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

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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

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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)?

1 — Atomic number  
H — Symbol  
1.01 — Atomic weight (rounded value)

IA																	Rea Metals						VIIIA
1	2																	10					
H	He																	Ne					
1.01	4.0																	20.2					
3	4																	5	6	7	8	9	10
Li	Be																	B	C	N	O	F	Ne
6.94	9.01																	10.8	12.0	14.0	16.0	19.0	20.2
11	12																	13	14	15	16	17	18
Na	Mg	IIIB	IVB	VB	VIB	VIIIB	VIII B		IB	IIB	III A	IV A	V A	VIA	VII A	He							
23.0	24.3										Al	Si	P	S	Cl	Ar							
23.0	24.3										27.0	28.1	31.0	32.1	35.5	39.9							
19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36						
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr						
39.1	40.1	45.0	47.9	50.9	52.0	54.9	55.8	58.9	58.7	63.5	65.4	69.7	72.6	74.9	79.0	79.9	83.8						
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54						
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe						
85.5	87.6	88.9	91.2	92.9	95.9	98.9	101.1	102.9	106.4	107.9	112.4	114.8	118.7	121.8	127.6	126.9	131.3						
55	56	57	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86						
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn						
132.9	137.3	138.9	168.5	180.9	183.9	186.2	190.2	192.2	195.1	197.0	200.6	204.4	207.2	209.0	(210)	(210)	(222)						
87	88	89	104	105	106	107	108	109	110	111	112												
Fr	Ra	Ac	Rf	Db	Sg	Bh	Hs	Mt															
(223)	226.0	(227)	(261)	(262)	(266)	(264)	(269)	(268)	(269)	(272)	(277)						(293)						
																	PGMs						
58	59	60	61	62	63	64	65	66	67	68	69	70	71										
Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu										
140.1	140.9	144.2	(145)	150.4	152.0	157.3	158.9	162.5	164.9	167.3	168.9	173.0	175.0										
90	91	92	93	94	95	96	97	98	99	100	101	102	103										
Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr										
232.0	(231)	238.0	(237)	(244)	(243)	(247)	(247)	(251)	(252)	(257)	(258)	(259)	(260)										

( ) represents an isotope

Rea Earth

# MRT-SPE Method for PGMs from High Matrix

## Typical current Technique

Open column with ion Ex resin for separation of PGMs

Anion Ex resin is not good for high selective separation from matrix

Not enough clean up for determination of PMGs by ICP-OES

Spectral Interference  
Physical Interference  
Chemical Interference

## Present method

Separate PGMs by MRT-SPE  
SPE cartridge is much easier handling than open column

MRT-SPE can separate target PGM element from main metal matrix

Reduce each interference

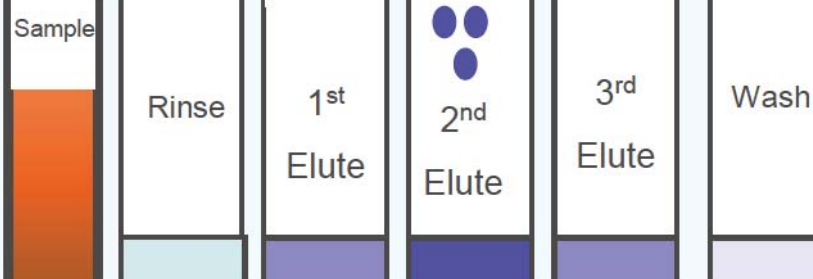
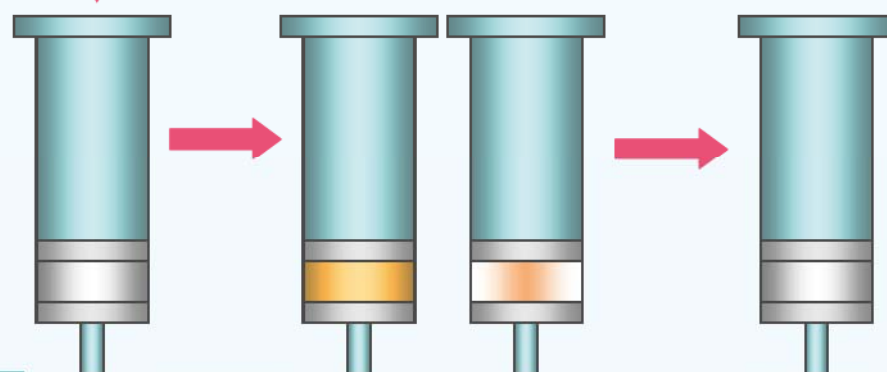
## MRT-SPE can separate PGMs



