



Determination of Metal Elements such as Potassium, Sodium, Calcium and Magnesium in Glass

1 Sample solution preparation :

1.0g sample (precision to 0.0001g) was weighed and placed in a PTFE beaker. 10mL HF, 3mL HNO₃ were added, heated to dissolve. 10mL HF, 2mL HClO₄ were added, evaporated to paste form, the beaker wall was washed with water, and 1mL HClO₄ was added, then heated until white smoke stops. 25 mL deionized water, 8 mL (1+1) HCl were added, heated until all residue dissolve completely, cooled down, transferred to a 100 mL volumetric flask, washed with water up to volume.

2 Experimental equipment and reagents :

AA7000 series atomic absorption spectrophotometer (with K, Na, Ca, Mg, Fe, Al, Ba, Sb hollow cathode lamp, EWAI Inc.)

Temperature-controlled hot plate

PTFE beaker

PTFE volumetric flask

Hydrochloric acid (HCl): excellent grade purity

Nitric acid (HNO₃): excellent grade purity

Hydrofluoric acid (HF): excellent grade purity

Perchloric acid (HClO₄): excellent grade purity

100 g/L lanthanum chloride solution: 11.73g lanthanum oxide was weighed and placed in a 100mL volumetric flask, first wet with small amount of water, and then 37.5mL hydrochloric acid was added. Deionized water was added to dilute it up to the volume.

Potassium standard solution (National Reference Materials Research Center)

Sodium standard solution (National Reference Materials Research Center)

Calcium standard solution (National Reference Materials Research Center)

Magnesium standard solution (National Reference Materials Research Center)

Iron standard solution (National Reference Materials Research Center)

Aluminium standard solution (National Reference Materials Research Center)

Barium standard solution (National Reference Materials Research Center)

Antimony standard solution (National Reference Materials Research Center)

3 Instrument conditions

| Parameter | Wavelength (nm) | Slit width (nm) | Burner height (mm) | Fuel gas flow rate (L/min) | Lamp current (mA) | Flame type |
|-----------|-----------------|-----------------|--------------------|----------------------------|-------------------|---------------------------|
| K | 766.5 | 0.2 | 10 | 1.5 | 3 | Air – acetylene |
| Na | 589.0 | 0.2 | 10 | 1.5 | 3 | Air – acetylene |
| Ca | 422.7 | 0.2 | 10 | 1.5 | 3 | Air - acetylene |
| Mg | 285.2 | 0.2 | 10 | 1.5 | 2 | Air – acetylene |
| Fe | 248.3 | 0.2 | 10 | 1.5 | 3 | Air - acetylene |
| Al | 309.3 | 0.2 | 5 | 3 | 3 | Nitrous oxide – acetylene |
| Ba | 553.6 | 0.2 | 10 | 4 | 3 | Nitrous oxide - acetylene |
| Sb | 217.6 | 0.2 | 8 | 1.5 | 4 | Air – acetylene |

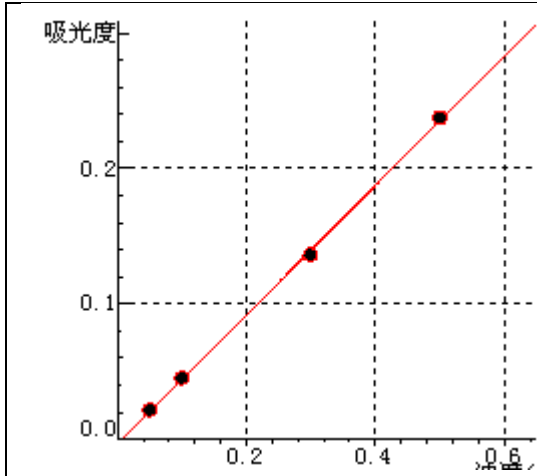
4 Standard solution preparation

For each 100mL Mg and Ca standard solution, 1.5 mL 100 g/L lanthanum chloride solution should be added respectively. K and Na standard solutions can be mixed.

| Element | Concentration ($\mu\text{g/mL}$) | | | | |
|---------|------------------------------------|------|------|------|------|
| K | 0 | 0.05 | 0.1 | 0.3 | 0.5 |
| Na | 0 | 0.05 | 0.1 | 0.3 | 0.5 |
| Ca | 0 | 0.5 | 1.0 | 2.0 | 3.0 |
| Mg | 0 | 0.01 | 0.05 | 0.1 | 0.2 |
| Fe | 0 | 0.1 | 0.3 | 0.6 | 1.2 |
| Al | 0 | 5 | 10 | 15 | 20 |
| Ba | 0 | 2.0 | 4.0 | 8.0 | 10.0 |
| Sb | 0 | 5.0 | 10.0 | 15.0 | 20.0 |

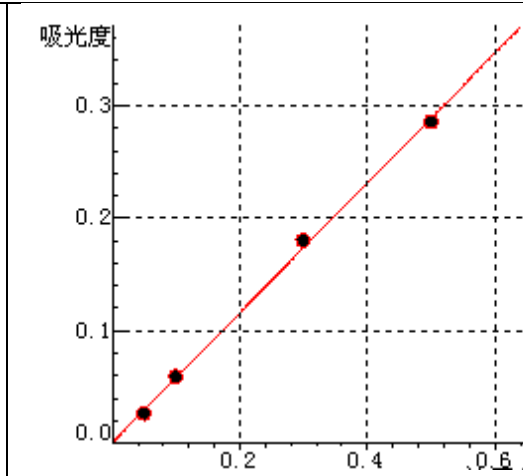
5 Standard curve

When measuring Ca, Mg sample solution, the concentration of lanthanum chloride needs to be the same as that in standard solution.



