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JAPANESE INDUSTRIAL STANDARD

Testing Methods for Boiler Feed Water and Boiler Water

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Contents

	Page
1. Scope	1
2. Common Matters	1
2.1 General Matters	1
2.2 Common Matters of This Standard	1
2.3 Marking of Test Results	5
2.4 Test Error	5
3. Sample	5
3.1 Sampling	5
3.2 Storing Treatment of Sample	13
3.3 Handling of Sample	13
3.4 Testing Time	14
4. Pretreatment of Sample	14
4.1 Decomposition by Hydrochloric Acid or Nitric Acid	14
4.2 Decomposition by Nitric Acid and Perchloric Acid	15
4.3 Decomposition by Nitric Acid and Sulfuric Acid	16
4.4 Pretreat in the Case of Applying Atomic Absorption Spectrophotometry	17
5. Appearance	17
6. Turbidity	17
6.1 Visual-Sense Turbidity	17
6.2 Transmitted-Light Turbidity	17
6.3 Integral Spherical Bulb Turbidity	18
7. pH	18
8. Electric Conductivity	18
8.1 General Test	18
8.2 Electric Conductivity after Passing through Strong Acid Cation-Exchange Resin Layer (Hydrogen Ion Type)	18
9. Acid Consumption Quantity (Alkalinity)	19
9.1 Acid Consumption Quantity (pH 4.8) (M-Alkalinity)	19
9.2 Acid Consumption Quantity (pH 8.3) (P-Alkalinity)	19
10. Alkali Consumption Quantity (Acidity)	19
10.1 Alkali Consumption Quantity (pH 8.3) (Total Acidity)	19
10.2 Alkali Consumption Quantity (pH 4.8) (Mineral Acid Acidity) ..	19

	Page
11. Hardness	21
11.1 Total Hardness	21
11.2 Calcium Hardness	21
11.3 Magnesium Hardness	21
12. Suspensoids and Evaporation Residuals	21
12.1 Suspensoids	21
12.2 Total Evaporation Residuals	21
12.3 Soluble Evaporation Residuals	21
12.4 Ignition Residuals	21
12.5 Ignition Reducing Quantity	21
13. Oxygen Consumption Quantity by Potassium Permanganate at 100°C (COD _{Mn})	21
14. Organic Carbon (TOC)	21
15. Hexane Extract	21
16. Carbon Tetrachloride Extract	21
17. Dissolved Oxygen (O)	21
17.1 Winkler's Method	22
17.2 Indigo Carmine Colorimetric Method	31
18. Hydrazine (N ₂ H ₄) [Hydrazinium Ion (N ₂ H ₅ ⁺)]	39
18.1 <i>p</i> -Dimethylaminobenzaldehyde Absorptiometry	39
18.2 Iodine Titrimetric Method	41
19. Chloride Ion (Cl ⁻)	42
19.1 Mercury (II)-Thiocyanate Absorptiometry	42
19.2 Mercury (II)-Nitrate Titrimetric Method	42
19.3 Silver-Nitrate Titrimetric Method	43
19.4 Ion Electrode Method	43
20. Sulfurous Acid Ion (SO ₃ ²⁻)	43
20.1 Iodine Titrimetric Method	43
21. Sulfuric Acid Ion (SO ₄ ²⁻)	46
21.1 Barium Chromate-Diphenylcarbazide Absorptiometry	46
21.2 Barium Chromate Absorptiometry	46
21.3 Gravimetric Method	46
22. Phosphoric Acid Ion (PO ₄ ³⁻)	46

	Page
22.1 Phosphoric Acid Ion (PO_4^{3-})	46
22.2 Phosphoric Acid Ion Other than Orthophosphoric Acid (Hydrolytic Phosphoric Acid)	51
23. Silica (SiO_2)	52
23.1 Ionic Silica	52
23.2 Dissolved and Colloidal Silica	56
23.3 Total Silica	56
24. Ammonium Ion (NH_4^+)	59
24.1 Indophenol Blue Absorptiometry	59
24.2 Ion Electrode Method	61
25. Sodium (Na)	62
25.1 Flame Photometry	62
25.2 Ion Electrode Method	63
26. Calcium (Ca)	63
26.1 Chelatometric Titration Method	63
26.2 Atomic Absorption Spectrophotometry	63
27. Magnesium (Mg)	63
27.1 Chelatometric Titration Method	64
27.2 Atomic Absorption Spectrophotometry	64
28. Copper (Cu)	64
28.1 Diethyldithiocarbamic Acid Absorptiometry	64
28.2 2, 9-Dimethyl-1, 10-Phenanthroline Absorptiometry	64
28.3 Atomic Absorption Spectrophotometry	69
29. Zinc (Zn)	72
29.1 Dithyzone Absorptiometry	72
29.2 Atomic Absorption Spectrophotometry	72
30. Nickel (Ni)	74
30.1 Dimethylglyoxime Absorptiometry	74
30.2 Atomic Absorption Spectrophotometry	74
31. Iron (Fe)	76
31.1 1, 10-Phenanthroline Absorptiometry	76
31.2 2, 4, 6-Tri-2-Pyridil-1, 3, 5-Triazine Absorptiometry	76

	Page
31.3 4, 7-Diphenyl-1, 10-Phenanthroline Absorptiometry	78
31.4 Atomic Absorption Spectrophotometry	80
32. Aluminium (Al)	82
32.1 8-Quinolinol Absorptiometry	82
32.2 Atomic Absorption Spectrophotometry	82

JAPANESE INDUSTRIAL STANDARD

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Testing Methods for Boiler Feed Water
and Boiler Water

B 8224-1986

1. Scope

This Japanese Industrial Standard specifies testing methods for boiler feed water, hereinafter referred to as the "feed water", boiler water and steam.

Remark: In this Standard the units and numerical values shown in { } are in accordance with the conventional unit system and are appended for informative reference.

2. Common Matters

2.1 General Matters In this Standard matters not specified specially shall be as follows:

- (1) General Rules General matters common to chemical analysis shall be in accordance with JIS K 0050.
- (2) Testing Methods Testing methods not specified in this Standard shall be in accordance with JIS K 0101.
- (3) Sampling Methods General matters common to sampling shall be in accordance with JIS K 0094.
- (4) Absorptiometry General matters common to absorptiometry shall be in accordance with JIS K 0115.
- (5) Atomic Absorption Spectrophotometry General matters common to atomic absorption spectrophotometry shall be in accordance with JIS K 0121.
- (6) Infrared Spectroscopy General matters common to infrared spectroscopy shall be in accordance with JIS K 0117.
- (7) Ion-Selective Electrode Method General matters common to ion-selective electrode method shall be in accordance with JIS K 0122.

