HUMAN HEALTH

ENVIRONMENTAL HEALTH

GREAT RESULTS BEGIN WITH GOOD PREPARATION

Titan MPS™ Microwave Sample Preparation System

A Reference Notebook of Microwave Applications

PerkinElmer®
For the Better

INTRODUCTION

At PerkinElmer, we understand that sample preparation is one of the most critical steps in the analytical process. Often accounting for 60% of your analytical timetable, it has a fundamental impact on laboratory throughput and analytical performance. Any errors within the sample preparation process will undermine the quality of your data at all subsequent stages of your analysis. Great results begin with good preparation, and our Titan MPS™ Microwave Sample Preparation System delivers the clean, clear solutions you need for reliable results.

The Titan MPS system is ideal for difficult environmental and common industrial applications while also providing high performance for a wide range of digestion needs.

This document is intended to provide you with the tools you need to quickly and efficiently develop digestion methods for your unique sample preparation needs.

These applications represent starting points for successful digestions and may require modification for your specific samples.

PerkinElmer Titan MPS Microwave Sample Preparation System.

Answering your needs. Empowering Your Lab.

The process of sample digestion is the chemical reaction of the sample with reagents (typically strong acids) in an effort to remove the sample matrix and leave analyte ions suspended in solution. The sample and reagents are heated in an appropriate container to speed the reaction. In many cases, the energy threshold requirements for reaction cannot be met by the temperatures reached using open-vessel digestion. Closed-vessel microwave-assisted digestion creates a pressurized, high-temperature environment by containing the gases produced during sample heating. The result is faster, more complete digestion than possible at atmospheric pressure.

During digestion, there is always some vessel-to-vessel temperature variation, the result of microwave field flux variabilities, differing microwave absorption rates among the samples, and chemical reactions during digestion of the sample. The PerkinElmer Titan MPS Microwave Sample Preparation System is designed to minimize these effects, and any remaining variation in vessel temperatures should not affect digestion success.

The Titan MPS system accurately measures the temperature of each vessel, ensuring that the temperature of any individual vessel never exceeds the target temperature in the method. This feature allows setting high digestion temperatures while ensuring that the vessel and sample remain under the method limits.

In general, during method development it is beneficial to design the method to use the smallest sample weight that will deliver the detection limits needed during subsequent instrumental analysis. Sample amounts larger than necessary can negatively affect the method by developing excessive pressure and increasing the potential for unwanted temperature variability, as well as shortening consumable lifetimes.

The process of sample digestion commonly results in the evolution of gas from the sample. Samples with high carbon content and metal oxides will evolve the most gas, and for samples of this type, the sample weights should be kept very small until the performance and parameters of the digestion are well known. In addition, most digestion reactions are exothermic in nature; this "self-heating" can rapidly cause the target temperature to be overrun, resulting in the pressure within the vessel to exceed the desired limit. Once the heating, gas evolution, and reaction profile of the sample has been determined, sample weights can be gradually increased, if needed.

Use of Hydrofluoric Acid (HF)

While the use of HF for sample digestion can be very beneficial, and in some cases is the only effective reagent, using HF requires careful consideration and oversight as there are a number of safety and usability impacts. HF will dissolve glass and quartz causing damage to nebulizers, spray chambers, injectors, or any other wetted items in the instrument sample introduction system. Complexation will prevent damage from occurring. HF-resistant sample introduction systems are available for most instruments.

HF is typically used in the digestion of silicates, rare-earth metal alloys, geologic samples, and occasionally for ceramics and polymers that will not respond to more typical digestion acids.

The advantage of HF is that it has a vapor pressure similar to water, so does not cause excessive pressure during digestion. HF also does not typically form molecular species that cause interferences during ICP-MS analysis.

However, HF has several disadvantages. HF is very dangerous in contact with skin or when the vapors are inhaled. A complete understanding of HF hazards is required prior to use. When HF is used for digestion, it must be neutralized or complexed prior to use in any instrument sample introduction system that contains glassware (see "Complexation of HF" in the Supplemental section). Additionally, HF will form compounds with Group I and II metals which can precipitate as a fine cloudy haze. These precipitates can be brought back into solution by complexing with boric acid in a secondary digestion.

