

小麦中元素的 ICP-MS 分析

1 实验部分

1.1 仪器与试剂

OptiMass 9500 型电感耦合等离子体飞行时间质谱仪, 超纯水仪, 分析天平, 温控电热板, 粉碎机, 压力消解罐, 容量瓶。

硝酸, $\rho=1.42\text{ g/ml}$, 优级纯

B、Ti、V、Cr、Mn、Co、Ni、Cu、Zn、As、Se、Sr、Mo、Cd、Sn、Sb、Ba、Tl、Pb, Ca、Mg、Fe、Al 元素标准溶液 (1000mg/L), 使用时采用 2% 硝酸水溶液稀释至所需浓度; 高纯氩气。

1.2 仪器条件

表 1 仪器条件

Cal Curve Type	Linear Least Squares
Sample Introduction	Manual
SmartGate Ranges	(11.6-21),(28.5-42.5), (55.5-56.5) (79-81)
Acquisition Time	5
Replicates	3
Sample Intro Time	30
Sample Pump Speed	6
Flush Pump Speed	30
Flush Time	10
Rinse Time	10
Rinse Pump Speed	10
Torch X position (mm)	9
Torch Y position (mm)	0
Torch Z position (mm)	0
Gasbox nebulizer flow (l/min)	0.68
Gasbox plasma flow (l/min)	10
Gasbox auxiliary flow (l/min)	0.5
Generator set power (W)	1200
Skimmer (V)	-1500
Extraction (V)	-1400
Z1 (V)	-900
Y Mean (V)	-250
Y Deflection (V)	-2
Z Lens Mean (V)	-1000
Z Lens Deflection (V)	-30
Lens Body (V)	-160
Fill (V)	-30
Fill Bias (V)	0
Fill Grid (V)	-13
Pushout Grid (V)	-380
Pushout Plate (V)	690
Blanker (V)	150
Reflectron (V)	700
Multiplier Gain (V)	3100
Detector Time C0 Work (ns)	-825.226
Detector Time C1 Work	2013.48
Blanker Time C0 (ns)	158.339
Blanker Time C1	247.582

1.3 标准溶液的配制

准确移取 0.25ml B、Ti、V、Cr、Mn、Co、Ni、Cu、Zn、As、Se、Sr、Mo、Cd、Sn、Sb、Ba、Tl、Pb 1000ug/ml 母液，2ml Ca、Mg、Fe、Al 1000ug/ml 母液于 50ml 容量瓶中，加入 1ml 浓硝酸，用水定容至刻度摇匀，即配成 B、Ti、V、Cr、Mn、Co、Ni、Cu、Zn、As、Se、Sr、Mo、Cd、Sn、Sb、Ba、Tl、Pb 浓度为 5ug/ml，Ca、Mg、Fe、Al 浓度为 40 ug/ml 的标准中间溶液；分别取适量多元素标准中间液于 50mL 容量瓶中，用 2% 硝酸溶液定容至刻度稀释至所需浓度摇匀备用。

1.4 样品处理

1.4.1 压力罐消解法

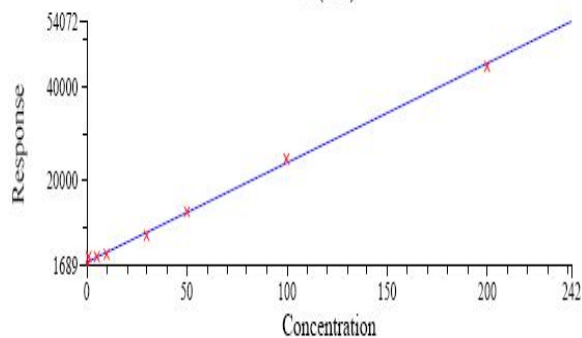
准确称取 0.5g 粉碎后的小麦粉样品于消解罐中，加入 5ml 硝酸，放置过夜，旋紧不锈钢外套，放入恒温干燥箱消解，于 160℃消解 4h，冷却后，缓慢旋松不锈钢外套，将消解罐取出，在控温电加热板上于 100℃加热 30min，冷却后用水定容至 50ml，混匀备用，同时做空白试验。

消解方式	步骤	控制温度	恒温时间
压力罐消解	1	80℃	2h
	2	120℃	2h
	3	160℃	4h

2 实验结果

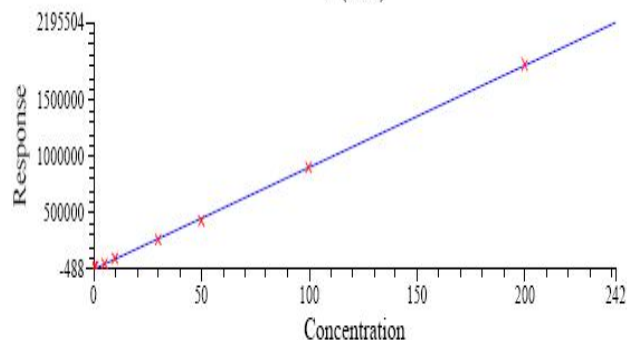
2.1 标准曲线

B (B11)



