

JAPANESE
INDUSTRIAL
STANDARD

JIS K 0101 : 1998

Testing methods for industrial water

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Translated

by

Japanese Standards Association

1-24, Akasaka 4, Minato-ku Tokyo 107-8440 Japan



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JAPANESE INDUSTRIAL STANDARD

JIS K 0101: 1998

Testing methods for industrial water

March, 2000

ERRATA

Page 261 line 11

Error: 20 ml of sulfuric acid(1+1)

Correct: 20 ml of sulfuric acid(1+10)

Remarks: This erratum is for correcting the first edition of this Standard.

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ERRATA(1/2)

Page 11, line 17

In Notes(7), replace “...and suspended matters” by “...or suspended matters”.

Page 17

In 8.2 (4)(c), V is changed as follows:

Error V : sample used for dilution (ml)

Correct V : sample used for the test (ml)

Page 32, line 4

The numerical value is replaced as follows:

Error $x = 0.310\ 0$

Correct $x = 0.310\ 1$

Page 59, line 9

Sentences in 16.1(2)(a) are changed as follows:

Error ...and wash sufficiently with water by suction. Thereafter, place this filter medium⁽¹⁾ on a watch...

Correct ...and wash sufficiently with water by suction⁽¹⁾. Thereafter, place this filter medium on a watch...

Page 68, line 19

In 18 (3)(c), replace “heat” by “boil”.

Page 94, line 6

Error *p*-hydrazinobenzene diazonium salt

Correct *p*-sulfo benzene diazonium salt

Remarks: This erratum is for correcting the first edition of this Standard.

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ERRATA(2/2)

Page 254, line 3

42.1(3)(a) is changed as follows:

Error Put 10 ml of sample (containing 2 to 50 μg as SO_4^{2-}) in a precipitation tube for a centrifuge, and keep it at 20 to 30 $^{\circ}\text{C}^{(1)}$. Add 4 ml...

Correct Add water into appropriate sample (containing 2 μg to 50 μg as SO_4^{2-}) in a precipitation tube for a centrifuge to make total 10 ml, and keep it at 20 $^{\circ}\text{C}$ to 30 $^{\circ}\text{C}^{(1)}$. Add 4 ml...

Page 255, line 10

42.2(3)(a) is changed as follows:

Error Put 10 ml of sample (containing 50 to 500 μg as SO_4^{2-}) in a precipitation tube for a centrifuge, and keep it at 20 to 30 $^{\circ}\text{C}^{(1)}$. Add 4 ml...

Correct Add water into appropriate sample (containing 50 μg to 500 μg as SO_4^{2-}) in a precipitation tube for a centrifuge to make total 10 ml, and keep it at 20 $^{\circ}\text{C}$ to 30 $^{\circ}\text{C}^{(1)}$. Add 4 ml...

Page 268, line 1

Error chloride ion

Correct bromide ion

Page 268, line 4

Error chlorine

Correct bromine

Page 453

In clause 3, replace " Informative reference VI " by " Informative reference IV".

Remarks: This erratum is for correcting the first edition of this Standard.

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Testing methods for industrial water

1 Scope This Japanese Industrial Standard specifies the testing methods for industrial water.

Remarks : Normative references to this Standard are shown in Attached Table 1.

2 Common items The common items shall be as follows:

- (1) **General rule** The general items common to the chemical analysis shall be in accordance with **JIS K 0050**.
- (2) **Definitions** For the purposes of this Standard, the definitions in **JIS K 0102**, **JIS K 0211** or **JIS K 0215** apply.

In addition, the inductively coupled plasma mass spectrometry is hereafter referred to as "ICP mass spectrometry".

- (3) **Gas chromatography** The general items common to the gas chromatography shall be in accordance with **JIS K 0114**.
- (4) **Absorptiometry** The general items common to the absorptiometry shall be in accordance with **JIS K 0115**.
- (5) **Inductively coupled plasma atomic emission spectrometry** The general items common to inductively coupled plasma atomic emission spectrometry (hereafter referred to as "ICP atomic emission spectrometry") shall be in accordance with **JIS K 0116**.
- (6) **Infrared spectrophotometry** The general items common to the infrared spectrophotometry shall be in accordance with **JIS K 0117**.
- (7) **Atomic absorption method** There are a flame absorption method, an electric heating system atomic absorption method (hereafter referred to as "electric heating atomic absorption method") and other atomic absorption methods. The general items common thereto shall be in accordance with **JIS K 0121**.
- (8) **Ion-selective electrode method** The general items common to the ion-selective electrode method shall be in accordance with **JIS K 0122**.
- (9) **Ion chromatography** The general items common to the ion chromatography shall be in accordance with **JIS K 0127**.
- (10) **Determination range** The determination ranges indicated in respective test methods are expressed by the mass (mg, μ g or ng) in the final solution. However, in the atomic absorption method, flame emission photometry, ICP atomic emission spectrometry, ICP mass spectrometry, ion chromatography, ion-selective electrode method, and testing methods of total organic carbon (TOC), total oxygen demand (TOD), dissolved oxygen and residual chlorine, the determination range is expressed by the concentration (mg/l or μ g/l) of the final solution.

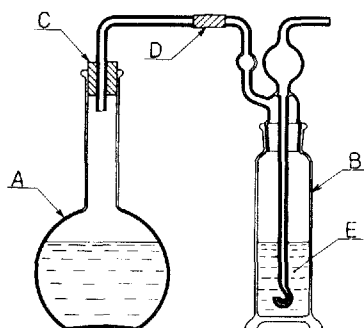
- (11) **Repeatability** The repeatability for the standard solution shall be indicated by coefficient of variation (%)⁽¹⁾ obtained by repetitive tests within the determination range of respective testing methods.

Note (1) Coefficient of variation (%) = $\frac{\sigma}{\bar{x}} \times 100$

where, σ : standard deviation
 $\bar{x}$: average

- (12) **Water** The water to be used in this Standard shall be water A1 to A4 specified in **JIS K 0557**, however, where it is specified in relevant items, the water shall be in accordance therewith.

- (a) **Dissolved-oxygen-free water** Transfer water A2 or A3 specified in **JIS K 0557** into a flask, boil for about 5 min to remove the dissolved oxygen, then connect the gas washing bottle containing alkaline pyrogallol solution⁽²⁾ as in Fig. 2.1, and allow to cool while shielding from oxygen in the atmosphere. Otherwise, allow to remove dissolved oxygen by blowing high purity nitrogen grade 2 specified in **JIS K 1107** for approx. 15 min instead of boiling.



- A: 1 000 ml Flat bottom flask
B: 250 ml Gas-washing bottle
C: Rubber stopper
D: Rubber tube
E: Alkaline pyrogallol solution

Fig. 2.1 An example for cooling and preservation of dissolved-oxygen-free water

- (b) **Carbonic acid-free water** Transfer water A2 or A3 specified in **JIS K 0557** into a flask. After removing dissolved gas and carbon dioxide by boiling for approx. 5 min, use the same apparatus as that in Fig. 2.1, put potassium hydroxide solution (250 g/l) into a gas washing bottle, shield from carbon dioxide in the atmosphere, and allow to cool.

Note (2) Dissolve 6 g of pyrogallol (1,2,3-benzenetriol) specified in **JIS K 8780** in 50 ml of water, and reserve in a coloured bottle. Separately, dissolve 30 g of potassium hydroxide specified in **JIS K 8574** in 50 ml of water. Mix both liquids in use. 1 ml of this solution absorbs approx. 12 ml of oxygen (approx. 17 mg).

(13) **Reagents**

- (a) In the case where an item is designated, JIS marked reagent of the highest quality shall be used. Where JIS marked reagent does not exist, reagents without impairing the test shall be used⁽³⁾.

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

- (b) The concentration of solution of reagents shall be indicated g/l or mg/l for mass concentration and mol/l or mmol/l for mol concentration unless otherwise specified.

For compounds, masses as anhydride are used.

The concentration of standard solution shall be indicated mass in 1 ml (mg/ml or µg/ml) except ion-selective electrode method and flame emission photometry.

- (c) The concentration indicated in parentheses after the name of the solution means the approximate concentration except standard solutions. For example, the sodium hydroxide solution (0.1 mol/l) means the concentration of sodium hydroxide solution of approx. 0.1 mol/l.

The concentration indicated before the name of solution means the correct concentration. In general, it shall be indicated with numerical value without fraction, and the factor be separately obtained.

- (d) The water to be used for preparation of reagents shall be the water specified in (12), however, where it is specified in respective items, the water shall be in accordance with specifications of the item.
- (e) When preparing the standard solution of low concentration by diluting the standard solution, use a 10 ml or over transfer pipette unless otherwise specified.
- (f) The names of reagents shall be, as a rule, conformed to the compound nomenclature determined by The Chemical Society of Japan in accordance with the inorganic chemical nomenclature and organic chemical nomenclature of International Union of Pure and Applied Chemistry (IUPAC) and the names of JIS reagents.
- (g) For handling of reagents, waste solution, etc. sufficient cares shall be taken following laws and regulations relating thereto.

Note (3) For the test using the reagent of very small quantity in such determination method as electric heating atomic absorption method, ICP mass spectrometry, etc., specially high pure reagents shall be used.

- (14) **Appliances** Glassware, porcelain crucible, porcelain evaporating dish and filter paper used in this Standard are as follows.

- (a) Glassware specified in **JIS R 3503** and **JIS R 3505** shall be used. If special appliances are required, they are exemplified or explained in respective items.

For heating procedures, borosilicate glass-1 specified in **JIS R 3503** shall be used.

Desiccant used in a desiccator shall be silica gel⁽⁴⁾ unless otherwise specified.

- (b) Porcelain crucible and porcelain evaporating dish specified in **JIS R 1301** and **JIS R 1302** shall be used.
- (c) Filter paper for quantitative analysis specified in **JIS P 3801** shall be used. The type of filter papers shall be specified in respective items.

Note (4) Packaging silica gel desiccant type A grade 1 specified in **JIS Z 0701** shall be used.

Remarks : In the case where silica, boron, sodium, potassium, arsenic, zinc, etc. are tested, cares shall be taken sufficiently on the elution of these components from borosilicate.

- (15) **Measurement of absorbance (Absorptiometry)** If not specified on absorption cell, the absorption cell of 10 mm in optical path length shall be used.
- (16) **Working curve [absorptiometry, atomic absorption method, flame emission photometry, ICP atomic emission spectrometry, ICP mass spectrometry, ion chromatography, ion-selective electrode method, total organic carbon (TOC), and total oxygen demand (TOD)]** When a working curve is prepared, divide the determination range given by a test method into 4 to 6 stages, and take standard solutions so as to conform thereto. Prepare the working curve within the determination range.

