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**Testing methods for industrial water and
industrial wastewater — Part 1: Test
methods for general physics and
chemistries**

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Foreword

This Japanese Industrial Standard has been established by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Act.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, published patent application or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, published patent application or utility model rights.

JIS K 0102 series consists of the following 5 parts under the general title *Testing methods for industrial water and industrial wastewater* — :

Part 1 : Test methods for general physics and chemistries

Part 2 : Anion and cation analysis (to be published)

Part 3 : Metal analysis (to be published)

Part 4 : Organic analysis (to be published)

Part 5 : Methods for acute toxicity test by fish, bacterial test, and biological test (to be published)

Testing methods for industrial water and industrial wastewater — Part 1 : Test methods for general physics and chemistries

1 Scope

This Japanese Industrial Standard specifies the general physical and chemical analysis methods for industrial water and for wastewater discharged from factories (including workplaces) (hereafter referred to as industrial wastewater). Unless otherwise specified in the specific test method subclause, tests given in this Standard are applicable to both industrial water and industrial wastewater.

For test methods in this Standard for which a corresponding International Standard exists, the number of the International Standard and the symbol of degree of its correspondence with **JIS** are shown in relevant test clauses.

Where modifications have been made to the technical contents of the corresponding International Standard, the modifications and their explanations are given in Annex F.

2 Normative references

Part or all of the provisions of the standards listed in Annex E, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated in the said annex shall be applied.

3 Terms and definitions

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

4 General requirements

4.1 Chemical analysis

General requirements of chemical analysis are provided in **JIS K 0050**.

4.2 Absorptiometry

General requirements of absorptiometry are provided in **JIS K 0115**.

4.3 Repeatability

The repeatability of each test method is indicated by the approximate value of the coefficient of variation (%) obtained by performing repetitive tests using the standard solution to be used in the dynamic range of that test method.

4.4 Water used for tests

The water to be used for the tests described in this Standard shall be of A1 to A4 specified in **JIS K 0557**, unless otherwise specified in the particular test clause. Dis-

solved oxygen-free water and carbon dioxide-free water shall be as follows.

- a) **Dissolved oxygen-free water** Prepare using one or a combination of the following methods 1) to 5) immediately before use. For storage, connect the flask to a gas washing bottle containing alkaline pyrogallol solution as shown in Figure 1 to shield it from oxygen in the air. Prepare alkaline pyrogallol solution by mixing, immediately before use, a solution obtained by dissolving 6 g of pyrogallol (1,2,3-benzenetriol) specified in **JIS K 8780** in 50 ml of water and putting in a coloured bottle, and a solution obtained by dissolving 30 g of potassium hydroxide specified in **JIS K 8574** in 50 ml of water. One millilitre of this solution absorbs about 12 ml (about 17 mg) of oxygen (see Annex E of **JIS K 0050**).

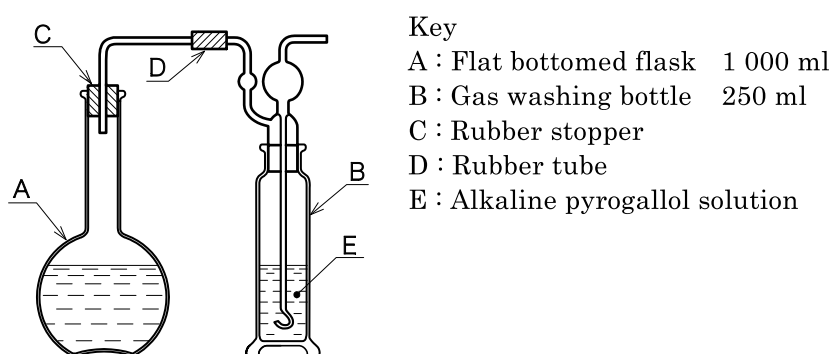


Figure 1 Example of cooling and storing dissolved oxygen-free water

- 1) Place water of A2 or A3 specified in **JIS K 0557** in a flask and boil for about 5 min to expel dissolved oxygen. Connect the flask to a gas washing bottle containing alkaline pyrogallol solution as shown in Figure 1 to shield from oxygen in the air and allow to cool (see NOTE 3 to Clause 4 of **JIS K 0557**).
 - 2) Place water of A2 or A3 specified in **JIS K 0557** in a flask and aerate with nitrogen of Grade 2 specified in **JIS K 1107** for about 15 min (see NOTE 3 to Clause 4 of **JIS K 0557**) to expel dissolved oxygen.
 - 3) Pass water of A2 or A3 specified in **JIS K 0557** through a gas separation pipe using an oxygen separation membrane to expel dissolved oxygen.
 - 4) Thoroughly deaerate water of A2 or A3 specified in **JIS K 0557** using an ultrasonic vibration apparatus to expel dissolved oxygen.
 - 5) Take water of A2 or A3 specified in **JIS K 0557**, which has just been refined, in an Erlenmeyer flask aerated with nitrogen of Grade 2 specified in **JIS K 1107** with care not to foam.
- b) **Carbon dioxide-free water** Prepare using one or a combination of the following methods 1) to 4) immediately before use. For storage, connect the flask to a gas washing bottle containing potassium hydroxide solution (250 g/L) (prepared using potassium hydroxide specified in **JIS K 8574**) or soda lime for carbon dioxide absorption of No. 1 specified in **JIS K 8603** similarly to the apparatus shown in Fig-

ure 1 to shield it from carbon dioxide in the air (see Annex E of **JIS K 0050**).

- 1) Place water of A2 or A3 specified in **JIS K 0557** in a flask, and boil for about 5 min to expel dissolved gas and carbon dioxide. Connect the flask to a gas washing bottle containing potassium hydroxide solution (250 g/L) or soda lime for carbon dioxide absorption of No. 1 specified in **JIS K 8603** similarly to the apparatus shown in Figure 1 to shield it from carbon dioxide in the air and allow to cool (see NOTE 4 to Clause 4 of **JIS K 0557**).
- 2) Perform the procedure in a) 2) to expel carbon dioxide.
- 3) Pass water of A2 or A3 specified in **JIS K 0557** through a gas separation pipe using an carbon dioxide separation membrane to expel carbon dioxide.
- 4) Perform the procedure in a) 4) to expel carbon dioxide.

4.5 Reagents used for tests

- a) Reagents conforming to relevant Japanese Industrial Standards (**JISs**) and not interfering with the analysis shall be used. In the absence of relevant **JIS** specifications, reagents not interfering with the analysis shall be used.

For standardization of titrants, reference materials for volumetric analysis specified in **JIS K 8005** shall be used if applicable.

- b) Standard solutions shall be prepared by the methods specified in the particular test clause, or shall be solutions with a guaranteed traceability to the National Metrological Standard used by diluting to a specified concentration if necessary. The types and concentrations of the chemical compounds used or acids added in the preparation of standard solutions shall not be such that they create interferences for the analysis.

NOTE Standard solutions with guaranteed traceability are most typically those bearing a JCSS (Japan Calibration Service System) mark.

- c) The concentration of a reagent solution, unless otherwise specified, is expressed by g/L or mg/L in the case of mass concentration, and by mol/L or mmol/L in the case of molar concentration. The mass of a compound is indicated in parentheses after the name of the compound, using the value of the mass as an anhydride. The concentration of a standard solution is expressed by the mass in 1 ml or 1 L (mg/ml, µg/ml, ng/ml, g/L, mg/L or µg/L).
- d) The concentration of a reagent solution indicated in parentheses following the name of the solution, except for when it is a standard solution, is an approximate value. For example, “sodium hydroxide solution (0.1 mol/L)” indicates that the sodium hydroxide solution is approximately 0.1 mol/L in concentration. Further, the concentration of a liquid reagent is indicated by the ratio to the volume of water; for example, “reagent (a + b)” indicates that it is a mixture of a ml of the reagent and b ml of water. The concentration indicated before the name of the solution is an exact value which is generally a whole number. In this case the factor of the solution is separately determined.

- e) For preparation of reagents, the water specified in **4.4** shall be used.
- f) For diluting a standard solution to a lower concentration, unless otherwise specified, a volumetric pipette of 10 ml or larger capacity shall be used.
- g) The designations of reagents in this Standard, wherever possible, are in accordance with the nomenclature of compounds specified by the Chemical Society of Japan based on the inorganic chemicals nomenclature and organic chemicals nomenclature of the International Union of Pure and Applied Chemistry (IUPAC), and the designations of reagents specified in **JIS**.
- h) Care shall be taken against room contamination, inhalation and adherence to human body, of reagents and wastewater, etc. The handling of reagents and wastewater, etc. shall follow the related laws and regulations.

4.6 Implements used for tests and methods for washing them

4.6.1 Implements

Glassware, porcelain crucible, porcelain basin, platinum crucible, platinum dish and filter paper to be used in this Standard shall be as follows :

- a) Glassware, unless otherwise specified, shall be as given in **JIS R 3503** and **JIS R 3505**. Where a special implement(s) needs to be used, its illustration or description will be given in the particular test clause. For heating procedures, glassware made of borosilicate glass-1 specified in **JIS R 3503** shall be used.

The desiccant to be used for a desiccator, unless otherwise specified, shall be the silica gel desiccant for packaging of Type A Class 1 specified in **JIS Z 0701**.

- b) The porcelain crucible and porcelain basin shall be as given in **JIS R 1301** and **JIS R 1302**, respectively.
- c) The platinum crucible and platinum dish shall be as given in **JIS H 6201** and **JIS H 6202**, respectively.
- d) The filter paper shall be that for quantitative analysis as given in **JIS P 3801**. See the particular test clause for the specific type of filter paper to be used.

4.6.2 Washing of implements

All of the glassware, porcelain crucibles and porcelain basins used for the test methods specified in this Standard shall be washed with water of A2, and then with water of A3 before being used for the test, unless otherwise specified in the particular test clause.

4.7 Management of quality of test results

In order to manage the quality of test results, the accuracy (trueness and precision) of measured values should be periodically evaluated using the reference materials with guaranteed traceability for each test method or the working curve standard solutions that are prepared using such materials. When there are no reference materials with guaranteed traceability, the reference materials or working curve standard solutions that are used for each test method shall be used. Furthermore, this checking of accu-

racy should be also done by a periodical addition recovery test etc. using these reference materials or working curve standard solutions.

4.8 Sample

4.8.1 Sampling, sample container, water sampler and sampling procedure

For the purpose of this Standard, a sample is the water that is collected for testing. Sampling, sample container, water sampler, and sampling procedure shall be in accordance with **JIS K 0094**.

4.8.2 Handling of sample

Unless otherwise specified, carry out the determination on the whole amount contained in the sample. When the sample water contains suspended matter, therefore, agitate it thoroughly until homogeneous and then collect a suitable volume of it for testing. Where the target component is in the dissolved state, filter the collected sample through a filter paper of Class 5 C immediately after collection, discard the initial filtrate of about 50 ml, and use the subsequent filtrate as a sample. Alternatively, a filter paper of Class 6 or a filtering material with a pore diameter of 1 μm or less may be used. If other filtration method is specified in the particular test clause, that method shall be applied.

4.8.3 Storage of sample

Unless otherwise specified, test the sample immediately after collection. Where this is not possible, store the sample in accordance with Clause 7 of **JIS K 0094**, and test it as soon as possible. When storing in a cold place at a temperature close to 0 °C, take care not to freeze the sample. If other storage method is specified in the particular test clause, that method shall be applied.

5 Flow rate

The measurement of flow rate used for testing of industrial wastewater shall be in accordance with Clause 8 of **JIS K 0094**.

6 Temperature

6.1 General

The atmospheric temperature and the water temperature shall be both measured at the time of sampling.

6.2 Temperature

6.2.1 Implements

- a) **Thermometer**, as specified in **JIS B 7414**, **JIS C 1602**, **JIS C 1604**, **JIS C 1605** or **JIS C 1611**, capable of measuring $-30\text{ }^{\circ}\text{C}$ to $+50\text{ }^{\circ}\text{C}$ with a permissible error of within $\pm 1\text{ }^{\circ}\text{C}$. The thermometer specified in **JIS B 7414** shall be that for total immersion and shall use an organic liquid as the temperature sensitive liquid. A maximum-minimum thermometer shall be used for measuring the maximum and minimum temperatures during the measuring period.

6.2.2 Procedure

Find a well-ventilated place without direct sunshine or any strong ambient thermal radiation in the sampling site, and hold the thermometer at a position 1.2 m to 1.5 m above the ground and read the scale when the temperature sensitive liquid has stopped moving.

6.3 Water temperature

6.3.1 Implements

- a) **Thermometer**, as specified in **JIS B 7414**, **JIS C 1602**, **JIS C 1604**, **JIS C 1605** or **JIS C 1611**, capable of measuring $-30\text{ }^{\circ}\text{C}$ to $+100\text{ }^{\circ}\text{C}$ with a permissible error of within $\pm 1\text{ }^{\circ}\text{C}$. The thermometer specified in **JIS B 7414** shall be that for total immersion and shall use an organic liquid as the temperature sensitive liquid. A bar thermometer, if used, may be a glass rod thermometer or a Pettenkofer water temperature gauge.

