

JIS

JAPANESE INDUSTRIAL STANDARD

**Sampling methods for
industrial water and
industrial wastewater**

JIS K 0094—1994

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In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

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J I S

Sampling methods for industrial water
and industrial wastewater

K 0094-1994

1. Scope This Japanese Industrial Standard specifies the sampling of samples for industrial water and industrial wastewater (hereafter referred to as samples) and the works accompanied with this operation.

Remarks 1. The standards cited in this Standard are shown in Attached Table 1.

2. The sampling methods for the tests of dissolved oxygen, hexane extracts, carbon tetrachloride extracts, non-volatile hydrocarbons, bacterial examination, and biological examination shall be in accordance with JIS K 0101 and JIS K 0102.

The sampling methods for ultrapure water shall be in accordance with JIS K 0550, JIS K 0551, JIS K 0552, JIS K 0553, JIS K 0554, JIS K 0555, and JIS K 0556. Additionally, if there are prescriptions on sampling in other standards concerning water quality, follow them.

2. Matters in common The matters in common shall be as follows:

(1) General rules The general matters applied commonly to chemical analysis shall be in accordance with JIS K 0050.

(2) Definitions For the purposes of this Standard, the definitions given in JIS K 0101 or JIS K 0102 and the following definitions apply.

(a) sample Sample means the water sampled for tests.

(b) water sampler Water sampler means the implement for sampling water.

(c) water sampling apparatus Water sampling apparatus means the one by which water is sampled automatically according to the procedures which have been previously set. They are classified into an intermittently sampling apparatus and the sampling apparatus for mixed sample.

(3) Water Water shall be A1, A2, or A3 specified in JIS K 0557.

(4) Reagents The matters in common about reagents shall be as follows:

(a) When reagents are specified in Japanese Industrial Standard, they shall be the highest quality one or the one adequate for use, and when they have no Japanese Industrial Standard, the reagents giving no hindrance for test shall be employed.

- (b) The concentration of reagent solution shall be generally expressed with mass concentration g/l (in case of compound, use the mass of anhydrous compound), and it be placed behind the solution name of reagent with parentheses. For instance, sodium hydroxide solution (200 g/l) means it has about 200 g/l concentration.
- (c) The concentration of liquid reagent shall be expressed by mixed ratio with water as in [reagent (a+b)]. This expression means mixing a ml of reagent and b ml of water, and following JIS K 0050, this expression can be used for hydrochloric acid, nitric acid, sulfuric acid, phosphoric acid, aqueous ammonia, hydrogen peroxide, and others. When these are used without dilution, however, only the name of reagent shall be shown.
- (d) As for handling of reagents and their wastes, pay full attention in accordance with the laws and regulations related them.
- (5) Glassware Principally, glassware shall be used borosilicate glass-1 or borosilicate glass-2 specified in JIS R 3503.

Remarks: When testing silica, boron, sodium, potassium, arsenic, zinc, or others, be careful about dissolving of these elements from borosilicate glass.

- (6) Note, remarks, figure, and table Note, remarks, figure, and table are serially numbered at each clause.

3. Sample container and washing They shall be a polyethylene bottle with a mated stopper or a colorless glass bottle with a mated stopper for closely stoppering, and be used after satisfactorily washing so as to get rid of the hindrance for test.

Remarks: When sample container is specified in the test method in JIS K 0101, JIS K 0102, and JIS B 8224, the sample container specified in the above standard shall be used.

3.1 Sample container Sample container shall be as follows:

- (1) Polyethylene bottle with mated stopper Specified in JIS Z 1703⁽¹⁾⁽²⁾⁽³⁾⁽⁴⁾⁽⁵⁾.
- (2) Colorless glass bottle with mated stopper⁽²⁾⁽⁶⁾⁽⁷⁾⁽⁸⁾.

Notes ⁽¹⁾ JIS Z 1703 specifies only a narrow-mouthed bottle, but a wide-mouthed bottle or a bottle with easily washable shoulder may be used, and a polypropylene bottle with a mated stopper, polystyrene bottle with a mated stopper, or polycarbonate bottle with a mated stopper may be also serviceable.

