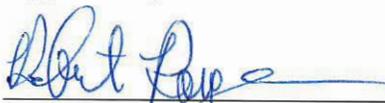


Status: Final

Approved by:



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Technical SOP for Operation of Milestone UltraWAVE Microwave Digestion Unit

1. SCOPE

This SOP describes the procedure to prepare samples of various matrices (liquid, soil, fabrics, foams, furnishing materials, plastics, glass, waste materials and their mixtures) via microwave assisted acidic digestion in the Milestone UltraWAVE for appropriate metal analysis.

This procedure is recommended for use only by laboratory technicians working directly under chemists experienced in sample preparation for inorganic analysis or for use by chemists trained in sample preparation.

Analysis performed for waste classification must be done in a California certified hazardous waste testing laboratory.

2. RESPONSIBILITIES

ECL Staff: Responsible for preparing samples via microwave digestion to be analyzed for metals, and performing routine maintenance on the digestion unit.

3. PROCEDURE

3.1. Batch QC Requirements

Refer to "Microwave Assisted Digestion", DCN:03.3051.00 for applicable batch QC requirements.

3.2. Instrument QC Requirements

Refer to the appropriate analytical method for additional QC requirements.

3.3. Sample Preparation

Refer to "Microwave Assisted Digestion", DCN:03.3051.00 for sample preparation procedures.

3.4. Procedure

3.4.1 Turn ON microwave, use the switch on lower front side of the instrument.

- Turn on nitrogen by turning the valve of the regulator clockwise until it is fully open.
- Turn on the chiller, using the power switch on the front panel.
- Log into the UltraWAVE using the ID “Administrator” and “123456” password on Terminal, hit “OK”.
- If a maintenance message appears, acknowledge by hitting “OK” and alerting the QAO.
- Press “UltraWAVE” in upper-right corner that will open “Method” tab automatically.
- Press “folder & arrow” button in lower-left and pick one of the methods.
- Press “folder & arrow” button again to confirm your selection.
- Method parameters such as N₂ pressure, vessel cooling, ramp, holding temp and time are now displayed in sub-tabs. Please don’t overwrite methods. Use “Save As” with a different name if change of any parameter is necessary.

Digestion times and temperatures as recommended; current parameters are as follows:

- Method “**3015A**” (for liquid samples): Ramp to 170° C for 10 minutes; hold at 170° C for 10 minutes, N₂ pressure at 35 bars;
- Method “**3051A**” (for soils, shredder waste, etc.): Ramp to 175° C for 5.5 minutes; hold at 175° C for 4.5 minutes, N₂ pressure at 35 bars;
- Method “**3051A 1h ramp**” (for polymers, foams, fabrics, etc.): Ramp to 175° C for 60 minutes; hold at 175° C for 30 minutes; cool down for at least 5 minutes (normally takes about half an hour until samples reach ambient temperature & pressure), N₂ pressure at 35 bars;

If the samples react when acids are added as described in section 3.4.2, then adjust temperature and time: Ramp to 150°C for 60 minutes; hold at 150°C for 4.5 minutes, N₂ pressure at 35 bars;

- Method “**3052**” (for glass and other siliceous materials): Ramp to 180° C for 5.5 minutes; hold at 180° C for 9.5 minutes, N₂ pressure at 40 bars.

3.4.2 Weigh a portion of homogenized sample ($\leq 150 \mu\text{m}$ fraction for glass, $\leq 2\text{mm}$ for other solid/ground samples for TTLC analysis) using a spatula or aliquot well-shaken liquid sample such as TCLP leachate via pipette and transfer into a vessel quantitatively. Use Milestone’s vessel stand for convenience, it fits both quartz and Teflon vessel types. Add spikes, DI and all acids needed for digestion, cap the vessel and place inside 15-position carousel. Recommended parameters are as follows:

- Sample size: 0.2-0.25 grams for solids, 5mL for liquids
- Acids used:
 - 1:1 ratio of conc. HNO₃ & conc. HF (2.5+2.5 mL) for glass.
 - 9:5 ratio of conc. HNO₃ & conc. HCl (4.5+2.5 mL) for soils, aqueous samples. If silver and antimony are not the analytes of interest, then use 5 mL of conc. HNO₃ instead of HNO₃/HCl mix.
 - 8:1:1 ratio of conc. HNO₃ & conc. HCl & 35% H₂O₂ (4 + 0.5 + 0.5 mL) for reactive materials such as polymers, foams, fabrics, etc., try to use small pieces of sample. Let capped vials with samples, spike and reagents settle for up to one hour in a fume hood. Once samples have settled, add extra 100-300 µL of DI in each vessel, which helps to dissipate extra heat and reduces reactivity inside the vessel(s) at high temperatures.