In the tests for atomic absorption method, flame emission photometry, ICP atomic emission spectrometry, ICP mass spectrometry, ion chromatography, ion-selective electrode method, total organic carbon (TOC), and total oxygen demand (TOD), use the working curve newly prepared in testing. When the same item is continuously tested on many samples, use appropriately standard solution halfway in testing, and confirm an indication value.

In absorptiometry, allow the preliminarily prepared working curve to be used.

- (17) **Note, remarks, figure, table, and formula** For the note, remarks, figure, table, and formula, serial number is annexed to each clause.

3 Sample

3.1 Sampling, sample container, water sampler and water-sampling operation

The sample means the water sampled for carrying out the test. Sampling, sample container, water sampler and water-sampling operation shall be in accordance with JIS K 0094.

3.2 Handling of sample Test total quantity contained in a sample unless otherwise specified. Therefore, where suspended matters are contained in the sample, sufficiently mix by shaking to make the sample homogeneous. Thereafter, take the sample to be used for the test. However, for the test of an anion, use the filtrated sample unless otherwise specified. Where the total quantity is obtained, specify the handling in each item.

Besides, where only those in dissolved state are tested, immediately after taking the sample, filter with filter paper of grade 5C⁽¹⁾, discard approx. 50 ml of the initial filtrate, and take the filtrate thereafter as the sample.

Note (1) Filter paper grade 6 or the filter material with 1 μm or under in pore diameter may be used.

3.3 Preservation treatment of sample Perform a test immediately after sampling unless otherwise specified. In the case where the test can not be performed immediately and the sample is preserved, carry out the operation as follows in accordance with 7 (preservation treatment of sample) of JIS K 0094, and test as soon as possible. In the case where the sample is preserved in a cold place, do not freeze.

(1) **Reagents** The following reagents shall be used.

- (a) **Hydrochloric acid** As specified in JIS K 8180.
- (b) **Hydrochloric acid (for analysis of arsenic)** As specified in JIS K 8180.
- (c) **Nitric acid** As specified in JIS K 8541.
- (d) **Sulfuric acid** As specified in JIS K 8951.
- (e) **Phosphoric acid** As specified in JIS K 9005.
- (f) **L(+)-ascorbic acid** As specified in JIS K 9502.
- (g) **Sodium hydroxide solution (200 g/l)** Dissolve 20 g of sodium hydroxide specified in JIS K 8576 in water to make 100 ml.
- (h) **Basic zinc carbonate suspension** Dissolve 20 g of zinc sulfate heptahydrate specified in JIS K 8953 in water to be mixed with sodium carbonate solution (100 g/l) of the same volume as that. Prepare at service.
- (i) **Copper (II) sulfate pentahydrate** As specified in JIS K 8983.
- (j) **Chloroform** As specified in JIS K 8322.

(2) **Preservation treatment** Perform the preservation treatment as follows.

- (a) Preserve samples to be used for the tests of oxygen demand by potassium permanganate (COD_{Mn}) at 100 °C, oxygen demand by potassium dichromate (COD_{Cr}), biochemical oxygen demand (BOD), total organic carbon (TOC), total oxygen demand (TOD), and surface active agent in a dark place at 0 to 10 °C.
- (b) Add hydrochloric acid or sulfuric acid to the samples used for the tests of ammonium ion, organic nitrogen, and total nitrogen, regulate pH at 2 to 3, and preserve in a dark place at 0 to 10 °C. For a short number of days, allow the sample to be preserved in a dark place at 0 to 10 °C as it is without preservation treatment.
- (c) Add about 5 ml of chloroform to the sample to be used for the test of nitrite ion and nitrate ion per 1 l of the sample, and preserve in a dark place at 0 to 10 °C. For a short number of days, allow the sample as it is to be preserved in a dark place at 0 to 10 °C without preservation treatment.
- (d) Add sodium hydroxide solution (200 g/l) to the sample to be used for the tests of iodide ion, and bromide ion to make pH approx. 10 and preserve (allow 2 to 4 grains of sodium hydroxide per 1 l of the sample to be added).
- (e) Add sodium hydroxide solution (200 g/l) to the sample to be used for the test of cyanide compound and sulfide ion to make pH approx. 12, and preserve (allow 4 to 6 grains of sodium hydroxide per 1 l of the sample to be added). In the case where oxidizing matters such as residual chlorine or the like coexist with the sample to be used for the cyanide compound test, after reducing by adding L(+)-ascorbic acid, make pH approx. 12.

Further, for sulfide ion, take a sample into a dissolved oxygen measuring bottle, add basic zinc carbonate suspension at a rate of approx. 2 ml per 100 ml of the sample to fix as zinc sulfide, and allow to be preserved.

- (f) Add phosphoric acid to the sample to be used for the phenols test to make pH approx. 4, add 1 g of copper (II) sulfate pentahydrate per 1 l of the sample, mix by shaking, and preserve in a dark place at 0 to 10 °C.
- (g) Perform preservation treatment for the sample to be used for the tests of phosphide compound and total phosphorus as follows. Add chloroform at a rate of approx. 5 ml per 1 l of a sample under a state as it is, and preserve in a dark place at 0 to 10 °C. In that case, for a short number of days, allow to be preserved in a dark place at 0 to 10 °C under a state as it is without preservation treatment. For the sample to be used for the test of dissolved phosphide compound, after filtration in accordance with 3.2, add chloroform at a rate of approx. 5 ml per 1 l of the sample, and preserve in a dark place at 0 to 10 °C. In the case, for a short number of days, allow to be preserved in a dark place at 0 to 10 °C under a state as it is without preservation treatment.

The sample to be used for total phosphorus may be preserved making the pH value about 2 by adding sulfuric acid or nitric acid.

- (h) Add nitric acid to the samples to be used for the tests of metallic elements such as copper, zinc, lead, cadmium, manganese, iron, aluminium, nickel, cobalt, arsenic, tin, total chromium, mercury, vanadium, etc. to make pH approx. 1, and preserve.

In the case where treatments with sulfuric acid and nitric acid, or nitric acid and potassium permanganate are not performed in testing for the sample to be used for testing arsenic and organic matters, a great quantity of nitrates, nitrites, etc. do not contained, add hydrochloric acid (for analysis of arsenic) to make pH approx. 1, and preserve.

Preserve the sample to be used for the test of chromium (VI) in a dark place at 0 to 10 °C under a state as it is.

For the sample to be used for the test of dissolved state metal elements, after filtering a sample in accordance with 3.2, add nitric acid to make pH approx. 1, and preserve.

4 Pretreatment of sample Though the pretreatment of a sample is specified in each test item, since the pretreatment operations in the test of metal element are almost common independently of the kind of metal elements, the operations are specified collectively hereafter. However, the pretreatments for the tests of sodium, potassium, calcium, magnesium, arsenic, chromium (VI), mercury, etc. among metal elements are specified in respective test items.

The pretreatments in the test of metal elements have the purpose mainly to decompose the coexisting organic substance, suspended matters and metal complex.

Though the method of heating after adding various acids to the sample is used, a suitable method is selected according to the state of the sample and the kind of test.

4.1 Boiling with hydrochloric acid or nitric acid This method applies to the sample containing an extremely small amount of organic substances and suspended matters.

(1) **Reagents** The following reagents shall be used.

(a) **Hydrochloric acid** As specified in **JIS K 8180**.

(b) **Nitric acid** As specified in **JIS K 8541**.

(2) **Operation** The operation shall be carried out as follows.

(a) Add hydrochloric acid or nitric acid at a rate of 5 ml per 100 ml of the sample⁽¹⁾.

(b) Boil for approx. 10 min by heating.

(c) After standing to cool, add water to make the specified amount, as required.

Note (1) In the case of testing dissolved metal elements, the sample filtered in accordance with 3.2 shall be used.

4.2 Decomposition with hydrochloric acid or nitric acid This method applies to the sample containing little organic substance and hydroxide, oxide, sulfide, phosphate, etc., as suspension.

(1) **Reagents** The following reagents shall be used.

(a) **Hydrochloric acid** As specified in **JIS K 8180**.

(b) **Nitric acid** As specified in **JIS K 8541**.

(2) **Operation** The operation shall be carried out as follows.

(a) After mixing the sample⁽²⁾ sufficiently by shaking, immediately take the suitable amount of sample in a beaker and add hydrochloric acid or nitric acid at a rate of 5 ml per 100 ml of the sample.

(b) Heat to concentrate the sample to approx. 15 ml.

(c) When the insoluble matters remain, filter the sample with filter paper of grade 5B, and then wash with water sufficiently.

(d) After standing to cool, transfer the filtrate and washings into a suitable capacity of volumetric flask, and dilute to the marked line with water.

Note (2) For testing the dissolved metal elements, the sample filtered in accordance with 3.2 shall be used, and the method of 4.1 be used.

Remarks : For the sample to which decomposition with mixed acid of hydrochloric acid and nitric acid is favorable, after performing the operation to (b), stand to cool to room temperature. When hydrochloric acid is used in (a), add 5 ml of nitric acid, when nitric acid is used, add 5 ml of hydrochloric acid, cover with a watch glass, and heat again. When violent reaction ends, remove the watch glass, expel nitrogen oxide by heating further, and concentrate to approx. 5 ml. In the case where acid is in short supply in this operation, add an appropriate quantity of hydrochloric acid and nitric acid, heat by the same operation, and dissolve. When insoluble matters remain, add 15 ml of warm water, and carry out the operations of (c) and (d).

4.3 Decomposition with nitric acid and perchloric acid This method applies to the sample containing organic matters that are difficult to be oxidized.

(1) **Reagents** The following reagents shall be used.

(a) **Perchloric acid** As specified in JIS K 8223.

(b) **Nitric acid** As specified in JIS K 8541.

(2) **Operation** The operation shall be carried out as follows.

- (a) Mix the sample⁽²⁾ by shaking thoroughly, immediately take a proper amount into a beaker or porcelain evaporating dish.
- (b) Add 5 to 10 ml of nitric acid, heat it on a heating plate gently to concentrate up to about 10 ml⁽³⁾, and stand to cool.
- (c) Add 5 ml of nitric acid and then add 10 ml of perchloric acid⁽⁴⁾ little by little. Continue heating. When the white fume of perchloric acid begins to generate, cover the container with a watch glass, and decompose organic substances while keeping the conditions in which the perchloric acid flows down along the wall of the container.
- (d) When organic substances remain not composed, further add 5 ml of nitric acid and repeat the operation specified in (c) until the organic substances decompose.
- (e) After standing to cool, dilute the solution with water to approx. 50 ml. When insoluble matters remain, filter the solution with filter paper of grade 5B, then wash with water. Transfer the filtrate and washings into a proper capacity of volumetric flask, and add water up to the marked line.

Notes (3) The sample may be transferred into a Kjeldahl flask to be decomposed.

(4) The decomposing procedure by heating using perchloric acid may be dangerous to explode in some kinds of samples, therefore pay attention to the following.

