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Solid Phase Extraction using Molecular Recognition Technology for Highly Selective Platinum Group Metals Separations

Session 1290 - Sample Preparation: General
Day and Time: Tuesday, March 04, 2008, Morning

Molecular Recognition Technology – Solid Phase Extraction (MRT-SPE)

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Abstract

Platinum Group Metals (PGMs) have scarcity value, and are broadly used as purification catalysts in fuel cells and diesel engine oils in recent years. The demand of PGMs for these and other applications is increasing.

However, there is concern that, in the future, the production of PGMs may not keep up with this increasing demand. As a result, recycling of PGMs from spent fuel catalysts or from base metal by-product refining is becoming important.

For the separation of PGMs for analysis, traditional methods use sedimentation, liquid-liquid-extraction, or ion-exchange extraction. Such techniques require many extraction steps and are labor-intensive.

In this study, an efficient way to separate PGMs was demonstrated using solid phase extraction (SPE) with molecular recognition technology (MRT). For the separation resin, AnaLig® PM-01, 05, 07 and 08 were used, and the adsorption behavior of PGMs to AnaLig® and the elution performance after rinse steps were examined.

Non-metal needle modified ASPEC XLi, which is an automatic solid phase extraction pretreatment method, was used for the metal analysis. After SPE extraction, the metal extracts were injected into inductivity coupled plasma optical emission spectroscopy (ICP-OES), after which the recovery rate of PGM from the AnaLig® was calculated. Finally, the selectivity of this method versus interfering metals was examined. AnaLig® PM-08 was found to be highly selective for PGMs in acid solution..

Background and Objective



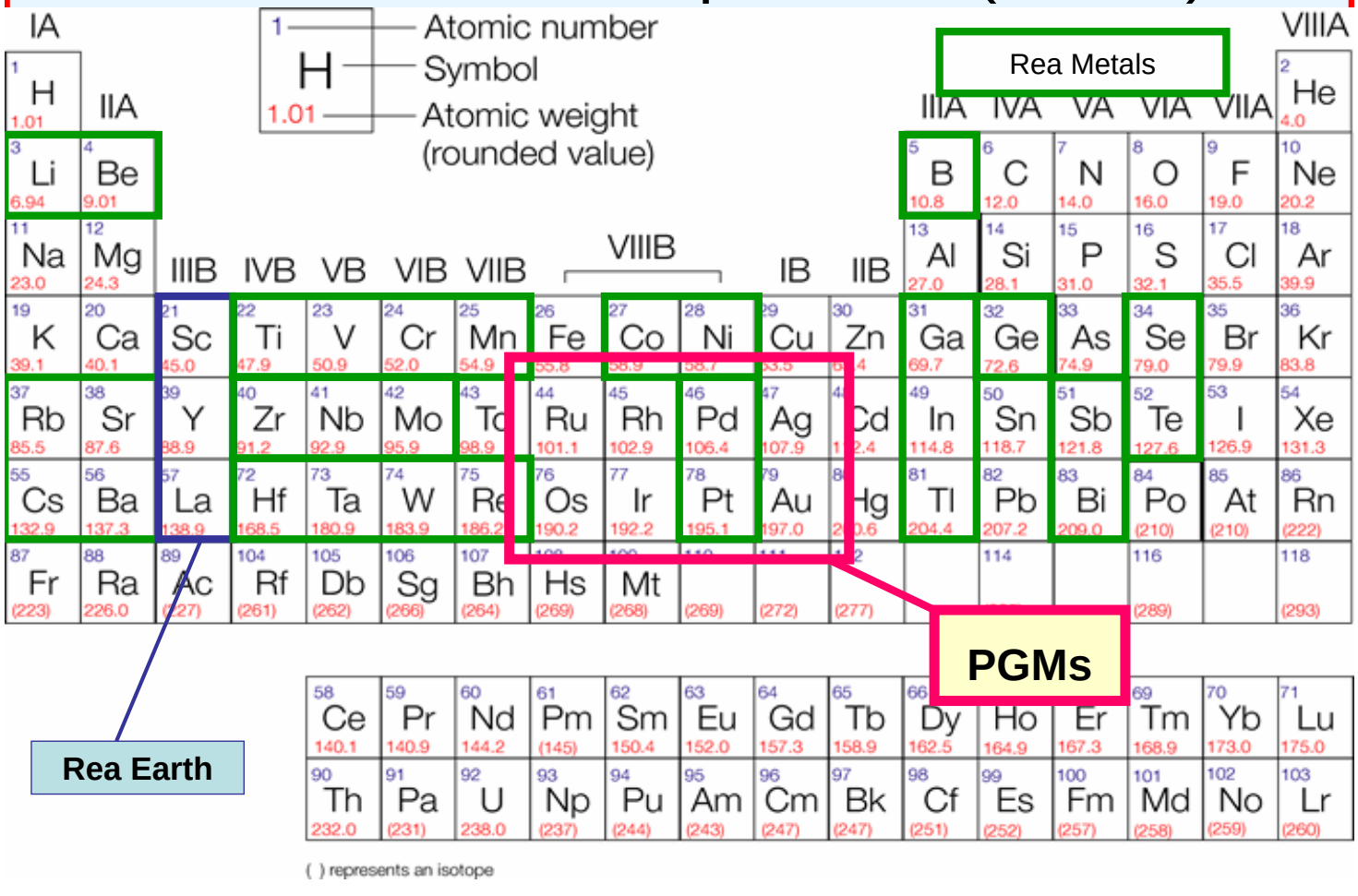

Purification of Platinum Group Metals (PGMs) by SPE

Re-Cycling demand increase about PGM

Current ICP-OES technique is poor for this topics

Develop a useful method for PGM analysis using MRT-SPE-ICP-OES

What is Precious Group Metals (PGMs)?



