

Determination of Ca and Fe Contents in Solid Fibers

1 Sample solution preparation :

0.2g sample (precision to 0.0001g) was weighed and placed in PTFE inner can, added with 5mL nitric acid, mixed well and left overnight. 3mL hydrogen peroxide was added, covered with inner lid and placed into stainless steel jacket, outer cover was tightened. The digestion can was placed in an oven, heated at 140 °C for 3.5h until the digestion completes, then it was cooled down naturally to room temperature. The inner can was removed, placed on hot plate, heated to reduce acid to about 1mL, removed for cooling. It was transferred to a 50mL volumetric flask with deionized water and diluted up to the volume, shaken well & spared for later use.

2 Experimental equipment and reagents :

AA7000 series atomic absorption spectrophotometer (with Ca, Fe hollow cathode lamp, EWAI Inc.)

High pressure digestion can

Constant temperature blast drying oven

Nitric acid (HNO₃): excellent grade purity

Hydrogen peroxide (H₂O₂): excellent grade purity

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

Calcium standard solution (National Reference Materials Research Center)

Iron 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
Ca	422.7	0.2	10	1.5	3	Air - acetylene
Fe	248.3	0.2	10	1.5	3	Air - acetylene

4 Standard solution preparation

For each 100mL Ca standard solution, 1.5 mL 100 g/L lanthanum chloride solution is to be added.

Element	Concentration (µg/mL)				
Ca	0	0.5	1.0	1.5	2.0
Fe	0	0.1	0.25	0.5	1.0



5 Standard curve

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

