Measurement of minerals and metals by Inductively Coupled Plasma –Atomic Emission spectrophotometric (ICP-AES) Technique [1]

Preparation of standard solutions

Individual standard solutions (1000 mg/L) of Na, K, Ca, Mg, P, Al, Fe, Cu, Zn, Co, Mo, Mn, Pb, Cd, and As: Alternatively, multi-element standard solution (1000 mg/L of each standard) can be used. The linear range of the standard curve may differ based upon the instrument used and operation mode of the torch (axial or radial).

Prepare a series of mixed working standard solutions in the range of 0.1 to 50 µg/ml following serial dilution technique from the mixed standard solution (100 µg/ml). Ensure to limit dilution factor under each dilution step to less than 25.

Instrumental conditions

Select appropriate emission wavelengths to be used with each element under consideration. The recommended settings for various instrumental parameters may vary from one instrument to the other as well as model to model. Certain parameters require optimization at the time of use to obtain the best results. Instrumental conditions should therefore be optimized as described by the manufacturer. Typical emission wavelengths, curve types and calibration ranges given below are for guidance purposes and analyst needs to select the right emission wavelength and other parameters based on the instrument used and type of sample analyzed (expected interferences).

S.No	Analyte	Emission Wavelength (nm)	Curve Type	Calibration Range, µg/ml
1	Na	589.520, 588.995	Quadratic	0.50 – 20.0
2	K	766.491	Quadratic	0.50 – 20.0
3	Ca	318.127	Quadratic	0.50 – 20.0
4	Mg	279.079	Quadratic	0.50 – 20.0
5	P	213.618	Linear	0.10 – 20.0
6	Al	257.509, 308.215, 396.152	Linear	0.10 – 20.0
7	Fe	259.940	Linear	0.10 – 20.0
8	Cu	224.700, 324.754	Linear	0.10 – 10.0
9	Zn	213.857	Linear	0.10 – 20.0
10	Co	228.616, 235.341	Linear	0.10 – 10.0
11	Mo	202.032	Linear	0.10 – 10.0
12	Mn	257.610	Linear	0.10 – 10.0
13	Pb	220.353	Linear	0.10 – 10.0
14	Cd	226.502	Linear	0.10 – 5.0
15	As	188.98, 193.696	Linear	0.10 – 5.0
16	Sb	206.833	Linear	0.10 – 20.0
17	Ba	455.403	Linear	0.10 – 20.0
18	Cr	267.716	Linear	0.10 – 20.0

[1] This method is added to the section *Inorganic Components*, subsection *Metallic Impurities* of Volume 4 of the Combined Compendium of Food Additive Specifications (on page 66).

S.No	Analyte	Emission Wavelength (nm)	Curve Type	Calibration Range, µg/ml
19	Si	251.611	Linear	0.10 – 20.0
20	Ni	231.604	Linear	0.10 – 20.0
21	Ti	334.941	Linear	0.10 – 20.0

Procedure:

Set the instrumental parameters suitable for the analysis of analytes under consideration and the likely interferences. Setup the instrument, aspirate the standard blank solution and set the instrument to zero. Aspirate standards and construct standard curve for each element using the emission intensity and concentration of the element in the working standard. Check the coefficient of determination (R^2) and it shall be >0.99. Aspirate sample solution (or diluted sample solution) and deduce the concentration of the element in the solution (µg/ml).

Element (mg/kg) = (A-B) x V x DF/ x W

Where:

A is the concentration of element in the sample solution, µg/ml
B is the concentration of element in the corresponding reagent blank solution, µg/ml
V is the volume of sample made up, ml
DF is the dilution factor
W is the weight of sample, g