Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Tutorial

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Principle

The working principle of ICP-MS is illustrated in Fig. 1. The liquid sample is first introduced into the nebulizer, where it is converted into an aerosol and carried into the plasma torch. In the torch, an inductively coupled plasma is generated, which atomizes the elements and ionizes them into free ions. These ions are then extracted through a sampling interface and guided by an ion lens system, which focuses and adjusts their trajectory. The ions then enter the quadrupole mass filter, where they are separated according to their mass-to-charge ratio ($\frac{m}{q}$). The selected ions pass through the quadrupole and are detected by a detector, where the signal is amplified and measured.

The output from the instrument is given as counts per second (cps). To determine the concentration of elements in the original liquid sample, the measured cps must be compared to a reference line, which is calibrated using standard solutions with known concentrations.

This technique allows for parts per trillion (ppt)-level detection sensitivity.

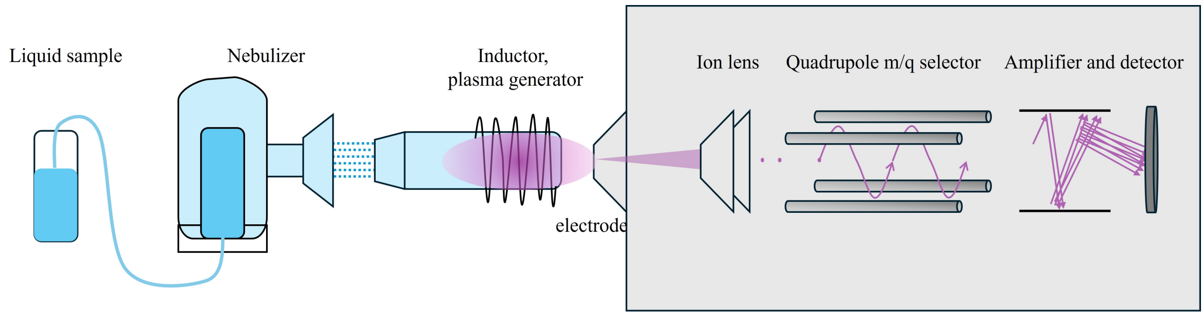

Figure 1: The schematic of ICP-MS equipment.

Process

Sequence	Sample	Annotation
1	1% HNO_3	washing, CPS data not recorded
2	1% HNO_3	washing, CPS data not recorded
3	UPW	washing, CPS data recorded
4	1% HNO_3	ref point 1, blank data (0,0)
5	100 ppt	ref point 2
6	200 ppt	ref point 3
7	500 ppt	ref point 4
8	1000 ppt	ref point 5
9	UPW	washing before QC
10	QC1	quality control
11	SAMPLE	the real sample to be measured
12	UPW	washing, CPS data recorded
13	UPW	washing, CPS data recorded
14	UPW	washing, CPS data recorded

Table 1: The experiment process

Reference line

In this section, we will show how the reference line is built by taking Mg^{2+} as an example. TABLE 2 shows the data for generating the reference line of Mg^{2+}. ReferLine ID are the designed concentration (unit ppt) level; Weight conc. are the actual concentration by weight, which depends on the accuracy of the weighing machine; CPS are the detected flux (count per second) of the samples; Regression conc. values are obtained by inputting the five ”y” CPS data points as the independent variable, resulting in the ”x” outcomes.

ReferLine ID	weight conc.(ppt)	CPS	regress conc.(ppt)
0	0	3.67	-1.743352949
100	101.1570672	4548.38	102.7007552
200	170.7122409	7597.66	172.7776864
500	504.8849092	21927.85	502.1064916
1000	979.6110274	42745.37	980.5236645

Table 2: The data for generating the reference line.

The parameters for regression equation $y = ax + b$ is given in TABLE3.

a	43.51332
b	79.52908
Correlation Coefficient	0.999986

Table 3: Linear Regression Parameters

FIGURE 2 is the plot of the reference line of Mg^{2+} ions. The Reference Line (blue line), represents the linear regression derived from the five data points indicating weight concentration (blue dots). The regression concentration values (red squares) are obtained by inputting the five "y" CPS data points as the independent variable, resulting in the "x" outcomes. This linear regression serves as a guide, illustrating the relationship between weight concentration and CPS data with precision.