- i) For oxidizable organic substances, add nitric acid of (b) before adding perchloric acid, and decompose sufficiently by heating procedure.

- ii) When perchloric acid is added, the concentrated liquid shall be cooled beforehand without fail.
- iii) The decomposition by heating shall be carried out under the coexist state of perchloric acid with nitric acid without fail.
- iv) The concentrated solution shall be not made dryness.

4.4 Decomposition with nitric acid and sulfuric acid This method is applicable⁽⁵⁾ for various samples.

(1) **Reagents** The following reagents shall be used.

- (a) **Nitric acid** As specified in **JIS K 8541**.
- (b) **Sulfuric acid (1+1)** Take one volume of water into a beaker, cool it and add gradually one volume of sulfuric acid specified in **JIS K 8951** while stirring.

(2) **Operation** The operation shall be carried out as follows.

- (a) Mix the sample⁽²⁾ thoroughly by shaking, immediately take its proper amount into a beaker or porcelain evaporating dish and add 5 to 10 ml of nitric acid.
- (b) Heat to approx. 10 ml⁽³⁾ of solution, then add again 5 ml of nitric acid and 10 ml of sulfuric acid (1+1), and heat until the white fume of sulfuric acid generates to decompose organic substances.
- (c) When the decomposition of organic substances is difficult, add further 10 ml of nitric acid, and repeat the operation specified in (b) to decompose the organic substances.
- (d) After standing to cool, dilute the solution with water to approx. 50 ml. If insoluble substances⁽⁶⁾ remain, filter with filter paper of grade 5B, wash with water, then transfer the filtrate and washings into a proper capacity of volumetric flask, and add water up to the marked line.

Notes ⁽⁵⁾ In the case of applying the flame atomic absorption method in which the water solution is sprayed as it is, this method is not desirable.

- ⁽⁶⁾ Where lead is contained and precipitates are generated, carry out the operation specified in 4.3 or the following operation:

Carry out the operation specified in (b) to evaporate the solution to almost dryness, add approx. 30 ml of water and 15 ml of hydrochloric acid, and heat to dissolve. Where insoluble matters exist, filter with filter paper of grade 5B, then wash with warm hydrochloric acid (1+10). After standing to cool, transfer the filtrate and washings into a proper capacity of volumetric flask, and add water up to the marked line.

4.5 Pretreatment in flame atomic absorption method, electric heating atomic absorption method, ICP atomic emission spectrometry or ICP mass spectrometry The pretreatment shall be carried out by selecting the optimum method of the methods given in 4.1 to 4.4 taking into consideration the quantity of organic substances and suspensions contained in the sample, their existing state, the method of

atomic absorption method, ICP atomic emission spectrometry or ICP mass spectrometry to be applied, sufficiently⁽⁷⁾ ⁽⁸⁾.

In the case where the flame atomic absorption method or ICP atomic emission spectrometry in which the direct spraying of prepared sample is performed, the sample shall be of hydrochloric acid or nitric acid⁽⁹⁾. In the case of electric heating atomic absorption method or ICP mass spectrometry, it shall be of nitric acid, and the acid concentration shall be a suitable concentration⁽¹⁰⁾.

Notes ⁽⁷⁾ The pretreatment in the case where the solvent extraction method is applied prior to the flame atomic absorption method or ICP atomic emission spectrometry, unless otherwise designated, shall be as shown in the text, and the interfering organic substances and other materials which are likely to interfere shall be thoroughly decomposed.

In the case where the flame atomic absorption method or ICP atomic emission spectrometry is carried out by spraying the sample as it is, the following pretreatment may be carried out.

In the case of a sample containing an extremely small amount of organic substances ~~and~~ ^{or} suspended matters, carry out the operation specified in 4.1. As general method of pretreatment for the sample containing organic substances and suspended matters, apply the specifications of 4.3 or 4.4. In this case, expel the most part of sulfuric acid and perchloric acid by generating white fume sufficiently.

For ICP mass spectrometry, the blank test value cannot be ignored in some kinds and concentrations of acid, so that the effect of the kind and concentration of acid of elements shall be verified in advance.

It should be judged based on the results of the test which is carried out for recovery test by adding a specified amount of objective component to the sample, which pretreatment method is to be selected.

- ⁽⁸⁾ Refer to Note ⁽³⁾ of clause 2. For highly purified reagents, there are nitric acid, hydrochloric acid, perchloric acid and sulfuric acid specified in JIS K 9901, JIS K 9902, JIS K 9904 and JIS K 9905, respectively, and others.
- ⁽⁹⁾ In the case of an ICP atomic emission spectrometry, since when the sample is of sulfuric acid, the introduction amount of the sample is little and the sensitivity becomes occasionally worse, application of 4.4 is limited to an inevitable case.
- ⁽¹⁰⁾ In the cases of a flame atomic absorption method and an electric heating atomic absorption method, the concentration shall be 0.1 to 1 mol/l. In the case of an ICP atomic emission spectrometry, and tin (Sn) is not for object, the concentration shall be 0.1 to 0.5 mol/l. And tin (Sn) is for object, it shall be 1 to 1.5 mol/l. In the case of ICP mass spectrometry, it shall be 0.1 to 0.5 mol/l. However, in these cases, it shall be almost the same concentration as that in the case where a working curve is prepared.

5 Marking of results In the case where there are two or more test methods, it shall be described clearly which method is used.

6 Temperature Temperature is divided into atmospheric temperature and water temperature. Then, both the atmospheric temperature and the water temperature shall be measured at the time of sampling.

6.1 Atmospheric temperature The atmospheric temperature shall be measured according to the following:

(1) **Implement** The implement shall be as follows.

(a) **Thermometer** 50 °C thermometer of solid-stem general purpose liquid-in-glass thermometer specified in **JIS B 7411**.

(2) **Operation** The operation shall be carried out as follows.

(a) By use of the etched-stem liquid-in-glass thermometer, read out the scale when the temperature-sensitive liquid has stopped. The measurement shall be carried out at sampling site of good ventilation, avoiding direct rays of the sun and strong heat radiation from the surroundings, keeping at a position 1.2 to 1.5 m high above the ground.

Remarks 1 Use the maximum and minimum thermometer (see Fig. 6.1) for measuring the maximum and minimum temperature during the measuring period.

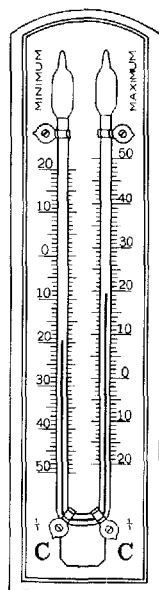


Fig. 6.1 An example of max. and min. thermometer

6.2 Water temperature The water temperature shall be measured according to the following:

(1) **Implement** The implement shall be as follows.

(a) **Thermometer** 50 or 100 °C thermometer of solid-stem general purpose liquid-in-glass thermometer specified in **JIS B 7411**.

(2) **Operation** The operation shall be carried out as follows.

- (a) Immersing the etched-stem liquid-in-glass thermometer directly into the water on site or into the sample⁽¹⁾ immediately after sampling, and while keeping the temperature-sensitive liquid under the surface of water, read out the scale when the temperature-sensitive liquid has stopped.

Note ⁽¹⁾ In order to avoid influences of the container and the atmospheric temperature, a great quantity of the sample shall be taken.

Remarks 2 In the case where Pettenkofer water thermometer (see Fig. 6.2) is used, after changing the sample in a metal cylinder three times, fill the cylinder with the sample, and read out the scale when the temperature-sensitive liquid has stopped.

- 3 For a thermistor thermometer and a metal resistance thermometer, keep the temperature detecting part in water to be measured, and read out the scale when the pointer of an indication part is stabilized at a constant value.

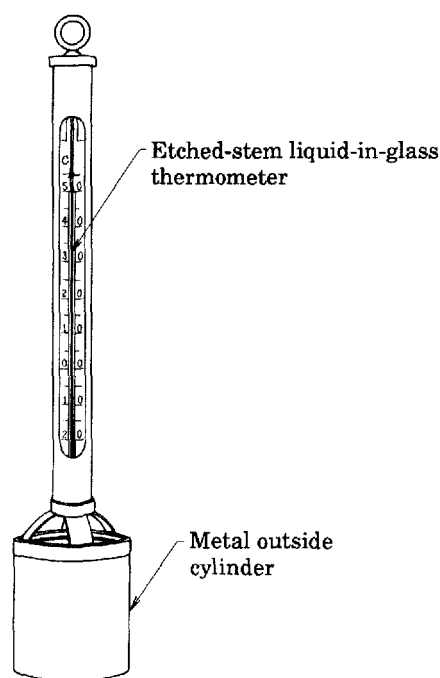


Fig. 6.2 Pettenkofer water thermometer

7 Appearance Observe the appearance of the sample immediately after sampling.

(1) **Implement** The implement shall be as follows.

(a) **Beaker** 300 to 500 ml (colourless)

(2) **Operation** Carry out the operation as follows.

(a) Take the sample immediately after sampling into a beaker, and observe the following items with the naked eye:

i) Kind of colour and its degree of the whole sample

ii) Kind of colour and its degree of supernatant water

iii) Kind of colour and degree of quantity of floating matters, suspended matters, etc.

iv) State and degree of oils, tars, etc.

v) Other particular state such as bubbles, odours, etc. of the sample

8 Odour and threshold odour number (TON) The test of odour shall be classified into the detection of odour and the threshold odour number (TON)⁽¹⁾.

The odours of water are caused by the effects of increase and extinction of bacteria, algae, micro-organisms, etc., of mixing-in of municipal sewage, cattle shed drainage, and industrial waste-water, of elution of inner surface treating substances of a water storage tank and piping system, and of residual chlorine due to chlorine treatment.

Because the test of odour is performed by the personal sense of smell, individual difference is large. The temperature and humidity, taking food and smoking of the inspector also affect the results.

Note (1) TON is the abbreviation of threshold odour number, which is the dilution multiple of odour threshold, i.e. the multiple value of dilution when odour is apparently sensed.

8.1 Odour The odour shall be tested for its kind and degree by warming the sample to approx. 40 °C.

(1) **Implement** The implement shall be as follows.

(a) **Erlenmeyer flask with ground stopper** 300 ml

(2) **Operation** Carry out the operation as follows.

- (a) Transfer 200 ml of the sample into a 300 ml Erlenmeyer flask with ground stopper, stopper lightly, and warm to approx. 40 °C.
- (b) Take off the stopper while shaking the flask, and immediately test for the existence of odour and its kind and degree.
- (c) The odour shall be indicated as shown in Table 8.1, so that the kind and degree of odour of the sample can be approximately understood.