- ⁽²⁾ Stopper shall be made of the same material as that of bottle, or be a screw lid made of synthetic resin. The stopper made of rubber or cork shall not be used.

- (³) Some bottles made by polyethylene may give a trace dissolution of metal such as molybdenum, chromium, or titanium, therefore pay attention them when special purpose is targeted.
- (⁴) Polyethylene bottles have a tendency to adsorb or to adhere suspension, phosphorus compounds, organic compounds, heavy metal elements, or others in sample on it. The adsorption of heavy metal elements can be prevented by the treatment for storing by adding nitric acid specified in JIS K 8541 or hydrochloric acid specified in JIS K 8180 (refer to 7).
- (⁵) In a polyethylene bottle, algae easily propagate themselves because of its permeability of air, therefore be careful about them.
- (⁶) JIS R 3503 specifies a wide-mouthed bottle and narrow-mouthed bottle, but either can be serviceable, and the bottle with easily washable shoulder can be serviceable, too.
- (⁷) Stopper shall be of ground type or screw type. In case of using a ground type, ground condition shall be superior. In this case, be careful about the dirt which easily adheres at ground place.

In case of a screw type, use a screw lid made of polyethylene, polypropylene or the one inside coated with polytetrafluoroethylene. An inner lid made of polyethylene is convenient to use because it does not contaminate sample.

- (⁸) While sample is kept inside a glass bottle, sodium, potassium, boron, silica, aluminum, or the like may slightly dissolve out. Some type of glass bottles may slightly dissolve out arsenic or zinc from it. The first class of hard glass gives less dissolving out of its components.

3.2 Method for washing The general method for washing shall be as follows:

(1) Washing of polyethylene bottle with a mated stopper

- (a) Prior to use(⁹), wash it with tap water or the like, and then wash sufficiently with A1 water.
- (b) When sampling(¹⁰) is made for the test of metal elements or organic substances, after treatment in (a), wash it with warm nitric acid (1+10) or warm hydrochloric acid (1+5), fill it with nitric acid (1+65), stopper closely, allow it to stand for 16 h or more, wash sufficiently with A1 water, and then wash sufficiently with A2 water or A3 water.
- (c) When sampling is made for the test of other items than those in (b) such as anion, after treatment in (a), fill it with A2 water, stopper closely, allow it to stand for 16 h or more, and wash sufficiently with A2 water or A3 water.
- (d) If there are prescriptions on washing methods in relevant standards concerning water quality, follow them.

- (e) The sample container, which has been washed, shall be stored until sampling will be made after evacuating inside and stoppering closely.

Notes (⁹) Generally speaking, use the one newly prepared. The condition, under which it has been used, gives no influences to next test, it may be used again.

- (¹⁰) When the sample for a trace of metal elements is sampled, use newly prepared one.

(2) Washing of colorless glass bottle with a mated stopper

- (a) Follow (1)(¹¹).

Note (¹¹) When glass bottles are reused, follow Note (⁹).

4. General operation of sampling The general operation of sampling shall be classified into sampling with a water sampler, sampling with a water sampling apparatus, and the procedure using a sampling valve.

4.1 Sampling with water sampler Sampling with water sampler shall be classified into sampling carried directly with a sample container, sampling with a bucket such as polyethylene-made bucket, sampling with a Heyroth water sampler, and sampling with a Vandorn water sampler.

4.1.1 Sampling with a sample container This is a method to sample surface water using a sample container.

- (1) Implement Implement shall be as follows:

- (a) Sample container Follow 3.

- (2) Operation Operation shall be carried out as follows:

- (a) Wash(¹) a sample container 3 or 4 times with the water at sampling site.
- (b) Immerse slowly the sample container to let the water at sample site flow in until it gets full(²), and stopper it closely.

Notes (¹) Pay attention not to contaminate the water at sampling site by the water which washed the container.

- (²) When it is feared that sample may be frozen, leave about 10 % space of whole volume of a sample container.

4.1.2 Sampling with buckets This is a method to sample surface water using a bucket or a water sampler (a ladle) with a handle or the like.

