

## Determination of Mg, Ni, K and Na in Molybdenum Powder

### 1 Sample solution preparation :

2g sample was weighed and placed in a PTFE beaker, added with 10mL hydrogen peroxide, placed on a hot plate and heated at low heat for digestion. After digestion, it was removed to cool back to room temperature, then 3mL nitric acid was added. As the reaction completes, it was transferred into a 50mL volumetric flask, the beaker inner wall was rinsed with deionized water at least 3 times, then collected in the volumetric flask. It was made up to the volume, spared for later use.

### 2 Experimental equipment and reagents :

AA7000 series atomic absorption spectrophotometer (with Mg, Ni, K, Na hollow cathode lamp, EWAI Inc.)

Hydrogen peroxide: excellent purity

PTFE beaker

Hot plate

Nitric acid (HNO<sub>3</sub>): excellent grade purity

Mg standard solution (National Reference Materials Research Center)

Ni standard solution (National Reference Materials Research Center)

K standard solution (National Reference Materials Research Center)

Na 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	4	Air – acetylene
Mg	285.2	0.2	10	1.5	3	Air - acetylene

Element	Wavelength ( $\lambda$ /nm)	Lamp current (I/mA)	D2 current (I/mA)	Spectral bandpass width ( $\Delta\lambda$ /nm)	Background correction
Ni	232.0	1.3	100	0.2	D2 lamp



### Graphite furnace temperature curve

No	Step	Start temp (°C)	End temp (°C)	Heating time (s)	Inner gas path	Auxiliary gas path	Mode	Alarm
1	Drying	40	100	35	Open	Off	Power	Open
2	Drying	100	120	10	Open	Off	Power	Open
3	Ashing	120	1000	10	Open	Off	Power	Open
4	Ashing	1000	1000	8	Open	Off	Power	Open
5	Ashing	1000	1000	6	Off	Off	Power	Open
6	Atomization	2700	2700	3	Off	Off	Power	Open
7	Cleaning	2700	2700	2	Open	Off	Power	Open
8	Cooling	0	0	25	Open	Off	Power	
9	Cooling	0	0	1	Off	Off	Power	Open

#### 4 Standard solution preparation

Each 100 mL K and Na standard solution should be added with 1.5 mL 100 g/L cesium chloride solution.

Each 100 mL Mg standard solution should be added with 1.5 mL 100 g/L La solution.

Element	Concentration				
K (µg/mL)	0	0.3	0.6	0.9	1.2
Na (µg/mL)	0	0.004	0.008	0.012	0.016
Mg (µg/mL)	0	0.03	0.06	0.09	0.12
Ni (µg/L)	0	5.0	10.0	20.0	30.0

### 5 Standard curve