2.2 Common Matters of This Standard Common matters of this Standard shall be as follows:

- (1) Determination Range The determination ranges indicated in respective testing methods are marked by mass (mg) in final solution. However, in atomic absorption spectrophotometry, ion-selective electrode method, and testing methods of organic carbon (TOC) and dissolved oxygen their determination ranges are marked by concentration (mg/l) in final solution.
- (2) Water The water in this Standard, unless specially specified⁽¹⁾, shall be distilled water or ion-exchanged water (water refined by ion-exchanged resin) and, as follows:
- (a) Distilled Water The distilled water shall be not more than 2 $\mu\text{S}/\text{cm}$ (25°C)⁽²⁾ in electric conductivity and not less than 60 min in coloration holding time⁽³⁾ by potassium permanganate. However, the water to be used for preparation of standard liquid to be stored for a long period shall, at the time of distillation, be added by potassium permanganate solution (3 w/v %) and, in the state holding faint red purple colour, refined.

For preparation and test of reagents to be used for determination of heavy metal element, the water refined by using distillation apparatus made of hard glass or quartz shall be used.

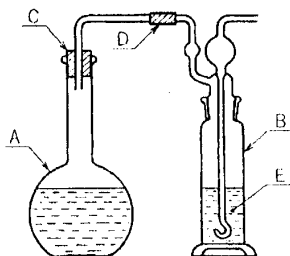
For preservation of water, depending upon the test purpose vessel with ground-in stopper of polyethylene tetrafluoride resin, polyethylene, quartz glass, hard glass, etc. shall be used.

- (b) Ion-Exchanged Water The ion-exchanged water shall be refined by using mixture of strong acidic cation-exchange resin (hydrogen ion type) and strong basic anion-exchange resin (I type)⁽⁴⁾ (hydroxide ion type), have electric conductivity of not more than 1 $\mu\text{S}/\text{cm}$ (25°C)⁽⁵⁾, have holding time of coloration⁽³⁾ by potassium permanganate of not less than 60 min⁽⁶⁾, and filtered by filter film of not more than 0.45 μm ⁽⁷⁾ in aperture size.

The ion-exchanged water may be used for test for other than oxygen consumption amount (COD_{Mn}) by potassium permanganate at 100°C, hexane extracts, carbon tetrachloride extracts, organized carbon (TOC), ammonium ion, etc. According to the test purpose, similarly to the case of (a), a vessel of suitable quality material shall be selected to be stored.

- (c) Water Containing No Dissolved Oxygen Put distilled water or ion-exchanged water into a flask, boil for about 5 min, after removing dissolved oxygen, connect a gas washing bottle containing alkaline pyrogallol solution⁽⁸⁾ as shown in Fig. 1, and shut off from oxygen in atmospheric air to leave cooling.

Fig. 1. An Example of Cooling and Storing of Water Containing No Dissolved Oxygen



- A: flat bottom flask 1000 ml
- B: gas washing bottle 250 ml
- C: rubber stopper
- D: rubber tube
- E: alkaline pyrogallol solution

- (d) Water Containing No Carbonic Acid Put distilled water or ion-exchanged water into a flask, after removing dissolved gas and carbonic acid by boiling for about 5 min, put potassium hydroxide solution (25 w/v %) in a gas washing bottle by using an apparatus similar to Fig. 1, shut off carbon dioxide in air and leave cooling.

- Notes (1) Preparation of reagents to be used for test for oxygen consumption amount (COD_{Mn}) by potassium permanganate at 100°C , organic carbon (TOC), etc., and preparation method for water to be used for test are specified in respective test methods.
- (2) Let the condensate water flow through a cell of direct flow liquid type, and measure the electric conductivity.
- (3) It means the period of time until coloration extinguishes when potassium permanganate solution (N/100) 0.2 ml and sulfuric acid (1 + 1) 2 ml is added to water 500 ml to be mixed thoroughly by stirring and is stood still.
- (4) In the case where coexistence of silica of not less than 0.1 mg SiO_2 / l does not cause influence (for example, water to be used for washing of glass devices and the like), strong basic anion exchange resin (type II) may be used.
- (5) Measure the electric conductivity by flowing the flowing out water from column to direct flowing liquid type cell.
- (6) In the case where coexistence of organic substance of micro-quantity does not cause influence, the ion-exchanged water of which coloration holding time by potassium permanganate is about 10 min may be used.

- (⁷) As to the ion-exchanged water, because non-electrolyte, suspensoid, etc. are not almost removed, filter by using a filter film of not more than 0.45 μm in aperture size.
- (⁸) Dissolve pyrogallol 6 g in water 50 ml, and put into a brown bottle. Separately, dissolve potassium hydroxide 30 g in water 50 ml, and at the time of use, mix the both solutions to use. This solution 1 ml absorbs oxygen about 12 ml (about 17 mg).

Remark: The distilled water is the refined water by using distillation method at the final process of refining of water. Further, the ion-exchanged water is the refined water by using ion-exchange method at the final process of refining. In the ion-exchanged water, the non-electrolyte, suspensoid, etc. in the original water coexist. Further, during the strong basic anion-exchange resin is new, micro-amount amines may elute.

(3) Reagents Reagents to be used in test shall be as follows:

- (a) Unless particularly specified, the reagent of quality of the highest grade of reagents specified in JIS or that of quality of the highest grade based on the reagent mark certification system of the Ministry of International Trade and Industry shall be used.

For standardization of specified liquids, reagents specified in JIS K 8005 shall be used.

Further, in case where not specified in JIS, the reagent of highest quality equivalent to this shall be used.

- (b) In preparation of reagents, the concentration indicated after the name of solution in parentheses means the approximate concentration.

For example, sodium hydroxide (N/10) indicates that it is the concentration of sodium hydroxide solution of about N/10.

Further, the concentration indicated before the name of solution means the correct concentration. However, generally, it is indicated by a numerical value having no fraction and separately the factor is to be obtained.

