



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0121 : 2006

(JAIMA/JSA)

**General rules for atomic absorption
spectrometry**

ICS 71.040.50

Reference number : JIS K 0121 : 2006 (E)

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Analytical Instruments Manufacturers Association (JAIMA)/ Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14. Consequently **JIS K 0121 : 1993** is replaced with this Standard.

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Date of Establishment: 1970-10-01

Date of Revision: 2006-10-20

Date of Public Notice in Official Gazette: 2006-10-20

Investigated by: Japanese Industrial Standards Committee

Standards Board

Technical Committee on Chemical Analysis

JIS K 0121 : 2006, First English edition published in 2007-04

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

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Printed in Japan

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General rules for atomic absorption spectrometry

1 Scope This Japanese Industrial Standard specifies general rules for quantitative analysis using an atomic absorption spectrometric apparatus.

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0050 *General rules for chemical analysis*

JIS K 0083 *Methods for determination of metals in flue gas*

JIS K 0101 *Testing methods for industrial water*

JIS K 0102 *Testing methods for industrial wastewater*

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 0212 *Technical terms for analytical chemistry (optical part)*

JIS K 0215 *Technical terms for analytical chemistry (analytical instrument part)*

JIS K 0222 *Methods for determination of mercury in stack gas*

JIS K 0557 *Water used for industrial water and wastewater analysis*

3 Terms and definitions For the purposes of this Standard, the definitions given in **JIS K 0050**, **JIS K 0211**, **JIS K 0212** and **JIS K 0215**, and the following definitions apply.

- a) **atomic absorption spectrometry** a method using the atomic absorption spectrometric apparatus to determine the concentration of an analyte element by converting it in a sample to an atom in ground state by means of flame, electric heating or chemical reaction, and by measuring the absorbance of its atomic vapour layer
- b) **flame atomic absorption spectrometry** an atomic absorption spectrometry using flame
- c) **deuterium lamp** a hydrogen discharge lamp filled with deuterium
Since it emits intensive continuous spectrum all over the area of an ultraviolet region, it is used as a light source for a background correction
- d) **low-pressure mercury lamp** a mercury discharge lamp of at most 133 Pa in pressure of mercury vapour
Since it emits intensively a resonance line of 253.7 nm, it is used as a light source for measurement of mercury
- e) **electrothermal atomic absorption spectrometry** a method of atomic absorption spectrometry by drying, incinerating and atomizing the sample solution in a furnace electrically heated of which heating elements are graphite or heat resistant metal

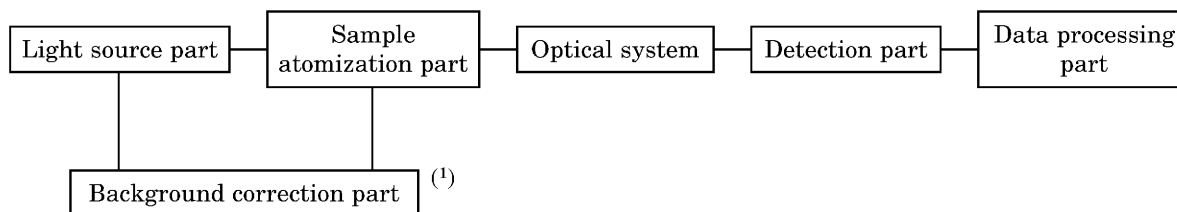
- f) **cold vapour atomic absorption spectrometry** a method of atomic absorption spectrometry for the atomic vapour obtained by reduction-vaporizing or heat-vaporizing the sample solution
- g) **atomic vapour generation device by reduction** a device to generate atomic vapour by reduction-vaporizing the analyte in the sample
- h) **atomic vapour generation device by heating** a device to generate atomic vapour by heat-vaporizing the analyte in the sample
- i) **background-correction system using continuous-spectrum sources** an optical system using a light source producing continuous spectrum for the background correction
- j) **background-correction system using Zeeman splitted spectrum** an optical system using a Zeeman splitted spectrum by a magnetic field for the background correction
- k) **background-correction system using nonresonance spectrum** an optical system using a neighbouring spectral line to the resonance line of an analyte element generated from the hollow cathode lamp for the background correction
- l) **background-correction system using self-reversal spectrum** an optical system using a self-reversal spectral line generated by discharge of a high current by applying alternately an ordinary current and the high current flow to the hollow cathode lamp for the background correction
- m) **mercury analyzer** measuring apparatus intended for mercury only in which the atomic absorption spectrometry for mercury atomic vapour generated by reduction-vaporizing or heat-vaporizing of mercury in a sample is performed
- n) **thermal vaporization-gold amalgamation-cold vapour type** a system in which the atomic absorption spectrometry for atomic vapour obtained in such a way that mercury in a sample is made to produce atomic vapour by heat-vaporizing, this atomic vapour is concentrated in a mercury collection tube and then heating is performed

4 Type of analysis method The type of the analysis method includes atomic absorption spectrometry and atomic absorption spectrometry intended for mercury only.

