



PRECIOUS METAL DISSOLUTION CHALLENGES AND MICROWAVE DIGESTION



MILESTONE
HELPING
CHEMISTS

March 2, 2023

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ABOUT MILESTONE

- Established in 1988
- Over 25,000 instrument installations worldwide ranging from Fortune 500, Academia, Government Agencies, Independent Contact Labs, Manufacturing
- Global Footprint
 - US Headquarters: Shelton, CT
 - Full sales and service support
 - Applications lab and technical support
 - Parts inventory
 - European Headquarters: Bergamo, Italy
 - R&D/Manufacturing: facilities in Germany and Switzerland



Milestone Inc. USA: Shelton, CT



Milestone srl: Bergamo, IT

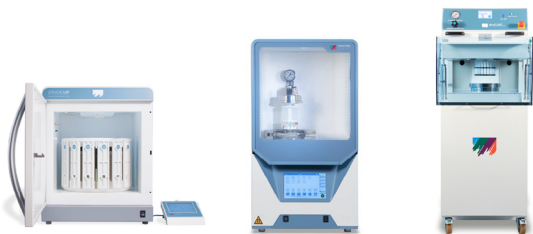


YOUR PARTNER IN TRACE METALS ANALYSIS



MICROWAVE DIGESTION

Effective sample preparation is the first step for successful AAS/ ICP-OES/ ICP-MS analysis.



CLEAN CHEMISTRY

Trace metals analysis require dedicated tools to control and reduce the blanks.



MERCURY ANALYSIS

Direct mercury analysis allow to analyze any matrices in few minutes without any sample preparation.



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

SAMPLE PREP CHALLENGES WITH PGE/CATALYST SAMPLES

- Sample prep goal:
 - Get all elements of interest into solution for analysis via ICP-OES/ICP-MS
- Platinum Group Metals
 - Challenge:
 - Requires high temperature/long time to digest
 - Typical reagents: HCl / Aqua Regia / H₂O₂
- Catalysts
 - Challenge:
 - Can require time at temperature (>2 hours) to achieve complete digestion
 - Typical reagents: HCl / HNO₃ / HF / H₃PO₄ / H₂SO₄
- Difficult to digest with a traditional rotor-based microwave digestion system
- Typical sample prep techniques used:
 - Hot block / open-vessel digestion, Fire Assay, Fusion



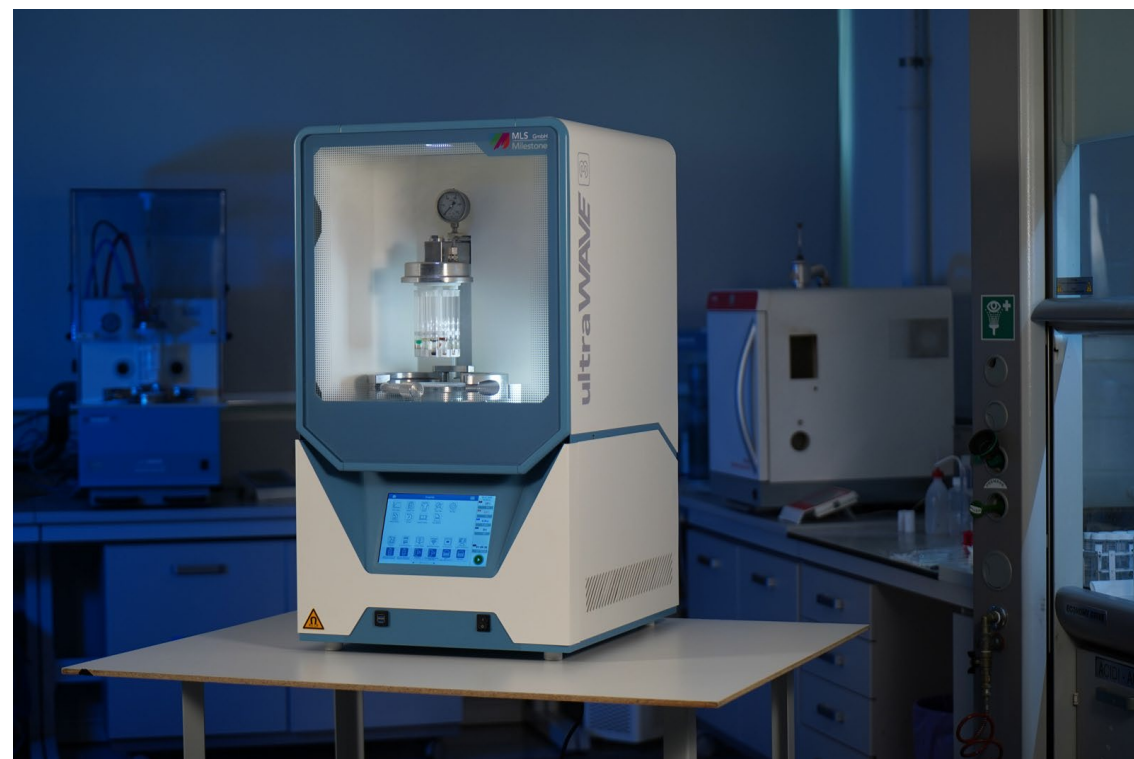
HOT PLATE/HOT BLOCK CHALLENGES

- High acid consumption
 - Acid impurities become more concentrated over time
 - Increased analytical blank
- Susceptible to environmental contamination
- Sample cross-contamination possible
- Limited temperature + pressure capability
 - Limited by boiling points of acids used at atmospheric pressure
 - Long digestion times
 - Incomplete digestions
- Requires 'babysitting'
 - Continuous addition of acids
- Loss of volatile elements



ADVANTAGES OF CLOSED-VESSEL DIGESTION

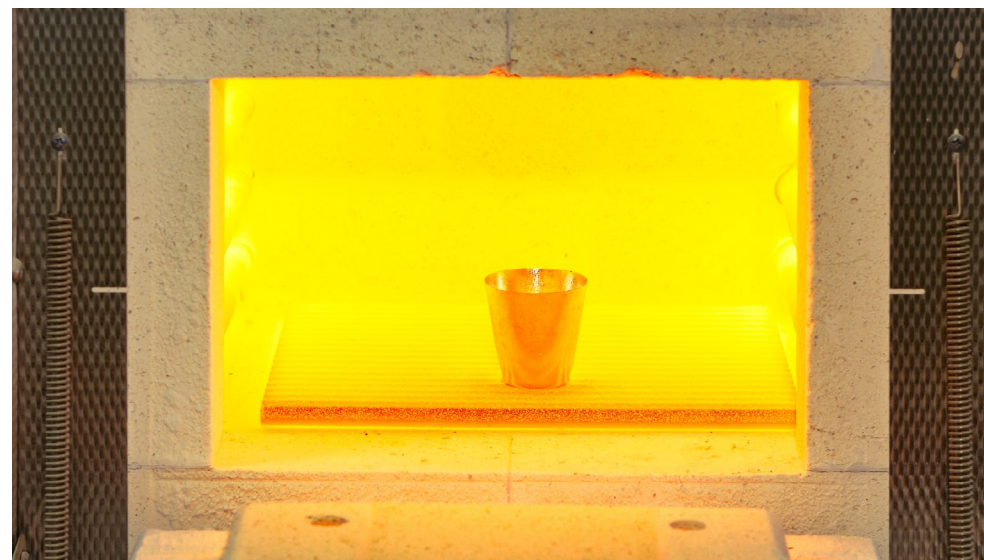
- Superior digestion quality
 - Can work at high temperature and pressure, thus enhancing digestion quality and reducing time required to digest sample
- Speed/productivity
 - Allows for fast digestion of multiple samples simultaneously
- Enables lower detection limits
 - Closed to the environment
 - Less acid usage
 - No sample cross-contamination