Table 8.1 Example of classification and kind of odour

Classification	Kind of odour
(1) Fragrant odour	Odours of melon, violet, garlic, cucumber, aromatics, spices, etc.
(2) Botanical odour	Odours of algae, green grass, timber, seaweed, etc.
(3) Earthy odour, mouldy odour	Odours of soil, swamp, mould, etc.
(4) Fishery odour	Odours of fish, liver oil, clam, etc.
(5) Chemical odour	Odours of phenol, tar, oil, fats and oils, paraffin, chlorine, hydrogen sulfide, chlorophenol, pharmacy odour, chemicals odour, etc.
(6) Metallic odour	Odours of iron, metal, etc.
(7) Putrescent odour	Odours of garbage, sewage, pigpen, putrescence, etc.
(8) Unpleasant odour	Unpleasant odours such as strong odours of fish, pigpen, putrescence, etc.

Remarks 1 To minimize individual difference of the sense of smell, the same sample should be tested by several persons.

2 The odour should be tested at the time of sampling without warming, and the results are recorded. (This is called "odour at the time of cold".)

8.2 Threshold odour number (TON) The threshold odour number means the intensity of odour, and shall be expressed by multiple of dilution “dilution multiple of odour threshold” when the sample is added into water maintained at about 40 °C and the definitely perceptible odour is sensed. To minimize the individual difference of the sense of smell, the same sample shall be tested by at least 5 people, preferably by about 10 people.

(1) **Reagent** Use the following reagents.

- (a) **Odour-free water** Pass water A3 specified in **JIS K 0557** at a rate of 5 l/(l-active carbon · h) into the apparatus as shown in Fig. 8.1.

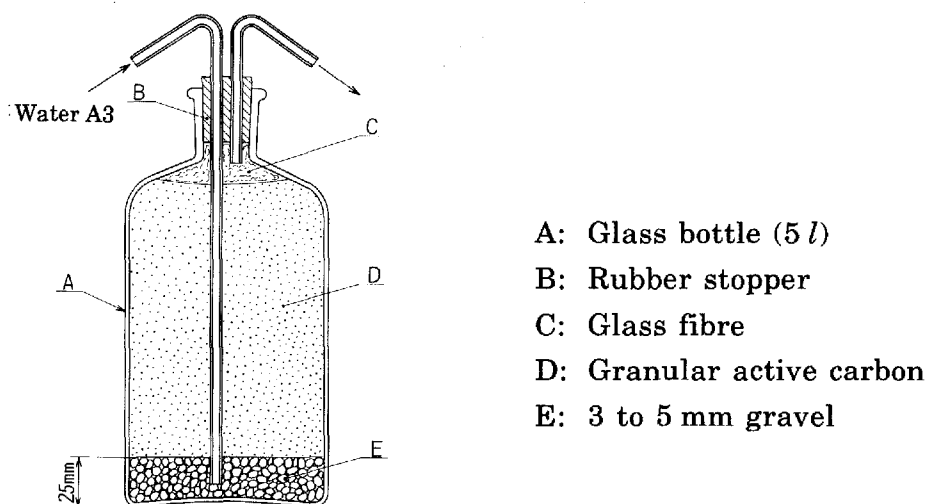


Fig. 8.1 Method for preparation of odour-free water (an example)

(2) **Implements** The implements shall be as follows.

- (a) **Erlenmeyer flask with ground stopper** 300 ml
(b) **Long leg burette** 50 ml
(c) **Water bath** The water bath with temperature regulator

(3) **Preliminary test** Carry out the preliminary test as follows.

- (a) Transfer 200, 40, 10 and 4 ml of the sample respectively into Erlenmeyer flasks with ground stopper, and dilute with odour-free water to 200 ml to make the sample for the preliminary test.
(b) Separately, transfer 200 ml of odour-free water into an Erlenmeyer flask with ground stopper as reference water.
(c) Warm the sample for the preliminary test and the reference water to 40 to 50 °C on the water bath, then shake to mix the reference water, and smell the odour generated at the same time of taking-off the stopper.
(d) Thereafter, operate the same as before-mentioned in the order from the smaller quantity of the sample, then compare the odour of the sample for the preliminary test with that of the reference water, and obtain the minimum quantity (ml) of the sample perceptible the odour.

(4) **Operation** Carry out the operation as follows.

- (a) Obtain the volume of the sample to be used for the test as the number of ml shown in longitudinal series according to Table 8.2 from the quantity of the sample obtained in (d) of (3).
- (b) Transfer each volume of the sample obtained in (a) into separate Erlenmeyer flasks with ground stopper respectively, and add the odour-free water to make 200 ml to take it as the sample for the main test.
- (c) Next, operate the same as in (b) to (d) of (3) to obtain the number of ml of the minimum sample perceptible the odour, and calculate the threshold odour number (TON) from the following formula:

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

where, TON : threshold odour number (TON)

V : sample used for dilution (ml)

Table 8.2 Volume of sample to be used for test

	Unit : ml			
Volume of sample for the preliminary test	200	40	10	4
Volume of sample to be used for the test	200	40	10	4.0
	100	28.5	8.0	2.9
	67	20	6.7	2.0
	50	13.3	5.0	1.3
	40	10	4.0	1.0

Remarks 3 Where the odour of sample is too intensive, carry out the preliminary test and the main test after diluting the sample with water to ten times.

4 Wash the Erlenmeyer flask with ground stopper to be used for the test with water beforehand.

5 The test depends on the environmental conditions in most cases, and therefore carry out the test in an odour-free, quiet room.

6 Avoid smoking, tea drinking, meal, etc. just before the test, and further keep the hands and fingers free from the odour of soap, lotion, perfume, etc.

7 Because the sense of smell is weakened by 4 to 5 times consecutive tests, repose for (approx.) 15 to 30 min.

8 Obtain the degree of odour (*pO*) from the following formula, if required.

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

9 Turbidity Turbidity expresses the degree of turbidity of water, and is indicated by dividing into visual-sensation turbidity, transmitted-light turbidity, scattered-light turbidity, and integrating-sphere turbidity. Where the turbidity is measured by comparing with kaolin standard solution, it shall be expressed by using "degree (kaolin)" as the unit, and where the turbidity is measured by comparing with formagen standard solution, it shall be expressed by using "degree (formagen)" as the unit.

The turbidity varies easily, and therefore it is desirable to test immediately after sampling.

9.1 Visual-sensation turbidity The visual-sensation turbidity shall be obtained by comparing the turbidity of the sample with the kaolin standard solution with the naked eye.

Measuring range: 1 to 10 degrees (kaolin)

(1) **Reagents** The following reagents shall be used.

- (a) **Water** Filter water A3 specified in **JIS K 0557** by using filter of approx. 0.1 μm pore diameter, and take the filtrate after discarding the first approx. 200 ml.
- (b) **Refined kaolin** Take approx. 10 g of kaolin into a 500 ml beaker, add to it 300 ml of water and 0.2 g of sodium diphosphate + hydrate (sodium pyrophosphate + hydrate) specified in **JIS K 8785** and mix by stirring vigorously with a magnetic stirrer for approx. 3 min. Transfer this into 1 000 ml measuring cylinder with ground stopper, add water up to the marked line of 1 000 ml, stopper it, and mix by shaking violently for approx. 1 min. After standing still at room temperature for approx. 1 h, discard the solution of 250 ml from the above part by using syphon, and separately take the solution up to the next 500 ml.

Centrifugalize the separately taken solution at approx. 3 000 min^{-1} (the number of rotations depends on the radius of rotating part of centrifugal separator), for approx. 20 min or filter to separate kaolin by using filter of not more than 1 μm pore diameter. Heat the filtered kaolin at 105 to 110 $^{\circ}\text{C}$ for approx. 3 h, and after standing to cool in a desiccator, preserve in a wide-mouthed bottle.

- (c) **Kaolin standard solution [1 000 degrees (kaolin)]** After dispersing 1.00 g of refined kaolin into water of proper amount, transfer into a 1 000 ml volumetric flask, add approx. 800 ml of water and approx. 10 ml of formaldehyde solution specified in **JIS K 8872**, and furthermore add water up to the marked line.
- (d) **Kaolin standard solution [100 degrees (kaolin)]** After mixing by stirring thoroughly the kaolin standard solution [1 000 degrees (kaolin)], immediately take its 100 ml into a 1 000 ml volumetric flask, and add water up to the marked line.

(2) **Implement** The implement shall be as follows.

- (a) **Dark box** It is convenient when comparing the turbidity with the naked eye to use a dark box as shown in Fig. 9.1. The measurement by approaching an electric lamp to the under window of the dark box becomes easily visible.

- (b) **Colorimetric tube** The flat bottomed colorimetric tube with a ground stopper with a marked line of 100 ml at a height of (270 ± 1.5) mm from the bottom as shown in Fig. 9.2 having colourless glass. The tubes aligned in height of marked lines (± 1.5 mm) shall be used.

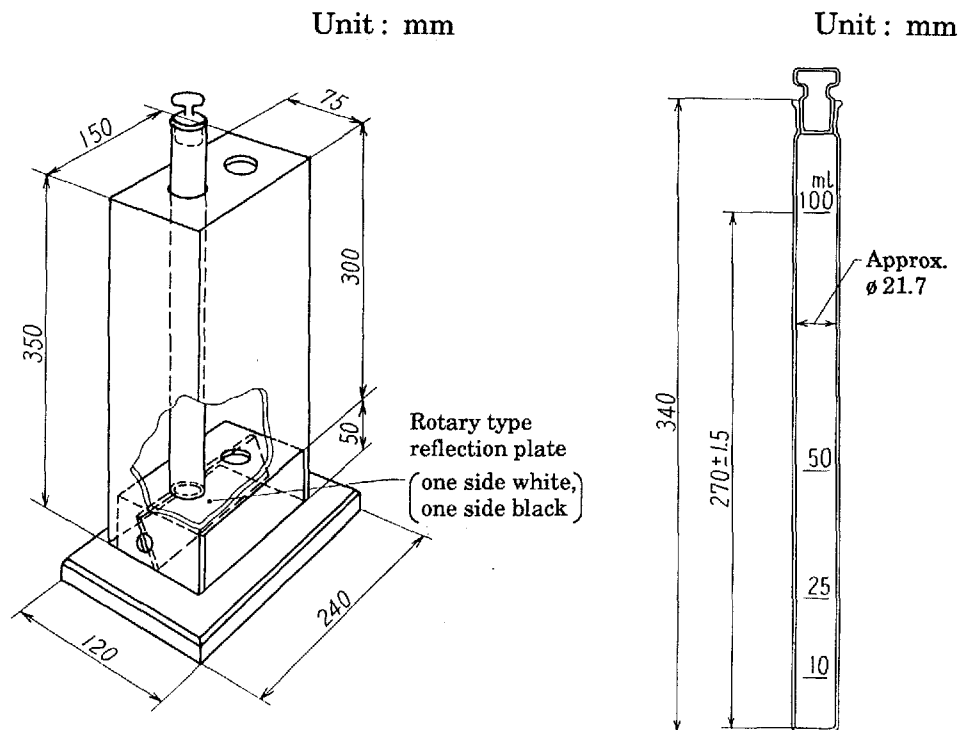


Fig. 9.1 An example of dark box

Fig. 9.2 Colorimetric tube

(3) **Operation** Carry out the operation as follows.

