# Rapid, Efficient, and Safe Sample Preparation of Mixed Cannabis Samples in a Single Microwave Digestion Batch

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## Introduction

Labs that test cannabis and hemp typically work in a high throughput environment and receive samples that include plant material, extracts, tinctures, inhaled products, and a wide variety of edibles. Typically, these samples are batched by sample type and then run as batches in a microwave digestion system. Previously, the only way to batch all of the sample types listed above required a high pressure device which are very expensive.

CEM has developed a method to prepare all of these samples in a single batch in a MARSXpress vessel equipped with a simple, three-part MARSXpress vessel. Up to forty mixed samples can be prepared in a single batch. It provides the highest throughput and most cost effective solution available to laboratories. For this study, eleven different sample types, of all varieties, were digested in a single batch, in duplicate. All together 24 samples and blanks were run simultaneously in a single batch for maximum throughput.

## Experimental Conditions

Samples: Eleven samples were chosen to simulate the wide variety of samples that are tested in the cannabis lab. These were sourced from local hemp dispensaries. The samples are shown in this photograph and include: hemp flower, hemp oil, MCT oil, topical cream, crude CBD oil, beef jerky, peanut butter, ghee (butter), granola bar, gummy snacks, hard candy.
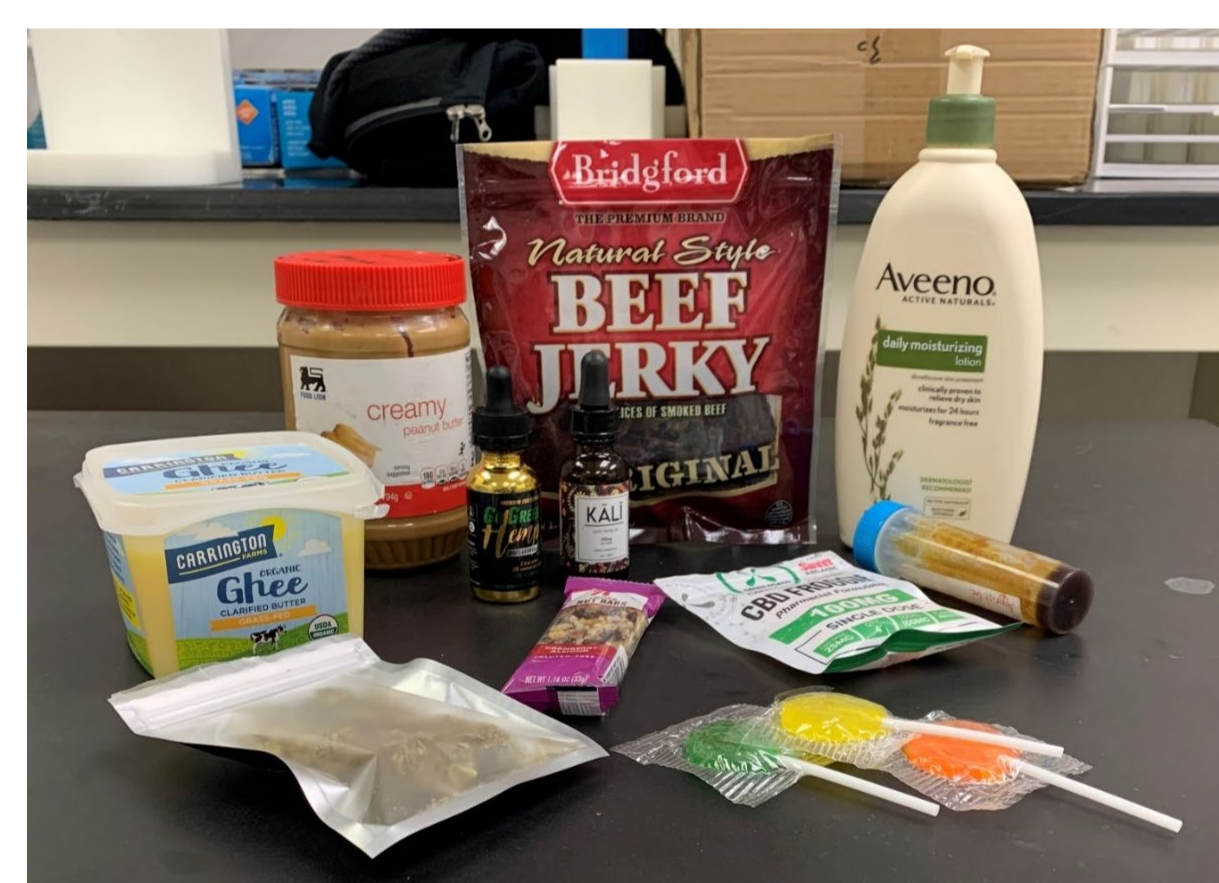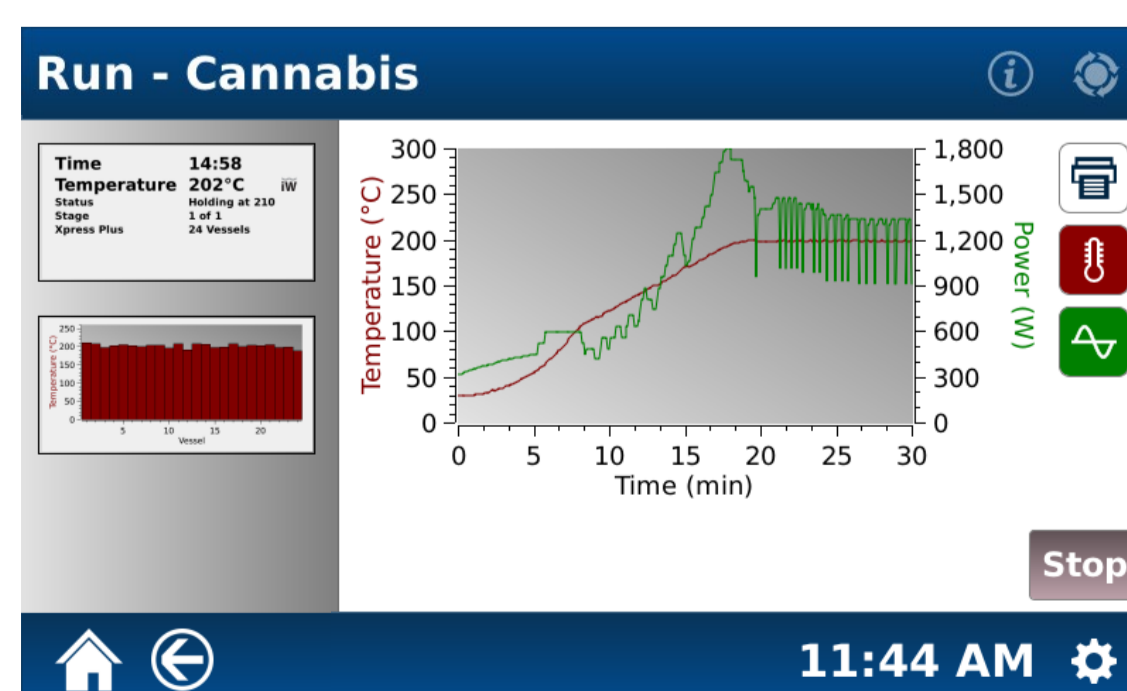
Sample Preparation: 0.5 grams of each sample were digested in a MARS 6 with MARSXpress Plus vessels in order to meet the sampling requirements for California heavy metals testing. An acid mixture of 9 mL nitric acid and 1 mL HCl was then added and the vessels were sealed. A pre-programmed Cannabis method was selected on the MARS 6 touchscreen interface. This method was developed to heat the samples to 210 °C and hold them for a specified time in order to achieve a complete digestion of all sample types. The samples were then diluted to 50 mL volume with DI water and analyzed via ICP-MS.



