Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)

Instrumentation and Sample Preparation

Instrumentation / Model:

- Agilent 5110 ICP-OES
- Uses a plasma torch (10,000 K) to excite atoms and ions
- These emit characteristic wavelengths of light that are unique to each element (optical emission) that can be detected.

Sample Preparation:

1. Alloy is crushed, snapped, or milled into a powder or grain.

2. Sample is weighed and dissolved using hotplate digestion with a mixture of strong mineral acids (HCl, HNO_3, HF, H_2SO_4, H_3PO_4) – typically aqua regia is used.

3. Sample is diluted to volume using a volumetric flask, with deionised water.

Figure 2: Inductively Coupled Plasma

Figure 1: Theory of Operation of an ICP-OES Diagram

Figure 3: Aqueous Inorganic Samples

Figure 4: Agilent 5110 ICP-OES

How does it work?

Sample Introduction:

An autosampler takes up the sample using a peristaltic pump (up to 84 samples per analysis). *(Figure 1)*

The aqueous sample is sprayed through a concentric nebuliser which produces an ultra-fine mist. Any large droplets condense in the spray chamber and are drained to the waste.

The Plasma:

Argon (Ar) gas is pumped into the torch, where a radiofrequency (RF) coil operates at 40 MHz. This produces a chain reaction that creates an Ar-plasma, which is approximately 10,000 K. *(Figure 2)*

The sample enters the plasma (via the torch) where it is:
1. Vapourised
2. Desolvated
3. Atomised
4. Ionised

During stages 3 and 4 the analyte enters an optically excited state – after cooling this excited state emits a photon of characteristic wavelength unique to that element.

Detection:

The emitted light passes through a prism and an echelle grating – this separates the light into individual wavelengths. These wavelengths are detected by a solid-state chip which converts the intensity into a concentration value.

Figure 5: Typical Optical Emission Spectra and Calibrations for Samarium (left) and Iron (right)

What does it detect?

Elements of Interest:

The ICP-OES can detect all metallic and metalloid elements. *(Figure 4)* At Less Common Metals some elements of interest are **Sm, Co, Fe, Nd, Pr, Dy, Ti, B.** *(Figure 5)*

These elements can be detected from 5 parts-per-million (ppm) all the way up to 100 % due to a dual-mode of viewing the atomic emission spectra (axial and radial viewing).

ICP-OES analysis is simultaneous, and it can analyse up to 72 elements quickly (1-2 minutes) making it a high throughput measurement for trace- and major-element analysis. (C, S, O, N, H cannot be detected d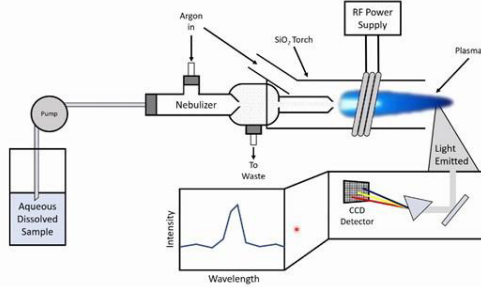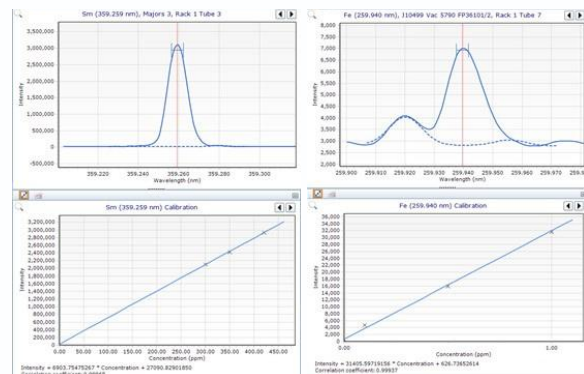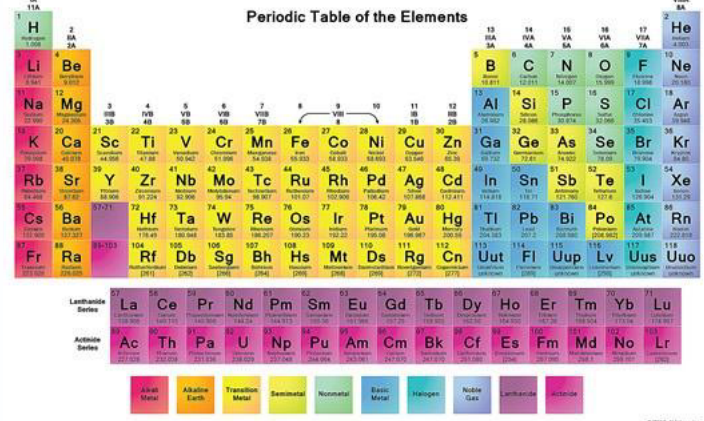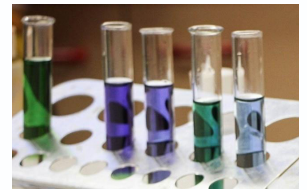ue to their presence in common solvents and acids).