this method is 1ppb, except for Mercury which has an LoQ of 0.1ppb.
III. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.
IV. The LoD for each heavy metal in this method is 0.01ppb.

### 3.5 Summary

Table 3.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Average $R^2$ | 0.9997 |
| Calibration Range | 5-50ppb |
| LoQ | 1ppb (0.1ppb for Hg) |
| LoD | 0.01ppb |
| Specificity | Tested in various matrices, shown to be specific. Only CRMs used for calibrations. |
| Accuracy | ±2%, with an RSD <5% |
| Repeatability | RSD <0.1-2% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times across several matrices. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Method tested with different extraction solvents, different pH, etc. Additional experiments are currently in progress. |

## 3.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

# 4) PESTICIDE ANALYSIS ON GC-MS

## 4.1 Method Details

I. Analysis of pesticide residues by Gas Chromatography – Mass Spectrometry (GC-MS)
II. This method is derived from the United States Pharmacopoeia (USP) General Chapter <561>.
III. Complete method details are outlined in LAB-SOP-ANA-004 Pesticide Residues Analysis in Cannabis and Cannabis Products by GC-MS.
IV. This method is capable of identifying 48 pesticide active ingredients included on the "Mandatory cannabis testing for pesticide active ingredients – List and Limits" published by Health Canada, and quantifying 11 of them.
V. The remaining 85 pesticide active ingredients can be quantified by LC-MS (see Section 5 of this document).
VI. The complete list of pesticide active ingredients that are quantifiable with this method can be found in Appendix A.

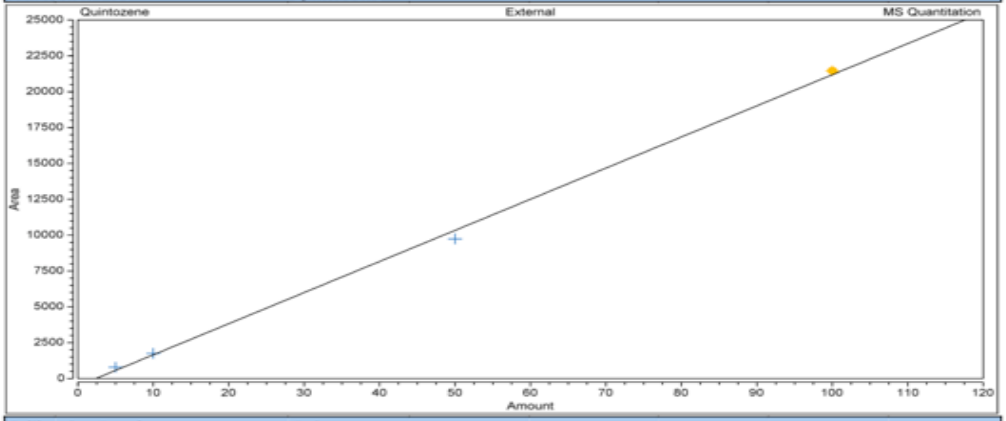
## 4.2 Linearity & Range

I. This method has a low calibration range (5-100ppb) and a high calibration range (100-2000ppb).

Figure 4.2.1 below shows the low calibration range for Quintozene, as an example:

| Calibration Details | Quintozene | Matrix_5ppb-to-2000ppb | |
|---|---|---|---|
| Calibration Type | Lin, WithOffset | Offset (C0) | -516.6755 |
| Evaluation Type | Area | Slope (C1) | 217.0373 |
| Number of Calibration Points | 4 | Curve (C2) | 0.0000 |
| Number of disabled Calibration Points | 0 | R-Square | 0.99824 |

Calibration Plot — Quintozene



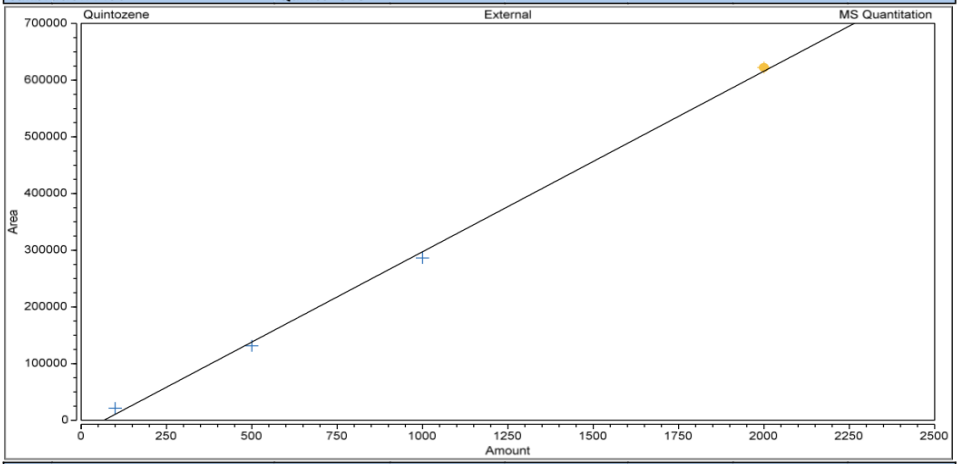

Calibration Results — Quintozene

| No. | Injection Name | Calibration Level | X Value MS Quantitation Peak Quintozene | Y Value MS Quantitation Peak Quintozene | Y Value MS Quantitation Peak Quintozene | Area counts*min MS Quantitation Peak Quintozene | Height counts MS Quantitation Peak Quintozene |
|---|---|---|---|---|---|---|---|
| 4 | 5 ppb | 1 | 5.0000 | 786.8494 | 786.8494 | 786.849 | 30701.749 |
| 5 | 10 ppb | 2 | 10.0000 | 1752.4060 | 1752.4060 | 1752.406 | 68043.468 |
| 6 | 50 ppb | 3 | 50.0000 | 9742.6693 | 9742.6693 | 9742.669 | 376578.266 |
| 7 | 100 ppb | 4 | 100.0000 | 21462.5236 | 21462.5236 | 21462.524 | 842242.165 |

Figure 4.2.2 below shows the high calibration range for Quintozene, as an example:

| Calibration Details | Quintozene | Matrix 5ppb-to-2000ppb | |
|---|---|---|---|
| *Calibration Type* | Lin, WithOffset | *Offset (C0)* | -21319.2339 |
| *Evaluation Type* | Area | *Slope (C1)* | 318.5908 |
| *Number of Calibration Points* | 4 | *Curve (C2)* | 0.0000 |
| *Number of disabled Calibration Points* | 0 | *R-Square* | 0.99843 |

**Calibration Plot** **Quintozene**




**Calibration Results** **Quintozene**

| No. | Injection Name | Calibration Level | X Value MS Quantitation Peak Quintozene | Y Value MS Quantitation Peak Quintozene | Y Value MS Quantitation Peak Quintozene | Area counts*min MS Quantitation Peak Quintozene | Height counts MS Quantitation Peak Quintozene |
|---|---|---|---|---|---|---|---|
| 7 | 100 ppb | 4 | 100.0000 | 21462.5236 | 21462.5236 | 21462.524 | 842242.165 |
| 8 | 500 ppb | 5 | 500.0000 | 131339.0075 | 131339.0075 | 131339.007 | 5130299.702 |
| 9 | 1000 ppb | 6 | 1000.0000 | 286474.1205 | 286474.1205 | 286474.121 | 11013566.554 |
| 10 | 2000 ppb | 7 | 2000.0000 | 622374.1143 | 622374.1143 | 622374.114 | 24133879.357 |

II. Calibrations are available for all 11 quantifiable pesticide active ingredients for this method (see Appendix A for the complete list).