Figure 1. Cannabis and hemp containing samples

Table 1. MARS 6 Cannabis One Touch Method Conditions

| Parameter | Setting |
|---|---|
| Power | 1800 W |
| Ramp Time | 20 min |
| Hold Time | 10 min |
| Temperature | 210 °C |



MARS 6 with MARSXpress Plus vessels

## Experimental Conditions

Table 2. ICP-MS 2030 Operating Conditions

| Parameter | Setting |
|---|---|
| Radio Frequency Power | 1.20 kW |
| Sampling Depth | 5.0 mm |
| Plasma Gas | 8.0 L/min |
| Auxiliary Gas | 1.10 L/min |
| Carrier Gas | 0.70 L/min |
| Mix Gas | 0.00 L/min |
| Cell Gas | 6.0 mL/min |
| Cell Voltage | -21 V |
| Energy Filter | 7.0 V |
| Chamber Temperature | 5 °C |



Shimadzu ICP-S-2030M

## Results and Discussion

**Figure 3** illustrates the ramping of the varied sample types. Note that during the ramp certain samples are higher in temperatures than others. These samples are likely higher in sugars or carbohydrates as these bonds break first. The MARS 6 with iWave technology and advanced software makes continuous adjustments to modulate power in order to maintain the proper heating conditions. The robustness of the high throughout MARSXpress Plus vessel also contributes to the ability to run mixed samples in one batch. In **Figure 4** the samples are in the hold phase and the temperatures of the varied samples have equilibrated. **Figure 5** shows a subset of samples that have been completely digested and diluted to volume.
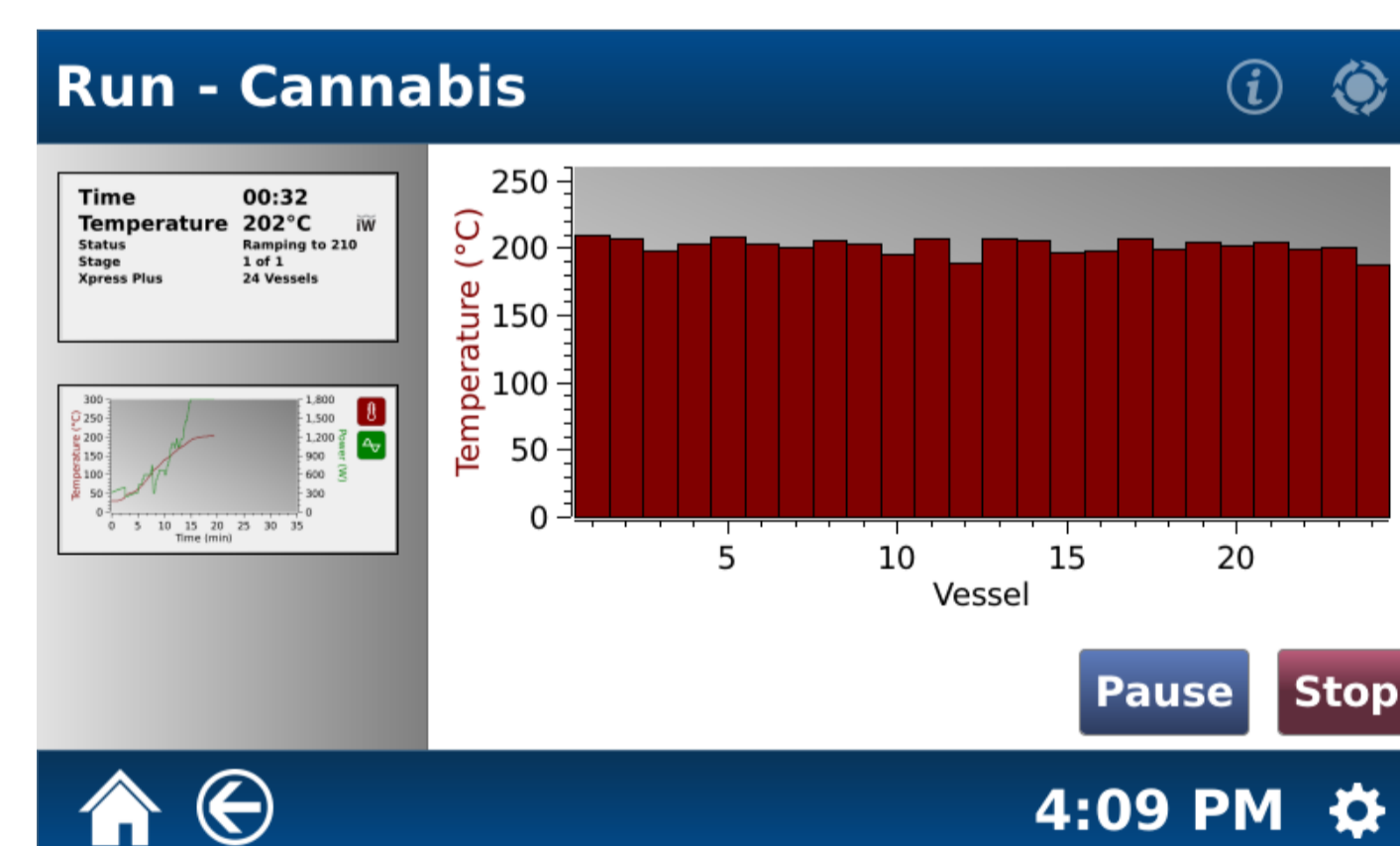