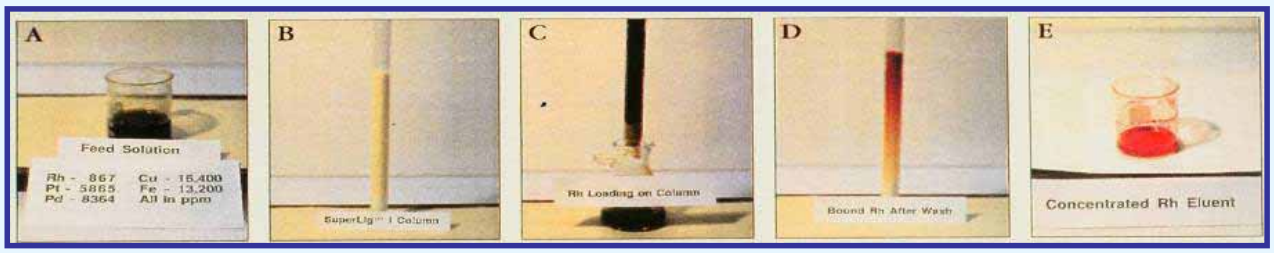
MRT-SPE Method for PGMs from High Matrix

Typical current Technique	Present method
Open column with ion Ex resin for separation of PGMs	Separate PGMs by MRT-SPE SPE cartridge is much easier handling than open column
Anion Ex resin is not good for high selective separation from matrix	MRT-SPE can separate target PGM element from main metal matrix
Not enough clean up for determination of PMGs by ICP-OES	