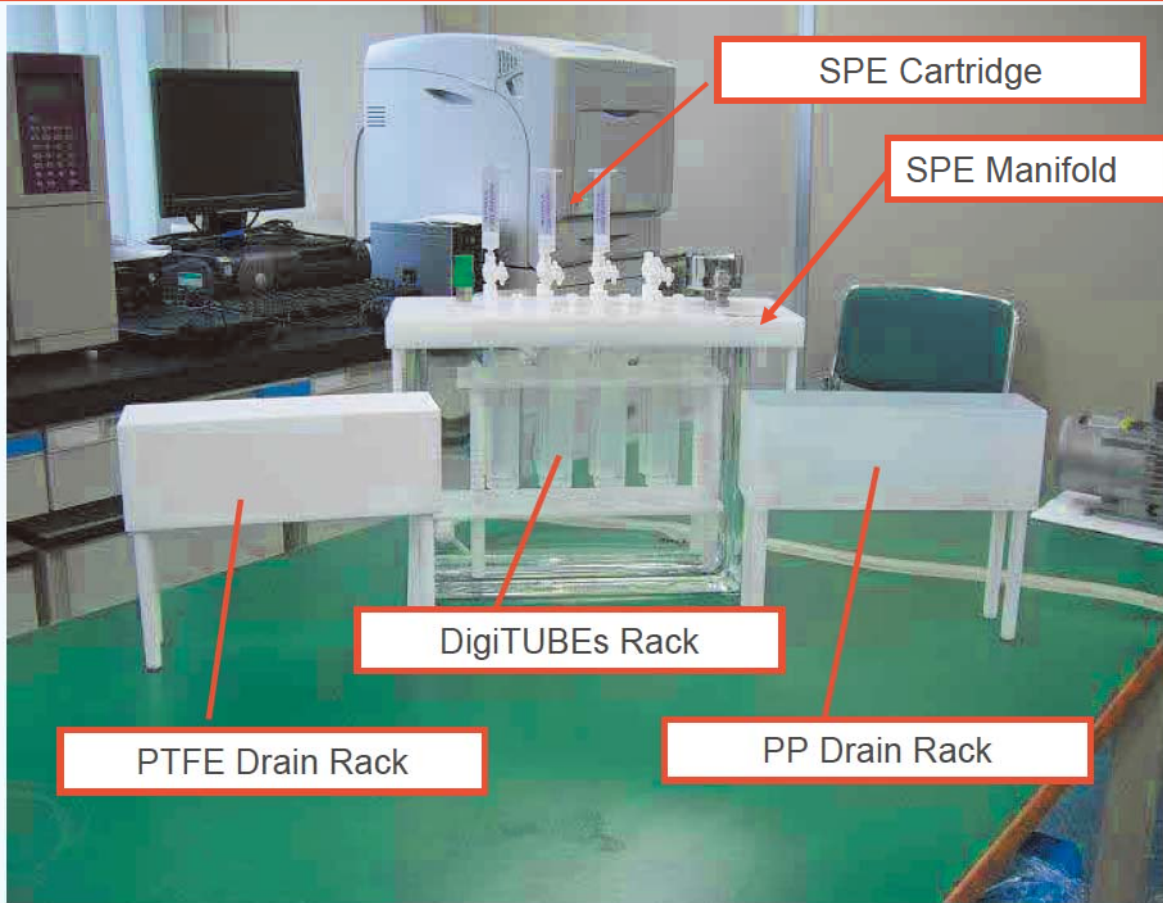
Open Column



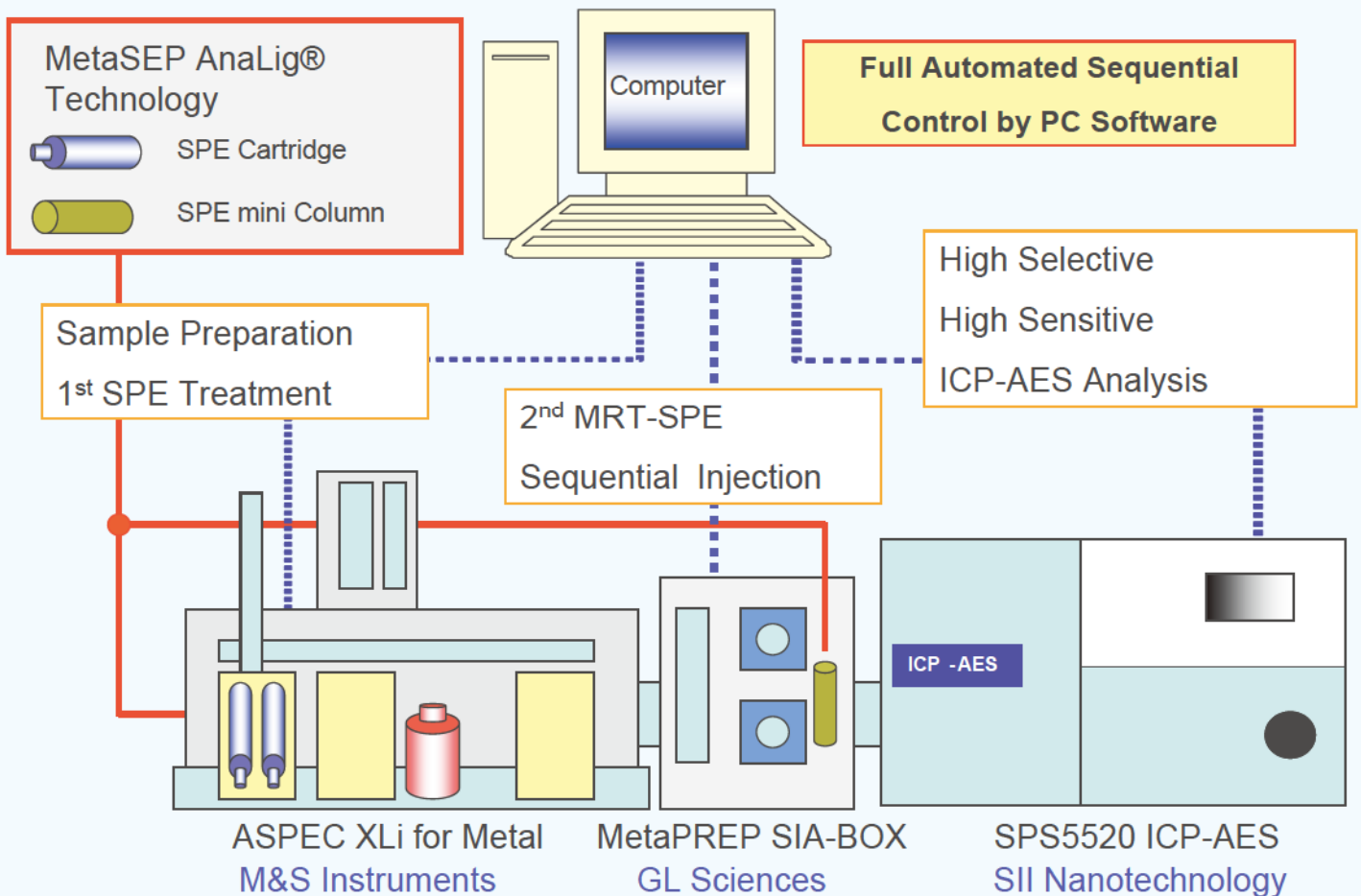
SPE

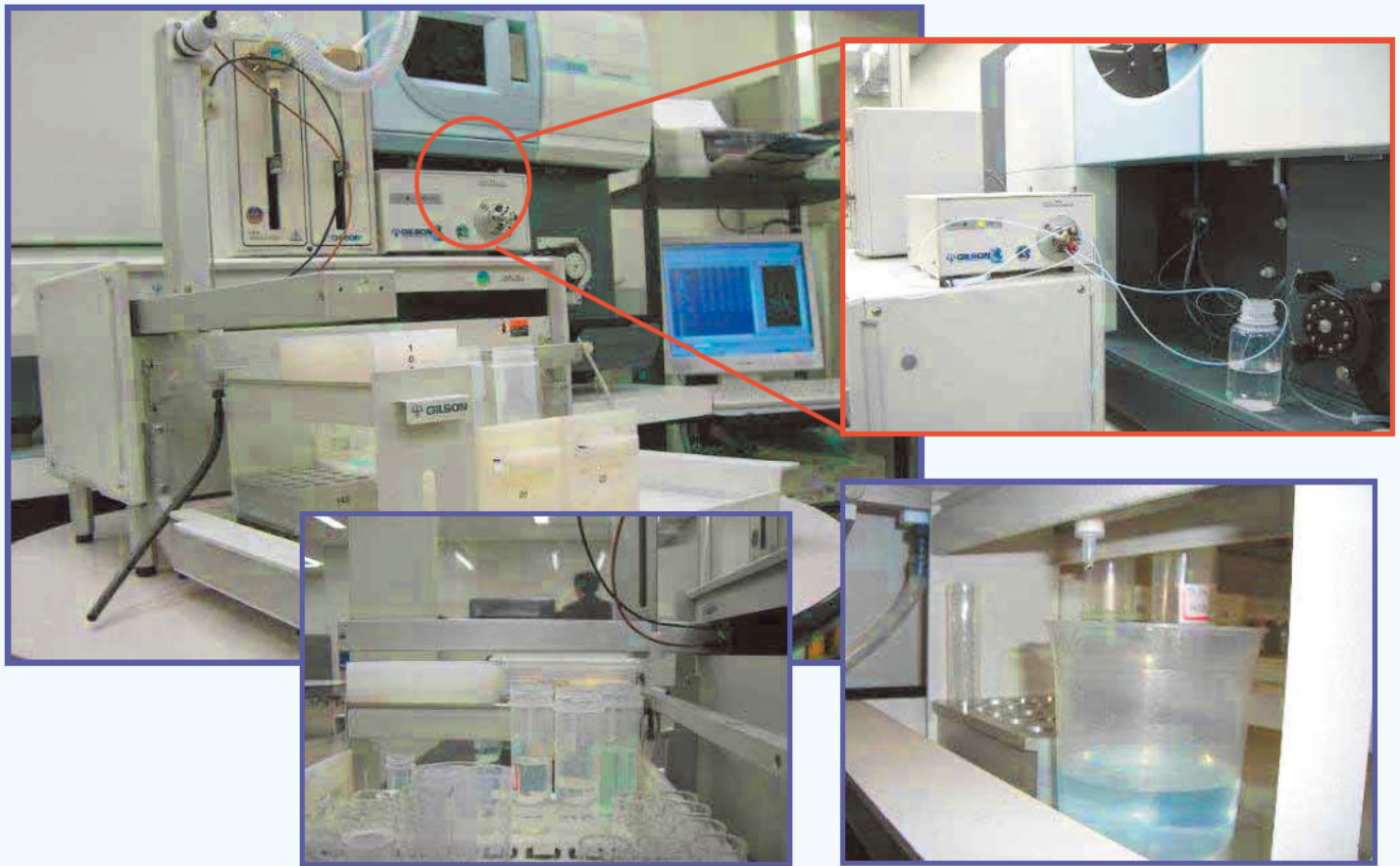


# SPE Manifold System for Metal Separation



## Evaluation System for Automation SPE Analysis