CHALLENGES OF FIRE ASSAY/FUSION

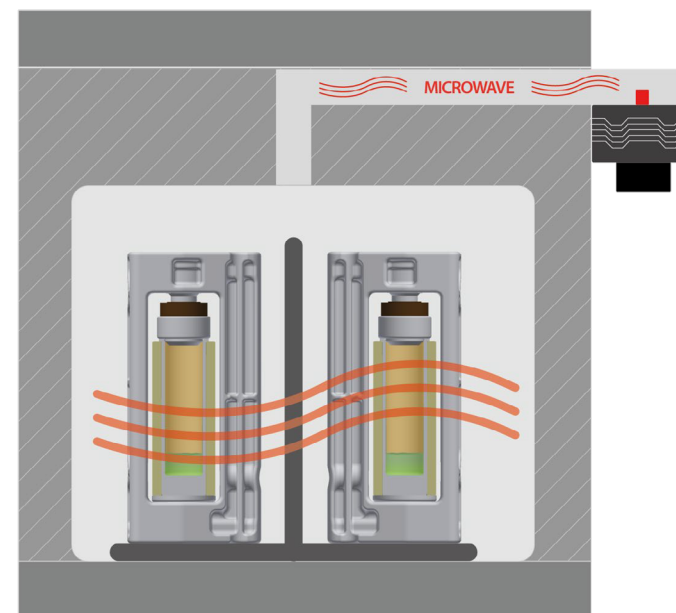
Open system that uses flux powders in a muffle furnace

- Time consuming
- Handling
 - Expensive Pt labware, funnels, filters, etc.
- Strong chance of contamination
- High blanks
- Not ideal for trace element quantitation
- High energy consumption due to the use of high-temperature muffle furnaces



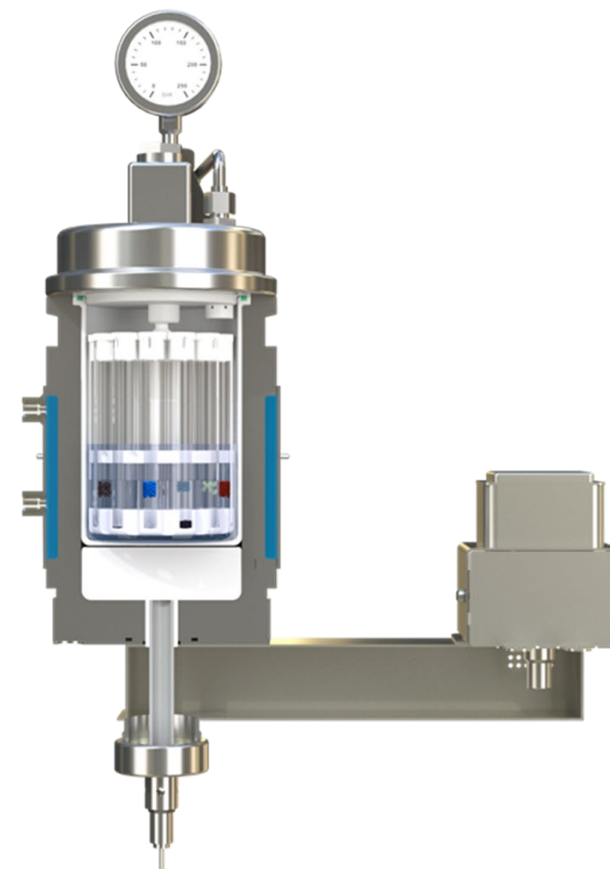
CONSIDERATIONS WITH USE OF ROTOR-BASED SYSTEMS

- Polymer based vessels, resulting in pressure and temperature limitations
 - Typically limited to 30 – 60 minutes at high temperature
- Each vessel at its own temperature and pressure
- Requires batching of similar matrices and chemistries
- Vessel handling requires multiple steps
- Vessel liners can require intensive cleaning depending on analyte levels

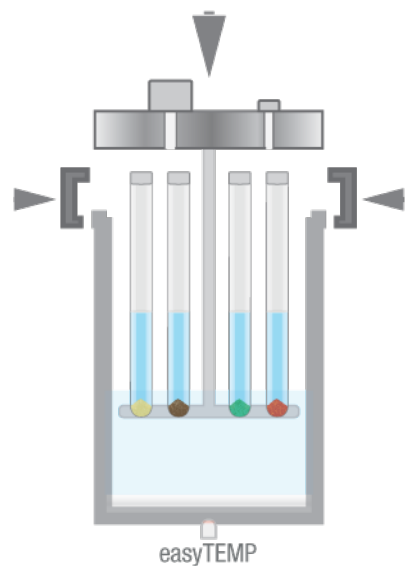


INTRODUCING THE SINGLE REACTION CHAMBER (SRC)

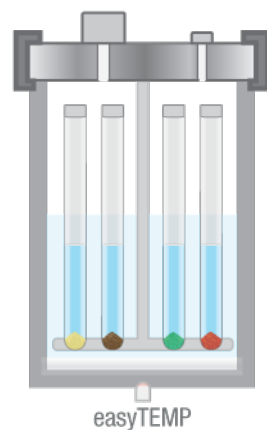
- SRC is a newer approach to microwave closed-vessel digestion
 - Developed and patented by Milestone in 2006 (ultraCLAVE)
 - ultraWAVE introduced in 2011
 - ultraWAVE 3 introduced in 2023
- Overcomes most limitations of rotor-based systems
- Rugged 1 L stainless-steel chamber
 - Fully protected by a PTFE liner and cover
 - Water-cooled to avoid overheating and acid diffusion
- Rack with vials sit in a water base load
 - Homogenous heating and temperature throughout
 - All samples at same temperature and pressure
 - Allows for mixed-batch runs



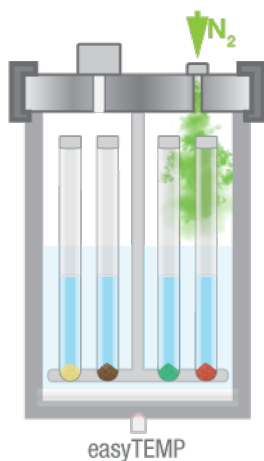
SINGLE REACTION CHAMBER – OPERATING SEQUENCE



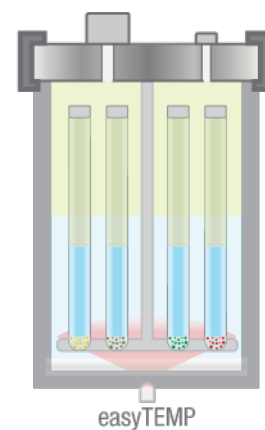
1
Sample rack is lowered automatically into the microwave chamber.



