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General rules for ion chromatography

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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 0127:2001** is replaced with this Standard.

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General rules for ion chromatography

1 Scope

This Japanese Industrial Standard specifies the general rules for qualitative and quantitative determination of analyte using the ion chromatography.

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) listed below shall be applied.

- JIS K 0050 *General rules for chemical analysis*
- JIS K 0211 *Technical terms for analytical chemistry (General part)*
- JIS K 0214 *Technical terms for analytical chemistry (Chromatography part)*
- JIS K 0215 *Technical terms for analytical chemistry (Analytical instrument part)*
- JIS K 0557 *Water used for industrial water and wastewater analysis*
- JIS K 8001 *General rule for test methods of reagents*
- JIS K 8005 *Reference materials for volumetric analysis*
- JIS K 8019 *Sodium nitrite (Reagent)*
- JIS K 8073 *Benzoic acid (Reagent)*
- JIS K 8116 *Ammonium chloride (Reagent)*
- JIS K 8121 *Potassium chloride (Reagent)*
- JIS K 8267 *Sodium formate (Reagent)*
- JIS K 8372 *Sodium acetate (Reagent)*
- JIS K 8443 *Potassium cyanide (Reagent)*
- JIS K 8495 *para-Dimethylaminobenzylidene rhodanine (Reagent)*
- JIS K 8506 *Potassium bromide (Reagent)*
- JIS K 8528 *Sodium oxalate (Reagent)*
- JIS K 8530 *Potassium bromate (Reagent)*
- JIS K 8548 *Potassium nitrate (Reagent)*
- JIS K 8617 *Calcium carbonate (Reagent)*
- JIS K 8875 *Magnesium (Reagent)*
- JIS K 8913 *Potassium iodide (Reagent)*
- JIS K 8987 *Sodium sulfate (Reagent)*
- JIS K 9001 *Potassium thiocyanate (Reagent)*
- JIS K 9007 *Potassium dihydrogen phosphate (Reagent)*
- JIS K 9501 *Sodium azide (Reagent)*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0050**, **JIS K 0211**, **JIS K 0214** and **JIS K 0215** and the following apply.

NOTE : Some of the following terms that are also defined in **JIS K 0211:2005**, **JIS K 0214:2006** or **JIS K 0215** are given supplemented or reinforced definitions for use in this Standard. Those terms are marked with asterisks.

3.1 ion chromatography*

an analysis method using ion chromatograph in which ion species in sample solution are developed and eluted in a separation column composed of stationary phase as ion exchanger and of mobile phase as eluent, and then are measured by a conductivity detector, electrochemical detector, spectrophotometric detector, or fluorometric detector

3.2 ion chromatograph*

an apparatus used for ion-chromatographic analysis

3.3 eluent*

liquid used for development and elution of ion species contained in the sample kept in the column

3.4 gradient elution method

method to develop and elute ion species in the sample while continuously varying the composition of the eluent

In some cases it is the flow rate of the eluent that is varied.

3.5 sample injector, injection port

part with a function to introduce the test sample solution

3.6 column oven

oven for accommodating the column

3.7 pre-column*

column set before the separation column for the purpose of concentration, pre-separation, removal of foreign matters, etc. of ion species to be measured

3.8 separation column*

column used for separation of ion species, having the separating performance suitable for each specific separation purpose

3.9 ion exchanger*

packing having ion-exchange groups on the surface

3.10 ion exchange capacity*

equivalent of ion-exchange group per unit mass or unit volume of ion exchanger, expressed in meq/g or meq/ml

3.11 eluate

liquid flowing out from a separation column as a result of development by the mobile phase (eluent)

3.12 suppressor

device used with a conductivity detector, which reduces the background conductivity without impairing the detection of the ion species to be measured

3.13 suppressor method

measurement method using a suppressor

3.14 non-suppressor method*

measurement method in which the ion species is separated using an eluent of low conductivity and measured by means of a conductivity detector, without the use of a suppressor

3.15 regenerant*

liquid for regeneration or continuous maintenance of the function of the suppressor

It is also called a cleaning liquid.