Method Development using Automated SPE system

## MetaSEP AnaLig® PM Series procedure for PGMs

**PM-01,05,07,08 SPE Cartridge**

**Sample** 4ml

0.5M thiourea/0.1MHCl 4ml

pH <1 by 0.1M HCl

Pure water 4ml × 3

Au, Pd, Pt, Rh, Ru

10ml/min

1ml/min

0.1M HCl溶液

**Loading**

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

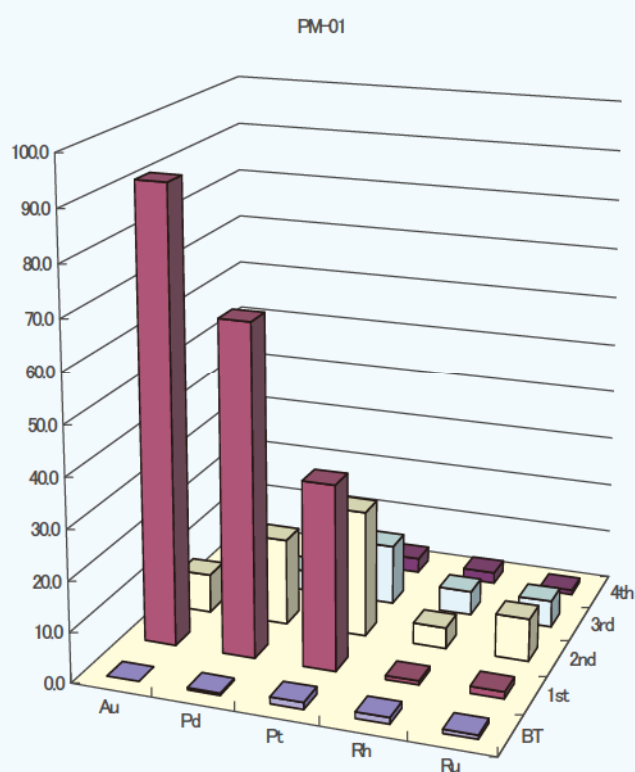
Cartridge size: 500mg/3mL

	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%

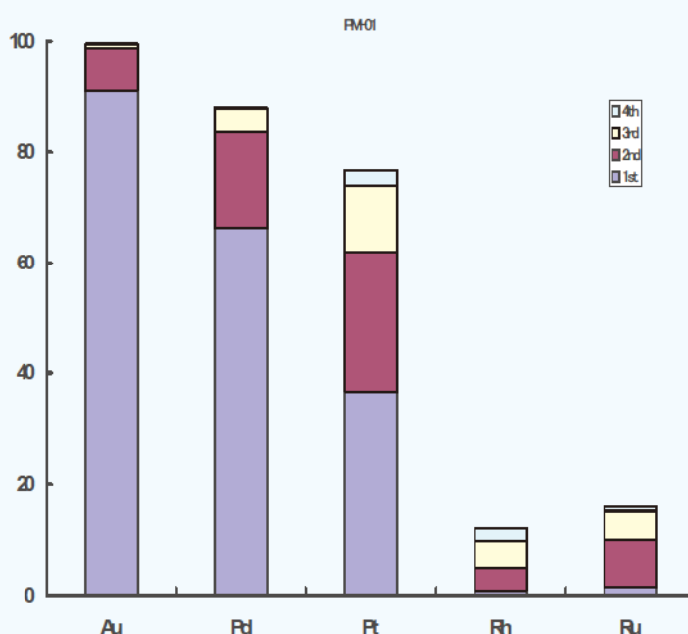