- (1) Implement Implements shall be as follows:

- (a) Water sampler It is a bucket or a water sampler (a ladle) with a handle or the like(³), which is made of polyethylene(⁴). Fig. 4.1 shows an example of a water sampler with a handle.

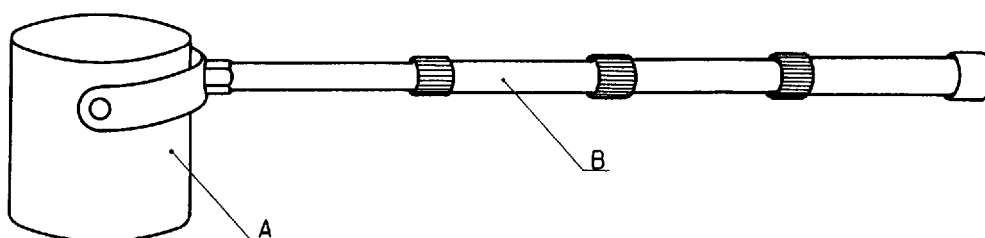
(b) Sample container Follow 3.

Notes (³) If occasion calls, a bucket equipped with rope can be used. In this case, be careful not to soil the rope with such as mud.

It would be convenient to use the handle with telescopic structure.

(⁴) It is permissible to use samplers made of other synthetic resin such as polypropylene. A stainless steel-made sampler is permissible in other case than sampling for trace analysis of heavy metals.

Fig. 4.1 Example of water sampler with a handle



A: Container made of polyethylene

B: Handle (telescopic type made of aluminum alloy or stainless steel)

(2) Operation Operation shall be as follows:

- (a) Wash a water sampler with the water at sampling site(¹).
- (b) Ladle the water at sampling site to fill a water sampler, wash quickly a sample container with the water just ladled, flow the water in sample container until it gets full(²), and stopper closely(⁵).

Note (⁵) When sample is in plenty of suspension, agitate it to make suspension homogeneous, and flow quickly it into a sample container.

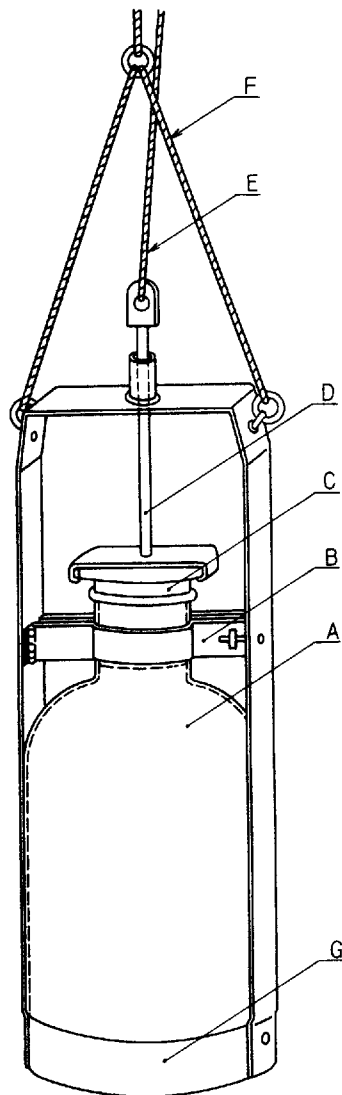
4.1.3 Sampling with a Heyroth water sampler This is a method to make sampling at each depth level in such as reservoir, water channel, river, lake and marsh, well, and sea area, using a Heyroth water sampler.

(1) Implement Implements shall be as follows:

- (a) Heyroth water sampler A sample container(⁶) is fixed at the frame equipped with a plumb. Fig. 4.2 shows its example.
- (b) Sample container Follow 3.

Note (⁶) There is a water sampler with 500 ml to 1000 ml capacity.