NOTE : The atomic absorption spectrometry intended for mercury only refers to the atomic absorption spectrometry with the function specialized in mercury analysis.

5 Atomic absorption spectrometry

5.1 Setup of apparatus The atomic absorption spectrometric apparatus consists of a light source part, a sample atomization part, an optical system, a detection part and a data processing part. An example of the standard setup is as given in figure 1.



Note (1) The background correction part is not necessarily an indispensable component and in some cases this is not provided.

**Figure 1 Setup of atomic absorption spectrometric apparatus
(an example)**

5.2 Light source part

5.2.1 Light source The light source part consists of a light source and a power source for lighting lamps and the light source shall be as follows.

- a) **Hollow cathode lamp** A hollow cathode lamp is the light source for analysis and the light source for the background correction, and an example of its construction is as given in figure 2.

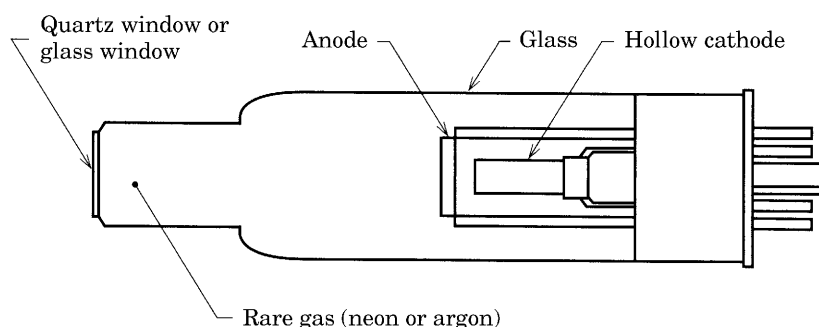


Figure 2 Structure of hollow cathode lamp (an example)

- b) **High intensity lamp** A light source for analysis of a specific element
- 1) High intensity hollow cathode lamp
 - 2) Electrodeless discharge lamp
- c) **Low pressure mercury lamp** A light source for mercury analysis
- d) **Xenon lamp** A light source for an analyte element analysis and for the background correction
- e) **Deuterium lamp** A light source for the background correction
- f) **Tungsten lamp** A light source for the background correction

5.2.2 Power source for lighting lamps The power source for lighting lamps in light source part shall have functions of lighting the light source lamps and stabilizing their luminance.

5.3 Sample atomization part

5.3.1 Atomization part of flame system The atomization part of a flame system consists of a burner and a gas flow rate controller.

- a) As the burner, there shall be a premix burner which blows sample solution into chamber and sends only fine particles into a flame and a total consumption burner which feeds total quantity of the atomized sample solution into the flame. An example of the premix burner is as given in figure 3. Here, the sample solution shall be introduced into a chamber in mist form by the flow of a supporting gas to the outside of the nebulizer composed of double pipes. This mist further runs into a disperser, and only finer particles go into the chamber with a fuel gas and supporting gas and are sent into flame from the slot of a burner head. Gases to be used for the flame of the premix burner shall be acetylene and air, acetylene and dinitrogen monoxide, hydrogen and argon, etc. The drain trap which prevents the fuel gas and supporting gas flowing out from the drain tube shall be used.
- b) The gas flow rate controller shall be that to keep the pressure and the flow rate of the fuel gas and supporting gas to be supplied to the burner constant, and consists of a pressure control valve, a flow rate control valve and others.

