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General rules for flow analysis

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Foreword

This Japanese Industrial Standard has been 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 0126:2009** is replaced with this Standard.

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General rules for flow analysis

1 Scope

This Japanese Industrial Standard specifies general requirements for flow analysis. The flow analysis indicates operations and technique for the quantitative determination of organic and inorganic matters by using reactions in the flow. This Standard specifies methods for flow injection analysis, continuous flow analysis and sequential injection analysis, which differ in the method for introducing samples and reagents into the analyser.

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 0213 *Technical terms for analytical chemistry (Electrochemistry 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*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS K 0050**, **JIS K 0211**, **JIS K 0212**, **JIS K 0213**, **JIS K 0214**, **JIS K 0215**, and the following apply.

3.1

double injection method

the method to introduce a sample of different concentration or amount into two places in a flow, or the method to introduce a sample and reagent simultaneously before and after a carrier

3.2

merging zone method

the method using two flows of carriers where a sample is introduced into a flow of carrier and at the same time a reagent into the other flow of carrier, both of which are merged downstream

3.3

sandwich injection method

the method to introduce Sample S and Reagent R in the order of R-S-R or S-R-S in series in the carrier flow passage

3.4

stopped flow method

the method to stop temporarily the mixed flow of a sample and reagent

The following two methods are available: the method to stop the sample and reagent in the flow cell of a detector to observe the change in reaction with the passage of time and the method to stop these in a mixer to restrain the dispersion and to prolong the reaction time.

3.5

flow injection titration method

the method to obtain the concentration from the width of response curve obtained after introducing a sample into the flow of reagent, dispersing sufficiently by using a mixer with a stirrer and producing reaction

In the case of using various buffer solutions as a reagent, the dispersion coefficient shall be three to ten without using a mixer with a stirrer, and the peak height of response curve shall be used for determination.

NOTE When a component with concentration C_0 is introduced into the fluid in a tube and the concentration becomes C due to dispersion, the value obtained by dividing C_0 by C is called dispersion coefficient, which is expressed as D and defined as $D = C_0/C$.

3.6

segment

the divided layer (band) generated in a tube when gas, organic solvent or the like which does not mix with the fluid in the tube is introduced

3.7

dispersion

the phenomenon where a sample and reagent introduced into fluid in a tube spread into the fluid due to the transportation by the laminar flow in the tube and the diffusion in the axial and radial directions of the flow

3.8

merging point

the place where two or more flows containing a sample, reagent, carrier, or the mixture of these join into one

3.9

response curve

a curve or group of data indicating the relation between the response signal intensity of detector and the time which are plotted on the ordinate and abscissa, respectively

3.10

carrier

the fluid to carry a sample or reagent introduced into a tube

3.11

tube

a fine pipe comprised of the flow passage of fluid

3.12

pump

an instrument to send fluid

3.13

injector

a device to introduce a sample or reagent into the fluid in a tube

3.14

divider

a device to divide one flow passage into plural flow passages

3.15

mixer

a device to mix a sample and reagent

3.16

concentrator

a device to separate and/or concentrate a target component in a sample

3.17

extractor

a device to extract a target component in a sample

3.18

segmentor

a device used to form a segment

3.19

phase separator

a device to separate water phase and organic phase

3.20

gas separator

a device to separate gas and liquid

3.21

reactor

a device to react a sample with a reagent

3.22

dialyzer

a device to separate an analyte or unnecessary coexisting component in a sample by using a semipermeable membrane, etc.

3.23

distiller

a device to separate and purify an analyte in a sample by distillation operation

4 Flow injection analysis

4.1 Overview

A sample is introduced into a reagent flowing at a constant flow rate in a tube to react an analyte with the reagent by dispersion or mixture in the tube, and the quantitative determination is carried out by detecting the reaction product with a detector installed downstream.

A reagent may be introduced into the flow of sample in some cases.

4.2 Composition of analyser

4.2.1 General

The flow injection analyser is composed of the flow system in which the liquid-feed part, sample introduction part, reaction part and detection part are connected by tubes and of the display/recording part. Examples of the basic schematic diagram and basic structural diagram of flow injection analysis are shown in Figures 1 and 2, respectively.