- After mixing with shaking the sample thoroughly, take a proper amount⁽¹⁾ from it into a colorimetric tube, and add water up to the marked line of 100 ml.
- Separately, take 1 to 10 ml of kaolin standard solution [100 deg. (kaolin)] step by step into a colorimetric tube, add water up to the marked line of 100 ml, and prepare the kaolin standard solution for nephelometry [1 to 10 deg. (kaolin)].
- After mixing by shaking thoroughly the colorimetric tube containing the sample and that containing kaolin standard solution for nephelometry, put them immediately into a dark box, see through from above to compare turbidity, and select the kaolin standard solution for nephelometry corresponding to the sample.
- From the corresponding turbidity [deg. (kaolin)] of the kaolin standard solution for nephelometry, calculate the visual-sensation turbidity of the sample [deg. (kaolin)] from the following formula:

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

where, T : visual-sensation turbidity [deg. (kaolin)]

T_s : corresponding turbidity of kaolin standard solution for nephelometry [deg. (kaolin)]

V : sample (ml)

Note (1) Where the turbidity of the sample is not more than 10 deg. (kaolin), take 100 ml as it is.

9.2 Transmitted-light turbidity Measure the intensity of transmitted-light near 660 nm in wavelength passed through the sample, and obtain the transmitted-light turbidity from the working curve prepared by using kaolin standard solution or formagen standard solution.

Measuring range: Where the absorption cell is 50 mm, 5 to 50 deg. (kaolin) or 4 to 80 deg. (formagen), and where the absorption cell is 10 mm, 25 to 250 deg. (kaolin) or 20 to 400 deg. (formagen).

(1) **Reagents** The following reagents shall be used.

- (a) **Water** As described in 9.1 (1) (a).
- (b) **Kaolin standard solution [100 deg. (kaolin)]** As described in 9.1 (1) (d).
- (c) **Formagen standard solution [400 deg. (formagen)]** Take 1.00 g of hydrazinium sulfate (hydrazine sulfate) specified in JIS K 8992, dissolve in a proper amount of water, transfer into a 100 ml volumetric flask and add water up to the marked line.

Separately take 10.0 g of hexamethylenetetramine specified in JIS K 8847 to dissolve in water of a proper amount, transfer it into a 100 ml volumetric flask, and add water up to the marked line.

Take respectively 10 ml of both these solutions in a 200 ml volumetric flask, and mix by shaking thoroughly. After standing as it is at $(25 \pm 3)^\circ\text{C}$ in solution temperature for approx. 24 h, add water up to the marked line.

(2) **Apparatus** The apparatus shall be as follows.

- (a) **Photometer** Spectrophotometer or photoelectric photometer

(3) **Operation** The operation shall be carried out as follows.

(3.1) **In the case of using kaolin standard solution**

- (a) After mixing by stirring the sample thoroughly, take it into a 50 mm⁽²⁾ absorption cell, and measure the intensity of transmitted light near 660 nm⁽³⁾ in wavelength by absorbance.
- (b) Obtain the transmitted-light turbidity [deg. (kaolin)] of the sample from the working curve prepared by using kaolin standard solution.

Working curve Take 5 to 50 ml of kaolin standard solution [100 deg. (kaolin)] step by step into a 100 ml volumetric flask, and add water up to the marked line to prepare the kaolin standard solution [5 to 50 deg. (kaolin)] for working curve⁽⁴⁾. Hereafter, carry out the operation of (a), and prepare the relation curve between the transmitted-light turbidity [deg. (kaolin)] and the absorbance of kaolin standard solution for working curve.

- Notes (2) Where the transmitted-light turbidity of the sample is 25 to 250 [deg. (kaolin)], use a 10 mm absorption cell.
- (3) Where the sample has a colour (specially where there is absorption near 660 nm in wavelength), measure the intensity of transmitted-light amount by absorbance by using the filtrate of the sample filtered by using film filter of 1 μ m or under of pore diameter or by using the supernatant precipitated centrifugally [at approx. 3 000 min⁻¹ (the number of rotations depends on the radius of rotating part of centrifugal separator) for approx. 20 min] as the reference solution.
- (4) Where the 10 mm absorption cell is used, take 2.5 to 25 ml of kaolin standard solution [1 000 deg. (kaolin)] of 9.1 (1) (c) stepwise, and prepare the kaolin standard solution [25 to 250 deg. (kaolin)] for working curve.

(3.2) In the case of using formagen standard solution

- (a) After mixing by shaking the sample thoroughly, carry out the operation⁽⁵⁾ of (3.1) (a).
- (b) Obtain the transmitted-light turbidity [deg. (formagen)] of the sample from the working curve prepared by using formagen standard solution.

Working curve Take 1 to 20 ml of formagen standard solution [400 deg. (formagen)] step by step into a 100 ml volumetric flask, and add water up to the marked line to prepare⁽⁶⁾ the formagen standard solution [4 to 80 deg. (formagen)] for working curve. Hereafter, carry out the operation of (a), and prepare the relation curve between the transmitted-light turbidity [deg. (formagen)] and the absorbance of the formagen standard solution for working curve.

- Notes (5) Where the transmitted-light turbidity of the sample is 20 to 400 deg. (formagen), use a 10 mm absorption cell.
- (6) Where a 10 mm absorption cell is used, take 5 to 100 ml of formagen standard solution [400 deg. (formagen)] of 9.2 (1) (c) step by step to prepare the formagen standard solution [20 to 400 deg. (formagen)] for working curve.

9.3 Scattered-light turbidity Measure the intensity of scattered light due to grains in the sample at near 660 nm wavelength, and obtain the scattered-light turbidity from the working curve prepared by using kaolin standard solution or formagen standard solution.

Measuring range: 1 to 5 deg. (kaolin) or 0.4 to 5 deg. (formagen) (different depending on the apparatus)

(1) **Reagents** The following reagents shall be used.

- (a) **Water** As described in 9.1 (1) (a).
- (b) **Kaolin standard solution [100 deg. (kaolin)]** As described in 9.1 (1) (d).

- (c) **Formagen standard solution [40 deg. (formagen)]** Take 10 ml of formagen standard solution [400 deg. (formagen)] of 9.2 (1) (c) into a 100 ml volumetric flask, and add water up to the marked line.

(2) **Apparatus** The apparatus shall be as follows.

- (a) **Scattered-light turbidity meter** An example of the scattered-light turbidity meter is given in Fig. 9.3.

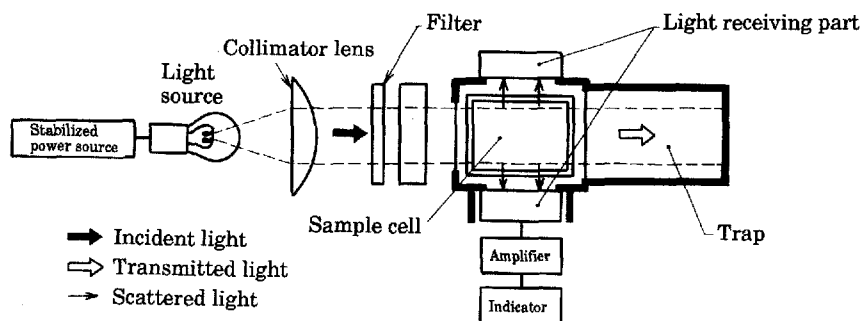


Fig. 9.3 Structural example of scattered-light turbidity meter

(3) **Operation** The operation shall be carried out as follows.

(3.1) In the case of using kaolin standard solution

- (a) Take water into an absorption cell, regulate the indication value⁽⁷⁾ of a scattered light turbidity meter at zero, then use kaolin standard solution for working curve [5 deg. (kaolin)], and regulate the indication value⁽⁷⁾ of the scattered light turbidity meter at 100 %.
- (b) After mixing the sample by shaking sufficiently, take into an absorption cell, and measure the indication value (intensity of scattered light) near 660 nm in wavelength.
- (c) Obtain the scattered light turbidity [deg. (kaolin)] of the sample from the working curve prepared by using kaolin standard solution.

Working curve Deal out step by step 1 to 5 ml kaolin standard solution [100 deg. (kaolin)] into a 100 ml volumetric flask, add water to the marked line, and prepare kaolin standard solution [1 to 5 deg. (kaolin)] for working curve. Hereafter carry out the operation of (a) and (b), and prepare the relation curve between the scattered light turbidity [1 to 5 deg. (kaolin)] of kaolin standard solution for working curve and the indication value (intensity of scattered light).

Note ⁽⁷⁾ For the intensity of scattered light, measurement at positions of 90° or 270° to incident light are generally carried out.

(3.2) In the case of using formagen standard solution

- (a) Take water into an absorption cell, regulate the indication value⁽⁷⁾ of a scattered light turbidity meter at zero, then use formagen standard solution [5 deg. (formagen)] for working curve, and regulate the indication value⁽⁷⁾ of the scattered light turbidity meter at 100 %.

- (b) After mixing the sample by shaking sufficiently, take into an absorption cell, and measure the indication value (intensity of scattered light) near 660 nm in wavelength.
- (c) Obtain the scattered light turbidity [deg. (formagen)] of the sample from the working curve prepared by using formagen standard solution.

Working curve Deal out step by step 1 to 12.5 ml of formagen standard solution [40 deg. (formagen)] into a 100 ml volumetric flask, add water to the marked line, and prepare formagen standard solution [1 to 5 deg. (formagen)] for working curve. Hereafter, carry out the operation of (a) and (b), and prepare the relation curve between the scattered light turbidity [1 to 5 deg. (formagen)] of formagen standard solution for working curve and the indication value (intensity of scattered light).

9.4 Integrating-sphere turbidity Obtain the ratio of the intensity of scattered light due to grains in water to the intensity of transmitted light, and obtain the integrating-sphere turbidity from the working curve prepared by using kaolin standard solution or formagen standard solution.

Measuring range: In the case of 50 mm absorption cell, 0.2 to 5 deg. (kaolin) or 0.2 to 5 deg. (formagen), and in the case of 10 mm absorption cell, 5 to 100 deg. (kaolin) or 5 to 100 deg. (formagen).

- (1) **Reagents** The following reagents shall be used.
 - (a) **Water** As described in 9.1 (1) (a).
 - (b) **Kaolin standard solution [100 deg. (kaolin)]** As described in 9.1 (1) (d).
 - (c) **Formagen standard solution [40 deg. (formagen)]** As described in 9.3 (1) (c).
- (2) **Apparatus** The apparatus shall be as follows.
 - (a) **Integrating-sphere turbidity meter** An example of the integrating-sphere turbidity meter is shown in Fig. 9.4.