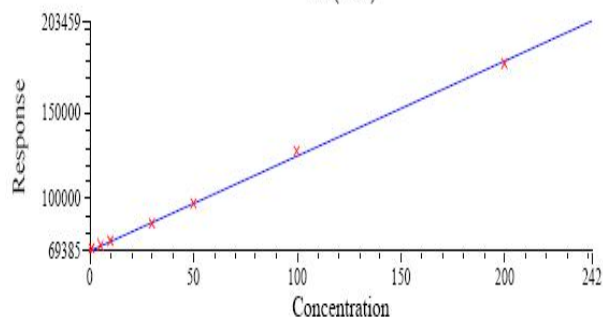
Conc = 0.00467819 * Resp - 10.9594
R² = 0.998086 Max Error = 4.173890

V (M51)



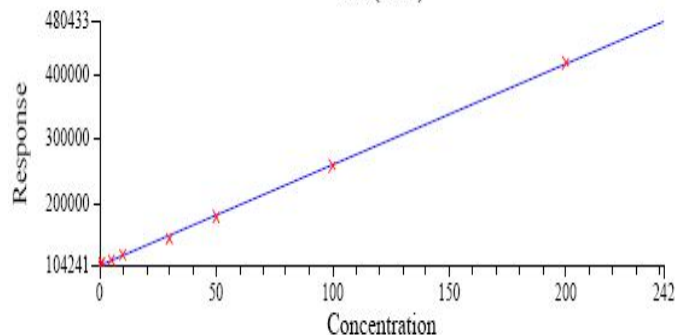
Conc = 0.000110201 * Resp + 0.0537906
R² = 0.999675 Max Error = 1.985031

Ti3 (Ti49)



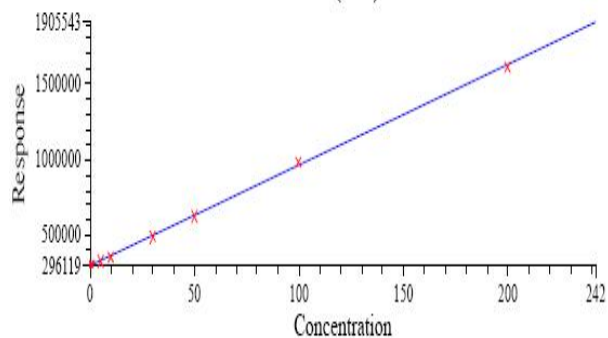
Conc = 0.00181808 * Resp - 127.904
R² = 0.999005 Max Error = 4.753625

Cr2 (Cr53)



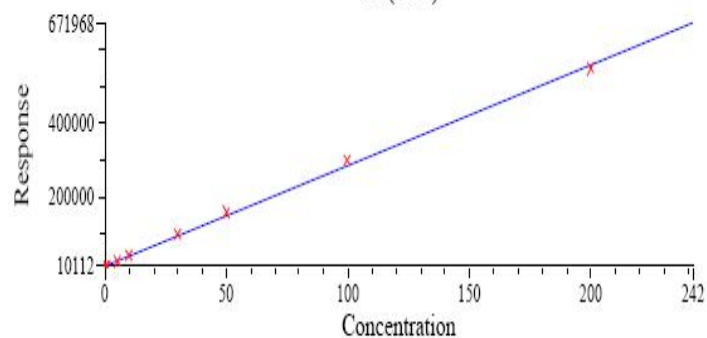
Conc = 0.000643288 * Resp - 67.0567
R² = 0.999512 Max Error = 2.524304

Mn (M55)

