



PYRO INFOPACK

SULFATED ASHING



Thank you for your interest in Milestone's PYRO microwave muffle for Sulfated Ashing. This Infopack will help you learn how the PYRO can enhance your lab's efficiency and safety for sulfated ashing applications.

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MILESTONE
H E L P I N G
C H E M I S T S



FAST AND SAFE
ASHING ANALYSIS
VIA MICROWAVE
MUFFLE FURNACE



PYRO

Advanced Microwave Muffle Furnace



FAST
ASHING



HIGH SAMPLE
THROUGHPUT



IMPROVED
WORKING CONDITIONS



FLEXIBLE
CONFIGURATIONS



ASTM, USP, SEMI AND ISO
COMPLIANCE

IMPROVING TIME AND THROUGHPUT WITH MICROWAVE ASHING

Dry ashing is based on the decomposition of organic material by the action of oxygen at elevated temperatures. The determination of the ash content, whether for process control or as a preparation method for other analytical techniques, is an important test performed daily in thousands of laboratories worldwide. The ashing process is done in conventional muffle furnaces that often offer inefficient, tedious, and time-consuming procedures. The use of microwave heating for dry ashing applications has been represented a breakthrough in the process and quality control of samples like polymers, petroleum, food and feeds, pulp paper and pharmaceuticals. The PYRO microwave muffle furnace, is available in three interchangeable configurations:

HIGH SAMPLE THROUGHPUT

SULFATED ASHING

ULTRAFAST HEATING



The PYRO is built to offer superior working conditions and a more efficient ashing procedure on a wide range of matrix. It incorporates in a single platform a rugged hardware, a reliable microwave-transparent muffle, an efficient exhaust, an accurate temperature control and a smart user interface.