3.16 conductivity detector

the detector to measure the conductivity of the object component in the eluate

3.17 electrochemical detector

the detector which applies a defined potential on a working electrode and measures the current or amount of electricity flow caused by electrochemical reaction of the object component in the eluate

3.18 ultraviolet-visible absorption detector*

the detector to measure the absorbance of the analyte absorbed within the ultraviolet-visible range

3.19 fluorometric detector*

the detector to measure the fluorescence of the object component in the eluate

3.20 waste reservoir

vessel for reserving liquid exhausted from the apparatus

3.21 standard solution*

the solution conditioned using reference material or standard reagents generally to 1 000 mg/L in the concentration of ion species to be measured

3.22 diluted standard solution

the solution prepared by diluting the standard solution to a specified concentration

3.23 mixed diluted standard solution

the solution prepared by mixing two or more standard solutions or diluted standard solutions such that each component is of a specified concentration

3.24 resolution*

the index to indicate the mutual separation of peaks of two ions

3.25 sensitivity, sensitiveness*

the minimum quantity (value) of detectable quantity or concentration change in measurement of ion species

3.26 working curve*

the line expressing the relation between the quantity or concentration of ion species to be measured and measured value

3.27 external standard method*

the method in which the working curve expressing the relation between the quantity or concentration of the ion species to be measured and the peak area or height is prepared and then the quantity or concentration in the sample is obtained by the working curve prepared by plotting the peak area or height of the sample measured under the same conditions

3.28 blank test*

the test to obtain the blank value

3.29 blank value*

value obtained by carrying out the same procedure as the sample on a solution not containing the ion species to be measured

3.30 minimum limit of detection, minimum limit of identification*

minimum detectable quantity (value) or concentration of the ion species to be measured

3.31 minimum limit of determination*

minimum quantity (value) or concentration of the ion species to be measured which can be determined

3.32 stationary phase*

one of the elements of a field where ion chromatography is carried out, which is in equilibrium with the mobile phase and interacts with the analyte

3.33 mobile phase*

one of the elements of a field where ion chromatography is carried out, which is a fluid that flows in contact with the stationary phase

4 Outline of ion chromatography

The ion chromatography is the method in which the sample is introduced by using an eluent as the mobile phase and the ion species is determined by utilizing the difference of interaction (ion exchanger, etc.) with the stationary phase. It is a type of high performance liquid chromatography. The ion species is mainly separated by the separation mechanism such as ion exchange, ion exclusion, and reversed phase ion pair. It is used for the measurement of inorganic ion, organic acid, low-molecular-weight amine, halogen and the like in solution. It is used in many fields such as the environmental monitoring of atmosphere, water, soil, etc., the control of food, the water quality control of drinking water, drainage, the control of exhaust gas, the analysis of living organic sample, the determination of halogen and sulphate element contained in inorganic and organic materials such as bromine series fire retardant in resinous product.

Sample introduction is performed by measuring a specified volume of the sample solution and injecting it into a column.

As a column, a tube of synthetic resin, metal or other material filled with ion exchanger is used.

For the detector, a conductivity detector, an ultraviolet-visible absorption detector, an electrochemical detector, a fluorometric detector, a mass spectrometer, an ICP emission spectrophotometer and the like are used. The detector used can be equipped with a pre-analysis treatment part for improving the sensitivity and/or selectivity to the ion species to be measured.

For the measurement of trace impurities, the pre-treatment such as solvent extraction, solid phase extraction, the improvement of sensitivity by mass spectrometric apparatus and/or derivation reagent is required.

5 Construction of apparatus

5.1 Construction

The ion chromatograph is composed of a vessel for eluent, a pump, a sample injection port, a column part, a detecting part, a data processing part and a waste reservoir. If necessary, it may be equipped with auxiliary devices such as a gradient device, degasifier, etc. An example of basic construction is shown in figure 1.