- (c) The water to be used for preparation of reagents shall, as a rule, be the water specified in (2), but in the case where it is specified in respective items, it shall be in accordance with them.
- (d) In the case where the standard solution is prepared by diluting the standard solution, not less than 10 ml of it shall be taken by using a transfer pipet.
- (e) Names of reagents shall be in accordance with the compound nomenclature specified based on the inorganic nomenclature and organic nomenclature of International Union of Pure and Applied Chemistry (IUPAC) by Corporate Juridical Person Japanese Chemical Association, as far as possible.

- (4) Glass Devices As to glass devices, those specified in JIS R 3503 and JIS R 3505 shall, as a rule, be used, but in the case where special device is required its example is shown in figure or explained in respective items.

Further, in the case of accompanying heating operation, the device of hard quality grade 1 of JIS R 3503 shall be used.

Remark: In the case where silica, boron, sodium, potassium, arsenic, zinc, etc. are tested, take care sufficiently to elution of glass component from hard glass.

- (5) Measurement of Absorbance (Absorptiometry) In the case where there is no description on absorption cell, that of 10 mm in optical path length shall be used.
- (6) Working Curve (Absorptiometry, Atomic Absorption Spectrophotometry) At the time of preparation of working curve, divide the determination range indicated in the testing method into 5 to 6 stages and take the standard solution so as to be coincident with this.

Prepare the working curve only to the determination range.

In absorptiometry, the preliminarily prepared working curve may be used. In atomic absorption spectrophotometry, use the newly prepared working curve, and in the case where many samples are tested, in the midway of testing, use adequately the standard solution to conduct affirmation of absorbance.

2.3 Marking of Test Results Marking of test results shall be as follows:

- (1) In the case of marking test results, when there are two or more testing methods, describe clearly either method is conducted.
- (2) Take the unit to indicate the concentration of test results as mg/l.

2.4 Test Error The error on the test results shall be in accordance with JIS Z 8402.

3. Sample

The sample shall be the water sampled from the feed water, boiler water, and steam for conducting each test, and the sample, capable of being representative of feed water, boiler water and steam at respective sampling positions.

3.1 Sampling Sampling shall be as follows:

- (1) Sampling Position Take the sample of feed water, boiler water and steam from the position indicating the highest value in the concentration of test component in the system or a suitable position to the test purpose, as a rule.

An example of sampling of feed water is shown in Table 1. Take the boiler water from the continuous blow tube set at steam drum, sampling inner tube of boiler water, etc.

- (2) Sample Vessel As to the sample vessel, unless particularly specified, use a colorless hard glass bottle with ground-in stopper (hard glass grade 1) or polyethylene bottle with ground-in stopper (or polypropylene bottle or the like) shown in Table 2, as a rule.

Further, use the sample vessel capable of stoppering tightly in order to prevent mixing-in of substances from outside into the sample and relief of test component from the sample. However, for stopper do not use rubber, cork, etc.

As to the new sample vessel not yet used, at first wash with warm nitric acid (1 + 10) or warm hydrochloric acid (1 + 10), and further wash⁽⁹⁾ sufficiently with water. In the case where the once used sample vessel is reused, wash with warm hydrochloric acid (1 + 5), and further wash sufficiently with water.

Note ⁽⁹⁾ It is preferable to promote washing effect by using detergent such as cleanser and the like. However, wash so sufficiently with water that alkali content, fats and oils, surface-active agent, etc. do not remain by adhering with adsorption, and, if possible, fill with water.

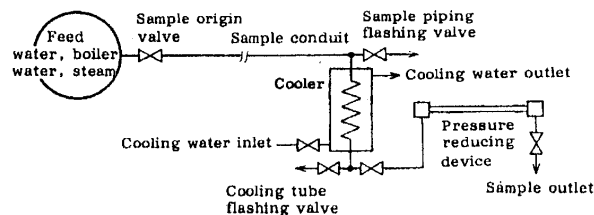
Table 1. An Example of Sampling Position of Feed Water

Test item ⁽¹⁰⁾	Sampling position
pH Electric conductivity Electric conductivity after passing through cation-exchange resin layer ⁽¹²⁾ Fats and oils ⁽¹³⁾ Hydrazine ⁽¹⁴⁾ Silica	Deaerator inlet or economizer inlet ⁽¹¹⁾
Iron, copper	Deaerator outlet or economizer inlet
Hardness	Feed water system before injection of de-hardening agent
Dissolved oxygen	In the case where deaerator exists, deaerator inlet and outlet. In the case where there is no deaerator, feed water system before injection of deoxygenation agent.

Notes ⁽¹⁰⁾ Other than the test item shown in Table 1, the feed water sample for determination of total evaporation residuals and phosphoric acid ion of feed water of once-through boiler of not more than 2.5 MPa {25 kgf/cm²} in pressure shall be taken from the feed water system after the returning water from separator has been combined with the supplementary water.

- (¹¹) As to the feed water system before injection of pH adjusting agent or recovery water and supplementary water individually, in the case where measuring watch of pH, and electric conductivity is required the feed water sample shall be taken out from the position corresponding to those purposes.
 - (¹²) It means the electric conductivity measured for the sample through hydrogen ion type strong acidic cation-exchange resin.
 - (¹³) These mean hexane extract or carbon tetrachloride extract.
 - (¹⁴) This is the test item in the case where hydrazine is injected as deoxygenation agent into feed water.
- (3) Sampling Apparatus In the case of taking the sample of feed water, boiler water and steam, the cooling part of sample and pressure-reducing system shall be, as a rule, in accordance with Fig. 2 and the following. However, if the sample is low temperature (room temperature) and low pressure, the cooler and pressure-reducing device may be omitted.

Fig. 2



- (a) Sample Conduit For the sample conduit to introduce the sample taken out from each system, the stainless steel pipe specified in JIS G 3463 or JIS G 3459 shall be used. However, depending upon the test item and its concentration, carbon steel pipe specified in JIS G 3454 or JIS G 3456 may be used.
- (b) Cooler In a high pressure boiler, for material of cooler, stainless steel shall be used. The capability of cooler shall be capable of taking the sample at not higher than room temperature (in the case of sampling for determination of dissolved oxygen, lower than room temperature by about 2°C) at a flow rate not less than 1 l/min.

