FIELD APPLICATION REPORT

ICP-Optical Emission Spectrometry
and ICP-Mass Spectrometry

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Multiwave 3000 Sample preparation system. System will do digestion, evaporation and solvent extraction.

Microwave Digestion – EPA Method 3052 on the Multiwave 3000

Introduction

Microwave sample preparation provides an efficient and clean sample preparation for multi-element analytical techniques such as ICP-OES and ICP-MS. As Microwave assisted digestion has evolved, so have the methodologies. EPA method 3052 is designed for the "total" analysis in a variety of matrices including soil, sediments, sludge, oils, biological and botanical materials. This method is the most versatile and has been well proven. It allows variations in reagents and methodology, making it ideal for a variety of matrices and elements. The variability of this method also requires a basic understanding of digestion chemistry.

Rotor systems for the Multiwave 3000. Rotors are designed for specific functions such as digestion, drying, or solvent extraction.

Benefits of Microwave Assisted Digestion

- Cleanliness of preparation environment
- Reproducible digestion,
- Improved QA/QC
- Reduces skill level as a factor
- Greatly reduces preparation time

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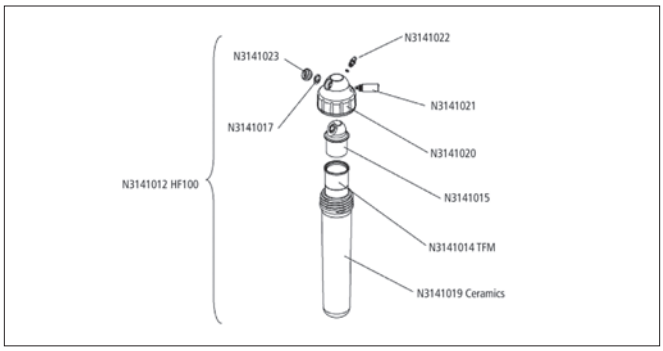
Instrument Configuration

The Multiwave™ 3000 is a completely modular system which can be configured to perform many sample preparation functions. To perform Method 3052 digestion the system should be equipped as follows;

- Multiwave 3000 Oven (N314-1001)
- High Pressure 16 position rotor (N314-1003)
- Pressure/Temp Probe (N314-1005)
- IR Temp Sensor (N314-1006) *Optional*

While larger capacity rotors are available, they should only be used with sample matrices which are known to digest with low pressures. (< 20 bar)

Spare parts for the sample vessels should be kept on hand to replace old or worn parts and to repair vessels which may have ruptured during a digestion.



Pressure Vessel HF100 MW3000.
Complete reaction vessel for versatile applications. The 100 mL liner is made of PTFE-TFM (N3141014) and supported by the Vessel Jacket H100 Ceramics (N3141019). A PTFE-TFM Seal for Pressure Vessel M&H (N3141015) with integrated release valve and Safety Disk M&H (N3141017) fixed in a PEK Screw Cap (N3141020) closes the vessel.

Reagent Selection

Method 3052 allows the analyst to select specific reagents for specific matrices and analytes of interest. Typically 9 mL of nitric acid are placed in the reaction vessel with the sample, and a combination of other reagents such as hydrochloric, hydrofluoric, or hydrogen peroxide may be added based on matrix and particular analytes.

Hydrofluoric and hydrochloric acids are both used as complexation reagents especially in the presence of silicates and precious metals respectively. The use of hydrogen peroxide enhances the oxidation properties of nitric acid especially in the digestion of organics.

The following table suggests reagents and their ratios using method 3052.

		Reagent and Volume (mL)			
Matrix	**Reference Material**	**HNO_3**	**HF**	**HCl**	**H_2O_2**
Soil	NIST SRM 2711 Montana Soil	9	3	2	1
Sediment	NIST SRM 2704 Buffalo River Sediment	9	3	2	1
Biological	NIST SRM 1577a Bovine liver	9	0	1	2
Botanical	NIST SRM 1547 Peach leaves	9	0.5	0.5	1
Botanical	NIST SRN 1567a Wheat flour	9	0	0.5	2
Waste Oil	NIST SRM 1054a Wear-metals in lubricating oil	9	0.5	0.5	2

The use of HCl in the digestion should be used anytime that Ag or Sb are analytes of interest. Studies have shown very poor recoveries without the use of HCl.

Peroxide may be used in all digestions, however be aware of the increased reactivity with organic materials.