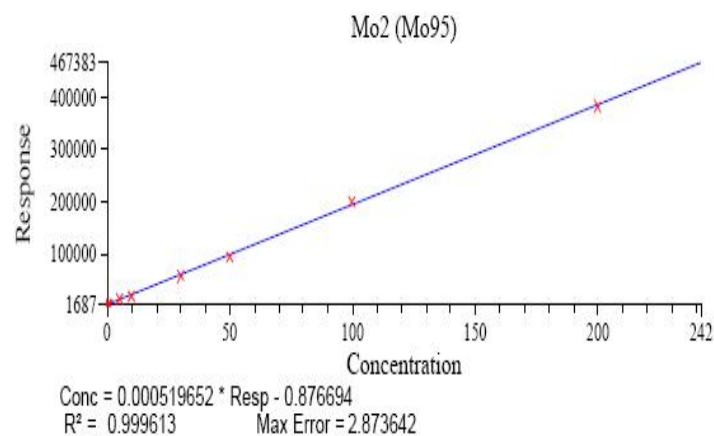
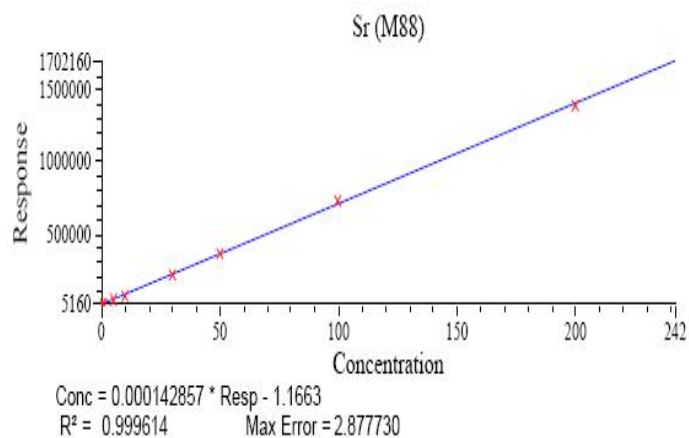
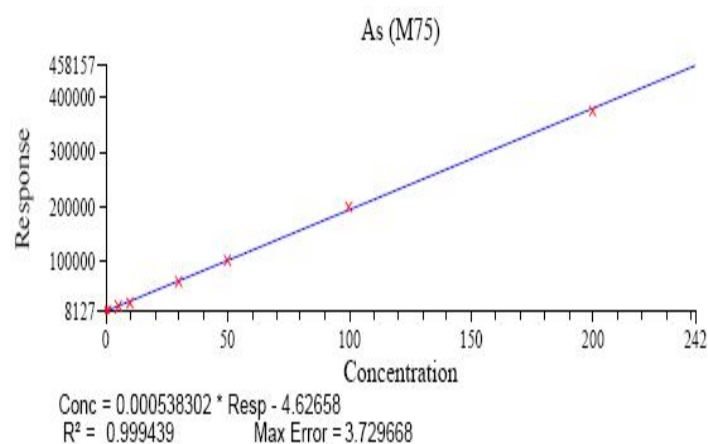
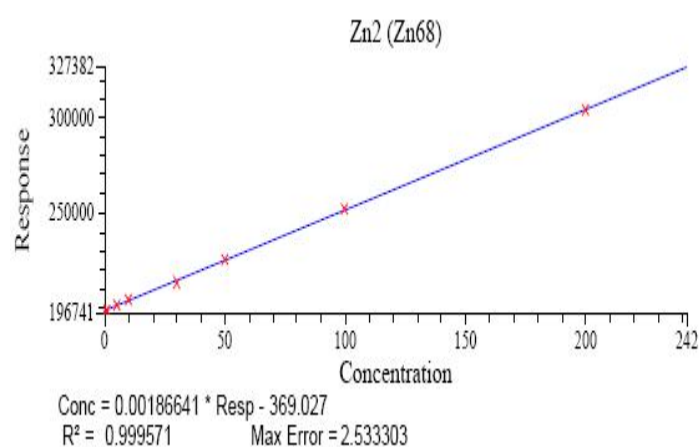
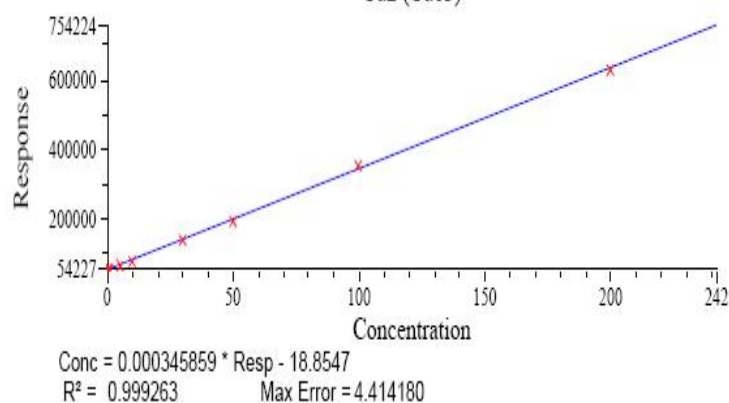
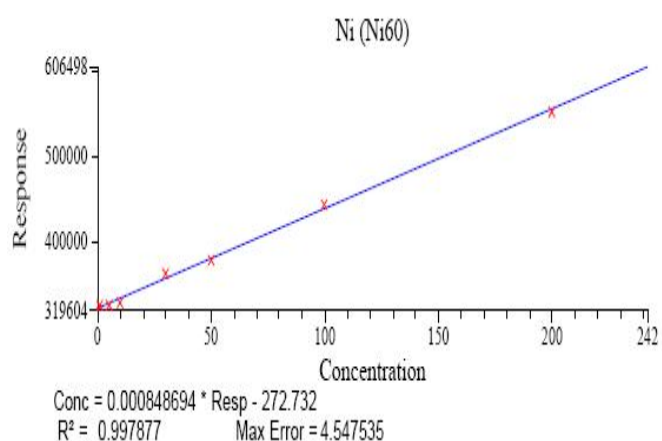


Conc = 0.000150364 * Resp - 44.5257
R² = 0.999475 Max Error = 3.495769

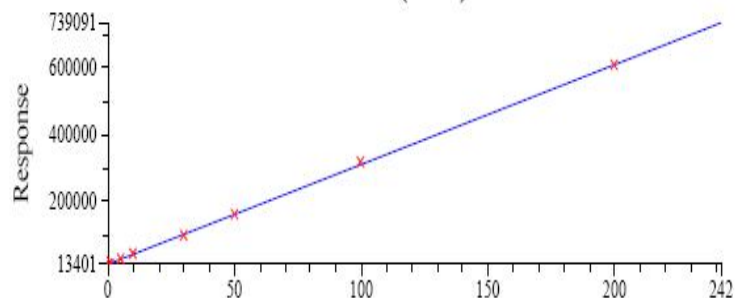
Co (M59)