6.3.2 Procedure

The measurement procedure for each type of thermometer shall be as follows.

- a) **Glass rod thermometer** Immerse the thermometer directly in the water at the site or in the sample immediately after collection, down to an approximate position of the graduation line around which the temperature sensitive liquid stops. Read the scale when the temperature sensitive liquid has stopped. Take a sufficient amount of sample to avoid the influence by the container and atmospheric temperature.
- b) **Pettenkofer water temperature gauge** (see Figure 2) Throw the water temperature gauge into the water at the site to draw sample, and then discard the sample. After performing this three times, throw in the water temperature gauge again to fill the metallic cylinder with the sample, and read the scale when the temperature sensitive liquid has stopped.
- c) **Electric thermometer** Hold the thermometer with its detector immersed in the water to be measured, and read the scale when the needle of the indicator has stabilized.

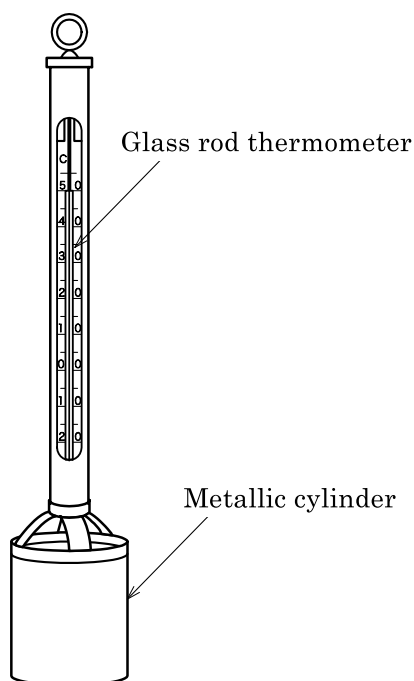


Figure 2 Example of Pettenkofer water temperature gauge

7 Appearance

7.1 General

The appearance of a sample shall be observed immediately after sampling.

7.2 Implements

a) **Beaker**, of 300 ml to 500 ml, and colourless.

7.3 Procedure

Place the sample in a beaker immediately after collection, and observe the sample with the naked eye to check and make a record of the following.

- Type and degree of colour of the whole sample
- Type and degree of colour of the supernatant
- Type of colour and degree of quantity of floating matter, suspended matter, etc.
- Condition and degree of oils, tars, etc.
- Other particularities of the sample such as presence of bubbles and odours.

8 Transparency

8.1 General

The transparency is an index of clarity of the sample and is measured as follows. Place the sample in a transparency meter, and observe it from above. Note the depth of water when the double cross on the mark plate placed at the bottom of the transpar-

ency meter becomes clearly visible. Express the result in degrees, with one degree being equal to 10 mm.

Measuring range : 1 degree to 30 degrees

NOTE The International Standard corresponding to this test method and the symbol of degree of correspondence are as follows.

ISO 7027 : 1990 *Water quality — Determination of turbidity* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

8.2 Implements

- a) **Transparency meter**, as illustrated in Figure 3 and detailed in Figure 4, consisting of a glass cylinder with a lower opening graduated in 5 mm up to 50 mm height from the mark plate, and in 10 mm from 50 mm to 300 mm height, and used with the mark plate placed at its bottom. In place of the cylinder with a lower opening illustrated in Figure 4, other cylinder, for example, that having a length of 600 mm $\pm$ 10 mm, an inside diameter of 25 mm $\pm$ 1 mm and 10 mm graduation, may be used.

NOTE Some commercially available cylinders are made of acrylic resin and 1 m in length.

8.3 Procedure

Measurement using a transparency meter shall be as follows.

- a) Fill the transparency meter with the sample which has been shaken well, observe the bottom of the transparency meter from above while letting the sample flow out from the lower opening by gradually loosening the pinch cock of the rubber tube, and read the scale at the water level just when the double cross on the mark plate becomes clearly visible. The opening of the pinch cock should be performed quickly if the sample contains a large amount of suspended matter which may form sediment at the bottom of the transparency meter and cause error in measurement. It should be noted that using different light sources in this measurement, even if they are identical in their illuminance, may cause change in the transparency if they have different chromas. For this measurement, use daylight as the light source, and avoid direct sunlight.
- b) Repeat the procedure in a) two or three times, each time noting the scale reading at the water level. Calculate the average of these readings, and report it as the degree of transparency.

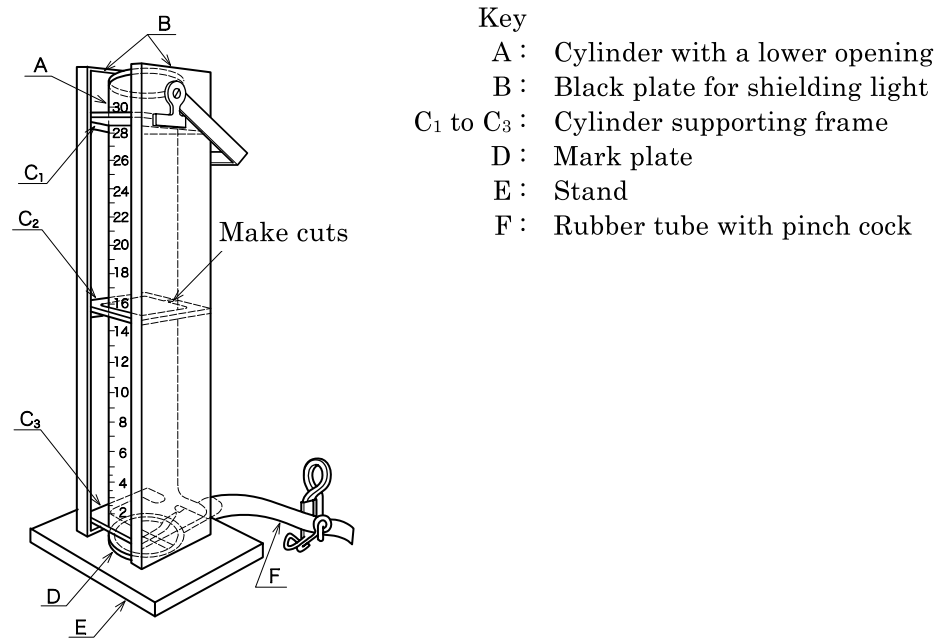


Figure 3 Example of transparency meter

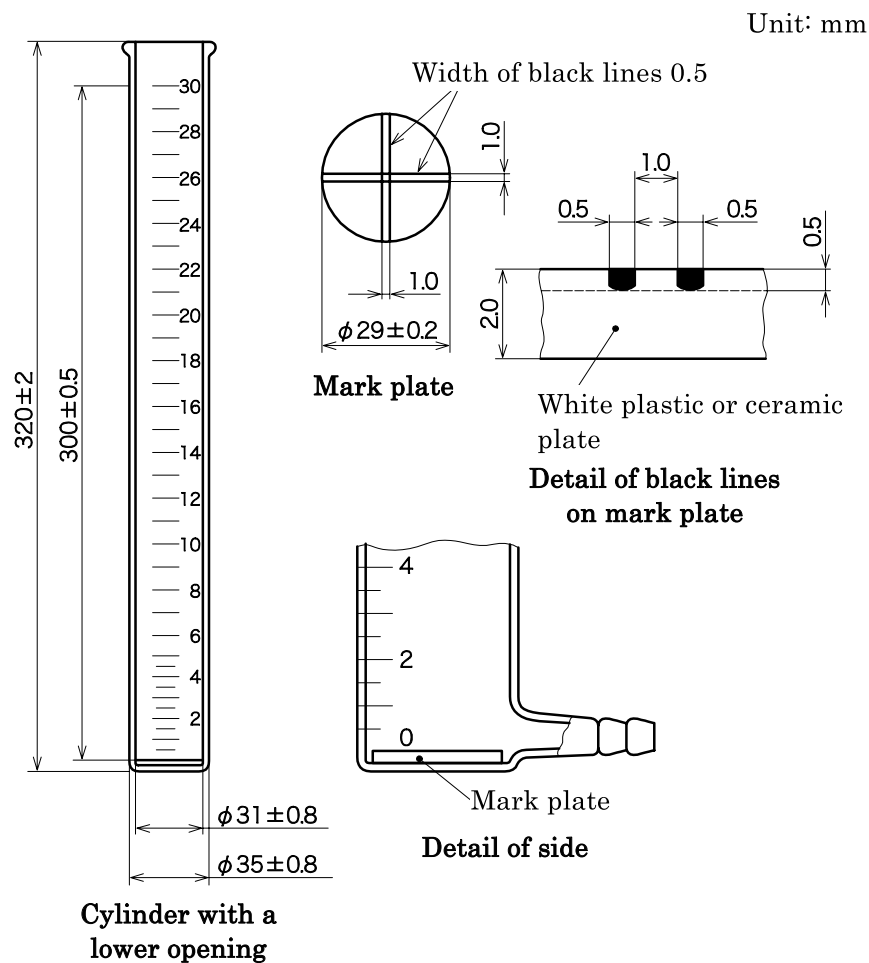


Figure 4 Details of transparency meter

9 Turbidity

9.1 General

The turbidity is a degree of cloudiness of water that is expressed in visual-sensation turbidity, transmitted-light turbidity, scattered-light turbidity and integrating-sphere turbidity which are measured according to respective methods given in 9.2 to 9.5. Turbidity measured in comparison with kaolin standard solutions is expressed in degrees (kaolin) and that measured in comparison with formazin standard solutions, in degrees (formazin). As turbidity easily changes, a sample should be tested immediately after collection.

9.2 Visual-sensation turbidity

9.2.1 Summary

The turbidity of a sample is compared against kaolin standard solutions with the naked eye.

Measuring range : 1 degree (kaolin) to 10 degrees (kaolin)

9.2.2 Reagents

- a) **Water** Filter water of A3 specified in **JIS K 0557** through a filtering material with a pore diameter of about 0.1 μm , discard the initial filtrate of about 200 ml, and use the ensuing filtrate.
- b) **Refined kaolin** Put about 10 g of china clay (kaolin) in a 500 ml beaker, add to this 300 ml of water and 0.2 g of sodium diphosphate decahydrate specified in **JIS K 8785**, and vigorously stir for about 3 min with a magnetic stirrer. Transfer this to a 1 000 ml measuring cylinder (with ground-in stopper), add water up to the 1 000 ml mark, apply the stopper and vigorously shake for about 1 min. After leaving for about 1 h in room temperature, discard the upper 250 ml of the solution using a siphon, and collect the next 500 ml.

Either centrifuge the collected solution at about 3 000 min^{-1} (adjusted according to the radius of the rotating part of the centrifugal separator) for about 20 min or filter it through a filtering material with a pore diameter of not more than 1 μm . Heat the centrifuged or filtered kaolin at 105 °C to 110 °C for about 3 h, allow to cool in the desiccator, and store in a wide-mouth bottle.

- c) **Kaolin standard solution [1 000 degrees (kaolin)]** Disperse 1.00 g of refined kaolin in an appropriate volume of water and transfer to a 1 000 ml volumetric flask. Add about 800 ml of water and about 10 ml of formaldehyde solution specified in **JIS K 8872**, and add water to the mark.
- d) **Kaolin standard solution [100 degrees (kaolin)]** Shake the kaolin standard solution [1 000 degrees (kaolin)] well, immediately transfer 100 ml of it to a 1 000 ml volumetric flask, and add water to the mark.

9.2.3 Implements

- a) **Dark box** For comparing turbidity with the naked eye, a dark box as illustrated in

Figure 5 should be used with the electric lamp brought close to its under window.

- b) **100 ml colorimetric tube** A flat-bottomed tube with ground-in stopper, made of colourless glass and bearing a 100 ml mark at a height of $270 \text{ mm} \pm 1.5 \text{ mm}$ from the bottom shall be used. The tubes aligned in height of the mark to an accuracy of $\pm 1.5 \text{ mm}$ should be used (Figure 6).