Spectral Interference
Physical Interference
Chemical Interference

→ Reduce each interference

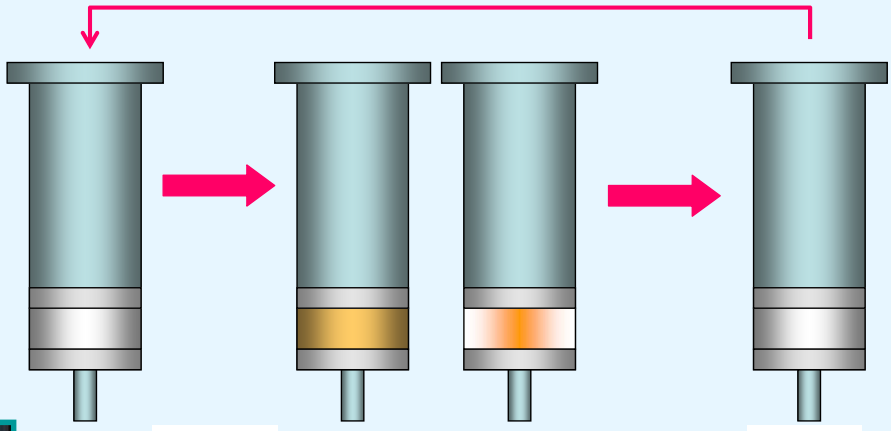
MRT-SPE can separate PGMs

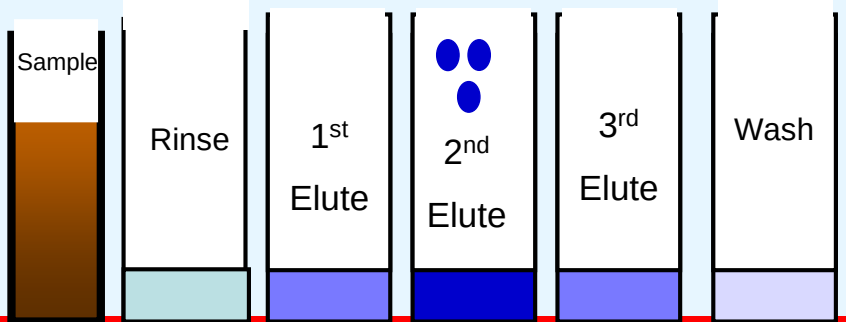


Open Column

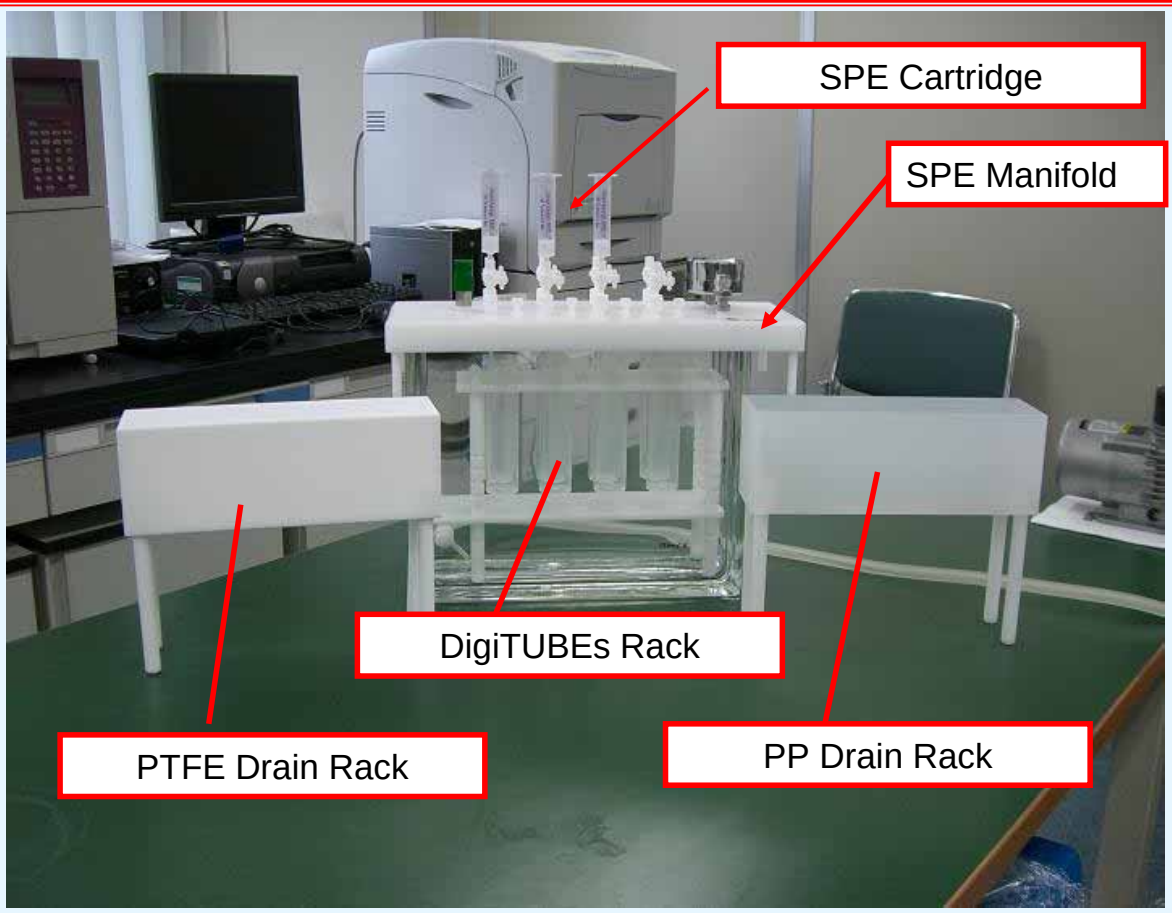
↓

SPE





SPE Manifold System for Metal Separation



Evaluation System for Automation SPE Analysis

MetaSEP AnaLig® Technology

SPE Cartridge

SPE mini Column

Computer

Full Automated Sequential Control by PC Software

High Selective

High Sensitive

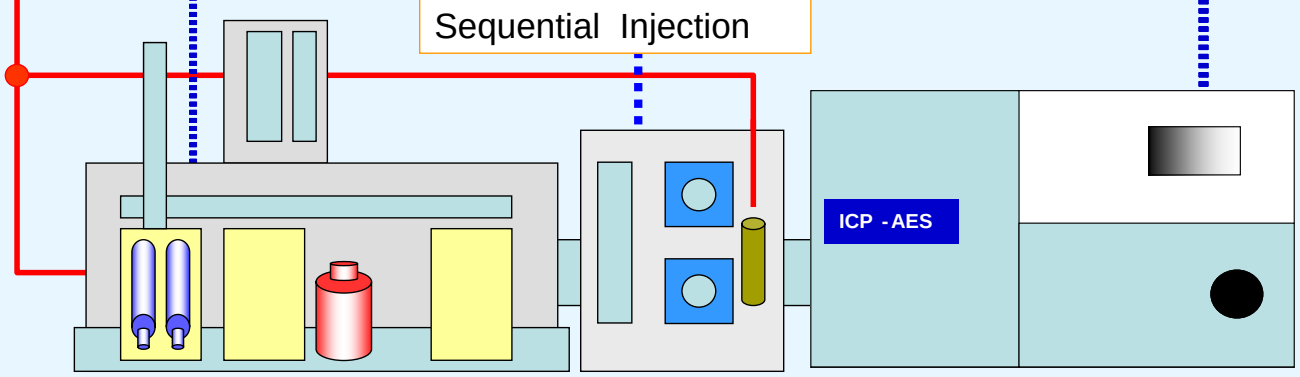
ICP-AES Analysis

Sample Preparation

1st SPE Treatment

2nd MRT-SPE

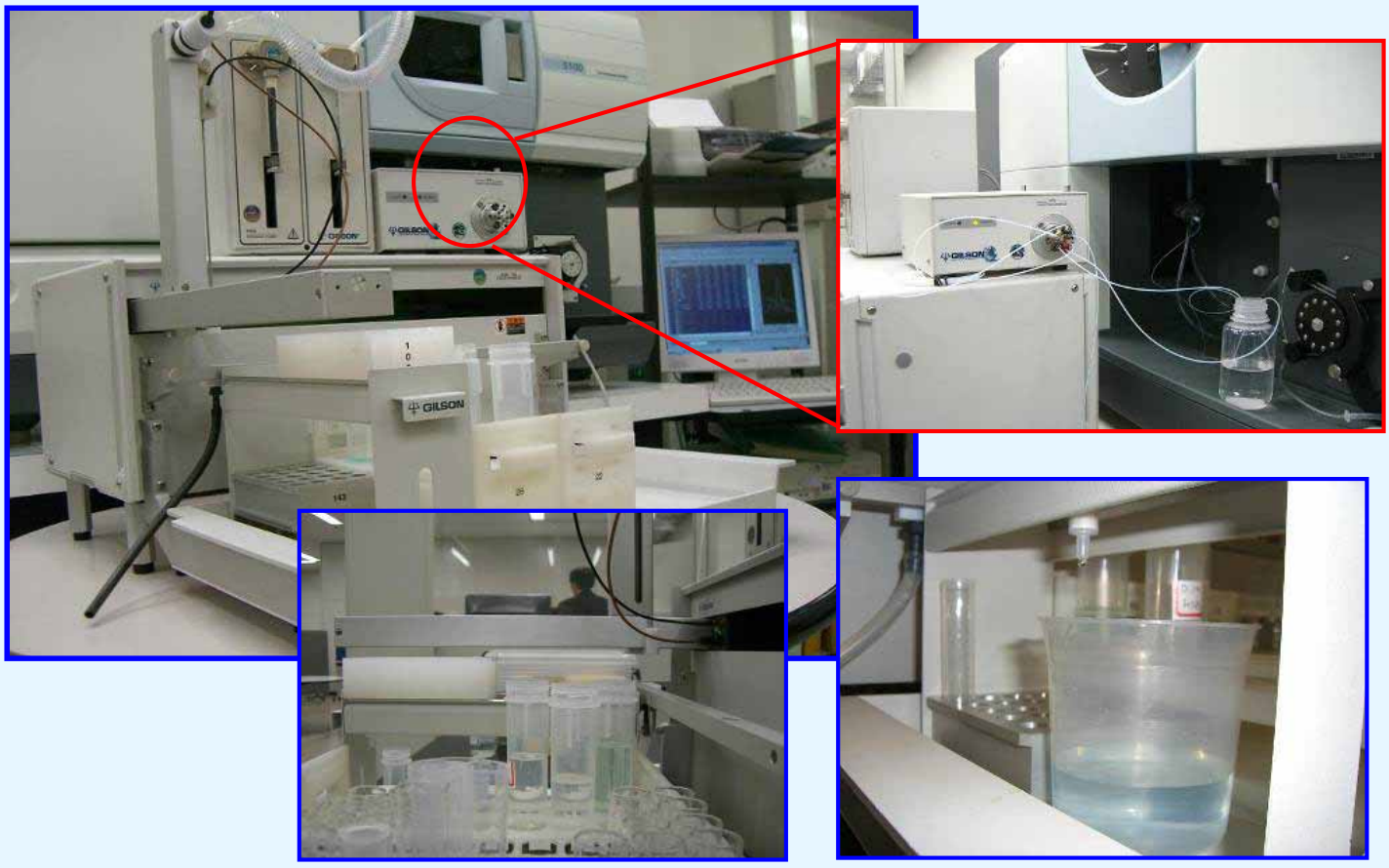
Sequential Injection



ASPEC XLi for Metal
M&S Instruments

MetaPREP SIA-BOX
GL Sciences

SPS5520 ICP-AES
SII Nanotechnology



Method Development using Automated SPE system

MetaSEP AnaLig® PM Series procedure for PGMs

PM-01,05,07,08 SPE Cartridge

Sample 4ml

pH <1 by 0.1M HCl

Au、Pd、Pt、Rh、Ru

0.1M HCl**溶液**

1ml/min

Loading

0.5M thiourea/0.1MHCl 4ml

Pure water 4ml ✕3

10ml/min

Pure water 4ml

0.5ml/min

Elute

Method 1) : 0.5M Thiourea / 0.1MHCl 2ml **x** 4
Method 2) : 0.5M Ammonium Chloride 2mL x 4