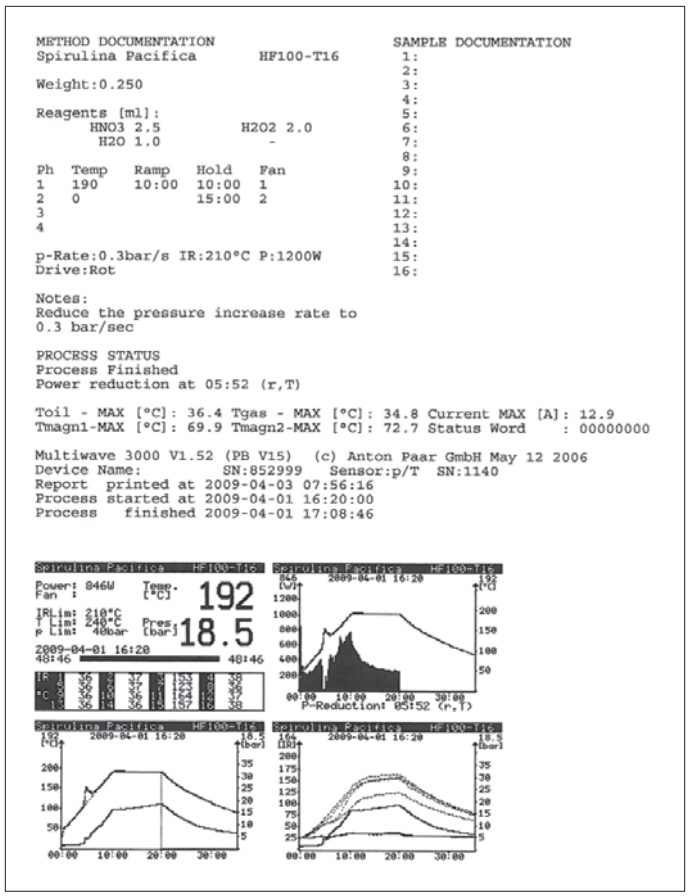
Method Procedure

A 0.25 to 1.0 g sample is weighed out in the reaction vessel. 9 mL of nitric acid are then added to each vessel. Then depending on the matrix the proper amount of hydrofluoric and/or hydrochloric acids are then added. Finally the hydrogen peroxide is added. The vessel is allowed to react for approximately one minute prior to sealing the vessels.

Vessels should then be placed in the rotor and placed in the microwave. The vessels should then be heated to at least 180 °C over 5.5 minutes and then held at 180 °C for at least 9.5 minutes. (The heating profile may be modified for reactive matrices). An example of the Multiwave program is shown below.

Analytical Results (ppb)

Analyte	**Expected Range**	**Spirulina-A**	**Spirulina-B**	**Spirulina Avg.**
As	150 to 300	269	274	272
Cd	10 to 75	16	15	15.5
Hg	10 to 75	16	14	15
Pb	50 to 150	77	74	75.5



Example of digestion and analytical results. Sample was a high organic matrix with volatile elements.

Results

The goal of this method is total sample decomposition and with judicious choice of acid combinations this is achievable for most matrices. Selection of reagents which give the highest recoveries for the target analytes is considered the optimum method condition.

Digests and alternative procedures produced by the method are suitable for analysis by flame atomic absorption spectrometry (FLAA), cold vapor atomic absorption spectrometry (CVAA), graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma atomic emission spectrometry (ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS) and other analytical elemental analysis techniques where applicable.

• Aluminum • Cadmium • Iron • Molybdenum • Sodium • Antimony • Calcium • Lead • Nickel • Strontium • Arsenic • Chromium • Magnesium • Potassium • Thallium • Boron • Cobalt • Manganese • Selenium • Vanadium • Barium • Copper • Mercury • Silver • Zinc • Beryllium

Other elements and matrices may be analyzed by this method if performance is demonstrated for the analyte of interest, in the matrices of interest, at the concentration levels of interest.

Conclusion

The Multiwave 3000 is designed for both high productivity and excellent digestion performance without compromising safety. Up to 16 PTFE-TFM vessels per run cope with a wide selection of samples, which require reaction conditions of up to 40 bar and 240 °C. An immersing pressure/temperature sensor in one reference vessel provides accurate reaction control. Tool-free and easy vessel handling allows for safe and convenient routine work.

The Multiwave 3000 is one of the most versatile sample preparation platforms available. It can easily perform the EPA method 3052 digestion procedure, bringing to the lab all of the benefits associated with microwave assisted closed vessel digestion. Any laboratory doing trace metals analysis would instantly benefit from the application of this digestion procedure. Samples would see less contamination, digestions would become more reproducible, and QC would be much tighter.

References

1. H.M. Kingston, P.J. Walter, "The Art and Science of Microwave Sample Preparations for Trace and Ultratrace Elemental Analysis" In Inductively Coupled Mass Spectrometry, A. Montaser, Wiley-VCH, 1997.

2. Environmental Protection Agency, Method 3052, SW-846, 1996.

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