Fig. 4.2 Example of a Heyroth water sampler



- A: Sample container
 B: Metal holder
 C: Stopper of sample container
 D: Metal holder for sampling
 E: Chain or string for stopper opening
 F: Rope for suspending
 G: Plumb

(2) Operation Operation shall be carried out as follows:

- (a) Confirm there is no unusualness at each part of Heyroth water sampler such as rope for suspending, and chain for stopper opening⁽⁷⁾⁽⁸⁾.
- (b) Attach the sample container which has been washed with the water at sampling site in advance, and fix the mated stopper to a metal holder for stopper opening⁽⁹⁾.
- (c) Sink slowly a water sampler at the depth decided for sampling using a rope for suspending, and confirm its depth.
- (d) Open the stopper by pulling the chain for stopper opening, and allow it to stand for a while to take sample in.
- (e) Loosen the chain for stopper opening, to close the stopper, draw up the water sampler, and remove the sample container, followed by close stoppering.

Notes (7) When handling it, follow the instruction manual of a water sampler.

(8) Mark previously the signs showing depth on the rope for suspending.

(9) It is advisable to fasten near end of the rope for suspending in order not to drop and lose the water sampler into water.

Remarks 1. Convenient type has a sample container which has been fixed to the suspending rope equipped with a plumb. Sample can be taken in by pulling a string for sampling to unfasten a soft polyvinyl chloride tube. In this case, surely fasten the sample container using the suspending rope equipped with a plumb, and be careful about it, because pulling a stopper may cause easily slipping out of sample container.

4.1.4 Sampling with Vandorn water sampler This is a method to make sampling at each depth level in such as reservoir, water channel, river, lake and marsh, well, and sea area, using a Vandorn water sampler.

(1) Implement Implements shall be as follows:

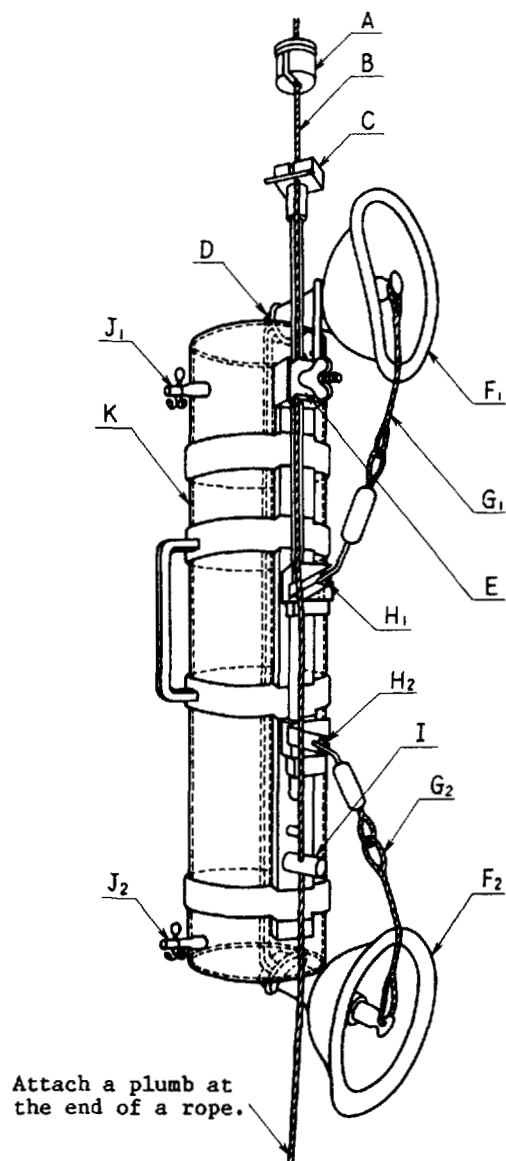
(a) Vandorn water sampler This is a synthetic resin-made cylinder (10)(11), upper and lower of which lids made of synthetic rubber are attached. Fig. 4.3 shows an example of a Vandorn water sampler.

(b) Sample container Follow 3.

Notes (10) The cylinder is made of such material as opaque polyethylene, or transparent acrylic resin or polycarbonate.

(11) There is a cylinder having 1 l to 20 l capacity.