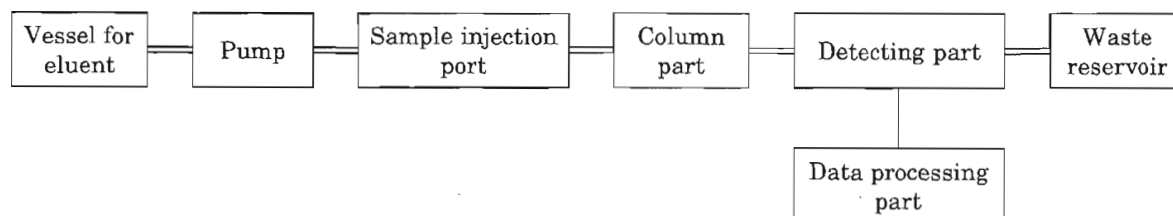


Figure 1 Example of basic construction of ion chromatograph

5.2 Vessel for eluent

The material of vessel for eluent shall not be corroded by or contaminate the eluent.

5.3 Pump

The pump is composed of a degasifier and a feeding pump. In the case of performing gradient elution, the feeding pump shall have a gradient function.

- a) **Degasifier**, used for continuously removing the air dissolved in the eluent, for preventing the troubles of air bubbles generated in the apparatus due to temperature and pressure change, and for obtaining stable flow rate and background. This device is not required if the use of an eluent prepared by water degassed offline is permissible.
- b) **Feeding pump**, capable of feeding the eluent to the column at an accurate and precise flow rate, preferably meeting the following conditions.
 - 1) It has a high precision on the uniformity of flow rate.
 - 2) It has sufficient feeding pressure.
 - 3) It has small pulsating flow rate.
 - 4) It is capable of controlling the flow rate.
 - 5) It has liquid contacting parts of a material that is not corroded by or that does not contaminate the eluent.
 - 6) It allows easy exchange of eluent.
- c) **Gradient device**, consisting of a control part in which two or more eluents are used and the composition of the eluents is changed with time, and a mixer in which the mixed solution is brought to uniformity. It is either a low pressure gradient device having the mixer before the feeding pump or a high pressure gradient device having multiple feeding pumps and the mixer after the feeding pump. In either device, the range of mixing ratio that can be set should be wide and the concentration should be precise.

5.4 Sample injection port (injector)

The sample injection port introduces a specified volume of sample solution into the apparatus with good reproducibility by any of the following methods. An automatic sample injector may be used where many sample solutions are required to be automatically introduced.

- a) **Method 1** The sample injection port is provided with a sample loop for storing the sample solution under test. The sample solution under test is filled into the sample loop, and then introduced into the separation column by switching of the eluent flow channel by six-way valve or the like. An example of the sample injection port by this method is shown in figure 2. Either the whole volume of the sample is filled in the loop, or only a specified volume is introduced in a part of the loop.

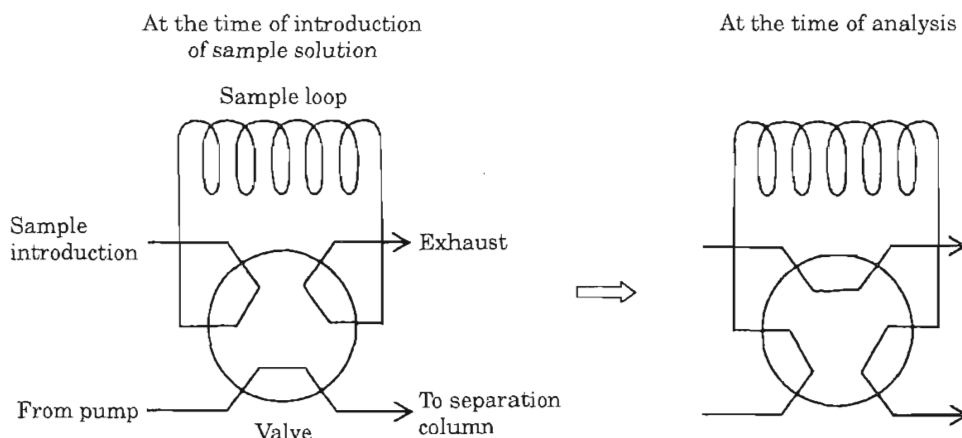


Figure 2 Example of sample injection port

- b) **Method 2** The sample injection port is provided with a concentration column for collection and concentration of the ion species. A specified volume of sample solution under test is introduced into the concentration column, and then ion species concentrated in the concentration column is introduced into the separation column by switching of the eluent flow channel by six-way valve or the like. An example of the sample injection port by this method is shown in figure 3.