3.4.3 Add 5mL ACS grade conc. HNO₃ to 130mL DI water, as an electrolyte solution, to the main teflon vessel, insert an o-ring at the top bezel and place it inside the steel reaction chamber. Lift up the steel knob to allow rotation of chamber lid with temperature probe.

- Assemble PTFE lid: the big part goes with o-rings side up and the little part is holding the whole lid in place and works as a screw, be careful with metal/plastic threads make sure all o-rings are not worn-out as chamber will not reach desired 35-40 bars N₂ and the digestion program may not start.
- Switch to “System” tab on Terminal and check if any interlocks are active, they’ll be shown in red.
- Load the carousel inside the chamber, align the notch with the temperature probe, make sure carousel is well-centered.
- Press “arrow down” button on the left in case if everything shown in green light (i.e. no interlocks) on “System” tab, this will lower the carousel down to chamber opening.
- Double-check if carousel is well-centered and press same button again, this will close the chamber completely.
- Hand-tighten the clamp and check if mechanical lock in the center of the clamp is activated/elevated.
- Hit “Start” button now to begin the digestion.
- Monitor digestion process in a “Run” tab, sudden peaks on pressure or temperature curves might indicate combustion inside the vessel(s).

3.4.4 Allow samples to cool in digestion vessels, make sure temperature is 25 ± 2°C and pressure is at 0 bars before opening microwave, main vessel temperature and pressure are displayed on Terminal’s “System” tab.

- Make sure that mechanical lock in the center of the clamp is deactivated, untighten the clamp.
- Press “arrow up” button on the left in case if everything shown in green light (i.e. no interlocks) on “System” tab, this will elevate the carousel up to chamber opening.
- Press “arrow up” button again, this **will elevate the carousel up** to the top.
- Transfer the whole carousel with 15 vessels to the fume hood (use **a plastic lid** at the bottom since half of carousel was submerged in diluted nitric/ electrolyte solution).
- **Wipe each vessel with paper towels and then transfer contents quantitatively to 50 mL digestion vials, rinse vessels and caps at least twice; use DI water for rinsing. Make up to the final volume.**
- **Samples ready for ICP-AES or FIMS analysis.**

Note: glass digests (EPA's 3052) shouldn't be brought to final volume. Set hot block at $95 \pm 5^{\circ}\text{C}$ and transfer digests to hot block, see “Microwave Assisted Digestion of Silicate Samples”, DCN:03.3052.00.

- Refer to tables 1 and 2 for regular maintenance procedures.
- Remove the main PTFE chamber and place it in a fume hood.
- Unscrew carousel holder and disassemble PTFE lid, place them in a fume hood.
- Monitor the condition of several o-rings (3x small, 1x medium and 1x large) which prevent microwave chamber from leaking, replace worn **out** o-rings when needed.
- Dry and clean the metallic components of the microwave with ethanol and apply some silicone oil #SP0006A using Kimwipes®.
- Place all vessel caps into plastic container with 5% nitric solution for cleanup, rinse with DI and air-dry before the next digestion. Please refer to “Operating Manual”, Milestone Ultrawave ECR Microwave Oven regarding vessels cleanup and disposal.
- Extra cleaning of the caps, big reaction vessel with its lid, o-rings, carousel/carousel holder and exhaust/purge tubing connectors must be performed if burning of the sample(s) happened during digestion batch. Use acetone or methanol to remove carbon and/or oily residuals.
- Cover the chamber with a plastic lid to keep the dust out for longer periods of time between the batches.
- **Turn OFF microwave, use the switch on lower front side of the instrument.**
- Make sure to turn off chiller, close nitrogen cylinder and turn off the vents (in Terminal's “System” tab) before leaving the lab.

4. DEFINITIONS

Holding Time – The maximum amount of time that a sample may be stored before preparation or analysis

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectrometry

FIMS – Flow-injection Mercury System

Preparation Batch – One (1) to twenty (20) environmental samples of the same quality system matrix that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. The maximum time between the start of processing of the first and last sample in the batch is twenty-four (24) hours.