BT: Cartridge Break Through

EL: Elution Recovery from Cartridge

## MetaSEP AnaLig® PM-01 for PGMs data



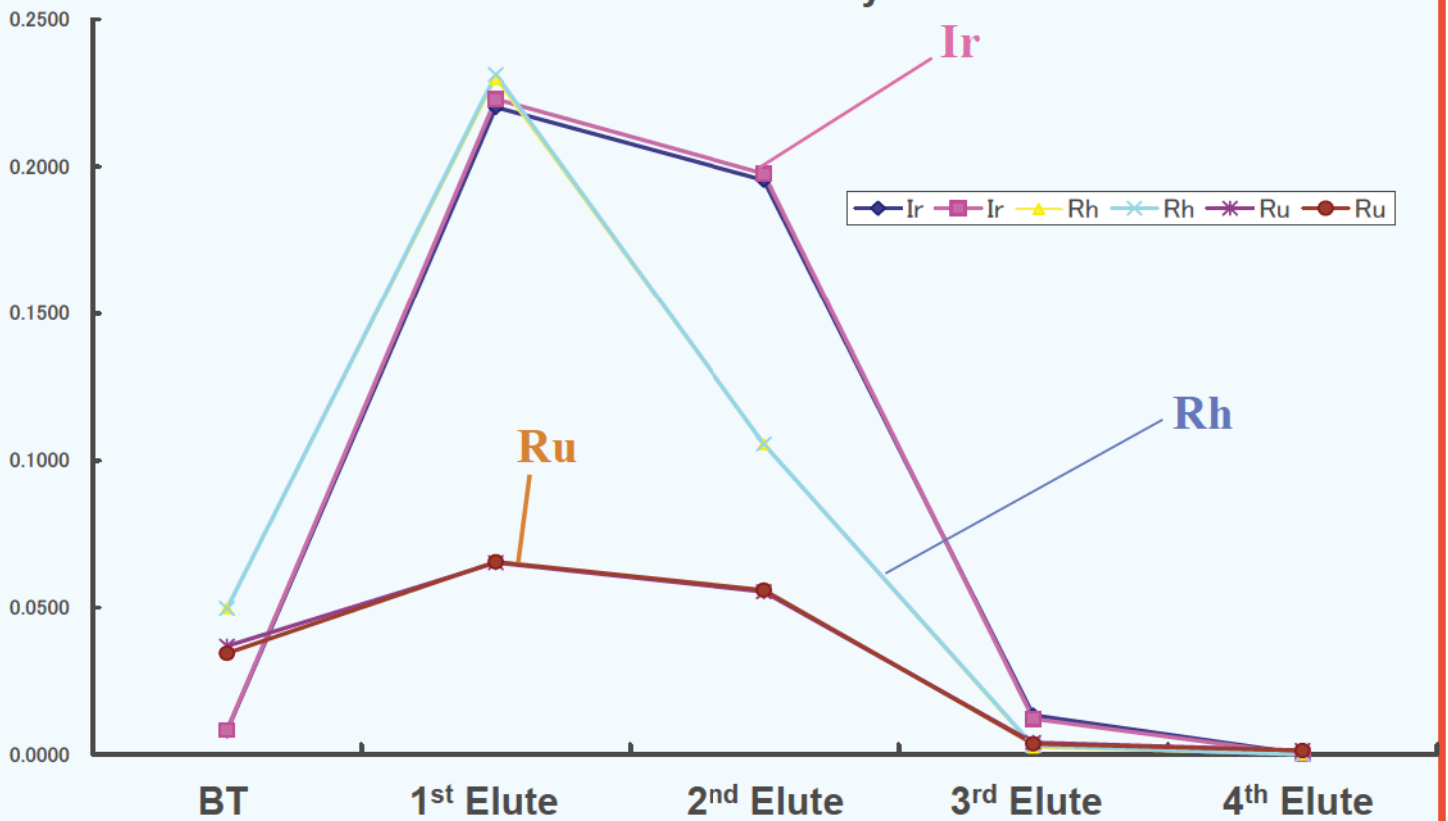
Eluted by 0.5M Thiourea/0.1 M HCl



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