Conc = 0.000367916 * Resp - 5.22803
R² = 0.998355 Max Error = 4.855919



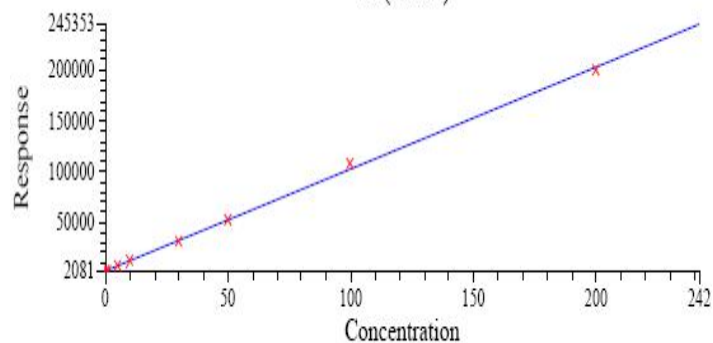
Sn2 (Sn118)



$$\text{Conc} = 0.000333501 * \text{Resp} - 4.48733$$

$$R^2 = 0.999655 \quad \text{Max Error} = 2.937079$$

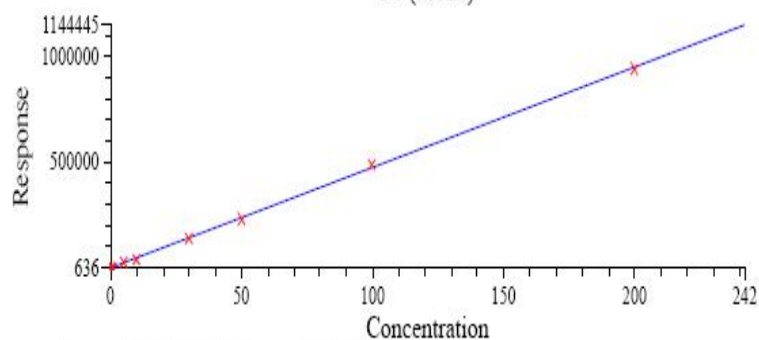
Cd (Cd111)



$$\text{Conc} = 0.00099642 * \text{Resp} - 2.47454$$

$$R^2 = 0.999333 \quad \text{Max Error} = 4.156512$$

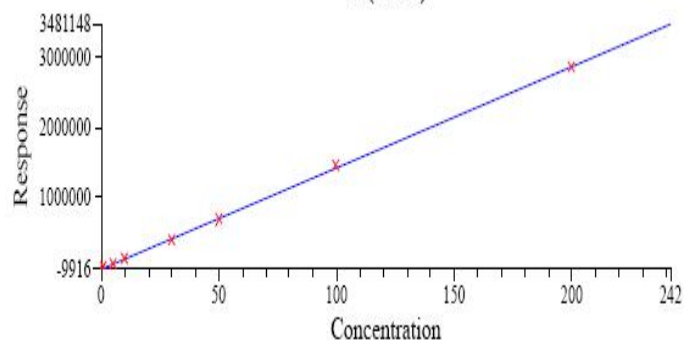
Sb (M121)



$$\text{Conc} = 0.000211574 * \text{Resp} - 0.134646$$

$$R^2 = 0.999694 \quad \text{Max Error} = 2.663989$$

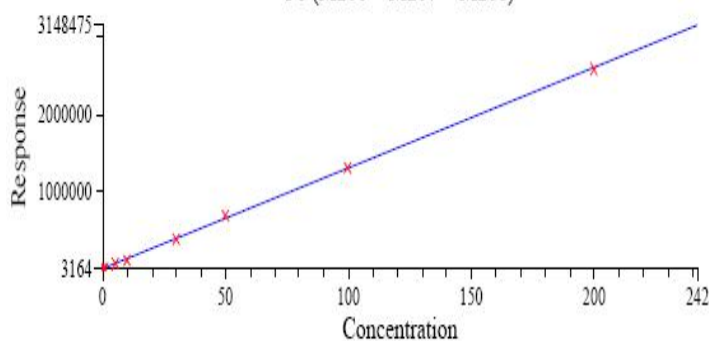
Tl (Tl205)



$$\text{Conc} = 6.93199e-005 * \text{Resp} + 0.687383$$

$$R^2 = 0.999766 \quad \text{Max Error} = 1.510686$$

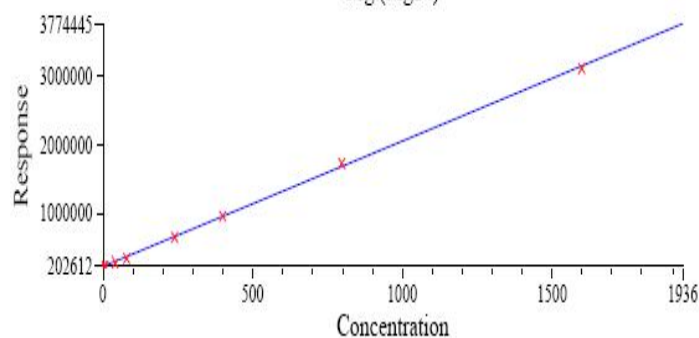
Pb (M206 + M207 + M208)



$$\text{Conc} = 7.71353e-005 * \text{Resp} - 0.858608$$

$$R^2 = 0.999698 \quad \text{Max Error} = 2.478703$$

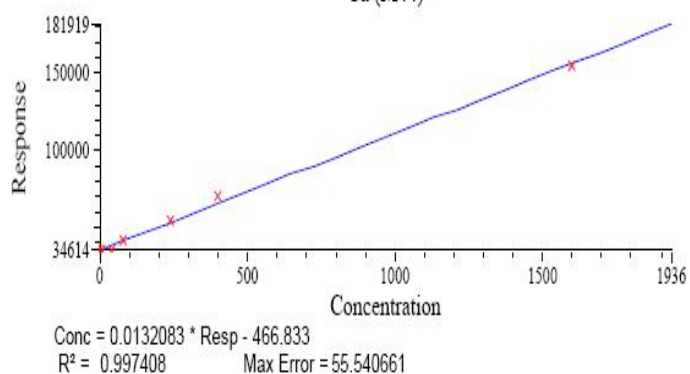
Mg (Mg24)



