



Introduction

Pharmaceutical corporations are tasked with testing their products extensively prior to distribution. Testing is performed on raw materials, excipients, and during the quality control process for the finished product. Traditional techniques require significant user interaction and are subject to error. The Phoenix™ Microwave Ashing System provides a safe and reliable alternative and meets all muffle furnace requirements for the United States, British, Japan, European, and other Pharmacopeias.

Traditional Methods

Traditional techniques require Bunsen burners, heat lamps, hot plates, and muffle furnaces kept at high temperature virtually continuously. This equipment is prone to safety risks, as users are directly exposed to hot surfaces and open flames. They are also more prone to error, as weights must be taken, transferred manually, and calculated. Other furnace systems exist, but temperature uniformity, temperature measurement, and exposure to hot surfaces continue to be a concern. The Phoenix is different.



Microwave Ashing

The Phoenix is a microwave furnace that meets all microwave and standard muffle furnace requirements listed in ASTM, USP (<281> ROI & <733> LOI), AOAC, FDA, ISO, DIN, ASTM D5630-94, and ASTM D1506-94b. The system is programmable with up to 20 methods and can ash up to 15 samples at a time. Microwave energy ensures rapid heating of the furnace while the design of the continuous silicon carbide heating element maintains temperature uniformity throughout the cavity. Because the element is in the side of the furnace walls, it will not damage metallic crucibles, which can be used in the Phoenix.

All temperature measurements are performed using NIST-traceable thermocouples. The air temperature is measured in the middle of the furnace, ensuring an accurate measurement. The exterior of the furnace is virtually cool enough to touch, even when the inside is at temperature. In order to access the interior of the furnace, the furnace door is placed in a holder that prevents the user from being exposed to the hot door surface, thereby reducing the operator exposure to hot surfaces. Upon method completion, cool-down is rapid.

All sulfated ashing steps can be performed in the Phoenix with the Sulfated Ashing Option (SAO), eliminating the

need for additional equipment. Acid can be added to the sample in the crucible and placed directly into the Phoenix. A lower temperature sulfating/ashing step can then proceed prior to the high-temperature dry ashing of the organic sample. The system can be programmed with a pause step to allow the sulfated crucible to be removed and weighed prior to the dry ashing step. The Phoenix SAO is designed to evacuate toxic acid vapor and neutralize it for a more environmentally sound, laboratory-friendly solution to the hazardous fumes. The system also helps the laboratory comply with ISO 14000 Environmental Management requirements to eliminate greenhouse gases.

The optional workstation allows quantification for document control and automatically determines the %ROI, %LOI or %Ash. Once generated, sample information cannot be modified, aiding in 21 CFR Part 11 compliance. All weighing and method information is included in the data file, as well as the final result, user-entered sample ID, and a date/time stamp indicating when the sample was ashed. All methods are stored automatically and the system provides the security of a password lock to reduce the possibility of erroneous method modification.

Results

The Phoenix complies with all requirements included in both USP <733> and <281>. It can easily be used to determine the LOI for any type of powder and will quickly ash samples to determine the ROI for trace metal or other analysis. To demonstrate the accuracy and precision of the Phoenix, several different pharmaceutical compounds were tested and the results shown in Table 1 below.

	Conventional Muffle Furnace			Microwave Muffle Furnace		
	Temperature (°C)	Time (min)	ROI (%)	Temperature (°C)	Time (min)	ROI (%)
Mycostatin	800	60	3.5	800	40	3.09
Amoxicillin Trihydrate (antibacterial)	800	540	0.1-0.2	800	30	0.12
Nicotinamide (feed grade)	800	60	1.10	800	10	1.08
Dextromethorphan	800	60	<0.1	800	30	0.04

Table 1: Trace metal analysis using Phoenix with Sulfated Ashing Option

As demonstrated above, the results from the Phoenix are all within what would be expected of the individual compounds. Interestingly, most of the tests were complete in half of the amount of time (or less) than required using a traditional muffle furnace. The time for the conventional muffle furnace does not include additional operator actions, such as using a hot plate or Bunsen burner, which may require an additional 30 minutes of operator time prior to insertion of the sample into the muffle furnace. In most laboratories, this step requires constant attention as the technician cannot leave an open flame. As noted above, the Phoenix SAO can be used to perform the sulfating/ashing step with sulfuric acid, reducing the amount of operator attention and creating a safer laboratory environment.

Conclusions

The Phoenix meets the requirements of USP <281> and <733>, while also providing a simple method of analysis with a high degree of accuracy. The safety and automated nature of the system make it very operator-friendly.

For additional information on how to use the Phoenix for Sulfated Ashing Determination, see <http://www.cem.com/phoenix-sulfated-ashing.html>

- "Efficiency improvement for sulfated ash determination by usage of a microwave muffle furnace" Hinz, D.C.; *J. Pharm. Biomed. Anal.*, **2007**, 43, 1881. <http://www.sciencedirect.com/science/article/pii/S0731708507000258>
- "Microwave Muffle Furnace Technology for Use in the Test for Residue on Ignition" Donaghy, C.; Smart, M.; *Pharmacopeial Forum*, **2002**, 28, 2029.

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APPLICATION NOTES