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**General rules for atomic emission
spectrometry**

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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 0116:2003** is replaced with this Standard.

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General rules for atomic emission spectrometry

1 Scope

This Japanese Industrial Standard specifies general rules when quantitative analysis is conducted using atomic emission spectrometer.

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 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 0216 *Technical terms for analytical chemistry (Environmental part)*

JIS K 0553 *Testing methods for determination of metallic elements in highly purified water*

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

JIS K 0970 *Piston pipettes*

JIS K 1105 *Argon*

JIS K 8001 *General rule for test methods of reagents*

JIS Z 8402-1 *Accuracy (trueness and precision) of measurement methods and results—Part 1: General principles and definitions*

ISO 3696 *Water for analytical laboratory use—Specification and test methods*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0050**, **JIS K 0211**, **JIS K 0212**, **JIS K 0215**, **JIS K 0216**, **JIS K 0553**, **JIS K 0557**, **JIS K 1105**, **JIS K 8001**, **JIS Z 8402-1** and **ISO 3696**, and the following apply.

3.1 atomic emission spectrometry

quantitative analysis method carried out by measuring the emission intensity of atomic spectral line of target element of measurement contained in a sample obtained by a vaporizing and exciting discharge technique such as that by means of ICP (see **3.20**), MIP (see **3.21**) or spark discharge

Qualitative analysis is also possible by identifying the wavelength obtained.

3.2 sequential scanning spectrometer

spectrometer for measuring the intensity of a spectrum line or of a series of spectrum lines in sequence by dispersing the incident light

3.3 simultaneous spectrometer

spectrometer for measuring the intensity of plural spectral lines simultaneously by dispersing the incident light

3.4 integration time

measuring time when the emission intensity is measured and integrated for a certain time

3.5 analytical sample

sample which has been prepared to be ready for measurement

3.6 analytical portion

portion of analytical sample taken as the sample to be used for one-time analysis

3.7 sample for calibration graph

sample containing the target element of measurement whose concentration is known in order to create the calibration graph

3.8 solution for calibration graph

solution containing the target element of measurement whose concentration is known in order to create the calibration graph

3.9 sample for correction of calibration graph

sample used for the correction of calibration graph at a predetermined interval or each time a predetermined number of samples have been measured

3.10 solution for correction of calibration graph

solution used for the correction of calibration graph at a predetermined interval or each time a predetermined number of samples have been measured

3.11 blank solution

water, or a solution in which the target element of measurement is not contained but other components are same as the sample, wholly treated similar to the sample including the contact with the implements such as glass and apparatus used for the analytical operation, and the addition of solvents, reagents and internal standard element, for investigating the contamination of the target element of measurement or the interference element from implements and reagents, and the environment of laboratory

It is also called the laboratory reagent blank.

3.12 calibration blank solution

solution whose composition is same as that of the solution for calibration graph but the concentration of target element of measurement is zero

3.13 sample solution

solution which has been prepared by pretreatment of gaseous, liquid or solid samples to be ready for measurement

3.14 detection limit

minimum detectable concentration (amount) of target element of measurement, which is the concentration giving a signal of three times the standard deviation of the background intensities

3.15 instrument limit of detection (ILOD)

concentration giving a signal of three times the standard deviation of signals obtained when the blank solution for calibration graph is measured 10 times continuously

3.16 method limit of quantification (MLOQ)

concentration giving a signal of 14.1 times the standard deviation of signals obtained when the blank solution is measured 10 times continuously

3.17 short term stability

relative standard deviation of emission intensity or intensity ratio when the same samples are measured repeatedly in a short term

3.18 long term stability

relative standard deviation of emission intensity or intensity ratio when the same samples are measured repeatedly in a long term

3.19 resolution

ability of spectrometer to separate two spectral lines adjacent to each other

3.20 inductively coupled plasma (ICP)

plasma generated by inductive coupling of high frequency power in radio frequency region

3.21 microwave induced plasma (MIP)

plasma generated by being induced by high frequency electric power in the region of microwave

3.22 torch

tube used for supplying the gas flow necessary for lighting and sustaining plasma

3.23 nebulizer

implement for making the sample solution into fine liquid drops

3.24 spray chamber

implement used to introduce only very fine liquid drops to an emission part by separating and removing drops of large particle size

3.25 plasma gas, coolant gas

main forming gas of plasma in ICP

It is supplied through the most exterior peripheral tube of torch for the purpose of cooling the torch.

3.26 auxiliary gas

gas subsidiarily used to prevent the plasma from contacting the torch in ICP

It is supplied through the middle tube of torch.