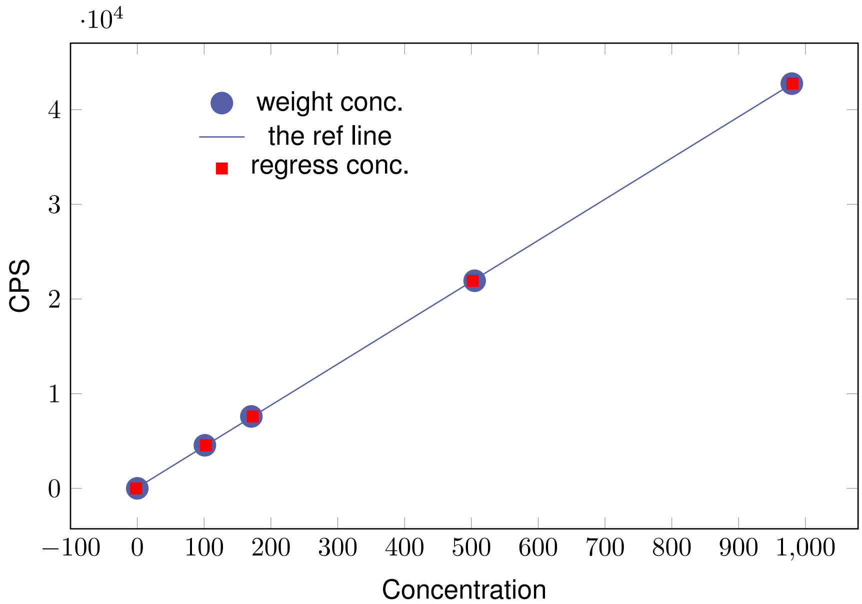

Figure 2: The Reference Line (blue line), the weight concentration (blue dots) and the regression concentration values (red squares).

Quality Control (QC)

Quality Control (QC) is a pre-test before measuring real samples. The QC sample concentration should be within the reference range (i.e., 0 to 1000 ppt). Using the measured CPS in the regression equation gives us the regression concentration. If the difference between the regression and weight concentrations is less than 0.2 (20%), the reference line can serve as a reliable guide. Here, the deviation is 0.0153, for Mg^{2+}.

weight conc.(ppt)	CPS	regress conc.(ppt)	deviation
604.8263382	26800.32	614.0830057	0.01530467

Table 4: The QC results of Mg^{2+}.

Results

The step 11 is the real sample in this measurement. The measured CPS of Mg^{2+} in the sample is 11.33, which is the average value of 3 repeat recording results: 13, 10 and 11. Put the value 11.33 back to the regression equation $y = ax + b$, we can get its regress concentration: 0.176 ppt. Using the same method, the concentration of other elements are obtained, as shown in TABLE 5.

Element	Regress Conc. (ppt)	Element	Regress Conc. (ppt)
7 Li [Warm H2]	-0.022	89 Y [HEHe]	-0.026
9 Be [No Gas]	-0.204	90 Zr [He]	2.743
11 B [No Gas]	16.336	93 Nb [HEHe]	0.297
23 Na [Warm H2]	1.157	95 Mo [HEHe]	0.411
24 Mg [Warm H2]	0.176	105 Pd [HEHe]	0.204
27 Al [Warm H2]	0.4	105 Pd [He]	0.078
39 K [Warm H2]	0.921	107 Ag [He]	1.658
40 Ca [Warm H2]	3.779	111 Cd [HEHe]	-0.09
48 Ti [HEHe]	0.165	115 In [HEHe]	1.605
51 V [He]	-0.04	118 Sn [Warm H2]	1.377
52 Cr [Warm H2]	0.111	118 Sn [HEHe]	0.729
55 Mn [Warm H2]	0.1	121 Sb [HEHe]	0.728
56 Fe [Warm H2]	0.59	138 Ba [HEHe]	-0.029
58 Ni [Warm H2]	-0.712	181 Ta [HEHe]	0.099
59 Co [Warm H2]	0.07	182 W [He]	0.68
63 Cu [Warm H2]	0.224	195 Pt [No Gas]	-6.325
64 Zn [He]	0.589	197 Au [He]	4.421
71 Ga [Warm H2]	0.035	201 Hg [HEHe]	1.204
72 Ge [HEHe]	0.624	205 Tl [He]	0.732
75 As [He]	-0.294	208 Pb [HEHe]	7.721
88 Sr [HEHe]	0	209 Bi [He]	1.195