III. Average $R^2$ for all pesticide calibrations was 0.997.

## 4.3 Accuracy & Precision

Table 4.3.1 below shows a spiked sample of 1000ppb of Canadian Pesticide Mix to a dried flower cannabis matrix:

| PESTICIDE | EXPECTED (ppb) | OBTAINED (ppb) | % RECOVERY |
|---|---|---|---|
| Etridiazole | 1000 | 1016.226 | 101.6% |
| Quintozene | 1000 | 1019.907 | 102.0% |
| MGK-264 | 1000 | 1019.872 | 101.9% |
| α-Endosulfan | 1000 | 1010.726 | 101.1% |
| Chlorfenapyr | 1000 | 1028.866 | 102.9% |
| β-Endosulfan | 1000 | 1013.450 | 101.3% |
| Endosulfan sulfate | 1000 | 1005.925 | 100.6% |

Table 4.3.2 below shows repeatability data for a sample of pesticide active ingredients in a dried flower cannabis matrix:

| PESTICIDE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| Etridiazole | 4.98 | 4.95 | 4.50 | 4.81 | 0.27 | 5.59% |
| MGK-264 | 5.08 | 5.08 | 4.75 | 4.97 | 0.19 | 3.83% |
| α-Endosulfan | 5.93 | 5.81 | 5.58 | 5.77 | 0.18 | 3.08% |
| Quintozene | 8.09 | 7.74 | 7.57 | 7.80 | 0.27 | 3.40% |

*All values in ppb, unless otherwise indicated

Table 4.3.3 below shows repeatability data for a sample of pesticide active ingredients in a cannabis oil matrix:

| PESTICIDE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| Cyfluthrin | 48.30 | 47.71 | 47.93 | 47.98 | 0.298 | 0.62% |
| Parathion-methyl | 5.69 | 5.66 | 6.27 | 5.87 | 0.344 | 5.85% |
| Cypermethrin | 10.41 | 9.97 | 9.75 | 10.04 | 0.336 | 3.35% |
| Chlorphenapyr | 3.39 | 3.95 | 3.71 | 3.68 | 0.281 | 7.63% |

*All values in ppb, unless otherwise indicated

Table 4.3.4 below shows intermediate precision data for a sample of pesticide active ingredients in a cannabis oil matrix, run by different analysts on different days:

| ANALYST 1 | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| Cyfluthrin | 85.98 | 85.74 | 86.35 | 86.02 | 0.307 | 0.36% |
| Parathion-methyl | 80.07 | 80.07 | 80.04 | 80.06 | 0.015 | 0.02% |
| Cypermethrin | 73.06 | 79.10 | 73.74 | 75.30 | 3.308 | 4.39% |
| **ANALYST 2** | **RUN 1** | **RUN 2** | **RUN 3** | **MEAN** | **SD** | **RSD** |
| Cyfluthrin | 85.95 | 85.84 | 85.87 | 85.89 | 0.057 | 0.07% |
| Parathion-methyl | 80.40 | 80.04 | 80.51 | 80.32 | 0.246 | 0.31% |
| Cypermethrin | 72.87 | 72.01 | 73.86 | 72.91 | 0.926 | 1.27% |

*All values in ppb, unless otherwise indicated

I. This dataset is an example, and it has been repeated for all pesticide active ingredients detectable under this method, and across several matrices.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 4.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.
III. The LoQ & LoD for each pesticide active ingredient in this method can be found in Appendix A.

## 4.5 Summary

Table 4.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Average $R^2$ | 0.997 |
| Calibration Range | 5-100ppb (low) & 100-2000ppb (high) |
| LoQ | See Appendix A |
| LoD | See Appendix A |
| Specificity | Tested in various matrices, shown to be specific. Only CRMs used for calibrations. |
| Accuracy | ±3%, with an RSD ~2.5% |
| Repeatability | RSD <0.5-2.5% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times across several matrices. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Tested with different extraction solvents, different pH and different matrices Additional experiments are currently in progress. |

## 4.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

# 5) PESTICIDE ANALYSIS ON LC-MS

## 5.1 Method Details

I. Analysis of pesticide residues by Liquid Chromatography – Mass Spectrometry (LC-MS)
II. This method is derived from the United States Pharmacopoeia (USP) General Chapter <561>.
III. Complete method details are outlined in LAB-SOP-ANA-005 Pesticide Residues Analysis in Cannabis and Cannabis Products by LC-MS.
IV. This method is capable of identifying and quantifying 85 pesticide active ingredients included on the "Mandatory cannabis testing for pesticide active ingredients – List and Limits" published by Health Canada.
V. The complete list of pesticide active ingredients that are detectable and quantifiable with this method can be found in Appendix B.

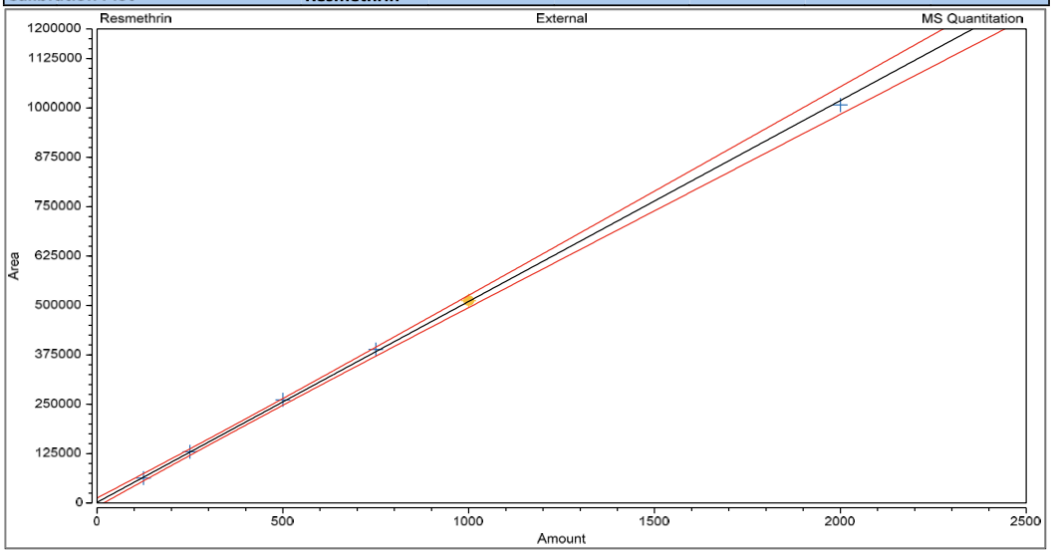
## 5.2 Linearity & Range

Figure 5.2.1 below shows a calibration curve for Resmethrin, as an example:

**Calibration**

| Calibration Details | Resmethrin | | |
|---|---|---|---|
| *Calibration Type* | Lin, WithOffset, 1/A | *Offset (C0)* | 1734.9040 |
| *Evaluation Type* | Area | *Slope (C1)* | 508.4617 |
| *Number of Calibration Points* | 6 | *Curve (C2)* | 0.0000 |
| *Number of disabled Calibration Points* | 0 | *R-Square* | 0.9997 |

**Calibration Plot** **Resmethrin**




I. Calibrations are available for all pesticide active ingredients included in this method (see Appendix B for the complete list).
II. The average $R^2$ value for all pesticide active ingredients was 0.996 across a range of 5-2000ppb.

## 5.3 Accuracy & Precision

Table 5.3.1 shows an example of recovery and repeatability data for several pesticide active ingredients spiked at 10ppb and added to a dried flower cannabis matrix:

| PESTICIDE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| Acephate | 10.6568 | 10.7642 | 10.6415 | 10.6875 | 0.0660 | 0.63% | 106.9% |
| Azadirachtin | 12.1751 | 12.5720 | 11.6071 | 12.1180 | 0.4848 | 4% | 121.2% |
| Iprodione | 9.1905 | 9.7724 | 9.1398 | 9.3657 | 0.3515 | 3.75% | 93.7% |
| Permethrin | 8.5320 | 8.2112 | 7.9545 | 8.2325 | 0.2893 | 3.51% | 82.3% |
| Phenothrin | 11.1841 | 10.9633 | 10.9320 | 11.0264 | 0.1374 | 1.25% | 110.3% |
| Dodemorph | 12.0986 | 12.2267 | 12.0798 | 12.1350 | 0.0799 | 0.66% | 121.3% |
| Fluopyram | 13.2536 | 13.4809 | 13.0327 | 13.2557 | 0.2241 | 1.69% | 130.3% |
| Hexythiazox | 14.6192 | 14.2692 | 14.2132 | 14.3672 | 0.2200 | 1.53% | 143.7% |

