Instrumental Methods of Analysis

Atomic Emission Spectroscopy

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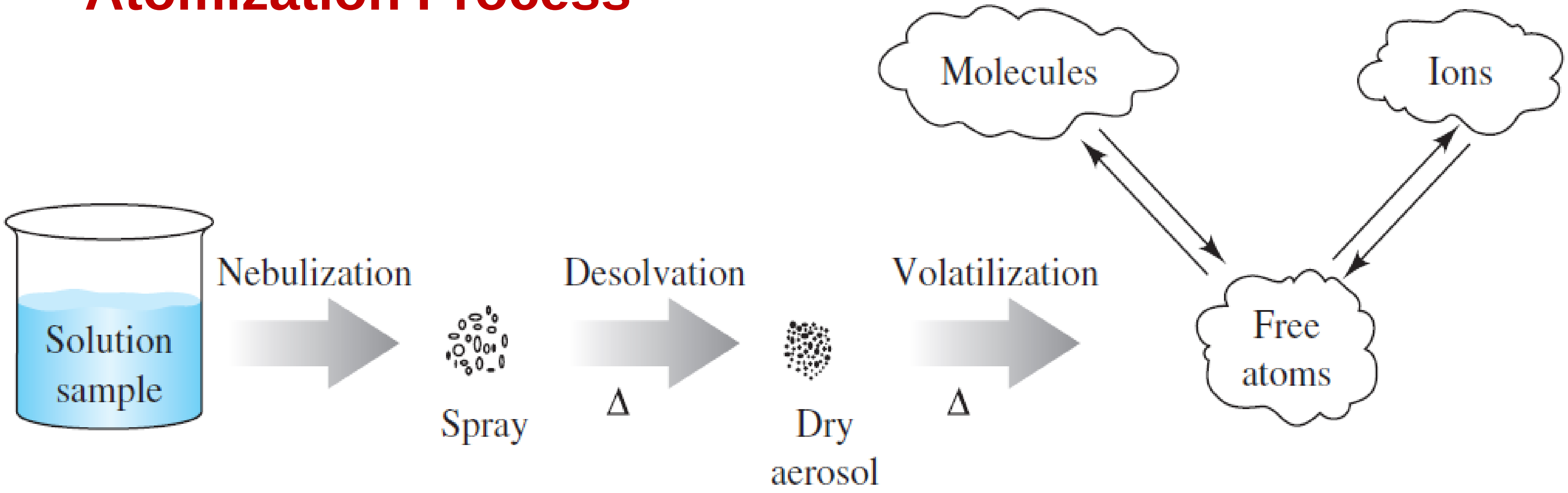
Atomization Process



Processes leading to atoms, molecules, and ions with continuous sample introduction into a **plasma** or **flame**. The solution sample is converted into a spray by the nebulizer. The high temperature of the flame or plasma causes the solvent to evaporate leaving dry aerosol particles. Further heating volatilizes the particles producing atomic, molecular, and ionic species. These species are often in equilibrium at least in localized regions.

Several methods that are used to atomize samples for atomic spectroscopy. Inductively coupled plasmas, flames and electrothermal atomizers are the most widely used atomization methods. Flames and electrothermal atomizers are found in atomic absorption spectrometry, while the inductively coupled plasma is used in optical emission and in atomic mass spectrometry.

Classification of Atomic Spectroscopic Methods

Atomization Method	Typical Atomization Temperature ºC	Types of Spectroscopy
Flame	1700–3150	-Absorption -Emission -Fluorescence
Electrothermal	1200–3000	-Absorption -Fluorescence
Inductively coupled plasma	6000–8000	-Emission -Mass
Direct-current plasma	5000–10,000	-Emission
Electric arc	3000–8000	-Emission
Electric spark	Varies with time and position	-Emission

Flame Sources

Both atomic and molecular emission and absorption can be measured when a sample is atomized in a flame.

A sample of a material (analyte) is brought into the flame as either a gas, sprayed solution, or directly inserted into the flame by use of a small loop of wire, usually platinum.

The heat from the flame evaporates the solvent and breaks chemical bonds to create free atoms. The thermal energy also excites the atoms into excited electronic states that subsequently emit light when they return to the ground electronic state. Each element emits light at a characteristic wavelength, which is dispersed by a grating or prism and detected in the spectrometer.

A frequent application of the emission measurement with the flame is the regulation of alkali metals for pharmaceutical analytics.

Plasma Sources

A **plasma** consists of a hot, partially ionized gas, containing an abundant concentration of cations and electrons that make the plasma a conductor.