Fig. 4.3 Example of Vandorn water sampler



- A: Messenger
 B: Rope (or wire) for suspending
 C: Messenger receiver
 D: Rubber string
 E: Fastening metal for rope
 F₁, F₂: Rubber lid
 G₁, G₂: Wire for rubber lid
 H₁, H₂: Fixing metal of wire for rubber lid
 I: Fixing place of rope
 J₁, J₂: Soft polyvinyl chloride tube with a pinch cock for taking out sample
 K: synthetic resin-made cylinder

(2) Operation Operation shall be carried out as follows:

- (a) Confirm whether or not there is unusualness about such part as a messenger receiver, upper lid and lower lid, wire for rubber lid, rope for suspending of Vandorn water sampler⁽⁷⁾⁽⁸⁾.
- (b) Wash previously the cylinder, upper lid and lower lid of a Vandorn water sampler, and buckets⁽¹⁾ with the water of sampling site.
- (c) Attach the upper lid and lower lid on the fixing metal of wire⁽⁹⁾⁽¹²⁾.
- (d) Sink slowly a Vandorn water sampler to depth for sampling using rope for suspending, and confirm its depth.
- (e) Move up and down 2 or 3 times the Vandorn water sampler to make replace the water kept inside of a cylinder, drop a messenger, close the upper lid and lower lid, and take sample.

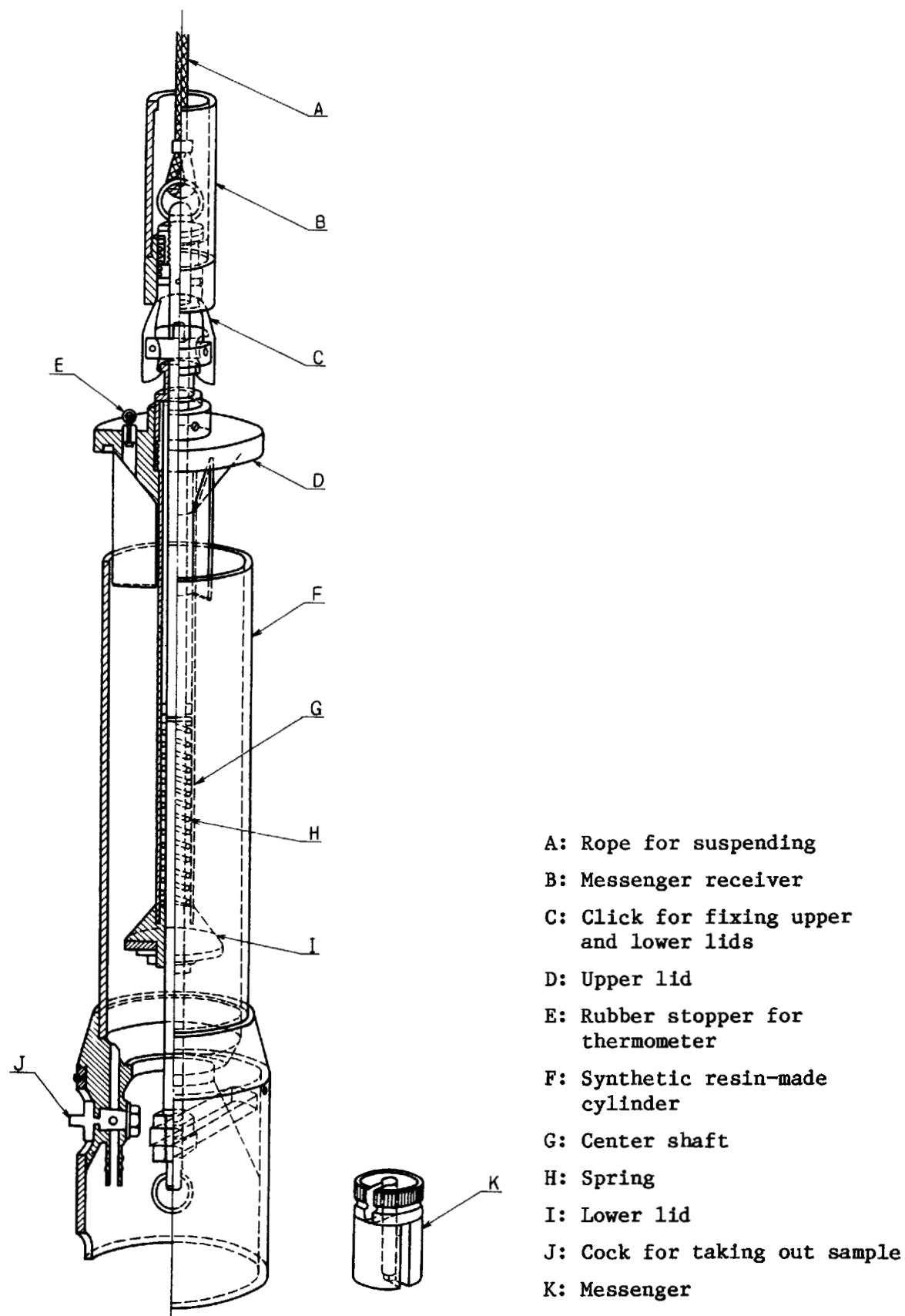
- (f) Draw up the Vandorn water sampler, open the soft polyvinyl chloride tube equipped with a pinch cock for sample taking from the cylinder, wash buckets with a part of sample, and transfer the sample into the buckets⁽¹³⁾.
- (g) While stirring sample, wash the sample container a couple of times with this sample, and fill the sample container with the sample⁽²⁾, followed by close stoppering.