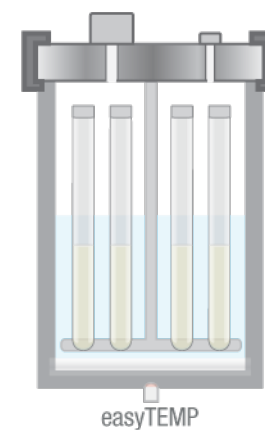
2
Chamber clamp is secured by the operator. Interlocks prevent operation without clamp in place.



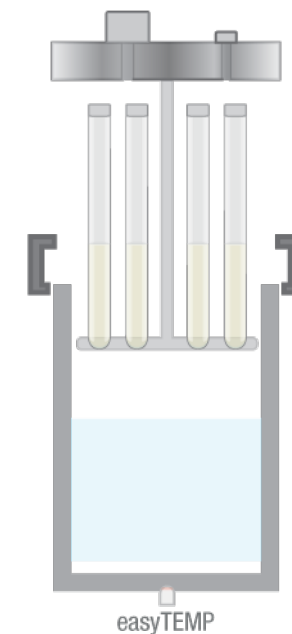
3
Chamber is pre-pressurized with inert gas to prevent sample boiling. Cross contamination is eliminated.



4
Microwave energy is applied. All samples under same temperature and pressure conditions.

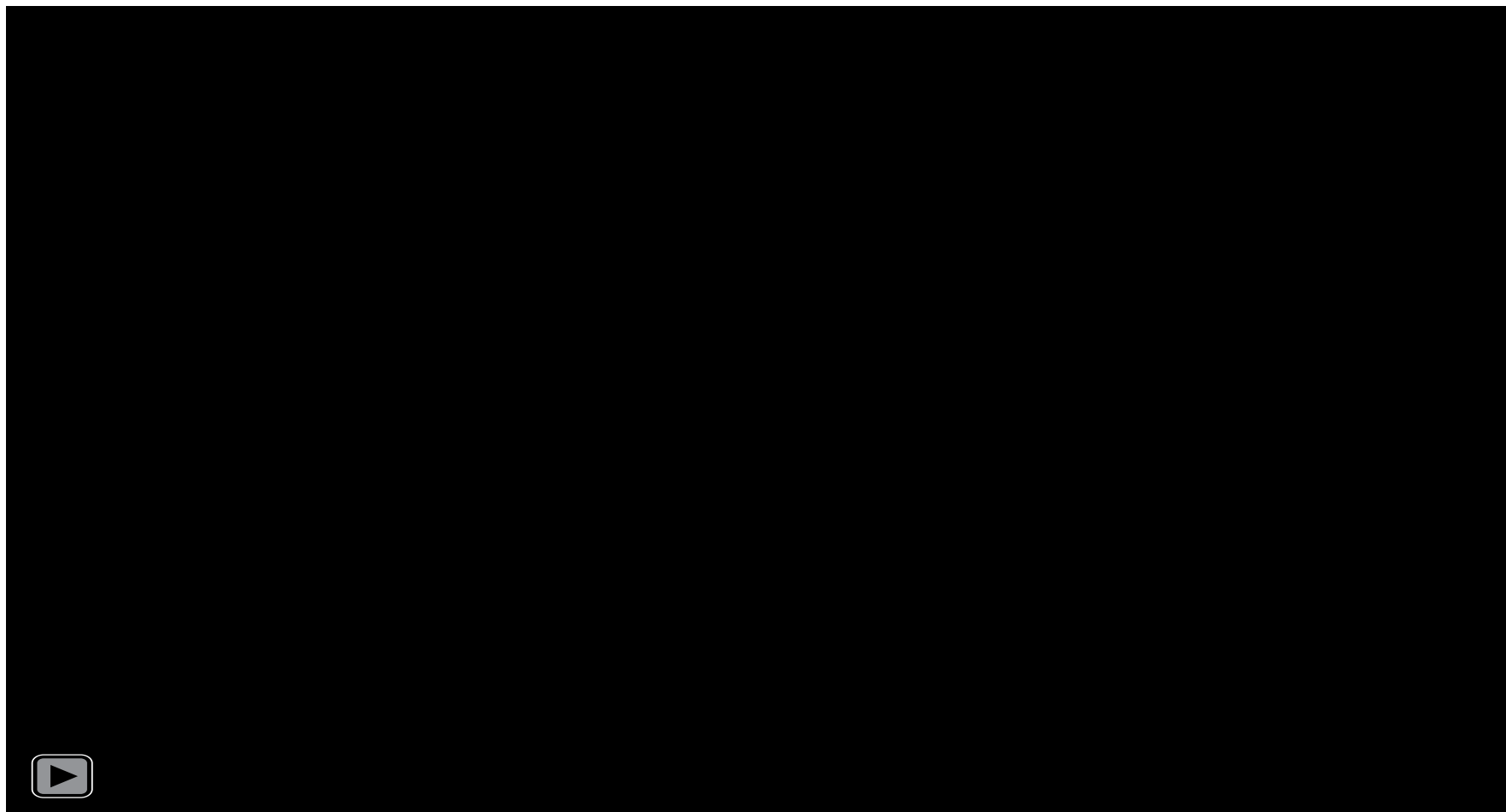


5
Very fast cooling step due to water cooling of chamber. Chamber is vented and acid vapors extracted.



6
Clamp is released and sample rack automatically rises from chamber.

ULTRAWAVE 3 WORKFLOW



BENEFITS OF THE ULTRAWAVE 3 FOR PGE/CATALYST ANALYSIS

- Highest temperature/pressure capability
 - Surpasses temperature and pressure limitations of rotor-based systems
 - Lower analytical blanks and reduced interferences
 - Complete digestion of challenging samples
- Greatly simplified workflow
 - Reduced number of steps
 - Run any mixture of samples together (mixed batch)
 - Suitable for all reagents (including aqua regia, HCl, HF, etc.)



RACKS AND VIALS

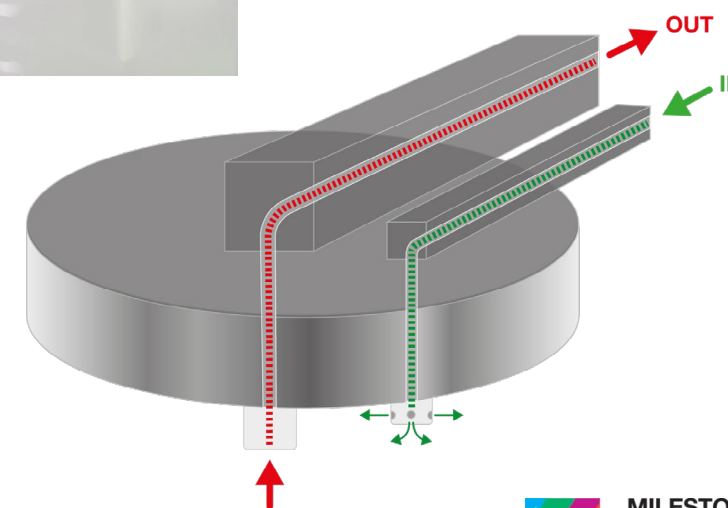
- Most positions available in an SRC system
- Vials available in:
 - PTFE-TFM
 - Quartz
 - Disposable Glass

