Use of ICP-Mass Spectrometry for Precious Metals

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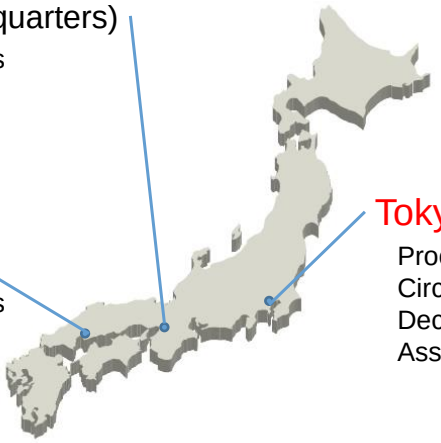
JAPAN MINT

Overview

- Introduction of Japan Mint
- Explanation of ICP-MS features
- Report of analysis for our refined silver with ICP-MS

Our Offices & Factories

Osaka (Headquarters)
- Circulation coins
- Proof coins
- Decorations
- Refining
- R & D (Assay)



Hiroshima
- Circulation coins
- Melting

Tokyo
- Proof coins
- Circulation coins
- Decorations
- Assay

Our Products & Business

Circulation Coins

Collector Coins (Proof coin sets)

Collector Coins (commemorative coins)

Gold Coin

Silver Coin with print

Bicolor- clad Coin

Decorations & Medals

Order of Sacred treasure

Gold Medal with hologram

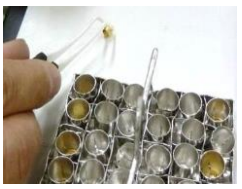
Our Products & Business

Refining

Gold bar

Hallmark

Assay



LBMA & GD-referees Visit

7th November 2012

Analysis for Precious Metals

Use	Material	Method	Remarks
Coin	Gold(≧999)	Fire assay	In case of ≧9999 : ICP-OES
	Silver(≧925)	Potentiometric Method	In case of ≧9999 : ICP-MS
Refined bullion	Gold(≧9999)	ICP-OES	
	Silver(≧9999)	ICP-MS	
Precious metal products for certification	Gold	Fire assay	
	Silver	Potentiometric Method	In case of ≧9999 : ICP-MS

ICP-Mass Spectrometer

Agilent Technologies 7700

Advantages

①high sensitivity (<ppt)
②high-matrix analysis
③wide dynamic range (9 orders)
④multi-elemental analysis

Disadvantages

①isobaric interferences
②polyatomic interferences
③ultra pure water and reagents

Detection Limits

ICP-MS

H																	He
Li	Be	<1ppt					<10ppt		<100ppt		<1ppb		B	C	N	O	F	Ne
Na	Mg												Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn		Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd		In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg		Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac																

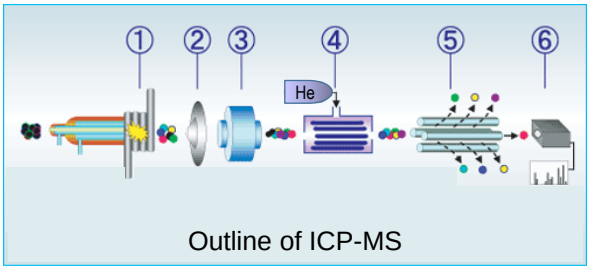
Agilent Technology

ICP-OES

H																	He
Li	Be	<1ppb			<10ppb			<100ppb			>100ppb		B	C	N	O	F	Ne
Na	Mg												Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn		Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd		In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg		Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac																

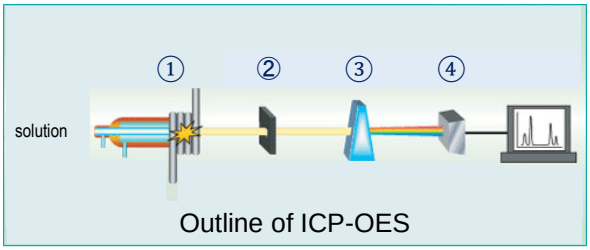
Foundation for Promotion of Material Science and Technology of Japan

Structure



Outline of ICP-MS

①ICP
②interface
③ion lens
④CRC
Collision Reaction Cell
⑤mass spectrometer
⑥detector



Outline of ICP-OES

①ICP
②slit
③spectroscope
④detector

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Isobaric Interferences

Relative Isotopic Abundance Table

	mass number											(%)
	55	56	57	58	59	60	61	62	63	64	65	66
Mn	100											
Fe		91.75	2.12	0.28								
Co					100							
Ni				68.08		26.22	1.14	3.63		0.93		
Cu									69.15		30.85	
Zn										48.27		27.98

Recommended mass number

Agilent Technologies Inc. 2009

Selected mass number for analysis

Ni ・・・ 58 (68.08%) → 60 (26.22%)

Zn ・・・ 64 (48.27%) → 66 (27.98%)