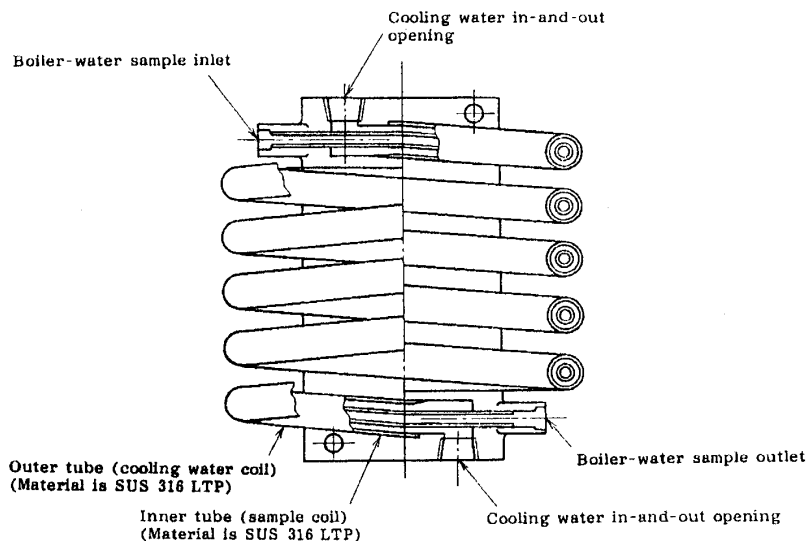
Before attaching the cooler, the hydraulic test shall be carried out by applying a pressure of not less than 1.5 times the highest working pressure for about 10 min, and the absence of leakage, confirmed. The representative example of structure and material of cooler is shown in Fig. 3.

Table 2. Test Items and Sample Vessels

Test item	Sample vessel
Hardness Silica Acid consumption quantity (pH 8.3) (P-alkalinity) Acid consumption quantity (pH 4.8) (M-alkalinity) Total evaporation residual Chloride ion	Polyethylene bottle
Iron Copper Phosphoric acid ion	Hard glass bottle
pH Electric conductivity Electric conductivity after passing through cation- exchange resin layer Hydrazine	Polyethylene bottle or those specified in respective testing methods ⁽¹⁵⁾
Fats and oils ⁽¹³⁾ Dissolved oxygen Sulfurous acid ion	Those specified in respective testing methods

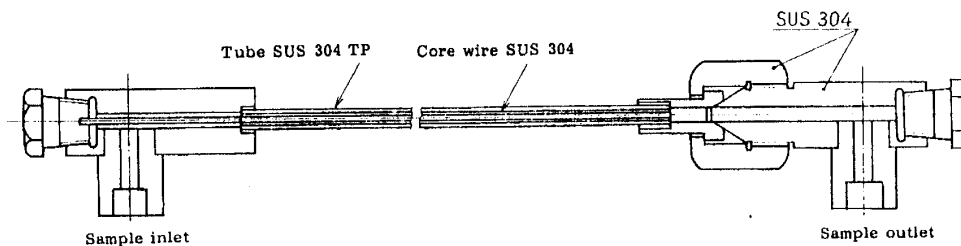
Note ⁽¹⁵⁾ In the case where pH is not less than 10, electric conductivity and that after passing through the cation-exchange resin layer are not less than 10 $\mu\text{S}/\text{cm}$ (25°C), and the concentration of hydrazine is not less than 1 mg $\text{N}_2\text{H}_4/\text{l}$, polyethylene bottle may be used.

Fig. 3. An Example of Cooler



- (c) **Pressure Reducer** The pressure reducer shall be able to reduce pressure so that the sample can be taken at a rate of 1 to 2 l /min in the state where each valve of sampling system is fully opened. The representative example of its structure and material is shown in Fig. 4.

Fig. 4. An Example of Pressure Reducer

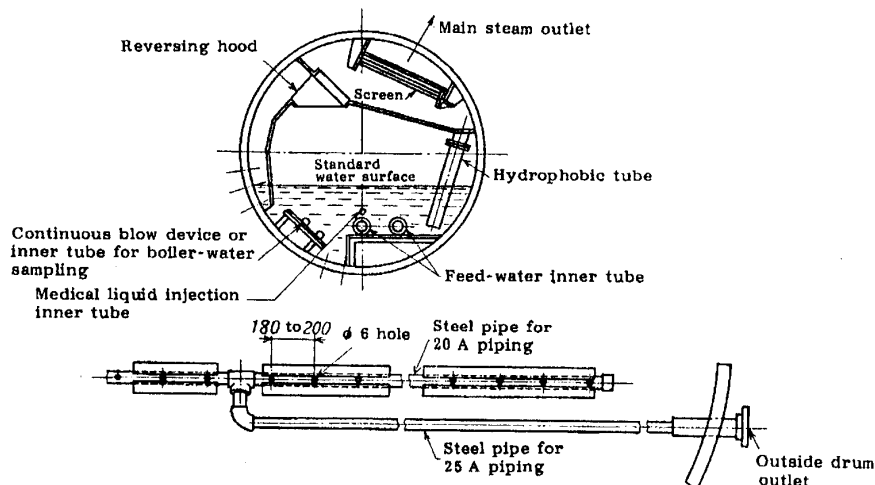


- (d) **Sampling Tube in Boiler** The boiler shall be provided with a continuous blow device capable of sampling, inner tube for sampling of boiler water, etc.

The representative example of setting position of continuous blow device or inner tube for sampling of boiler water in a drum type boiler and of its structure is shown in Fig. 5. The length of steel pipe for 20 A piping of Fig. 5, in the case where a gas-water separator exists in the drum, shall be made equal to the length of this separator.

Fig. 5. Example of Position of Sampling Tube in Steam Drum and Structure

Unit: mm



- (e) **Steam Taking Nozzle** The steam sampling nozzle shall be of such structure that it has steam in-taking hole, is fit in steam tube, and is capable of taking by introducing steam flown from steam in-taking hole of nozzle into sample conduit.

The material of steam-taking nozzle shall have heat resistance not less than that of JIS G 3458 (STPA 24) and its design reference, be in accordance with Table 3. Further, the representative example of structure is shown in Fig. 6.

The nozzle attaching position shall be, as a rule, the position where steam has flown in straight tube part having no bore variation of not less than 10 times the bore of steam tube and further where no bending tube part, bore variation, and other conditions to disturb the steam flow at adjacent distance directly behind its position.