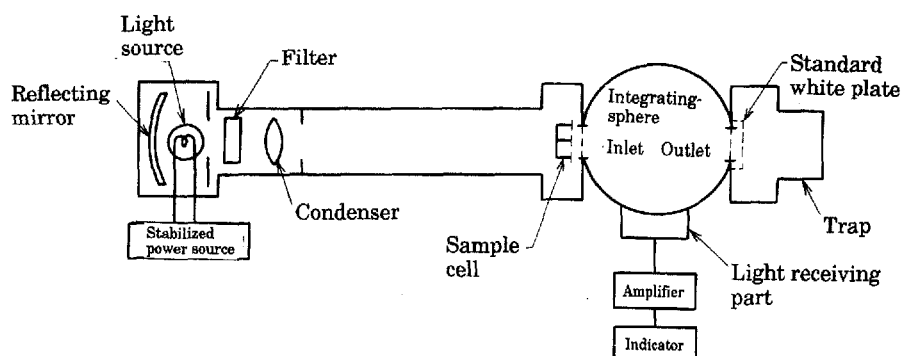


Fig. 9.4 Structural example of integrating-sphere turbidity meter

(3) **Operation** The operation shall be carried out as follows.

(3.1) **In the case of using kaolin standard solution**

- (a) Put a 50 mm absorption cell containing water and a trap in the optical path, and adjust the indicating value to 0. Then, insert a standard white plate into the trap, and adjust so that the indicating value becomes 100.
- (b) Next, substitute for 50 mm absorption cell containing water, put a 50 mm absorption cell containing the sample mixed by shaking thoroughly and a trap (containing no standard white plate) in the optical path, and measure the intensity of scattered light T_d .
- (c) Successively, insert a standard white plate into the trap, and measure the intensity T_t of total transmitted light of the sample.
- (d) Calculate the value of $T_d/T_t \times 100$, and obtain the integrating-sphere turbidity [deg. (kaolin)] of the sample from the working curve prepared by using kaolin standard solution.

Working curve Take 0.2 to 5 ml of kaolin standard solution [100 deg. (kaolin)] step by step into a 100 ml volumetric flask, and add water up to the marked line to prepare⁽⁸⁾ the kaolin standard solution [0.2 to 5 deg. (kaolin)] for working curve. Hereafter, carry out the operation of (a) to (d), and prepare the relation curve between the integrating-sphere turbidity [deg. (kaolin)] of the kaolin standard solution for working curve and the value of $T_d/T_t \times 100$.

Note ⁽⁸⁾ In the case of measuring by using a 10 mm absorption cell, prepare the kaolin standard solution [5 to 100 deg. (kaolin)] for working curve.

Remarks 1 **Method without calculation of $T_d/T_t \times 100$** After carrying out the operation of (a), put a cell containing the sample substituting for the absorption cell containing water and adjust so that the indicating value at that time becomes 100. Successively, read out the indicating value with the standard white plate detached. For working curve, operate in the same manner on the kaolin standard solution (or formagen standard solution), take the integrating-sphere turbidity [deg. (kaolin)] or [deg. (formagen)] on the abscissa and the indicating value, (corresponding to $T_d/T_t \times 100$) on the ordinate to prepare and obtain the integrating-sphere turbidity of the sample from this working curve.

(3.2) **In the case of using formagen standard solution**

- (a) Carry out the operation specified in (3.1) (a) to (c).
- (b) Calculate the value of T_d/T_t , and obtain the integrating-sphere turbidity [deg. (formagen)] of the sample from the working curve prepared by using formagen standard solution.

Working curve Take 0.5 to 12.5 ml of formagen standard solution [40 deg. (formagen)] step by step into a 100 ml volumetric flask, and add water up to the marked line to prepare the formagen standard solution [0.2 to 5 deg.

(formagen)] for working curve⁽⁹⁾. Hereafter, carry out the operation of (a) to prepare the relation curve between the integrating-sphere turbidity [deg. (formagen)] of the standard solution for working curve and the value of $T_d/T_i \times 100$.

Note (9) When measuring by using 10 mm absorption cell, prepare the formagen standard solution [5 to 100 deg. (formagen)] for working curve.

10 Colour For colour marking, the method according to the chromaticity according to platinum/cobalt or the method according to the stimulus value Y and chromaticity coordinates x, y shall be used. The chromaticity according to platinum/cobalt applies only where the sample is of colour system from faint yellow to yellow brown.

10.1 Chromaticity according to platinum/cobalt The chromaticity according to platinum/cobalt indicates the degree from faint yellow to yellow brown due to dissolved-in-water or colloidal substances and the colour appearing when 1 ml of platinum/cobalt chromaticity standard solution (1 mg of platinum and 0.5 mg of cobalt) is added in 1 l of water shall be taken as platinum/cobalt chromaticity one degree.

(1) **Reagents** The following reagents shall be used.

- (a) **Water** Filter water A3 specified in **JIS K 0557** by using filter of approx. 0.1 μm pore diameter, and take the filtrate after discarding the first approx. 200 ml.
- (b) **Platinum/cobalt chromaticity standard solution (1 000 deg.)** Take 2.49 g of potassium hexachloroplatinate specified in **JIS K 8163**⁽¹⁾, 2.00 g of cobalt II chloride hexahydrate specified in **JIS K 8129** and 200 ml of hydrochloric acid specified in **JIS K 8180**, add water to dissolve, transfer into a 1 000 ml volumetric flask, and add water up to the marked line. Preserve it in a brown bottle.

Note (1) Where platinum is used instead of potassium hexachloroplatinate specified in **JIS K 8163**, dissolve 1.00 g of platinum in a proper amount of aquaregia (hydrochloric acid 3 + nitric acid 1), add hydrochloric acid in excess and evaporate to dryness on a water bath. Repeat this operation two or three times to remove nitric acid, thereafter dissolve it together with 2.00 g of cobalt II chloride hexahydrate specified in **JIS K 8129** and 200 ml of hydrochloric acid specified in **JIS K 8180**, put into a 1 000 ml volumetric flask, and add water up to the marked line.

(2) **Apparatus** The apparatus shall be as follows.

- (a) **Dark box** As described in 9.1 (2) (a).
- (b) **Colorimetric tube** 100 ml As described in 9.1 (2) (b).

(3) **Operation** The operation shall be carried out as follows.

- (a) Filter the sample with filter paper of class 5C or filter of pore diameter of 1 μm or less, or separate the sample centrifugally at about 3 000 min^{-1} (the number of rotations depends on the radius of rotating part of centrifugal separator) for 20 min to remove turbidity.
- (b) Take a proper amount of this sample into a 100 ml colorimetric tube, and add water up to the marked line of 100 ml.
- (c) Take step by step 0.1 to 2.0 ml of platinum/cobalt chromaticity standard solution (1 000 deg.) into a 100 ml colorimetric tube, add water up to the marked line of 100 ml, stopper it and mix by shaking thoroughly to prepare platinum/cobalt chromaticity standard solution series of 1 to 20 deg.

- (d) Place the sample and chromaticity standard solution series on a white paper or put into a dark box to see through from above, and compare the colour of the sample with the platinum/cobalt chromaticity standard solution series to obtain the corresponding platinum/cobalt chromaticity standard solution.
- (e) Calculate the platinum/cobalt chromaticity of the sample from the following formula:

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

where, C : platinum/cobalt chromaticity
 C_s : corresponding platinum/cobalt chromaticity standard solution (deg.)
 V : sample (ml)

10.2 Marking by stimulus value Y and chromaticity coordinates x, y The percentage transmission of the sample at each wavelength from 400 to 700 nm is measured using water as reference solution to obtain tristimulus value⁽²⁾ X, Y, Z , and the chromaticity coordinates x, y is calculated, then the colour is marked with stimulus value Y and chromaticity coordinates x, y .

Note ⁽²⁾ The value obtained by totalizing the percentage transmission in series of X, Y , and Z according to the kinds of wavelength giving respective stimuli, considering that there are three kinds of sensitivities of the optic nerve to colour, and the sensitivity to colour changes by mixing rate of the three kinds of stimuli to the optic nerve.

- (1) **Reagent** The following reagent shall be used.
 - (a) **Water** As described in 10.1 (1) (a).
- (2) **Apparatus** The apparatus shall be as follows.
 - (a) **Centrifugal separator**
 - (b) **Photometer** The spectrophotometer capable of using 100 mm absorption cell and of measuring the total visible field with the wavelength interval of 10 nm or less, or equivalent chromaticity meter in performance.
- (3) **Operation** The operation shall be carried out as follows.
 - (a) Remove turbidities by filtering the sample with filter paper of class 5C or filter of 1 μm pore diameter or less, or by centrifugal separation at about 3 000 min^{-1} (the number of rotations depends on the radius of rotating part of centrifugal separator) for 20 min.
 - (b) Transfer a portion of this sample into a 100 mm absorption cell⁽³⁾, and measure the percentage transmission at each wavelength specified in Table 10.1⁽⁴⁾ using water as reference solution.

Notes ⁽³⁾ When the measurement using 100 mm absorption cell is impossible because of the dark colour of the sample, use an absorption cell of proper length of optical path to measure. In this case, measure the absorbance at each wavelength, convert the value to the absorbance of 100 mm absorption cell, and calculate by obtaining the percentage transmission from this absorbance.

- (4) Table 10.1 follows Attached Table 5-1-1 (20 nm in wavelength interval) of the evaluation method of metamerism index/degree of illuminant metamerism specified in **JIS Z 8719**.

Table 10.1 Polyvalues function f_x, f_y, f_z for calculating tristimulus values X, Y, Z by 20 nm in wavelength interval

Wavelength λ (nm)	Specified chromatic light 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	0.356
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	$x=0.310\ 1$	$y=0.316\ 2$	$z=0.373\ 7$

- (c) **Method for obtaining tristimulus value and chromaticity coordinates x, y** Totalize the percentage transmission at each specific wavelength of X, Y , and Z in Table 10.1 respectively, and calculate the tristimulus value X, Y , and Z , and chromaticity coordinates x, y from the following formula,

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

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

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

$$x = \frac{X}{X+Y+Z}$$

$$y = \frac{Y}{X+Y+Z}$$

where, X : stimulus value X

Y : stimulus value Y

Z : stimulus value Z

K : 100.000

$f_X(\lambda)$: f_X at wavelength λ

$f_Y(\lambda)$: f_Y at wavelength λ

$f_Z(\lambda)$: f_Z at wavelength λ

$\tau(\lambda)$: percentage transmission at wavelength λ

- (d) **Method for obtaining stimulus value Y** Stimulus value Y , as it is, is used as the stimulus value.