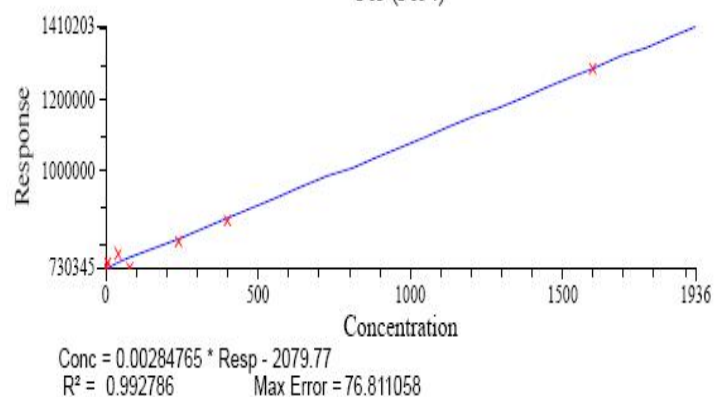
$$\text{Conc} = 0.000543064 * \text{Resp} - 113.767$$

$$R^2 = 0.999417 \quad \text{Max Error} = 29.927820$$

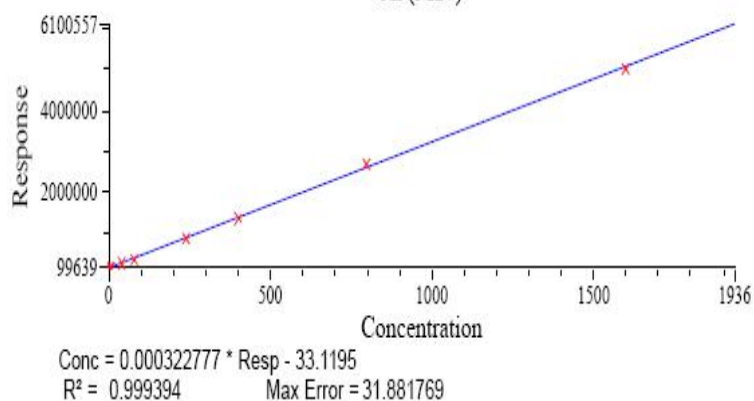
Ca (M44)



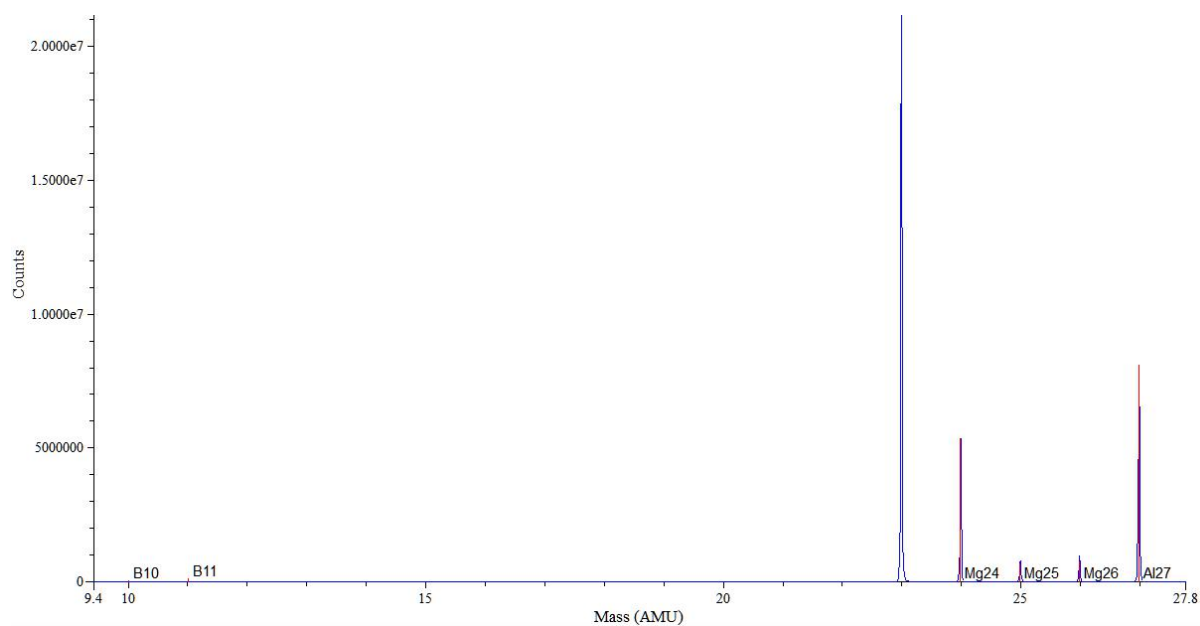
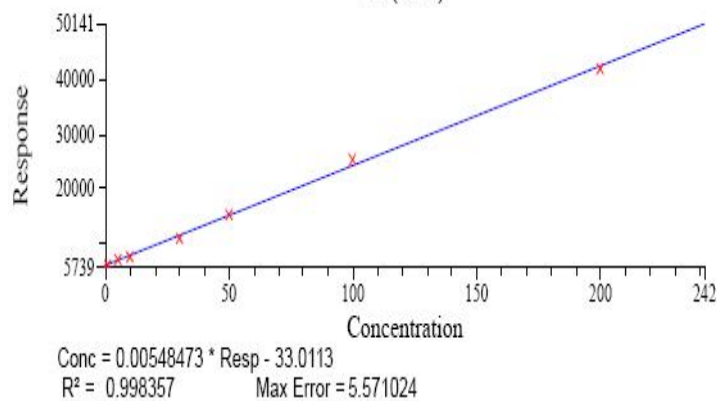
Fe3 (Fe54)