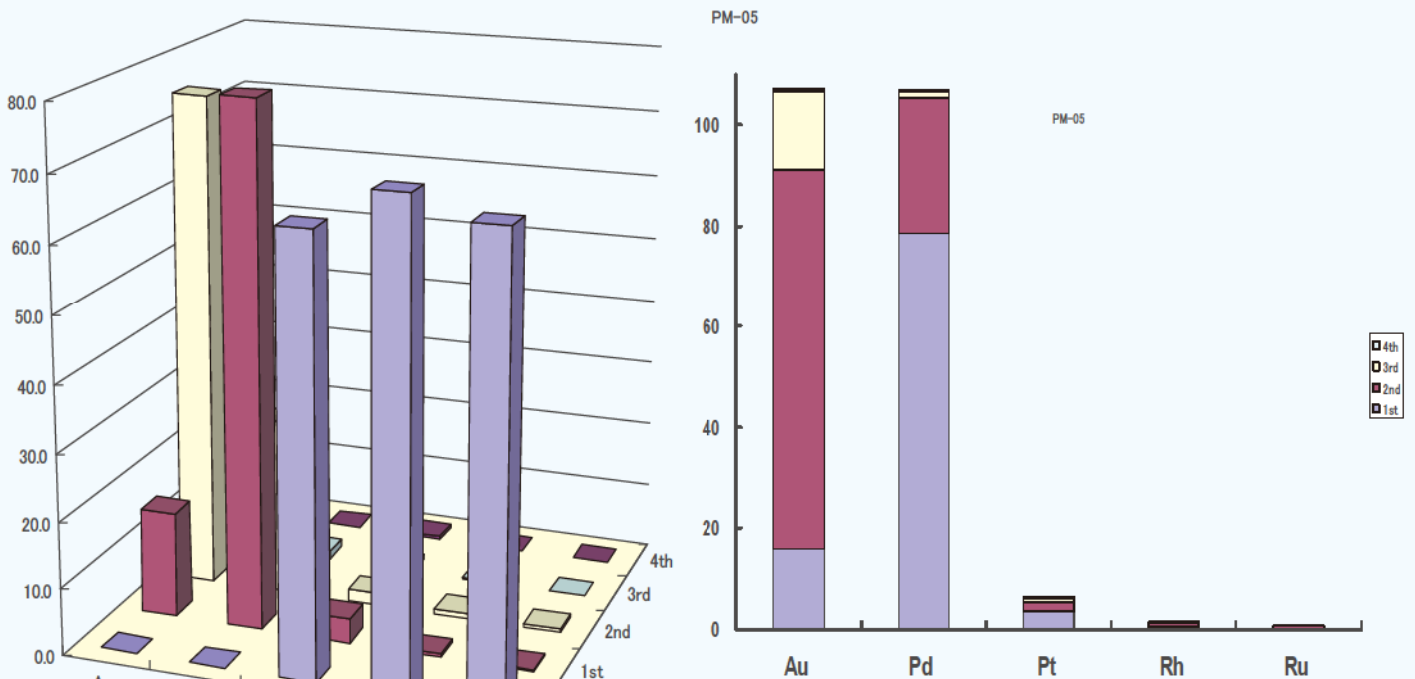
## MetaSEP AnaLig® PM-01 for PGMs data

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**

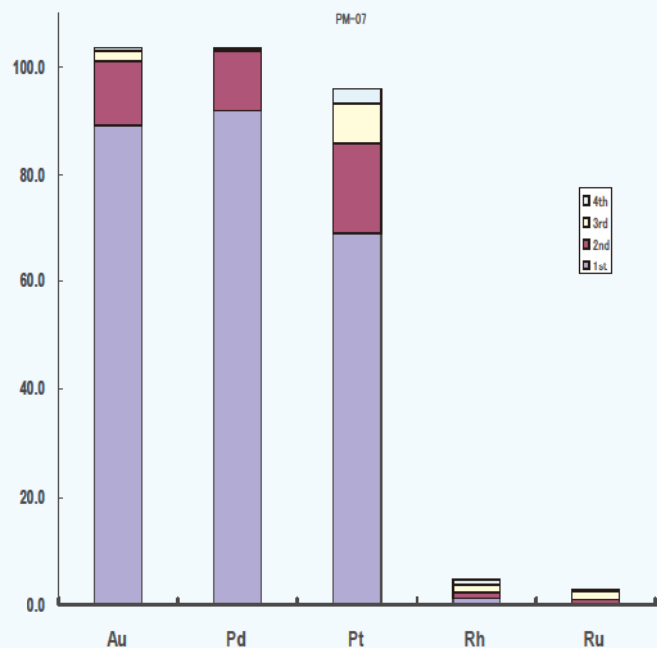
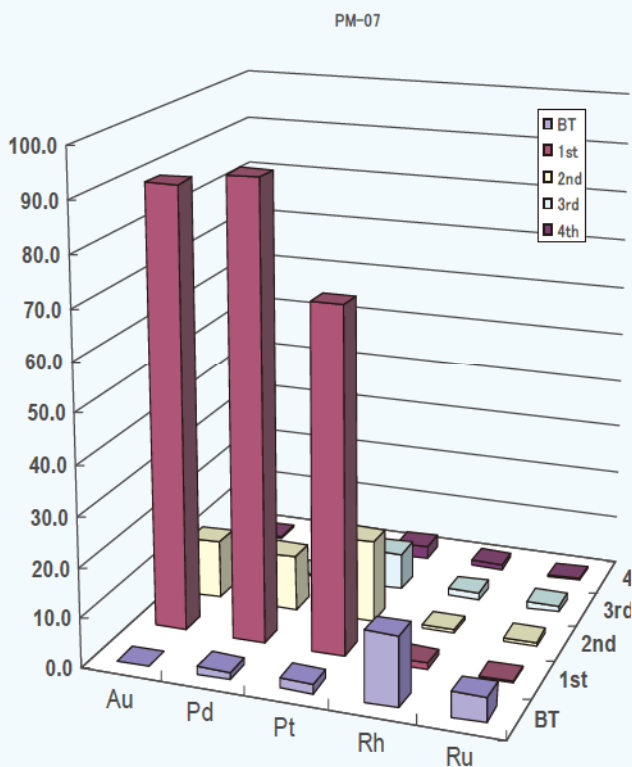
# MetaSEP AnaLig® PM-05 for PGMs data

