

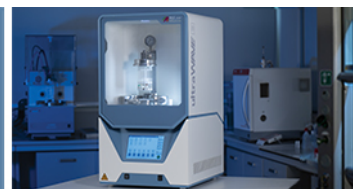
Procedure for complete digestion of Rh, Ru, Ir sponge and pure Rhodium powder, using ultraWAVE 3 system

I INTRODUCTION

Platinum Group Elements (PGEs) are considered as the most valuable precious metals. For this reason, they are often used for jewelry and ornament purpose, but they also play an important role in the automotive catalytic converter production, thanks to their properties to convert cars emissions in less toxic compounds. Samples with PGEs content are chemically inert materials and, for this reason, they are difficult to be prepared for ICP analysis. Hot plate systems are often not the right solution, since they don't provide with enough temperature and pressure capabilities to assure the complete digestion. Moreover, they suffer from airborne contamination, long programs and poor recovery of volatile compounds. The closed vessel microwave digestion, has proven to be an effective technique with better, fast digestion, clean environment, and full recovery of volatile compounds but PGEs samples requires high temperature and pressure conditions that, in a conventional microwave unit, lead to frequent consumables replacement.

Milestone's ultraWAVE 3, thanks to its unique Single reaction chamber (SRC) technology, is the only system able to achieve high temperature and pressure conditions, necessary for a better digestion of PGEs samples, preventing high consumables cost. ultraWAVE 3 stainless steel reactor and its unique technology allows to perform a simultaneous digestion of samples at high temperature for long time.

This industry report describes how a variety of PGEs samples were digested simultaneously using the Milestone's ultraWAVE 3, and this can be replicated in previous ultraWAVE generation, without sample-to-sample cross contamination.



EXPERIMENTAL

INSTRUMENT

- Milestone's ultraWAVE 3
- 7-positions rack with 40 mL quartz vials
- Analytical balance
- ICP-MS

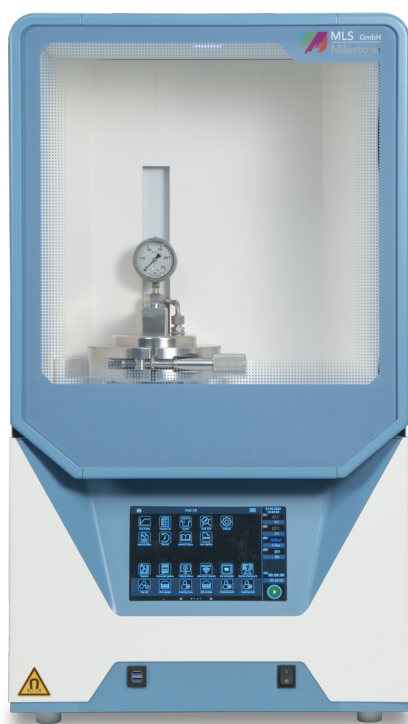


Figure 1 – Milestone's ultraWAVE 3

Developed and patented by Milestone, the ultraWAVE 3 represents another significant step forward for SRC technology and embrace Milestone 20+ years of experience. The stainless-steel reactor with a high-purity PTFE-TFM liner and cover, enables to achieve, higher pressures and temperatures regardless the sample type and acid mixture. The digestion process is continuously controlled by easyTEMP, an advance contactless sensor that measure the temperature directly of the reaction chamber. The simplified rack construction eliminates the time need to assemble and disassemble the vessels. Just as important, dissimilar samples can be

processed simultaneously using disposable glass, quartz or PTFE-TFM vials, thus saving time and money. The ultraWAVE 3 addressed all the sample preparation challenges related to performance, time, workflow, and cost of ownership.

SAMPLES

Pure Rhodium powder and Rh, Ru and Ir sponge samples, frequently used for automotive catalytic converter production, were digested.

SAMPLE PREPARATION

An amount of 0.1 g of each Rh, Ru and Ir sponge were weighed inside a 40 mL quartz vial and 10 mL of 37% HCl were added.

An amount of 0.07g of Pure Rhodium powder was weighed inside a 40 mL quartz vial and 10 mL of 37% HCl + 150 μ L Br₂ were added.

The rack with the 7 quartz vials was positioned inside the SRC TFM liner inside the reaction chamber, which was previously filled with 120 mL of H₂O and 5 mL of HNO₃. The SRC system was pressurized up to 40 bar with Nitrogen gas to close the glass vials.

The following microwave heating program was applied for the sponges:

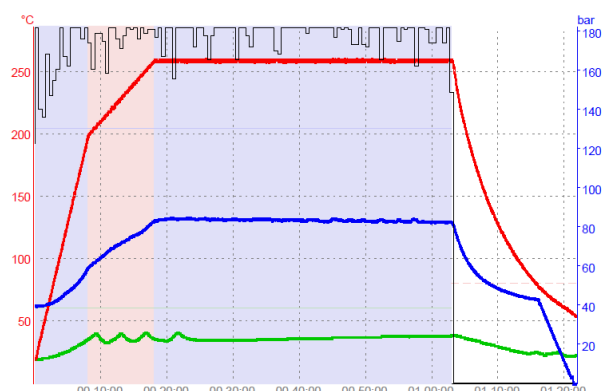
Step	Time (min)	Power (W)	T1 (°C)	T2 (°C)	P (bar)
1	25	1500	280	60	90
2	60	1500	280	60	90

Table 1 - Microwave Program for the sponges

The following microwave heating program was applied for the pure Rhodium powder:

Step	Time (min)	Power (W)	T1 (°C)	T2 (°C)	P (bar)
1	08	1500	200	60	90
2	10	1500	260	60	110
3	45	1500	260	60	110

Table 2 - Microwave Program for the pure Rh powder



After cooling the sample digests were diluted with deionized water up to 50 mL and then analyzed by ICP-MS.

RESULTS AND DISCUSSION

Samples were completely dissolved using the ultraWAVE 3 without any residue thus assuring full recovery of the elements of interest.

CONCLUSIONS

ultraWAVE 3 is able to achieve the complete digestion of the most challenging inorganic samples (with PGEs content, for example) such as the Rh, Ru and Ir sponges and the pure Rhodium powder. ultraWAVE 3 achieves higher unmatched temperature and pressure capabilities compared to rotor-based systems, even for long processing time assuring their complete digestion.

Consumable cost is strongly reduced thanks to the use of inexpensive vials and to the performances granted by SRC technology.

Milestone's ultraWAVE 3 is the perfect solution for preparation of samples with PGEs content.



Figure 1 - Rh, Ru, Ir sponges

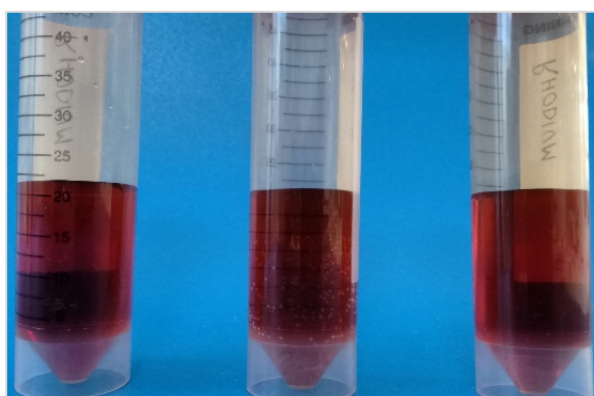


Figure 2 – Pure Rhodium powder