# of position	Volume (mL)	Vials material
7	40	PTFE-TFM, Quartz, Disposable glass
20	15	
27	8	
40	4.5	Glass

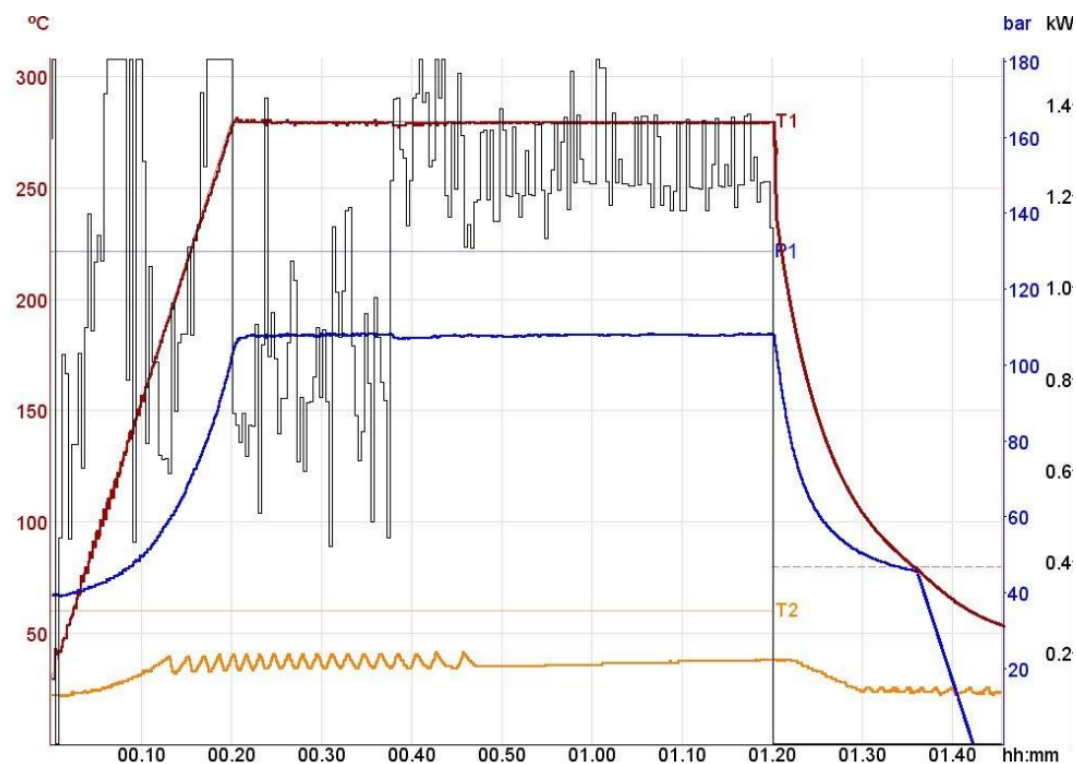


ULTRAWAVE 3 CONSTRUCTION

- High-pressure lines made of:
 - Corrosion-resistant steel (Hastelloy)
- Separated high-pressure lines:
 - Inlet for gas pre-pressurization
 - Outlet for venting at the end of the run
 - No risk of sample contamination
 - Lower blanks
 - Longer lifetime of the pressure lines



ULTRAWAVE 3 COOLING AND EFFICIENCY



- Liquid-cooled reactor and magnetron
 - Delivers higher power for a longer period of time
 - Ensures longer lifetime of the high voltage components
 - Prevents diffusion of corrosive gasses to stainless steel reactor
 - Allows for back-to-back high-temperature runs
 - Silent operation (32 dB) allows for better working conditions within the lab
 - Water vs air-cooling
 - Allows for faster turnaround

EASYCONTROL 3.0

- Intuitive, Fully icon-driven software
- Customizable home page
- Shortcuts to any page
- Sidebar with run info
- Quick start of a digestion
 - Recent methods always listed in the bottom



ALL INFO WITHIN OPERATOR REACH

Milestone Connect:

- Instrument guides
 - User manual, tips and techniques, etc.
- Video tutorials built right into the system
- Instrument info
 - QC reports, SN, etc.
- Spare parts list
- Direct connection to the Milestone Connect website – www.milestoneconnect.com/NA

The screenshot displays the Milestone Connect operator interface for the 'Lignin and cellulose (biopolymers)' system. The interface is organized into several sections:

- Top Navigation:** Home icon, system name 'Lignin and cellulose (biopolymers)', and a menu icon.
- Primary Function Grid:** A 2x5 grid of icons for Run Profile, Sample List, System, Audit Trail, Settings, Method Editor, Recent, Method Library, New Method, and Login.
- Secondary Function Row:** A row of icons for Reports, Instrument guides, Tutorial videos, Milestone Connect, My instrument, and System Dashboard.
- Bottom Method Row:** A row of icons for various methods: Lignin and cellulose (biopolymers), General Method of Environmental Sa..., USP <232> <233> and ICH Q3D elem..., Manganese doped zirconia, Ferberite, and Pine needles (SRM NIST 1575).
- Right Panel:** Real-time monitoring data including date/time (16.09.2022 13:56:57), temperature (T1: 1°C, T2: 1°C), pressure (P1: 0.0bar), and motor power (MW: 0W).
- Control:** A large green play button at the bottom right.

MILESTONE EXPERIENCE WITH PGE'S

The procedure used is reported here below:

Number of positions	Sample name	Sample Amount	Reagents
5	Rh, Ru, Ir sponges	Up to 100 mg	10 mL HCl, 37%

Microwave program:

Step	Time	T1	Power
1	00:25:00	280 °C	1500 W
2	01:00:00	280 °C	1500 W

PROCEDURE FOR PURE RHODIUM POWDER

For this test, 40 mL quartz vials were used. The procedure used is reported below:

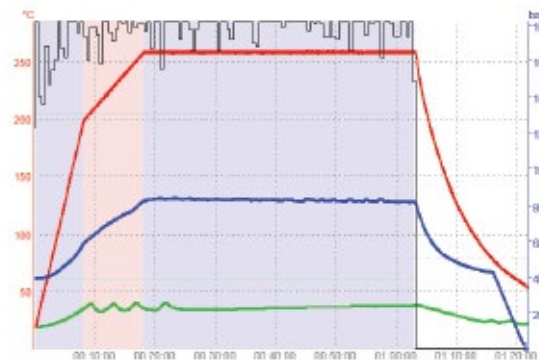
Number of positions	Sample name	Sample Amount	Reagents
5	Rhodium powder	Up to 70 mg	10 mL HCl, 37%, 150 µL of Br ₂

Microwave program:

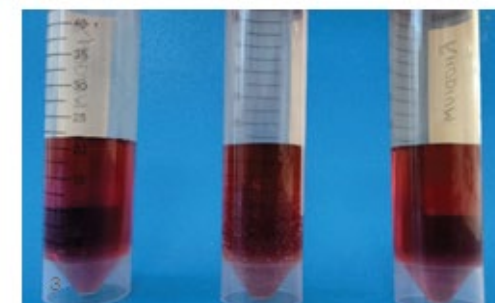
Step	Time	T1	Power
1	00:08:00	200 °C	1500 W
2	00:10:00	260 °C	1500 W
3	00:45:00	260 °C	1500 W

RESULTS FOR Rh, Ru, Ir SPONGES AND RHODIUM POWDER

Samples were completely dissolved without any residue, thus assuring full recovery of the elements of interest.