*All values shown are in ppb, unless otherwise indicated

Table 5.3.2 shows an example of repeatability data for several pesticide active ingredients in a cannabis oil matrix:

| PESTICIDE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| Carbofuran | 2.35 | 2.33 | 2.32 | 2.33 | 0.015 | 0.65% |
| Metalaxyl | 2.82 | 2.77 | 2.74 | 2.78 | 0.040 | 1.46% |
| Dimethomorph | 4.35 | 4.30 | 4.29 | 4.31 | 0.032 | 0.75% |
| Fluopyram | 2.48 | 2.44 | 2.55 | 2.49 | 0.056 | 2.24% |
| Boscalid | 1.75 | 1.75 | 1.57 | 1.69 | 0.104 | 6.15% |
| Tebuconazole | 1.55 | 1.52 | 1.55 | 1.54 | 0.017 | 1.12% |

*All values shown are in ppb, unless otherwise indicated

Table 5.3.3 shows intermediate precision data for a sample of pesticide active ingredients spiked to 100ppb in a dried cannabis matrix, run by different analysts on different days:

| ANALYST 1 | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| Clothiandin | 75.88 | 76.44 | 75.97 | 76.10 | 0.30 | 0.40% |
| Acetamiprid | 131.00 | 131.77 | 131.12 | 131.30 | 0.41 | 0.32% |
| Dinotefuran | 88.83 | 89.58 | 89.83 | 89.41 | 0.52 | 0.58% |
| Chlorantraniliprole | 84.33 | 83.17 | 82.37 | 83.29 | 0.99 | 1.18% |
| Paclobutrazol | 118.31 | 116.79 | 119.55 | 118.22 | 1.38 | 1.17% |
| Myclobutanil | 108.33 | 110.71 | 112.14 | 110.39 | 1.92 | 1.74% |
| **ANALYST 2** | **RUN 1** | **RUN 2** | **RUN 3** | **MEAN** | **SD** | **RSD** |
| Clothiandin | 97.11 | 96.87 | 96.59 | 96.86 | 0.26 | 0.27% |

| | | | | | | |
|---|---|---|---|---|---|---|
| Acetamiprid | 103.7 | 104.66 | 103.48 | 103.95 | 0.63 | 0.60% |
| Dinotefuran | 98.68 | 99.39 | 99.57 | 99.21 | 0.47 | 0.47% |
| Chlorantraniliprole | 109.05 | 108.33 | 109.31 | 108.90 | 0.51 | 0.47% |
| Paclobutrazol | 124.34 | 126.34 | 124.88 | 125.19 | 1.03 | 0.83% |
| Myclobutanil | 134.58 | 134.72 | 135.16 | 134.82 | 0.30 | 0.22% |

*All values in ppb, unless otherwise indicated

I. The above data is an example, and was repeated across multiple concentrations for all pesticide active ingredients and cannabis matrices.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 5.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The LoQ for each pesticide active ingredient in this method is 1ppb.
III. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.
IV. The LoD for each pesticide active ingredient in this method is 0.1ppb.

## 5.5 Summary

Table 5.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Average $R^2$ | 0.9993 |
| Calibration Range | 5-2000ppb |
| LoQ | 1ppb |
| LoD | 0.1ppb |
| Specificity | Tested in various matrices, shown to be specific. Only CRMs used for calibrations. |
| Accuracy | ±2%, with an RSD ~2% |
| Repeatability | RSD <0.1-2% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times across several matrices. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Method tested with different extraction solvents & different pH. Additional experiments are currently in progress. |

## 5.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

# 6) AFLATOXIN ANALYSIS

## 6.1 Method Details

I. Analysis of aflatoxins by Liquid Chromatography – Mass Spectrometry (LC-MS)
II. This method is derived from the United States Pharmacopoeia (USP) General Chapter <561>.
III. Complete method details are outlined in LAB-SOP-ANA-006 Analysis of Aflatoxins in Cannabis and Cannabis Products by LC-MS.

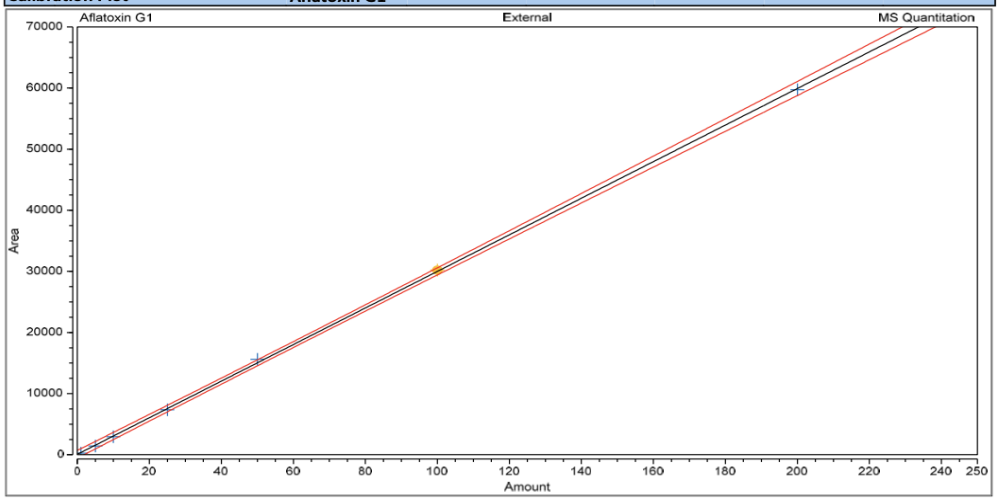
## 6.2 Linearity & Range

Figure 6.2.1 below shows a calibration curve for Aflatoxin G1, as an example:

**Calibration**

| Calibration Details | Aflatoxin G1 | | |
|---|---|---|---|
| *Calibration Type* | Lin, WithOffset | *Offset (C0)* | 69.1470 |
| *Evaluation Type* | Area | *Slope (C1)* | 299.3089 |
| *Number of Calibration Points* | 7 | *Curve (C2)* | 0.0000 |
| *Number of disabled Calibration Points* | 0 | *R-Square* | 0.9998 |

**Calibration Plot** Aflatoxin G1




I. Calibration curves are available for Aflatoxin B1, B2, G1 & G2 as well as Ochratoxin A.
II. Average $R^2$ value for all aflatoxins was 0.9998 with an average RSD of 1.86%.
III. The range for Aflatoxin B1 & G1 is 1-200ppb, while the range for Aflatoxin B2 & G2 is 0.3-60ppb.

## 6.3 Accuracy & Precision

Table 6.3.1 below shows recovery and repeatability data for a dried cannabis flower matrix spiked with 30ppb of Aflatoxin B2 & G2, and 100ppb of B1 & G1:

| AFLATOXIN | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| B1 | 115.437 | 114.574 | 115.820 | 115.280 | 0.640 | 0.56% | 115.3% |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| G1 | 128.034 | 128.475 | 129.096 | 128.535 | 0.420 | 0.42% | 128.5% |
| B2 | 34.077 | 33.623 | 34.060 | 33.920 | 0.760 | 0.76% | 113.1% |
| G2 | 37.232 | 38.390 | 38.603 | 38.075 | 0.737 | 1.94% | 126.9% |

*All values shown are in ppb, unless otherwise indicated

Table 6.3.2 below shows recovery and repeatability data for a dried cannabis flower matrix spiked with 3ppb of Aflatoxin B2 & G2, and 10ppb of B1 & G1:

| AFLATOXIN | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| B1 | 9.724 | 9.594 | 9.500 | 9.606 | 0.110 | 1.17% | 96.0% |
| G1 | 10.472 | 10.404 | 10.191 | 10.355 | 0.140 | 1.42% | 103.6% |
| B2 | 2.960 | 2.876 | 2.852 | 2.896 | 0.050 | 1.96% | 96.5% |
| G2 | 3.257 | 3.326 | 3.278 | 3.287 | 0.030 | 1.08% | 109.6% |