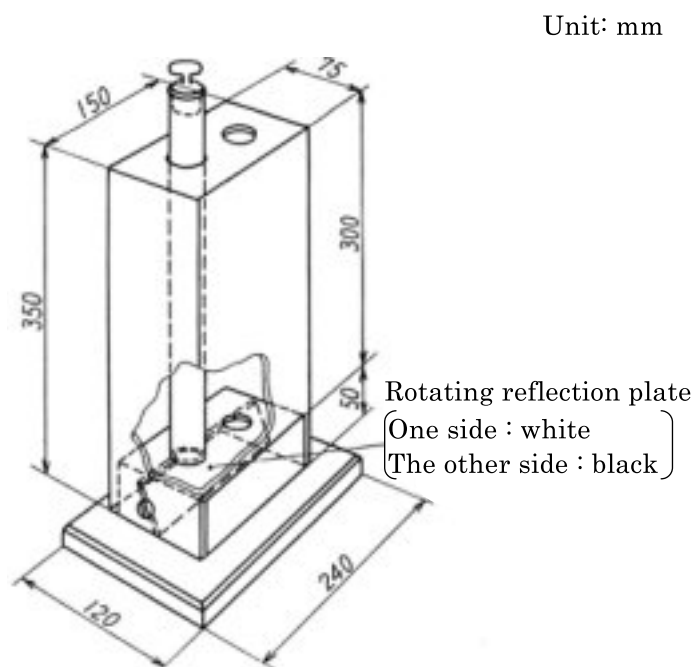


Figure 5 Example of dark box

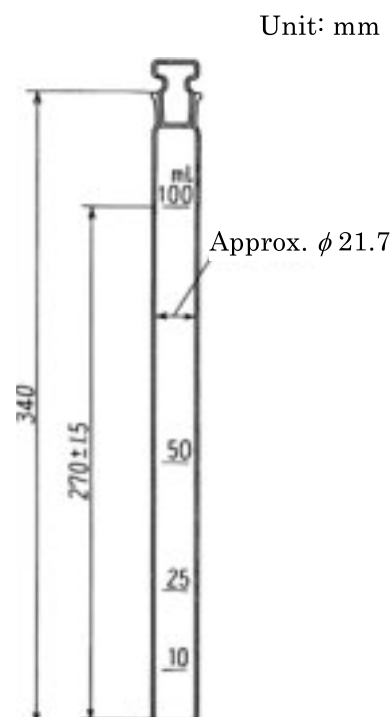


Figure 6 Colorimetric tube

9.2.4 Procedure

- Shake the sample well, transfer an appropriate amount of it to a colorimetric tube, and add water to the 100 ml mark. If the turbidity of the sample is 10 degrees (kaolin) or lower, use 100 ml of the sample as it is.
- Separately, place 1 ml to 10 ml of the kaolin standard solution [100 degrees (kaolin)] in incremental steps in separate colorimetric tubes, add water to each of them to the 100 ml mark to prepare kaolin standard solutions for nephelometry [1 degree (kaolin) to 10 degrees (kaolin)].
- Shake well both the colorimetric tube containing the sample and those containing standard kaolin solutions for nephelometry, and immediately place them in the dark box. Viewing from above the dark box, compare the cloudiness of the solutions and select the kaolin standard solution that matches the cloudiness of the sample.
- Calculate the visual-sensation turbidity [degrees (kaolin)] of the sample from the turbidity [degrees (kaolin)] of the selected kaolin standard solution using the following formula :

$$T = T_s \times \frac{100}{V}$$

where, T : visual-sensation turbidity [degrees (kaolin)]
 T_s : turbidity of the matching kaolin standard solution [degrees (kaolin)]
 V : volume of sample (ml)

9.3 Transmitted-light turbidity

9.3.1 Summary

The transmitted-light turbidity of a sample is determined by measuring the intensity of the light transmitted through the sample near the wavelength of 660 nm and comparing the measured intensity against the working curve prepared using kaolin standard solutions or formazin standard solutions.

Measuring range : 5 degrees (kaolin) to 50 degrees (kaolin) or 4 degrees (formazin) to 80 degrees (formazin) in the case of a 50 mm absorption cell, and 25 degrees (kaolin) to 250 degrees (kaolin) or 20 degrees (formazin) to 400 degrees (formazin) in the case of a 10 mm absorption cell

9.3.2 Reagents

- a) **Water**, in accordance with 9.2.2 a).
- b) **Kaolin standard solution [100 degrees (kaolin)]**, in accordance with 9.2.2 d).
- c) **Formazin standard solution [400 degrees (formazin)]** Dissolve 1.00 g of hydrazinium sulfate specified in **JIS K 8992** in an appropriate volume of water, transfer to a 100 ml volumetric flask, and add water to the mark.

Separately, dissolve 10.0 g of hexamethylenetetramine specified in **JIS K 8847** in an appropriate volume of water, transfer to a 100 ml volumetric flask, and add water to the mark.

Take 10 ml of each of these solutions in a 200 ml volumetric flask, and shake well to mix. Allow to stand for about 24 h while maintaining the liquid temperature at $25\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$, and add water to the mark.

9.3.3 Apparatus

- a) **Centrifugal separator**
- b) **Photometer** Spectrophotometer or photoelectric photometer

9.3.4 Procedure

9.3.4.1 Procedure using kaolin standard solutions

- a) Shake the sample well, transfer to a 50 mm absorption cell, and measure the intensity of the transmitted light near the wavelength of 660 nm by apparent absorbance.
 - Use a 10 mm absorption cell if the transmitted-light turbidity of the sample is 25 degrees (kaolin) to 250 degrees (kaolin).

- If the sample has a colour (especially when absorption is observed near the wavelength of 660 nm), centrifuge the sample [at about 3 000 min⁻¹ (adjusted according to the radius of the rotating part of the centrifugal separator) for about 20 min] or filter it through a filtering material with a pore diameter of not more than 1 µm, and using the resultant supernatant or the filtrate as the contrast solution, measure the intensity of the transmitted light by absorbance.
- b) Determine the transmitted-light turbidity [degrees (kaolin)] of the sample by referring to the working curve prepared using kaolin standard solutions.

9.3.4.2 Procedure using formazin standard solutions

- a) Shake the sample well, transfer to a 50 mm absorption cell, and measure the intensity of the transmitted light near the wavelength of 660 nm by apparent absorbance.
 - Use a 10 mm absorption cell if the transmitted-light turbidity of the sample is 20 degrees (formazin) to 400 degrees (formazin).
 - If the sample has a colour (especially when absorption is observed near the wavelength of 660 nm), centrifuge the sample [at about 3 000 min⁻¹ (adjusted according to the radius of the rotating part of the centrifugal separator) for about 20 min] or filter it through a filtering material with a pore diameter of not more than 1 µm, and using the resultant supernatant or the filtrate as the contrast solution, measure the intensity of the transmitted light by absorbance.
- b) Determine the transmitted-light turbidity [degrees (formazin)] of the sample by referring to the working curve prepared using formazin standard solutions.

9.3.5 Working curve

9.3.5.1 Procedure using kaolin standard solutions

- a) Prepare kaolin standard solutions for working curve according to the following.
 - Place 5.0 ml to 50 ml of the kaolin standard solution [100 degrees (kaolin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare kaolin standard solutions for working curve [5 degrees (kaolin) to 50 degrees (kaolin)].
 - When using a 10 mm absorption cell, place 2.5 ml to 25 ml of the kaolin standard solution [1 000 degrees (kaolin)] in incremental steps in separate 100 ml volumetric flasks to prepare kaolin standard solutions for working curve [25 degrees (kaolin) to 250 degrees (kaolin)].
- b) Plot the relation between the transmitted-light turbidity [degrees (kaolin)] of the kaolin standard solutions for working curve and the absorbance according to the procedure in 9.3.4.1 a).

9.3.5.2 Procedure using formazin standard solutions

- a) Prepare formazin standard solutions for working curve according to the following.
 - Place 1.0 ml to 20 ml of the formazin standard solution [400 degrees (formazin)]

in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare formazin standard solutions for working curve [4 degrees (formazin) to 80 degrees (formazin)].

- When using a 10 mm absorption cell, place 5.0 ml to 100 ml of the formazin standard solution [400 degrees (formazin)] in incremental steps in separate 100 ml volumetric flasks to prepare formazin standard solutions for working curve [20 degrees (formazin) to 400 degrees (formazin)].
- b) Plot the relation between the transmitted-light turbidity [degrees (formazin)] of the formazin standard solutions for working curve and the absorbance according to the procedure in 9.3.4.2 a).

9.4 Scattered-light turbidity

9.4.1 Summary

The scattered-light turbidity of a sample is determined by measuring the intensity of the light scattered due to the particles in the sample near the wavelength of 660 nm and comparing the measured intensity against the working curve prepared using kaolin standard solutions or formazin standard solutions.

Measuring range : 1 degree (kaolin) to 5 degrees (kaolin) or 0.4 degrees (formazin) to 5 degrees (formazin) (depend on the apparatus used)

9.4.2 Reagents

- a) **Water**, in accordance with 9.2.2 a).
- b) **Kaolin standard solution [100 degrees (kaolin)]**, in accordance with 9.2.2 d).
- c) **Formazin standard solution [40 degrees (formazin)]** Take 10 ml of formazin standard solution [400 degrees (formazin)] prepared according to 9.3.2 c) in a 100 ml volumetric flask and add water to the mark.

9.4.3 Apparatus

- a) **Scattered-light turbidity meter**, as illustrated in Figure 7.

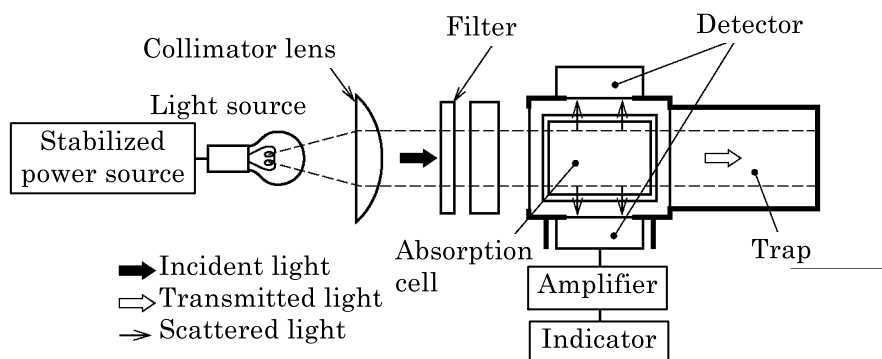


Figure 7 Example of construction of scattered-light turbidity meter

9.4.4 Procedure

9.4.4.1 Procedure using kaolin standard solutions

- a) Adjust the reading of the scattered-light turbidity meter to zero by taking water in the absorption cell, and then adjust it to 100 % by using the kaolin standard solution for working curve [5 degrees (kaolin)].

NOTE The intensity of the scattered light is commonly measured at 90° or 270° to the incident light.

- b) Shake the sample well, take the sample in the absorption cell, and take the reading (intensity of the scattered light) near the wavelength of 660 nm.
- c) Determine the scattered-light turbidity [degrees (kaolin)] of the sample by referring to the working curve prepared using kaolin standard solutions.

9.4.4.2 Procedure using formazin standard solutions

- a) Adjust the reading of the scattered-light turbidity meter to zero by taking water in the absorption cell, and then adjust it to 100 % using the formazin standard solution for working curve [5 degrees (formazin)].

NOTE The intensity of the scattered light is commonly measured at 90° or 270° to the incident light.

- b) Shake the sample well, take the sample in the absorption cell, and take the reading (intensity of the scattered light) near the wavelength of 660 nm.
- c) Determine the scattered-light turbidity [degrees (formazin)] of the sample by referring to the working curve prepared using formazin standard solutions.

9.4.5 Working curve

9.4.5.1 Procedure using kaolin standard solutions

- a) Place 1.0 ml to 5.0 ml of the kaolin standard solution [100 degrees (kaolin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare kaolin standard solutions for working curve [1 degree (kaolin) to 5 degrees (kaolin)].
- b) Plot the relation between the scattered-light turbidity [1 degree (kaolin) to 5 degrees (kaolin)] of the kaolin standard solutions for working curve and the meter readings (intensity of the scattered light) according to the procedure in **a)** and **b)** of 9.4.4.1.

9.4.5.2 Procedure using formazin standard solutions

- a) Place 1.0 ml to 12.5 ml of the formazin standard solution [40 degrees (formazin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare formazin standard solutions for working curve [0.4 degrees (formazin) to 5 degrees (formazin)] .
- b) Plot the relation between the scattered-light turbidity [0.4 degrees (formazin) to 5 degrees (formazin)] of the formazin standard solutions for working curve and the meter readings (intensity of the scattered light) according to the procedure in **a)**

and b) of 9.4.4.2.

9.5 Integrating-sphere turbidity

9.5.1 Summary

The integrating-sphere turbidity of a sample is determined by obtaining the ratio between the intensity of light scattered due to the particles in the sample and the intensity of light transmitted through the sample, and comparing the obtained ratio against the working curve prepared using kaolin standard solutions or formazin standard solutions.

Measuring range : 0.2 degrees (kaolin) to 5 degrees (kaolin) or 0.2 degrees (formazin) to 5 degrees (formazin) in the case of using a 50 mm absorption cell, and 5 degrees (kaolin) to 100 degrees (kaolin) or 5 degrees (formazin) to 100 degrees (formazin) in the case of using a 10 mm absorption cell

9.5.2 Reagents

- a) **Water**, in accordance with 9.2.2 a).
- b) **Kaolin standard solution [100 degrees (kaolin)]**, in accordance with 9.2.2 d).
- c) **Formazin standard solution [200 degrees (formazin)]** Take 50 ml of formazin standard solution [400 degrees (formazin)] prepared according to 9.3.2 c) in a 100 ml volumetric flask and add water to the mark.
- d) **Formazin standard solution [40 degrees (formazin)]**, in accordance with 9.4.2 c).