Table 5: The concentration of elements

In this table, the "Element" column includes the tested isotope mass of the element, the element label, and the reative gas. The reactive gases are used to resolve interferences, leading to lower detection limits and better accuracy for some difficult analytes.

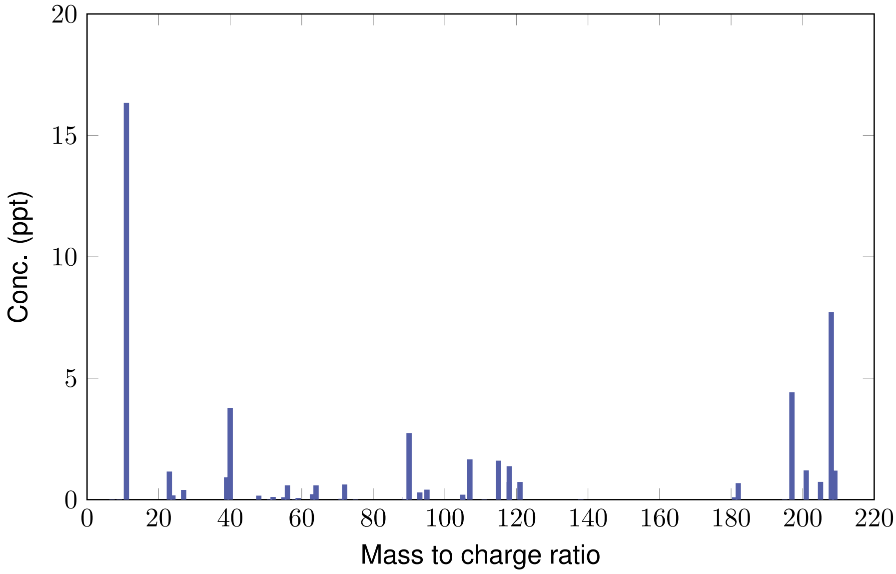

Figure 3: The mass spectrum

As a mass spectrometric technique, ICP-MS can also separate and measure the individual isotopes of an element, so it can be used for applications where isotopic abundances or isotope ratios are of interest. "FIGURE 3 depicts a "mass spectrum", providing a concise representation of element concentrations at various mass-to-charge ratios. The isolated 'peaks' in the plot correspond to distinct elements or isotopes."

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Appendix-Relative Isotopic Abundance Table

Relative Isotopic Abundance Table

(unit: %)

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	
H	99.99	0.011															H
He				100													He
Li						7.59	92.41										Li
Be									100								Be
B										19.9	80.1						B
C												98.93	1.07				C
N														99.63	0.368		N
O																99.76	O

	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	
O	0.038	0.205															O
F			100														F
Ne				90.48	0.27	9.25											Ne
Na							100										Na
Mg								78.99	10.00	11.01							Mg
Al											100						Al
Si												92.23	4.683	3.087			Si
P															100		P
S																94.93	S

	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	
S	0.76	4.29		0.02													S
Cl			75.78		24.22												Cl
Ar				0.337		0.063		99.60									Ar
K							93.26	0.012	6.730								K
Ca								96.94		0.647	0.135	2.086		0.004		0.187	Ca
Sc													100				Sc
Ti														8.25	7.44	73.72	Ti

	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	
Ti	5.41	5.18															Ti
V		0.250	99.75														V
Cr		4.345		83.79	9.501	2.365											Cr
Mn							100										Mn
Fe						5.845		91.75	2.119	0.282							Fe
Co											100						Co
Ni										68.08		26.22	1.140	3.634		0.926	Ni
Cu															69.17		Cu
Zn																48.63	Zn