*All values shown are in ppb, unless otherwise indicated

Table 6.3.3 below shows recovery and repeatability data for a dried cannabis flower matrix spiked with 1.5ppb of Aflatoxin B2 & G2, and 5ppb of B1 & G1:

| AFLATOXIN | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| B1 | 4.186 | 4.377 | 4.433 | 4.332 | 0.129 | 2.99% | 86.7% |
| G1 | 4.418 | 4.517 | 4.693 | 4.540 | 0.140 | 3.07% | 90.8% |
| B2 | 1.504 | 1.418 | 1.461 | 1.461 | 0.043 | 2.94% | 97.4% |
| G2 | 1.591 | 1.571 | 1.612 | 1.591 | 0.020 | 1.29% | 106.1% |

*All values shown are in ppb, unless otherwise indicated

Table 6.3.4 below shows recovery and repeatability data for a cannabis matrix spiked with 3 different concentrations of Ochratoxin A:

| OCHRATOXIN | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| 1ppb Spike | 1.39 | 1.38 | 1.27 | 1.30 | 0.08 | 5.78% | 130.0% |
| 10ppb Spike | 11.49 | 11.25 | 11.41 | 11.39 | 0.12 | 1.06% | 113.9% |
| 20ppb Spike | 25.15 | 25.62 | 24.90 | 25.22 | 0.36 | 1.44% | 126.1% |

*All values shown are in ppb, unless otherwise indicated

Table 6.3.5 shows intermediate precision data for a sample of aflatoxins spiked to 50ppb of Aflatoxins B1 & G1, 15ppb of Aflatoxins B2 & G2 & 20ppb of Ochratoxin A in a dried cannabis matrix, run by different analysts on different days:

| ANALYST 1 | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| Aflatoxin B1 | 59.79 | 60.41 | 59.98 | 60.06 | 0.32 | 0.53% |
| Aflatoxin G1 | 64.75 | 67.27 | 66.16 | 66.06 | 1.26 | 1.91% |
| Aflatoxin B2 | 17.15 | 17.76 | 17.43 | 17.45 | 0.31 | 1.75% |

| | | | | | | |
|---|---|---|---|---|---|---|
| Aflatoxin G2 | 18.90 | 19.71 | 19.66 | 19.42 | 0.45 | 2.34% |
| Ochratoxin A | 25.15 | 25.62 | 24.90 | 25.22 | 0.36 | 1.44% |
| **ANALYST 2** | **RUN 1** | **RUN 2** | **RUN 3** | **MEAN** | **SD** | **RSD** |
| Aflatoxin B1 | 56.36 | 54.58 | 56.54 | 55.83 | 1.08 | 1.94% |
| Aflatoxin G1 | 60.03 | 59.03 | 60.98 | 60.01 | 0.98 | 1.62% |
| Aflatoxin B2 | 16.05 | 16.11 | 16.33 | 16.16 | 0.15 | 0.91% |
| Aflatoxin G2 | 16.97 | 16.46 | 16.43 | 16.62 | 0.30 | 1.83% |
| Ochratoxin A | 21.52 | 23.46 | 22.47 | 22.48 | 0.97 | 4.33% |

*All values shown are in ppb, unless otherwise indicated

I. The above data are examples, and they were repeated across multiple concentrations for all aflatoxins and cannabis matrices.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 6.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The LoQ for each aflatoxin was found to be 0.3ppb.
III. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.
IV. The LoD for each aflatoxin was found to be 0.1ppb.

## 6.5 Summary

Table 6.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Average $R^2$ | 0.9996 |
| Calibration Range | Aflatoxin B1 & G1: 1-200ppb<br>Aflatoxin B2 & G2: 0.3-60ppb |
| LoQ | 0.3ppb |
| LoD | 0.1ppb |
| Specificity | Tested in various matrices, shown to be specific. Only CRMs used for calibrations. |
| Accuracy | ±1%, with an RSD <0.4-1.5% |
| Repeatability | RSD <0.4-1.5% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times across several matrices. |

| | |
|---|---|
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Tested with different extraction solvents, different pH and different matrices Additional experiments are currently in progress. |

## 6.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

# 7) TERPENE ANALYSIS

## 7.1 Method Details

I. Analysis of terpenes using Gas Chromatography – Flame Ionization Detector (GC-FID).
II. This method is a modified United Nations Office on Drugs and Crime (UNODC) method.
III. Complete method details are outlined in LAB-SOP-ANA-003 Analysis of Terpenes from Different Matrices by GC-FID.
IV. The complete list of terpenes that are detectable with this method can be found in Appendix C.

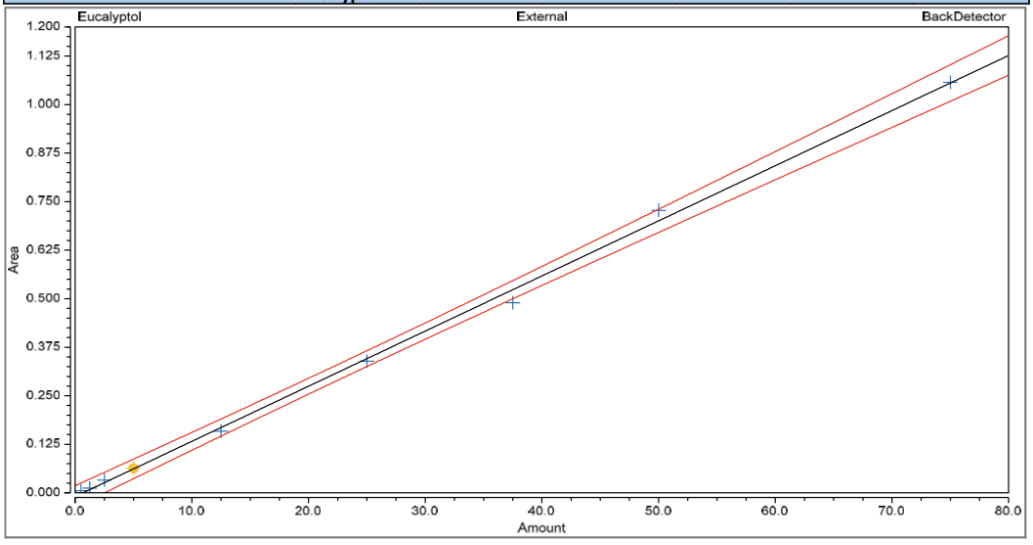
## 7.2 Linearity & Range

Figure 7.2.1 below shows a Eucalyptol calibration curve, as an example:

**Calibration**

| Calibration Details | Eucalyptol | | |
|---|---|---|---|
| *Calibration Type* | Lin, WithOffset | *Offset (C0)* | -0.0093 |
| *Evaluation Type* | Area | *Slope (C1)* | 0.0142 |
| *Number of Calibration Points* | 9 | *Curve (C2)* | 0.0000 |
| *Number of disabled Calibration Points* | 0 | *R-Square* | 0.9981 |

**Calibration Plot** **Eucalyptol**




I. A seven-point calibration curve was chosen (1ppm, 5ppm, 10ppm, 50ppm, 100ppm, 150ppm & 300ppm) to select the linear part of the calibration curves. The average $R^2$ value across this range was 0.994.

## 7.3 Accuracy & Precision

Table 7.3.1 shows an example of 50ppm of several different terpenes spiked to a dried flower cannabis matrix:

| TERPENE | EXPECTED (ppm) | OBTAINED (ppm) | % RECOVERY |
|---|---|---|---|
| Camphene | 50 | 48.026 | 96.1% |
| β-Myrcene | 50 | 44.713 | 89.4% |
| Phellandrenes | 50 | 50.061 | 100.1% |
| 3-Carene | 50 | 48.276 | 96.6% |
| Eucalyptol | 50 | 53.712 | 107.4% |
| Ocimenes | 50 | 54.915 | 109.8% |