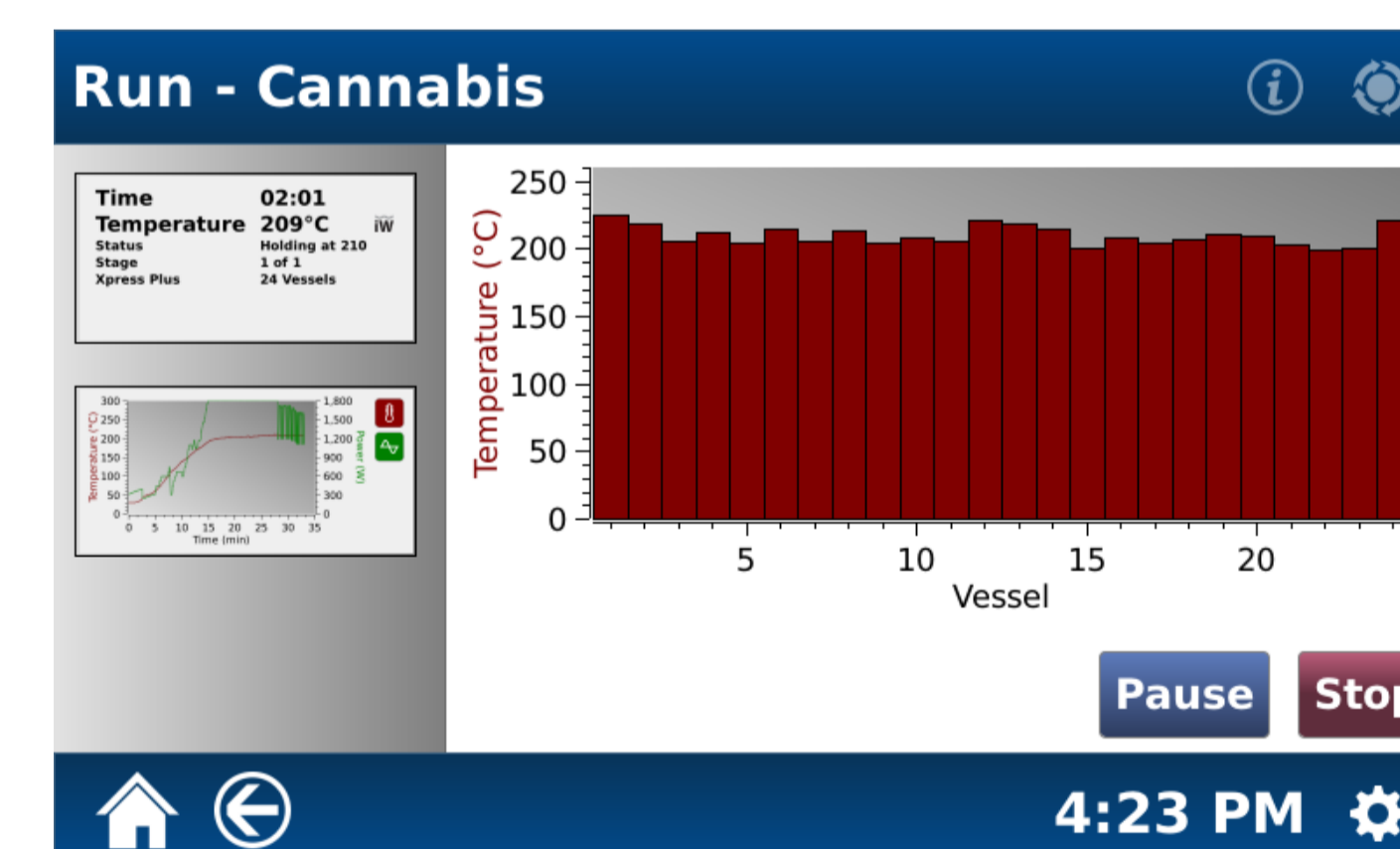Figure 3. Temperature of all vessels during ramp



