



A New Microwave Digestion System Designed for Small Volumes as Low as 0.5 mL

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Introduction

Traditional microwave digestion systems have been available for over thirty years. These systems are designed to process samples up to 1 g in weight or more, using large digestion vessels. Clinical analyses, such as blood, urine, and hair, often use just a few milligrams of sample and 1 mL of reagent or less. These cannot be processed in the batch-style microwave digestion system. Recently, CEM has introduced a new Discover SP-D™ Clinical system, designed specifically for digestion of smaller clinical sample types. The system utilizes a small, focused cavity and produces only 300 watts of energy, as opposed to nearly 2000 watts for many batch systems. Samples are placed in either 4 or 10 mL vials made of glass or Quartz, acid is added, then the vials are closed with a simple snap-on cap. The samples are then loaded into an autosampler that can prepare up to 48 samples with varied sample sizes, acids, and digestion conditions in a sequential fashion, as shown in Figure 1. Total digestion time for each sample is typically 7 – 10 minutes, making this a high-throughput, unattended system. We will show examples of various clinical samples with digestion conditions and analysis.

Figure 1. Discover SP-D Clinical with Mixed Clinical Samples



Procedure and Method

Each sample was weighed into a 10 mL Pyrex vial, as shown in Figure 2. Concentrated HNO₃ was added according to Table 1 and each vial was capped with a disposable cap. The samples were placed into the Discover SP-D Clinical automation rack and the software was programmed according to Table 1 for each sample. The Discover SP-D Clinical digested all 4 samples in about 30 minutes. Upon complete digestion, each sample was diluted up to 50 mL with deionized H₂O.

Figure 2. Weighing Hair Sample into Vial



Results and Discussion

The Discover SP-D Clinical was successful in digesting all of the samples. Figure 3 shows the temperature graph of the hair sample, indicating that the target temperature was achieved and held for the programmed 3 minutes. Upon dilution, each sample was clear and colorless, and free of particulate. As shown in Figure 4. As pressure built up during the digestion, the Discover SP-D Clinical vented the samples at pre-programmed vent points to ensure volatile elements would not be lost.

Samples

Four common clinical samples were digested including: blood, hair, finger nails, and brain tissue. Each sample was digested using HNO₃ in a 10 mL Pyrex vessel with snap-on cap. The Discover SP-D Clinical uses an automation rack for holding the samples, so it is able to digest samples with different acids, acid volumes, and heating profiles, sequentially and record digestion conditions for each sample. Table 1 shows the sample weight and acid volume of each sample, as well as the digestion method.

Table 1. Samples, Weight, Acid Volumes, and Digestion Method

Sample	Weight/Volume	Acid Volume (mL)	Temperature (°C)	Ramp	Hold	Cooling
Hair	0.1 g	2 mL	160	3	3	1
Finger Nails	0.1 g	2 mL	175	3	1	
Blood	1 mL	1 mL	160	4	3	1
Brain Tissue	0.3 g	1 mL	180	4	3	1

Figure 3. Temperature Graph of Digestion of Hair Sample



Figure 4. Clear and Particulate-free Digests



Conclusions

The Discover SP-D Clinical is ideally suited for digestion of clinical samples. The automation rack allows for mixing of samples, acids, acid volumes, and digestion methods. This is of great benefit for research and development labs that are working to optimize digestion conditions of many different samples. The automation rack is also very helpful for labs with high-throughput, as the technician can prep the entire rack and program the unit, then start the run and leave the Discover SP-D Clinical running unattended, while they tend to other tasks. The system can even be run overnight so that a technician has up to 48 digested samples ready for analysis, first thing in the morning. The two vial sizes, shown in Figure 5, and material options give additional flexibility to labs that require ultra clean digestions of any size clinical sample. Samples as small as 0.05 g and 0.5 mL acid can be digested in the 4 mL Quartz vial and ppt detection levels can still be achieved.

Figure 5. 4 and 10 mL Vials



Instrumentation

The Discover SP-D Clinical is the only microwave digestion system designed for small clinical samples. The small microwave cavity and sequential operation focuses all of the microwave energy on the sample, while the small vessels allow for as little as 0.5 mL of reagent to be used for the digestion. The system comes with an automation deck, allowing for up to 48 samples to be run, unattended. Digestion vials are available in 4 and 10 mL volumes, in Pyrex or Quartz. Table 2 shows allowable acid volume.

Table 2. Minimum and Maximum Acid Volume for Vials

Vessel Size	Minimum Acid Volume	Maximum Acid Volume
4 mL	0.5 mL	1.5 mL
10 mL	1.0 mL	4.0 mL