Atomic Absorption Spectrophotometry

Atomic Absorption Spectrophotometry is designed to determine the amount (concentration) of an object element in a sample, utilizing the phenomenon that the atoms in the ground state absorb the light of characteristic wavelength passing through an atomic vapor layer of the element.

Apparatus Usually the apparatus consists of a light source, a sample-atomizer, a spectroscope, and a photometer, and a recording system. Some are equipped with a background compensation system. For the light source, a hollow cathode lamp and a discharge lamp are mainly used. To the sample-atomizer, the flame type, electrothermal type, and the cold-vapor type are applied.

The cold-vapor flameless type is categorized as the two methods: reduction vaporizing method and heat vaporizing method. The flame type is composed of a burner and a gas-flow regulator, the electrothermal type is composed of an electric furnace and a power source, and the cold-vapor type is composed of a mercury generator by chemical reduction-vaporization and thermal reduction-vaporization and an absorption cell. For the spectroscope, a grating for light diffraction or an interference filter prism is used. The photometer mainly consists of a detector and a signal treatment system. A recording system is composed of a display and a recording device. A background compensation system is employed for the correction of matrix effects on the measuring system. Several principles can be utilized for background compensation, using the continuous spectrum sources, the Zeeman split spectrum, the nonresonance spectrum, or the self-inversion phenomena.

Procedure Unless otherwise specified, proceed by either of the following methods:

(1) Flame Type Fit the specific light source lamp to the lamp housing, and switch on the instrument. Light the source lamp, adjust the wavelength dial of the spectroscope to the wavelength of the analytical line specified, and set at an appropriate current value and slit-width. Using the supporting gas and combustible gas specified, ignite the mixture of these gases, adjust the gas flow rate and pressure, and make the zero adjustment after nebulizing the solvent into the flame. Nebulize the test solution or the standard solution or control solution prepared by the method prescribed elsewhere, and measure the absorbance.

(2) Electrothermal type Fit the specific light source to the lamp housing and switch on the instrument. After lighting the lamp and selecting the analytical wavelength specified in the individual monograph, set an appropriate electric current and slit-width. A suitable amount of sample solution, standard solution, or control solution, prepared as specified in the individual monograph, is injected to the furnace and an appropriate stream of inert gas is made to flow through the furnace. The

specimen is dried and ashed, and the element included is atomized, on heating at appropriate temperature for an appropriate time in appropriate mode. The atomic absorption specified is observed and the intensity of absorption is measured.

(3) Cold-vapor Type Fit the light source lamp specified on the photometer. Light the source lamp, adjust the wavelength dial of the spectroscope to the wavelength of the analytical line specified, and set at an appropriate current value and a slit-width. Then, in the reduction vaporizing method, transfer the test solution or the standard solution or control solution to the closed vessel, reduce to the element by addition of a proper reducing agent, and vaporize. In the heat vaporizing method, vaporize the sample by heating. Measure the absorbance of the atomic vapor generated by these methods.

Usually, the determination can be done by an appropriate one of the methods given below. In the determination, the interference and background should be considered.

(1) Calibration Curve Method Prepare standard solutions of at least three different concentrations, measure the absorbances of these standard solutions, and prepare a calibration curve from the obtained values. Then measure the absorbance for the test solution adjusted in concentration 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 more than 2 of different test solutions, add 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 absorbance for each solution, and plot the amounts (concentration) of added standard object element on the abscissa and the absorbances on the ordinate on 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 in the case that the calibration curve drawn as directed in (1) above passes through the origin.

(3) Internal Standard Method Prepare several solutions containing a constant amount of the prescribed internal standard element, and known, graded amounts of the standard object element. Using these solutions, measure the absorbances of the standard object element and the absorabance of internal standard element at the analytical wavelength of each element under the same measuring condition, and obtain the ratios of each absorbance of standard object element to the absorbance 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 absorbance 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 absorbance 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 and test solutions which can interfere with the determination.