Table 7.3.2 shows repeatability data for a sample of terpenes in a dried flower cannabis matrix:

| TERPENE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD |
|---|---|---|---|---|---|---|
| α-Pinene | 8.894 | 9.198 | 8.674 | 8.922 | 0.263 | 2.95% |
| Camphene | 54.439 | 56.970 | 52.745 | 54.718 | 2.126 | 3.88% |
| Sabinene | 0.699 | 0.686 | 0.673 | 0.686 | 0.013 | 1.89% |
| β-Pinene | 18.222 | 18.874 | 17.782 | 18.292 | 0.550 | 3.01% |
| β-Myrcene | 68.073 | 71.715 | 65.578 | 68.455 | 3.086 | 4.51% |
| Phellandrenes | 49.722 | 52.368 | 48.094 | 50.061 | 2.157 | 4.31% |
| 3-Carene | 54.735 | 56.754 | 53.354 | 54.948 | 1.710 | 3.09% |
| α-Terpinene | 4.006 | 4.091 | 3.949 | 4.014 | 0.072 | 1.79% |
| o-Cymene | 6.224 | 6.471 | 6.071 | 6.255 | 0.202 | 3.23% |
| D-Limonene | 30.350 | 31.579 | 29.389 | 30.439 | 1.097 | 3.60% |
| Eucalyptol | 59.475 | 62.624 | 57.406 | 59.835 | 2.627 | 4.39% |
| Ocimenes | 54.438 | 57.691 | 52.615 | 54.915 | 2.572 | 4.68% |
| γ-Terpinene | 3.063 | 3.136 | 3.031 | 3.077 | 0.054 | 1.75% |
| Sabinene hydrate | 1.662 | 1.763 | 1.616 | 1.680 | 0.075 | 4.46% |

*All values in ppm, unless otherwise indicated

I. The above data are examples, and they were repeated across multiple concentrations for all terpenes and cannabis matrices.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 7.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The LoQ for each terpene was found to be 1ppm.
III. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.

IV. The LoD for each terpene was found to be 0.1ppm.

## 7.5 Summary

Table 7.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Average $R^2$ | 0.994 |
| Calibration Range | 1-300ppm |
| LoQ | 1ppm |
| LoD | 0.1ppm |
| Specificity | Tested in various matrices, shown to be specific. One overlapping pair was identified: alpha-Cedrene & Caryophyllene. This pair can be further assigned by GC-MS. Only CRMs used for calibrations. |
| Accuracy | ±5%, with an RSD <1-10% |
| Repeatability | RSD <1-5% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times across several matrices. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Additional experiments are currently in progress. |

## 7.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

II. There is one pair for which this method was found not to be specific: alpha-Cedrene & Caryophyllene. Samples containing those terpenes will be further assigned by GC-MS.

# 8) RESIDUAL SOLVENTS

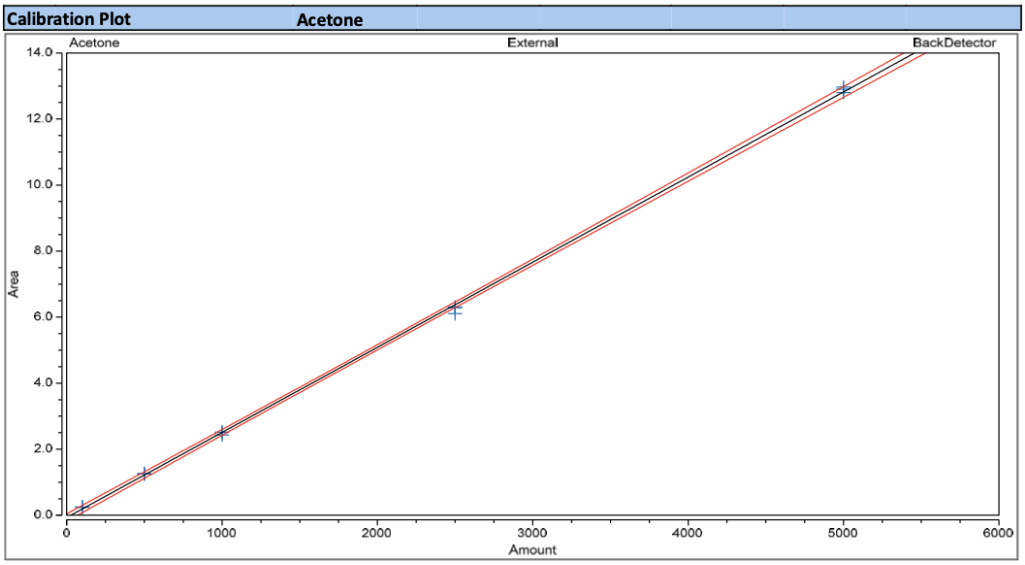
## 8.1 Method Details

I. Analysis of residual solvents using Gas Chromatography – Flame Ionization Detector (GC-FID).
II. This method is derived from the United States Pharmacopoeia (USP) General Chapter <467>.
III. Complete method details are outlined in LAB-SOP-ANA-008 Residual Solvent Analysis for Cannabis and Cannabis Products by GC-FID.
IV. The complete list of residual solvents that are detectable and quantifiable with this method can be found in Appendix D.

## 8.2 Linearity & Range

Figure 8.2.1 below shows a calibration curve for Acetone, as an example:

| Calibration | | | |
|---|---|---|---|
| **Calibration Details** | **Acetone** | | |
| *Calibration Type* | Lin, WithOffset | *Offset (C0)* | -0.0705 |
| *Evaluation Type* | Area | *Slope (C1)* | 0.0026 |
| *Number of Calibration Points* | 15 | *Curve (C2)* | 0.0000 |
| *Number of disabled Calibration Points* | 0 | *R-Square* | 0.9996 |




I. Calibration curves are available for all other residual solvents. A complete list of residual solvents detected and quantified by this method is available in Appendix D.
II. The average $R^2$ value across the liquid residual solvent range (100-5000ppm) was 0.9997.

## 8.3 Accuracy & Precision

Table 8.3.1 shows recovery and repeatability data for a cannabis oil matrix spiked with 2250ppm of various residual solvents:

| ANALYTE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| Methanol | 2469.9 | 2431.9 | 2429.7 | 2443.8 | 22.60 | 0.92% | 108.6% |
| Ethanol | 2469.6 | 2426.6 | 2442.5 | 2446.2 | 21.74 | 0.89% | 97.1%** |
| Isopropanol | 2437.8 | 2405.0 | 2412.9 | 2418.6 | 17.12 | 0.71% | 107.5% |
| 1-propanol | 2481.2 | 2381.2 | 2432.8 | 2431.7 | 50.01 | 2.06% | 108.1% |
| 2-butanone | 2329.5 | 2282.5 | 2277.6 | 2296.5 | 28.65 | 1.25% | 102.1% |
| Ethyl acetate | 2219.9 | 2282.4 | 2171.3 | 2224.6 | 55.69 | 2.50% | 98.9% |
| Chloroform | 2348.8 | 2336.1 | 2339.2 | 2341.4 | 6.62 | 0.28% | 104.1% |
| Benzene | 2209.8 | 2200.7 | 2188.2 | 2199.6 | 10.84 | 0.49% | 97.8% |

*All values shown are in ppm, unless otherwise indicated
**Note that the theoretical value for ethanol is 2515ppm.

Table 8.3.2 shows recovery and repeatability for a cannabis extract matrix spiked with 125ppm of three gaseous solvents:

| ANALYTE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| Propane | 159.1 | 155.1 | 154.4 | 156.22 | 2.54 | 1.63% | 125.0% |
| Iso-Butane | 128.92 | 128.29 | 127.27 | 128.16 | 0.83 | 0.65% | 102.5% |
| Butane | 112.64 | 111.05 | 109.38 | 111.02 | 1.63 | 1.47% | 88.8% |

*All values shown are in ppm, unless otherwise indicated

Table 8.3.3 shows recovery and repeatability data for a cannabis oil matrix spiked with 1000ppm of various residual solvents:

| ANALYTE | RUN 1 | RUN 2 | RUN 3 | MEAN | SD | RSD | % REC. |
|---|---|---|---|---|---|---|---|
| Methanol | 887.6 | 713.3 | 857.3 | 819.4 | 93.12 | 11.37% | 81.9% |
| Ethanol | 1046.6 | 906.0 | 1026.0 | 992.9 | 75.93 | 7.65% | 78.6%** |
| Isopropanol | 907.7 | 825.9 | 903.5 | 879.0 | 46.06 | 5.24% | 87.9% |
| 1-propanol | 1064.5 | 1012.5 | 1075.7 | 1050.9 | 33.72 | 3.21% | 105.1% |
| 2-butanone | 821.2 | 778.9 | 785.9 | 795.3 | 22.67 | 2.85% | 79.5% |
| Ethyl acetate | 796.5 | 793.5 | 789.8 | 793.3 | 3.36 | 0.42% | 79.0% |
| Chloroform | 865.7 | 858.7 | 867.6 | 864.0 | 4.69 | 0.54% | 86.4% |
| Benzene | 829.9 | 847.5 | 811.6 | 829.7 | 17.95 | 2.17% | 83.0% |

*All values shown are in ppm, unless otherwise indicated
**Note that the theoretical value for ethanol is 1263ppm.