For digesting samples with HF, the following should be considered:

1. What are the analytes of interest and would they require HF to be freed from their matrix?
2. Is the sample introduction system on the analytical instrument HF compatible?
3. Does the lab have the capacity to perform two digestions for every sample (one for the sample, a second shorter one for complexing the HF)?
4. Is the infrastructure needed for the safe handling and response to an HF exposure present?

The typical process of using HF for digestion is as follows:

1. There is rarely a need for more than 1-2 mL of HF (49%) per sample. The balance of the reagents (around 8-10 mL) will commonly be nitric acid, hydrochloric acid (HCl) acid or a blend of the two.
2. HF can be added first or last to the sample in the vessel.
3. The normal procedure to re-dissolve the fluoride salts is to add 6 mL saturated boric acid solution for each mL of HF, close the vessel, and heat to 190 °C for 10 minutes.

Digestion Tips

The following tips are general guidelines and outline considerations and responses to common digestion challenges.

1. During digestion, pressure is generated as part of the heating and decomposition of the sample. If vessel pressure during digestion is excessive, reduce the sample weight or the temperature of the method.

2. Sample decomposition during digestion can be strongly exothermic and can occur rapidly. If vessel pressure or temperature rises too quickly, reduce the sample weight and increase the ramp time of the step where this occurs. A longer ramp allows the Titan MPS to more easily discern a strong exothermic event from the normal heating ramp and provides additional time for the system to respond to and control the event.
3. Successful digestion of a sample is a combination of chemistry, heating, and time. Raising the temperature of the sample and reagents increases the rate of reaction and therefore shortens the digestion time. High temperatures are also needed to crest the reaction threshold and begin the digestion process, and enough time is needed for the reaction to run to completion. If a sample is not completely digested, raise the method temperature or increase the digestion time in 10 °C and/or 10 minute increments and re-evaluate. If the hottest digestion step exceeds 60 minutes or an increase of 30 °C is still not successful, the chemistry of the sample should be re-evaluated and a change to the acids should be made.
4. Without the use of HF, samples containing silicon and silicates will typically have this material remaining after digestion. Most geologic samples and a surprising number of plant samples contain silicon and silicate material. Unless silicon or the elements bound to it (commonly Ca or Mg) are analytes of interest, this material is usually not analytically significant. Analytes of interest can be successfully extracted without HF, and the remaining silicon/silicate solids can be filtered or centrifuged with the supernatant liquid used for successful analysis.
5. To dissolve silicon and silicates, HF is included in the acid mixture used for digestion. After the digestion is complete, there may be remaining fluoride compounds that can appear as cloudy white or milky material. A "Complexation of HF" step (located in the Supplemental section) will bring these solids back into solution as well as complex the HF so that the solution can be used with sample introduction systems made of glass.
6. If performing digestion in preparation for arsenic (As) analysis by Hydride-AA or Hydride-ICP-OES, be aware that the use of nitric acid alone may not place the As in an appropriate oxidative state after digestion. The use of HCl or other agents after digestion may not be able to overcome the residual nitric acid concentration in the sample. Digestion using only 2 mL of HNO_3 with the balance (8-10 mL) of deionized water (DI H_2O) may be necessary. Trials using nitric acid with varying ratios of hydrochloric acid should be performed to find the optimal reagent balance for follow-up hydride analysis.
7. The length of the digestion can be critical to its success. Ending a digestion too quickly may result in incomplete digestion. The default limit for digestion time is 75 minutes. This digestion time includes all ramp, hold, and cooling times. If additional time is required, login to the Titan as "admin", enter "Setup" and increase the maximum digestion time (listed in minutes) to a value slightly larger than is expected to be needed.
8. Many metals and metal alloys can be very effectively digested in a solution combining three parts HCl with one part HNO_3 by volume (commonly referred to as Aqua Regia). Note that the reaction of the solution with metals can be vigorous, and the Aqua Regia evolves a lot of gas. The vapor pressure and the evolution of gas from Aqua Regia will raise the digestion pressure significantly. This should be carefully considered when choosing the amount of sample and the target digestion temperature. The inverse solution (three parts HNO_3 to one part HCl by volume) is useful for organic samples.
9. To boost the oxidative strength of the digestion, hydrogen peroxide (H_2O_2) can be added to a nitric acid digestion. Doing so will also convert spent oxides of nitrogen back to nitric acid, thereby boosting the total acidity of the reagents during digestion. This is at the expense of increased digestion pressure and the potential to enhance rapid exothermic sample reactions.
10. Nitric acid is the most commonly used digestion reagent. It does not generate interferences or spectral difficulties on most inorganic analytical instruments and is compatible with nearly all sample introduction systems for atomic absorption (AA), inductively coupled plasma optical emission spectroscopy (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS) instruments. Nitric acid is readily obtainable in high-purity form, reducing baseline contamination.
11. Hydrochloric acid is a strong alternative to nitric acid and is typically used when digesting metal alloys, oxides, or ceramic samples that do not respond well to nitric acid. Hydrochloric acid can potentially cause elemental interferences in ICP-MS analysis. Chloride salts can form during digestion, which may affect analyte recovery. The addition of hydrochloric acid is also recommended when mercury is one of the analytes of interest. Adding 1 mL of concentrated hydrochloric acid (35-37%) to the digestion will complex the mercury and prevent it from volatilizing. Like nitric acid, hydrochloric acid is readily available in high-purity form.
12. Sulfuric acid is a very powerful reagent for use in digesting plastic samples and stubborn organic samples with multiple or cyclic bonds. If a digestion with nitric acid results only in the blackening of the sample or the sample forms a dark paste, the addition of sulfuric acid will usually bring this digestion to completion. As a side benefit, the addition of sulfuric acid also reduces the overall digestion pressure due to its very low vapor pressure. The ability to reduce digestion pressure can be used to aid other digestions that may not require sulfuric acid normally. Sulfuric acid has many potential interferences and can change the viscosity of the sample solution, lowering sample signal and therefore should be considered carefully prior to inclusion in a digestion. Sulfate salts can form during digestion which can affect analyte recovery. Like nitric acid, sulfuric acid is readily available in pure form though it can be more costly.