9.5.3 Apparatus

- a) **Integrating-sphere turbidity meter**, as illustrated in Figure 8.

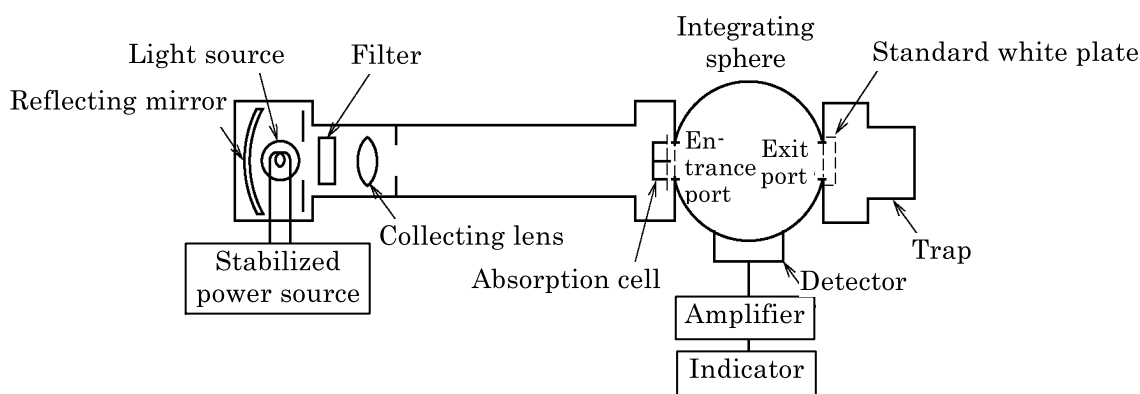


Figure 8 Example of construction of integrating-sphere turbidity meter

9.5.4 Procedure

9.5.4.1 Procedure using kaolin standard solutions

- a) Adjust the meter reading to zero with a 50 mm absorption cell containing water and a trap set up in the optical path, and then adjust it to 100 with a standard white plate inserted into the trap.

- b) Then, with a 50 mm absorption cell containing sample previously shaken well, used in place of the 50 mm absorption cell containing water, and a trap (without the standard white plate) set up in the optical path, measure the intensity of the scattered light, T_d .
- c) Insert the standard white plate into the trap and measure the intensity of the total transmitted light of the sample, T_t .
- d) Calculate the value of $(T_d/T_t) \times 100$ and determine the integrating-sphere turbidity [degrees (kaolin)] of the sample by referring to the working curve prepared using kaolin standard solutions.

9.5.4.2 Procedure using formazin standard solutions

- a) Perform the procedures in a) to c) of 9.5.4.1.
- b) Calculate the value of $(T_d/T_t) \times 100$ and determine the integrating-sphere turbidity [degrees (formazin)] of the sample by referring to the working curve prepared using formazin standard solutions.

9.5.4.3 Method without calculation of $(T_d/T_t) \times 100$

- a) Perform the procedure in 9.5.4.1 a). Set up an absorption cell containing sample instead of an absorption cell containing water, and adjust the meter reading to 100.
- b) Remove the standard white plate and note the reading on the meter.
- c) Determine the integrating-sphere turbidity of the sample by referring to the working curve.

9.5.5 Working curve

9.5.5.1 Procedure using kaolin standard solutions

- a) Prepare kaolin standard solutions for working curve according to the following.
 - Place 0.2 ml to 5.0 ml of the kaolin standard solution [100 degrees (kaolin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare kaolin standard solutions for working curve [0.2 degrees (kaolin) to 5 degrees (kaolin)].
 - When using a 10 mm absorption cell, place 5.0 ml to 100 ml of the kaolin standard solution [100 degrees (kaolin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare kaolin standard solutions for working curve [5 degrees (kaolin) to 100 degrees (kaolin)]. For preparing a 100 degrees (kaolin) solution, add the whole volume to the 100 ml volumetric flask.
- b) Perform the procedures in a) to c) of 9.5.4.1, calculate the value of $(T_d/T_t) \times 100$ and plot the relation between this value and the integrating-sphere turbidity [degrees (kaolin)] of the kaolin standard solutions for working curve.

9.5.5.2 Procedure using formazin standard solutions

- a) Prepare formazin standard solutions for working curve according to the following.

- Place 0.5 ml to 12.5 ml of the formazin standard solution [40 degrees (formazin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare formazin standard solutions for working curve [0.2 degrees (formazin) to 5 degrees (formazin)].
 - When using a 10 mm absorption cell, place 2.5 ml to 50 ml of the formazin standard solution [200 degrees (formazin)] in incremental steps in separate 100 ml volumetric flasks, and add water to each of them to the mark to prepare formazin standard solutions for working curve [5 degrees (formazin) to 100 degrees (formazin)].
- b) Perform the procedures in **a)** to **c)** of **9.5.4.1**, calculate the value of $(T_d/T_l) \times 100$ and plot the relation between this value and the integrating-sphere turbidity [degrees (formazin)] of the formazin standard solutions for working curve.

9.5.5.3 Method without calculation of $(T_d/T_l) \times 100$

Perform the same procedure as described above using kaolin (or formazin) standard solutions, plot the integrating-sphere turbidity [degrees (kaolin)] [or [degrees (formazin)]] on the abscissa and the meter readings [equivalent to $(T_d/T_l) \times 100$] on the ordinate to prepare a working curve.

10 Chromaticity

10.1 General

The methods for colour indication of a sample and their applications are shown in Table 1.

Table 1 Methods for colour indication of sample and their applications

Method	Applicable to	Test clause
Method using stimulus values and chromaticity coordinates	Industrial water and industrial wastewater	10.2
Method using platinum and cobalt	Industrial water	10.3
Method using the three wavelengths	Industrial wastewater	10.4
NOTE The International Standard corresponding to the method using the three wavelengths and the symbol of degree of correspondence are as follows. ISO 7887 : 1994 <i>Water quality — Examination and determination of colour</i> (MOD) In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and JIS are IDT (identical), MOD (modified), and NEQ (not equivalent) according to ISO/IEC Guide 21-1.		

10.2 Method using the stimulus values and chromaticity coordinates

10.2.1 Summary

Using water as the contrast solution, the transmittance (percentage) of a sample is

measured at a specified wavelength from 400 nm to 700 nm, and the tristimulus values ¹⁾, X, Y and Z, and then the chromaticity coordinates x and y are calculated. The colour of the sample is expressed by the stimulus value Y and chromaticity coordinates x and y .

NOTE For expression of chromaticity by the dominate wavelength (or complementary wavelength) and the excitation purity, see Annex A.

Note ¹⁾ The tristimulus values are based on the understanding that the optical nerve perceives colour in three different ways and the mixing ratio of the three stimuli to the optical nerve determines the colour perception by the optical nerve, and are obtained by arranging the wavelengths giving the three respective stimuli into X, Y and Z series, and totalizing the transmittance (percentage).

10.2.2 Reagents

- a) **Water** Filter the water of A3 specified in **JIS K 0557** through a filtering material with a pore diameter of about 0.1 μm , discard the initial filtrate of about 200 ml, and use the ensuing filtrate.

10.2.3 Apparatus

- a) **Centrifugal separator**
- b) **Photometer** A spectrophotometer which can use a 100 mm absorption cell and can cover the entire visible field with 10 nm or less wavelength range, or a colorimeter with performance equivalent to this.

10.2.4 Procedure

- a) Either centrifuge the sample at about $3\,000\text{ min}^{-1}$ (adjusted according to the radius of the rotating part of the centrifugal separator) for about 20 min to remove turbidity or filter it through a filter paper of Class 5 C or a filtering material with a pore diameter of not more than 1 μm .
- b) Transfer a part of this liquid into a 100 mm absorption cell, and measure the transmittance (percentage) at each wavelength given in Table 2 by using water as a contrast solution.
- c) If a 100 mm absorption cell cannot be used because of the darkness of the sample, other absorption cell with a suitable optical path length shall be used. In this case, measure the absorbance at the specified wavelength, convert the measured absorbance into the absorbance given by a 100 mm absorption cell, and determine the transmittance (percentage) from this absorbance.

Table 2 Weighting functions, f_x , f_y , f_z , for calculating tristimulus values X , Y and Z at the wavelength interval 20 nm

Wavelength λ (nm)	Standard illuminant C		
	f_x	f_y	f_z
400	0.019	−0.003	0.062
420	2.993	0.087	14.387
440	7.634	0.510	38.438
460	6.642	1.382	38.130
480	2.360	3.206	19.545
500	0.068	6.907	5.746
520	1.196	12.876	1.444
540	5.590	18.261	0.359
560	11.751	19.592	0.073
580	16.795	15.991	0.026
600	17.897	10.694	0.013
620	14.021	6.261	0.002
640	7.453	2.901	0.000
660	2.731	1.003	0.000
680	0.756	0.273	0.000
700	0.166	0.059	0.000
Total	98.072	100.000	118.225
Chromaticity coordinates	$x = 0.310\ 1$	$y = 0.316\ 2$	$z = 0.373\ 7$
NOTE Table 2 is in accordance with light source C given in Attached Table 5-1-1 of JIS Z 8719 .			

- d) Using the values of f_x , f_y and f_z , corresponding to the transmittance (percentage) at the specified wavelengths given in Table 2, calculate the tristimulus values X , Y and Z and chromaticity coordinates x and y according to the following formulas :

$$X = \frac{1}{K} \sum_{400}^{700} f_x(\lambda) \cdot \tau(\lambda) \dots\dots\dots (1)$$

$$Y = \frac{1}{K} \sum_{400}^{700} f_y(\lambda) \cdot \tau(\lambda) \dots\dots\dots (2)$$

$$Z = \frac{1}{K} \sum_{400}^{700} f_z(\lambda) \cdot \tau(\lambda) \dots\dots\dots (3)$$

$$x = \frac{X}{X + Y + Z} \dots\dots\dots (4)$$

$$y = \frac{Y}{X + Y + Z} \dots\dots\dots (5)$$

where, X : stimulus value X
 Y : stimulus value Y
 Z : stimulus value Z
 K : 100.000

$f_X(\lambda)$: f_X at the wavelength λ

$f_Y(\lambda)$: f_Y at the wavelength λ

$f_Z(\lambda)$: f_Z at the wavelength λ

$\tau(\lambda)$: transmittance (percentage) at the wavelength λ

10.3 Platinum-cobalt chromaticity

10.3.1 Summary

The platinum-cobalt chromaticity is a degree of pale yellow to yellowish brown colour generated by matters dissolved in water or suspended in water as colloids. The colour obtained when 1 ml of the platinum-cobalt chromaticity standard solution (1.0 mg of platinum and 0.5 mg of cobalt) is added to 1 000 ml of water is one degree in platinum-cobalt chromaticity.

10.3.2 Reagents

- a) **Water**, in accordance with 10.2.2 a).
- b) **Platinum-cobalt chromaticity standard solution (1 000 degrees)**
 - 1) Add 2.49 g of potassium hexachloroplatinate (IV) specified in **JIS K 8163** and 2.00 g of cobalt (II) chloride hexahydrate specified in **JIS K 8129** to 200 ml of hydrochloric acid (guaranteed grade) specified in **JIS K 8180**, add water to dissolve, transfer to a 1 000 ml volumetric flask, and add water to the mark. Store this solution in a coloured bottle.
 - 2) When using platinum instead of the potassium hexachloroplatinate (IV) specified in **JIS K 8163**, dissolve 1.00 g of platinum in an appropriate amount of aqua regia [mixture of three volumes of hydrochloric acid (guaranteed grade) specified in **JIS K 8180** and one volume of nitric acid specified in **JIS K 8541**], add excess hydrochloric acid and evaporate to dryness on a boiling water bath. Repeat this procedure two or three times to remove nitric acid, and dissolve 2.00 g of cobalt (II) chloride hexahydrate specified in **JIS K 8129** together with 200 ml of hydrochloric acid (guaranteed grade) specified in **JIS K 8180**, transfer to a 1 000 ml volumetric flask, and add water to the mark. Store this solution in a coloured bottle.

10.3.3 Implements and apparatus

- a) **Dark box**, in accordance with 9.2.3 a).
- b) **Colorimetric tube**, in accordance with 9.2.3 b).
- c) **Centrifugal separator**

10.3.4 Procedure

- a) Centrifuge the sample at about 3 000 min⁻¹ (adjusted according to the radius of the rotating part of the centrifugal separator) for 20 min to remove turbidity or filter it through a filter paper of Class 5C or a filtering material with a pore diameter of not more than 1 µm.
- b) Transfer an appropriate amount of this sample to a 100 ml colorimetric tube, and add water to the 100 ml mark.