I. The above data are examples, and they were repeated across multiple concentrations for all residual solvents.
II. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 8.4 Limit of Detection & Limit of Quantification

I. The Limit of Quantification (LoQ) was calculated based on the smallest concentration from the linear area of the calibration curve.
II. The LoQ for each residual solvent was found to be 100ppm.
III. The Limit of Detection (LoD) was determined based on the smallest concentration detected on the chromatogram which is distinguishable from the baseline.
IV. The LoD for each residual solvent was found to be 10ppm.

## 8.5 Summary

Table 8.5.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Average $R^2$ | 0.996 |
| Calibration Range | 100-5000ppm for liquid solvent<br>100-2000ppm for gaseous solvent |
| LoQ | 100ppm |
| LoD | 10ppm |
| Specificity | Tested in various matrices, shown to be specific. |
| Accuracy | ±1%, with an RSD <1.54% |
| Repeatability | RSD <0.4-1.5% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |
| Robustness | Tested with different extraction solvents. Additional experiments are currently in progress. |

## 8.6 Conclusion

I. The results of internal validation studies have found this method to be specific, linear, precise, accurate and repeatable.

# 9) MICROBIOLOGICAL TESTING – UNITED STATES PHARMACOPOEIA

## 9.1 Method Details

I. Identification and enumeration of microbiological contaminants by conventional culture-based method.
II. This method is derived from the United States Pharmacopoeia (USP) General Chapter <61> and <62>.
III. Complete method details are outlined in LAB-SOP-MIC-002 & LAB-SOP-MIC-003.
IV. This method is capable quantifying the Total Aerobic Plate Count (TAPC), Total Yeast & Mould Count (TYMC), Bile-Tolerant Gram-Negative Bacteria (BTGNB), and identifying objectionable organisms such as *Escherichia coli*, *Salmonella* spp., *Pseudomonas aeruginosa* & *Staphylococcus aureus* as well as pathogenic species of *Aspergillus*.
V. Sterility, inclusivity and exclusivity tests were performed on all media, reagents and buffers before use. Each test was validated with a positive and negative control.

## 9.2 Selectivity & Identification

Table 9.2.1 below shows identification results for objectionable microbes in various cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REPEATABILITY |
|---|---|---|---|---|
| Flower | *S. aureus* | Positive | Positive | 100% |
| Flower | *S. aureus* | Positive | Positive | |
| Oil | *S. aureus* | Positive | Positive | 100% |
| Oil | *S. aureus* | Positive | Positive | |
| Flower | *E. coli* | Positive | Positive | 100% |
| Flower | *E. coli* | Positive | Positive | |
| Oil | *E. coli* | Positive | Positive | 100% |
| Oil | *E. coli* | Positive | Positive | |
| Flower | *Salmonella* spp. | Positive | Positive | 100% |
| Flower | *Salmonella* spp. | Positive | Positive | |
| Oil | *Salmonella* spp. | Positive | Positive | 100% |
| Oil | *Salmonella* spp. | Positive | Positive | |
| Flower | *P. aeruginosa* | Positive | Positive | 100% |
| Flower | *P. aeruginosa* | Positive | Positive | |
| Oil | *P. aeruginosa* | Positive | Positive | 100% |
| Oil | *P. aeruginosa* | Positive | Positive | |
| Flower | *A. niger* | Positive | Positive | 100% |
| Flower | *A. niger* | Positive | Positive | |

| | | | | |
|---|---|---|---|---|
| Oil | *A. niger* | Positive | Positive | 100% |
| Oil | *A. niger* | Positive | Positive | |
| Flower | *A. terreus* | Positive | Positive | 100% |
| Flower | *A. terreus* | Positive | Positive | |
| Oil | *A. terreus* | Positive | Positive | 100% |
| Oil | *A. terreus* | Positive | Positive | |
| Flower | *A. fumigatus* | Positive | Positive | 100% |
| Flower | *A. fumigatus* | Positive | Positive | |
| Oil | *A. fumigatus* | Positive | Positive | 100% |
| Oil | *A. fumigatus* | Positive | Positive | |

I. The method indicated in LAB-SOP-MIC-003 identified specific organisms using selective and differential media, and further reconfirmed the species using biochemical and molecular techniques. Molecular analysis, biochemical analysis, and growth on selective media confirm the above-listed results. To spike the different matrixes, the ATCC strains (five passages) were used as a reference material.
II. These experiments have been repeated across multiple matrices and cannabis samples, and have been found to be 100% repeatable for identifying and detecting any objectionable organisms listed in the compendium.

## 9.3 Accuracy & Precision

Table 9.3.1 shows recovery and repeatability data for the Total Aerobic Plate Count (TAPC) of spiked samples in different cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REC. | AVG % REC. |
|---|---|---|---|---|---|
| Flower | *S. aureus* | 15,000 | 14,324 | 95.50% | 96.60% |
| Flower | *S. aureus* | 15,000 | 14,645 | 97.63% | |
| Flower | *E. coli* | 75,000 | 74,950 | 99.90% | 99.85% |
| Flower | *E. coli* | 75,000 | 74,125 | 98.80% | |
| Flower | *S. aureus* | 14,000 | 13,829 | 98.77% | 98.66% |
| Flower | *S. aureus* | 14,000 | 13,939 | 99.56% | |
| Flower | *E. coli* | 28,000 | 27,978 | 99.90% | 100.36% |
| Flower | *E. coli* | 28,000 | 28,234 | 100.83% | |
| Flower | *S. aureus* | 15,000 | 14,624 | 97.49% | 97.50% |
| Flower | *S. aureus* | 15,000 | 14,754 | 98.36% | |
| Flower | *E. coli* | 82,000 | 82,728 | 100.88% | 100.00% |
| Flower | *E. coli* | 82,000 | 81,284 | 99.12% | |
| Flower | *S. aureus* | 12,000 | 11,699 | 97.49% | 98.69% |

| | | | | | |
|---|---|---|---|---|---|
| Flower | *S. aureus* | 12,000 | 11,998 | 99.90% | |
| Flower | *E. coli* | 89,000 | 89,145 | 100.16% | 100.01% |
| Flower | *E. coli* | 89,000 | 88,889 | 99.87% | |
| Flower | *S. aureus* | 10,000 | 9,899 | 98.99% | 99.19% |
| Flower | *S. aureus* | 10,000 | 9,940 | 99.40% | |
| Oil | *E. coli* | 5,500 | 5,534 | 100.61% | 96.85% |
| Oil | *E. coli* | 5,500 | 5,123 | 93.14% | |

*All values shown in Colony Forming Units (CFU)/gram, unless otherwise indicated

I. The TAPC was enumerated using one strain of gram positive (*Staphylococcus aureus* ATCC 6538) and one strain of gram negative (*Escherichia coli* ATCC 25922) bacteria, covering a wide range of pathogenic bacteria.

Table 9.3.2 shows recovery and repeatability data for the Bile-Tolerant Gram-Negative Bacteria (BTGNB) of spiked samples in different cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REC. | AVG % REC. |
|---|---|---|---|---|---|
| Flower | *E. coli* | 66,000 | 66,700 | 101.06% | 100.45% |
| Flower | *E. coli* | 66,000 | 65,899 | 99.84% | |
| Flower | *E. coli* | 26,000 | 25,749 | 99.03% | 98.46% |
| Flower | *E. coli* | 26,000 | 25,453 | 97.89% | |
| Flower | *E. coli* | 72,000 | 72,245 | 100.34% | 99.64% |
| Flower | *E. coli* | 72,000 | 71,245 | 98.95% | |
| Flower | *E. coli* | 69,000 | 68,945 | 99.92% | 99.57% |
| Flower | *E. coli* | 69,000 | 68,455 | 99.21% | |
| Oil | *E. coli* | 6,300 | 6,295 | 99.92% | 99.96% |
| Oil | *E. coli* | 6,300 | 6,300 | 100.00% | |

*All values shown in Colony Forming Units (CFU)/gram, unless otherwise indicated

II. The population of bile-tolerant Gram-Negative Bacteria was analyzed using *Escherichia coli* (ATCC 25922), which is more prevalent than all other Gram-Negative bacteria tolerant of bile.