13. Phosphoric acid is useful in the digestion of many types of geologic or ore samples and also benefits from the ability to dramatically reduce the pressure during digestion. Like sulfuric acid, phosphoric acid has many potential interferences and will affect the viscosity of the final sample solution. Phosphoric acid has extended rinse-out times and can form a residue on ICP-OES and ICP-MS torches, injectors, as well as ICP-MS cones. Phosphoric acid is expensive in its purest form.

Vessel and System Maintenance

To maintain top performance, vessels should be cleaned once per week following the "Vessel Cleaning" method in the Supplemental section.

At a minimum, once per month the vessels should be re-conditioned following the "Vessel Re-Conditioning" method in the Supplemental section.

Over time, the gases generated during digestion can permeate the internal surface layer of the digestion vessels or liners. This is true of all PTFE materials and does not indicate adsorption or permeation of analytes. This gaseous permeation will generally be noticed as a "yellowing" of the vessel/liner or the acquisition of a slightly orange tint to the vessel. As these gases accumulate, they reduce the microwave transparency of the vessel itself and can cause the vessel to be directly heated by the microwave energy applied. This can result in uneven heating of samples during digestion and potentially cause damage to the vessel itself.

Vessels and liners can be de-gassed by placing in a lab oven and baking overnight at 200-220 °C. Optionally, the frequent use of the "Vessel Re-Conditioning" method will help slow the accumulation of gases and may even prevent the need for de-gassing of the vessels or liners.