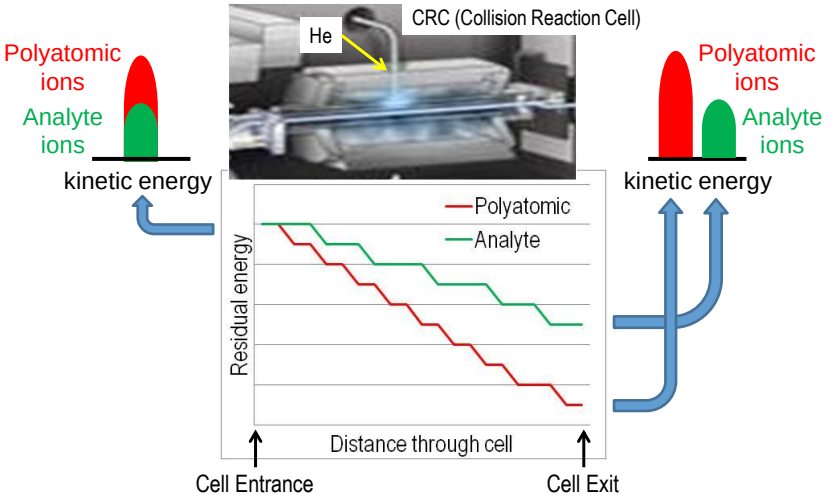
Polyatomic Interferences

	Mass number	Abundance (%)	Polyatomic ion	Detection limit (ppt)			remarks
				Normal plasma	Cool plasma	CRC	
K	39	93.26	$^{38}Ar^{1}H$	5000	2	0.3	
Ca	40	96.941	^{40}Ar	no	6	1.3	○
	44	2.086	–	1000	–	30	※
Mn	55	100	$^{40}Ar^{14}N^{1}H$	5	1.3	0.6	
Fe	54	5.85	$^{40}Ar^{14}N$	560	–	–	
	56	91.75	$^{40}Ar^{16}O$	4000	15	2.5	○
	57	2.12	–	250	–	–	※
Ni	58	68.08	$^{40}Ar^{18}O$	130	–	–	
	60	26.22	–	2	1.7	4	※○
Co	59	100	$^{40}Ar^{18}O^{1}H$	0.5	0.3	0.3	

Selection ・・・※ old equipment (with only normal plasma), ○ new one (with CRC)

Polyatomic Interference Removal

using He mode and CRC



Analysis for Refined Ag with ICP-MS

Sample preparation

ICP-MS

Weighing 100mg

Disposable bottle

Dissolution in HNO_3 5ml

for precision analysis

Making up to 50ml

Ultrapure water

ICP-OES

Weighing

Dissolution in HNO_3

Addition HCl

Precipitation of AgCl

Filtration of AgCl

Making up

Advantages of ICP-MS

1.Avoiding impurities adsorption on AgCl

2.Minimizing contaminations after dissolution

3.Time saving for precipitation and filtration

Analysis for Refined Ag with ICP-MS

element	mass number
Al	27
Bi	209
Cd	111
Cu	63
Fe	56
Mg	24
Mn	55
Ni	60
Pb	208
Pd	105
Sb	121
Sn	118
Zn	66

Qualitative analysis to determine impurities elements with DC-Arc-OES

↓

Calibration curves for each element with Ag matrix and Y internal standard

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Quantitative analysis of impurities in samples with ICP-MS

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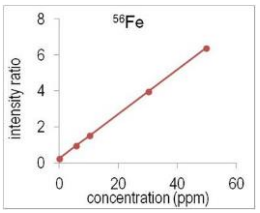
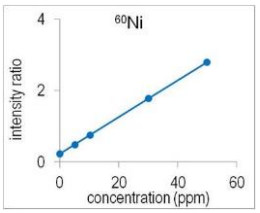
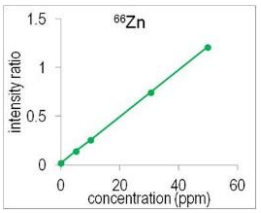
Fineness (‰) = subtraction of the total content of impurities from 1000

Results for Refined Ag with ICP-MS

Calibration curves







	Concentration of impurities (ppm)															Ag	
No.	Mg	Al	Mn	Fe	Ni	Cu	Zn	Pd	Cd	Sn	Sb	Pb	Bi	total ppm	total ‰	‰	Ave. ‰
1-1	<0.000	<0.000	0.113	<0.000	0.390	2.700	<0.000	0.927	0.000	<0.000	<0.000	0.148	0.091	4.369	0.004	999.996	999.996
1-2	<0.000	<0.000	0.119	<0.000	0.349	3.124	<0.000	0.880	0.002	<0.000	<0.000	0.150	0.092	4.716	0.005	999.995	
2-1	<0.000	<0.000	0.275	0.954	<0.000	1.883	<0.000	5.385	0.001	<0.000	<0.000	<0.000	0.983	9.481	0.009	999.991	999.991
2-2	<0.000	<0.000	0.274	1.085	<0.000	1.925	<0.000	4.710	<0.000	<0.000	<0.000	<0.000	0.988	8.982	0.009	999.991	

Conclusions

1. We introduced the features of ICP-MS .
   - ICP-MS has higher sensitivity than ICP-OES.
   - ICP-MS has isobaric interference and polyatomic interference.
   - It is important to eliminate interferences for analysis of the precious metal using ICP-MS.
2. We introduced the analysis of refined silver with ICP-MS.
   - ICP-MS is very useful to analyze our refined silver >999.99 by recent technological advances.

Thank you for your attention

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