Notes (¹²) Elastically weakened spring of the messenger receiver may cause to close the lid of a cylinder while sampling is being made, therefore pay attention to it.

(¹³) It is permissible to attach soft polyvinyl chloride tube at the sample exit of Vandorn water sampler and to make the sample flow in the sample container. However, suspensoid may become ununiformity due to sedimentation of suspension in sample onto the cylinder bottom, therefore pay attention to it.

Remarks 2. When using a water sampler, there is a method to sample by an insulated water sampler (example shown in Fig. 4.4). In this case, the sample container and operation shall follow 4.1.4.

Fig. 4.4 Example of insulated water sampler



4.2 Sampling with water sampling apparatus This is a method to raise automatically water at sampling site using a water sampling apparatus, and to take the sample into a sample container.

4.2.1 Sampling with intermittent sampling apparatus Carry out sampling with an intermittent sampling apparatus.

(1) Apparatus and implement Apparatus and implements shall be as follows:

- (a) Intermittent sampling apparatus The methods to sample with an intermittent sampling apparatus⁽¹⁴⁾⁽¹⁵⁾⁽¹⁶⁾⁽¹⁷⁾⁽¹⁸⁾ shall be classified into sampling method interlocked with a water-quality tester, intermittent (or periodical) sampling method, and centralized piping method.

Sampling method interlocked with a water-quality tester Interlocking with a water-quality automatic tester, the signal from the water-quality automatic tester, issued when the change of water quality is detected, shall be received, and the sample of specified amount shall be automatically taken. If necessary, a preserving apparatus at lower temperature shall be annexed. When unusual value of water quality is detected, this is used for sampling water. Fig. 4.5 shows an example of sampling method interlocked with water-quality tester.

Intermittent (or periodical) sampling method Interlocking with a water level gauge, the signal from water level shall be received when water level gets a specified value, and the sample of specified amount shall be automatically taken. At every time that is set previously, a specified amount of water shall be automatically sampled into a sample container. If necessary, a preserving apparatus at lower temperature shall be annexed. This can be used to sample the water which flows out intermittently.

Sampling method by centralized piping Water from various sampling places is always sent to such as a laboratory, and required amount of sample can be freely taken at arbitrary time. This method has an advantage to centralize samples from lot of sites to one place. This method can be used when water quality control is necessitated at such place as factory or workshop⁽¹⁹⁾⁽²⁰⁾. Fig. 4.6 shows an example of sampling method by centralized piping.

(b) Sample container Follow 3.

Notes ⁽¹⁴⁾ There are various types; sampling method at intermittent sampling apparatus; the range and recording method of set conditions about sampling time; the kind, volume, and number of sample containers that were being built in; and preserving method at lower temperature, therefore they shall be chosen depending on the purpose of use.