- c) Place 0.1 ml to 2.0 ml of the platinum-cobalt chromaticity standard solution (1 000 degrees) in incremental steps in separate 100 ml colorimetric tubes, add water to each of them to the 100 ml mark, stopper and shake well to prepare a 1-20 degrees series of platinum-cobalt chromaticity standard solutions.
- d) Place the sample and the platinum-cobalt chromaticity standard solution series on a white paper or in a dark box, and observe from above to compare the colour of the sample against those of the standard solution series. Select a standard solution with a matching colour to the sample, and determine the corresponding platinum-cobalt chromaticity.
- e) Calculate the platinum-cobalt chromaticity of the sample using the following formula :

$$C = C_s \times \frac{100}{V}$$

where, C : platinum-cobalt chromaticity
 C_s : chromaticity (degrees) of the matching platinum-cobalt chromaticity standard solution
 V : volume of sample (ml)

10.4 Method using the three wavelengths

10.4.1 Summary

The sample is filtered through a membrane filter, and the absorbance at the three wavelengths 436 nm, 525 nm and 620 nm are measured. The absorption factor $\alpha(\lambda)$ [hereafter referred to as $\alpha(\lambda)$] of each of these absorbances is calculated and used to express the colour of the sample.

10.4.2 Reagents

- a) **Water**, in accordance with 10.2.2 a).

10.4.3 Apparatus

- a) **Photometer**, in accordance with 10.2.3 b).
- b) **pH meter**, of Type II specified in 5.2 of JIS Z 8802.
- c) **Thermometer**, in accordance with 6.3.1 a).

10.4.4 Procedure

- a) Filter the sample through a membrane filter with a pore diameter of 0.45 μm . In parallel with the colour measurement, measure the pH and temperature of the filtered sample. If the sample has a dark colour, dilute the filtered sample using a given volume of water, and factor in this dilution in calculating the value of $\alpha(\lambda)$. Note the pH and temperature values alongside the chromaticity measurement results for information.
- b) Transfer a part of this solution to an absorption cell, and measure the absorbance at the wavelengths 436 nm, 525 nm and 620 nm using water as the contrast solution. If $\alpha(\lambda)$ is smaller than 10 m^{-1} , use an absorption cell with an optical path

length of 10 mm or more.

- c) From the measured absorbance, calculate $\alpha(\lambda)$ [absorbance (m^{-1}) per 1 m of optical path length] at each wavelength according to the following formula :

$$\alpha(\lambda) = \frac{A}{d} \times 1\,000$$

where, $\alpha(\lambda)$: absorbance per 1 m of optical path length (m^{-1})

A : absorbance of filtered sample at the wavelength λ

d : optical path length of absorption cell (mm)

1 000 : factor for converting to absorbance per 1 m of optical path length

11 Odour and threshold odour number (TON)

11.1 General

The test of odour is performed either by the detection of odour or the determination of the threshold odour number (TON).

The causes of odour in water include: propagation and extinction of bacteria, algae, microorganisms, etc.; mixing-in of municipal sewage, cattle shed drainage, and industrial wastewater; elution of inner surface treatment substances of water-storage tank and piping system; and residual chlorine after chlorine treatment.

The test of odour is dependant on the personal sense of smell, and therefore prone to variation of results between individual testers, and furthermore can be affected by the temperature and humidity, as well as the type of diet and smoking habit of the tester. To reduce the effect of this variation, it is desirable that at least five, and preferably about ten, testers participate in the testing of the same sample.

TON (Threshold Odour Number) is the threshold dilution ratio for odour, that is, the multiple of dilution at which the odour is clearly detectable.

11.2 Odour

11.2.1 Summary

The type and degree of odour of the sample when the sample is heated to about 40 °C is determined.

11.2.2 Implements

- a) **Erlenmeyer flask with ground-in stopper**, of 300 ml.

11.2.3 Procedure

- a) Place 200 ml of sample in a 300 ml Erlenmeyer flask with ground-in stopper, loosely apply the stopper, and warm to about 40 °C.
- b) While swirling the flask, remove the stopper, and immediately test the sample for the presence, type and degree of odour. It is recommended to examine and record the odour of the unheated sample (odour at the time of cold) at the time of collection of the sample.

- c) The odour of the sample shall be roughly indicated by its type and degree according to Table 3.

Table 3 Example of classification and types of odour

Gross classification of odours	Types of odour
1) Fragrant odour	Odours of melon, violet, garlic, cucumber, aromatics, spice, etc.
2) Botanical odour	Odours of algae, green grass, timber, seaweed, etc.
3) Earthy odour and mouldy odour	Odours of earth, swamp, mould, etc.
4) Fishy or shellfishy odour	Odours of fish, lever oil, clam, etc.
5) Chemical odour	Odours of phenols, tar, oil, fats, paraffin, chlorine, hydrogen sulfide, chlorophenol, drugstore, chemicals, etc.
6) Metallic odour	Odours of iron, metal, etc.
7) Putrescent odour	Odours of garbage, sewage, pigpen, putrescence, etc.
8) Unpleasant odour	Intensified unpleasant odours of fish, pigpen, putrescence, etc.

11.3 Threshold odour number (TON)

11.3.1 Summary

The threshold odour number (TON) is the index of the intensity of an odour, expressed by the dilution ratio of the sample that yields a clearly detectable odour when the sample is added to the odour-free water maintained at about 40 °C.

11.3.2 Reagents

- a) **Odour-free water** Pass water of A3 specified in **JIS K 0557** at an approximate rate of 5 L/h per 1 L of active carbon through the apparatus shown in Figure 9.

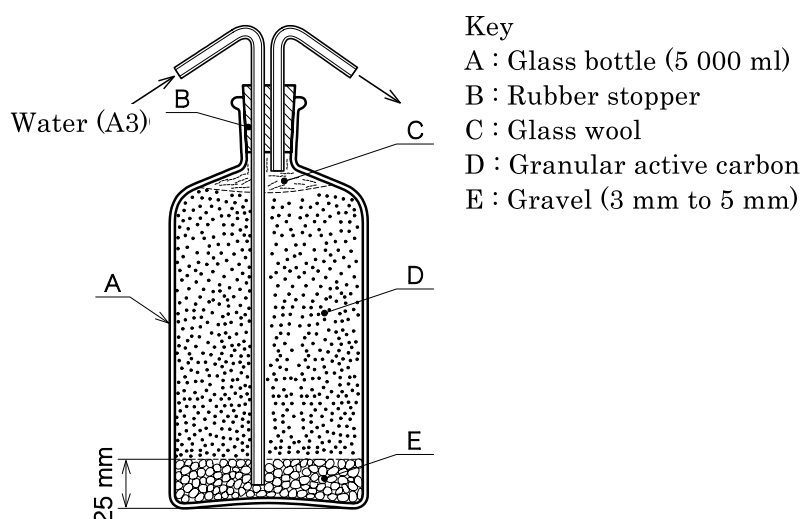


Figure 9 Example of method for preparing odour-free water

11.3.3 Implements

- a) **Erlenmeyer flask with ground-in stopper**, of 300 ml, washed with odour-free water prior to use.
- b) **Water bath**, equipped with a temperature controller.

11.3.4 Procedure

a) Preliminary test

- 1) Place 200 ml, 40 ml, 10 ml and 4 ml of sample in separate 300 ml Erlenmeyer flasks with ground-in stopper, and add odour-free water to each of these to make 200 ml to prepare the samples for preliminary test.
- 2) In a separate 300 ml Erlenmeyer flask with ground-in stopper, place 200 ml of odour-free water to use as a reference water.
- 3) Heat the samples for preliminary test and the reference water to 40 °C to 50 °C on a water bath. Shake the reference water and smell its odour upon removing the stopper.
- 4) Then, perform the same procedure starting with the most diluted sample, and successively compare the odour of the series of samples with that of the reference water. Determine the smallest volume (ml) of sample at which the odour was detectable.

b) Test

- 1) Select the series of sample volumes in ml(s) to use for the test based on the sample volume determined in **a) 4)** according to Table 4.

Table 4 Sample volumes for test

	Unit: ml			
Sample volume determined by preliminary test	200	40	10	4
Sample volumes to be used for test	200	40	10	4.0
	100	28.5	8.0	2.9
	67	20	6.7	2.0
	50	13.3	5.7	1.3
	40	10	4.0	1.0

- 2) Put the volumes of sample determined in **a)** in separate 300 ml Erlenmeyer flasks with ground-in stopper and add odour-free water to make 200 ml to use as samples for the test.
- 3) Perform the procedures in **2)** to **4)** of **a)** on these samples and determine the smallest volume in ml(s) of sample at which the odour was detectable. Based on this volume, calculate the threshold odour number (TON) according to the following formula :

$$TON = \frac{200}{V}$$

where, V : volume of sample used for test (ml)

- 4) In the case of determining the degree of odour (pO), use the following formula :

$$pO = \frac{1}{\log 2} \times \log TON = 3.32 \times \log TON$$

- c) **Instructions regarding the test** The following instructions shall be borne in mind in performing the test procedures in **a)** and **b)**.
- Samples with a strong odour should be diluted ten times with odour-free water prior to the preliminary test and the test.
 - As the test is easily influenced by the ambient conditions, it should be carried out in an odourless and noise-free room.
 - Testers should refrain from smoking, drinking tea or coffee, eating, and so on immediately before the test, and also make sure that their hands are free from smells of soap, lotion, perfume, and so on.
 - The tester should take a recess of about 15 min to 30 min after four or five successive tests in order to regain fresh sense of smell.

12 pH

12.1 General

The pH is measured by the glass-electrode method specified in **JIS Z 8802**. Since the pH of a sample is quickly changed by chemical, physical and biochemical action and other influences, it should be measured immediately after collection of the sample.

NOTE The International Standard corresponding to this test method and the symbol of degree of correspondence are as follows.

ISO 10523 : 1994 *Water quality — Determination of pH* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

12.2 Reagents

- a) **Water**, of A2 or A3 specified in **JIS K 0557**. For preparing a borate pH standard solution or carbonate pH standard solution, the carbon dioxide-free water as specified in **4.4 b)** shall be used.
- b) **Potassium trihydrogen dioxalate dihydrate**, specified in **JIS K 8474**.
- c) **Potassium hydrogen phthalate**, for pH standard solution, specified in **JIS K 8809**.
- d) **Potassium dihydrogen phosphate**, for pH standard solution, specified in **JIS K 9007**.
- e) **Disodium hydrogenphosphate**, for pH standard solution, specified in **JIS K 9020**.

- f) **Sodium tetraborate decahydrate**, for pH standard solution, specified in **JIS K 8866**.
- g) **Sodium hydrogen carbonate**, for pH standard solution, specified in **JIS K 8622**.
- h) **Sodium carbonate**, for pH standard solution, specified in **JIS K 8625**.
- i) **pH standard solutions** Prepared solutions or certified solutions as described in the following.
 - 1) Prepared pH standard solutions are those prepared in accordance with **7.3.2** of **JIS Z 8802**.
 - 2) Certified pH standard solutions are Class 2 certified pH standard solutions traceable to the National Metrological Standard.
 - 3) The pH values of pH standard solutions at each temperature shall be in accordance with Table 5 and Table 6. The pH values at temperatures not given in the tables below shall be obtained by interpolation.

Table 5 Typical pH values of prepared pH standard solutions at each temperature

Temperature °C	pH value				
	Oxalate	Phthalate	Neutral phosphate	Borate	Carbonate ^{a)}
0	1.67	4.01	6.98	9.46	10.32
5	1.67	4.01	6.95	9.39	(10.25)
10	1.67	4.00	6.92	9.33	10.18
15	1.67	4.00	6.90	9.27	(10.12)
20	1.68	4.00	6.88	9.22	(10.07)
25	1.68	4.01	6.86	9.18	10.02
30	1.69	4.01	6.85	9.14	(9.97)
35	1.69	4.02	6.84	9.10	(9.93)
38	—	—	—	—	9.91
40	1.70	4.03	6.84	9.07	—
45	1.70	4.04	6.83	9.04	—
50	1.71	4.06	6.83	9.01	—
55	1.72	4.08	6.84	8.99	—
60	1.73	4.10	6.84	8.96	—
70	1.74	4.12	6.85	8.93	—
80	1.77	4.16	6.86	8.89	—
90	1.80	4.20	6.88	8.85	—
95	1.81	4.23	6.89	8.83	—
Note ^{a)} The values in parentheses are the secondary interpolated values.					