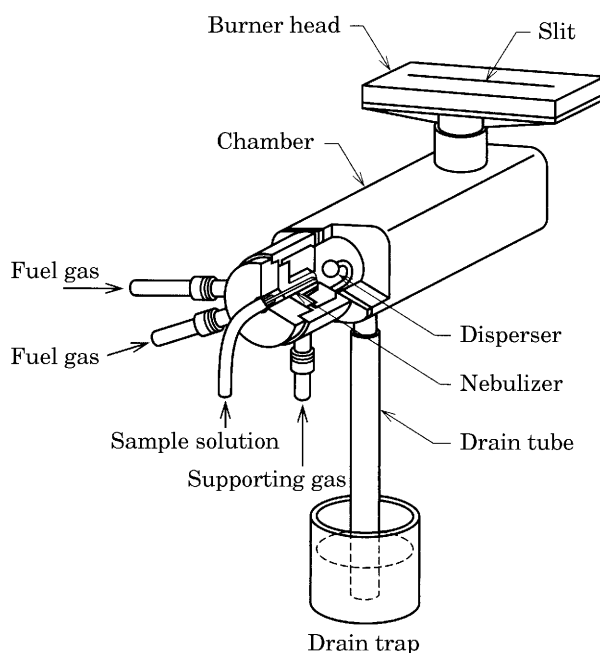


Figure 3 Premix burner (an example)

5.3.2 Atomization part of electrothermal type The atomization part of an electrothermal type consists of an electric heating furnace and a power source.

- a) The electric heating furnace is used to dry, incinerate, and atomize the sample solution by applying a current flow to the heating element of the electric heating furnace, and its heating element shall be made of graphite or heat resisting metal. In some cases, it is so constructed as to flow argon, nitrogen, a mixed gas of argon and hydrogen, etc., into the furnace in order to prevent oxidation and transfer sample vapour and others. An example of the electric heating furnace (graphite) is as given in figure 4.

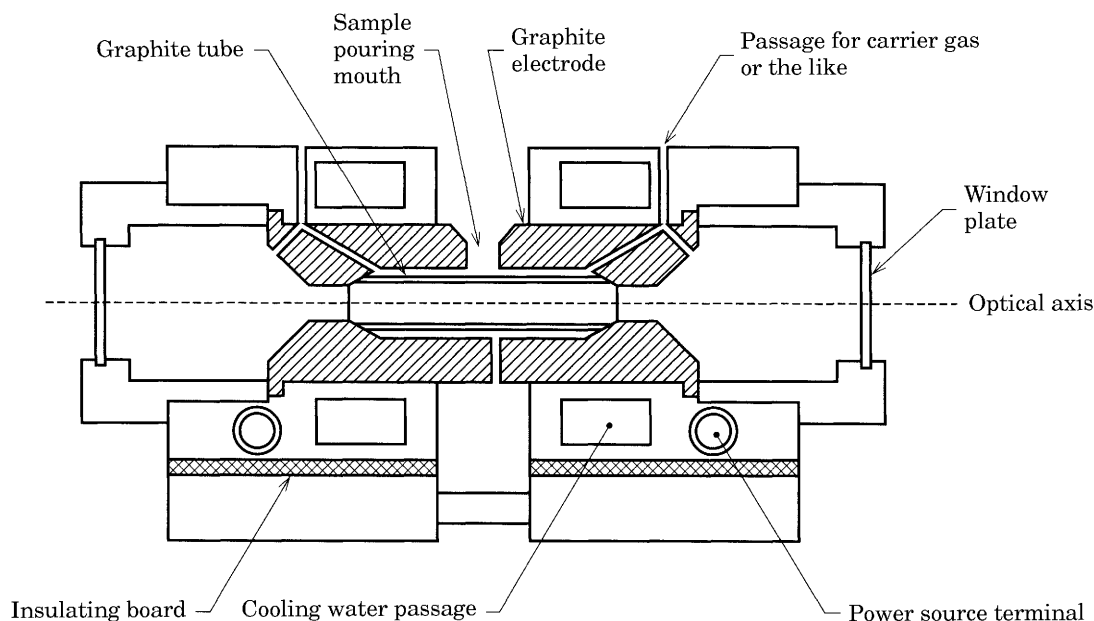


Figure 4 Electric heating furnace (graphite) (an example)

- b) The power source heats the heating element of the electric heating furnace stepwise or continuously to a required temperature.

5.3.3 Atomization part of cold vapour type The atomization part of a cold vapour type consists of a mercury vapour generating part and an absorption cell.

- a) The mercury vapour generating part shall be an atomic vapour generation device by reduction in which mercury is reduction-vaporized by adding a reducing agent to a sample solution.
- b) The absorption cell shall be of a gas flow-through structure having quartz glass windows at both ends.

5.4 Optical system

5.4.1 Photometry system The photometry system includes a single-beam system (figure 5) and a double-beam system (figure 6). For the single-beam system, measurement is performed with one luminous flux, and for the double-beam system, the luminous flux is divided by a half mirror etc., and the one is made to pass through an atomization part, and the other is bypassed. The light intensity change is corrected taking the latter as a reference beam.