Vessel Cleaning Between Digestions

During normal digestions, vessels or liners do not require cleaning between samples as the sample has been completely digested; simply rinsing with DI H_2O is enough to prep the vessels for the next digestion. This remains true as long as the concentrations of elements in the sample remain similar and all digestions result in total sample digestion. If analyzing samples where the concentration of the elements of interest is expected to differ by more than two orders of magnitude, it is advisable to run the "Vessel Cleaning" method in the Supplemental section.

If the previous digestion was not successful and material remains in the vessel, cleaning will be required to prepare the vessel for the next sample. There are typically two reasons for material to remain after digestion. The first is that the peak temperature and length of time at the peak temperature were inadequate to completely digest the sample. If additional time and temperature are unable to digest the sample, then it is likely that the acids used for digestion must be changed. Please see the Digestion Tips section for insight on how to attack the residue and enable effective cleaning of the vessel.

Cautions

Microwave ovens are not spark-proof devices. As such, caution must be taken when preparing samples for microwave digestion that are fine powders or that may be prone to spontaneous reaction or ignition. If these samples remain on the sides of the vessel, inside the vessel, or float on top of the reagents as a mass, there is the possibility of direct microwave interaction and generation of a spark. This spark initiates ignition of the remaining sample which can damage the vessel and/or the Titan MPS systems itself. While the Titan MPS is designed to capture this energy and reduce danger to personnel and the lab environment, preventing an event of this nature is important.

When handling fine powder or reactive samples, it is important that the sample be completely rinsed to the bottom of the vessel and either well mixed with the reagents or completely submerged by the reagents. Placing the sample into the vessel first and then using the reagents to wash down the walls of the vessel during addition is a common solution. Optionally, a small amount of DI H_2O can be used to rinse the sample from the vessel walls and, normally, will not affect the digestion. Additionally, PerkinElmer offers PTFE weigh boats that, after weighing the sample, can be placed into the vessel and remain there during digestion.

When trying to break up floating sample and swirling the vessel is not effective, stirring of the sample into the reagents can be done using a long PTFE stir stick which is then rinsed off with a small amount of DI H_2O.

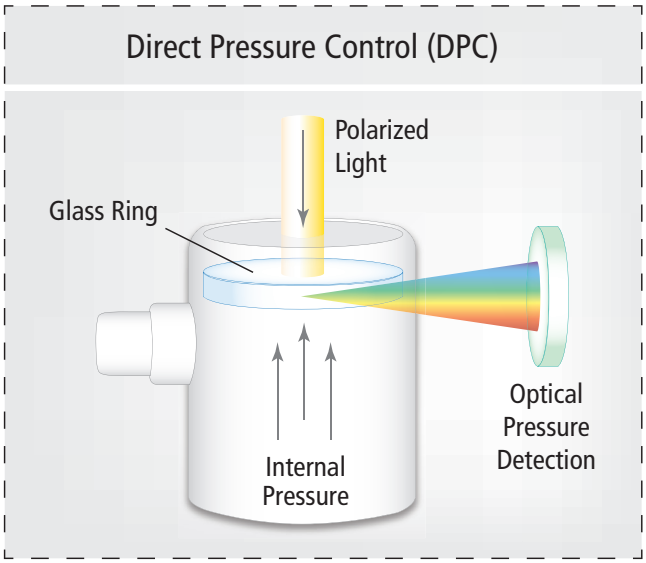
When heating samples in the microwave, any microwave energy not absorbed by the samples or reagent will either be reflected back to the magnetrons, resulting in less efficient operation, or will be absorbed by a part of the microwave system not intended to be heated. For this reason, each published microwave method indicates that the maximum power of the first step should be reduced when using fewer than the maximum number of vessels.