Dilution

Analysis by ICP-OES

SPS5520 SII nanotechnology

Recovery Test of MetaSEP AnaLig PM Series for PGMs

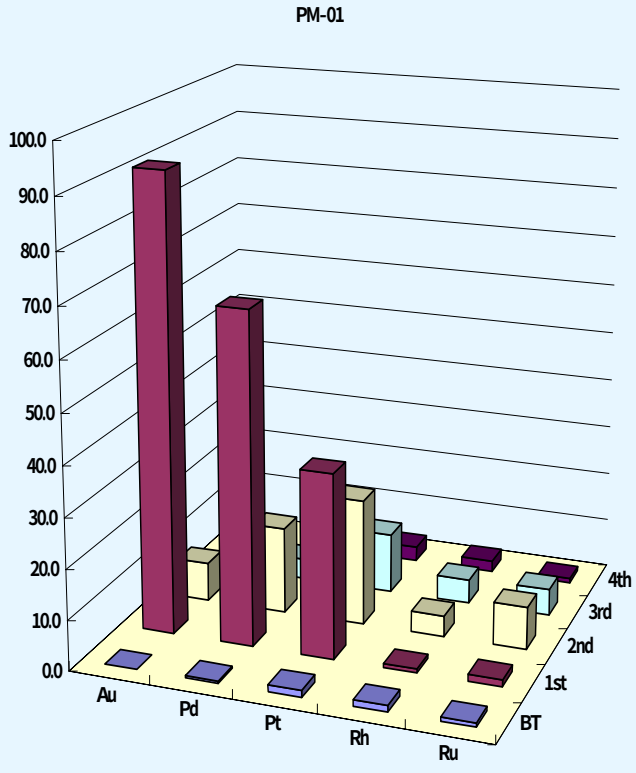
Cartridge size: 500mg/3mL

	Au		Pd		Pt		Rh		Ru		Ir	
	BT	EL	BT	EL	BT	EL	BT	EL	BT	EL	BT	EL
PM-01	0.0%	99.5%	0.4%	88.0%	1.4%	76.9%	1.3%	12.1%	0.8%	16.1%	-	-
PM-02	0.1%	103.1%	0.4%	105.9%	0.4%	73.3%	14.3%	10.3%	7.0%	3.4%	4.4%	4.4%
PM-03	0.1%	104.2%	0.2%	101.2%	68.0%	12.2%	77.0%	0.6%	66.1%	7.1%	75.6%	1.7%
PM-04	0.1%	103.4%	0.2%	105.5%	0.5%	95.0%	20.2%	42.5%	14.8%	19.9%	2.4%	15.5%
PM-05	0.0%	107.1%	0.0%	106.9%	64.6%	6.6%	70.6%	1.5%	67.5%	0.8%	-	-
PM-07	0.0%	103.3%	1.5%	103.5%	1.9%	95.8%	14.0%	4.7%	4.9%	2.7%	-	-
PM-08	0.0%	108.3%	0.0%	106.5%	2.0%	96.5%	15.5%	15.9%	36.6%	16.6%	-	-
PM-12	0.0%	37.3%	0.2%	103.3%	0.3%	87.3%	48.2%	22.7%	17.0%	10.9%	12.3%	8.9%

