

Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

- elemental analysis
- main to trace components
- quantitative analysis

Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) is one of the most common techniques for elemental analysis. Its high specificity, multi-element capability and good detection limits result in the use of the technique in a large variety of applications. All kinds of dissolved samples can be analyzed, varying from solutions containing high salt concentrations to diluted acids. A plasma source is used to dissociate the sample into its constituent atoms or ions, exciting them to a higher energy level. They return to their ground state by emitting photons of a characteristic wavelength depending on the element present. This light is recorded by an optical spectrometer. When calibrated against standards the technique provides a quantitative analysis of the original sample.

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Principle of ICP-AES

In fig. 1 a schematic of an ICP-AES is shown. In the ICP-AES a plasma source is used to make specific elements emit light, after which a spectrometer separates this light in the characteristic wavelengths.

Sample introduction

A solid sample is normally first dissolved and mixed with water. The technique is robust enough to allow direct analysis of liquids. The sample solution is transformed into an aerosol by a so-called nebuliser. The bigger droplets are separated from the smallest in a specially spray-chamber. The smallest droplets (1-10 μm) are transferred by an argon flow into the heart of the ICP-AES, the argon plasma. The bigger droplets (>90%) are pumped to waste.

Plasma

To produce strong atomic emission from all chemical elements it is necessary to attain temperatures considerably above those available from simple flames. The highest amount of atomic emission is reached at temperatures in the range of 7,000 K to 10,000 K.

A convenient means of obtaining these temperatures is to generate an inert-gas plasma. Plasma is a gaseous state of matter containing major concentrations of essential free electrons and highly charged ions. It is a very effective medium for volatilization and atomization (and ionization) of liquid droplets. When the aerosol droplets enter the hot area of the plasma (front page) they are converted into salt particles by

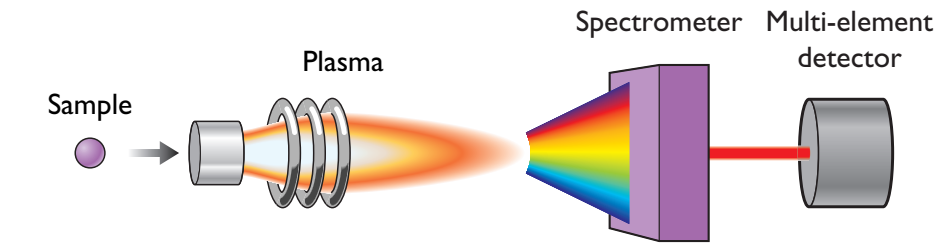


Fig. 1: Schematic of an ICP-AES.

desolvation. These salt particles are split into individual molecules that will subsequently fall apart to atoms and ions (fig. 2). Most elements get ionized very efficiently (> 90%). Almost no molecules and ground state atoms are present in a plasma.

In the plasma, even more energy is transferred to the atoms and ions, promoting the excitation of their electrons to higher energy levels.

When these excited atoms and ions return to

their ground state or to lower excitation states they will emit electromagnetic radiation (fig. 3) in the ultra-violet/visible range of the spectrum.

Each excited element emits specific wavelengths (λ), i.e. has a typical emission spectrum. The intensity of the radiation is proportional to the element concentration. Commercially available standards can be used to calibrate the ICP-AES, which makes it possible to perform highly quantitative analysis.

Fig. 2: Processes that take place in an ICP.

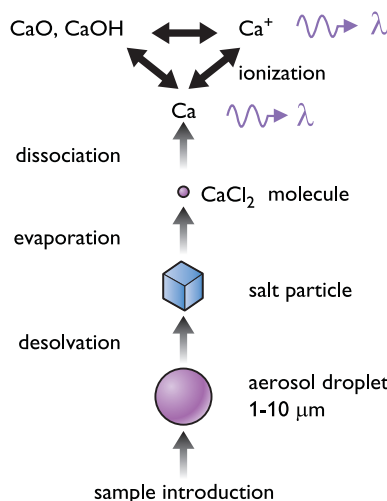


Fig. 3: Process of excitation of atoms resulting in generation of element characteristic wavelengths.

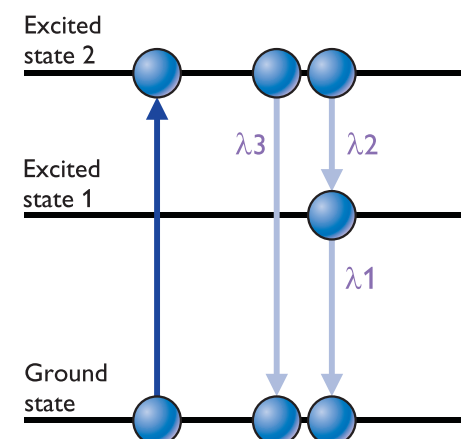




Fig. 4: Schematic of the different parts of a monochromator.