Remarks 1 Method for obtaining dominant wavelength and complementary wavelength The point in the chromaticity (Fig. 10.1) is non-coloured chromaticity coordinates ($x = 0.310\ 1$, and $y = 0.316\ 2$).

For the colour of which the chromaticity coordinates are represented by the point S_1 in the area enclosed by the straight line RC , the straight line VC , and the spectrum locus, obtain the wavelength corresponding to the intersecting point S_1' of the extension of the straight line CS_1 and the spectrum locus from Fig. 10.3. Call this wavelength the dominant wavelength of the said colour and express by the symbol λ_d .

Further, for the colour of which the chromaticity coordinates are represented by the point S_2 inside the triangle CRV , obtain the wavelength corresponding to the intersecting point S_2'' of the extension of the straight line CS_2 and the spectrum locus from Fig. 10.3. Call this wavelength the complementary wavelength of the said colour, and express by the symbol λ_e .

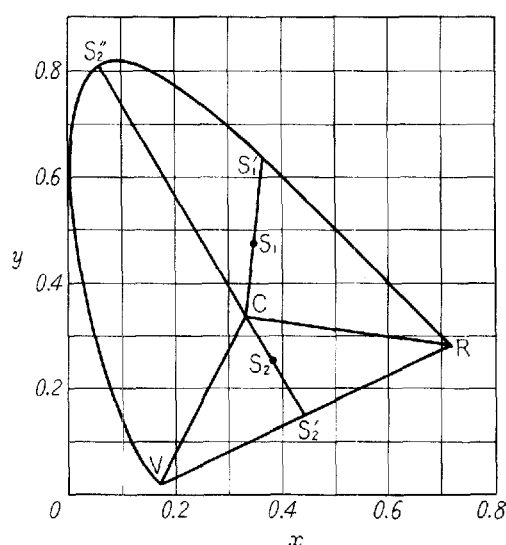


Fig. 10.1 Chromaticity diagram

Remarks 2 Method for obtaining excitation purity For the colour of which the chromaticity coordinates are represented by the point S_1 in Fig. 10.1, obtain the excitation purity P_e from the following formula(*), and annex % to the said value.

$$P_e = \frac{x - x_c}{x_{\lambda} - x_c} \times 100 \dots\dots\dots (1)$$

or

$$P_e = \frac{y - y_c}{y_{\lambda} - y_c} \times 100 \dots\dots\dots (2)$$

where, x, y : coordinates of point S_1

x_c, y_c : coordinates of point C

x_{λ}, y_{λ} : coordinates of point S_1'

Further, for the colour of which the chromaticity coordinates are represented by the point S_2 , obtain the excitation purity P_e from the following formula(*), and annex % to the said value.

$$P_e = \frac{x - x_c}{x_p - x_c} \times 100 \dots\dots\dots (3)$$

or

$$P_e = \frac{y - y_c}{y_p - y_c} \times 100 \dots\dots\dots (4)$$

where, x, y : coordinates of point S_2

x_c, y_c : coordinates of point C

x_p, y_p : coordinates of point S_2' (intersecting point of the extension of straight line CS_2 and purple boundary)

Note (*) In order to calculate P_e , obtain by the formula of which the absolute value of a denominator or a numerator is large.

An example of method of obtaining excitation purity and dominant wavelength If the tristimulus value obtained by measuring the sample, X is 40.14, Y , 76.50, and Z , 34.25, the chromaticity coordinates x is 0.266, y , 0.507 and the stimulus value Y , 76.50. Obtain the chromaticity coordinates as a point (S_1) on Fig. 10.2 and then the colourless point as C ($x = 0.310\ 1$, $y = 0.316\ 2$). Connect C and S_1 with a straight line, and obtain the intersecting point of the extension of straight line and the spectrum locus as S_1' . S_1' is located at 534 nm on the spectrum locus, i.e. the dominant wavelength λ_d is 534 nm.

Further, since the coordinates of S_1' x_{λ} is 0.200 and y_{λ} is 0.785, the excitation purity P_e becomes $\frac{0.507 - 0.316\ 2}{0.785 - 0.316\ 2} \times 100 = 40.7\ \%$.

Therefore, the colour of the sample is expressed by the dominant wavelength $\lambda_d = 534 \text{ nm}$ and the excitation purity $Y = 76.50 \%$.

Further, it is convenient that the excitation purity is obtained by using Fig. 10.4 when the colour of the sample is thin and by using Fig. 10.5 when the colour of the sample is dense.

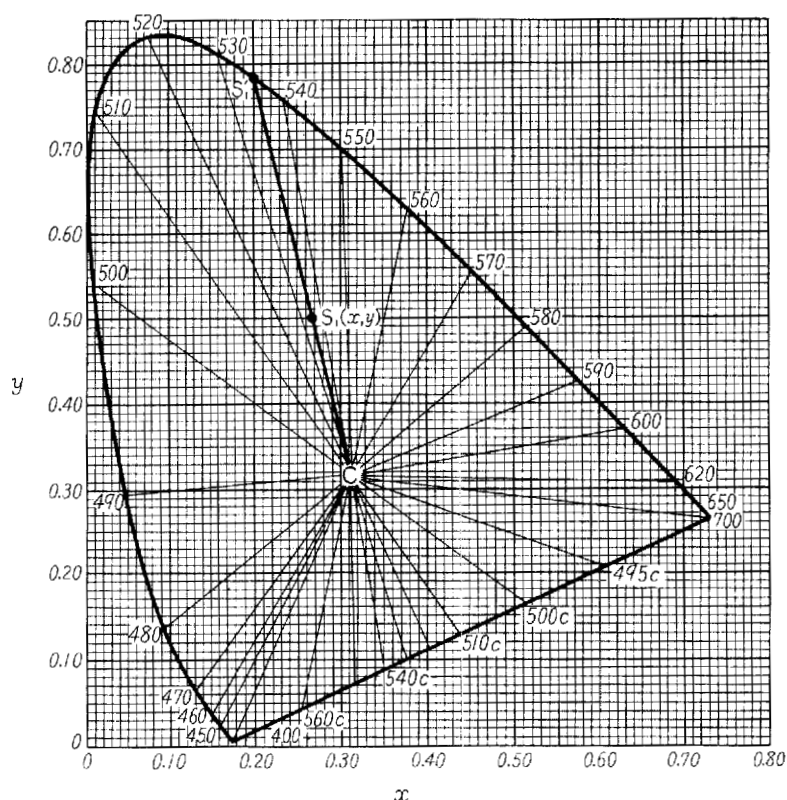
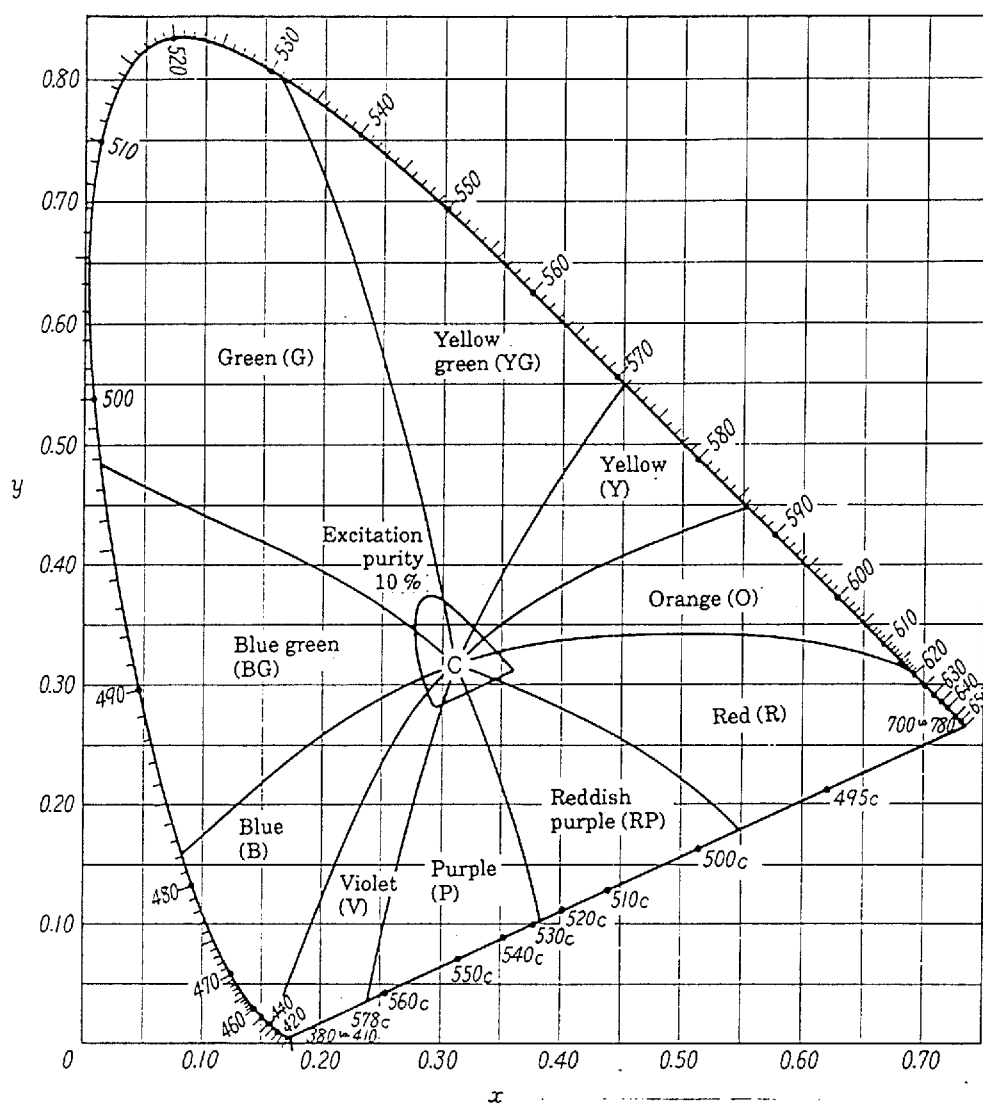


Fig. 10.2 Relation diagram between chromaticity diagram and dominant wavelength according to 2 degree visual field XYZ system



The curve indicated with wavelength scale mark is the spectrum locus, and the curve connecting both the ends of the spectrum locus is the purple boundary. The point C indicates the chromaticity coordinates of standard light C ($x = 0.3100$, $y = 0.3162$).

Fig. 10.3 Chromaticity diagram according to 2 degree visual field XYZ system (dominant wavelength and hue name)

Remarks 3 Dominant wavelength and hue The relationship between the dominant wavelength and the hue is shown in Table 10.2 and Fig. 10.3.