The plasmas used in atomic emission are formed by ionizing a flowing stream of argon, producing argon ions and electrons. The high temperatures in a plasma result from resistive heating that develops due to the movement of the electrons and argon ions.

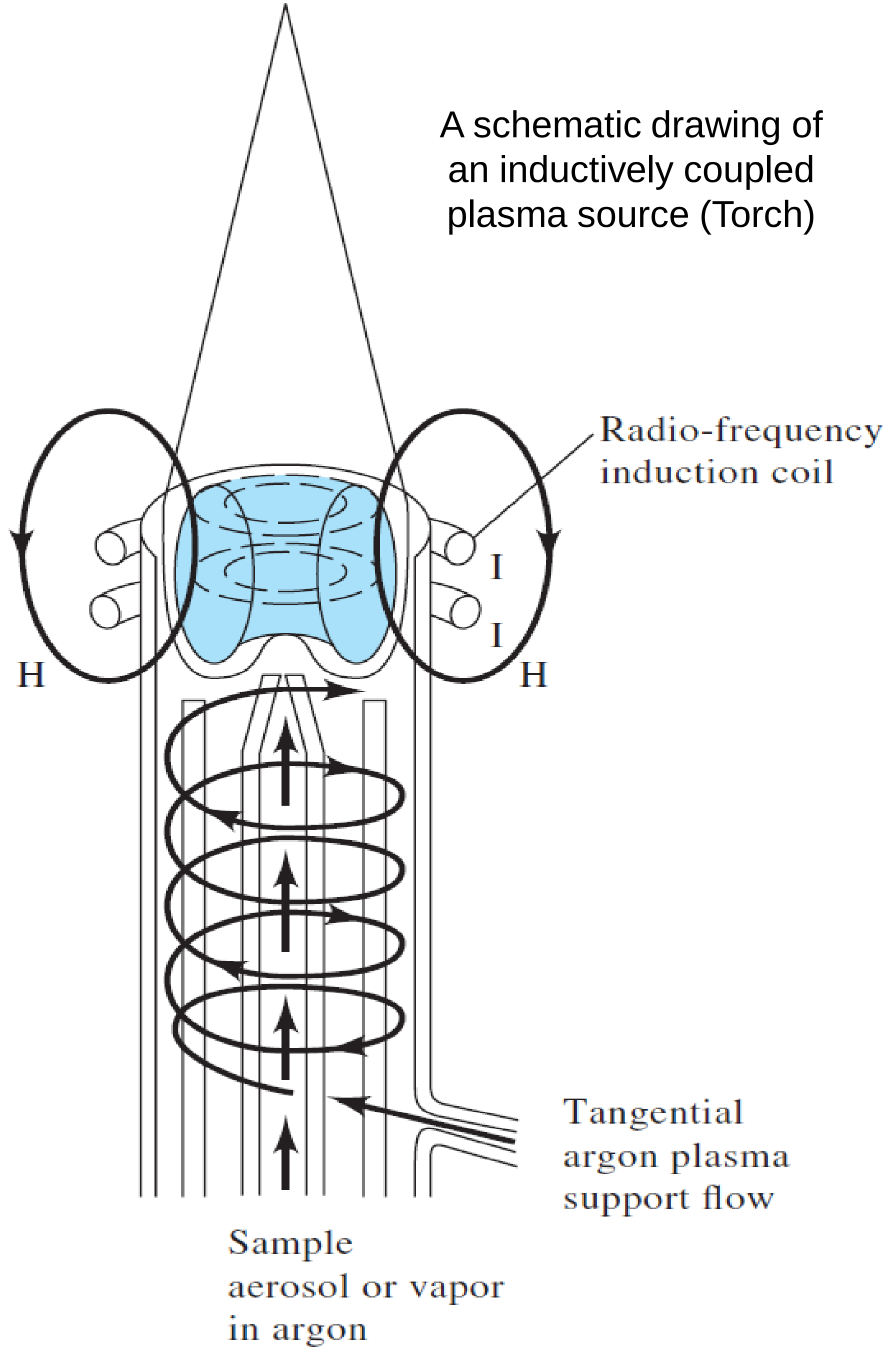
Because plasmas operate at much higher temperatures than flames, they provide better atomization and more highly populated excited states. Besides neutral atoms, the higher temperatures of a plasma also produce ions of the analyte.

Inductively Coupled Plasma (ICP)

The source consists of three concentric quartz tubes through which streams of argon flow at a total rate of between 11 and 17 L/min. Surrounding the top of the largest tube is a coil powered by a radio-frequency generator.