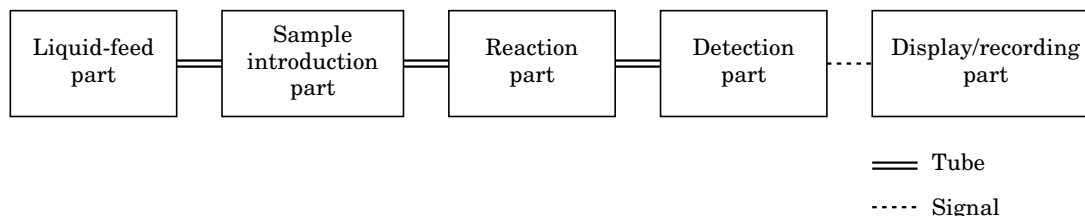


Figure 1 Basic schematic diagram for flow injection analysis (example)

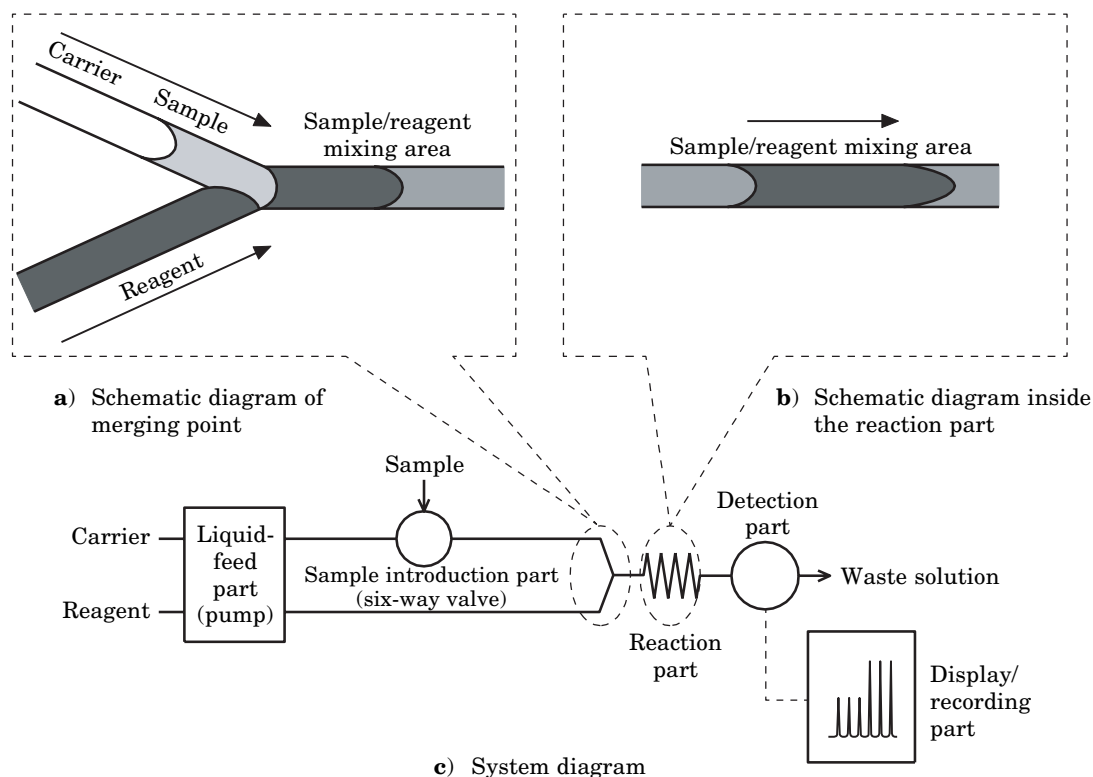


Figure 2 Basic structural diagram for flow injection analysis (example)

4.2.2 Devices composing analyser

The devices composing the flow injection analyser shall be as specified in **a)** to **g)**.

- a) Tube**, composing the flow passage of liquid, which satisfies the conditions in **1)** to **4)**.

- 1) Excellent in chemical resistance
- 2) Capable of resisting working pressure
- 3) Having a uniform inside diameter
- 4) Less sensitive to temperature

NOTE 1 The tube usually has an inside diameter of 0.25 mm to 2.0 mm and is generally made of fluororesin, polyethylene, polypropylene, stainless steel, glass, etc.