Rh, Ru, Ir sponges



Pure Rhodium Powder

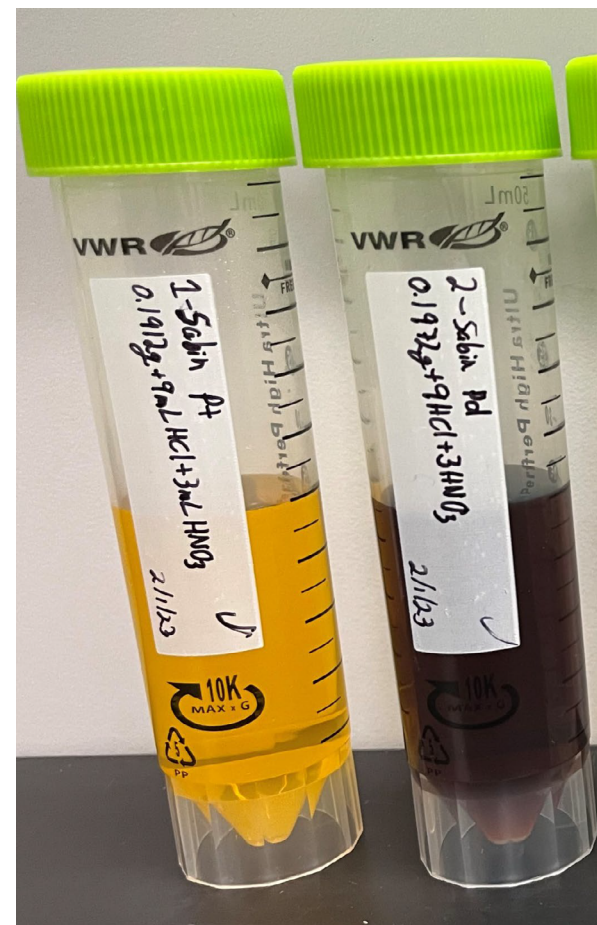
PARTNERSHIPS WITH AURIS NOBLE AND SABIN METAL

- Auris Noble - uniquely difficult sample matrices
 - Interest in use of ultraWAVE for alleviating bottlenecks in their lab
 - Samples for proof of concept and method development, work in progress
 - Auris Noble partnering with our application team on digesting difficult PGE matrices
- Connected to Sabin through Auris Noble
 - Provided samples for digestion
 - Results to be presented at IPMI



PGE DIGESTIONS

- Pure Pt and Pd chunks
- 0.2 g sample + 9 mL HCl + 3 mL HNO₃
- Digested using 40-mL TFM vials
- Microwave program:
 - 20 minutes to 260 °C
 - 30-minute hold at 260 °C
- Result: Complete digestion



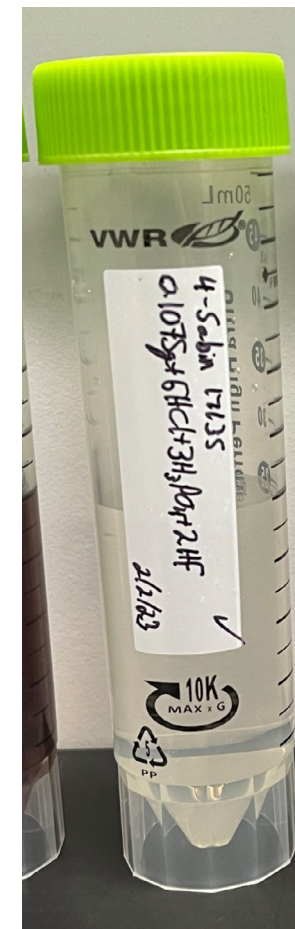
PGE DIGESTION RESULTS – PT AND PD VIA ICP-OES

	Palladium		Platinum	
	Sabin	Milestone	Sabin	Milestone
Ag	10	4	9	8
Al	3	3	1	2
Au	6	9	8	11
Bi	7	8	73	75
Ca	6	66	11	60
Cd	61	65	26	28
Co	9	9	9	6
Cr	5	8	4	5
Cu	10	8	10	9
Fe	18	25	15	11
Ir	10	10	11	11
Mg	nd	3	nd	4
Mn	7	8	10	11
Mo	nd	nd	7	6
Na	nd	nd	nd	nd
Ni	9	9	9	8
Pb	8	8	9	8
Pd	-	-	10	11
Pt	12	10	-	-
Rh	9	11	8	8
Ru	8	4	12	11
S	nd	nd	nd	nd
Sb	18	15	nd	nd
Si	43	27	25	123
Sn	6	9	15	nd
Te	10	13	13	6
Ti	13	14	18	18
V	2	3	4	4
W	nd	nd	6	6
Zn	8	6	10	9
Zr	Nd	nd	5	4
Purity	99.970	99.964	99.966	99.953



PGE DIGESTIONS - 17135

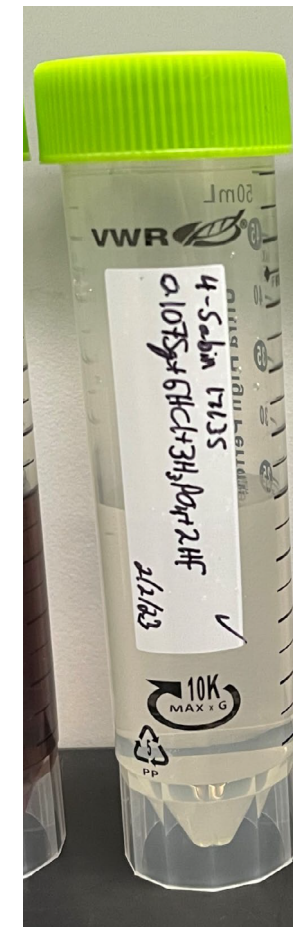
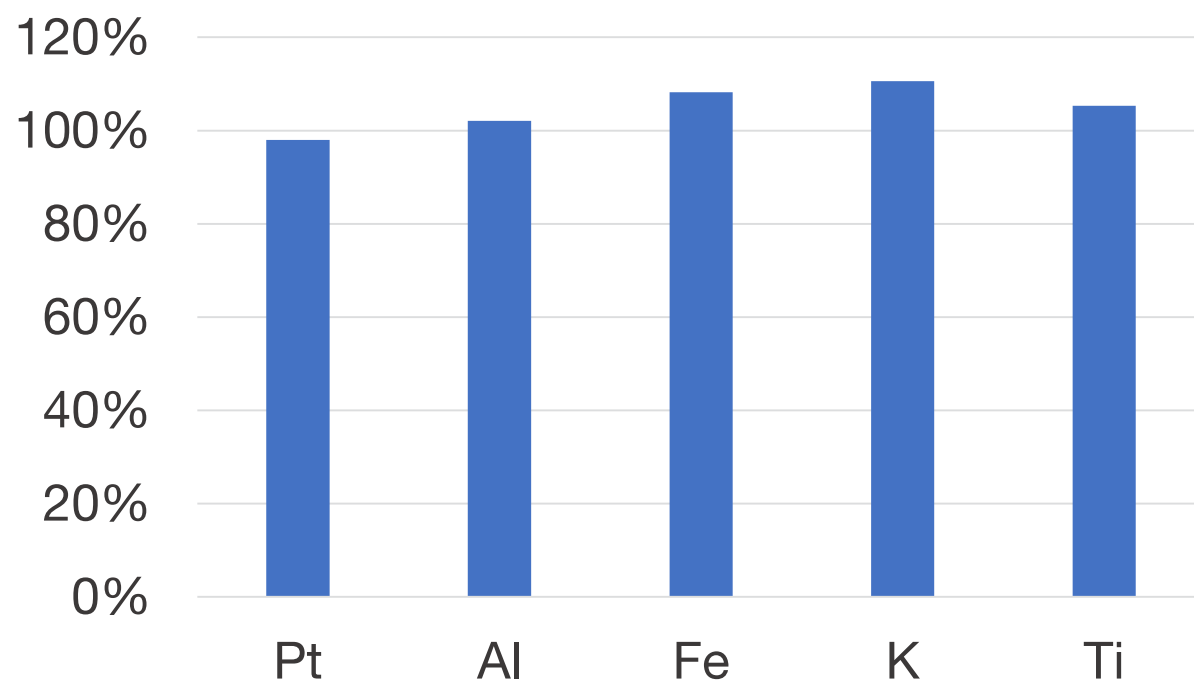
- Light grey powder, spent Pt catalyst on $\text{SiO}_2 / \text{Al}_2\text{O}_3$ substrate
- Sample composition:
 - 63% SiO_2 , 15% Al_2O_3 , 12% K_2O , 1% Pt
- 0.1 g sample + 6 mL HCl + 3 mL H_3PO_4 + 2 mL HF
- Digested using 40-mL TFM vials
- Microwave program:
 - 30 minutes to 280 °C
 - 60-minute hold at 280 °C
- Result: Complete digestion