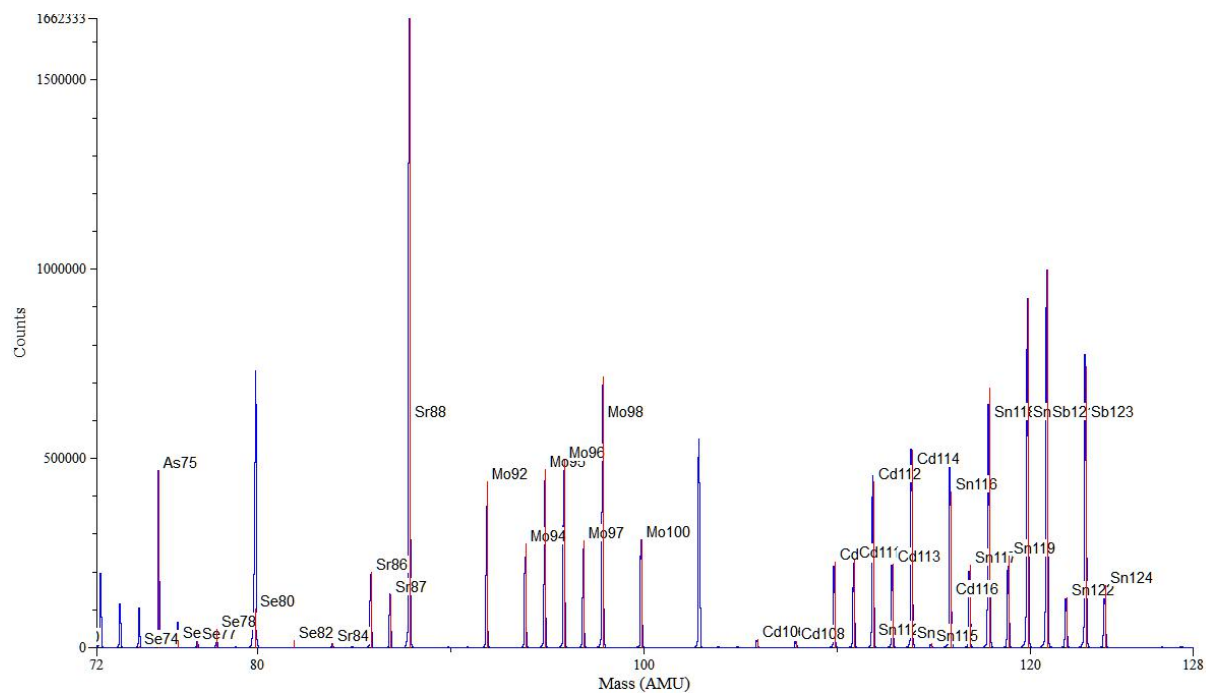
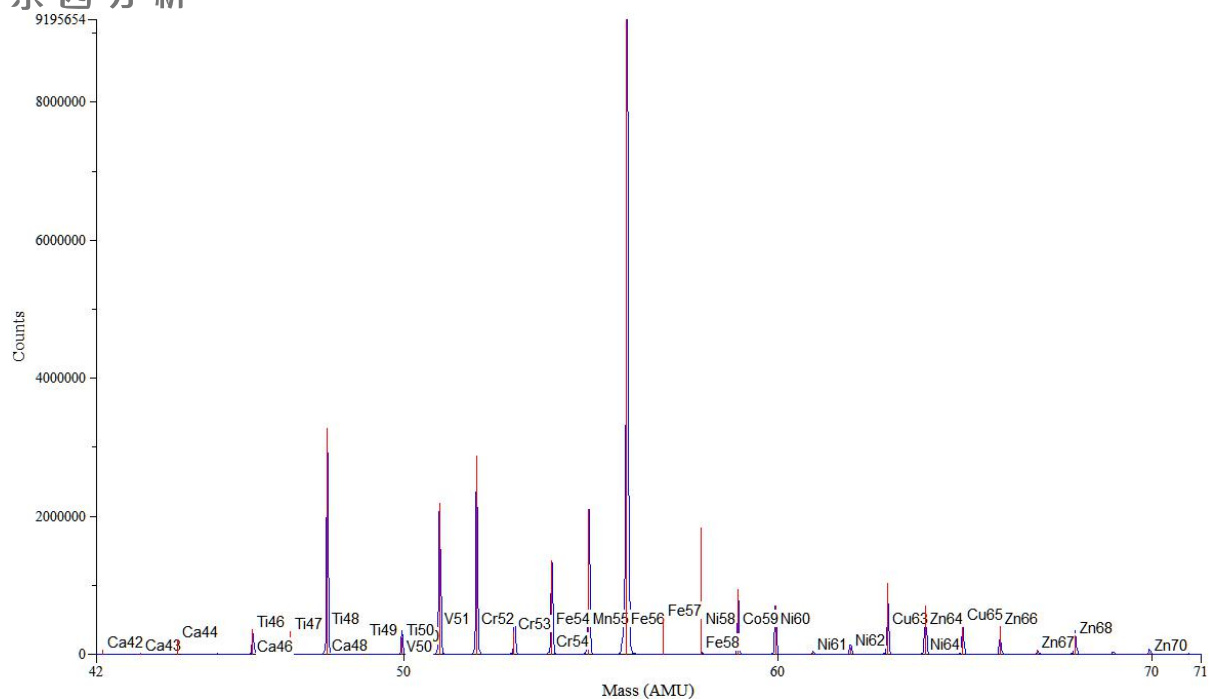


