检测 Feed 中金属元素

1 样品溶液制备：

称取一定量样品（精确到万分之一）于锥形烧杯中，加入硝酸+高氯酸混合酸（硝酸：高氯酸=9：1体积比）10-20mL，盖上表面皿，电热板低温加热至溶液澄清透明，高温继续加热至冒白烟，取下冷却至室温，去掉表面皿，用去离子水冲洗杯壁，继续加热溶液至0.5mL，取下冷却，用去离子水转移定容至25mL，摇匀备用。

2 实验设备及试剂：

GBC Quantima 电感耦合等离子体发射光谱仪

可调加热板

盐酸（HCl）：优级纯

钾单元素标准溶液（国家标准物质研究中心）

钠单元素标准溶液（国家标准物质研究中心）

镁单元素标准溶液（国家标准物质研究中心）

钙单元素标准溶液（国家标准物质研究中心）

镉单元素标准溶液（国家标准物质研究中心）

铅单元素标准溶液（国家标准物质研究中心）

磷单元素标准溶液（国家标准物质研究中心）

砷单元素标准溶液（国家标准物质研究中心）

汞单元素标准溶液（国家标准物质研究中心）

3 仪器条件

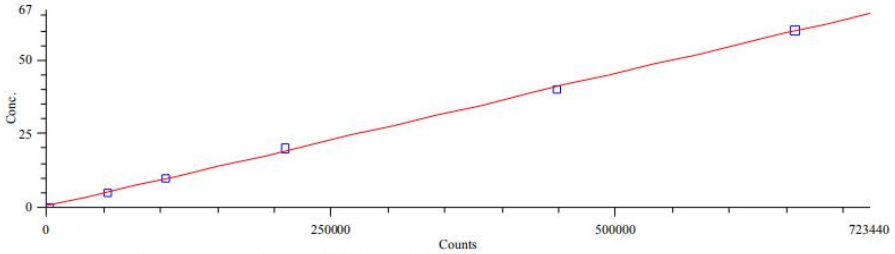
El	nm	Type	Mono	Order	Power (W)	Neb (L/min)	Height (mm)	Plasma (L/min)	Aux (L/min)	Pump (RPM)	PMT (V)	Int. (s)	Reps	Bkgd Corr
Na I	589.592	An	1	1	1000	0.80	6.0	10.0	0.5	10	600	0.50	3	Fixed
Mg II	279.553	An	1	2	1000	0.80	6.0	10.0	0.5	10	300	0.50	3	Fixed
K I	769.896	An	1	1	900	0.80	6.0	10.0	0.5	10	750	0.50	3	Fixed
P I	253.565	An	1	2	1000	0.80	6.0	10.0	0.5	10	550	0.50	3	Fixed
Pb II	220.353	An	1	2	1000	0.80	6.0	10.0	0.5	10	600	0.50	3	Fixed
Cd I	228.802	An	1	2	1000	0.80	6.0	10.0	0.5	10	500	0.50	3	Fixed
Ca II	393.366	An	1	1	1000	0.80	6.0	10.0	0.5	10	300	0.50	3	Fixed
Hg I	253.652	An	1	2	900	0.80	6.0	10.0	0.5	10	500	0.50	3	Fixed
As I	228.812	An	1	2	900	0.80	6.0	10.0	0.5	10	550	0.50	3	Fixed

4 标准溶液的配制

元素	浓度（mg/L）					
Na	0	5	10	20	40	60
Mg	0	0.5	1	5	10	30
K	0	10	20	40	60	/
P	0	1	5	10	30	/
Pb	0	0.5	1	2	5	10
Cd	0	0.1	0.5	1	4	/
Ca	0	1	5	10	20	40
Hg	0	1	5	10	20	/
As	0	1	5	10	20	/

5 标准曲线

Na I 589.592 nm (mg/L)

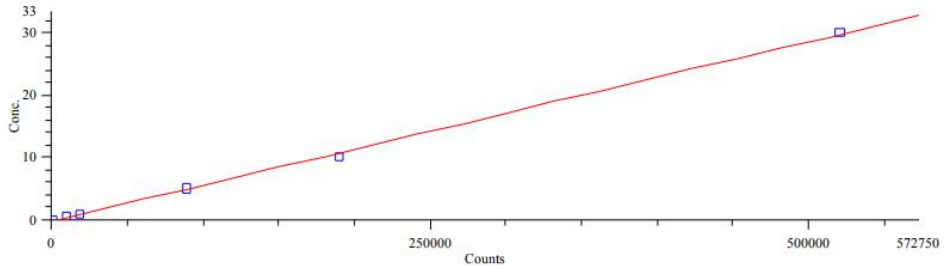


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	657673	60.00	59.75	-0.422
Standard 2	448023	40.00	40.79	1.99
Standard 3	209264	20.00	19.21	-3.94
Standard 4	104263	10.00	9.719	-2.81
Standard 5	52914.9	5.000	5.078	1.55
Blank	1731.08	0	0.4506	***