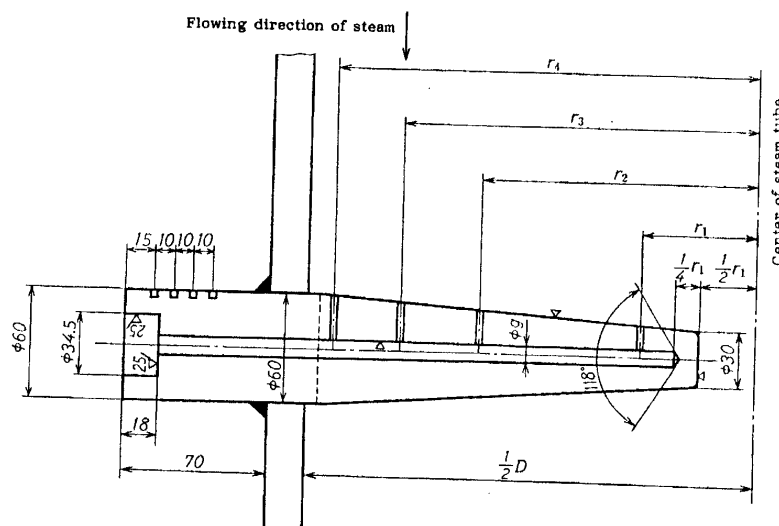
Table 3. Design Reference of Nozzle

Main item	Bore of steam tube D (mm)	Steam taking-in hole of nozzle					Nozzle bore g (mm)	
		Number of pieces n	Arranging position					Hole diameter d (mm)
			r_1	r_2	r_3	r_4		
Design reference	150 max.	2	$0.177D$	$0.427D$	—	—	$d \times \sqrt{1.5n}$ min.	
	Exceeding 150	3	$0.144D$	$0.337D$	$0.454D$	—		

Note (16) s in the calculation formula for nozzle hole diameter is the steam sampling quantity (kg/min), and E is the steam flow rate (t/h) in steam tube.

Fig. 6. An Example of Structure of Nozzle

Unit: mm



- (f) Washing at New Setting of Apparatus After attaching sample conduit of each system of new setting apparatus, while flowing cooling water to the cooler, take out sample from each system, cool to flow out from the cooler outlet and wash.

Carry out this washing at first only the sample conduit, then the sample conduit part and cooler, and at last the whole system for not less than 24 h in total.

- (4) Sampling Operation The sampling operation shall be as follows:

- (a) Washing of Apparatus While flowing the cooling water to cooler, flow the sample to the whole system, and thereafter adjust the flow rate of cooling water so as to make taking of sample at the designed taking flow rate (for example about 1 l/min) possible. In this state wash the system for not less than 1 h, and successively carry out the following operation.

In the case where the sample conduit is long and there is a fear for deposited substances to adhere inside of conduit, carry out flashing of the system equivalent to that at the new setting of apparatus.

When the boiler is in the working condition, flow continuously the sample at a rate of about 1 l/min.

- (b) Taking Operation After washing 5 times the sample vessel and its stopper by the sample flowing out from sampling apparatus, make the end of sample outlet tube (attach a soft vinyl chloride tube) to contact the bottom face of sample vessel, and after the sample of not less than 5 times the volume of sample vessel has overflowed, take the water in the sample vessel as the sample.

Tightly stopper⁽¹⁷⁾ the sample vessel as it is filled with water.

The taking amount of sample shall be in accordance with (5). Further, in the case where it is impossible to conduct the test directly after taking, depending upon the purpose of test, conduct the storage treatment according to 3.2.

In the case of sample to test fats and oils, let the end of sample outlet contact the bottom face of sample vessel (do not wash with the sample to be taken), and during space (about 10 % of volume) remains on the sample vessel, take out the sample vessel to stopper tightly.

Note ⁽¹⁷⁾ Sampling in the case of testing dissolved oxygen, sulfurous acid ion, and hydrazine ion shall be in accordance with the respective test items.

- (5) Taking Quantity of Sample Select the taking quantity of sample from the range of 1 to 2 l according to the number of test items and test purpose.

In the case of taking sample for testing of fats and oils, as required, take up to 2 l.

- (6) Recording Items at Sampling At the time of sampling, the following items shall be recorded on the recording paper for sample vessel and test results:

- (a) Name of sample
- (b) Sampling position
- (c) Date and time
- (d) Appearance of sample
- (e) Names of sampling person and person in duty of test

- (f) Operating record relating to water quality (for example, starting time, supplementary water quantity load in plant, etc.)
- (g) Injection conditions of medical product, operating state, of blow and supplementary water treatment device, etc. (for example, during injection of medical product, before boiler blow, the operating state of aggregation, precipitation device and the like)
- (h) Other items to be informative reference

3.2 Storing Treatment of Sample In the case where storage and transportation of sample are required, these shall be divided according to the purpose of test as follows and, the storing treatment shall be carried out.

- (1) The sample for testing heavy metal elements (copper, zinc, nickel, iron, etc.), metal elements such as aluminium and the like shall be added with nitric acid or hydrochloric acid to make pH about 1. For the sample for testing iron (II), hydrochloric acid shall be used.
The sample for testing metal element in dissolved state, immediately after sampling, shall be filtered by using filter paper of class 5 C or filter material⁽¹⁸⁾ of aperture size of 1 μm (or less), and thereafter, added with hydrochloric acid or nitric acid to make pH about 1.
- (2) The sample for testing ammonium ion and hydrazine (hydrazinium ion) shall be added with hydrochloric acid to make pH about 2.
- (3) The sample for testing of fats and oils (hexane extract or carbon tetrachloride extract) shall be added with hydrochloric acid to make pH about 4.
- (4) The sample for testing of oxygen consumption quantity (COD_{Mn}) by potassium permanganate at 100°C shall be stored at a dark place at 0 to 10°C and, tested within two days.

Note (¹⁸) The filter paper of class 6 or filter material of 0.45 to 1 μm in aperture size may be used.

3.3 Handling of Sample As to the test of metal element, determine the total quantity in the sample. When taking separately the sample, in the case where suspensoid exists in the sample, mix by shaking sufficiently to make uniform, thereafter take the sample separately and after conducting pretreatment specified in 4. or in respective testing methods, carry out the test.

In the case where only the metal element in dissolved state is tested, immediately after sampling, filter by filter paper of class 5 C or filter material⁽¹⁸⁾ of 1 μm or less in aperture size, discard the primary filtrate of about 50 ml, and take the filtrate thereafter as the sample. As to the anion ion test, as a rule, use the filtered sample.

