Increasing Laboratory Productivity using Microwave Digestion with an Inexpensive Disposable Glass Vessel

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Abstract

Disposable glass inserts are the latest breakthrough in microwave digestion vessel technology. The system incorporates hydrogen peroxide to create a pressure equilibrium inside the digestion vessel thus enabling the use of a thin walled glass insert. This low cost disposable insert can be used to digest a wide variety of sample materials. In the past, disposable liners were only available using very expensive digestion systems that operate at pressures up to 3000 psi. CEM's disposable glass technology incorporates a patented vent and reseal design that works at much more moderate and safer pressures of around 500 psi. Once samples are digested, they are diluted in the same glass insert and placed in a rack for ICP or ICP-MS analysis. This eliminates all transfers and significantly increases laboratory productivity. The technology behind this innovative design will be presented and analytical results for a variety of sample types will be shown.

Figure 1. Glass insert components

Introduction

The disposable insert has revolutionized microwave digestion by improving lab efficiency and reducing transfers in microwave digestion. By utilizing an inexpensive, disposable, glass insert; weighing, digestion, and sample dilution can all be performed in the same vessel without the need to transfer. After the digestion is complete, the glass insert can simply be thrown away and the vessel is ready to digest the next sample without the need for washing.

Technology

The disposable glass insert works by creating a moderate pressure mini chamber wherein sample digestion is performed. As shown in Figure 2, the disposable glass insert is placed into the vessel containing a small amount of hydrogen peroxide.

After the glass insert containing the sample and acid is placed into the vessel, the system is sealed to eliminate the risk of cross contamination during digestion as shown in Figure 3.

The sealed vessels are placed into a turntable and placed into the microwave chamber and as microwave energy is applied, heat is generated. This energy first heats the hydrogen peroxide, which produces vapors that apply pressure to the glass insert plug. This pressure equilibrium between the inside and outside of the glass insert lends structural rigidity to the system.

As pressure builds up inside the vessel the amount of force inside the glass insert overcomes the external pressurization and the sample is allowed to vent into the microwave chamber where the noxious vapors are safely removed from the system. After venting of excess pressure the vessel reseals to eliminate the risk of losing volatile elements. Figure 5 illustrates the patented dual vent and reseal technology.

Technology continued

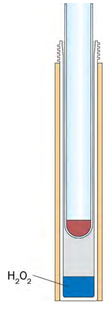

Figure 2. Vessel with hydrogen peroxide and disposable glass insert

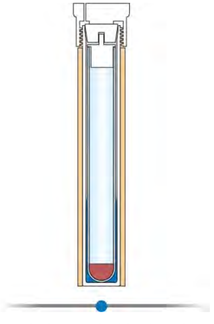

Figure 3. Sealed vessel with glass insert and plugs

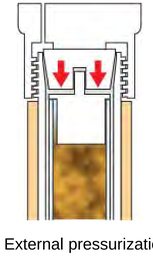

Figure 4. External pressurization of glass insert

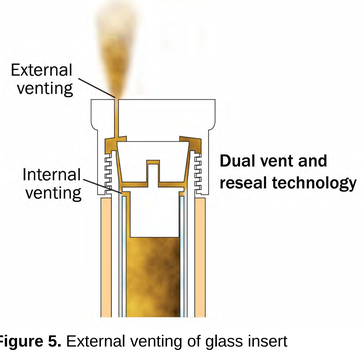

Figure 5. External venting of glass insert

Experimental Conditions

Twenty four hemp and hemp oil samples were digested in a MARS 6 Microwave Digestion System in the Disposable Glass Inserts. Samples weighing 0.25 g were added to the glass inserts along with 10 mL HNO_3 and a 0.4 – 60 ppm spike solution containing As, Hg, Cd, Pb. The vessels were assembled and placed into the MARS 6 where the Glass Cannabis One Touch Method was selected. Digestion conditions for the One Touch Method are as follows:

- Ramp: 20 min
- Hold: 10 min
- Temp: 200 °C

Upon completion of digestion each sample was diluted to 50 mL using the Glass Insert Dilution Stand, eliminating sample transfer. Samples were analyzed by ICP-OES on an Agilent 5110 with the settings shown in Table 1.

Experimental Conditions continued

Table 1. ICP-OES Conditions

Parameter	Setting
Plasma viewing mode	Axial
Read time	30 s
Measurement replicates	3
RF incident power	1400 W
Plasma argon flow rate	12 L/min
Nebulizer argon flow rate	0.7 L/min
Auxiliary argon flow rate	1.0 L/min
Inner diameter of the torch injector	1.8 mm
Nebulizer type	Seaspray
Spray chamber type	Glass cyclonic double-pass
Sample tubing	White/white 1.04 mm ID
Internal standard tubing	Orange/green 0.38 mm ID

Results

All samples were completely digested in the MiniClave vessels with disposable glass inserts and produced clear, particulate free digestates as shown in Figure 6. ICP analysis in Table 2 below shows that all spiked samples were recovered and that no contamination from the glass occurred.

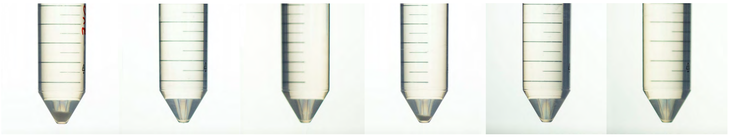

Figure 6. Clear particulate free digested samples

Table 2. Spike recovery for samples digested in Disposable Glass Inserts

Sample	As (%)	Cd (%)	Hg (%)	Pb (%)
Topical Cream	104.7	105.6	101.8	102.2
Fruit Snack	105.0	106.1	99.7	91.0
Cannabis Flower	106.8	102.0	95.6	102.5
CBD Oil 4.5k	101.5	101.3	92.3	102.6
CBD Oil 6k	100.9	100.4	91.9	101.8

Conclusion

The disposable glass inserts completely digested a variety of samples in a single batch at lower pressures than traditional SRC technology yielding a safer work environment for lab technicians.

The vent and reseal design of the disposable glass inserts allows excess pressure to be safely vented off while retaining volatile elements in solution.

The workflow was dramatically improved by using the Disposable Glass Insert because the sample was weighed, digested, and diluted in the glass insert without the need to transfer the sample from vessel to vessel. The glass inserts were then disposed of upon completion of analysis eliminating the need for vessel washing.