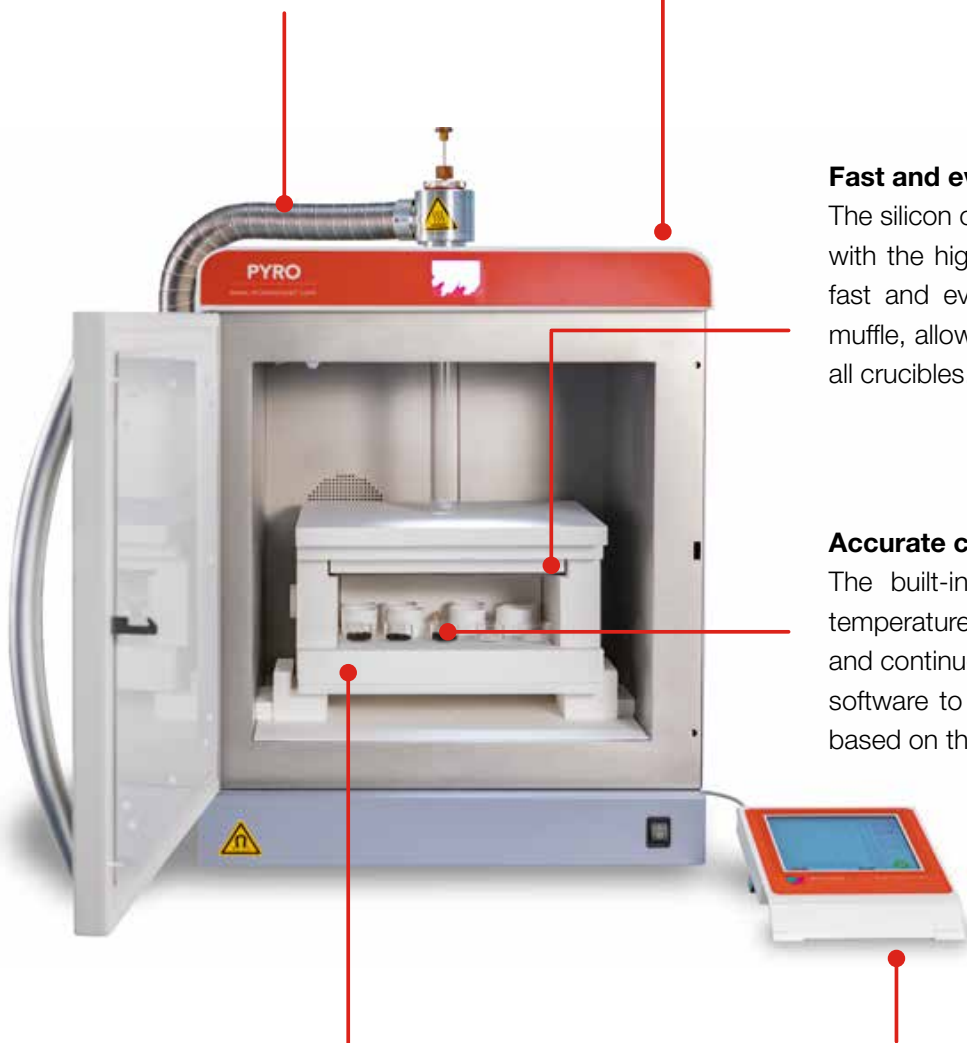
HIGH-CAPACITY FOR SUPERIOR PRODUCTIVITY

Efficient airflow

The unique design of the venturi system enables faster ashing and safer conditions without the need of an additional gas line. The continuous and adjustable airflow expedites the sample combustion and the removal of fumes during the ashing process.

Rugged and powerful hardware

The PYRO is designed to last! The stainless-steel construction and the high microwave power offer high reliability, fast heating rate, and superior working conditions as it does not expose the operator to heat or fumes.



Fast and even heating

The silicon carbide heating element, along with the high microwave power, provides fast and even heating across the entire muffle, allowing reproducible conditions in all crucibles.

Accurate control

The built-in sensor controls the muffle temperature during the entire program and continuously dialogues with the smart software to adjust the microwave energy based on the desired temperature profile.

Microwave transparent muffle

The microwave transparent muffle meets high throughput and large sample mass needs. It is equipped with a quartz plate for easy cleaning and filters to ensure proper sample combustion.

Smart user interface

All the process parameters are controlled by the advanced terminal and software. Simply select a built-in method or create a new one and press "START" to begin the ashing procedure.

SHORT ASHING TIME

Sample	Temperture (°C)	Time (min)
Pet Food	575	20
Milk Powder	550	20
Wheat Flour	900	50
Polypropylene	650	20
PVC	900	15
Coal	750	20
Heavy Oil	550	45
Rubber Tyre	550	45
Lactose	600	60
Antibiotics	850	60

Typical ashing time with PYRO

MILESTONE GREEN TECHNOLOGIES

The PYRO embraces the values and benefits of the green extraction approach, in fact it offers more efficient heating and saves energy. The accurate microwave emission and the great insulating capacity of the microwave-transparent muffle minimize energy waste.



ULTRAFAST MUFFLE FOR UNMATCHABLE ASHING TIME

The PYRO with ultrafast muffle reaches a temperature of 800°C in just 5 minutes, starting from room temperature and enables applications up to 1200°C. The temperature is monitored and controlled via a contactless infrared sensor. The unmatched heating rate of this PYRO configuration offers a solid option for QC labs to provide rapid feedback to their production and to any lab dealing with rush samples. Due to its heating rate, the ultrafast muffle has a natural fit for many high-temperature applications requiring no heating/cooling inertia, such as sintering or fusion.



FAST ASHING



RUSH SAMPLES



HIGH TEMPERATURE APPLICATIONS

EFFICIENT WORKFLOW IN SULFATED ASHING DETERMINATION

MICROWAVE SULFATED ASHING

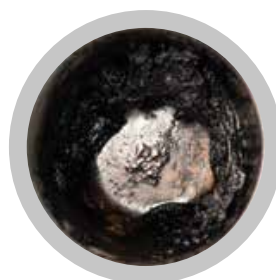
Ashing of samples like pharmaceutical and petroleum often involves the use of sulfuric acid that leads to hazardous acid vapours and is a labour-intensive procedure. The traditional approach requires the manual fuming of acid for several hours with high risks for the operator. The PYRO Sulfated Ashing configuration enables the operator to complete this test in a single step. Sulfuric acid is added to the sample, and the crucibles are placed into the muffle without any preliminary charring step. The acid fumes are continuously collected, condensed, and neutralized by the dedicated water-cooled scrubber module. With the PYRO Sulphated Ashing configuration the procedure is typically completed in less than two hours, eliminating any tedious and unsafe handling.



Sample



Sample with sulfuric acid



Charring step



At the completion of the ashing process



FASTER ASHING TIME

HIGHER THROUGHPUT

SUPERIOR SAFETY

SINGLE STEP PROCEDURE

FULL TRACEABILITY

SMART USER INTERFACE

All the PYRO configurations are controlled via a compact full-colour, touchscreen display. The user interface runs a user-friendly, icon-driven, multi-language software to provide easy control of the process. The software automatically regulates the microwave power according to the temperature profile desired, ensuring great reproducibility. The dedicated Milestone Validation Package, along with the 21 CFR part 11 compliance, meet the GMP requirements and provide high standards of operation. The PYRO is compliant with several official methods such as ASTM, USP, SEMI and ISO, preloaded into the terminal.



ultraFAST CRUCIBLES

The PYRO can accommodate crucibles made of metal, quartz, porcelain, etc. However the use of these commonly available crucibles impact the overall ashing time due to the long cooling time in the desiccator. The Milestone ultraFAST crucibles are made of an innovative material that accelerates the ashing process by enhancing the airflow also through the crucible's fibers. The negligible cooling time, only a few seconds from 1000°C to room temperature, further expedites the ashing procedure and removes the cooling time in the desiccator.



