

9	0.99978	19	0.99841	29	0.99594	39	0.99259
10	0.99970	20	0.99820	30	0.99565	40	0.99222

(2) Solid samples

A pycnometer which is suitable for solid samples is used. Use the sample pulverized appropriately. If the sample is in the form of powders, granules, pellets or flakes of measurable size, use the sample as it is.

Weigh a pycnometer, previously cleaned and dried, to determine its mass W down to the degree of 1.0 mg.

Place an adequate amount of sample in the pycnometer and measure its mass W_1 . Add the immersion liquid, keeping the temperature of the liquid 1 to 3°C lower than the specified temperature $t^\circ\text{C}$, to immerse the sample. Place the pycnometer in a desiccator and hold it with vacuum to remove air completely. Return the pressure to ordinal, fill the pycnometer with the immersion liquid, place the pycnometer in a water bath and keep it at the specified temperature $t^\circ\text{C}$. Next, exactly fill the pycnometer to its capacity with the immersion liquid, wipe out the attached liquid thoroughly and measure its mass W_2 . Empty the pycnometer, clean, wipe it. Then, fill the pycnometer with boiled and cooled distilled water, deaerate it in the same manner as described above, and measure the mass W_w at the specified temperature $t^\circ\text{C}$. When water is used as the immersion liquid, the specific gravity d'_i ($t' = t$) can be calculated by the following equation.

$$d'_i = \frac{W_1 - W}{(W_w - W) - (W_2 - W_1)}$$

When the immersion liquid other than water is used, follow the same procedure using the given immersion liquid, measure the mass W_{IL} and calculate the density (ρ'_{IL}) of that immersion liquid by using the following equation.

$$\rho'_{IL} = \frac{W_{IL} - W}{W_w - W} \times \rho'_w$$

ρ'_w : Density (g/cm^3) of water at the specified temperature t (see Table 1)

The density of the sample (ρ'_T) can be calculated by the equation.

$$\rho'_T = \frac{(W_1 - W) \times \rho'_{IL}}{(W_{IL} - W) - (W_2 - W_1)}$$

When t' is not equal to t , the specific gravity d'_i of the sample can be calculated by using the density of water at t (ρ'_w , Table 1) by the following equation.

$$d'_i = \frac{\rho'_T}{\rho'_w}$$

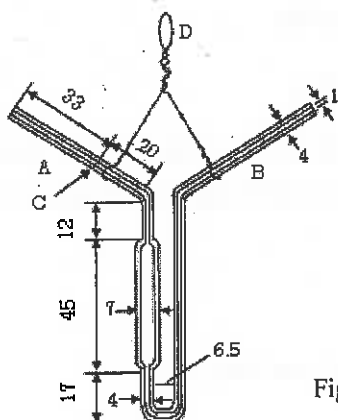
Immersion liquid

The immersion liquid is the liquid with known density which does not dissolve, swell or react with the sample and whose specific gravity is lower than that of the sample. Usually, new distilled water, purified mineral oil, etc. are used. No more than 0.1% of an appropriate moistening agent may be added to the immersion liquid to expel any entrapped air bubbles.

Method 2: Measurement using a Sprengel-Ostwald pycnometer

A Sprengel-Ostwald pycnometer is a glass vessel with a capacity of usually 1 mL to 10 mL. As shown in Fig. 1, both ends are thick-walled fine tubes (inside diameter: 1-1.5 mm, outside diameter: 3-4 mm), one of which, tube A, has a line marked C on it. Determine the mass of a pycnometer, W , previously cleaned and dried, by hanging it on the arm of a chemical balance with a platinum or aluminum wire D. Immerse the fine tube B in the sample solution, which is at a lower temperature by 3°C to 5°C than the specified temperature t °C. Attach rubber tubing or a ground-glass tube to the end of A, and suck up the sample solution until the meniscus is above the marked line C, taking care to prevent bubble formation. Immerse the pycnometer in a water bath kept at the specified temperature t °C for about 15 minutes, and then, by attaching a piece of filter paper to the end of B, adjust the level of the sample solution to the marked line C. Take the pycnometer out of the water bath, wipe thoroughly the outside surface and determine the mass W_1 . By use of the same pycnometer, perform the same procedure for the standard solution of water. Weigh the pycnometer containing water at the specified temperature t °C, and note the mass W_w . Calculate the specific gravity d_t^t according to the following equation.

$$d_t^t = \frac{W_1 - W}{W_w - W}$$



(The figures are in mm.)