Figure 4. Temperature of all vessels during hold



Figure 5. Diluted samples

## ICP-MS Analysis

Table 3. Instrument Measurement Parameters

| Element | Mass | Internal Standard | Cell Gas | Cal. Range (ppb) | Cal. R² | Detection Limit (ppb) | Integ. Time (sec) | No. of Scan | Repeat No. | Spiked Concen. (ppb)[a] |
|---|---|---|---|---|---|---|---|---|---|---|
| As | 75 | Sc (45) | On | 0.6 - 10 | 0.99946 | 0.00818 | 2.0 | 10 | 3 | 5 |
| Cd | 111 | In (115) | Off | 0.6 - 10 | 0.99992 | 0.00131 | 2.0 | 10 | 3 | 5 |
| Hg | 200 | Bi (209) | Off | 0.6 - 10 | 0.99978 | 0.00226 | 2.0 | 10 | 3 | 5 |
| Pb | 208 | Bi (209) | Off | 0.6 - 10 | 0.99986 | 3.14E-4 | 2.0 | 10 | 3 | 5 |

a. Spiked concentration is the spiked concentrations of different elements in the final measurement solutions after dilution.

Table 4. ICP-MS Results

| | | $^{75}$As | $^{111}$Cd | $^{200}$Hg | $^{208}$Pb | | | $^{75}$As | $^{111}$Cd | $^{200}$Hg | $^{208}$Pb |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Blank | Mean value | n.d. | n.d. | n.d. | n.d. | Lotion | Mean value | n.d. | n.d. | n.d. | n.d. |
| | RSD (n = 3) | --- | --- | --- | --- | | RSD (n = 3) | --- | --- | --- | --- |
| Fortified blank | Mean value | 5.23 | 5.23 | 5.1 | 5.08 | Fortified lotion | Mean value | 4.96 | 5.09 | 5.16 | 5.03 |
| | RSD (n = 3) | 2.03 | 0.76 | 3.57 | 1.59 | | RSD (n = 3) | 11.08 | 1.21 | 3.12 | 2.62 |
| Recovery (%) | | 105 | 105 | 102 | 102 | Recovery (%) | | 99 | 102 | 103 | 101 |
| Hard candy | Mean value | n.d. | n.d. | n.d. | 0.0116 | Froggy | Mean value | n.d. | n.d. | n.d. | 0.0407 |
| | RSD (n = 3) | --- | --- | --- | 1.79 | | RSD (n = 3) | --- | --- | --- | 0.66 |
| Fortified hard candy | Mean value | 5.12 | 4.92 | 5.05 | 5.13 | Fortified froggy | Mean value | 4.76 | 4.9 | 4.83 | 5.07 |
| | RSD (n = 3) | 3.51 | 1.68 | 0.19 | 1.74 | | RSD (n = 3) | 0.95 | 0.74 | 1.46 | 1.92 |
| Recovery (%) | | 102 | 98 | 101 | 102 | Recovery (%) | | 95 | 98 | 97 | 101 |
| Granola bar | Mean value | n.d. | 0.125 | n.d. | 0.0186 | Hemp flower | Mean value | 0.0231 | n.d. | n.d. | 0.163 |
| | RSD (n = 3) | --- | 1.63 | --- | 3.02 | | RSD (n = 3) | 6.07 | --- | --- | 1.09 |