Ionization of the flowing argon is initiated by a spark from a Tesla coil. The resulting ions and their associated electrons then interact with the fluctuating magnetic field (labeled **H**) produced by the induction coil (labeled **I**). This interaction causes the ions and electrons within the coil to flow in the closed annular paths. The resistance of the ions and electrons to this flow of charge causes ohmic heating of the plasma (6000 to 8000 K) at a height of 15–20 mm above the coil, where emission is usually measured.

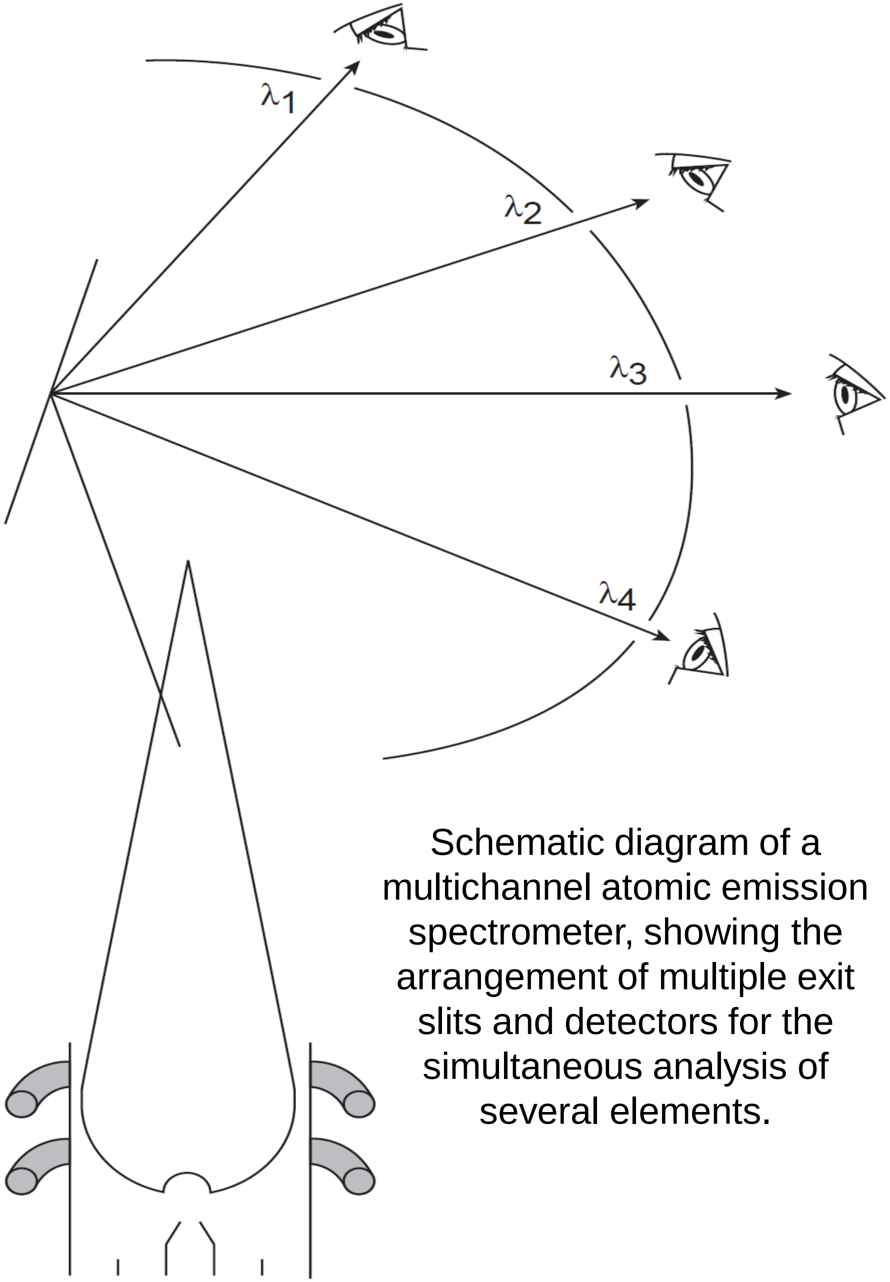
A schematic drawing of an inductively coupled plasma source (Torch)



Multielemental Analysis

Atomic emission spectroscopy is ideally suited for multielemental analysis because all analytes in a sample are excited simultaneously. A scanning monochromator can be programmed to move rapidly to an analyte's desired wavelength, pausing to record its emission intensity before moving to the next analyte's wavelength.

Another approach to multielemental analysis is to use a multichannel instrument that allows for the simultaneous monitoring of many analytes. A simple design for a multichannel spectrometer consists of a standard diffraction grating and 48–60 separate exit slits and detectors positioned in a semicircular array around the diffraction grating at positions corresponding to the desired wavelengths.