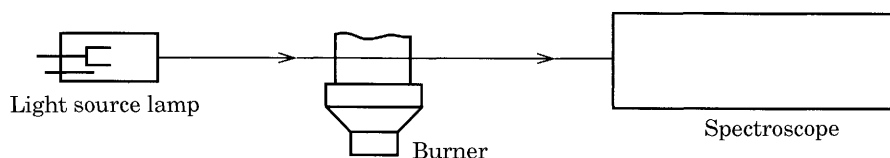


Figure 5 Example of optical system of single-beam system

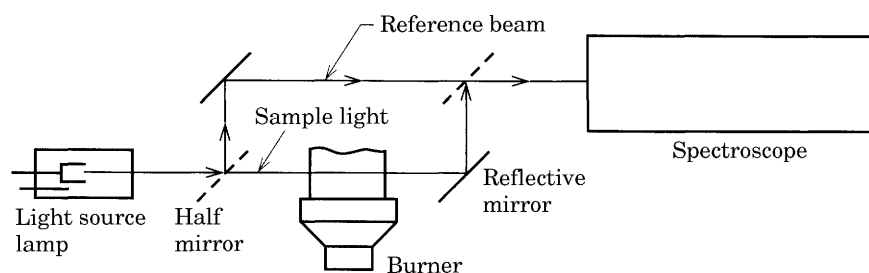


Figure 6 Example of optical system of double-beam system

5.4.2 Spectroscope The spectroscope shall be for selecting a required analytical line only from the spectrum emitted from the light source and equipped with the spectroscope using a diffraction grating and shall have the sufficient resolution which is capable of separating a neighbouring line. The spectroscope includes a Littrow spectroscope (figure 7), a Czerny-Turner spectroscope (figure 8), Ebert spectroscope (figure 8), Echelle spectrograph (figure 9) and others.

NOTE : For the element of which the light source spectrum distribution is simple, there is a case where an interference filter is used. Or, there is also the case where only a certain wave can be selectively detected without using a diffraction grating and an interference filter.