| Fortified granola bar | Mean value | 4.55 | 5.07 | 5.09 | 5.06 | Fortified hemp flower | Mean value | 4.81 | 5.05 | 5.11 | 5.15 |
| | RSD (n = 3) | 13.53 | 1.61 | 1.58 | 1.15 | | RSD (n = 3) | 9.68 | 1.22 | 0.6 | 1.48 |
| Recovery (%) | | 91 | 99 | 102 | 101 | Recovery (%) | | 96 | 101 | 102 | 100 |
| MCT oil | Mean value | n.d. | n.d. | n.d. | n.d. | Beef jerky | Mean value | n.d. | 0.0482 | n.d. | 0.057 |
| | RSD (n = 3) | --- | --- | --- | --- | | RSD (n = 3) | --- | 0.77 | --- | 1.65 |
| Fortified MCT oil | Mean value | 5.39 | 5.04 | 5.13 | 4.94 | Fortified beef jerky | Mean value | 4.81 | 5.07 | 5.06 | 5.22 |
| | RSD (n = 3) | 1.36 | 1.54 | 1.24 | 1.89 | | RSD (n = 3) | 7.54 | 1.17 | 1.25 | 1.86 |
| Recovery (%) | | 108 | 101 | 103 | 99 | Recovery (%) | | 96 | 100 | 101 | 103 |
| Ghee | Mean value | n.d. | n.d. | n.d. | n.d. | Peanut butter | Mean value | n.d. | 0.111 | n.d. | 0.0511 |
| | RSD (n = 3) | --- | --- | --- | --- | | RSD (n = 3) | --- | 3.92 | --- | 1.32 |
| Fortified ghee | Mean value | 5.46 | 5.05 | 5.15 | 5 | Fortified peanut butter | Mean value | 5.22 | 5.28 | 5.13 | 5.11 |
| | RSD (n = 3) | 3.8 | 2.01 | 1.19 | 1.73 | | RSD (n = 3) | 5.32 | 1.08 | 1.45 | 2.93 |
| Recovery (%) | | 109 | 101 | 103 | 100 | Recovery (%) | | 104 | 103 | 103 | 101 |
| Hemp oil | Mean value | n.d. | n.d. | n.d. | 0.0723 | Conc. CBD oil | Mean value | n.d. | n.d. | n.d. | 0.0662 |
| | RSD (n = 3) | --- | --- | --- | 1.6 | | RSD (n = 3) | --- | --- | --- | 0.78 |
| Fortified hemp oil | Mean value | 5.27 | 5.18 | 5.2 | 5.09 | Fortified Conc. CBD oil | Mean value | 5.41 | 5.17 | 4.93 | 5.05 |
| | RSD (n = 3) | 11.91 | 1.53 | 2.1 | 1.5 | | RSD (n = 3) | 10.43 | 0.7 | 2.03 | 2.74 |
| Recovery (%) | | 105 | 104 | 104 | 100 | Recovery (%) | | 108 | 103 | 99 | 100 |

## Conclusion

- The MARS 6 was able to completely digest a wide variety of cannabis samples in a single batch
- Up to 24 samples can be run at a time in a single batch
- The MARSXpress Plus vessel provides the combination of high throughput and robustness necessary for this work
- The Glass Expansion Niagara Rapid Rinse accessory significantly reduced our analysis time
- The Shimadzu ICP-MS 2030 provided superior analysis and showed excellent recoveries and RSD's