Eluted by 0.5M Ammonium chloride



# MetaSEP AnaLig® PM-07 for PGMs

Eluted by 0.5M Thiourea/0.1 M HCl

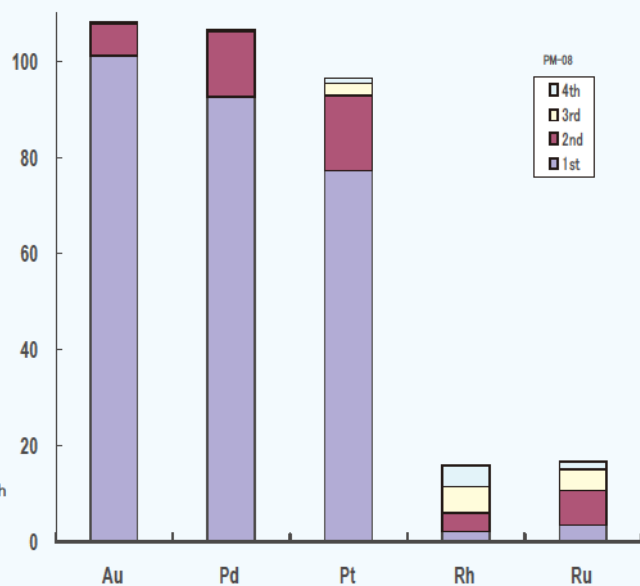
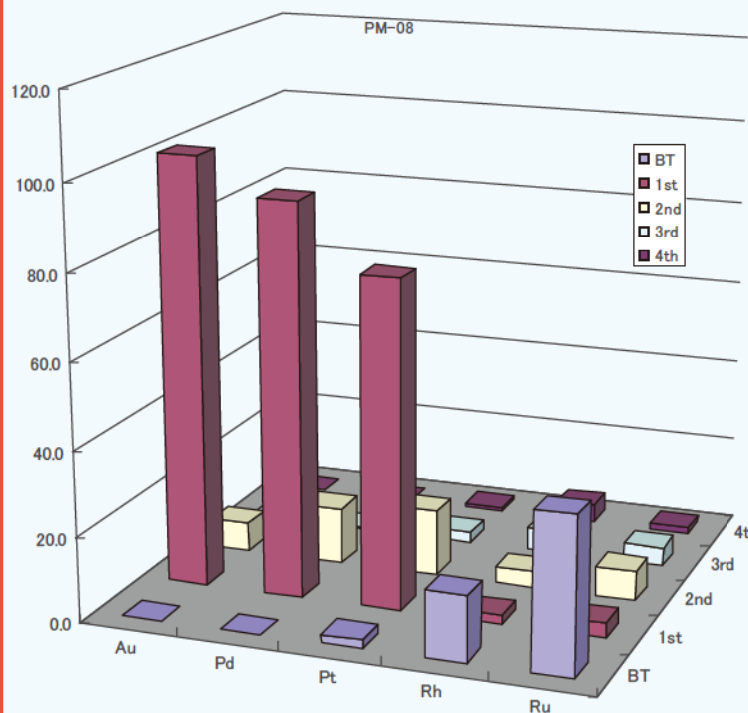