Table 9.3.3 shows recovery and repeatability data for Total Yeast & Mould Count (TYMC) of spiked samples in different cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REC. | AVG % REC. |
|---|---|---|---|---|---|
| Flower | *A. niger* | 3,000 | 2,989 | 99.63% | 96.28% |
| Flower | *A. niger* | 3,000 | 2,788 | 92.93% | |

| | | | | | |
|---|---|---|---|---|---|
| Flower | *A. niger* | 2,500 | 2,455 | 98.20% | 96.28% |
| Flower | *A. niger* | 2,500 | 2,359 | 94.36% | |
| Flower | *A. niger* | 4,500 | 4,233 | 94.06% | 95.80% |
| Flower | *A. niger* | 4,500 | 4,389 | 97.53% | |
| Flower | *A. niger* | 3,000 | 2,899 | 96.63% | 94.07% |
| Flower | *A. niger* | 3,000 | 2,745 | 91.50% | |
| Oil | *A. niger* | 2,000 | 1,800 | 90.00% | 93.63% |
| Oil | *A. niger* | 2,000 | 1,945 | 97.25% | |

*All values shown in Colony Forming Units (CFU)/gram, unless otherwise indicated

III. In Total Yeast and Mold Count, *A. niger* (ATCC 16888) was used, which is one of the most hazardous species of *Aspergillus*.
IV. This method has been enhanced by incorporating cutting-edge technologies, including gravimetric sample preparation, semi-automated spiral plating, and automatic colony counting. The overall results show above 96% accuracy for enumeration with all different matrices.
V. The above data are examples, and they were repeated across multiple concentrations for all microbes and cannabis matrices.
VI. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 9.4 Summary

Table 9.4.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Selectivity & Identification | Method has been tested for eight different types of micro-organisms across several cannabis matrices and found to be specific for selectivity and identification. |
| Accuracy | ±5% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |

## 9.5 Conclusion

I. The results of internal validation studies have found this method to be specific, precise, accurate and repeatable.

# 10) MICROBIOLOGICAL TESTING – EUROPEAN PHARMACOPOEIA

## 10.1 Method Details

I. Identification and enumeration of microbiological contaminants by conventional culture-based method.
II. This method is derived from the European Pharmacopoeia (Ph. Eur.) General Chapters 2.6.12 and 2.6.13.
III. Complete method details are outlined in LAB-SOP-MIC-006 & LAB-SOP-MIC-007.
IV. This method is capable quantifying the Total Aerobic Plate Count (TAPC), Total Yeast & Mould Count (TYMC), Bile-Tolerant Gram-Negative Bacteria (BTGNB), and identifying objectionable organisms such as *Escherichia coli*, *Salmonella* spp., *Pseudomonas aeruginosa* & *Staphylococcus aureus* as well as pathogenic species of *Aspergillus*.
V. Sterility, inclusivity and exclusivity tests were performed on all media, reagents and buffers before use. Each test was validated with a positive and negative control.

## 10.2 Selectivity & Identification

Table 10.2.1 below shows identification results for objectionable microbes in various cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REPEATABILITY |
|---|---|---|---|---|
| Flower | *S. aureus* | Positive | Positive | 100% |
| Flower | *S. aureus* | Positive | Positive | |
| Oil | *S. aureus* | Positive | Positive | 100% |
| Oil | *S. aureus* | Positive | Positive | |
| Flower | *E. coli* | Positive | Positive | 100% |
| Flower | *E. coli* | Positive | Positive | |
| Oil | *E. coli* | Positive | Positive | 100% |
| Oil | *E. coli* | Positive | Positive | |
| Flower | *Salmonella* spp. | Positive | Positive | 100% |
| Flower | *Salmonella* spp. | Positive | Positive | |
| Oil | *Salmonella* spp. | Positive | Positive | 100% |
| Oil | *Salmonella* spp. | Positive | Positive | |
| Flower | *P. aeruginosa* | Positive | Positive | 100% |
| Flower | *P. aeruginosa* | Positive | Positive | |
| Oil | *P. aeruginosa* | Positive | Positive | 100% |
| Oil | *P. aeruginosa* | Positive | Positive | |
| Flower | *A. niger* | Positive | Positive | 100% |
| Flower | *A. niger* | Positive | Positive | |

| | | | | |
|---|---|---|---|---|
| Oil | *A. niger* | Positive | Positive | 100% |
| Oil | *A. niger* | Positive | Positive | |
| Flower | *A. terreus* | Positive | Positive | 100% |
| Flower | *A. terreus* | Positive | Positive | |
| Oil | *A. terreus* | Positive | Positive | 100% |
| Oil | *A. terreus* | Positive | Positive | |
| Flower | *A. fumigatus* | Positive | Positive | 100% |
| Flower | *A. fumigatus* | Positive | Positive | |
| Oil | *A. fumigatus* | Positive | Positive | 100% |
| Oil | *A. fumigatus* | Positive | Positive | |

I. The method indicated in LAB-SOP-MIC-007 identified specific organisms using selective and differential media, and further reconfirmed the species using biochemical and molecular techniques. Molecular analysis, biochemical analysis, and growth on selective media confirm the above-listed results. To spike the different matrixes, the ATCC strains (five passages) were used as a reference material.
II. These experiments have been repeated across multiple matrices and cannabis samples, and have been found to be 100% repeatable for identifying and detecting any objectionable organisms listed in the compendium.

### 10.3 Accuracy & Precision

Table 10.3.1 shows recovery and repeatability data for the Total Aerobic Plate Count (TAPC) of spiked samples in different cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REC. | AVG % REC. |
|---|---|---|---|---|---|
| Flower | *S. aureus* | 1000 | 960 | 96.00% | 95.00% |
| Flower | *S. aureus* | 1000 | 940 | 94.00% | |
| Flower | *P. aeruginosa* | 20,200 | 19,400 | 96.04% | 95.05% |
| Flower | *P. aeruginosa* | 20,200 | 19,000 | 94.06% | |
| Flower | *S. aureus* | 1,000 | 980 | 98.00% | 97.00% |
| Flower | *S. aureus* | 1,000 | 960 | 96.00% | |
| Flower | *P. aeruginosa* | 20,200 | 19,800 | 98.03% | 100.01% |
| Flower | *P. aeruginosa* | 20,200 | 20,600 | 101.98% | |
| Flower | *S. aureus* | 1,000 | 960 | 96.00% | 94.00% |
| Flower | *S. aureus* | 1,000 | 920 | 92.00% | |
| Flower | *P. aeruginosa* | 20,200 | 19,400 | 96.04% | 97.03% |
| Flower | *P. aeruginosa* | 20,200 | 19,800 | 98.02% | |
| Oil | *S. aureus* | 1,000 | 960 | 96.00% | 94.00% |

| | | | | | |
|---|---|---|---|---|---|
| Oil | *S. aureus* | 1,000 | 920 | 92.00% | |
| Oil | *P. aeruginosa* | 20,200 | 18,600 | 92.08% | 93.07% |
| Oil | *P. aeruginosa* | 20,200 | 19,000 | 94.06% | |

*All values shown in Colony Forming Units (CFU)/gram, unless otherwise indicated

I. The TAPC was enumerated using one strain of gram positive (*Staphylococcus aureus* ATCC 6538) and one strain of gram negative (*Pseudomonas aeruginosa* ATCC 27853) bacteria, covering a wide range of pathogenic bacteria.