PGE DIGESTION RESULTS FOR 17135 VIA ICP-OES

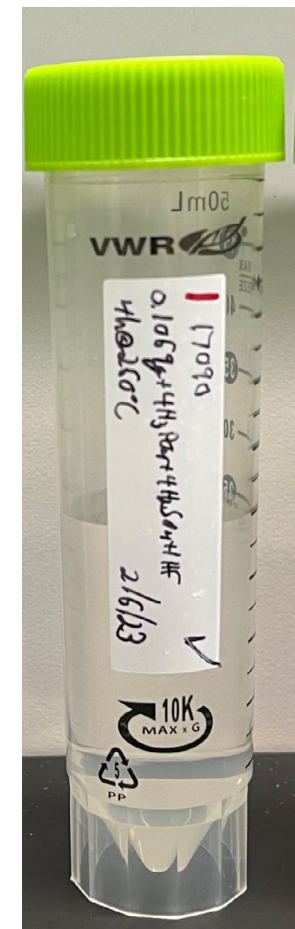
Percent recoveries as compared to results typically obtained by Sabin

- Pt – ICP-OES
- Al, Fe, K, Ti – ED-XRF



PGE DIGESTIONS - 17090

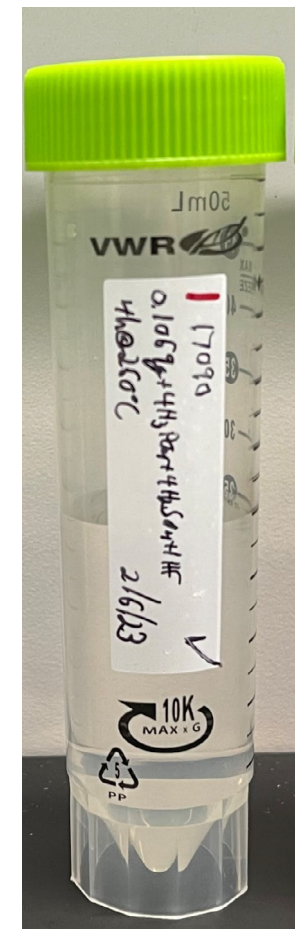
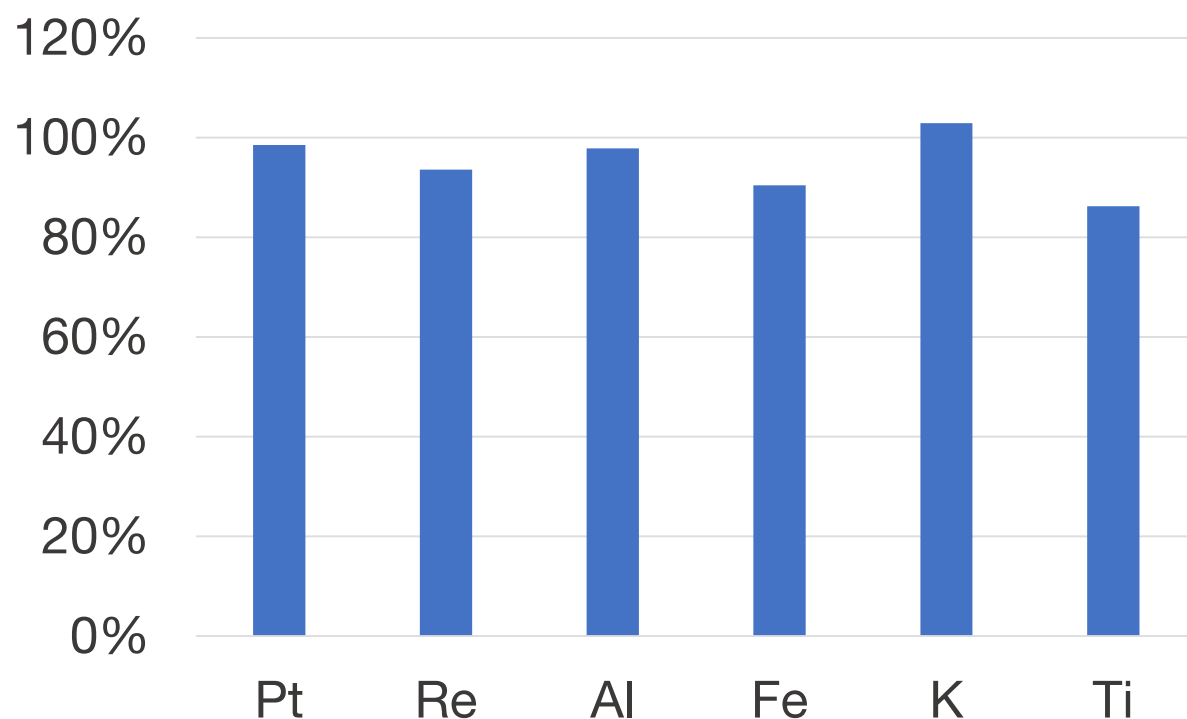
- Light grey powder, spent Pt / Re catalyst on Al_2O_3 substrate
- Sample composition:
 - 93% Al_2O_3 , 5% SiO_2 , 0.2% Pt, 0.2% Re
- 0.1 g sample + 4 mL H_3PO_4 + 4 mL H_2SO_4 + 1 mL HF
- Digested using 40-mL TFM vials
- Microwave program:
 - 20 minutes to 260 °C
 - 4-hour hold at 260 °C
- Result: Complete digestion