Spectrometer and Detector

A spectrometer, a multi-component part (fig. 4) containing mirrors, prisms etc, is used to separate the specific wavelengths of interest. Because atomic emission lines are very narrow lines, a high-resolution detector is essential. Simultaneous detection makes it possible to measure all elements of interest at the same time. This is an advantage because it limits signal variations introduced by sample preparation. Most often a CCD detector is used, which provides both advantages (high resolution and simultaneous detection).

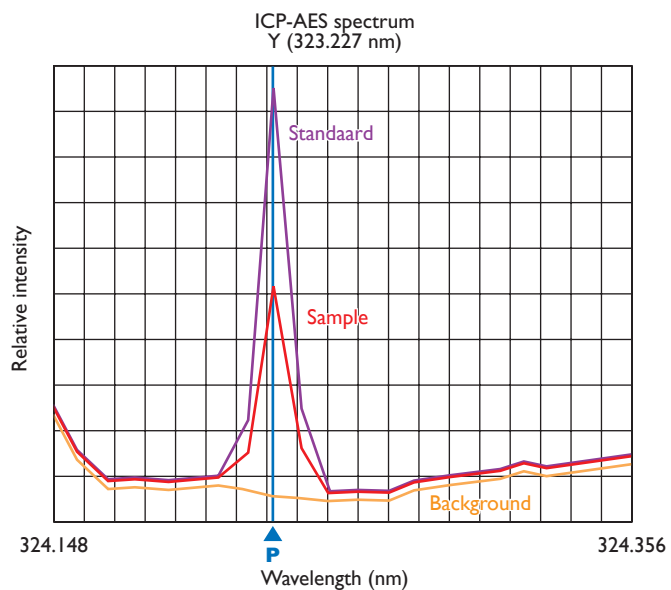
The emission spectrum

An emission spectrum is a plot of the radiation intensity (y-axis) versus the wavelength (x-axis) (fig. 5). In a plasma predominantly singly charged ions are produced, but also free atoms are present. Both will be excited and emit radiation. Because of the many different transitions possible between the various excited states (sodium has six excited states which produce fifteen wavelengths) an emission spectrum can be complicated. The different wavelengths emitted by the atoms and ions can overlap with wavelengths produced by other elements. For each element the natural emitted wavelengths are constant in nature. Therefore overlap of emission from different elements can be predicted and taken into account.

Applications

- analysis of glass
- monitoring elements of interest in process solutions
- quantitative analysis of traces of catalyst in polymers
- quantitative analysis of contaminations in thin layers

Fig. 5: Typical emission spectrum.



Time in months	Fe (µg)			Ni (µg)		
	0	1	3	0	1	3
Material 1	< 1	145	199	< 1	64	87
Material 2	< 1	45	51	< 1	9	10
Material 3	< 1	< 1	< 1	< 1	< 1	< 1

Table 1: Dissolution of Fe and Ni in ultra-pure water.

Typical analyses

Material balance in a glass oven

In glass factories massive amounts of glass are produced. During this process raw materials are mixed with cullet and this so-called batch is brought into a melting tank. Here the materials melt to liquid glass at a temperature of about 1500°C. During this process material is lost due to evaporation and the formation of dust clouds. Dust clouds are formed immediately after the batch has been brought into the melting tank, at the beginning of the furnace.

To determine the origin of such dust clouds the atmosphere of the melting tank is sucked through a set of washing bottles, filled with water, where the particles dissolve or precipitate. The sampled material is then treated with acids so that all particles are dissolved. From this solution the composition of the dust is determined using ICP-AES (table 2). The relative accuracy of the analysis is 3%.

The analysis clearly helps understand the processes involved in the loss of raw material in dust clouds and through evaporation. This in turn is very important to learn more about the process of melting and the effect of evaporation and dust on the corrosion of the materials that are used to build a glass furnace.

Corrosion behaviour of metal compounds

In many production processes parts of machinery are cooled using ultra pure water. The water flows through pipes inside the

machinery absorbing heat. During cooling the water is in contact with metal parts of the machinery that can suffer from corrosion.

In this typical example the corrosion behaviour of different parts of machinery was tested. As during corrosion the main components (Fe and Ni) dissolve, their concentration in the water can be used to quantify the problem. Metal parts were put into ultra pure water, and analyzed using ICP-AES after different periods of time. The results of the analysis are shown in table 1, which shows that only "material 3" has no detectable corrosion problems.

Table 2: Typical ICP-AES analysis of glass dust clouds.

	Sample 1 (µg/ml)
Na	45.0
K	150
Mg	1.59
Ca	2.92
Ba	33
Sr	42.1
Zr	4.4
V	0.11
Cr	0.15
Fe	0.23
Ni	0.16
Zn	0.08
Si	13.9
Pb	50.6
As	0.05
Sb	23
S	15.8
Al	2.9
P	0.4
Co	0.003

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Characteristics

Obtained information

- elemental composition

Sample type

- solids (after dissolution) and liquids
- bulk material and thin films
- both organic and inorganic materials

Detection limits

- most elements less than 0.005 mg/l

Accuracy

- 1-3 % relative

Analytical range

- trace (µg/g) to main (%) components