	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	
Cu	30.83																Cu
Zn		27.90	4.10	18.75		0.62											Zn
Ga					60.11		39.89										Ga
Ge						20.84		27.54	7.73	36.28		7.61					Ge
As											100						As
Se										0.89		9.37	7.63	23.77		49.61	Se
Br															50.69		Br
Kr														0.35		2.28	Kr

	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	
Se		8.73															Se
Br	49.31																Br
Kr		11.58	11.49	57.00		17.30											Kr
Rb					72.17		27.83										Rb
Sr				0.56		9.86	7.00	82.58									Sr
Y									100								Y
Zr										51.45	11.22	17.15		17.38		2.80	Zr
Nb													100				Nb
Mo												14.84		9.25	15.92	16.68	Mo
Ru																5.54	Ru

	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	
Mo	9.55	24.13		9.63													Mo
Ru		1.87	12.76	12.60	17.06	31.55		18.62									Ru
Rh							100										Rh
Pd						1.02		11.14	22.33	27.33		26.46		11.72			Pd
Ag											51.84		48.16				Ag
Cd										1.25		0.89		12.49	12.80	24.13	Cd
Sn																0.97	Sn

	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	
Cd	12.22	28.73		7.49													Cd
In	4.29		95.71														In
Sn		0.66	0.34	14.54	7.68	24.22	8.59	32.58		4.63		5.79					Sn
Sb									57.21		42.79						Sb
Te								0.09		2.55	0.89	4.74	7.07	18.84		31.74	Te
I															100		I
Xe												0.09		0.09		1.92	Xe

	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	
Te		34.08															Te
Xe	26.44	4.08	21.18	26.89		10.44		8.87									Xe
Cs					100												Cs
Ba		0.106		0.101		2.417	6.592	7.854	11.23	71.70							Ba
La										0.090	99.91						La
Ce								0.185		0.251		88.45		11.11			Ce
Pr													100				Pr
Nd														27.2	12.2	23.8	Nd
Sm																3.07	Sm

	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	
Nd	8.3	17.2		5.7		5.6											Nd
Sm			14.99	11.24	13.82	7.38		26.75		22.75							Sm
Eu							47.81		52.19								Eu
Gd								0.20		2.18	14.80	20.47	15.65	24.84		21.86	Gd
Tb															100		Tb
Dy												0.06		0.10		2.34	Dy

	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	
Dy	18.91	25.51	24.90	28.18													Dy
Ho					100												Ho
Er		0.14		1.61		33.61	22.93	26.78		14.93							Er
Tm									100								Tm
Yb								0.13		3.04	14.28	21.83	16.13	31.83		12.76	Yb
Lu															97.41	2.59	Lu
Hf														0.16		5.26	Hf

	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	
Hf	18.60	27.28	13.62	35.08													Hf
Ta				0.012	99.99												Ta
W				0.12		26.50	14.31	30.64		28.43							W
Re									37.40		62.60						Re
Os								0.02		1.59	1.96	13.24	16.15	26.26		40.78	Os
Ir															37.3		Ir
Pt														0.014		0.782	Pt

	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	
Ir	62.7																Ir
Pt		32.97	33.83	25.24		7.163											Pt
Au					100												Au
Hg				0.15		9.97	16.87	23.10	13.18	29.86		6.87					Hg
Tl											29.52		70.48				Tl
Pb												1.4		24.1	22.1	52.4	Pb

	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	
Bi	100																Bi

	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	
Th								100									Th
U										0.006	0.720			99.27			U

: normal plasma recommended

: normal plasma / cool plasma

: recommended mass number

: recommended mass number for ORS / cool
(ORS: Octopole Reaction System)

Figure 4: Isotopic abundance table from the **Agilent** Mass Spectrometer manual.

Other Techniques

- **Spectroscopic Ellipsometry (SE)**
  https://www.atomsolve.com/detailed-introductions/se-details

- **FIB-SEM**
  https://www.atomsolve.com/detailed-introductions/fib

- **Transmission Electron Microscopy**
  https://www.atomsolve.com/detailed-introductions/tem-details

- **3D modeling and simulation**
  https://www.atomsolve.com/detailed-introductions/3drendering