PGE DIGESTION RESULTS FOR 17090 VIA ICP-OES

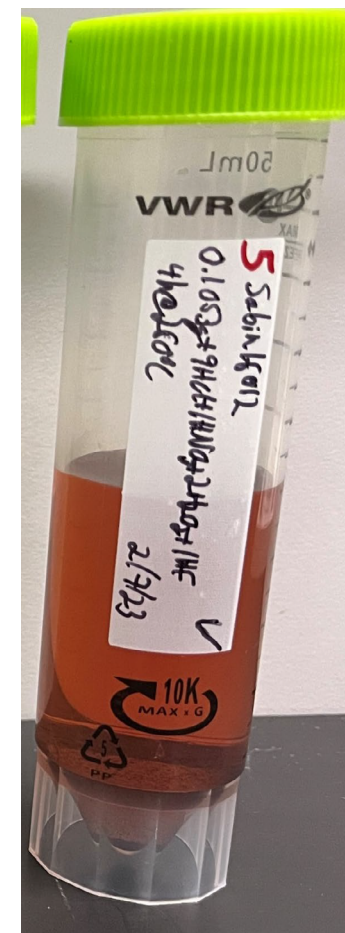
Percent recoveries as compared to results typically obtained by Sabin

- Pt, Re – ICP-OES
- Al, Fe, K, Ti – ED-XRF



PGE DIGESTIONS - 16012

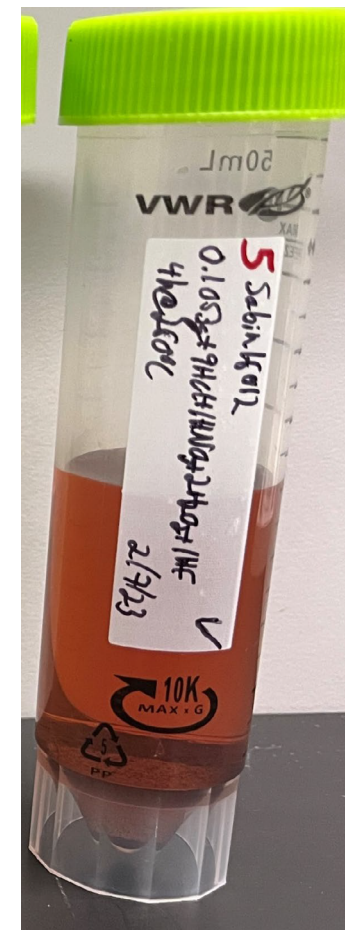
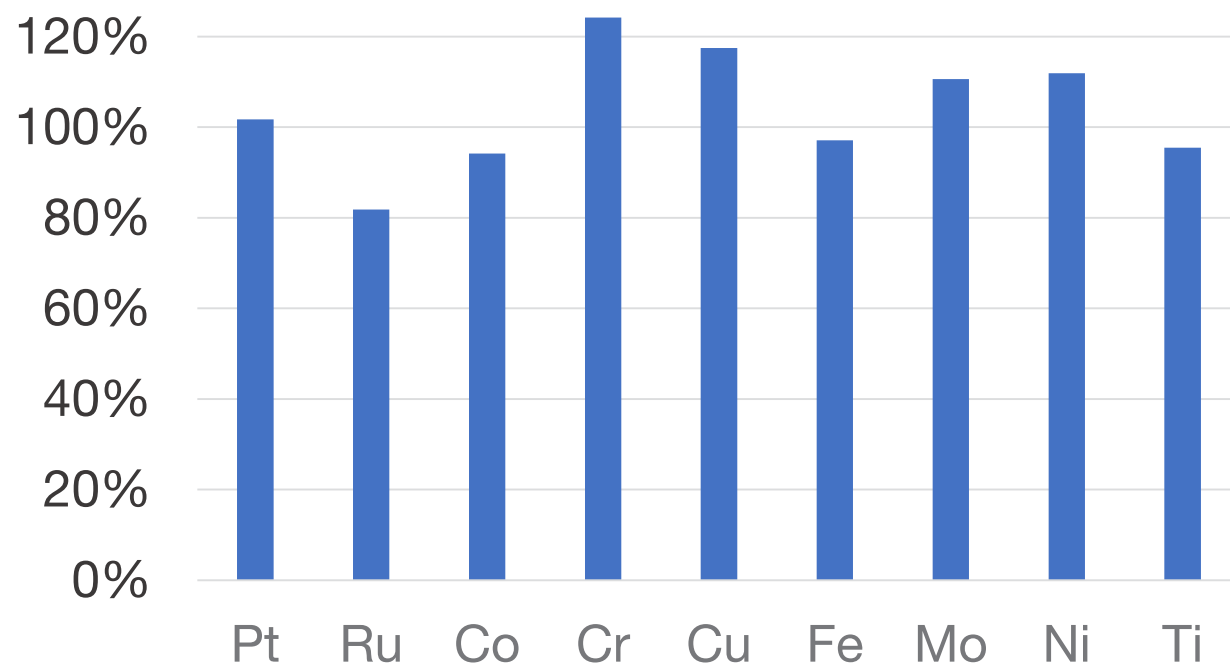
- Dark grey powder, metal flake
- Sample composition:
 - 37% Ru, 20% Pt, 17% Co, 4% Mo, 4% Nb, 4% Fe, 2% Cr
- 0.1 g sample + 9 mL HCl + 1 mL HNO₃ + 2 mL H₂O₂ + 1 mL HF
- Digested using 40-mL TFM vials
- Microwave program:
 - 20 minutes to 260 °C
 - 4-hour hold at 260 °C
- Result: Black precipitate, possibly RuO_x



PGE DIGESTION RESULTS FOR 16012 VIA ICP-OES

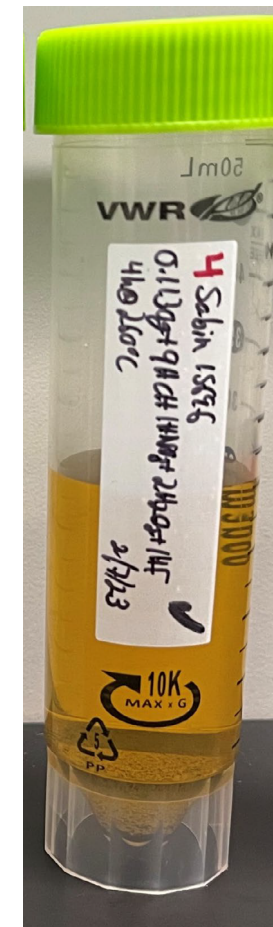
Percent recoveries as compared to results typically obtained by Sabin