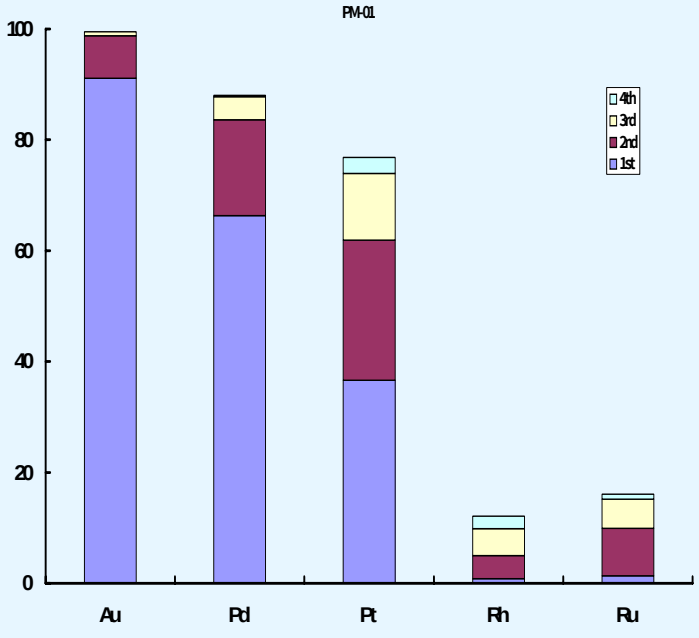
BT: Cartridge Break Through

EL: Elution Recovery from Cartridge

MetaSEP AnaLig® PM-01 for PGMs data



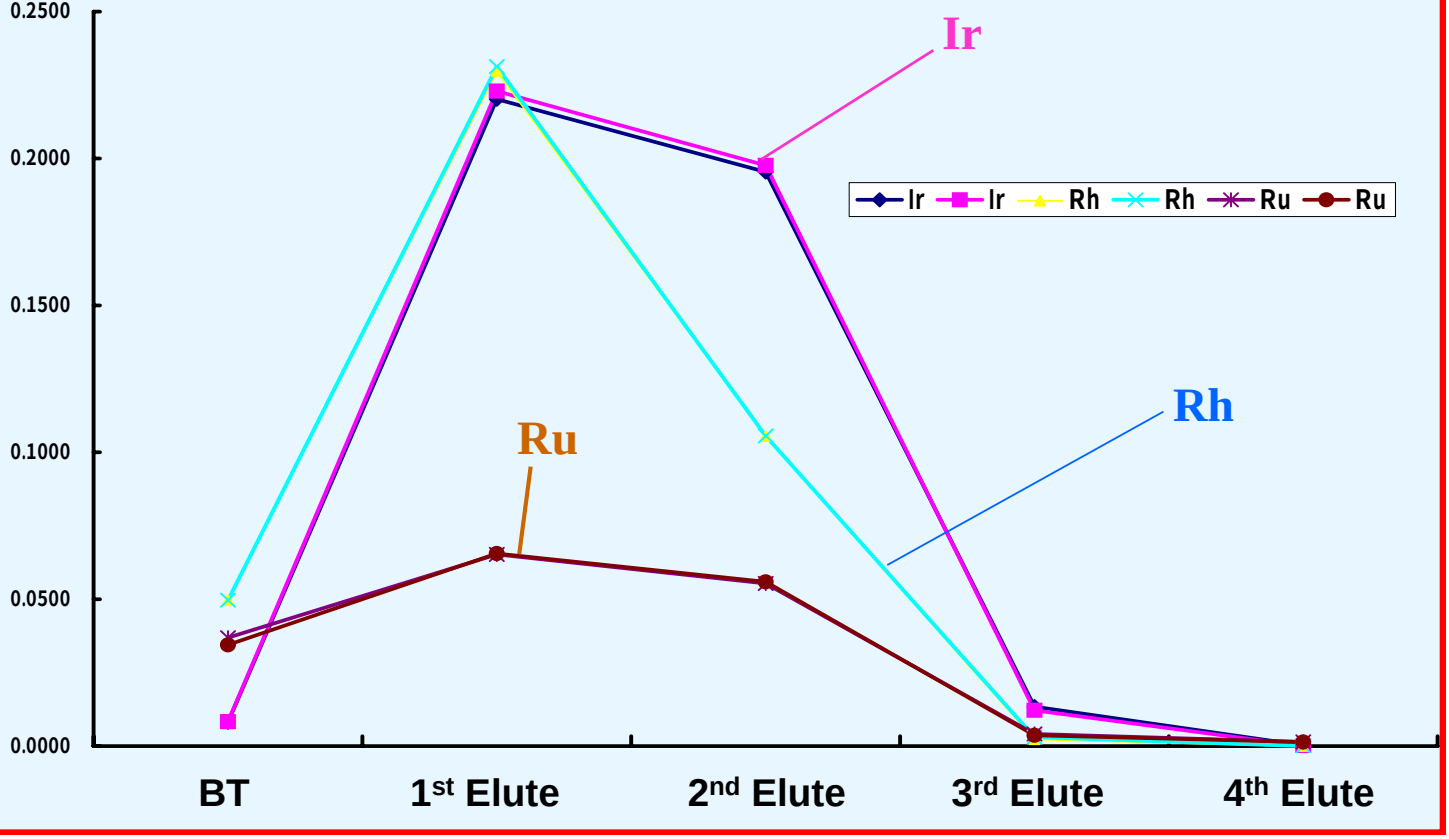
Eluted by 0.5M Thiourea/0.1 M HCl



Au 100%, Pd 90%, Pt 75% Recovery

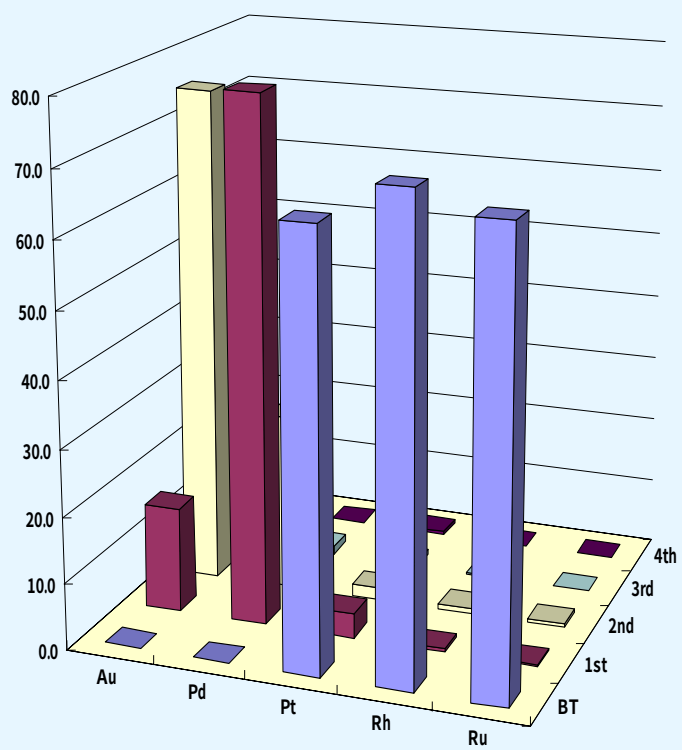
MetaSEP AnaLig® PM-01 for PGMs data