Further, in the case of obtaining the total quantity, follow the specifications of respective test methods.

3.4 Testing Time Conduct the test immediately after sampling. As to the test of sample capable of preventing variation of quality of component in sample, after conducting the operation of 3.2, conduct the test as soon as possible. As to the dissolved oxygen (in Winkler's method, until fixing), and sulfurous acid ion, conduct the test immediately after sampling at the sampling site.

As to pH and electric conductivity, conduct the test immediately after sampling. However, in the case of sample of weak buffer action, measure by using a liquid flow type cell with preventing contact with air as far as possible.

4. Pretreatment of Sample

Metal elements (excluding alkali metal) in feed water and boiler water exist in complicated state such as oxide, hydroxide, phosphate, complex, etc. The pretreatment operation in test of metal element is to make metal element the ion state and therefore is almost common independent upon the kind of metal element, and hereinafter specified collectively.

The pretreatments in tests of metal elements have the purpose to decompose mainly coexisting organic substances and metallic complexes. As to the treating method, mainly the method to evaporate by heating with adding each kind acid to sample is used. The suitable method shall be selected according to the state of sample and kind of test.

4.1 Decomposition by Hydrochloric Acid or Nitric Acid The decomposition by hydrochloric acid or nitric acid applies to the sample containing hydroxides, oxides, phosphates, etc. as suspensoids.

(1) Reagents

- (a) Water Distilled water
- (b) Hydrochloric Acid
- (c) Nitric Acid

(2) Operation

- (a) After mixing the sample⁽¹⁹⁾ by shaking, immediately take its 200 ml in a beaker⁽²⁰⁾ of 300 ml, and add hydrochloric acid 10 ml or nitric acid 10 ml.
- (b) Gently heat on a hot plate, and concentrate until the liquid quantity becomes about 20 ml⁽²¹⁾.
- (c) In the case where nondissoluble substance⁽²²⁾ has remained, after filtering by filter paper of class 5 B⁽²³⁾, wash thoroughly by warm water.
- (d) After cooling, put the filtrate and washings together with water into a transfer flask of a suitable capacity, and add water up to the marked line.

- (e) As for blank test take the same quantity of hydrochloric acid or nitric acid as that added to the sample, boil for several min, after leaving cool put it together with water into a transfer flask of the same quantity as the one used in (d), and add water up to the marked line.

- Notes (¹⁹) In the case where metal element in the dissolved state is tested, after sampling, use the sample filtered according to 3.3.
- (²⁰) As to the glass devices to be used for this test, after immersing into hydrochloric acid (1 + 10) for one night, wash sufficiently with water, and then use it.
- (²¹) In the case where precipitate of metal oxide is confirmed, further add hydrochloric acid or nitric acid of 5 ml, and successively heat to dissolve the precipitate.
- (²²) Because if a large amount of silica coexists it may deposit by becoming insoluble matter, filter to separate.
- (²³) Before using, wash thoroughly with warm hydrochloric acid (1 + 10).

4.2 Decomposition by Nitric Acid and Perchloric Acid The decomposition by nitric acid and perchloric acid applies to the sample containing organic substance difficult to be oxidized.

(1) Reagents

- (a) Water Distilled water
- (b) Nitric Acid
- (c) Perchloric Acid (60 %)

(2) Operation

- (a) After mixing the sample(¹⁹) by shaking, immediately take its 200 ml into a beaker(²⁰) of 300 ml, and add 5 to 10 ml of nitric acid.
- (b) Heat on a hot plate gently, when the liquid quantity has become about 10 ml, again add nitric acid 5 ml and then add, little by little, perchloric acid (60 %)(²⁴) 10 ml.
- (c) Continue heating on the hot plate, when white fume of perchloric acid begins to generate, cover the beaker with a watch glass, and keep the state where the perchloric acid flows down along the vessel wall to decompose.
- (d) When organic substance has remained without decomposing, further add nitric acid 5 ml to repeat the operation of (c), and decompose the organic substance.

- (e) After cooling, add water to dilute to about 50 ml in liquid quantity, in the case where insoluble substance⁽²²⁾ has remained, filter by using filter paper class 5 B⁽²³⁾, wash with warm water, put the filtrate and washings into a transfer flask of suitable capacity, and add water up to the marked line.
- (f) As for blank test, take the same quantities of nitric acid and perchloric acid (60 %) as those added to the sample, after cooling, put together with water into the transfer flask of the same capacity as that used in (e) and add water up to the marked line.

Note (²⁴) In the case of adding perchloric acid, let nitric acid coexist without fail.

4.3 Decomposition by Nitric Acid and Sulfuric Acid The decomposition by nitric acid and sulfuric acid is applicable⁽²⁵⁾ to many kind samples.

(1) Reagents

- (a) Water Distilled water
- (b) Nitric Acid
- (c) Sulfuric Acid (1 + 1)

(2) Operation

- (a) After mixing sample⁽¹⁹⁾ by shaking, immediately take its 200 ml into a beaker⁽²⁰⁾ of 300 ml, add nitric acid 5 to 10 ml and heat on a hot plate.
- (b) When the liquid quantity has become about 10 ml, again add nitric acid 5 ml and sulfuric acid (1 + 1) 10 ml, and heat until the white fume of sulfuric acid is generated and organic substance is decomposed.
- (c) When the decomposition of organic substance is difficult, further add nitric acid 10 ml, and repeat the operation of (b) to decompose the organic substance.
- (d) After cooling, dilute the liquid quantity to about 50 ml by water. In the case where insoluble substance⁽²⁶⁾ has remained, filter by using filter paper of class 5 B⁽²³⁾, wash with warm water, put the filtrate and washings into a transfer flask of a suitable capacity, and add water up to the marked line.
- (e) As for blank test, take nitric acid and sulfuric acid (1 + 1) of the same quantity as those added to the sample, and heat on a hot plate to generate the white fume of sulfuric acid sufficiently. After cooling, put it together with water into a transfer flask of the same capacity as that used in (d), add water up to the marked line.

Notes (²⁵) In the case where atomic absorption spectrophotometry to spray the aqueous solution as it is, is applied, it is not preferable.