Al (M27)



Se (Se78)





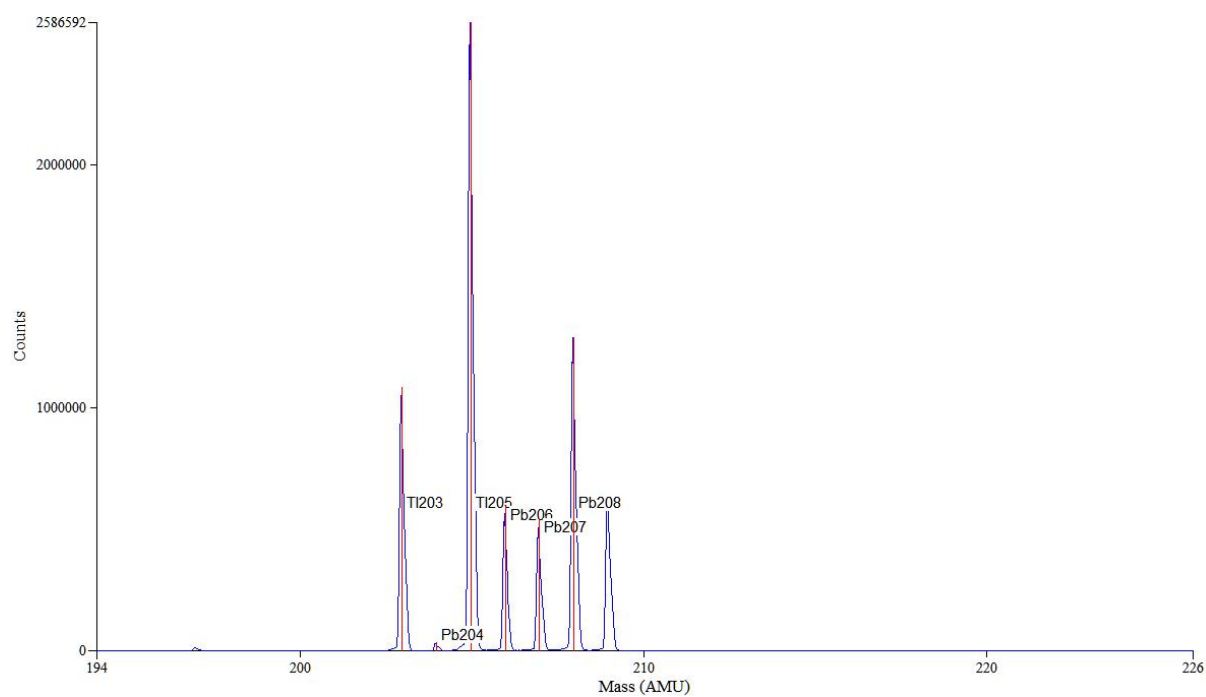
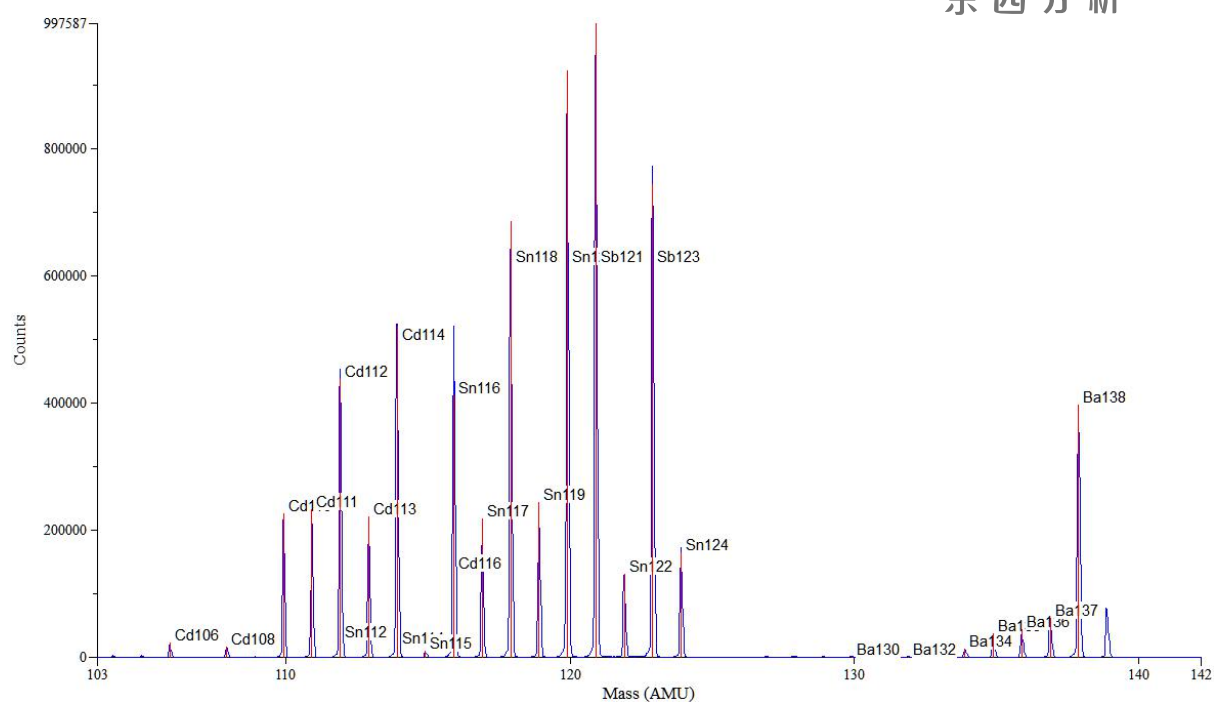


