Inductively Coupled Plasma-Atomic Emission Spectrometry

Inductively coupled plasma-atomic emission spectrometry is designed to determine the amount (concentration) of a test element in a sample, by atomizing and exciting the element by inductively coupled plasma (ICP), and determining the intensity of atomic emission spectral line.

Apparatus Usually, the apparatus consists of an excitation source part, a sample introduction part, a light emission part, a spectroscope part, a photometry part, an indication and recording part. The excitation source part is composed of an electric power source, a control system, and circuit to supply and control the electric energy which excites and emits an element in a sample. This part also includes a gas source and a cooling system. The sample introduction part is composed of a nebulizer and a spray chamber. The light emission part is composed of a torch and an high-frequency induction coil. The spectroscope part is composed of a light-converging system and a spectroscope such as a diffracting grating. The photometry part is composed of a detector and a signal processing system. The indication and recording part is composed of a display and a recording system. The ICP-atomic emission spectrometry includes single-element-sequential-type- and multiple-element-sequential-type-measuring methods using a wavelength scanning spectroscope, and a simultaneously measuring method using a wavelength-fixed-type polychrometer.

Procedure Confirm that all live parts are normal. Switch on the excitation source part and the cooling system. When a vacuum-type spectroscope is used to measure the emission line in vacuum-ultraviolet region, purge sufficiently the light-path between the light emission part and the spectroscope with argon or nitrogen gas. Set the flow rate of argon or nitrogen gas to the specified rate, switch on the high frequency power, and generate the plasma. Correct the wavelength of spectroscope with the emission spectral line of a mercury lamp. Introduce the test solution and the standard solution or control solution prepared as specified in the individual monograph and measure the emission intensity of an appropriate emission line of the object element.

Usually, the determination is done using one of the following methods. In the determination, the interference and background should be corrected.

(1) Calibration Curve Method Prepare standard solutions of three or more different concentrations, measure the emission intensities of these standard solutions, and prepare a calibration curve from the obtained values. Then, measure the emission intensity for the test solution with a concentration adjusted to a measurable range, and determine the amount (concentration) of the object element from the calibration curve.

(2) Standard Addition Method To equal volumes of three or more test solutions, add to each the standard solution so that the stepwise increasing amounts of the object element are contained in the solutions, and add the solvent to make a definite volume. Measure the emission intensity for each solution, and plot the amounts (concentrations) of added standard object element on the abscissa and the emission intensities on the ordinate on the graph paper. Extend the calibration curve obtained by linking the plots, and determine the amount (concentration) of object element from the distance between the origin and the intersecting point of the calibration curve on the abscissa. This method is applicable only when the calibration curve drawn as directed in section (1) above is a straight line passing through the origin.

(3) Internal Standard Method Prepare several solutions containing a constant amount of the specified internal standard element, and known graded amounts of the standard object element. For these solutions, measure the emission intensities of the standard object element and internal standard element at the analytical wavelength of each element under the same measuring conditions, and obtain the ratios of each emission intensity of standard object element to the emission intensity of the internal standard element. Prepare a calibration curve by plotting the amounts (concentrations) of standard object element on the abscissa and the ratios of emission intensity on the ordinate. Then, prepare the test solutions, adding the same amount of internal standard element as in the standard solution. Proceed under the same conditions as for preparing the calibration curve, obtain the ratio of the emission intensity of standard object element to that of internal standard element, and determine the amount (concentration) of the object element from the calibration curve.

Note: For this test, avoid the use of reagents, test solutions, and gases which interfere with the determination.