- (²⁶) In the case where silica exists in a large quantity or lead is contained, precipitate may be generated. In the case where lead is contained, carry out the operation of 4.2 or as follows:

Carry out the operation of (b), evaporate the solution almost to dryness, add water about 30 ml and hydrochloric acid 15 ml, and heat to dissolve. In the case where insoluble substance exists, after filtering by filter paper of class 5 B, wash with warm hydrochloric acid (1 + 10). After cooling, put the filtrate and washings into a transfer flask of a suitable capacity, and add water up to the marked line.

4.4 Pretreat in the Case of Applying Atomic Absorption Spectrophotometry
Carry out the pretreatment(²⁷) by using the most suitable method out of the methods indicated in 4.1 to 4.3 by taking into consideration sufficiently the quantity of suspensoids and organic substances contained in the sample, their existing state, and system of atomic absorption spectrophotometry to be applied, etc.

In the case where the atomic absorption spectrophotometry is carried out by spraying the prepared sample as it is, generally make the sample hydrochloric acidic or nitric acidic and make the acid concentration the suitable concentration between 0.1 to 1 N.

- Note (²⁷) In the case where prior to atomic absorption spectrophotometry, solvent-extraction method is applied, the pretreatment shall be as specified in 4.1 to 4.3, and the organic substances having possibility of interfering and other interfering substances shall be decomposed sufficiently.

In the case of carrying out the atomic absorption spectrophotometry according to the method to spray the sample as it is, the pretreatment may be carried out as follows:

In the case of sample having extremely small organic substance and suspensoids, add at a rate of hydrochloric acid 5 ml or nitric acid 5 ml per 100 ml of sample, and heat to boil for about 10 min. After cooling, add water to make a definite quantity.

As for general pretreatment method of sample containing organic substance and suspensoids, 4.2 is applied. In this case, let the white fume generate sufficiently to remove the most part of perchloric acid.

5. Appearance

The appearance shall be in accordance with 7. Appearance of JIS K 0101.

6. Turbidity

6.1 Visual-Sense Turbidity The test for visual sense turbidity shall be in accordance with 9.1 Visual Sense Turbidity of JIS K 0101.

6.2 Transmitted-Light Turbidity The test for transmitted-light turbidity shall be in accordance with 9.2 Transmitted Light Turbidity of JIS K 0101.

- 0.04: oxygen equivalent quantity of N/200 sodium thiosulfate solution 1 ml (mg)
0.0104: correction value of dissolved oxygen in added reagent

Note (33) Use the factor of N/20 sodium thiosulfate solution.

- (b) In the case where the operation of (4.11) is carried out, calculate according to the formula (2):

$$O = \left[\frac{(a \times f - a_1 \times f_1)}{V_a} - \frac{(b \times f - b_1 \times f_1)}{V_b} \right] \times 1000 \times 0.04 - 0.0104 \dots \dots \dots (2)$$

- where
- O : dissolved oxygen (mg O/l)
 - a : N/200 sodium thiosulfate solution required for titration (ml)
 - a₁ : N/200 potassium iodate solution required for titration (ml)
 - b : N/200 sodium thiosulfate solution required for titration of blank test (ml)
 - b₁ : potassium iodate solution required for blank test (ml)
 - f : factor of N/200 sodium thiosulfate solution(33)
 - f₁ : factor of N/200 potassium iodate solution
 - V_a : titrated sample (ml) (quantity of capacity of sampler subtracted by the discarded quantity)
 - V_b : titrated sample for blank test (ml) (quantity of capacity of sampler subtracted by the discarded quantity)
 - 0.04: oxygen equivalent quantity of 1 ml of N/200 sodium thiosulfate solution (mg)
 - 0.0104: correction value of dissolved oxygen in added reagent

Remarks 1. For both samplers, parts A shall be used only for addition of iodine alkaline potassium iodide solution and parts B, only for manganese sulfate solution and sulfuric acid (3 + 1).

At the time of addition of reagent, if the liquid surface is not maintained correctly as shown by the upper and lower base lines, error is generated in determination value. If sulfuric acid (3 + 1) is added with the precipitate settled as it is, many precipitates are contained in the solution to be exhausted and cause the negative error.

2. In the operation when the electric current titrimetric method of (4.11) is used, the operation may be carried out as follows:

6.3 Integral Spherical Bulb Turbidity The test for integral spherical bulb turbidity shall be in accordance with 9.4 Integral Spherical Bulb Turbidity of JIS K 0101.

Remark: The test for scattered-light turbidity shall be in accordance with 9.3 Scattered-Light Turbidity of JIS K 0101.

7. pH

The test for pH shall be in accordance with 11. pH of JIS K 0101.

8. Electric Conductivity

The tests for electric conductivity shall be divided into two tests of general test and the test for electric conductivity after passing through the strong acidic cation-exchange resin (hydrogen ion type) layer.

8.1 General Test The test for general electric conductivity shall be in accordance with 12. Electric Conductivity of JIS K 0101.

8.2 Electric Conductivity after Passing through Strong Acid Cation-Exchange Resin Layer (Hydrogen Ion Type) The electric conductivity after passing through the column filled with strong acid cation-exchange resin transformed to hydrogen ion type shall be measured in order to know existence of generation steam, steam condensate and salts other than volatile substance in feed water of boiler in a boiler conducted with volatile-substance treatment.

The volatile substances, for example hydrazine (hydrazinium ion), morpholine (tetrahydroxy-1, 4-oxazine), ammonium ion, etc. are adsorbed by strong acid cation-exchange resin transformed to hydrogen ion type and the electric conductivity by these is extinguished. However, cations other than these amines conduct exchange reaction with hydrogen ions of hydrogen ion type strong acid cation-exchange resin and generate hydrogen ions of the equivalent quantity to the cations. The electric conductivity by this hydrogen ion shall be measured and the concentration of salts existing in micro quantity, estimated. The test shall be as follows:

(1) Reagents

(a) Hydrochloric Acid (1 + 11)

(b) Strong Acid Cation-Exchange Resin

The strong acid cation-exchange resin of 355 to 1180 μm in particle size and of containing quantity of divinyl benzene of about 8 %. It is transformed to hydrogen ion type by operating as follows:

Immerse a suitable quantity of strong acid cation-exchange resin in water (about three times the quantity of resin), after leaving as it is for 12 h or more, transfer into a suitable column with water, and use at a rate of hydrochloric acid (1 + 11) 25 l per 1 l of strong acid cation-exchange resin to flow at a flow rate of about 10 l/(l-resin·h). Next, flow water at a rate of about 20 l/(l-resin·h) to wash, and wash until blue colour appears relative to mixed solution⁽²⁸⁾ of Methyl Red and Bromocresol Green. Fill this in a column shown in Fig. 7 so as air bubbles not to enter with water.