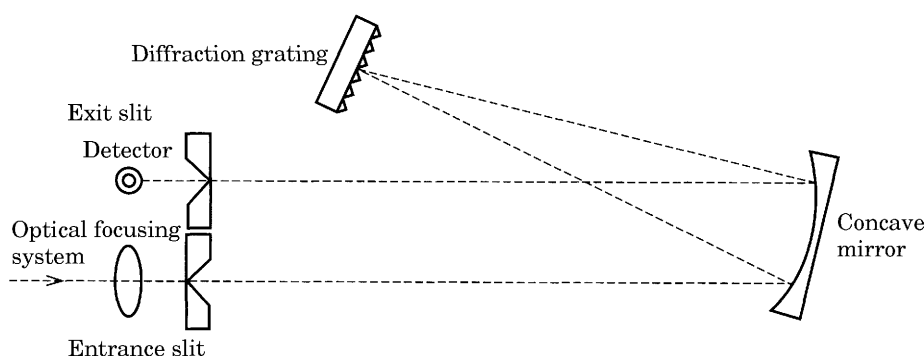


Figure 7 Spectroscopy section using Littrow type

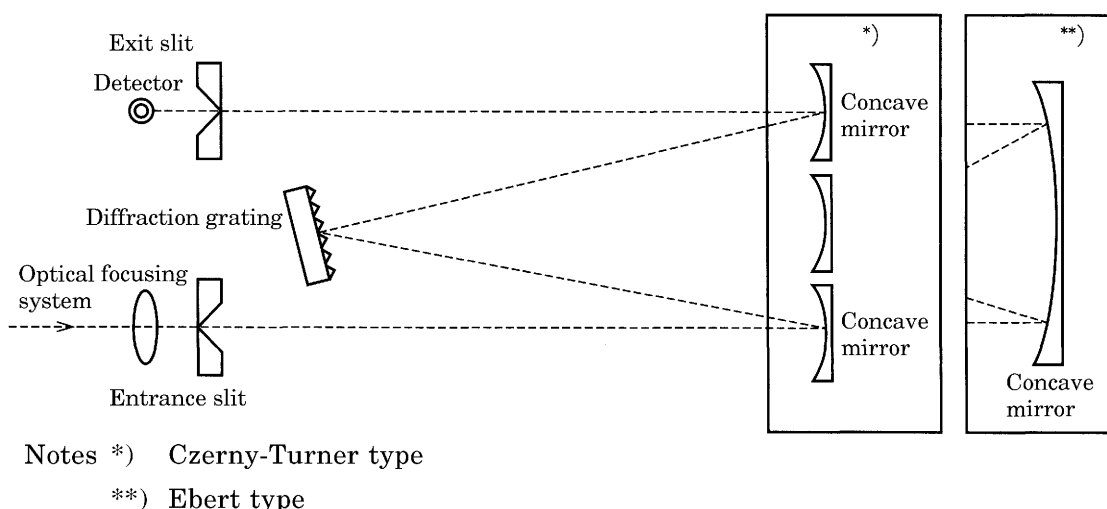


Figure 8 Spectroscopy section using Czerny-Turner type and Ebert type

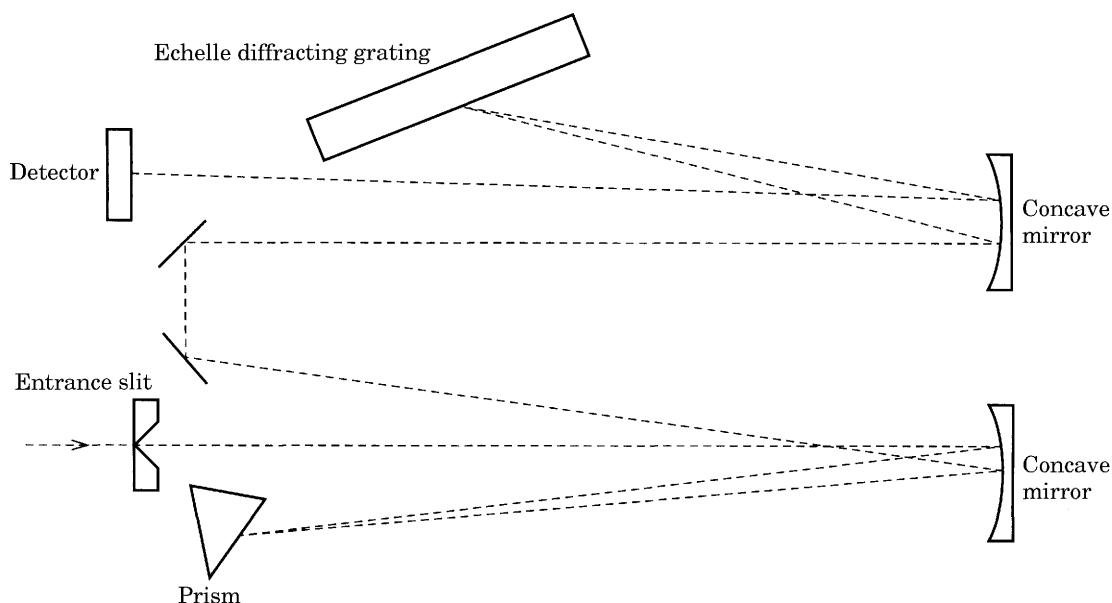


Figure 9 Spectroscopy section using Echelle type

5.5 Detection part The detection part changes the light intensity of the incident light to a detector into an electric signal according to the intensity. A photomultiplier tube, a photoelectric tube, or a semiconductor detector is used.

5.6 Data processing part The data processing part performs data processing⁽²⁾, and displays and records the absorbance, concentration, working curve and measurement result. The data processing part includes an analog system and a digital system. For the display, a display, printer, recorder, etc., are used.

Note ⁽²⁾ The data processing includes that which has the integration function of a signal, the reading function of an absorption peak value and the preparation and memory function of a working curve in order to attain the precision improvement and speeding up of reading of measured values.

5.7 Background correction part This part is for the purpose of correcting the background. There are four types of background correction systems: continuous-spectrum light source correction system, Zeeman splitted spectrum correction system, nonresonance spectrum correction system and self-reversal spectrum correction system.

5.7.1 Background correction system using continuous-spectrum light sources

This is a system to be used for background correction of a light source emitting continuous spectra (for example, a deuterium lamp, a tungsten lamp, etc.). It is composed of a continuous-spectrum light source lamp and an optical system⁽³⁾ which conforms its light to an optical axis of a light source lamp for analysis (a hollow cathode lamp). An example of the background correction system using a continuous-spectrum light sources is as given in figure 10.

Note ⁽³⁾ A half mirror is generally used.