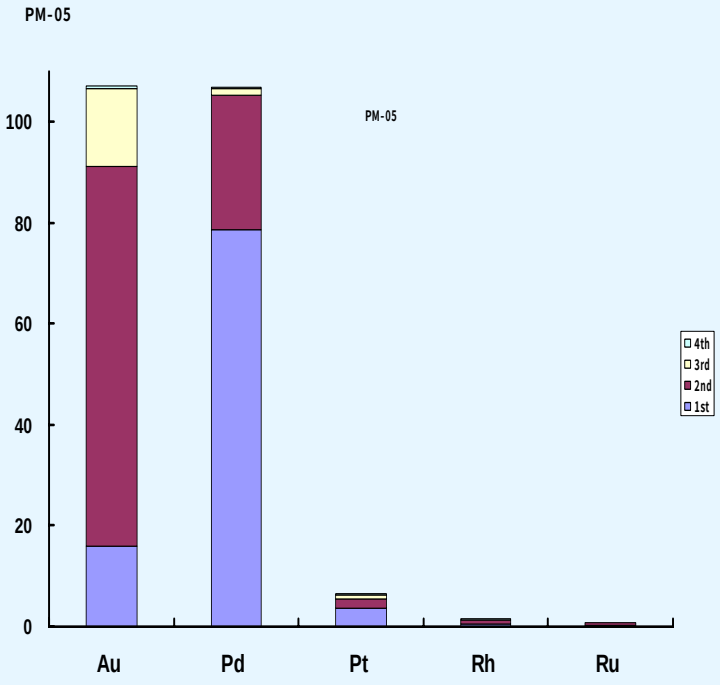
Eluted by 0.5M Ammonium chloride



MetaSEP AnaLig® PM-05 for PGMs

Eluted by 0.5M Thiourea/0.1 M HCl

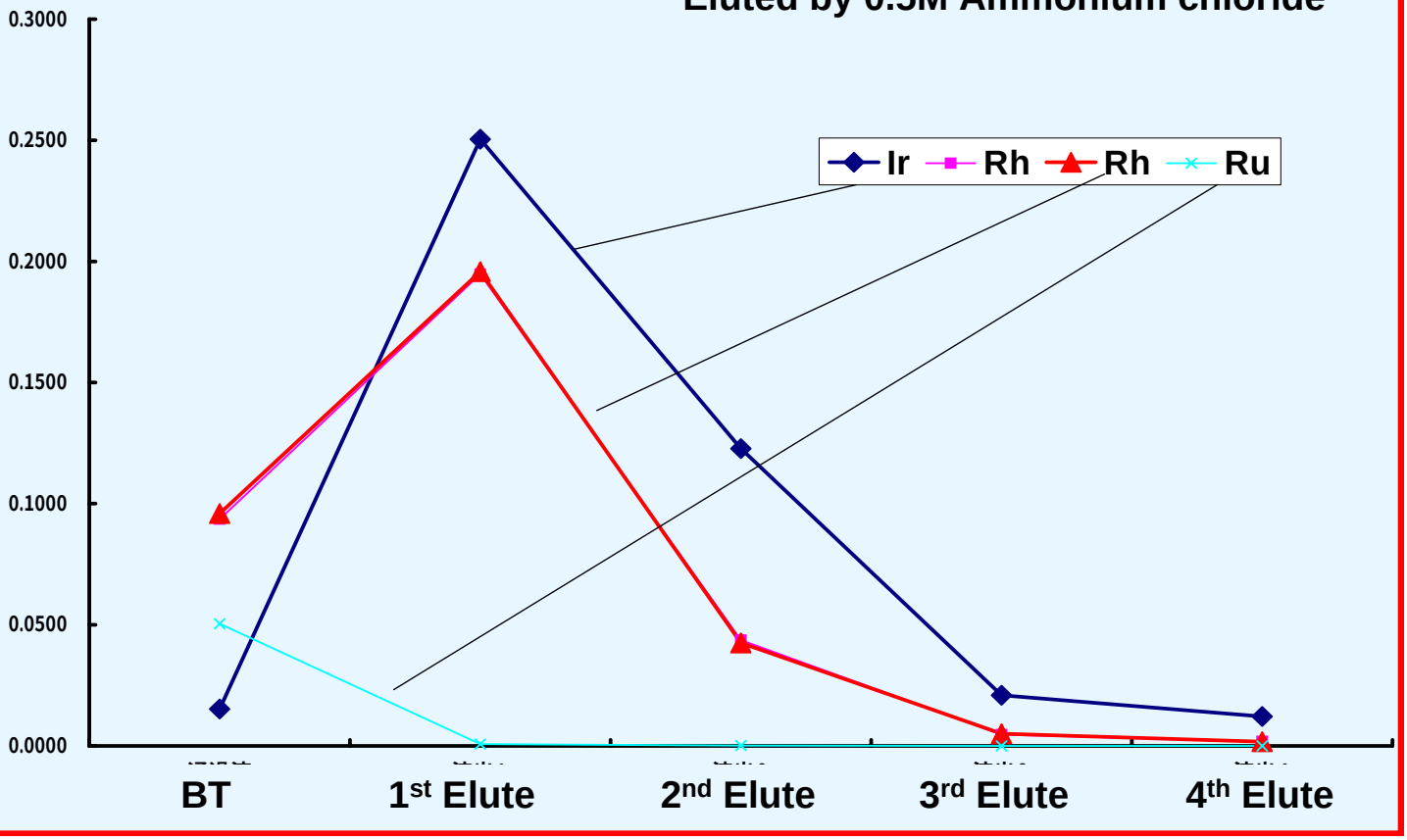




Au, Pd 100%recovery
Pt, Rh, Ru 60～70% break through

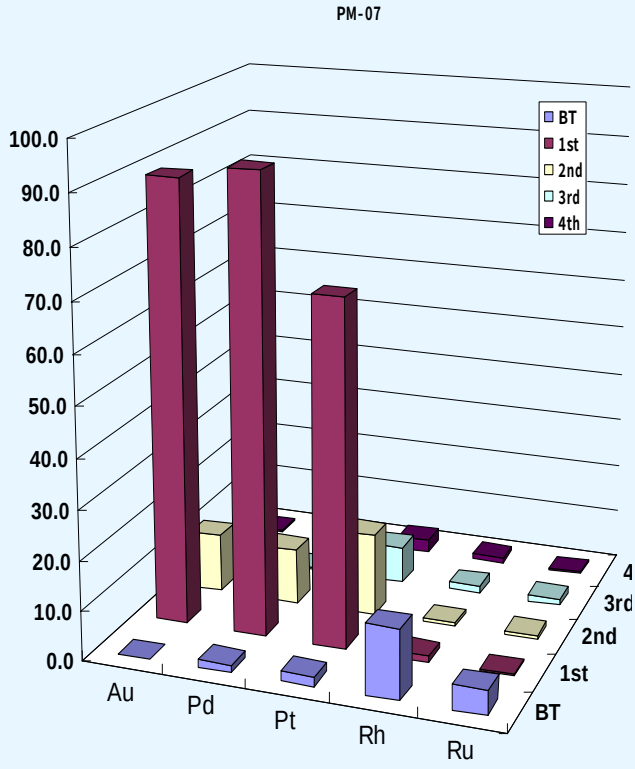
MetaSEP AnaLig® PM-05 for PGMs data

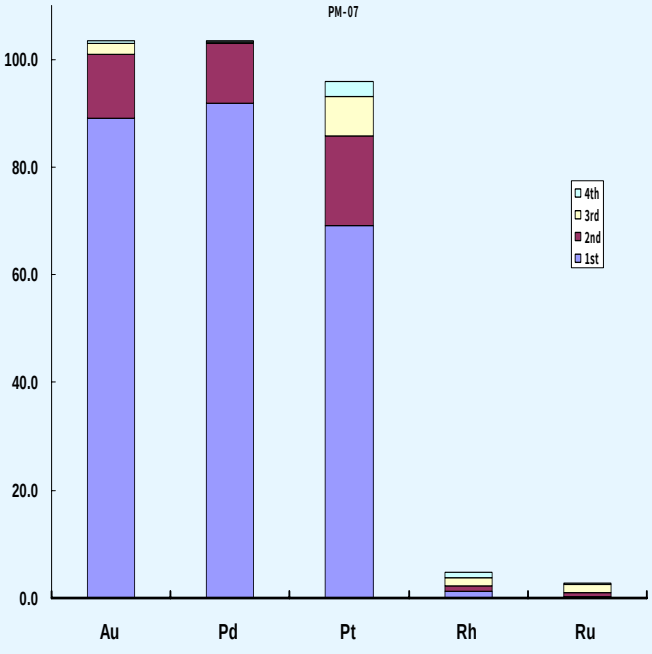