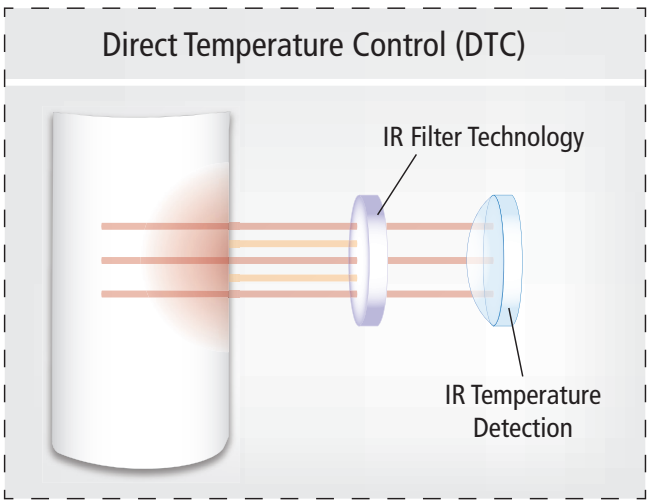
Additionally, very fast temperature ramps should be avoided as a great deal of excess microwave energy will be generated in an effort to rapidly heat the sample. In all cases, a more moderate temperature ramp is more efficient and will generate superior digestion results.

To ensure safe, accurate and reproducible digestions, the Titan MPS employs sophisticated Direct Pressure Control™ (DPC)™ and Direct Temperature Control™ (DTC)™ technologies. With these technologies, the Titan MPS is able to deliver outstanding reaction and digestion control.

By employing sophisticated electronics, DTC eliminates the external temperature interference emitted by the surface of the digestion vessel, allowing the accurate measurement of the internal sample temperature within each digestion vessel.

DPC measures sample pressure as a shift in the polarization of a beam of light sent through a prism and glass ring in the vessel cap. This polarization shift is a result of the internal vessel pressure applied directly to the glass ring. DPC and DTC provide sensing with no special assembly required, no fragile or expensive probes to insert and no connections needed.





PERSONAL CARE PRODUCT DIGESTION METHODS

Body Lotion

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%) 10 mL

SAMPLE WEIGHT

0.250 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	30	3	30	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%) 10 mL

SAMPLE WEIGHT

0.500 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	60	3	20	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Face Cream

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%) 10 mL

SAMPLE WEIGHT

0.250 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	30	3	30	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%) 10 mL

SAMPLE WEIGHT

0.500 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	60	3	20	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Lipstick

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	8 mL
HF (49%)	2 mL

SAMPLE WEIGHT

0.250 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	30	3	30	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	10 mL
HF (49%)	2 mL

SAMPLE WEIGHT

0.500 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	60	3	20	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

When using HF, if the sample is to be analyzed in an instrument containing glassware, the "Complexation of HF" secondary digestion (located in the Supplemental section) must be performed.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Shampoo

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%) 10 mL

SAMPLE WEIGHT

0.250 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	30	3	30	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%) 10 mL

SAMPLE WEIGHT

0.500 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	60	3	20	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Soap (Bar, Liquid)

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	8 mL
HF (49%)	2 mL

SAMPLE WEIGHT

0.250 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	30	3	30	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	10 mL
HF (49%)	2 mL

SAMPLE WEIGHT

0.500 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	60	3	20	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

When using HF, if the sample is to be analyzed in an instrument containing glassware, the "Complexation of HF" secondary digestion (located in the Supplemental section) must be performed.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Sunscreen

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	6 mL
HCl (35-37%)	3 mL
HF (49%)	1 mL

SAMPLE WEIGHT

0.150 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	35	5	25	100
3	50	35	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	6 mL
HCl (35-37%)	3 mL
HF (49%)	1 mL

SAMPLE WEIGHT

0.350 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	220	70	5	15	100
3	50	70	1	40	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

When using HF, if the sample is to be analyzed in an instrument containing glassware, the "Complexation of HF" secondary digestion (located in the Supplemental section) must be performed.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Talcum Powder

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	8 mL
HF (49%)	3 mL

SAMPLE WEIGHT

0.100 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	200	35	5	35	100
3	50	35	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	10 mL
HF (49%)	3 mL

SAMPLE WEIGHT

0.300 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	70	5	25	100
3	50	70	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