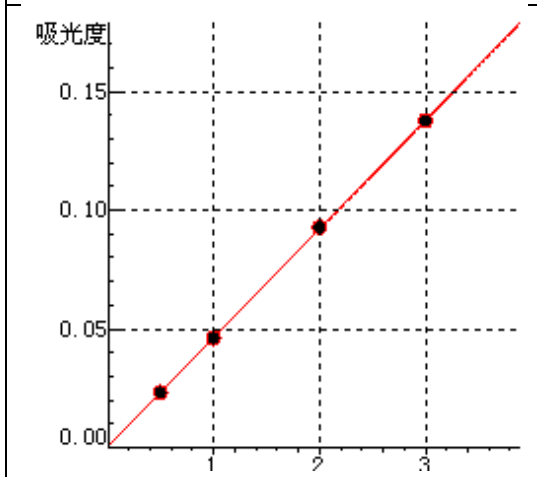
K Curve equation: $Y=0.47709*X-0.00328$

linearity coefficient: 0.99960



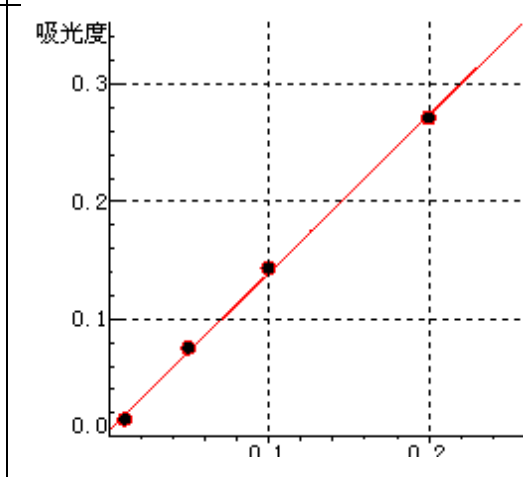
Na curve equation: $Y=0.57620*X+0.00075$

linearity coefficient: 0.99930



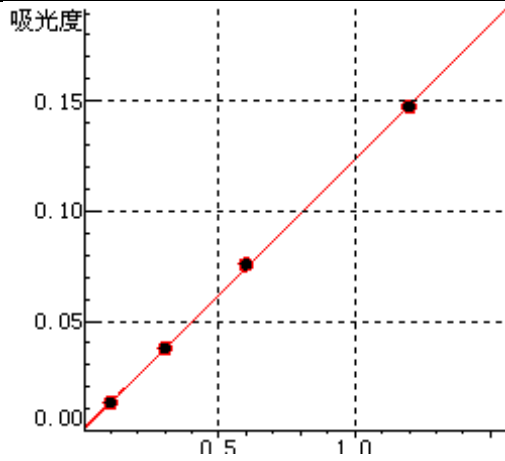
Ca curve equation: $Y=0.04572*X+0.00056$

linearity coefficient: 1.00000



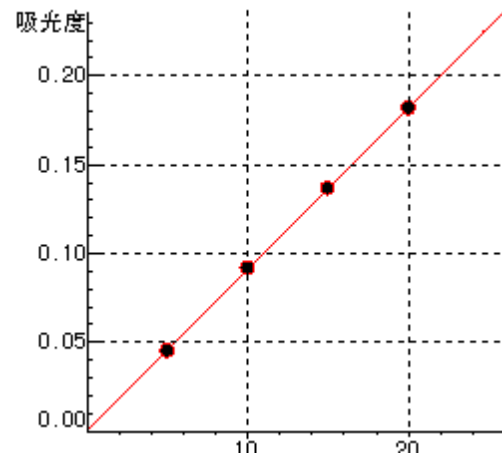
Mg curve equation: $Y=1.33995*X+0.00523$

linearity coefficient: 0.99940



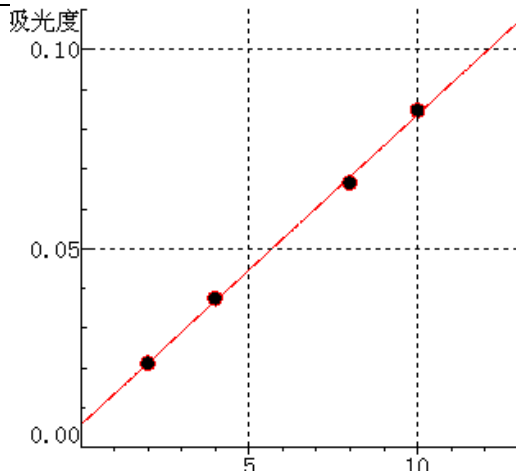
Fe curve equation: $Y=0.12233*X+0.00107$

linearity coefficient: 0.99990



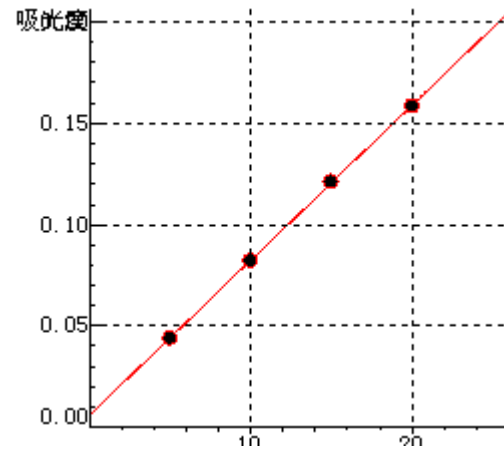
Al curve equation: $Y=0.00909*X+0.00025$

linearity coefficient: 1.00000



Ba curve equation: $Y=0.00781*X+0.00564$

linearity coefficient: 0.99920



Sb curve equation: $Y=0.00764*X+0.00595$

linearity coefficient: 1.00000