3.27 carrier gas

gas used to introduce a sample into plasma in ICP

It is supplied through the central tube of torch.

3.28 background equivalent concentration (BEC)

concentration of the target element of measurement giving signal intensity equal to the background intensity

3.29 spark discharge

discharge caused by making the electrical charge flow momentarily, which is stored in a capacitor of main discharge circuit between the electrodes by making an electric break down occur by applying about 10 kV of high voltage between the counter electrode and the sample electrode by an ignition circuit

The peak current of this momentary discharge reaches several tens A to 200 A (ampere) and this makes the sample be excited and emitted.

3.30 spark light source

excited and emitted sample by spark discharge

3.31 atmospheric gas

gas flowed around the electrode of spark discharge for the purposes such as stabilization of discharge, removal of influence owing to sample hysteresis, absorption owing to oxygen in air, and removal of band spectrum, etc.

Argon or nitrogen, or these gases mixed with oxygen respectively is used according to its purpose.

3.32 pre-burn time, pre-discharge time

time set as the period when photometry is not carried out, during the time from the start of discharge to the stabilization of emission intensity in spark discharge atomic emission spectrometry

3.33 time resolved photometric method

method for obtaining the concentration in such a way that the photoelectric current obtained at every discharge pulse and time resolved is integrated, and the individual integrated value is statistically processed and analysed

4 ICP atomic emission spectrometry

4.1 Composition of apparatus

ICP atomic emission spectrometer is composed of the excitation source part, sample introduction part, the emission part, the spectrophotometry part, data processing part and the control part. Figure 1 shows an example of the basic composition of the apparatus.

The MIP atomic emission spectrometer has a similar composition.

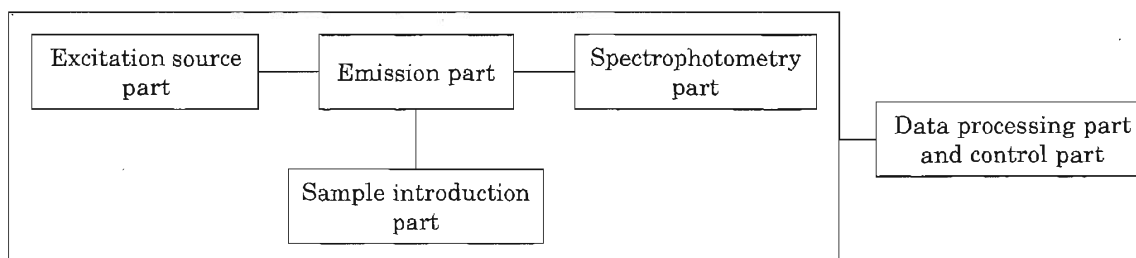


Figure 1 Composition of ICP atomic emission spectrometer (example)

4.1.1 Excitation source part

This part is composed of power supply circuit and control circuit to supply/control the electric energy in order to maintain the emission part.

4.1.2 Sample introduction part

This is a part introducing the sample into the emission part and is composed of a nebulizer and a spray chamber. An example of the sample introduction part is shown in figure 2. The part treated with a material resistant to hydrofluoric acid is also available.