NOTE 2 The tube is generally connected with connectors made of various types of synthetic resin, etc.

- b) Liquid-feed part**, used to feed a sample, reagent and/or carrier, which is composed of a pump, damper, etc. and satisfies the conditions in **1)** to **3)**. Pumps used for this part include plunger pumps, syringe pumps, peristaltic (roller) pumps and solenoid pumps.

- 1) Constant flow rate is highly accurate.
- 2) Pulsating flow is small.

- 3) Materials of part in contact with the solution is excellent in chemical resistance and corrosion resistance.
- c) **Sample introduction part**, used for introducing the sample into the analyser, which is composed of an injector and tube. For example, a six-way valve equipped with a loop having a fixed volume is used for the injector. Flow passages constituting the introduction part may be arranged as follows.

- 1) Those arranged to introduce the sample into the fluid in a tube by using the injector, confluence type of flow passage, etc.

NOTE A device may be used for introducing a reagent into the flow of sample or carrier.

- 2) Those arranged so that the flow of sample and reagent are merged by using the injector and plural flow passages.

- d) **Reaction part**, carrying out the division, mixture, dilution, decomposition, concentration, separation, extraction, gas-liquid separation, chemical reaction, etc. in combination with a tube and the following devices.

- 1) **Divider**, used to divide the flow passage for connecting multiple different reactors or introducing the sample into multiple analytical instruments systems. Y connectors and T connectors are frequently used.

- 2) **Mixer**, used for mixing the carrier or reagent and sample. Usually, the mixing coil of tube wound in a coil shape or 8 shape shall be used. The column packed with chemically inert matter such as glass beads and polymerbeads or the small mixing chamber (inside capacity 100 μ L to 1 000 μ L) is also used in some cases. For titration, the small-sized mixer with a stirrer shall be used.

NOTE The mixer may also be used as the reactor.

- 3) **Diluter**, used for diluting the sample. The mixer shall be used as the diluter.

- 4) **Decomposer**, used for decomposing analyte in the sample by irradiation with ultraviolet radiation or heating. A UV lamp or a heating element around which a tube is wound in a coil shape shall be used.

- 5) **Solid phase concentrator**, used for the separation or concentration of analyte. The column packed with solid phase such as ion exchange resin, chelate resin, hydrophobic resin shall be mainly used.

- 6) **Solvent extractor**, used for the separation or concentration of analyte mainly by utilizing the partitioning of solvents. The extractor consists of the segmentor, extracting coil and phase separator described as follows.

- 6.1) **Segmentor**, used to join the water phase and organic phase at this part and to form the regular segment of each phase. The T connector shall be used where different types of fluids that do not mix each other flow in from different directions. In this way, two types of segments are formed in an alternating sequence.

- 6.2) **Extracting coil**, used to extract the target component from water phase to organic phase or vice versa. For example, the tube wound in a coil shape is used.

- 6.3) **Phase separator**, used to divide the segments of water phase and organic phase into the flow of each phase. The device used shall be the organic phase separator which separates the flow of segments of water phase and organic phase from the flow of organic phase by using the separation membrane (e.g. hydrophobic fluoro-resin membrane having an affinity for organic phase).

NOTE The phase separation efficiency is influenced by the geometrical arrangement of separator, the flow rate of both phases, etc.

- 7) **Gas separator**, used to separate analyte in the sample as gas. The analyte in the sample evaporates into gas or reacts to turn into gas which is absorbed into absorbing solution through a gas permeable membrane. For the sample divided into liquid phase and gas phase, the device which separates analyte by the difference in specific gravity shall be used. The gas components dissolved in liquid phase are separated by passing these through a hydrophobic fluoro-resin membrane.

NOTE The gas separator mainly used is that designed so that the gas permeable membrane is placed between two blocks or that of double tube structure where a fluoro-resin tube permeable to gas passes through a glass tube.

- 8) **Reactor**, used for chemical reaction of sample and reagent. The mixer shall be usually used as the reactor. The column packed with ion exchange resin, reducing agent, enzyme, catalyst, etc. and the tube with an inner wall where enzyme is immobilized may also be used. Some reactors are equipped with temperature control function for maintaining the reactor at a definite temperature.
- e) **Detection part**, generating the response signal corresponding to the concentration of analyte. According to the purpose of analysis, the following detectors may be used for the detection part.