Table 6 Typical pH values of certified pH standard solutions at each temperature

Temperature °C	pH value					
	Oxalate	Phthalate	Neutral phosphate	Phosphate	Borate	Carbonate
	2nd grade	2nd grade	2nd grade	2nd grade	2nd grade	2nd grade
0	1.67	4.00	6.98	7.53	9.46	10.32
5	1.67	4.00	6.95	7.50	9.40	10.24
10	1.67	4.00	6.92	7.47	9.33	10.18
15	1.67	4.00	6.90	7.45	9.28	10.12
20	1.68	4.00	6.88	7.43	9.22	10.06
25	1.68	4.01	6.86	7.41	9.18	10.01
30	1.68	4.02	6.85	7.40	9.14	9.97
35	1.69	4.02	6.84	7.39	9.10	9.92
38	1.69	4.03	6.84	7.38	9.08	—
40	1.69	4.04	6.84	7.38	9.07	9.89
45	1.70	4.05	6.83	7.37	9.04	9.86
50	1.71	4.06	6.83	7.37	9.01	9.83
55	1.72	4.08	6.83	—	8.98	—
60	1.72	4.09	6.84	—	8.96	—
70	1.74	4.13	6.84	—	8.92	—
80	1.77	4.16	6.86	—	8.88	—
90	1.79	4.20	6.88	—	8.85	—
95	1.81	4.23	6.89	—	8.83	—

- 4) The pH standard solutions are prone to change of pH during storage, and shall not be used after being left in storage for a long period of time. This is especially true for borate and carbonate pH standard solutions, whose pH may be easily reduced over time due to their absorbing carbon dioxide in the atmosphere.
- 5) The pH standard solutions which have been used once or left exposed to the atmosphere shall not be used.
- j) **0.1 mol/L sodium hydroxide solution** Take about 30 ml of water in a polyethylene bottle, and while cooling, add gradually about 30 g of sodium hydroxide specified in **JIS K 8576** to dissolve. Tightly stopper, and allow to stand for four or five days. Transfer 5 ml of the supernatant of this solution to a 1 000 ml airtight polyethylene vessel, add carbon dioxide-free water specified in **4.4 b)** to make 1 000 ml, and mix. Store this solution while shielding from carbon dioxide.

12.3 Implements and apparatus

- a) **pH meter**, of Type II specified in **5.2** of **JIS Z 8802**, or of other type suitable for the specific purpose of test. The calibration of the pH meter shall be as follows.
 - 1) Attach a detector [an electrode (glass electrode or reference electrode), thermometer, etc.], and turn on the pH meter. A glass thermometer having been left dry for a long period of time shall be immersed in water and brought to equilibrium before use. As necessary, stains on a glass electrode shall be swiftly removed with a detergent, hydrochloric acid (1+20) (prepared using hydrochloric acid specified in

JIS K 8180), etc. and then thoroughly rinsed off with running water. Stain removal of a reference electrode shall be the same. The exchange of the internal liquid (potassium chloride solution) of the reference electrode shall follow the instruction manual. For avoiding the contamination of the liquid junction part, the electrolyte requires a hydrostatic pressure equivalent to that obtained by the water level difference of 2 cm or more.

- 2) Wash the detector three times or more with water, and wipe the moisture off with a clean soft paper.
- 3) Take the neutral phosphate pH standard solution in a beaker, and dip the detector in it. Adjust the temperature compensation dial or, in the case of a digital switch, adjust the display value on the switch to the temperature of the neutral phosphate pH standard solution. The temperature of the pH standard solution shall be as close to the temperature of the sample as possible. The temperature variation of each pH standard solution during this time shall be ± 2 °C.
- 4) Adjust the pH to the value corresponding to the temperature of the neutral phosphate pH standard solution (Table 5 or Table 6).
- 5) Wash the detector three times or more with water, and wipe the moisture off with a clean soft paper or the like.
- 6) Perform calibration according to the following using an appropriate pH standard solution for the pH of the sample.
 - 6.1) When the pH value of the sample is 7 or lower, dip the detector in a beaker containing either a phthalate pH standard solution or oxalate pH standard solution. Adjust the pH to the value corresponding to the temperature of the pH standard solution used (Table 5 or Table 6). The temperature variation of each pH standard solution during this time shall be ± 2 °C.
 - 6.2) When the pH value of the sample is over 7 and under 11, dip the detector in a beaker containing a phosphate pH standard solution, borate pH standard solution or carbonate pH standard solution. Adjust the pH to the value corresponding to the temperature of the pH standard solution used (Table 5 or Table 6). The temperature variation of each pH standard solution during this time shall be ± 2 °C.
 - 6.3) When the pH of the sample is 11 or more and the sample contains a particularly high concentration of alkaline metal elements, an alkaline error can easily be caused. For avoiding this, an electrode with a small alkaline error shall be used and the pH meter shall be calibrated with 0.1 mol/L sodium hydroxide solution containing no carbonate or with calcium hydroxide solution (saturated at 25 °C). These solutions shall be prepared immediately before use because their pH can be easily reduced due to their absorbing carbon dioxide from the air. Table 7 shows the pH value of these solutions at each temperature.

Table 7 pH value of 0.1 mol/L sodium hydroxide solution and calcium hydroxide solution saturated at 25 °C at each temperature

Temperature °C	0.1 mol/L sodium hydroxide solution	Calcium hydroxide solution saturated at 25 °C	Temperature °C	0.1 mol/L sodium hydroxide solution	Calcium hydroxide solution saturated at 25 °C
0	13.8	13.43	35	12.6	12.14
5	13.6	13.21	40	12.4	11.99
10	13.4	13.00	45	12.3	11.84
15	13.2	12.81	50	12.2	11.70
20	13.1	12.63	55	12.0	11.58
25	12.9	12.45	60	11.9	11.45
30	12.7	12.30			

- 7) Repeat the procedures in **2)** to **6)** until the indicated pH value agrees with the pH value corresponding to the temperature of the pH standard solution within the tolerance of ± 0.05 in the case of using a Type II pH meter, ± 0.005 in the case of using a Type 0 pH meter, ± 0.02 in the case of using a Type I pH meter, and ± 0.1 in the case of using a Type III pH meter.
- 8) When the pH meter shows a delay in reaching equilibrium in the pH reading of the pH standard solution after measurement, the contamination on its electrode system (measurement cell) should be removed by the following procedure.
 - 8.1) Wipe the electrode thoroughly with a soft paper regularly (once each day or each week). If the contamination is organic matter, remove with ethanol (volume fraction 70 %) [prepared by using ethanol (95) specified in **JIS K 8102**], acetone (specified in **JIS K 8034**), warmed detergent solution and so on.
 - 8.2) Clean the electrode with an organic solvent. The use of an organic solvent shall be limited to a short term because it may take away the moisture from the glass film surface and cause reduction in sensitivity. If this cleaning has been performed, immerse the electrode in water for at least several hours for recovery after the cleaning, and perform recalibration.
 - 8.3) If the contamination is adhesion of calcium carbonate, remove with diluted hydrochloric acid.
 - 8.4) In the case of a reference electrode or complex electrode, immerse in an electrolyte for reference electrode, or in the case of a glass electrode, immerse in water.

b) **Thermometer**, in accordance with **6.3.1 a)**.

12.4 Procedure

- a) Wash the detector of the calibrated pH meter three times or more with water, and wipe the moisture off with a clean soft paper or the like.
- b) Take the sample in a beaker, and immerse the detector in it. Adjust the temperature compensation dial or, in the case of a digital switch, adjust the display value on the switch to the temperature of the sample, and measure the pH of the sample.

The temperature variation of the sample shall be ± 2 °C.

- c) Remove the detector from the beaker, wash it three times or more with water, and wipe the moisture off with a clean soft paper or the like.
- d) Take the sample in a beaker again, and immerse the detector in it to measure the pH. The temperature variation of the sample shall be ± 2 °C.
- e) Repeat the procedures in **c)** and **d)** until three consecutive measurements agree within ± 0.1 , and calculate the average of the three measurements to obtain the pH value of the sample. The repeatability of ± 0.1 may be difficult to achieve for samples with low buffering property since their pH is prone to variation; for such samples, measurements agreeing within ± 0.2 shall be obtained and averaged to calculate the pH. Where the pH easily fluctuates due to carbon dioxide in the air, it is advisable to use a flow-through type electrode.
- f) The following notes should be borne in mind in carrying out the test procedure.
 - 1) The pH measurement of water of low ionic strength (not more than 5 mS/m in electrical conductivity) or water of low buffering property should be performed with special attention.
 - 2) When the pH value of the sample is 11 or higher, an electrode having a glass film of low solubility, that is, a high alkali electrode, should be used. Use of such an electrode is recommended for all measurements of water. The reference electrode should be a ground sleeve type using the potassium chloride solution (1 mol/L) (prepared by using the potassium chloride specified in **JIS K 8121**) as its electrolyte. For avoiding the influence of potassium chloride on the glass electrode, the solution should be gently agitated. For avoiding the influence of air, sample should be poured in a measurement container. For restricting the electrostatic effect, the measurement container should be electromagnetically screened, and a metallic electrode shall be inserted in the test solution to perform earthing.
 - 3) The temperature and certain types of gas and organic compounds interfere with the measurement of pH. Suspended matter can cause a significant error (suspension effect). To avoid these interferences, the electrode should be inserted into the supernatant of the solution after the solution has settled, or the ultrafiltration method should be applied. Some samples may be more likely to cause adhesion of oils and fats or other contaminants to the electrode surface, and contamination of the diaphragm. This can be prevented in the liquid junction part of the reference electrode. When the precipitation of, for example, silver sulfide or protein flock is generated in the diaphragm, a salt bridge of potassium nitrate (1 mol/L) (prepared by using potassium nitrate specified in **JIS K 8548**) should be used between the sample and the reference electrode.

13 Electrical conductivity

13.1 General

Electrical conductivity is the inverse number of electric resistivity ($\Omega \cdot \text{m}$) of a solu-

tion, expressed in S/m. Electric conductance is the inverse number of electric resistance (Ω) of a solution, expressed in S. In a test of water, the measurement at 25 °C is expressed in mS/m or mS, which are one-thousandth of S/m and S, respectively. “mS/m” reads as “millisiemens per metre”.

This test shall be carried out immediately after sampling. When this is not possible, the sample shall be poured in a sample container to the full capacity and stored in a dark place at 0 °C to 10 °C with the container tightly stoppered, and shall be tested as soon as possible.

NOTE The International Standard corresponding to this test method and the symbol of degree of correspondence are as follows.

ISO 7888 : 1985 *Water quality — Determination of electrical conductivity* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

13.2 Reagents

- a) **Water**, of A2 or A3 specified in **JIS K 0557**, having an electrical conductivity of not more than 0.2 mS/m (25 °C), adjusted to 20 °C $\pm$ 2 °C at the time of preparation.
- b) **Potassium chloride** Grind potassium chloride (for electrical conductivity measurement) specified in **JIS K 8121** in an agate mortar, heat it at 500 °C for about 4 h, and allow it to cool in a desiccator.
- c) **Potassium chloride standard solutions**, in accordance with 1) to 4) below. Store these potassium chloride standard solutions in tightly stoppered polyethylene bottles or in borosilicate glass bottles. The electrical conductivity of these solutions is shown in Table 8 and Table 9. Commercially available potassium chloride standard solutions may be used.
 - 1) **1 mol/kg potassium chloride standard solution**, as specified in 7.1 of **JIS K 0130**.
 - 2) **0.1 mol/kg potassium chloride standard solution**, as specified in 7.1 of **JIS K 0130**.
 - 3) **0.01 mol/kg potassium chloride standard solution**, as specified in 7.1 of **JIS K 0130**.
 - 4) **0.001 mol/kg potassium chloride standard solution**, as specified in 7.1 of **JIS K 0130**.
- d) **Hydrochloric acid (1+1)**, prepared by diluting the hydrochloric acid (guaranteed grade) specified in **JIS K 8180**.
- e) **Hydrochloric acid (1+100)**, prepared by diluting the hydrochloric acid (guaranteed grade) specified in **JIS K 8180**.
- f) **Sulfuric acid (1+360)** Place 360 volumes of water in a beaker and while stirring, add gradually 1 volume of sulfuric acid specified in **JIS K 8951**.

- g) **Electrolyte** Weigh 30 g of hydrogen hexachloroplatinate (IV) hexahydrate specified in **JIS K 8153** and 0.25 g of lead (II) acetate trihydrate specified in **JIS K 8374**, and add water of A2, A3 or A4 specified in **JIS K 0557** to these to make the total volume 1 000 ml. This solution is used whenever necessary.

Table 8 Electrical conductivity of potassium chloride standard solutions

Temperature °C	Electrical conductivity value and its uncertainty (mS/m)						
	0.01 mol/kg potassium chloride standard solution		0.1 mol/kg potassium chloride standard solution		1 mol/kg potassium chloride standard solution		CO ₂ saturated water
	Electrical conductivity	2 u_c	Electrical conductivity	2 u_c	Electrical conductivity	2 u_c	Electrical conductivity
0	77.292	0.023	711.685	0.285	6 348.8	2.5	0.058
5	89.096	0.027	818.370	0.327	7 203.0	2.9	0.068
10	101.395	0.030	929.172	0.372	8 084.4	3.2	0.079
15	114.145	0.034	1043.71	0.42	8 990.0	3.6	0.089
18	121.993	0.037	1114.06	0.45	—	—	0.095
20	127.303	0.038	1161.59	0.46	9 917.0	4.0	0.099
25	140.823	0.042	1282.46	0.51	10 862.0	4.3	0.110
30	154.663	0.046	1405.92	0.56	11 824.0	4.7	0.120
35	168.779	0.051	1531.60	0.61	12 797.0	5.1	0.130
40	183.127	0.055	1659.10	0.66	13 781.0	5.5	0.140
45	197.662	0.059	1788.06	0.72	14 772.0	5.9	0.151
50	212.343	0.064	1918.09	0.77	15 767.0	6.3	0.161
NOTE The conductivity values are derived from the data provided by the National Institute of Standards and Technology (NIST) [<i>Pure Appl. Chem.</i> , 73 , 1783 (2001)]; 2 u_c is the expanded uncertainty.							

