

检测钨材料中的 W、Y、K、La、Mo 等元素

1 样品溶液制备:

称取 1.0g (精确到万分之一) 样品于聚四氟烧杯中, 加入 10mL 硝酸和 25mL 氢氟酸, 加盖, 电热板低温加热溶解, 取下冷却至室温, 开盖, 继续加热蒸至湿盐状, 再加入 7mL 氢氟酸溶解, 溶解后取下冷却, 用去离子水转移定容至 50mL, 摇匀, 备用。

2 实验设备及试剂:

GBC Quantima 原子吸收分光光度计 (配有 W、K、Ce、La、Y、Zr、Mo、Cu 空心阴极灯, 北京东西分析仪器有限公司)

可调加热板

硝酸 (HNO₃): 优级纯

氢氟酸 (HF): 优级纯

W 单元素标准溶液 (国家标准物质研究中心)

K 单元素标准溶液 (国家标准物质研究中心)

Ce 单元素标准溶液 (国家标准物质研究中心)

La 单元素标准溶液 (国家标准物质研究中心)

Y 单元素标准溶液 (国家标准物质研究中心)

Zr 单元素标准溶液 (国家标准物质研究中心)

Mo 单元素标准溶液 (国家标准物质研究中心)

Cu 单元素标准溶液 (国家标准物质研究中心)

3 仪器条件

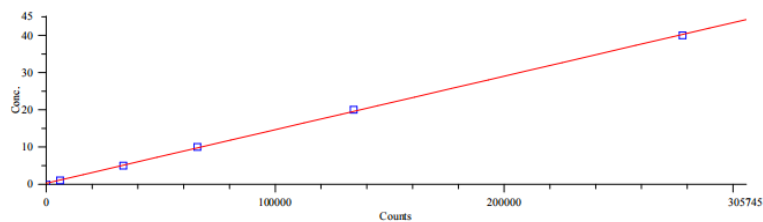
El	nm	Type	Mono	Order	Power (W)	Neb (L/min)	Height (mm)	Plasma (L/min)	Aux (L/min)	Pump (RPM)	PMT (V)	Int. (s)	Reps	Bkgd Corr
W II	239.709	An	1	2	1200	0.80	6.0	10.0	0.5	10	500	0.50	3	Fixed
Cu I	324.754	An	1	1	1000	0.80	6.0	12.0	0.5	10	500	0.50	3	Fixed
Zr II	349.621	An	1	1	1000	0.80	6.0	10.0	0.5	10	500	0.50	3	Fixed
Mo II	202.030	An	1	2	1000	0.80	6.0	12.0	0.5	10	500	0.50	3	Dyn
Ce II	418.660	An	1	1	1000	0.80	6.0	12.0	0.5	10	700	0.50	3	Fixed
Y II	378.870	An	1	1	1000	0.80	6.0	12.0	0.5	10	600	0.50	3	Fixed
La II	408.672	An	1	1	1000	0.50	6.0	12.0	0.5	10	700	0.50	3	Fixed
K I	769.896	An	1	1	900	0.80	6.0	10.0	0.5	10	600	0.50	3	Fixed

4 标准溶液的配制

元素	浓度 (mg/L)					
W	0	1	5	10	20	40
Cu	0	0.1	0.5	1	5	10
Zr	0	0.2	0.5	1	2	/
Mo	0	0.5	1	5	10	20
Ce	0	0.5	1	5	10	20
Y	0	0.1	0.5	1	5	10
La	0	0.05	0.1	0.5	1	5
K	0	5	10	20	40	/

5 标准曲线

W II 239.709 nm (mg/L)



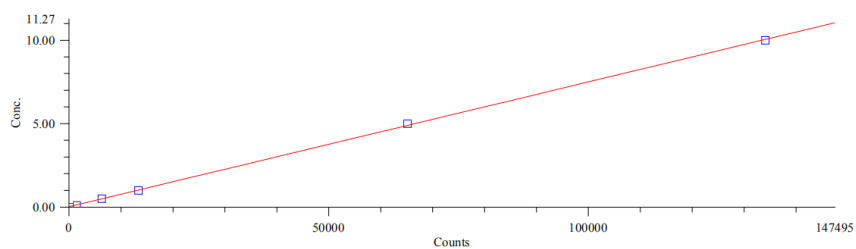
Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	277950	40.00	40.26	0.645
Standard 2	134237	20.00	19.57	-2.16
Standard 3	66000.9	10.00	9.744	-2.56
Standard 4	33564.2	5.000	5.075	1.49
Standard 5	6014.32	1.000	1.108	10.8
Blank	26.6243	0	0.2464	***