Schematic diagram of a multichannel atomic emission spectrometer, showing the arrangement of multiple exit slits and detectors for the simultaneous analysis of several elements.

Instrumentation

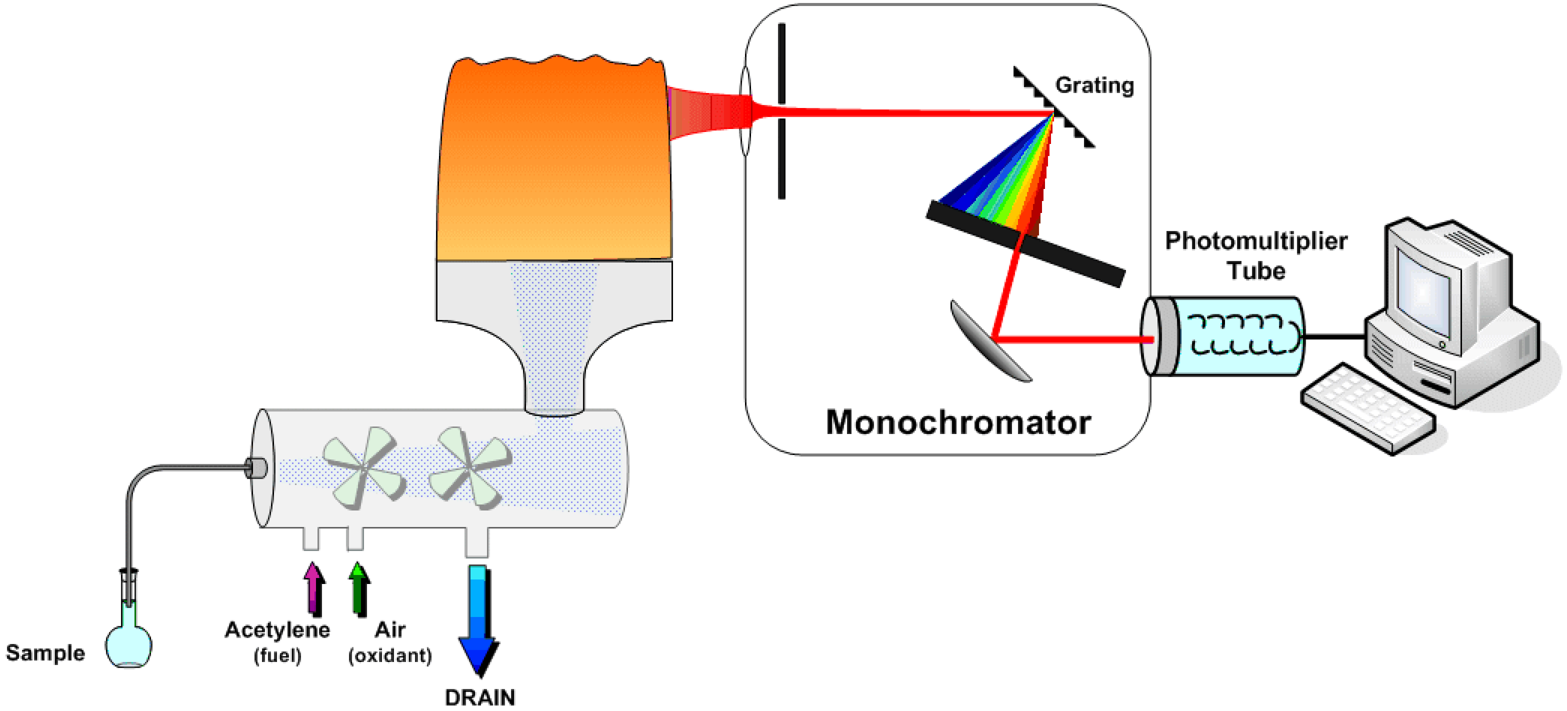
Instrumentation for atomic emission spectroscopy is similar in design to that used for atomic absorption.

In fact, most flame atomic absorption spectrometers are easily adapted for use as flame atomic emission spectrometers by turning off the hollow cathode lamp and monitoring the difference between the intensity of radiation emitted when aspirating the sample and that emitted when aspirating a blank.

Many atomic emission spectrometers, however, are dedicated instruments designed to take advantage of features unique to atomic emission, including the use of plasmas, arcs, sparks, and lasers, as atomization and excitation sources and have an enhanced capability for multielemental analysis.

Difference between
Atomic Absorption and Atomic Emission

Atomic Emission Spectroscopy



(Animation)


Thank You!