Fig. 1 Measurement using a Sprengel-Ostwald pycnometer

Further, when measurements for a sample solution and water are performed at the same temperature (t °C = t °C), the density of the sample solution at the temperature t °C (ρ_t^t) can be calculated from the measured specific gravity d_t^t and the density of water at the temperature t °C (ρ_w^t) indicated in Table 1 by using the following equation.

$$\rho_t^t = \rho_w^t d_t^t$$

Method 3: Measurement using an oscillator-type density meter

Density measurement with an oscillator-type density meter is a method for obtaining the density of liquid or gas by measuring the intrinsic vibration period T (s) of a glass tube cell filled with sample specimen. When a glass tube containing a sample is vibrated, it undergoes a vibration with an intrinsic vibration period T in proportion to the mass of the sample specimen. If the volume of the vibrating part of the sample cell is fixed, the relation of the square of intrinsic oscillation period and density of the sample specimen shall be linear. Before measuring a sample density, the respective intrinsic oscillation periods T_{S1} and T_{S2} for two reference substances (density: ρ_{S1} , ρ_{S2}) must be measured at a specified temperature t °C, and the cell constant K_t ($\text{g} \cdot \text{cm}^{-3}$)

s⁻²) must be determined by using the following equation.

$$K_r = \frac{\rho'_{S1} - \rho'_{S2}}{T_{S1}^2 - T_{S2}^2}$$

Usually, water and dried air are chosen as reference substances. Here the density of water at $t^\circ\text{C}$, ρ'_{S1} , is taken from the Table 1, and that of dried air ρ'_{S2} is calculated by using the following equation, where the pressure of dried air is at p kPa.

$$\rho'_{S2} = 0.0012932 \times \{273.15 / (273.15 + t)\} \times (p / 101.325)$$

Next, introduce a sample specimen into a sample cell having a cell constant K_r , the intrinsic vibration period, T_r , for the sample under the same operation conditions as employed for the reference substances. The density of a sample specimen at $t^\circ\text{C}$, ρ'_r , is calculated by use of the following equation, by introducing the intrinsic oscillation period T_{S1} and the density of water at a specified temperature $t^\circ\text{C}$, ρ'_{S1} , into the equation.

$$\rho'_r = \rho'_{S1} + K_r(T_r^2 - T_{S1}^2)$$

Further, the specific gravity of a sample specimen against water at a temperature $t^\circ\text{C}$, d'_t , can be obtained by using the equation below, by introducing the density of water at a temperature $t^\circ\text{C}$, ρ'_w , indicated in Table 1.

$$d'_t = \frac{\rho'_r}{\rho'_w}$$

Apparatus

An oscillator-type density meter is usually composed of a glass tube cell of about 1 mL capacity, the curved end of which is fixed to the vibration plate, an oscillator which applies an initial vibration to the cell, a detector for measuring the intrinsic vibration period, and a temperature controlling system.

A schematic illustration of the apparatus is depicted in Fig. 2.

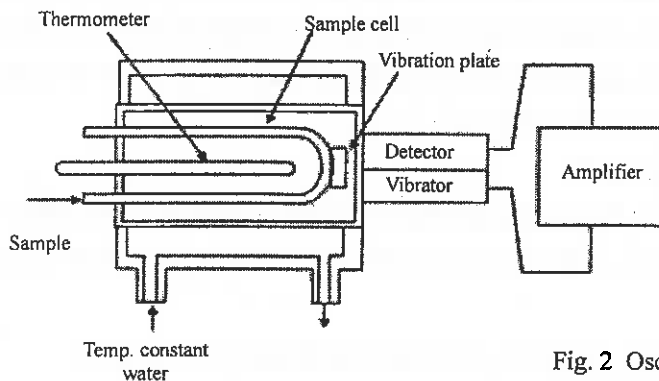


Fig. 2 Oscillator-type density meter

Procedure

A sample cell, water, and a sample specimen are previously adjusted to a specified temperature $t^\circ\text{C}$. Wash the sample cell with water or an appropriate solvent, and dry it thoroughly with a flow of dried air. Stop the flow of dried air, confirm that the temperature is at the specified value, and then measure the intrinsic oscillation period T_{S2} given by the dried air. Separately, the atmospheric pressure p (kPa) must be measured at the time and place of the examination. Next, introduce water into the sample cell and measure the intrinsic oscillation period T_{S1} given by water. Using these values of the intrinsic oscillation period and the atmospheric pressure,

the sample cell constant K_r can be determined by use of the above-mentioned equation.

Next, introduce a sample specimen into the glass cell, confirm the specified temperature, and measure the intrinsic oscillation period T_T given by the sample specimen. Using the intrinsic oscillation periods for water and the sample specimen, the density of water ρ'_{S1} , and the cell constant K_r , the density of the sample specimen ρ'_T can be obtained by use of the above equation. If necessary, the specific gravity of the sample specimen d'_t against water at a temperature $t^\circ\text{C}$, can be calculated by using the density of water ρ'_W shown in Table 1.