Sample Batch – A group of samples undergoing extraction at the same time and with the same equipment; the batch size is 20 or fewer samples.

TTLC – Total Threshold Limit Concentration (CA EPA)

TCLP – Toxicity Characteristic Leaching Procedure (US EPA)

PTFE – Polytetrafluoroethylene (Teflon®)

5. REFERENCES

- 5.1. “Microwave Assisted Digestion”, State of California Department of Toxic Substances Control, Environmental Chemistry Laboratory, DCN:03.3051.00
- 5.2. California Code of Regulations, Title 22, Division 4.5, Chapter 11, Article 3, 66261.24(a)(2)(A) Table II – List of Inorganic Persistent and Bioaccumulative Toxic Substances and Their Soluble Threshold Limit Concentration (STLC) and Total Threshold Limit Concentration (TTLC) Values
- 5.3. SW-846, US EPA, HW Test Methods:
 - (1) 3015A, Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
 - (2) 3051A, Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
 - (3) 3052, Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
 - (4) 6010C, Analysis of Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy
 - (5) 7470A, Mercury in Liquid Wastes (Manual Cold-Vapor Technique)
- 5.4. Operating Manual, Milestone Ultrawave ECR Microwave Oven
- 5.5. “Microwave Assisted Digestion of Silicate Samples”, State of California Department of Toxic Substances Control, Environmental Chemistry Laboratory, DCN:03.3052.00

6. TABLES

UltraWAVE Cleaning procedure

This is the cleaning guideline, more information can be found in the UltraWAVE user manual (**Service and maintenance chapter**).

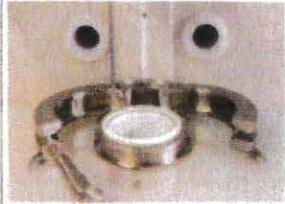
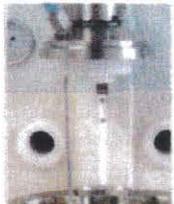
Frequency	Part/Component	Activity	Picture
Every run	PTFE Cover	Clean and dry the PTFE cover	
Every run	Metal clamps	Clean and dry the metal clamps	
Every run	Metal pressure vessel	Clean and dry the pressure vessel.	
Every run	Thermowell	Check the conditions of the thermowell, no cracks/bubbles must be present	
Every run	O-ring on the TFM vessel	Check the conditions of the o-ring. In case some drops of condense are found on the stainless steel reactor, change the o-ring.	
Every day	O-ring on the cover	Check the conditions of the o-ring on the PTFE cover	

Table 1: Regular maintenance of UltraWAVE digestion unit

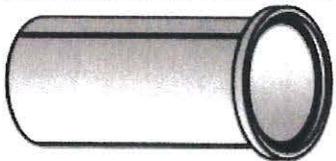
Every day or every 3 runs	Metal cover	Dry, clean with Ethanol and place some silicon grease spay on the metal cover	
Every day or every 3 runs	All metal parts (cover, clamps and reactor).	Dry and clean with Ethanol all metal parts. Clean the bottom of the Metal reactor with a cotton buds	 
Every day or every 3 runs	All metal parts (cover, clamps and reactor)	Spray a bit of silicon grease on paper and pass it on all metal parts	
Weekly	O-ring on the TFM cover	Place some silicon grease	Place a thin layer of silicon grease on the o-ring of the TFM cover
Monthly	Pressure test	Run a pressure test	See, its complete description in the user manual
Daily before unit switch off	Flushing of tubes	Use the Nitrogen gas	See, its complete description in the user manual
After long operation breaks	All internal parts	Run a program with water	See, its complete description in the user manual
Daily, before switch off the unit	Flushing of tubes	Use the Nitrogen gas. See chapter 7.1.3 of User Manual	
Everyday	TFM disc	Check conditions of TFM disc and replace them if necessary.	
Every months	TFM Vessel	<i>Cleaning procedure to remove NOx</i> Put the big PTFE vessel in an electrical oven at 140°C for about 4 hours	
Every year or after 600 runs	UltraWAVE	Service check	Contact local service engineer for the complete checking of the unit

Table 2: Regular maintenance of UltraWAVE digestion unit, continued

7. REVIEW

Signatures

Date