Au, Pd, Pt 90% recovery



# MetaSEP AnaLig® PM-08 for PGMs

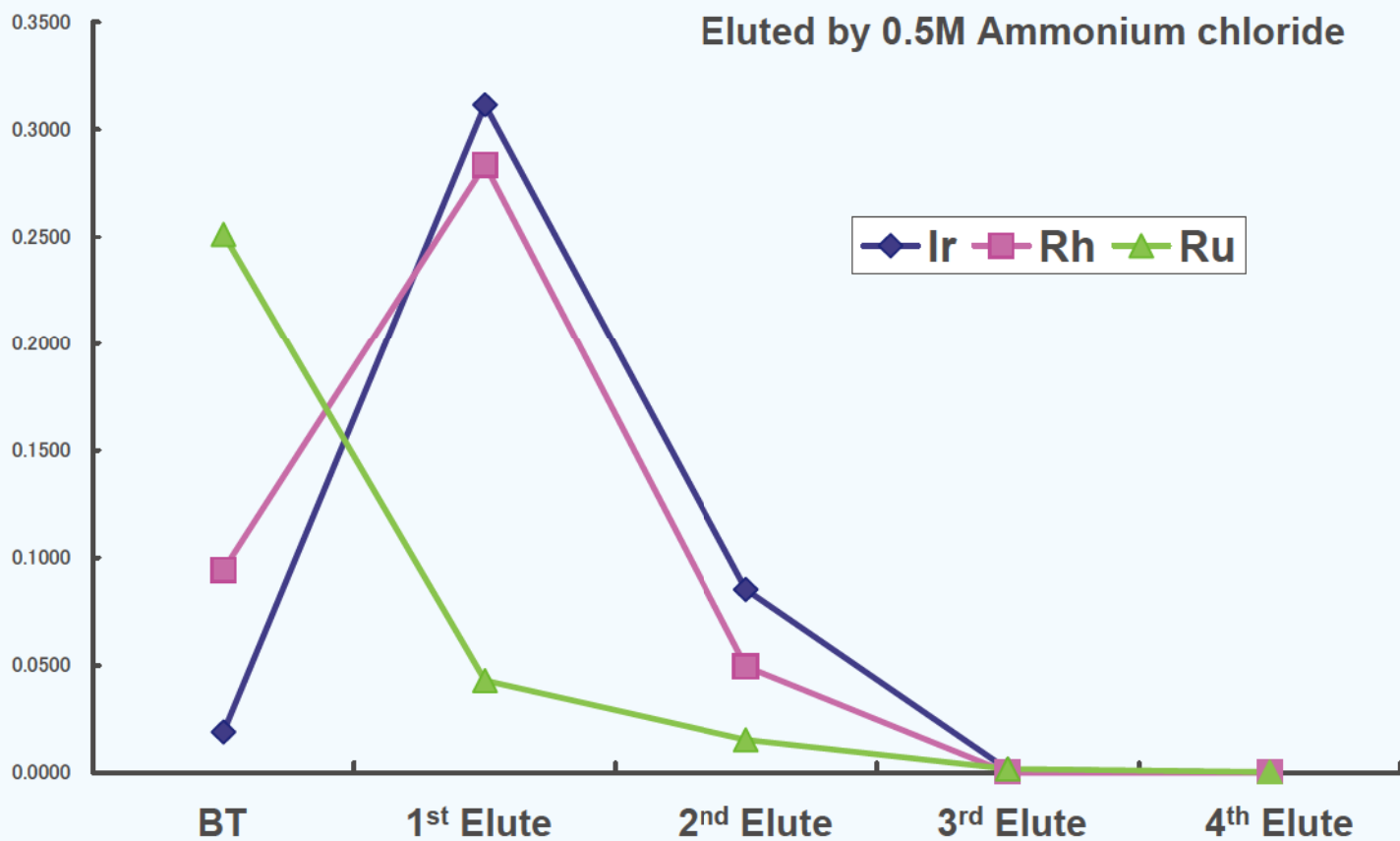
Eluted by 0.5M Thiourea/0.1 M HCl



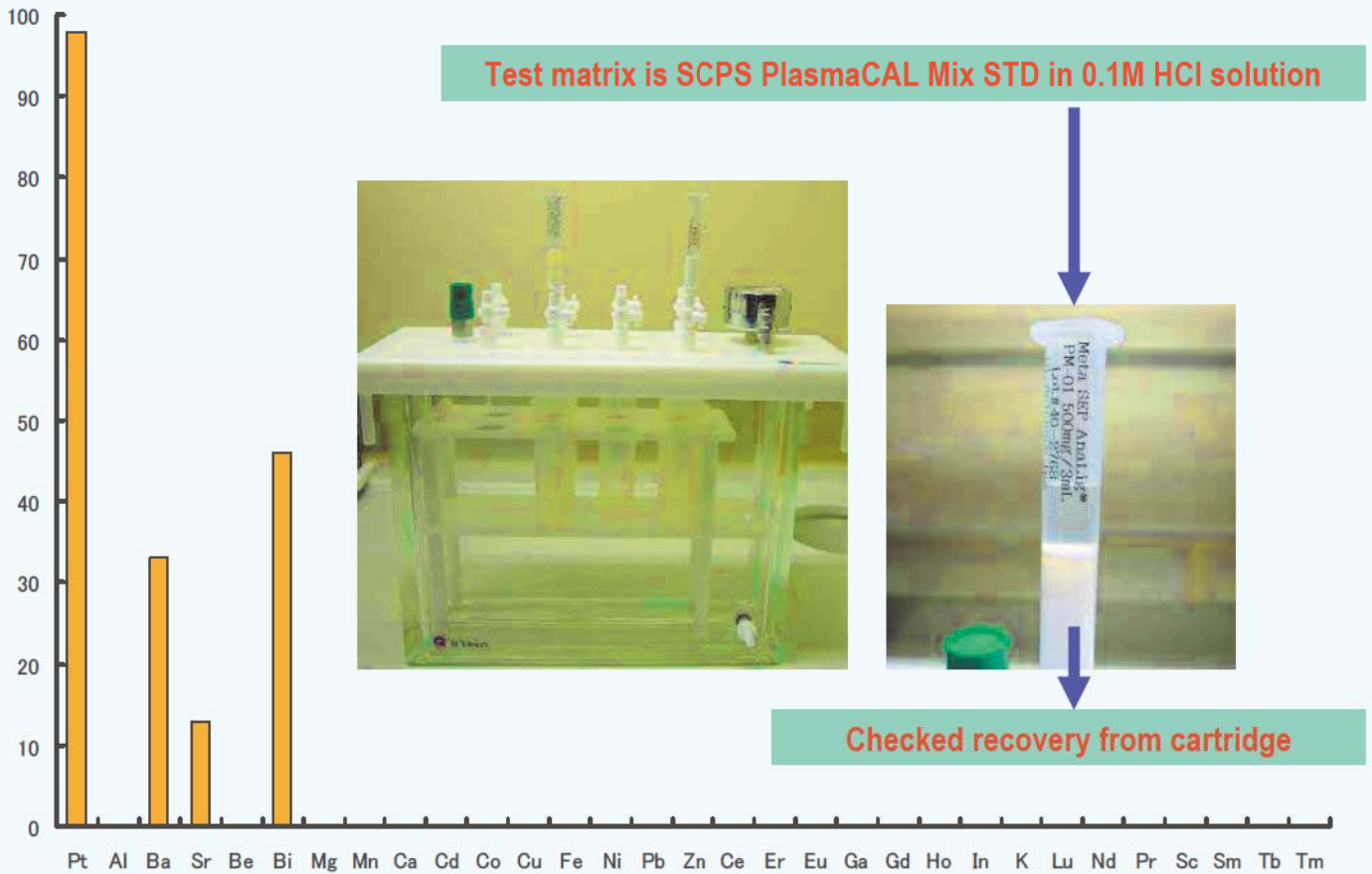
**Au, Pd, Pt 90% recovery**  
**Ru 30%, Rh 10% break through**

# 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.