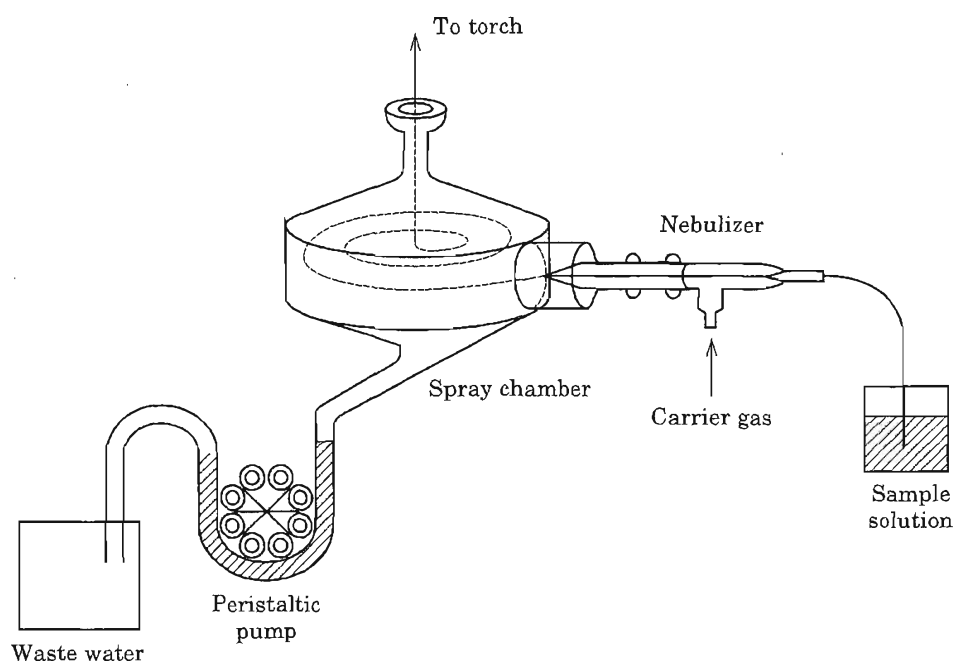


Figure 2 Sample introduction part (example)

4.1.3 Emission part

This is a part where the target element of measurement in the sample is excited/emitted, which is composed of the torch and the induction coil (see figure 3). The torch usually consists of a triple tube, and the sample is introduced from the central tube. ICP is used as the emission part and argon is used for the gas to form the plasma.

MIP is sometimes used in place of ICP. In such a case, the composition also differs and nitrogen is mainly used for the gas to form plasma.

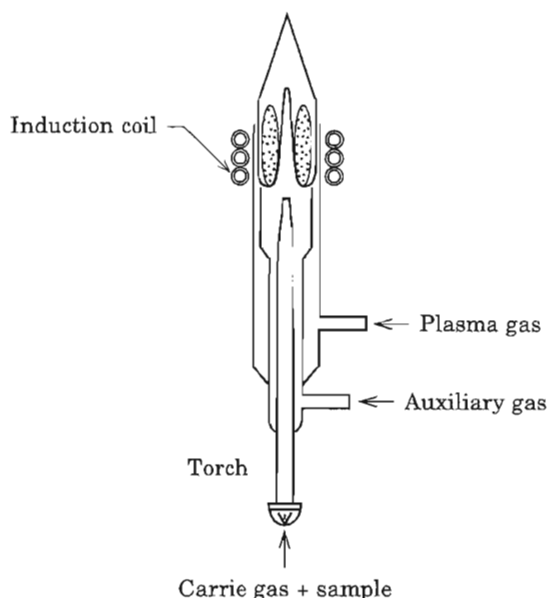


Figure 3 Emission part (ICP) (example)

There are two types of observation method from the emission part, which are the horizontal observation method and the axially observation method (see figure 4).

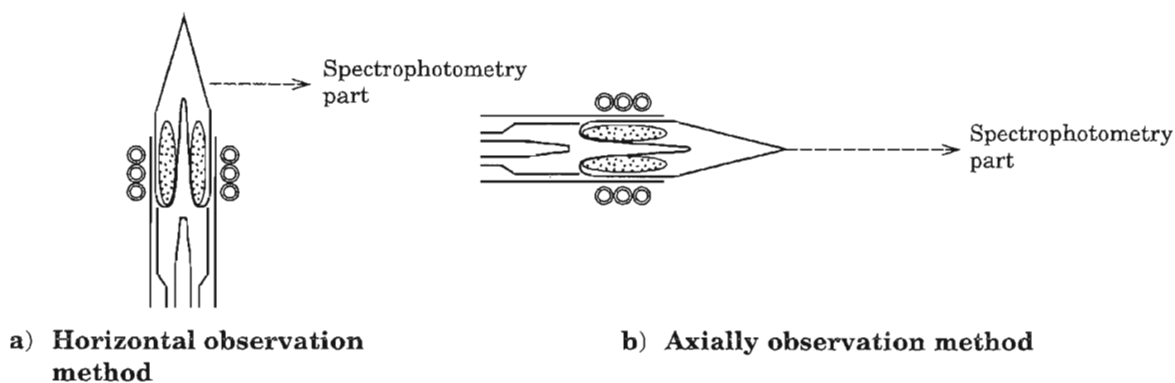


Figure 4 Observation method of light

4.1.4 Spectrophotometry part

The spectrophotometry part is composed of the light collection system which introduces light radiated from the emission part to a spectral part effectively, a spectral part which separates spectral lines and a detection part. There are Czerny Turner type spectrometer (figure 5), Paschen Runge type spectrometer (figure 6), Echelle type

spectrometer (figure 7) and the like. The Czerny Turner type spectrometer is used as a sequential scanning spectrometer, and the Paschen Runge type spectrometer and the Echelle type spectrometer are mainly used as a simultaneous spectrometer. The detector converts the incident light into an electric signal corresponding to its intensity, and a photo-multiplier or a solid state detector is used.