MILESTONE HELPING CHEMISTS

Established in 1988, Milestone is headquartered in Italy with its R&D and manufacturing centre in Germany and Switzerland and offices in the United States, China, Japan and Korea. We

operate worldwide through a network of over 100 exclusive distributors, all providing our customers with premium application and service support. Milestone's mission is to help chemists by offering them the most advanced instrumentation for sample preparation and direct mercury analysis in the world. Our industry-leading technology, in combination with fast, responsive service and applications support, allows Milestone to support our goal of giving you the highest return on investment possible.

MILESTONE SOLUTIONS FOR ELEMENTAL ANALYSIS



ETHOS UP

High Performance
Microwave Digestion



ultraWAVE

The Game Changer in
Microwave Digestion



DMA-80 *evo*

Direct Mercury Analyzer



traceCLEAN

Acid Steam
Cleaning



duoPUR/subCLEAN

Acid Purification

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ETHOS X VIDEO



The Milestone PYRO is an advanced microwave muffle furnace suitable for sulfated ashing applications. The Sulfated Ashing configuration ensures to have fast and safe procedures.

→ **see the PYRO Video**

INDUSTRY REPORT PYRO | PHARMACEUTICAL



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H E L P I N G
C H E M I S T S



An alternative and rapid method for measurement of inorganic contaminants in pharmaceutical samples

| INTRODUCTION

The USP chapter <281> describes the methodology for measurement of inorganic contaminants in pharmaceutical samples, where the adopted technique is the Sulfated Ashing.

This method consists in wetting the samples with few drops (1mL) of concentrated sulphuric acid at 98% prior the ashing step at $600\pm 50^{\circ}\text{C}$.

The reason for doing sulphated ash is to prevent volatilization of some important elements to be determined. When phosphorus is absent, barium, calcium, magnesium, sodium and potassium are converted to their sulphates. Tin and zinc are converted to their oxides.

Determination of such elements is important since some minerals are essential to a healthy diet (e.g., calcium, phosphorous, potassium and sodium), whereas others can be toxic (e.g., lead, mercury, cadmium and aluminum).

The conventional method requires to evaporate the H_2SO_4 98% acid using a hotplate or flame under a fume-hood. This operation is very dangerous for the operator since he has to manage very hot crucibles and he could be exposed to the highly toxic sulphuric acid fumes.

These fumes are also very corrosive and can easily damage all instruments and tools placed under the fume hood and the hood itself.

INDUSTRY REPORT PYRO | PHARMACEUTICAL



In addition, by the nature of this procedure it leads to poor reproducibility.

Milestone PYRO is an advanced sulfated ashing system, that allows to run this particular application in only one step, thus assuring operators' safety.

This industry report has the intent to show the performances of PYRO microwave sulphate ashing system on two different pharmaceutical samples.

EXPERIMENTAL INSTRUMENT



Picture 1 - Milestone PYRO Microwave ashing system

The new Milestone PYRO is an advanced microwave muffle furnace, suitable for sulfated ashing application.

It is equipped with a full stainless-steel door and the cavity has a volume in excess of 70 liters, thus allowing the use of a large muffle which in turn enhances the sample throughput.

The PYRO is equipped with two 950 Watt magnetrons for a total of 1900 Watt making it the most powerful microwave muffle furnace system available in the market. The system additionally employs a rotating diffuser that evenly distributes the

microwaves throughout the cavity, assuring a uniform temperature improving the reproducibility.



Picture 2 Milestone PYRO Sulfated Ashing muffle system

PYRO sulfated ashing muffle, enables the analyst to add sulfuric acid to the samples and directly place crucibles into the microwave muffle furnace, without a preliminary charring step. Tedious handling of samples and operator exposure to acid fumes are totally eliminated. Sulfuric acid fumes released from crucibles are continuously exhausted passing through a quartz tube and an air-cooled quartz collection vessel outside the unit.

Fumes are finally contained and neutralized in a dedicated acid scrubber module.

ANALYTICAL PROCEDURE

We have weighed accurately 1 g to 2 g of the substance in four porcelain crucibles (see details in Table 1).

The porcelain crucibles were initially preconditioned at constant weight (other types of crucibles can be used as silica, platinum or quartz).