Calibration Coefficients

C0	C1	R
0.242587	1.440e-4	0.9997

W

Cu I 324.754 nm (mg/L)



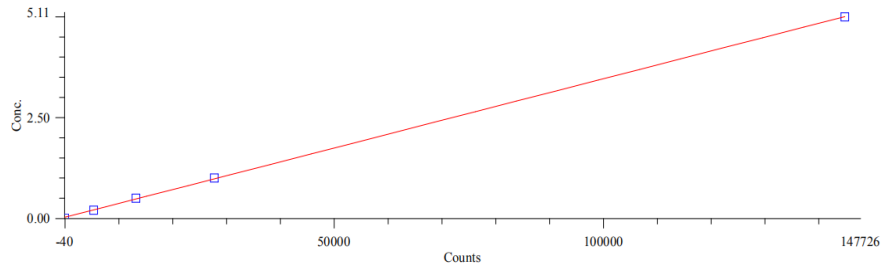
Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	134086	10.00	10.05	0.499
Standard 2	65175.0	5.000	4.895	-2.11
Standard 3	13324.7	1.000	1.016	1.56
Standard 4	6273.40	0.5000	0.4881	-2.37
Standard 5	1428.04	0.1000	0.1256	25.6
Blank	96.4199	0	0.02603	***

Calibration Coefficients

C0	C1	R
0.018814	7.481e-5	0.9998

Cu

Zr II 349.621 nm (mg/L)

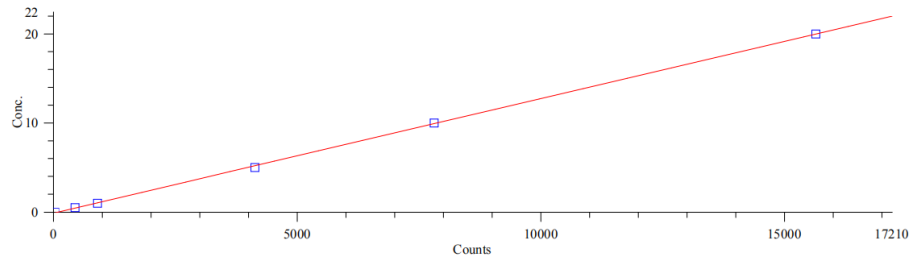


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	144829	5.000	5.005	0.106
Standard 2	27738.2	1.000	0.9804	-1.96
Standard 3	13173.0	0.5000	0.4797	-4.06
Standard 4	5302.87	0.2000	0.2092	4.58
Blank	-39.5075	0	0.02552	***

Calibration Coefficients	C0	C1	R
	0.026873	3.437e-5	0.9999

Zr

Mo II 202.030 nm (mg/L)

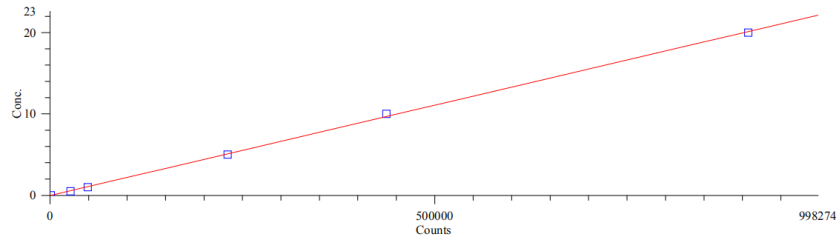


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	15645.8	20.00	19.99	-0.0576
Standard 2	7809.62	10.00	9.919	-0.809
Standard 3	4132.58	5.000	5.194	3.88
Standard 4	898.805	1.000	1.039	3.89
Standard 5	437.201	0.5000	0.4458	-10.8
Blank	22.9846	0	-0.08650	***

Calibration Coefficients	C0	C1	R
	-0.11603	0.001285	0.9998

Mo

Ce II 418.660 nm (mg/L)