Calibration Coefficients	C0	C1	R
	0.294087	9.040e-5	0.9994

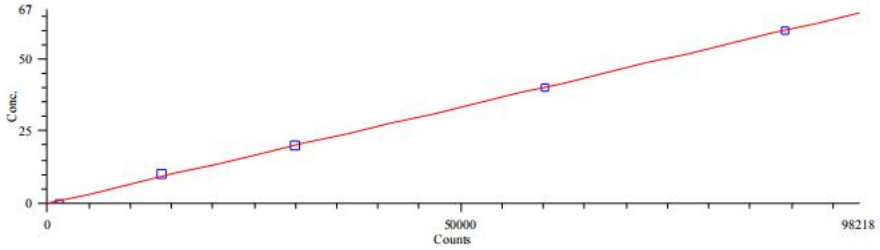
Na

Mg II 279.553 nm (mg/L)



Mg

K I 769.896 nm (mg/L)

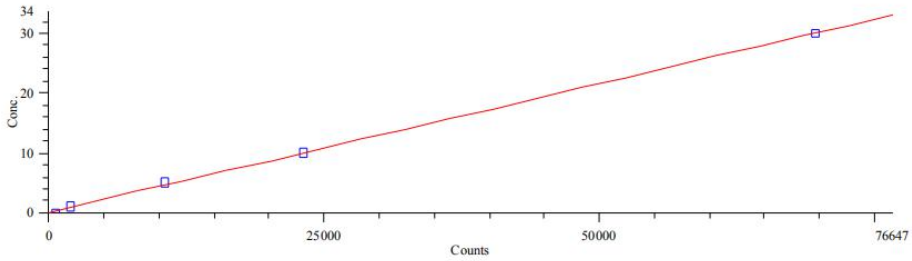


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	89288.9	60.00	59.92	-0.133
Standard 2	60244.1	40.00	40.35	0.874
Standard 3	29957.9	20.00	19.94	-0.289
Standard 4	13765.9	10.00	9.032	-9.68
Blank	1484.09	0	0.7562	***

Calibration Coefficients	C0	C1	R
	-0.24383	6.738e-4	0.9993

K

P I 253.565 nm (mg/L)

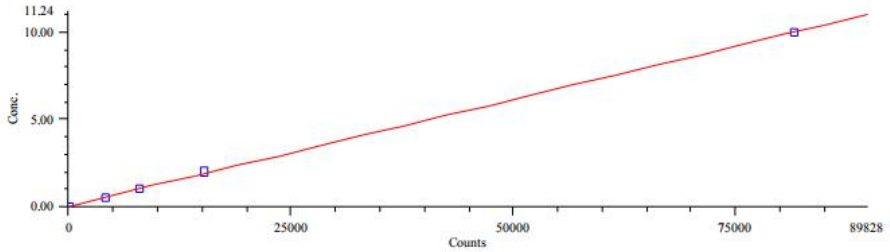


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	69679.0	30.00	30.05	0.153
Standard 2	23124.9	10.00	10.03	0.306
Standard 3	10535.2	5.000	4.618	-7.64
Standard 4	1989.83	1.000	0.9439	-5.61
Blank	635.720	0	0.3617	***

Calibration	C0	C1	R
Coefficients	0.088396	4.299e-4	0.9995

P

Pb II 220.353 nm (mg/L)

