

# Digestion of Platinum, Palladium, and Rhodium by Using PTFE-TFM Insert Vials for ICP-OES/MS Analysis

Relevant for: Mining, Metals, Catalysts

A novel digestion setup, using sealed quartz vessels with PTFE-TFM insert in Multiwave 7000 allows for complete digestion of platinum group metals (PGMs) and enables the additional determination of silicon in PGMs which is otherwise not possible.

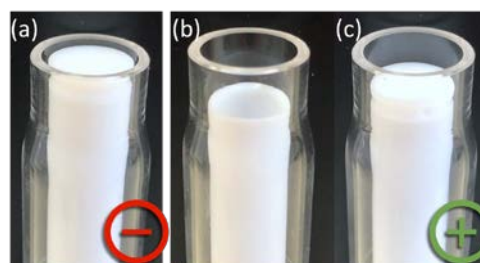


Figure 1: Quartz vessel with PTFE-TFM insert vial: (a) original with plug-on cap; (b) shorter 17 mL vial; (c) shorter vial with plug-on cap

## 1 Introduction

Digestion of Platinum Group Metals (PGMs) like platinum, palladium, and rhodium, is quite challenging. It requires high temperatures, long reaction times and highly corrosive conditions, like HCl or *aqua regia*.

Sealed quartz vessels in Multiwave 7000 allow for safe usage of these reagents, but can compromise accurate determination of Si.

A new digestion technique – performing PGM digestion in PTFE-TFM insert vials within the sealed quartz vessels – helps to overcome this limitation.

## 2 Instrumentation

Digestions were performed in Multiwave 7000 by using Rack 6 with sealed quartz vessels.

The PTFE-TFM insert vials have been taken from Rack 18, but have a reduced length and volume (Figure 1). This is necessary to avoid accidental opening of the sealed quartz vessels due to thermal expansion of the PTFE-TFM vials during a digestion run.

For complete digestion of rhodium, the magnetic stirrer device is required.

## 3 Experimental

### 3.1 Samples

Powders of platinum, palladium (up to 200 mg), and rhodium (up to 100 mg) were used in order to demonstrate the digestion of pure PGMs.

### 3.2 Reagents

HNO<sub>3</sub>, 65 %

HCl, 32 %

HClO<sub>4</sub>, 70-72 %

All reagents were of p.a. grade

### 3.3 Digestion Procedure

Samples were directly weighed into the shorter 17 mL PTFE-TFM vials. The vials containing rhodium were additionally loaded with a magnetic stir bar, as the digestion does not become complete without stirring. The acid mixtures listed in Table 1 were added while taking care that any sample residues were rinsed down from the vial wall.

Sample	Weight	Acid mixture
Platinum powder	200 mg	2 mL HNO <sub>3</sub> + 6 mL HCl
Palladium powder	200 mg	2 mL HNO <sub>3</sub> + 6 mL HCl
Rhodium powder*	100 mg	6 mL HCl + 1 mL HClO <sub>4</sub>

\*...A magnetic stir bar was added to the rhodium sample  
 Table 1: PGM samples, sample weight and acid mixtures

The 50 mL quartz vessel was filled with a solution of 1 mL HNO<sub>3</sub> and 2 mL H<sub>2</sub>O. This solution is essential to minimize the reagent loss from the PTFE-TFM vial.

The PTFE-TFM vial was carefully inserted into the quartz vessel (without damaging its sealing areas), and closed with a plug-on cap.

Finally, the quartz vessels were sealed with PTFE film by using the wrapping technique and loaded into Rack 6.

The loaded rack was put into the liner already filled with load solution (150 mL water and 5 mL HNO<sub>3</sub>). The liner was inserted into the Pressurized Digestion Cavity (PDC) of Multiwave 7000, and the temperature program (see chapter 3.4) was started. Figure 2 shows the pressure and temperature course of the digestion.

