A Method Development Process for Analysis of Metals in Cannabis by ICP-MS

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Introduction
As cannabis becoming legal in more states, testing requirements are also being pushed to ensure safety. One of the tests that is required in markets testing, is the prescribed metals content, to test for heavy metals such as arsenic and lead found in soil or water used to grow the plants. The metals, being限量性, are required to be monitored, especially when the plants are used as medicine. Shimadzu would like to thank ACT Laboratories for performing the sample preparation, data acquisition, and analysis.

Typical Method Development Process
1. Confident State Regulations
Always check current regulations in your state regarding required metals testing. This will include which metals must be tested, at what levels they must be tested, as well as other requirements that must be included in the method such as sampling, sample preparation, acceptable techniques, calibration requirements, etc. Because of the states required much different metals, it will help to have a low level of detection methods on hand, especially for metals such as Mercury, Cadmium, and Lead. However, most state’s regulations only call for testing of heavy metals.

2. Sample Preparation

Microwave digestion
Microwave digestion is the sample preparation technique that is most often used for preparation of cannabis samples. In this technique a sample and acid solution are added to a closed microwave digestion vessel. This is then heated in a microwave which allows rapid breakdown of the sample and the creation of a sample that can be digested in the acid solution.

Determination of the acid concentration is a balance of being strong enough to fully digest the sample into solution, but not so concentrated that it will require excessive dilution to bring the acid into typical analysis range. The final solution that is introduced into the instrument is typically 2% acid solution, but not so concentrated that it will require excessive dilution to bring the acid into typical analysis range.

As Shown in Table 3, this analysis provided good linear calibration curves for all elements with linearities at 0.99999 or better.

<table>
<thead>
<tr>
<th>Metal</th>
<th>Detection Limit (ppb)</th>
<th>Quantitation Limit (ppb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As</td>
<td>0.001</td>
<td>0.005</td>
</tr>
<tr>
<td>Cd</td>
<td>0.001</td>
<td>0.003</td>
</tr>
<tr>
<td>Hg</td>
<td>0.001</td>
<td>0.003</td>
</tr>
<tr>
<td>Pb</td>
<td>0.001</td>
<td>0.003</td>
</tr>
<tr>
<td>Tl</td>
<td>0.001</td>
<td>0.003</td>
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</tbody>
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Method Optimization and Data Analysis:
Once the data is collected the results of each analyte’s masses are compared to determine if masses have good isolate reach and provide required results. The mass list can be refined based on calibration, to a likely species, and high intensity. This can limit detection to masses that are more likely to be metals, and lower the theoretical detection limit (LOD).

Experimental
A method is developed using the outlined method development process. All samples were prepared by ACT Laboratories LLC and analyzed on a Shimadzu ICP-MS. Shimadzu would like to thank ACT Laboratories for performing the sample preparation, data acquisition, and analysis.

As Shown in Table 5, this analysis provided good linear calibration curves for all elements with linearities at 0.99999 or better.

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Conclusion
There are many factors when developing an ICP-MS method for metals analysis. Regulations that vary by state and can change over time make it difficult to create a single method that applies to all testing. This paper using several internal standard/additives to this method, that could be used for all states, but is not suitable for their regulations. An example method was developed using this approach providing a single universal method for this analysis.

References
“Astroz’s heavy metal contamination in cannabis flower using the Shimadzu ICP-MS/MS, Muhamed et al.”

Acknowledgments

Table 1

<table>
<thead>
<tr>
<th>Concentration</th>
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<tbody>
<tr>
<td>Unspiked</td>
<td>99.7%</td>
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<tr>
<td>Spiked</td>
<td>96.6%</td>
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Table 2

<table>
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<tr>
<th>Sample</th>
<th>Recovery</th>
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<tbody>
<tr>
<td>A</td>
<td>92.6%</td>
</tr>
<tr>
<td>B</td>
<td>94.7%</td>
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<tr>
<td>C</td>
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