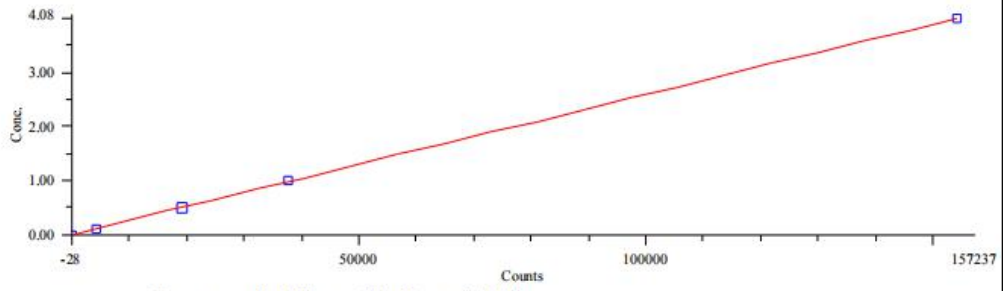


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	81661.6	10.00	10.02	0.171
Standard 2	15188.4	2.000	1.892	-5.41
Standard 3	7966.29	1.000	1.009	0.910
Standard 4	4147.96	0.5000	0.5424	8.47
Blank	34.6992	0	0.03960	***

Calibration	C0	C1	R
Coefficients	0.035359	1.222e-4	0.9998

Pb

Cd I 228.802 nm (mg/L)

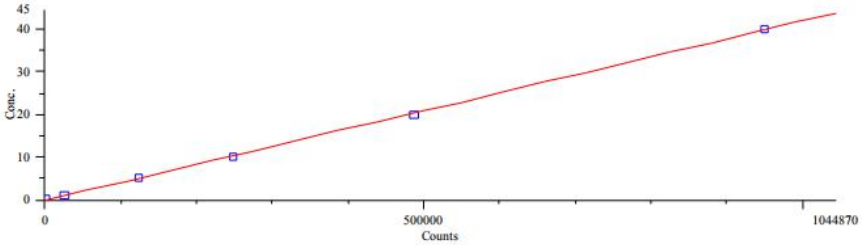


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	154154	4.000	4.004	0.102
Standard 2	37709.8	1.000	0.9813	-1.87
Standard 3	19224.0	0.5000	0.5014	0.287
Standard 4	4202.54	0.1000	0.1115	11.5
Blank	-27.7528	0	0.001680	***

Calibration Coefficients	C0	C1	R
	0.002400	2.596e-5	1.000

Cd

Ca II 393.366 nm (mg/L)

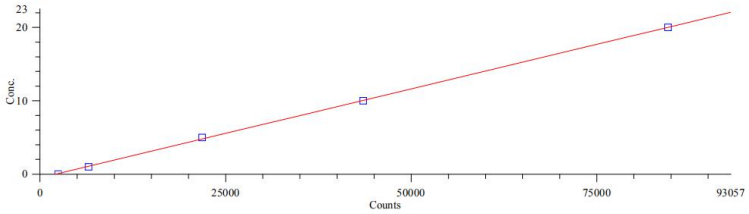


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	949882	40.00	39.78	-0.558
Standard 2	487387	20.00	20.30	1.51
Standard 3	249226	10.00	10.27	2.72
Standard 4	124301	5.000	5.012	0.237
Standard 5	25310.5	1.000	0.8434	-15.7
Blank	400.849	0	-0.2055	***

Calibration Coefficients	C0	C1	R
	-0.22241	4.211e-5	0.9998

Ca

Hg I 253.652 nm (mg/L)

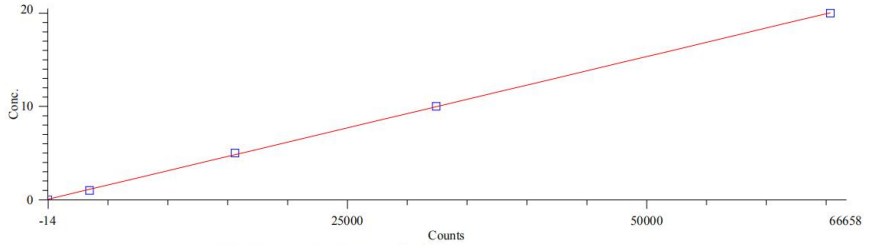


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	84597.4	20.00	20.02	0.108
Standard 2	43532.0	10.00	10.05	0.520
Standard 3	21829.8	5.000	4.783	-4.34
Standard 4	6536.59	1.000	1.070	7.04
Blank	2427.09	0	0.07272	***

Calibration Coefficients	C0	C1	R
	-0.51651	2.428e-4	0.9998

Hg

As I 228.812 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	65350.8	20.00	20.05	0.249
Standard 2	32420.9	10.00	9.972	-0.280
Standard 3	15606.8	5.000	4.826	-3.48
Standard 4	3453.01	1.000	1.107	10.7
Blank	-14.3975	0	0.04546	***

Calibration	C0	C1	R
Coefficients	0.049862	3.060e-4	0.9998

As