- Pt, Ru – ICP-OES
- Co, Cr, Cu, Fe, Mo, Ni, Ti – ED-XRF



PGE DIGESTIONS - 15696

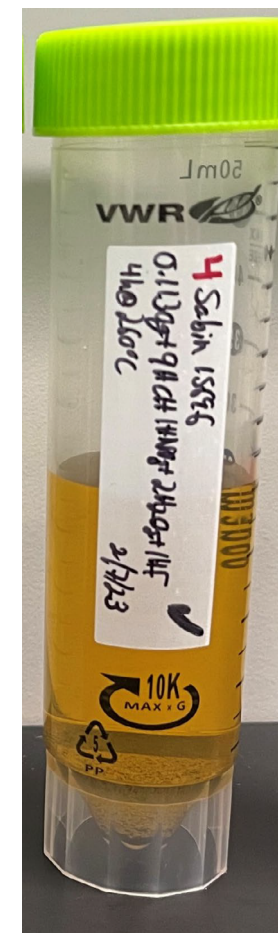
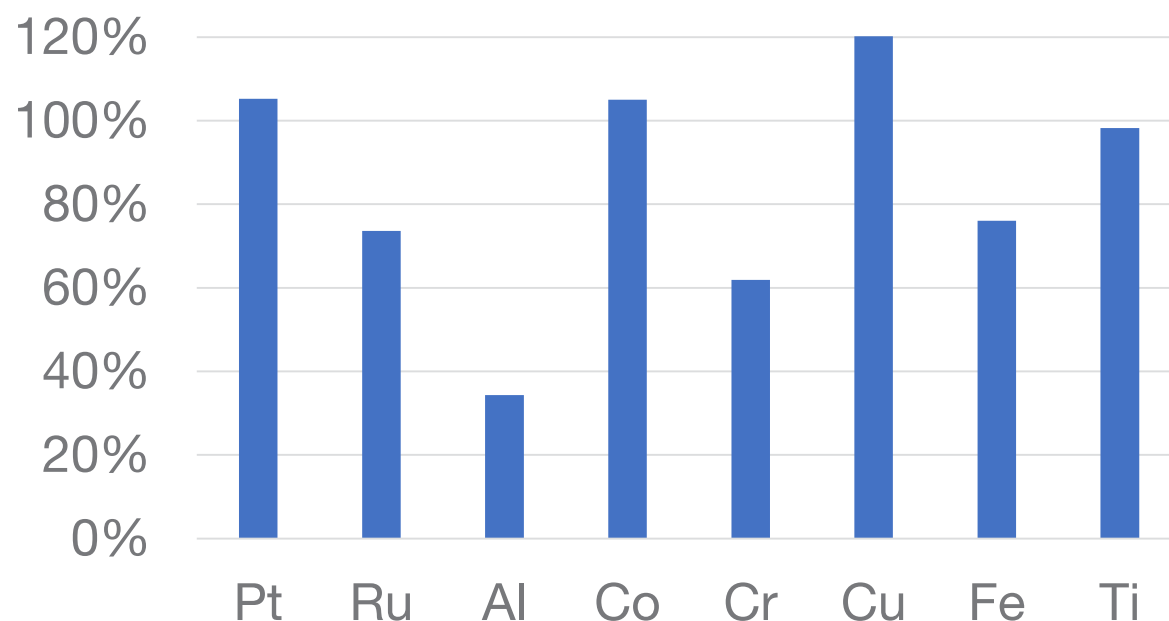
- Dark grey powder, metal flakes
- Sample composition:
 - 50% Pt, 23% Co, 14% Al, 4% Ru, 4% Si, 4% Cr
- 0.1 g sample + 9 mL HCl + 1 mL HNO₃ + 2 mL H₂O₂ + 1 mL HF
- Digested using 40-mL TFM vials
- Microwave program:
 - 20 minutes to 260 °C
 - 4-hour hold at 260 °C
- Result: Black precipitate, possibly RuO_x



PGE DIGESTION RESULTS FOR 15696 VIA ICP-OES

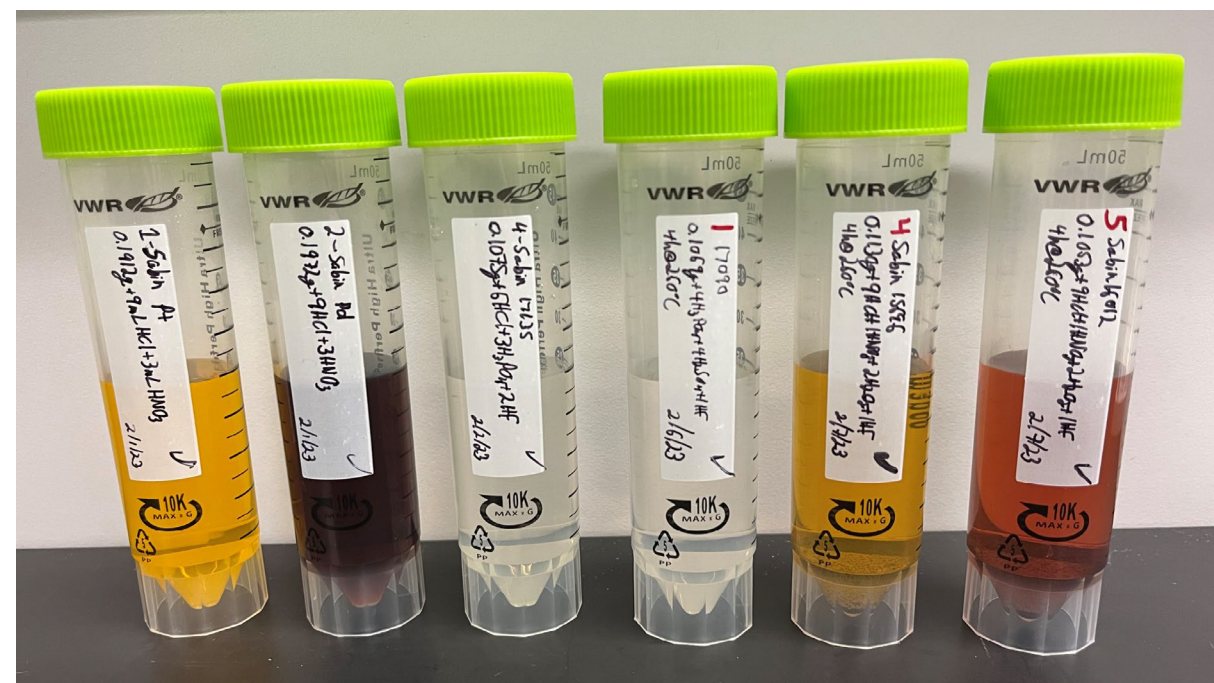
Percent recoveries as compared to results typically obtained by Sabin

- Pt, Ru – ICP-OES
- Al, Co, Cr, Cu, Fe, Ti – ED-XRF



OVERALL RESULTS

- Able to fully digest 4 of 8 samples
 - Some species highly-resistant to acid digestion, e.g, RuO_x
 - Strong correlation between data from digested samples on ultraWAVE and previous analyses by Sabin for most elements
 - Exploring additional method development to further optimize
- Method optimization possible though UW3's simplified method development capabilities
 - e.g, higher sample mass, lower acid volumes, shorter digestion times
- ultraWAVE 3's construction and components allow for high temps to be reached, and extended time at temperature, as needed
 - Water-cooled magnetrons
 - Powerful 1000 W external chiller
 - Quartz bottom prevents diffusion of reaction gases to SRC



WHY ULTRAWAVE 3 IS DIFFERENT?

- Performance

- Highest temperature and pressure capabilities of a microwave digestion system
- Enhanced Corrosion Resistance allows for use of any acid type using vials with loose-fitting caps
- Water-cooled magnetrons and quartz bottom allow for ultraWAVE 3's superior performance
 - High temperatures, extended time at temperature
- Better digestion control through easyTEMP
- Lower analytical blanks

- Productivity

- Higher number of positions than ultraWAVE 2
- Fast / efficient cooling with powerful external chiller

- Usability

- Vials with loose-fitting caps – less handling / operator labor
- No vessel assembly / disassembly
- Silent operation

- Reliability

- Acid-resistant, high-pressure lines
- Long lifetime of hardware components
- Low cost of ownership / operation