Table 10.3.2 shows recovery and repeatability data for the Bile-Tolerant Gram-Negative Bacteria (BTGNB) of spiked samples in different cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REC. | AVG % REC. |
|---|---|---|---|---|---|
| Flower | *S. typhi* | 5,560 | 5,330 | 95.86% | 96.90% |
| Flower | *S. typhi* | 5,560 | 5,445 | 97.93% | |
| Flower | *S. typhi* | 5,560 | 5,220 | 93.88% | 96.94% |
| Flower | *S. typhi* | 5,560 | 5,560 | 100.00% | |
| Flower | *S. typhi* | 5,560 | 5,330 | 95.86% | 96.90% |
| Flower | *S. typhi* | 5,560 | 5,445 | 97.93% | |
| Oil | *S. typhi* | 5,560 | 5,220 | 93.88% | 94.87% |
| Oil | *S. typhi* | 5,560 | 5,330 | 95.86% | |

*All values shown in Colony Forming Units (CFU)/gram, unless otherwise indicated

II. The population of bile-tolerant Gram-Negative Bacteria was analyzed using *Salmonella enterica serovar typhimurium* (ATCC 14028).

Table 10.3.3 shows recovery and repeatability data for Total Yeast & Mould Count (TYMC) of spiked samples in different cannabis matrices:

| SAMPLE TYPE | MICROBE | TRUE VALUE | RESULT | % REC. | AVG % REC. |
|---|---|---|---|---|---|
| Flower | *A. niger* | 440 | 420 | 95.45% | 95.45% |
| Flower | *A. niger* | 440 | 420 | 95.45% | |
| Flower | *A. niger* | 440 | 420 | 95.45% | 97.73% |
| Flower | *A. niger* | 440 | 440 | 100.00% | |
| Flower | *A. niger* | 440 | 420 | 95.45% | 95.45% |
| Flower | *A. niger* | 440 | 420 | 95.45% | |
| Oil | *A. niger* | 440 | 400 | 90.91% | 93.18% |
| Oil | *A. niger* | 440 | 420 | 95.45% | |

*All values shown in Colony Forming Units (CFU)/gram, unless otherwise indicated

III. In Total Yeast and Mold Count, *A. niger* (ATCC 16888) was used, which is one of the most hazardous species of *Aspergillus*.
IV. This method has been enhanced by incorporating cutting-edge technologies, including gravimetric sample preparation, semi-automated spiral plating, and automatic colony counting. The overall results show above 96% accuracy for enumeration with all different matrices.
V. The above data are examples, and they were repeated across multiple concentrations for all microbes and cannabis matrices.
VI. Reproducibility studies (between-laboratory comparisons) are currently in progress.

## 10.4 Summary

Table 10.4.1 below provides a validation summary:

| PARAMETER | RESULT |
|---|---|
| Selectivity & Identification | Method has been tested for eight different types of micro-organisms across several cannabis matrices and found to be specific for selectivity and identification. |
| Accuracy | ±5% |
| Intermediate Precision | Intra-laboratory comparisons done multiple times. |
| Reproducibility | Inter-laboratory comparisons currently in progress. |

## 10.5 Conclusion

I. The results of internal validation studies have found this method to be specific, precise, accurate and repeatable.

# 11) DEFINITIONS

ATCC – American Type Culture Collection

CRM – Certified Reference Material

% REC. – Percent recovery. Calculated by dividing the test result by the expected value and multiplying by 100. Used as an indicator of method accuracy.

# 12) APPENDICES

## 12.1 APPENDIX A – GC-MS PESTICIDE LIST

**Limits of Detection and Quantification for Specific Pesticide Residues Quantified by GC-MS:**

| Pesticide Active Ingredient | LoD (ppb) | LoQ (ppb |
|---|---|---|
| Etridiazole | 0.1 | 7.5 |
| Quintozene | 0.1 | 7.5 |
| Parathion-methyl | 1 | 25 |
| Kinoprene | 1 | 25 |
| MGK-264 | 1 | 25 |
| α-Endosulfan | 1 | 75 |
| Chlorphenapyr | 1 | 25 |
| β-Endosulfan | 1 | 25 |
| Endosulfan sulfate | 1 | 25 |
| Cyfluthrin | 1 | 100 |
| Cypermethrin | 1 | 250 |

## 12.2 APPENDIX B – LC-MS PESTICIDE LIST

**List of Pesticide Residues Quantified by LC-MS:**

| Pesticide Active Ingredient | | |
|---|---|---|
| Abamectin | Dodemorph | Permethrin |
| Acephate | Ethoprophos | Phenothrin |
| Acequinocyl | Etofenprox | Phosmet |
| Acetamiprid | Etoxazole | Piperonyl butoxide |
| Aldicarb | Fenoxycarb | Pirimicarb |
| Allethrin | Fenpyroximate | Prallethrin |
| Azadirachtin | Fensulfothion | Propiconazole |
| Azoxystrobin | Fenthion | Propoxur |
| Benzovindiflupyr | Fenvalerate | Pyraclostrobin |
| Bifenazate | Fipronil | Pyrethrins |
| Bifenthrin | Flonicamid | Pyridaben |
| Boscalid | Fludioxonil | Resmethrin |
| Buprofezin | Fluopyram | Spinetoram |
| Carbaryl | Hexythiazox | Spinosad |
| Carbofuran | Imazalil | Spirodiclofen |
| Chlorantraniliprole | Imidacloprid | Spiromesifen |
| Chlorpyrifos | Iprodione | Spirotetramat |
| Clofentezine | Kresoxim-methyl | Spiroxamine |
| Clothianidin | Malathion | Tebuconazole |
| Coumaphos | Metalaxyl | Tebufenozide |
| Cyantraniliprole | Methiocarb | Teflubenzuron |
| Cyprodinil | Methomyl | Tetrachlorvinphos |
| Daminozide | Methoprene | Tetramethrin |
| Deltamethrin | Mevinphos | Thiacloprid |
| Diazinon | Myclobutanil | Thiamethoxam |
| Dichlorvos | Naled | Thiophanate-methyl |
| Dimethoate | Novaluron | Trifloxystrobin |
| Dimethomorph | Oxamyl | |
| Dinotefuran | Paclobutrazol | |

## 12.3 APPENDIX C – TERPENE LIST

**List of Terpenes identifiable with GC-FID:**

| Terpene | | |
|---|---|---|
| α-Bisabolol | α-Pinene | β-Pinene |
| Borneol | Sabinene | β-Myrcene |
| Camphene | γ-Terpinene | Phellandrenes |
| Camphor | Sabinene hydrate | 3-Carene |
| Isoborneol | Terpinolene | α-Terpinene |
| dl-Menthol | Fenchone | o-Cymene |
| α-Terpineol | Linalool | D-Limonene |
| γ-Terpineol | Fenchol, exo- | Eucalyptol |
| Citronellol & Nerol | trans-Nerolidol | Ocimenes |
| Pulegone | Caryophyllene oxide | Cis-β-Farnesene |
| Geraniol | Gauiol | α-Humulene |
| Geranyl acetate | Cedrol | Valencene |
| α-Cedrene | β-Eudesmol | cis-Nerolidol |
| β-Cedrene | Phytol I & II | Caryophyllene |

## 12.4 APPENDIX D – RESIDUAL SOLVENT LIST

**List of Residual Solvents identifiable with GC-FID:**

| Residual Solvent | | |
|---|---|---|
| 1,1,1,2-Tetrafluoroethane | 2,3-Dimethylbutane* | Triethylamine |
| Methanol | 3-Methylpentane* | Heptane |
| 2-Methylbutane | n-Hexane* | Ethylene glycol |
| Pentane | 1-Propanol | Toluene |
| Ethanol | Hexane* | 1-Pentanol |
| Ethyl ether | 2-Butanone | Ethylbenzene |
| Acetone | Ethyl acetate | m-Xylene |
| 2,2-Dimethylbutane* | Chloroform | p-Xylene |
| Isopropanol | Cyclohexane* | o-Xylene |
| Acetonitrile | Benzene | Dimethyl sulfoxide |
| Methylene chloride | Acetic acid | Tridecane |
| 2-Methylpentane* | iso-Octane | Methyl acetate |
| tert-Butylmethyl ether | Methylethyl ketone | 1-Butanol |
| 2-Butanol | 2-Methyl-1-propanol | Isopropyl acetate |
| 3-Methyl-1-butanol | Isobutyl acetate | Butyl acetate |
| Propane | Butane | Iso-Butane |

*Hexanes