Samples were then moistened with a small amount (usually 1-2 mL) of sulfuric acid. The samples must be completely wet by sulphuric acid as shown in Picture 3 (second crucible).

INDUSTRY REPORT PYRO | PHARMACEUTICAL



Crucible were placed into PYRO microwave ashing unit and ashed with the microwave program described in Table 2.

Duplicates	Sample	Weight (g)	Type of Crucible	H2SO4 98% Volume
1	Nifuroxazide	2.0057	Porcelain	2 mL
2	Nifuroxazide	2.0247	Porcelain	2 mL
3	Nifuroxazide	2.0012	Porcelain	2 mL
1	Ibuprofen	1.0071	Porcelain	1 mL
2	Ibuprofen	1.0290	Porcelain	1 mL
3	Ibuprofen	1.0020	Porcelain	1 mL

Table 1 – Details on type sample amount, crucibles and volume of conc. H2SO4 98% used for the test.

Step	Time	T1	Power	Scrubber
1 (Charring)	00:10:00	250°C	1000 W	On
2 (Charring)	00:10:00	250°C	1000 W	On
3 (Ashing)	00:20:00	600°C	1800 W	Off
4 (Ashing)	00:30:00	600°C	1800 W	Off

Table 2 – PYRO microwave ashing program

RESULTS AND DISCUSSION



Picture 3- Samples during the steps of sulphate ashing run

The samples were completely ashed, obtaining a white residue of inorganic material. Crucibles were weighted and percentage of residue was then recalculated.

Sample	Crucible (g)	Crucible+Residue (g)	Residue %
Nifuroxazide (1)	53.2356	53.2394	0.189
Nifuroxazide (2)	58.6647	58.6685	0.187
Nifuroxazide (3)	53.0127	53.0164	0.185
Ibuprofen (1)	48.0675	48.0685	0.099
Ibuprofen (2)	44.1191	44.1199	0.078
Ibuprofen (3)	46.7442	46.7451	0.090

Table 3- Obtained results, % of residues

CONCLUSION

Milestone PYRO microwave ashing system offers the big advantage to run all samples in only one step ensuring full of operations and great performance.

Due to its muffle capacity and faster heating, the sample processing throughput is higher than conventional electrical muffle system.

The data shown in this technical note demonstrates that the ashing of samples in a PYRO is reproducible thanks to the great temperature homogeneity across all the muffle. The ability to remove and neutralize the H2SO4 fumes and reach high temperature in short time make the Pyro a great tool to perform sulfated ashing in compliance with the USP <281>.

ABOUT MILESTONE

At Milestone we help chemists by providing the most innovative technology for metals analysis, direct mercury analysis and the application of microwave technology to extraction, ashing and synthesis. Since 1988 Milestone has helped chemists in their work to enhance food, pharmaceutical and consumer product safety, and to improve our world by controlling pollutants in the environment.



OUR MISSION IS TO
MAXIMIZE
YOUR EFFICIENCY

MILESTONE VALIDATION PACKAGES

The compliance with regulations and data traceability are important values in the modern analytical laboratories of pharmaceutical, food and other regulated industries. The qualification of the instruments and of their related methods into these industries often goes through multiple steps in order to ensure that your instrument meets these regulations. Milestone, as a leading technology and solution provider in the microwave sample preparation, provides comprehensive Milestone Validation Packages (MVP) that meet these needs.

ENSURE HIGH QUALITY

GMP COMPLIANT

COST-EFFECTIVE

EXPERTISE AND KNOW-HOW

RESULTS RELIABILITY



MILESTONE
HELPING
CHEMISTS

COMPRHENSIVE SOLUTIONS THAT MEET THE QUALITY ASSURANCE STANDARD

The Milestone Validation Packages include several protocols in order to fulfill regulatory requirements, such as the Good Manufacturing Practice (GMP) guidelines that, along with our superior support and expertise, will be by your side throughout the whole process. Our experts will support you with the right configuration, with a dedicated procedure for your samples, with a demonstration of the system's capabilities and with a complete validation package that includes: DQ (Design Qualification), IQ (Installation Qualification), OQ (Operational Qualification), PQ (Performance Qualification) and the requalification. All the tests carried out by our service engineers are guaranteed by certified test equipment, providing high accuracy and meeting all the regulatory requirements.

Our software is fully compliant with the FDA regulation 21 CFR part 11, with an exhaustive audit trail for complete traceability. The MVP is one of the multiple services offered by Milestone to ensure the highest industry standards in customer support.



MILESTONE VALIDATION PACKAGES AVAILABLE FOR



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High Performance
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