Table 9 Electrical conductivity of 0.001 mol/kg potassium chloride standard solution

Concentration of potassium chloride standard solution mol/kg	Temperature °C	Electrical conductivity mS/m
0.001	25	14.65
NOTE The conductivity values are calculated from the data of Shedlovsky and the density data, and expressed in four digits [<i>J. Am. Chem. Soc.</i> , 54 , 1411 (1932)].		

13.3 Implements and apparatus

- a) **Conductance meter**, composed of a detector and an indicator. The detector shall consist of a cell having a built-in platinum electrode coated with platinum black, and having the cell constant shown in Table 10. The indicator shall incorporate a Wheatstone bridge circuit or the like. The cell shall be stored in water. The cell constant shall be periodically checked according to the method shown in **13.4.1**.

As required, the electrode shall be given a platinum black coating according to

the following.

- 1) Electrolyze the electrode in hydrochloric acid (1+1) using the platinum black electrode as an anode in order to remove platinum black from the electrode.
 - 2) Put this platinum electrode in the electrolyte [**13.2 g**], and apply a current with the d.c. voltage of about 6 V and the current density of 100 A/m² to 400 A/m² for about 10 min (60 kC/m² to 240 kC/m²) by changing the polarity several times while agitating the electrolyte by a suitable means.
 - 3) Then, put it in sulfuric acid (1+360) and apply the current for about 30 min while changing the direction of current occasionally to remove hexachloroplatinic (IV) acid and chlorine adhered to or occluded on the electrode.
- b) **Thermometer**, in accordance with **6.3.1 a)**, with a permissible error of within ± 0.5 °C.
- c) **Thermostatic bath**, capable of controlling to 25 °C $\pm$ 0.5 °C.

13.4 Procedure

13.4.1 Measurement of cell constant

The cell constant need not be measured after each testing of sample, but shall be checked at regular intervals by using the potassium chloride standard solutions given in Table 8 according to the following procedure.

- a) Wash the cell with water two or three times, and then with a potassium chloride standard solution (corresponding to the measuring range for the relevant cell constant as stated in Table 10) two or three times, and fill it with the same potassium chloride standard solution. Measure the electric conductance while keeping this cell at 25 °C $\pm$ 0.5 °C. Repeat the measurement several times, each time using a new potassium chloride standard solution, until three consecutive measurements agree within $\pm 3\%$. Where the measurements do not agree within $\pm 3\%$, perform a further plating of platinum black on the platinum black electrode according to **13.3 a)**, and then perform the above procedure again.
- b) Calculate the cell constant from the average of the three measured values according to the following formula :

$$J = \frac{\kappa_{\text{KCl}} + \kappa_{\text{H}_2\text{O}}}{L_{\text{XO}}}$$

where, J : cell constant (m⁻¹)

L_{XO} : measured electric conductance (mS)

κ_{KCl} : electrical conductivity of the employed potassium chloride standard solution at this temperature (mS/m)

$\kappa_{\text{H}_2\text{O}}$: electrical conductivity of water used for preparation of potassium chloride standard solution at this temperature (mS/m)

- c) Measure the cell constant by using other two types of potassium chloride standard solutions whose concentrations as shown in Table 8 are closer to each other than

the solutions used for measurement in a). If their values do not agree within ± 1 %, perform a further plating of platinum black according to 13.3 a), and then perform the measurement again.

13.4.2 Electrical conductivity measurement of sample

The electrical conductivity of the sample shall be measured as follows. When the accuracy of electrical conductivity is not necessary, the method described in 13.4.3 may be used.

- a) Turn on the power of the conductance meter in advance. Select a cell having a cell constant corresponding to the electrical conductivity of the sample as shown in Table 10 and wash it two or three times with water. When the contamination is serious, immerse the cell in hydrochloric acid (1+100), rinse thoroughly with running water, and finally wash two or three times with water.

Table 10 Cell constant and measuring range

Classification	Cell constant m^{-1}	Measuring range mS/m
		Material of detector (platinum black)
a.c. two-terminal system	0.1 to 1	0.005 to 100
	1 to 10	0.005 to 1 000
	10 to 100	0 to 10 000
	100 to 1 000	0 to 100 000
	1000 to 5 000	0 to 500 000

- b) Wash this cell two or three times with the sample, fill it with the sample, and measure the electric conductance of the sample while keeping the temperature at $25\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$. Repeat this measurement, each time using a new portion of sample, until three consecutive measurements agree within ± 3 %, and calculate the mean of the three measurements to determine the electric conductance. In the case of a sample with an electrical conductivity of less than 1 mS/m ($25\text{ }^{\circ}\text{C}$), it may be difficult to obtain three measurements agreeing within ± 3 % by this method. In this case, either use the test method specified in **JIS K 0552** or use a flow-through type cell.
- c) From the obtained electric conductance, calculate the electrical conductivity (mS/m) ($25\text{ }^{\circ}\text{C}$) of the sample according to the following formula :

$$\kappa = J \times L_x$$

where, κ : electrical conductivity of the sample (mS/m) ($25\text{ }^{\circ}\text{C}$)

J : cell constant (m^{-1})

L_x : measured electric conductance (mS)

- d) If the conductance meter indicates electric resistance, calculate the electrical conductivity (mS/m) of the sample according to the following formula :

$$\kappa = \frac{J}{R_x} \times 10^3$$

- where,
- κ : electrical conductivity of the sample (mS/m) (25 °C)
 - J : cell constant (m⁻¹). This value is 1 when the conductance meter directly indicates electric resistivity (Ω·m), in which case the value of electric resistivity is entered for R_x .
 - R_x : measured electric resistance (Ω)

13.4.3 Electrical conductivity measurement of sample (outside the temperature range of 25 °C ± 0.5 °C)

When measuring the sample outside the temperature range of 25 °C ± 0.5 °C, use a conductance meter incorporating a temperature compensating circuit, or convert the measured electrical conductivity to a value at 25 °C using the following temperature conversion formula. If the conductance meter indicates electric resistance, calculate the electrical conductivity of the sample according to **13.4.2 d)**. Electrical conductivity is affected by the temperature, such that it increases about 2 % per 1 °C rise in temperature. Note that the following conversion formula cannot be applied where the electrical conductivity is 1 mS/m or lower, since this produces a greater influence of hydrogen ion and hydroxide ion due to dissociation of water.

$$\kappa_{25} = \frac{\kappa_{\theta}}{1 + (\alpha / 100)(\theta - 25)}$$

- where,
- κ_{25} : electrical conductivity at 25 °C (mS/m)
 - κ_{θ} : electrical conductivity at measurement temperature θ (mS/m)
 - α : temperature coefficient of electrical conductivity (%)
 - θ : temperature of sample at the time of measurement (°C)

The temperature coefficient of electrical conductivity (change rate of electrical conductivity per 1 °C), α , can be obtained from the following formula according to an experimental measurement :

$$\alpha_{\theta,25} = \frac{1}{\kappa_{25}} \times \left(\frac{\kappa_{\theta} - \kappa_{25}}{\theta - 25} \right) \times 100$$

- where,
- $\alpha_{\theta,25}$: temperature coefficient of electrical conductivity for converting κ_{θ} to κ_{25} (%)
 - κ_{25} : electrical conductivity at 25 °C (mS/m)
 - κ_{θ} : electrical conductivity at measurement temperature θ (mS/m)
 - θ : temperature of sample at the time of measurement (°C)

NOTE See Annex B for examples of temperature correction factors for conversion of electrical conductivity.

14 Suspended matter and evaporation residue

14.1 General

The substances suspended in water and the residues after evaporation of water are classified into suspended matter, total evaporation residue, soluble evaporation residue,

ignition residue and ignition loss, and tested according to respective test methods.

This test shall be carried out immediately after sampling. When this is not possible, the sample shall be stored in a dark place at 0 °C to 10 °C and tested as soon as possible.

14.2 Suspended matter

14.2.1 Summary

The residue remaining on the filtering material after filtering of the sample is dried at 105 °C to 110 °C, then weighed.

14.2.2 Implements

a) **Filter (separate type)**, as exemplified in Figure 10.

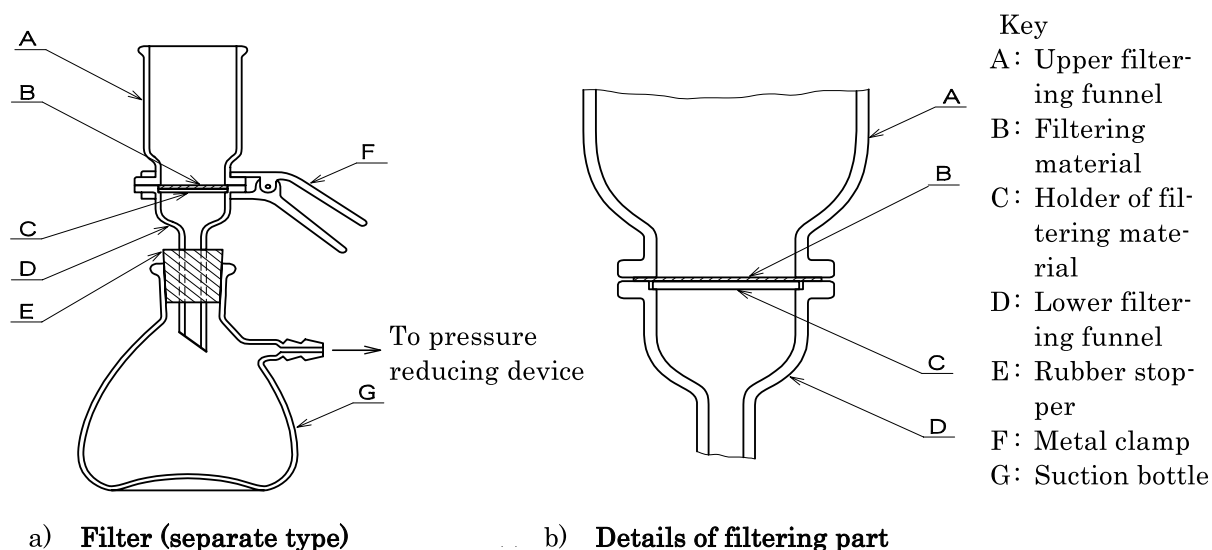


Figure 10 Example of filter (separate type)

- b) **Filtering materials** Glass-fibre filter paper, organic membrane filter, or metallic membrane filter, with a pore diameter of 1 µm and diameter of 25 mm to 50 mm. A glass-fibre filter paper seldom clogs, but may have flaking off of its fibre. Organic membrane filters can vary in chemical and thermal resistance and should be used with due care.
- c) **Watch glass** A light-weight watch glass or a container made of a light material such as aluminium foil.

14.2.3 Procedure

- a) For the mass measurement of the filtering material, use one of the following methods suitable for the filtering material used.
- In the case of using a glass-fibre filter paper, prior to use, attach it on a filter, and wash sufficiently with water using suction. Place this filtering material on a

watch glass, heat it at 105 °C to 110 °C for about 1 h, and after allowing to cool in a desiccator, weigh its mass.

- In the case of using a glass-fibre filter paper having been binder-treated with synthetic resin, place the filtering material on a watch glass without washing with water, heat at 105 °C to 110 °C for about 1 h, allow to cool in a desiccator, and weigh the mass.
 - For determination of ignition residue in suspended matter, use an organic membrane filter. In this case, place the filtering material on a watch glass without washing with water, heat at 105 °C to 110 °C for about 1 h, allow to cool in a desiccator, and weigh the mass. If the organic membrane filter deforms when heated at 105 °C to 110 °C, heat it at 90 °C.
 - In the case of using a metallic membrane filter, place the filtering material on a watch glass without washing with water, heat at 105 °C to 110 °C for about 1 h, allow to cool in a desiccator, and weigh the mass.
- b) Attach the filtering material on the filter after weighing. Pass through a sieve of 2 mm aperture an amount of sample that will give 2 mg or more of suspended matter after drying, shake well to disperse the suspended matter uniformly, quickly pour into the filter and filter using suction. Rinse down the substances adhering to the wall of the sample container and the filtering funnel with water and join them to the residue on the filtering material. Wash this several times with water.

When there is difficulty in filtering the sample, take a suitable amount of sample in a beaker, and pour it into the filter in divided amounts, while shaking well before each addition, such that each addition is made just before the last amount has drained through the filter. Stop the addition when the filtering speed has significantly lowered, and determine the amount of sample from the amount left in the beaker. When the sample contains suspended matter which may adhere to the wall of the sample container, collect a suitable amount of the sample, and employ the whole amount for the test. Scrape off the suspended matter adhering to the wall of the sample container using a policeman (a glass rod with rubber tube) or the like and gather it on the filtering material.