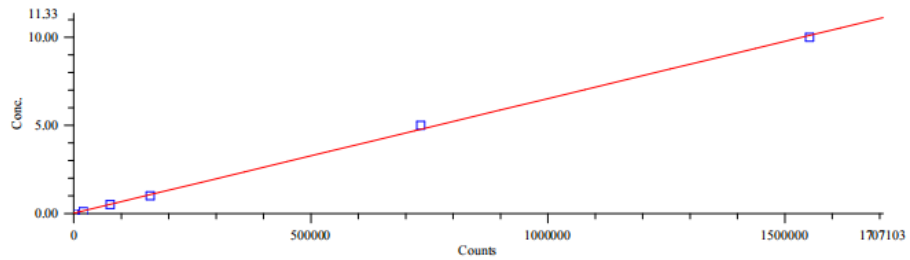


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	907522	20.00	20.13	0.639
Standard 2	436972	10.00	9.677	-3.23
Standard 3	230537	5.000	5.092	1.83
Standard 4	48888.0	1.000	1.057	5.70
Standard 5	26366.3	0.5000	0.5568	11.4
Blank	858.964	0	-0.009723	***

Calibration Coefficients	C0	C1	R
	-0.02880	2.221e-5	0.9996

Ce

Y II 378.870 nm (mg/L)

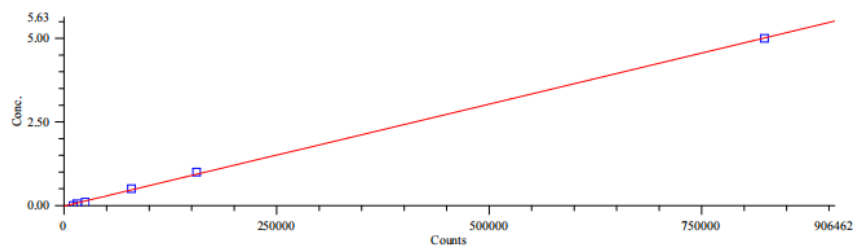


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	1551910	10.00	10.10	1.01
Standard 2	731568	5.000	4.770	-4.61
Standard 3	160851	1.000	1.060	6.04
Standard 4	76455.6	0.5000	0.5119	2.38
Standard 5	19435.4	0.1000	0.1413	41.3
Blank	67.4447	0	0.01546	***

Calibration Coefficients	C0	C1	R
	0.015020	6.499e-6	0.9991

Y

La II 408.672 nm (mg/L)

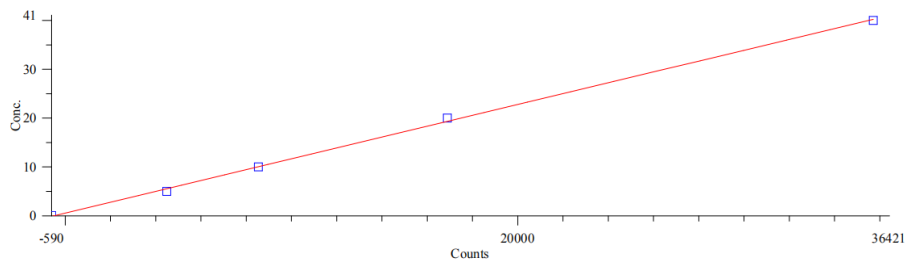


Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	82405.7	5.000	5.015	0.299
Standard 2	155702	1.000	0.9306	-6.94
Standard 3	79022.7	0.5000	0.4620	-7.60
Standard 4	24827.7	0.1000	0.1308	30.8
Standard 5	14532.1	0.05000	0.06787	35.7
Blank	10599.7	0	0.04384	***

Calibration Coefficients	C0	C1	R
	-0.02094	6.111e-6	0.9995

La

K I 769.896 nm (mg/L)



Standard	Counts	Real Conc	Calc Conc	% Diff
Standard 1	35706.7	40.00	40.25	0.618
Standard 2	16888.7	20.00	19.32	-3.41
Standard 3	8536.07	10.00	10.03	0.287
Standard 4	4488.29	5.000	5.527	10.5
Blank	-590.188	0	-0.1213	***

Calibration Coefficients	C0	C1	R
	0.535106	0.001112	0.9992

K