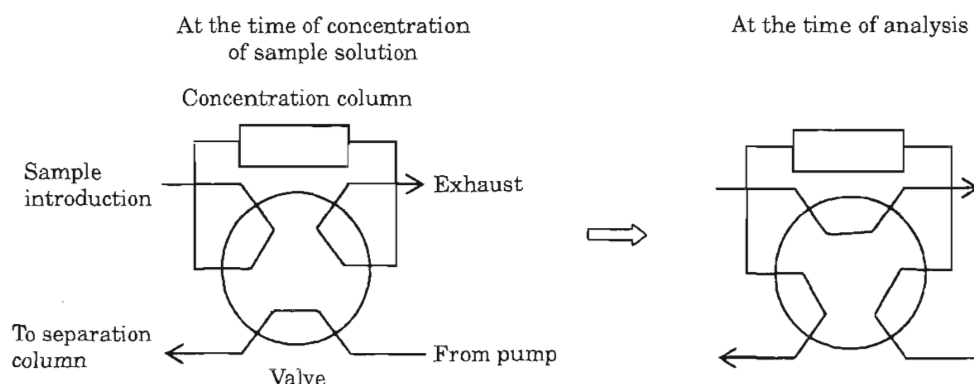


Figure 3 Example of sample injection port by method 2

5.5 Column part

The column part is composed of a column and a column oven. The column oven, however, if it is not required for the purpose of analysis, may be omitted as long as the analytical performance is not impaired.

- a) **Column**, either a pre-column¹⁾ or separation column, which shall have a performance suitable for the purpose of analysis.

Note ¹⁾ Provision of a pre-column is not mandatory; it should be provided as required for the purpose of protection of the column.

- b) **Column oven**, having an inner capacity sufficient for accommodating a column with the required length. Some column ovens also have a function of a constant temperature oven, for maintaining the temperature constant at a specified level.

5.6 Detecting part

The detecting part introduces the eluate from the column to a micro-flow cell, and detects the concentration or quantity of ion species component based on various principles by taking the eluent as the background. It consists of a detector and the part for carrying out the pre-treatment of eluate. The part for carrying out the pre-treatment of eluate as specified in the following, if it is not required for the purpose of analysis, may be omitted as long as the analytical performance is not impaired.

- a) **Detector**, of a material not corroded by the eluent and/or the components in the sample under test, suitable for detecting the ion species component under test, which is one of the followings.
 - 1) Conductivity detector
 - 2) Ultraviolet-visible absorption detector
 - 3) Electrochemical detector
 - 4) Fluorometric detector
 - 5) Mass spectrometer
 - 6) ICP atomic emission spectrometer
 - 7) ICP mass spectrometer
- b) **Part for carrying out the pre-treatment of eluate**, provided for enhancing the sensitivity or selectivity of the apparatus to the ion species component under test, and consisting of the following.
 - 1) **Suppressor**, to be provided where a conductivity detector is used, for reducing the conductivity of the eluent by ion exchange through the ion exchange site (membrane or resin), and improving the SN (signal noise) ratio by exchanging the pair ion of the ion to be measured with ion of higher conductivity to enhance the measuring sensitivity. In the anion analysis, cation interchange membrane or resin is used and the cation part of eluent and ion to be measured is exchanged with H^+ . On the other hand, in the cation analysis, anion interchange membrane or resin is used, and anion part of eluent and ion to be measured is exchanged with OH^- . Suppressor types, divided according to the type of ion exchange site, are the membrane type, column type, gel type, and fibre type as shown in table 1 or a type using a combination of these. When the ion exchange site of suppressor is continuously used, regeneration is necessary. Regeneration can be performed either by the chemical regeneration method using acidic aqueous solution (for measurement of anion) or alkali aqueous solution (for measurement of cation), or the electrical regeneration method to supply H^+ or OH^- by electrolyzing the water or the exhaust water from the detector.