⁽¹⁵⁾ Because an intermittent sampling apparatus is installed at high humid place, the body of apparatus shall be made of such as synthetic resin, reinforced material of synthetic resin, or stainless steel, and an electric circuit shall be constructed with moisture-resistant construction. Sample introducing pipe shall be corrosion-resistant material (such as stainless steel, synthetic resin, or synthetic-resin lined tube).

- (¹⁶) When employing an intermittent sampling apparatus for sampling, the mechanism to automatically evacuate the water staying in sample introducing pipe in advance, is needed. Generally, run a sampling pump prior to sampling procedure, evacuate the water staying in sample introducing pipe at the rate of 0.8 m/s or more, then switch a water-route changing valve at specified time, and take the sample. Alternatively, the water in a sample introducing pipe can be evacuated by compressed air.
- (¹⁷) The end point of sample introducing pipe of an intermittent sampling apparatus shall be attached to face downstream from sampling position. If a strainer is needed, the coarsely meshed one (with finer sieve opening than inside diameter of sample introducing pipe) shall be attached. In case of water channel or cesspool, attach it at the center of flow, choose the place where no floating of surface layer and deposit at bottom layer are not sucked in, and have its depth be tolerable enough to the fluctuation of water level.
- (¹⁸) When water contains a lot of suspension, it is advisable to use a large-capacity sampling pump or single-shaft eccentric pump and to prevent the blockage of a pump by suspended matter by means of attaching a back-washing mechanism or a washing mechanism by reverse rotation of a pump. Piping system shall be periodically cleaned because of easily sticking of slime.
- (¹⁹) In case of sampling method by centralized piping, send water using a large-capacity sampling pump at the rate of 0.8 m/s or more flow rate of water in sample introducing pipe. Install a branch pipe from sample introducing pipe at the point immediately before sampling. Attach soft polyvinyl chloride tube to this branch pipe, and make water flow out always from it. Set flow ratio of water between in sample introducing pipe and in branch pipe to be from 2:1 to 5:1. When the distance of sample introducing pipe is so long that it gives a large pressure loss in the pipe, install a line pump on the way of the pipe to pressure up. Clean periodically the sample introducing pipe.
- (²⁰) The sampling method by centralized piping makes pipe for sample introducing very long, so it is required to provide heat-insulating measures in order to prevent fluctuation of water temperature in the piping in air or under ground. In case of under-ground piping, measures to prevent damage due to use of flexible joints shall be made, and be careful about the depth of burying. At the rising point of piping, install an air vent valve.

(2) Operation Operation shall be carried out as follows:

- (a) Install the sample introducing pipe of an intermittent sampling apparatus⁽²¹⁾ at sampling site⁽²²⁾, and install a drain pipe at the place⁽²³⁾ where the water at sampling site is not contaminated. confirm there is no unusualness in an intermittent sampling apparatus and sample introducing pipe.
- (b) Wash sufficiently the sample introducing system of an intermittent sampling apparatus in advance, attach a cleaned sample container, if preserving apparatus⁽²⁴⁾ at low temperature has been provided, run the apparatus, and then run the sampling apparatus after setting the conditions for sampling.
- (c) After sampling finished, take out the sample container from the intermittent sampling apparatus⁽²⁾.

Notes (²¹) When handling an intermittent sampling apparatus, follow its instruction manual respectively. In case of sampling method by centralized piping, take samples according to 4.3.

(²²) Choose sampling site according to the purpose of test, but in case of sampling at water channel or cesspool, attach sample introducing pipe at the center of water flow so as to meet the variation of water level and so as not to suck in the deposit at bottom. Face the end point of sample introducing pipe toward downstream. If a strainer is needed, the coarsely meshed one (with finer sieve opening than inside diameter of sample introducing pipe) shall be attached, but if sample contains suspended matter whose size is larger than the sieve opening of the strainer and inside diameter of the sample introducing pipe, the sample just taken may be different from that taken by other methods.

(²³) Attach a drain pipe at the downstream far apart from sampling site.

(²⁴) Low-temperature preserving ability of an intermittent sampling apparatus is influenced with ambient temperature of the intermittent sampling apparatus and with direct sunshine, therefore be careful about them.

Fig. 4.5 Example of intermittent (or periodical) sampling method and method interlocked with water-quality tester