Eluted by 0.5M Ammonium chloride



MetaSEP AnaLig® PM-07 for PGMs

Eluted by 0.5M Thiourea/0.1 M HCl

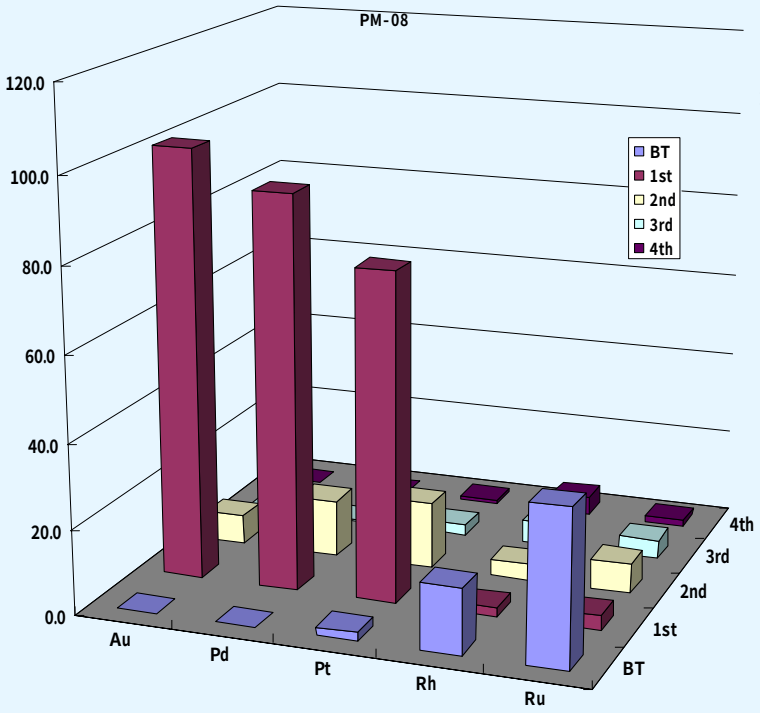


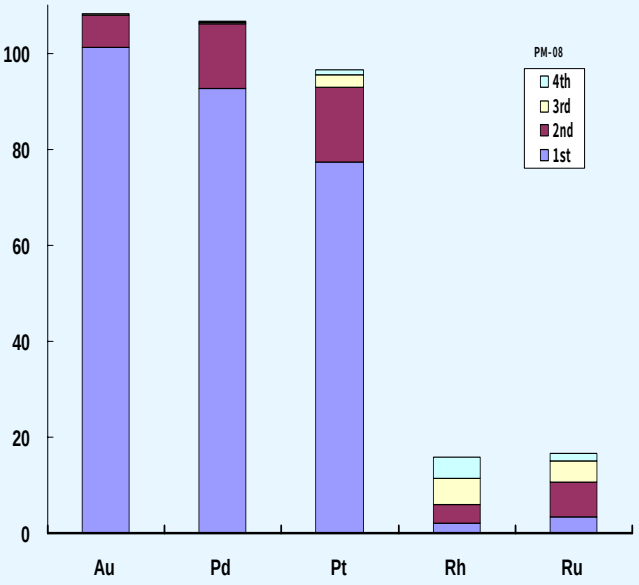


Au, Pd, Pt 90% recovery

MetaSEP AnaLig® PM-08 for PGMs

Eluted by 0.5M Thiourea/0.1 M HCl

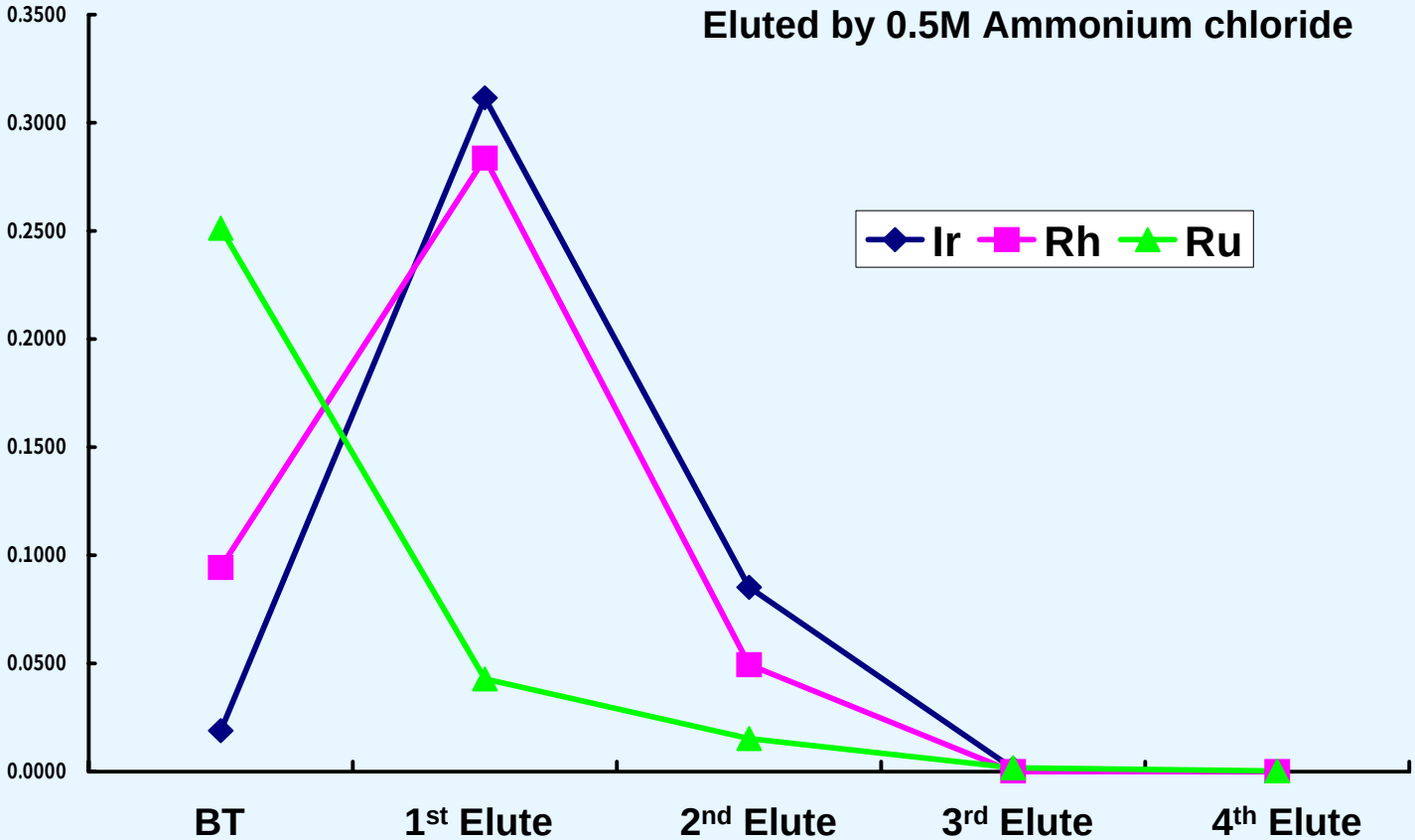




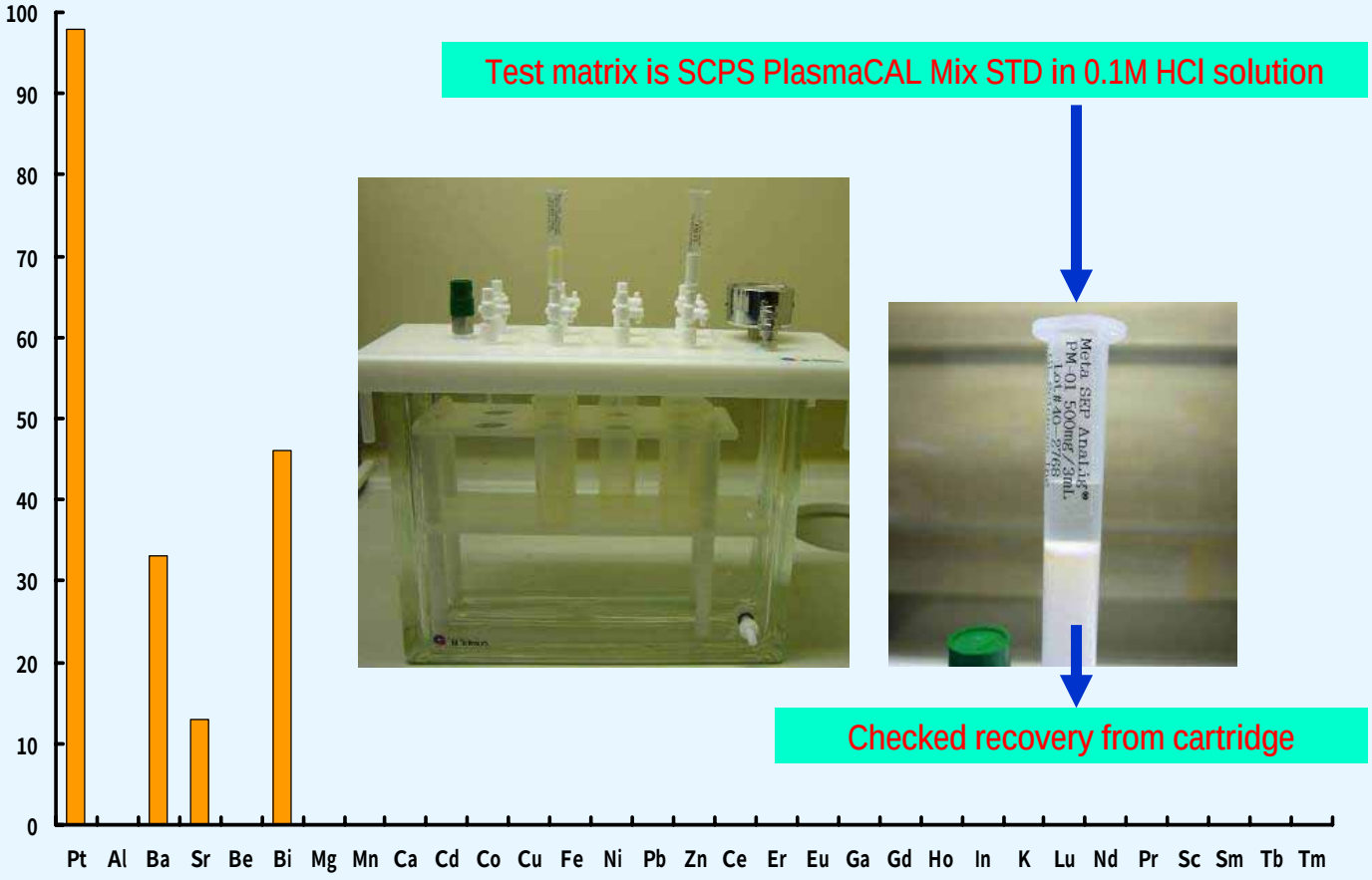
Au, Pd, Pt 90% recovery
Ru 30%、Rh 10% break through

MetaSEP AnaLig® PM-08 for PGMs data

Eluted by 0.5M Ammonium chloride



Element selectivity of AnaLig PM-08



MRT-SPE Columns scale up technology

Analytical SPE technique is useful for developing scale up column clean-up procedure in manufacturing PGMs from re-cycling solution.