图 1 23 种元素标样谱图

表 2 标准系列溶液浓度

序号	元素	标准系列质量浓度 ($\mu\text{g/L}$)							
		系列 1	系列 2	系列 3	系列 4	系列 5	系列 6	系列 7	系列 8
1	B	0	1	5	10	30	50	100	200
2	V	0	1	5	10	30	50	100	200
3	Ti	0	1	5	10	30	50	100	200
4	Cr	0	1	5	10	30	50	100	200
5	Mn	0	1	5	10	30	50	100	200
6	Co	0	1	5	10	30	50	100	200
7	Ni	0	1	5	10	30	50	100	200
8	Cu	0	1	5	10	30	50	100	200
9	Zn	0	1	5	10	30	50	100	200
10	As	0	1	5	10	30	50	100	200
11	Sr	0	1	5	10	30	50	100	200
12	Mo	0	1	5	10	30	50	100	200
13	Sn	0	1	5	10	30	50	100	200
14	Cd	0	1	5	10	30	50	100	200
15	Sb	0	1	5	10	30	50	100	200
16	Ba	0	1	5	10	30	50	100	200
17	Tl	0	1	5	10	30	50	100	200
18	Pb	0	1	5	10	30	50	100	200
19	Mg	0	8	40	80	240	400	800	1600
20	Ca	0	8	40	80	240	400	800	1600
21	Fe	0	8	40	80	240	400	800	1600
22	Al	0	8	40	80	240	400	800	1600
23	Se	0	1	5	10	30	50	100	200

表 3 方法线性方程及检测结果

序号	元素	相关系数	线性方程	元素含量 mg/kg	检测限 ng/mL
1	B	0.9981	$\text{Conc} = 0.00467819 * \text{Resp} - 10.9594$	0.5	2
2	V	0.9997	$\text{Conc} = 0.000110201 * \text{Resp} + 0.0537906$	0	0.1
3	Ti	0.9990	$\text{Conc} = 0.00181808 * \text{Resp} - 127.904$	0.3	1.8
4	Cr	0.9995	$\text{Conc} = 0.000643288 * \text{Resp} - 67.0567$	0	2.5
5	Mn	0.9995	$\text{Conc} = 0.000150364 * \text{Resp} - 44.5257$	10.2	3.0
6	Co	0.9984	$\text{Conc} = 0.000367916 * \text{Resp} - 5.22803$	0	0.5
7	Ni	0.9979	$\text{Conc} = 0.000848694 * \text{Resp} - 272.732$	0	12
8	Cu	0.9993	$\text{Conc} = 0.000345859 * \text{Resp} - 18.8547$	2.7	0.53
9	Zn	0.9996	$\text{Conc} = 0.00186641 * \text{Resp} - 369.027$	10.0	2.5
10	As	0.9994	$\text{Conc} = 0.000538302 * \text{Resp} - 4.62658$	0	0.47
11	Sr	0.9996	$\text{Conc} = 0.000142857 * \text{Resp} - 1.1663$	3.0	0.02
12	Mo	0.9996	$\text{Conc} = 0.000519652 * \text{Resp} - 0.876694$	0.7	0.26
13	Sn	0.9997	$\text{Conc} = 0.000333501 * \text{Resp} - 4.48733$	0	0.1
14	Cd	0.9993	$\text{Conc} = 0.00099642 * \text{Resp} - 2.47454$	0	0.72
15	Sb	0.9997	$\text{Conc} = 0.000211574 * \text{Resp} - 0.134646$	0	0.08
16	Ba	0.9995	$\text{Conc} = 0.000298918 * \text{Resp} - 0.353772$	12.7	0.20
17	Tl	0.9998	$\text{Conc} = 6.93199\text{e-}005 * \text{Resp} + 0.687383$	0	0.002
18	Pb	0.9997	$\text{Conc} = 7.71353\text{e-}005 * \text{Resp} - 0.858608$	0	0.02
19	Mg	0.9994	$\text{Conc} = 0.000543064 * \text{Resp} - 113.767$	438	4.6
20	Ca	0.9959	$\text{Conc} = 0.0132083 * \text{Resp} - 466.833$	318	13
21	Fe	0.9911	$\text{Conc} = 0.00279887 * \text{Resp} - 2047.24$	42.6	12.5
22	Al	0.9994	$\text{Conc} = 0.000322777 * \text{Resp} - 33.1195$	20.6	4.0
23	Se	0.9984	$\text{Conc} = 0.00548473 * \text{Resp} - 33.0113$	0.2	2.0