NOTE : For measuring spectral lines in a vacuum ultraviolet region (wavelength 120 nm to 190 nm), the spectrometer has a structure for evacuating the light collection system and the spectrometer, or a structure for substituting air by argon or nitrogen.

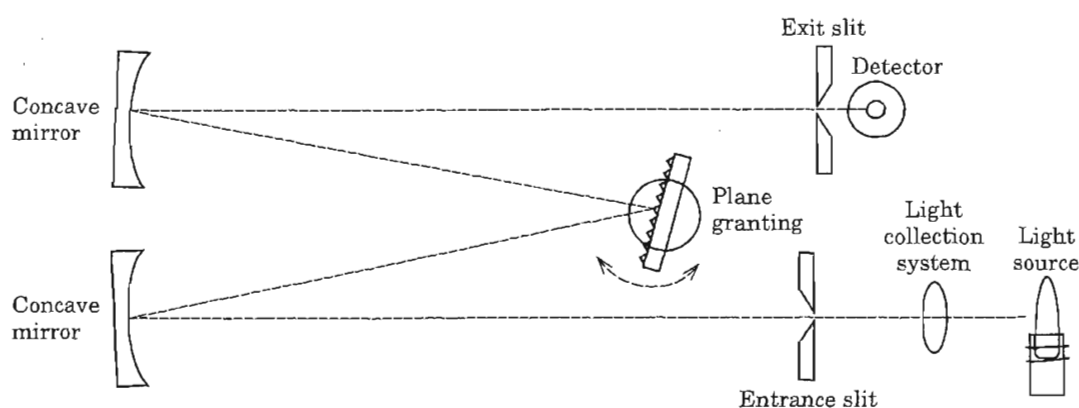


Figure 5 Spectrophotometry part with Czerny Turner type spectrometer (example)

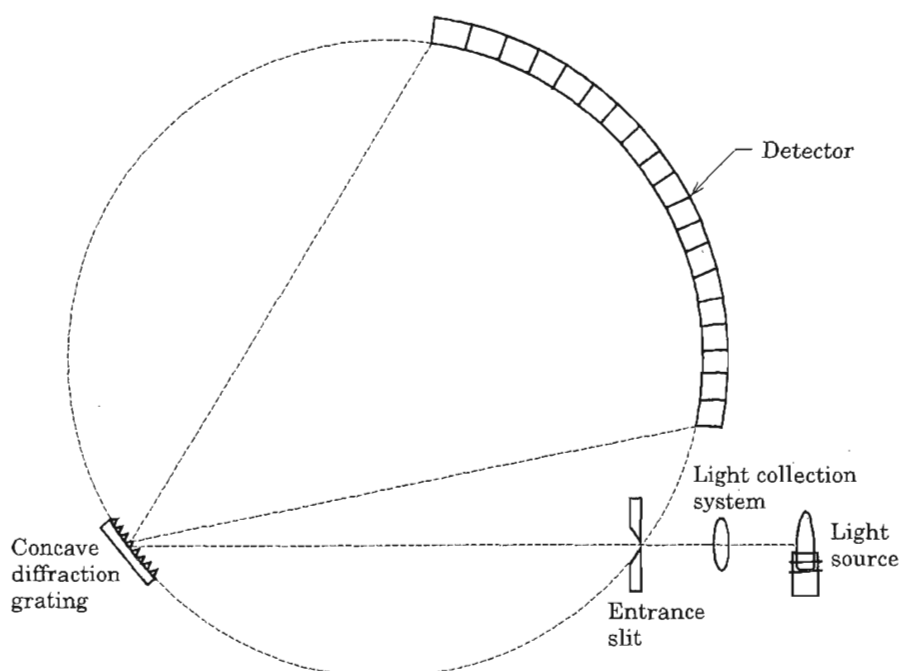


Figure 6 Spectrophotometry part with Paschen Runge type spectrometer (example)