### 3.4 Instrument Parameters

- Starting pressure: 55 - 60 bar
- Cooling temperature: 50 °C
- Pressure release rate: 10 bar/min

Step	Time/min	Temperature/°C	Max. Pressure/bar	Stirring
1	15:00	280	140	High
2	60:00	280	140	High

Table 2: Temperature program for the digestion of PGMs

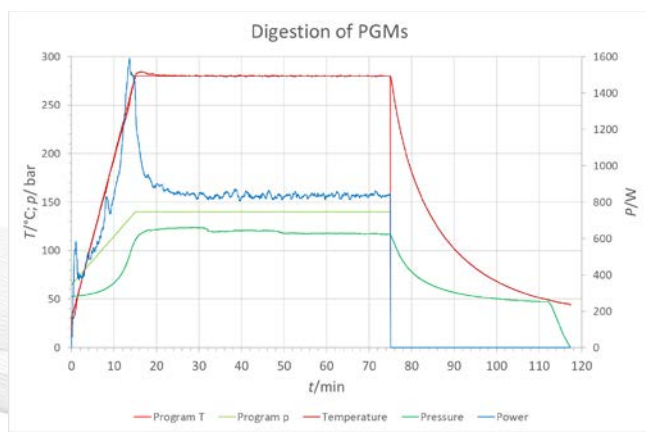


Figure 2: Run data

### 3.5 Post-Digestion Procedure

After the digestion the sealed quartz vessels were transferred from the liner and the PTFE sealing was removed.

Before extracting the PTFE-TFM insert vials from the quartz vessels, the plug-on caps had to be removed by means of a contact lens plunger (Figure 3) or pointed tweezers. Again, it is important not to damage the sealing area of the quartz vessels.

The dissolved PGM samples were transferred into 15 mL autosampler tubes.

The PTFE-TFM insert vials were rinsed with deionized water, and the rinsing solution was added to the sample vial.

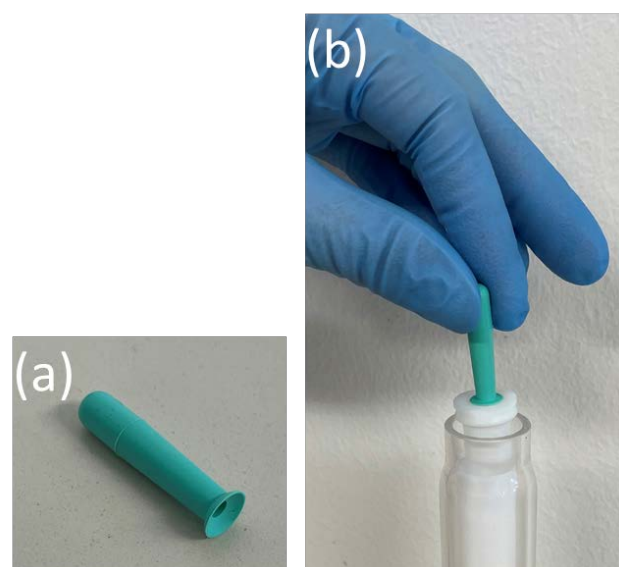


Figure 3: (a) lens plunger; (b) removing of the plug-on cap with the lens plunger

## 4 Results

Platinum and palladium were completely digested with aqua regia after 1 h at 280 °C.

Rhodium was completely digested as well, but with perchloric acid instead of nitric acid. Without stirring the rhodium powder is not fully exposed to the acid mixture, thus resulting in incomplete dissolution.

Digestion has been done with and without plug-on caps. While plug-on caps provide less reagent loss (approx. 15 %), the handling of the PTFE-TFM insert vials without plug-on caps (approx. 30 % loss) is more convenient.

It is clearly visible from Figure 4 that the reagent loss after digestion is lower if a plug-on cap is used.

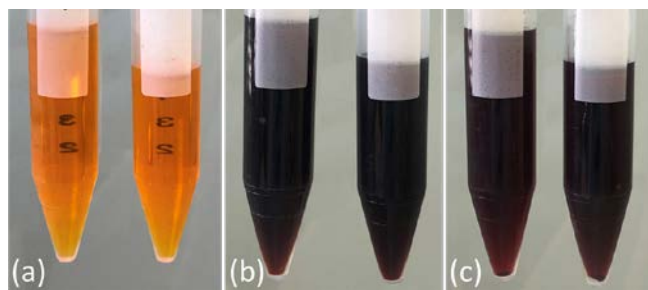


Figure 4: PGM samples after digestion: (a) platinum; (b) palladium; (c) rhodium.  
The left vials have been used with plug-on caps.

## 5 Conclusion

This new digestion approach – using a PTFE-TFM insert vial within a sealed quartz vessel in Multiwave 7000 – provides satisfactory digestions results for platinum, palladium and rhodium. As the sample does not come into contact with the quartz surface, this setup allows an unbiased determination of silicon in these samples.

## 6 Order Information

256223	17 mL TFM Vials
179041	Plug-on cap 18 mL Vial
70868	Vessel 50 mL Quartz
70867	Lid 22 mm Quartz

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