The parts which the solution passes through or comes in contact with, such as flow cell, are required to be excellent in chemical resistance and corrosion resistance.

- 1) Absorption detector
 - 2) Fluorescence detector
 - 3) Chemiluminescence detector
 - 4) Electrochemical detector (e.g. potential difference detector and current detector)
 - 5) Flame photometer
 - 6) Atomic absorption spectrometer
 - 7) Inductively coupled plasma atomic emission spectrometer
 - 8) Inductively coupled plasma mass spectrometer
- f) **Display/recording part**, used for the display and record of signals from the detector, etc.

NOTE Some recording devices are equipped with data processing function.

g) **Attachment**, equipped with, for example, the following items as required.

- 1) **Data processing device**, capable of processing signals from the detector by various data processing methods.

NOTE Some data processing devices are equipped with a recording device.

- 2) **Automatic sample introduction device (autosampler)**
- 3) **Degassing device**, capable of removing sufficiently the dissolved gas in solvent in order to ensure the stable liquid feeding with a pump.
- 4) **Thermostat**, capable of maintaining the reactor such as the tube of reaction part at an arbitrarily constant temperature and of maintaining a uniform distribution of internal temperature.
- 5) **Back pressure coil**, for raising the pressure in the flow passage in order to prevent the production of air bubbles in the liquid-feed part, sample introduction part, reaction part and tube. Normally, the tube with inside diameter smaller than that of the tube which constitutes the flow system shall be wound in coil form, and the tube in coil shall be installed between the reaction part and detector or downstream from the detector.
- 6) **Pressure gauge**

4.3 Setting of analyser

Since the method for setting analyser differs according to analysis items and analytical methods, select suitable settings accordingly while paying attention to items described in a) to l).

- a) Type of flow system
- b) Inside diameter and length of tube from sample introduction part to the merging point of reagents
- c) Inside diameter and length of tube between each merging point of reaction part, and from final merging point to detection part
- d) Temperature of heating and cooling parts, if any, and the inside diameter, length, etc. of tube
- e) Type of liquid-feed part
- f) Material and inside diameter of tube used for parts other than flow system
- g) Flow rate or flow speed of reagent. When a carrier is used, flow rate or flow speed of the carrier.
- h) Type of sample injector and amount of sample introduced
- i) Type of reaction part
- j) Type and sensitivity of detector
- k) Time required for analysis per sample
- l) Conditions of data processing part (e.g. data processing device and recorder)

4.4 Stability validation of analyser

Make sure that all parts are assembled appropriately and start the operation.

Introduce the standard solution or sample as a preliminary test, and confirm that the measurements are not hindered by noise level, drift of base line, etc. Check the shape and height of peak, and also the time required for analysis per sample.

4.5 Analytical operation

4.5.1 General

Among various available methods for flow injection analysis, select suitable analytical operations in terms of purposes, using a combination of the reactive system, introduction method, detection method, etc. Details are as follows.

4.5.2 Introduction of sample and reagent

The sample and reagent shall be introduced by either of the following methods.

- a) Method to introduce the sample into a flow of reagent
- b) Method to introduce the reagent into a flow of sample
- c) Double injection method
- d) Merging zone method
- e) Sandwich injection method

4.5.3 Type of operations

Perform the following operations in combination, as required, according to purposes.

- a) Dilution
- b) Chemical reaction, colouring reaction, oxidation-reduction reaction, enzyme reaction, oxidative decomposition, etc.
- c) Separation and/or concentration
- d) Extraction e.g. solvent extraction and solid phase extraction
- e) Gas-liquid separation e.g. flotation and separation through a gas permeable membrane
- f) Flow injection titration
- g) Stopped flow

4.5.4 Water, reagent, solvent and standard solution

Water, reagent, solvent and standard solution used shall be as specified in **a)** to **c)**.

- a) **Water**, of A2 or A3 specified in **JIS K 0557**.
- b) **Reagent and solvent**, delivering quality as specified in **JIS** or equivalent to that, and not influencing the detection method used.
- c) **Standard solution**, with established metrological traceability to International System of Units (SI), which is used for the preparation of the calibration curve.