When using HF, if the sample is to be analyzed in an instrument containing glassware, the "Complexation of HF" secondary digestion (located in the Supplemental section) must be performed.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

Toothpaste

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	8 mL
HF (49%)	2 mL

SAMPLE WEIGHT

0.250 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	5	90
2	190	30	3	30	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	10 mL
HF (49%)	2 mL

SAMPLE WEIGHT

0.500 g

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	5	80
2	210	60	3	20	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Accurately weigh the sample and transfer into the digestion vessel. Slowly add the reagents to the digestion vessel, rinsing the sample to the bottom of the vessel. Gently swirl the mixture and wait approximately 10 minutes before putting the seal in place and closing the vessel.

To avoid foaming and sample loss when opening the vessels after digestion, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

When using HF, if the sample is to be analyzed in an instrument containing glassware, the "Complexation of HF" secondary digestion (located in the Supplemental section) must be performed.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40% regardless of the number of vessels digested.

SUPPLEMENTAL

Complexation of HF

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

Boric Acid 1 g per 1 mL of HF

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	1	90
2	190	30	1	15	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

Boric Acid 1 g per 1 mL of HF

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	1	80
2	190	60	1	15	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Add approximately 1 gram of boric acid to the digestion vessel for every 1 mL of HF used during digestion.

To avoid foaming when opening the vessels after complexation, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40%, regardless of the number of vessels digested.

New Vessel Conditioning

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (1%) 25 mL

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	2	90
2	200	30	2	10	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (1%) 25 mL

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	2	80
2	210	60	2	10	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Add the 25 mL of a 1% nitric acid solution to the digestion vessel.

Wait until the vessels have cooled enough to be warm to the touch and then vent the residual pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40%, regardless of the number of vessels digested.

Vessel Cleaning

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	10 mL
H_2O_2 (30%)	2 mL (optional)

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	2	90
2	200	30	2	15	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (70%)	10 mL
H_2O_2 (30%)	2 mL (optional)

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	2	80
2	210	60	2	15	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Slowly add the reagents to the digestion vessel.

To avoid foaming when opening the vessels after cleaning, wait until the vessels have cooled enough to be warm to the touch and then vent the residual digestion pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

The addition of H_2O_2 will increase the cleaning potential of the solution but can be left out, if desired.

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40%, regardless of the number of vessels digested.

Vessel Re-Conditioning

EQUIPMENT - STANDARD VESSEL

PerkinElmer Titan MPS System

Standard 75 mL Digestion Vessel

REAGENTS

HNO_3 (1%)	25 mL
H_2O_2 (30%)	3 mL

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	30	5	2	100
2	200	30	2	15	100
3	50	30	1	15	0
4	-	-	-	-	-
5	-	-	-	-	-

EQUIPMENT - HIGH-PRESSURE VESSEL

PerkinElmer Titan MPS System

High-Pressure 100 mL Digestion Vessel

REAGENTS

HNO_3 (1%)	25 mL
H_2O_2 (30%)	3 mL

SAMPLE WEIGHT

-

TEMPERATURE PROGRAM

Step	Target Temp [°C]	Pressure Limit [bar]	Ramp Time [min]	Hold Time [min]	Power [%]
1	160	60	5	2	100
2	200	60	2	15	100
3	50	60	1	30	0
4	-	-	-	-	-
5	-	-	-	-	-

PROCEDURE

Add the 25 mL of a 1% nitric acid solution and 3 mL of H_2O_2 to the digestion vessel.

After heating is complete, wait until the vessels have cooled enough to be warm to the touch and then vent the residual pressure very slowly.

Always work in a fume hood wearing hand, eye and body protection since a large amount of gas can be produced during the digestion process.

NOTES

This application is designed for the maximum number of vessels (sixteen 75 mL vessels or eight 100 mL vessels). Decrease the power at the first step by 5% per vessel when using fewer than the maximum number of vessels. Minimum power is 40%, regardless of the number of vessels digested. the number of vessels digested.

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