In the case of a sample containing oils and fats, grease, wax or the like, in order to remove these substances, filter the sample, dry the filtering funnel without removing the filtering material, attach it back to the suction bottle and pour 10 ml of hexane specified in **JIS K 8848** several times.

- c) Carefully remove the residue from the filter together with the filtering material using tweezers or the like and transfer it onto the watch glass used in **a)**. Heat it at 105 °C to 110 °C for 2 h, and after allowing to cool in the desiccator in **a)**, weigh its mass.
- d) Calculate the suspended matter (mg/L) according to the following formula :

$$S_1 = (m_1 - m_2) \times \frac{1\,000}{V_{s1}}$$

where, S_1 : suspended matter (mg/L)
 m_1 : mass of filtering material containing suspended matter and watch glass (mg)
 m_2 : mass of filtering material and watch glass (mg)
 V_{s1} : volume of sample (ml)

14.3 Total evaporation residue

14.3.1 Summary

The residue obtained after heating the sample to dryness on an evaporating dish is weighed.

14.3.2 Implements

a) **Evaporating dish**, made of platinum, quartz glass or porcelain.

14.3.3 Procedure

- a) Select an evaporating dish of a material suitable for the properties of the sample being tested. A platinum dish shall not be used for samples containing both chloride ion and strong oxidative material. Heat the evaporating dish at 105 °C to 110 °C for about 1 h, allow to cool in a desiccator, and weigh its mass. Repeat this procedure until constant mass is reached.
- b) On this evaporating dish, place an amount of sample that will give 5 mg or more of total evaporation residue after drying, and evaporate to dryness, without boiling, using a heating plate, boiling water bath, infrared lamp or the like while taking care to prevent contamination from outside. When the whole amount of sample cannot be put on the evaporating dish at once, it may be split into several portions.

When the sample contains total evaporation residue which may adhere to the wall of the sample container, take a suitable amount of the sample, and employ the whole amount for the test. Scrape off the total evaporation residue adhering to the wall of the sample container using a policeman (a glass rod with rubber tube) or the like to gather it on the evaporating dish.

- c) Heat the evaporating dish at 105 °C to 110 °C for about 2 h, allow to cool in a desiccator in a), and weigh its mass. For successively determining the ignition residue, use an evaporating dish which has been previously brought to constant mass by heating at 600 °C ± 25 °C.
- d) Calculate the total evaporation residue (mg/L) according to the following formula :

$$R_t = (m_3 - m_4) \times \frac{1000}{V_{s2}}$$

where, R_t : total evaporation residue (mg/L)
 m_3 : mass of evaporating dish containing the residue (mg)
 m_4 : mass of evaporating dish (mg)
 V_{s2} : volume of sample (ml)

14.4 Soluble evaporation residue

14.4.1 Summary

The residue obtained after filtering the sample and heating the filtrate to dryness on an evaporating dish is weighed.

14.4.2 Implements

- a) **Evaporating dish**, in accordance with 14.3.2 a).
- b) **Filter**, in accordance with 14.2.2 a).
- c) **Filtration material**, in accordance with 14.2.2 b).

14.4.3 Procedure

- a) Attach the filtration material on the filter, and filter the sample. For determining soluble evaporation residue in suspended matter, use an organic membrane filter.
- b) Treat the filtrate according to a) to c) of 14.3.3.
- c) Calculate the soluble evaporation residue (mg/L) according to the following formula :

$$R_s = (m_5 - m_6) \times \frac{1\,000}{V_{s3}}$$

where, R_s : soluble evaporation residue (mg/L)
 m_5 : mass of evaporating dish containing the residue (mg)
 m_6 : mass of evaporating dish (mg)
 V_{s3} : volume of sample (ml)

14.5 Ignition residue

14.5.1 Ignition residue of suspended matter

14.5.1.1 Summary

The suspended matter obtained in 14.2 is ignited at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ and then weighed.

14.5.1.2 Reagents

- a) **Ammonium nitrate solution (250 g/L)** Dissolve 25 g of ammonium nitrate specified in JIS K 8545 in water to make total 100 ml.

14.5.1.3 Implements and apparatus

- a) **Crucible**, made of platinum or porcelain, 10 ml to 20 ml in capacity.
- b) **Electric furnace**, controllable to $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$.
- c) **Filtration material** An organic membrane filter

14.5.1.4 Procedure

- a) Transfer the suspended matter obtained in 14.2.3 c), together with the filtration material, to a crucible previously brought to constant mass by heating at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. After moistening it by dripping ammonium nitrate solution (250 g/L), place

it in an electric furnace. Gradually increase the temperature of the furnace and heat it at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ for about 30 min to ashes. Allow to cool in a desiccator and weigh the mass.

Where the organic membrane filter is made of nitro compound, moisten by dripping 2-propanol specified in **JIS K 8839**, and then ignite to ashes.

- b) Calculate the ignition residue (mg/L) according to the following formula :

$$R_{i1} = (m_7 - m_8) \times \frac{1000}{V_{s1}}$$

where, R_{i1} : ignition residue of suspended matter (mg/L)
 m_7 : mass of crucible containing the ignition residue (mg)
 m_8 : mass of crucible (mg)
 V_{s1} : volume of sample (ml)

14.5.2 Ignition residue of total evaporation residue

14.5.2.1 Summary

The total evaporation residue obtained in **14.3** is ignited at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ and then weighed.

14.5.2.2 Reagent

In accordance with **14.5.1.2**.

14.5.2.3 Implements and apparatus

In accordance with **14.5.1.3**.

14.5.2.4 Procedure

- a) Perform the procedure in **14.5.1.4 a)** on the total evaporation residue obtained in **14.3.3 c)**.
- b) Calculate the ignition residue of total evaporation residue (mg/L) according to the following formula :

$$R_{i2} = (m_9 - m_{10}) \times \frac{1000}{V_{s2}}$$

where, R_{i2} : ignition residue of total evaporation residue (mg/L)
 m_9 : mass of evaporating dish containing the residue (mg)
 m_{10} : mass of evaporating dish (mg)
 V_{s2} : volume of sample (ml)

14.5.3 Ignition residue of soluble evaporation residue

14.5.3.1 Summary

The soluble evaporation residue obtained in **14.4** is ignited at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ and then weighed.

14.5.3.2 Reagent

In accordance with **14.5.1.2.**

14.5.3.3 Implements and apparatus

In accordance with **14.5.1.3.**

14.5.3.4 Procedure

- a) Perform the procedure in **14.5.1.4 a)** on the soluble evaporation residue obtained in **14.4.3 b)**.
- b) Calculate the ignition residue of soluble residue (mg/L) according to the following formula :

$$R_{i3} = (m_{11} - m_{12}) \times \frac{1000}{V_{s3}}$$

where,

- R_{i3} : ignition residue of soluble residue (mg/L)
- m_{11} : mass of evaporating dish containing the residue (mg)
- m_{12} : mass of evaporating dish (mg)
- V_{s3} : volume of sample (ml)

14.6 Ignition loss

14.6.1 Summary

The loss in mass when the suspended matter obtained in **14.2**, total evaporation residue obtained in **14.3** and soluble evaporation residue obtained in **14.4** are each ignited at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ is measured and expressed as the ignition loss of each.

14.6.2 Procedure

The ignition losses of suspended matter, total evaporation residue and soluble residue are respectively calculated according to formulas given in **a)**, **b)** and **c)** below.

a) Ignition loss of suspended matter

$$E_{sus} = S - R_{i1}$$

where,

- E_{sus} : ignition loss of suspended matter (mg/L)
- S : suspended matter (mg/L)
- R_{i1} : ignition residue of suspended matter (mg/L)

b) Ignition loss of total evaporation residue

$$E_t = R_t - R_{i2}$$

where,

- E_t : ignition loss of total evaporation residue (mg/L)
- R_t : total evaporation residue (mg/L)
- R_{i2} : ignition residue of total evaporation residue (mg/L)

c) Ignition loss of soluble residue

$$E_{so} = R_s - R_{i3}$$

where,

- E_{so} : ignition loss of soluble residue (mg/L)
- R_s : soluble residue (mg/L)

R_{i3} : ignition residue of soluble residue (mg/L)

15 Acid consumption

15.1 General

Acid consumption is the amount of hydrogen ion (amount of acid) required to neutralize the alkalis dissolved in water such as hydrogen carbonate, carbonate, or hydroxide to a specified pH. It is expressed either by the amount of hydrogen ion in mmol(s) per 1 L of sample or by the amount of calcium carbonate in mg(s) per 1 L of sample converted from the amount of hydrogen ion (acid).

Test items of acid consumption and their applications are shown in Table 11.

Table 11 Test items of acid consumption and their applications

Test item	Applicable to	Test clause
Acid consumption (pH 4.8)	Industrial water	15.2.1
	Industrial wastewater	15.2.2
Acid consumption (pH 8.3)	Industrial water	15.3.1
	Industrial wastewater	15.3.2

15.2 Acid consumption (pH 4.8)

15.2.1 Acid consumption (pH 4.8) of industrial water

15.2.1.1 Summary

The sample is titrated with 10 mmol/L sulfuric acid using methyl red-bromocresol green mixed solution as an indicator.

15.2.1.2 Reagents

- Ethanol (95)**, specified in **JIS K 8102**.
- Methyl red-bromocresol green mixed solution** Dissolve 0.02 g of methyl red specified in **JIS K 8896** and 0.10 g of bromocresol green specified in **JIS K 8840** in 100 ml of ethanol (95).
- 50 mmol/L sulfuric acid** Add 3 ml of sulfuric acid specified in **JIS K 8951** in a beaker containing 100 ml of water, stir well, allow to cool, and add water to make 1 000 ml. Standardization of this solution shall be as follows.
 - Heat sodium carbonate of reference material for volumetric analysis specified in **JIS K 8005** at 600 °C for about 1 h, and allow to cool in a desiccator. Weigh 1.06 g of this to the nearest 1 mg, dissolve in water, transfer to a 200 ml volumetric flask, and add water to the mark.
 - Take 20 ml of this solution in a beaker, add three to five drops of methyl red-bromocresol green mixed solution as an indicator, and titrate with the above 50 mmol/L sulfuric acid. When the solution turns greyish violet, boil to expel carbon dioxide, allow to cool, and continue titration until the solution turns greyish violet.

Calculate the factor (f_1) of 50 mmol/L sulfuric acid from the volume in ml(s) of 50 mmol/L sulfuric acid required for this titration according to the following formula :

$$f_1 = m_1 \times \frac{A}{100} \times \frac{20}{200} \times \frac{1}{x_1 \times 0.005\ 30}$$

where, m_1 : mass of sodium carbonate (g)
 A : purity of sodium carbonate (mass fraction %)
 x_1 : volume of 50 mmol/L sulfuric acid solution required for titration (ml)
 0.005 30 : mass of sodium carbonate corresponding to 1 ml of 50 mmol/L sulfuric acid (g/ml)

- d) **10 mmol/L sulfuric acid** Take 200 ml of 50 mmol/L sulfuric acid in a 1 000 ml volumetric flask, and add water to the mark. Use the factor of 50 mmol/L sulfuric acid obtained in **15.2.1.2 c) 2)**.
- e) **Sodium thiosulfate solution (0.1 mol/L)** Dissolve 2.6 g of sodium thiosulfate pentahydrate specified in **JIS K 8637** in water to make 100 ml.

15.2.1.3 Implements and apparatus

- a) **pH meter**, in accordance with **12.3 a)**.
- b) **Magnetic stirrer**, not affecting the liquid temperature by the heat produced by its rotation.

15.2.1.4 Procedure

- a) Place 100 ml of sample (if turbid, filter through a Class 5 B filter paper, or centrifuge and use the supernatant) in a beaker, and add three to five drops of methyl red-bromocresol green mixed solution as an indicator. If the sample contains oxidizing substances such as residual chlorine, reduce it with sodium thiosulfate solution (0.1 mol/L) before adding this mixed solution.
- b) While stirring slowly with a magnetic stirrer, titrate the solution with 10 mmol/L sulfuric acid until it turns from blue to greyish violet (pH 4.8). If the colour change by the indicator is not clear due to the original colour of the sample or for other reasons, instead of using an indicator, prepare a titration curve between the sample pH and the volume of 10 mmol/L sulfuric acid by using a pH meter and determine the volume in ml(s) of 10 mmol/L sulfuric acid at pH 4.8 from the titration curve.
- c) Calculate the acid consumption (pH 4.8) according to the following formula :
- 1) For expressing by hydrogen ion concentration

$$C_1 = x_2 \times f_1 \times 0.02 \times \frac{1000}{V_{s1}}$$

where, C_1 : acid consumption (pH 4.8) [hydrogen ion concentration (mmol/L)]