Table 10.2 Dominant wavelength and hue name

Dominant wavelength nm	Hue name	Abbreviated symbol	Dominant wavelength nm	Hue name	Abbreviated symbol
498 _c to 700 to 618	Red	R	498 to 482	Blue green	BG
618 to 586	Orange	O	482 to 435	Blue	B
586 to 571	Yellow	Y	435 to 400 to 578 _c	Violet	V
571 to 531	Yellow green	YG	578 _c to 528 _c	Purple	P
531 to 498	Green	G	528 _c to 498 _c	Reddish purple	RP

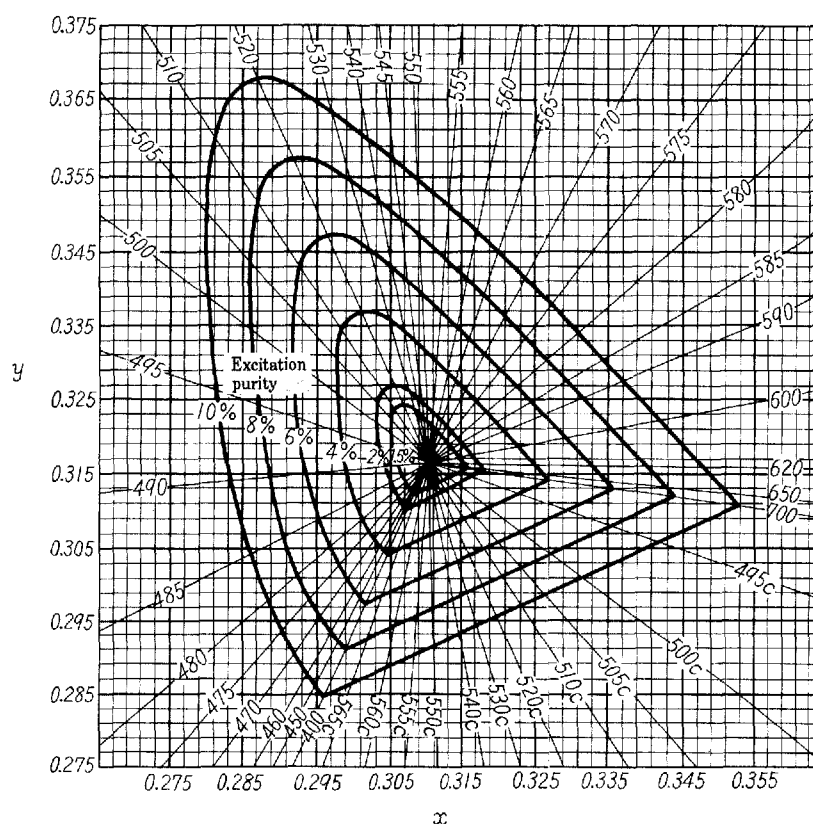


Fig. 10.4 Excitation purity (10 % marking) (in the case where the colour of sample is thin)

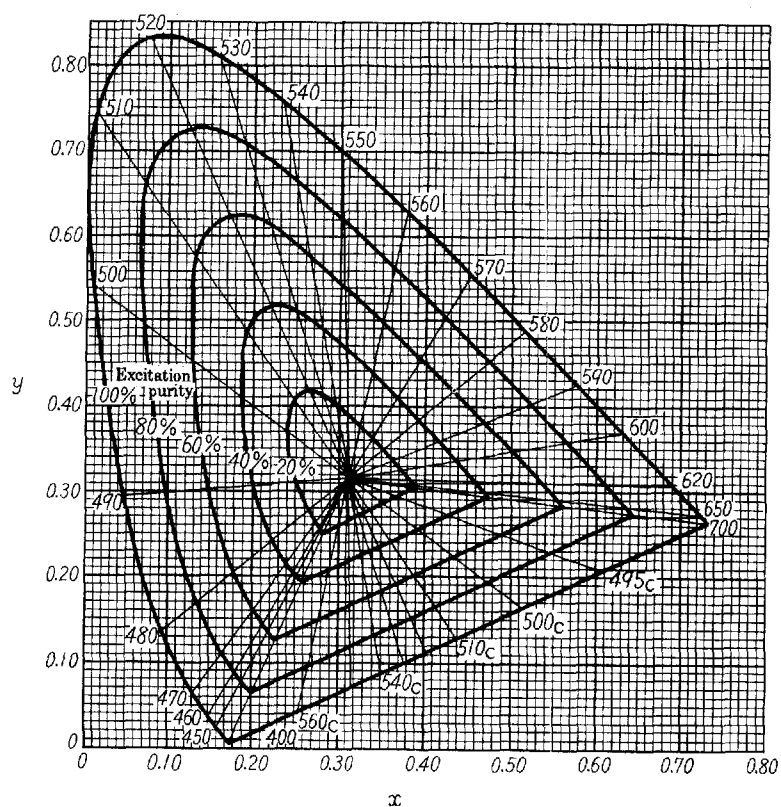


Fig. 10.5 Excitation purity (100 % marking) (in the case where the colour of sample is dense)

11 pH For measurement of pH, the glass electrode method specified in **JIS Z 8802** shall be applied.

Immediately after sampling, pH shall be measured.

11.1 Glass electrode method pH is measured with a pH meter using a glass electrode.

(1) **Reagents** The following reagents shall be used.

- (a) **Water** Water A2 or A3 specified in **JIS K 0557**. In the case where borate pH standard solution and carbonate pH standard solution are prepared, carbonic acid-free water of 2 (12) (b) is used.
- (b) **Potassium trihydrogen dioxalate dihydrate** Potassium trihydrogen dioxalate dihydrate specified in **JIS K 8474**.
- (c) **Potassium hydrogen phthalate** Potassium hydrogen phthalate specified in **JIS K 8809** for pH standard solution.
- (d) **Potassium dihydrogenphosphate** Potassium dihydrogenphosphate specified in **JIS K 9007** for pH standard solution.
- (e) **Disodium hydrogenphosphate** Disodium hydrogenphosphate specified in **JIS K 9020** for pH standard solution.
- (f) **Sodium tetraborate** Sodium tetraborate 10 hydrate specified in **JIS K 8866** for pH standard solution.
- (g) **Sodium hydrogen carbonate** Sodium hydrogen carbonate specified in **JIS K 8622** for pH standard solution.
- (h) **Sodium carbonate** Sodium carbonate specified in **JIS K 8625** for pH standard solution.

(2) **pH standard solution**⁽¹⁾ The following pH standard solutions shall be used.

- (a) **Oxalate pH standard solution** For the prepared pH standard solution, grind potassium trihydrogen dioxalate dihydrate with an agate mortar, preserve in a desiccator for 18 h or longer, dissolve its 12.71 g in a small quantity of water, transfer to a 1 000 ml volumetric flask, and add water to the marked line. Put it into a polyethylene bottle with ground stopper or a borosilicate glass bottle with ground stopper to be preserved. For specification pH standard solution, use grade 2 of oxalate pH standard solution specified in **JIS K 0018**.
- (b) **Phthalate pH standard solution** For the prepared pH standard solution, preliminarily heat potassium hydrogen phthalate at 120 °C for 1 h. After standing to cool in a desiccator, take its 10.21 g, dissolve in a small quantity of water, transfer to a 1 000 ml volumetric flask, and add water to the marked line. Put it into a polyethylene bottle with ground stopper or a borosilicate glass bottle with ground stopper to be preserved. For the specification pH standard solution, use grade 2 of phthalate pH standard solution specified in **JIS K 0019**.

- (c) **Neutral phosphate pH standard solution** For the prepared pH standard solution, preliminarily heat sodium dihydrogenphosphate and disodium hydrogenphosphate at 110 °C for 2 h. After standing to cool in a desiccator, take 3.40 g of potassium dihydrogenphosphate and 3.55 g of disodium hydrogenphosphate, dissolve in a small quantity of water, transfer to a 1 000 ml volumetric flask, and add water to the marked line. Put it into a polyethylene bottle with ground stopper or a borosilicate glass bottle with ground stopper to be preserved. For the specification pH standard solution, use grade 2 of neutral phosphate pH standard solution specified in **JIS K 0020**.
- (d) **Borate pH standard solution** For the prepared pH standard solution, grind sodium tetraborate 10 hydrate with an agate mortar. After making a constant quantity by leaving as it is in a desiccator containing the solution obtained by further adding sodium bromide specified in **JIS K 8514** to sodium bromide solution (saturated), take its 3.81 g, dissolve in a small quantity of carbonic acid free water [according to 2 (12) (b)], transfer to a 1 000 ml volumetric flask, and add the said carbonic acid free water to the marked line. Fill a polyethylene bottle with ground stopper or a borosilicate glass bottle with ground stopper to a full state therewith to be preserved. For the specification pH standard solution, use grade 2 of borate pH standard solution specified in **JIS K 0021**.
- (e) **Carbonate pH standard solution** For the prepared pH standard solution, make sodium hydrogen carbonate stand in a desiccator for approx. 3 h, and take its 2.10 g. Separately, preliminarily put sodium carbonate into a platinum crucible, heat at 600 °C to a constant weight, and take its 2.65 g. Dissolve both in a small quantity of carbonic acid free water [according to 2 (12) (b)], transfer to a 1 000 ml volumetric flask, and add the said carbonic acid free water to the marked line. Put it into a polyethylene bottle with ground stopper or a borosilicate glass bottle with ground stopper, and preserve in a desiccator containing soda lime specified in **JIS K 8603** or potassium hydroxide specified in **JIS K 8574**. For the specification pH standard solution, use grade 2 of carbonate pH standard solution specified in **JIS K 0022**.

Note (1) For the pH standard solution, use the prepared pH standard solution or grade 2 of the specification pH standard solution.

The prepared pH standard solution is prepared in accordance with the preparation method of each pH standard solution by a test operator himself. To the pH value at each temperature of each prepared pH standard solution, the pH value of Table 11.1 shall be applied.

The specification pH standard solution is systematized as the pH standard solution traceable to the primary standard substance (having traceability to an upper standard) kept by the national research institute. The pH standard solutions prepared by pH standard solution manufacturers are submitted to the public institute under national superintendence and direction (the institute having the secondary pH standard solution decided by the primary pH standard solution owned by the national research

institute), receives inspection to be accepted. The said accepted pH standard solution on the market shall be the specification pH standard solution.

Though these are grade 1 and grade 2 for the specification pH standard solution, since the grade 1 specification pH standard solution guarantees the pH value to three decimal places. For grade 2 specification pH standard solution to be used for this test, three decimal places of the pH value of grade 1 is rounded off to the second decimal place. The pH value at each temperature of grade 2 of each specification standard solution is given in Table 11.2.

Table 11.1 pH value of each temperature of the prepared pH standard solution

Temperature °C	pH value				
	Oxalate	Phthalate	Neutral phosphate	Borate	Carbonate (*)
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 (*) The values in parentheses indicate secondary interpolated values.