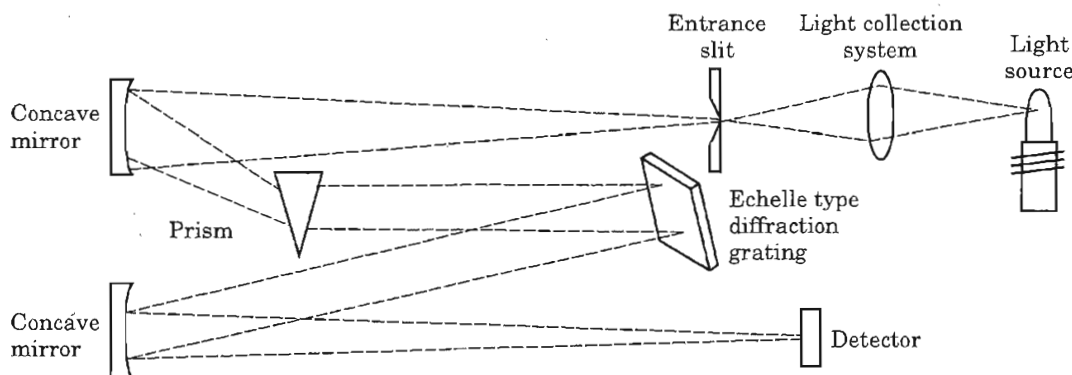


Figure 7 Spectrophotometry part with Echelle type spectrometer (example)

4.1.5 Data processing part and control part

The data processing part and the control part shall be as follows.

4.1.5.1 Data processing After data processing¹⁾, the calibration graph, the measurement result, etc. are displayed. For the display, the liquid crystal device (LCD), printer and the like are used.

Note ¹⁾ In order to improve the accuracy of data processing, it may have functions of background correction, spectral interference correction, correction by internal standard element and the like.

4.1.5.2 Control part For using the apparatus under the optimum conditions, this part controls the gas flow, torch position for measuring intensity of light, electric power for excitation source part and the like.

4.2 Attachments

The attachments shall be as follows.

- a) **Autosampler (or automatic sample introduction instrument)** An instrument to supply various samples automatically into the sample introduction part in sequence. Some may have the online functions of automatic dilution, automatic addition of internal reference solution and addition of solution for calibration graph.
- b) **Supersonic wave nebulizer** An instrument for atomizing the sample solution by an ultrasonic vibrator, deliquoring it by heating/cooling and introducing by carrier gas into the emission part.
- c) **Hydride generation instrument** An instrument that, after reducing the compounds such as arsenic, selenium and antimony in the sample solution into the volatile hydride with sodium tetrahydroborate etc., carries out the vapour-liquid separation and introduces only the gas components into the emission part with carrier gas.
- d) **Flow injection instrument** An instrument for injecting the sample solution of fixed small amount into the carrier solution flowing in a capillary tube by switching the valve and introducing it into the emission part. Some may carry out the online chemical reaction.

- e) **Introducing instrument with electric heating evaporation system** An instrument that, after injecting the small amount of sample solution into the graphite furnace or refractory metal heater, removes the solvent and a part of matrix component selectively in inert gas atmosphere, evaporates the residual target element of measurement instantaneously and introduces carrier gas into the emission part.
- f) **Laser ablation instrument** An instrument that introduces fine particles generated by evaporation when the laser beam is irradiated to the solid sample, by carrier gas into the emission part.
- g) **Others** Matrix separation column, chromatograph, etc.

4.3 Water, reagents and gas

4.3.1 Water

Water of A2 or better specified in **JIS K 0557** or that of Grade 2 or better specified in **ISO 3696** shall be used. It shall be confirmed that impurities contained in water above will not interfere with the target element of measurement.

4.3.2 Reagents

Reagents shall be as follows.

- a) When corresponding **JIS** exists, those of the highest grade or those appropriate for the purpose shall be used. When the corresponding **JIS** does not exist, those do not interfere with analysis in quality shall be used.
- b) For the reference solution for calibration graph, the reference solution with the metrological traceability secured [for example, the reference solution appended with the certificate with logo of JCSS (Japan Calibration Service Systems)] shall be used. When such a reference solution with the metrological traceability secured is not available or is not required depending on the purpose, the general reference solution on the market is used. It shall be confirmed that impurities contained in water above will not interfere with the target element of measurement.
- c) When using the reference solution for calibration graph other than **b)**, it shall be prepared by dissolving the metal of at least 99.99 % in purity or prepared by using the metal or the compound whose purity is measured and determined as the specification value with the confirmation that the coexisting substances do not interfere with the target element of measurement.

4.3.3 Gas

Generally, argon of 99.99 % or more (volume fraction) in purity as specified in **JIS K 1105** shall be used. When other gas than argon is used, that specified in **JIS** shall be used. When the gas specified in **JIS** is not available, that does not hinder the analysis shall be used.

4.4 Sampling and preparation of sample solution

4.4.1 Sampling

The sampling shall be carried out by the most suitable method for the analytical purpose, the characteristics of sample and the measuring items so as to represent the