Note ⁽²⁸⁾ Dissolve Methyl Red 0.02 g and Bromocresol Green 0.10 g in ethanol (95) 100 ml.

- (2) Ion Exchange Resin Column An example is shown in Fig. 7.
- (3) Operation Flow continuously the sample at a rate of 12 to 15 // (l-resin.h) into an ion-exchange resin column filled with strong acid cation-exchange resin (hydrogen ion type), and as to the flowing-out water, measure the electric conductivity.

9. Acid Consumption Quantity (Alkalinity)

9.1 Acid Consumption Quantity (pH 4.8) (M-Alkalinity) The acid consumption quantity (pH 4.8) (M-alkalinity) shall be in accordance with 13.1 Acid Consumption Quantity (pH 4.8) of JIS K 0101.

9.2 Acid Consumption Quantity (pH 8.3) (P-Alkalinity) The acid consumption quantity (pH 8.3) (P-alkalinity) shall be in accordance with 13.2 Acid Consumption Quantity (pH 8.3) of JIS K 0101.

10. Alkali Consumption Quantity (Acidity)

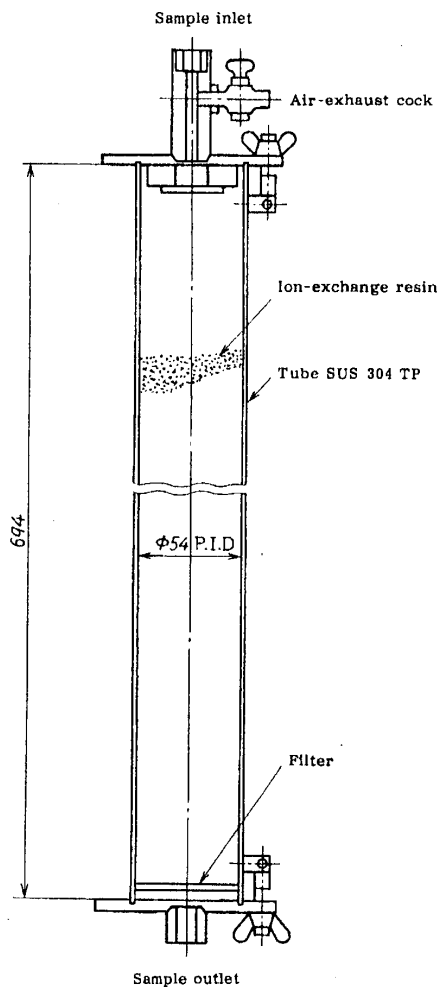
10.1 Alkali Consumption Quantity (pH 8.3) (Total Acidity) The alkali consumption quantity (pH 8.3) (total acidity) shall be in accordance with 14.1 Alkali Consumption Quantity (pH 8.3) of JIS K 0101.

10.2 Alkali Consumption Quantity (pH 4.8) (Mineral Acid Acidity) The alkali consumption quantity (pH 4.8) (mineral acid acidity) shall be in accordance with 14.2 Alkali Consumption Quantity (pH 4.8) of JIS K 0101.

Remark: In the alkali consumption quantity (pH 4.8) (mineral acid acidity), other than strong acids such as hydrochloric acid, sulfuric acid, nitric acid, etc. and strong organic acid, the alkali consumption quantities by salts such as iron, aluminium, etc. are contained. In the case where content quantities of strong acids such as hydrochloric acid, sulfuric acid, nitric acid, etc., strong organic acids, etc. are desired to know, the test shall be conducted according to 14.3 Alkali Consumption Quantity (Free Acid) of JIS K 0101.

Fig. 7. An Example of Ion-Exchange Resin Column

Unit: mm



- Description
1. Ordinary using pressure (gauge pressure) 0.1 MPa {1 kgf/cm²} max.
 2. Capacity 1.6 l
 3. Filled quantity of ion-exchange resin 1.5 l
 4. Flow rate 12 to 15 l (l-resin·h)
 5. Material SUS 304

11. Hardness

11.1 Total Hardness The total hardness shall be in accordance with 15.1 Total Hardness of JIS K 0101.

11.2 Calcium Hardness The calcium hardness shall be in accordance with 15.2 Calcium Hardness of JIS K 0101.

11.3 Magnesium Hardness The magnesium hardness shall be in accordance with 15.3 Magnesium Hardness of JIS K 0101.

12. Suspensoids and Evaporation Residuals

12.1 Suspensoids Suspensoids shall be in accordance with 16.1 Suspensoids of JIS K 0101.

12.2 Total Evaporation Residuals The total evaporation residuals shall be in accordance with 16.2 Total Evaporation Residuals of JIS K 0101.

12.3 Soluble Evaporation Residuals The soluble evaporation residuals shall be in accordance with 16.3 Soluble Evaporation Residuals of JIS K 0101.

12.4 Ignition Residuals The ignition residuals shall be in accordance with 16.4 Ignition Residuals of JIS K 0101.

12.5 Ignition Reducing Quantity The ignition reducing quantity shall be in accordance with 16.5 Ignition Reducing Quantity of JIS K 0101.

13. Oxygen Consumption Quantity by Potassium Permanganate at 100°C (COD_{Mn})

The oxygen consumption quantity (COD_{Mn}) by potassium permanganate at 100°C shall be in accordance with 17. Oxygen Consumption Quantity (COD_{Mn}) by Potassium Permanganate at 100°C of JIS K 0101.

14. Organic Carbon (TOC)

The organic carbon (TOC) shall be in accordance with 20. Organic Carbon (TOC) of JIS K 0101.

15. Hexane Extract

The hexane extract shall be in accordance with 26. Hexane Extract of JIS K 0101.

16. Carbon Tetrachloride Extract

The carbon tetrachloride extract shall be in accordance with 27. Carbon Tetrachloride Extract of JIS K 0101.

17. Dissolved Oxygen (O)

As for the determination of dissolved oxygen, Winkler's method or Indigo Carmine colorimetric method shall be used. This test shall be conducted immediately after sampling.