In this measurement, avoid the occurrence of bubble formation in the sample cell, when a sample specimen or water is introduced into the cell.

Method 4: Sink-float method

This method is used to measure the specific gravity of a chemical fiber sample or a plastic sample such as granule, pellet or flake.

(1) Chemical fiber samples

Immerse about 0.1 g of the fiber sample in about 20 mL of an appropriate deoiling liquid shown in Table 2 for about 1 hour. After remove oil, air-dry the sample, cut into 0.5 mm to 1.0 mm pieces using a razor etc., and using a vacuum desiccator with the pressure not exceeding 0.5 kPa dry these pieces completely before use. Place about 8 mL of the prepared specific gravity determination liquid in the sedimentation tube and put a small amount of the sample in it. Stopper the sedimentation tube, disperse the sample well, centrifuge to expel any entrapped air bubbles and immerse the tube in a water bath maintained at the specified temperature $t^\circ\text{C} \pm 0.1^\circ\text{C}$ for about 30 minutes. Observe whether the sample sinks or floats and add an adequate amount of either heavy or light liquid shown in Table 3 according to its condition to adjust the specific gravity of the specific gravity determination liquid so that the sample reaches equilibrium in the liquid. After adjustment, allow the tube to stand in the water bath for 30 minutes and confirm that the sample exists still in equilibrium in the liquid. Then, measure the specific gravity of the liquid in the cylinder as directed in Methods 1 to 3 and designate it as the specific gravity of the sample (d'_t).

Specific gravity determination liquid

Mix adequate amounts of a heavy liquid and a light liquid for fiber sample shown in Table 3 in a measuring cylinder so that the specific gravity of the mixture is within ± 0.02 of the expected specific gravity of the sample.

Table 2: Liquids used for deoiling chemical fiber samples

Fiber	Deoiling liquid
Polyester	Methanol
Acetate	Diethyl ether
Polypropylene	Ethanol
Other	Mixture of ethanol and benzene (capacity ratio 1:2)

Table 3: Heavy and light liquids

Fiber	Heavy liquid	Light liquid
Polypropylene	Water	Ethanol
Polyester	Perchloroethylene	n-Heptane
Other	Perchloroethylene	Toluene

(2) Plastic samples such as granules, pellets or flakes

Transfer exactly 100 mL of the prepared specific gravity determination liquid into a 250 mL measuring cylinder and immerse the cylinder in a water bath maintained at $t^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$. Put some of the samples in a cylinder, and sink them to the bottom to avoid formation of bubbles. Allow the cylinder to stand for about 5 minutes and stir several times so that the temperature remains stable at the specified temperature $t^{\circ}\text{C}$. Observe whether the sample sinks or floats and add an adequate amount of either heavy or light liquid according to its condition to adjust the specific gravity of the specific gravity determination liquid so that the sample reaches equilibrium in the liquid. After adjustment, allow the cylinder to stand in the water bath for a while and confirm that the sample exists still in equilibrium. Then, measure the specific gravity of the liquid in the cylinder as directed in Methods 1 to 3 and designate it as the specific gravity of the sample (d'_t).

Specific gravity determination liquid

The heavy and light liquids are freshly distilled liquids having different densities, which are compatible and do not dissolve, swell or react with the sample. Mix adequate amounts of a heavy liquid and a light liquid so that the specific gravity of the mixture is within ± 0.02 of the expected specific gravity of the sample, and use as the specific gravity determination liquid at the start of the measurement.

Method 5: Measurement using density gradient tube

When a solid sample is placed in the liquid, it sinks if its density is greater than that of the liquid and it floats if its density is smaller than that of the liquid, and it remains suspended if its density is equal to that of the liquid. The density gradient tube method works on this principle. This method is applicable for a sample having a volume not exceeding 0.5 cm^3 and when the density gradient liquid used for this test does not dissolve, swell or react with the sample.

The density gradient tube is a vertically-placed glass cylinder with graduations, and prepared by injecting two liquids having the different densities while continuously changing the mixing ratio. The relation between the graduation of the density gradient tube and the density should be calibrated using a standard float.

Apparatus

It is shown in Fig. 3.

- Glass cylinder: Use a glass cylinder 1 m in length and having an inside diameter of 4.5 cm, with a stopper on the top, having 1 mm graduation lines along the length of not less than 85 cm and 10 mm graduation lines around the circumference and the figures displayed at intervals of 1, 2 or 5 cm. If appropriate, glass cylinders of different sizes can be used.
- Glass containers: Use two 1000 mL or about 2000 mL glass containers having the same diameter.
- Siphon: Use a siphon as shown in Fig. 3.

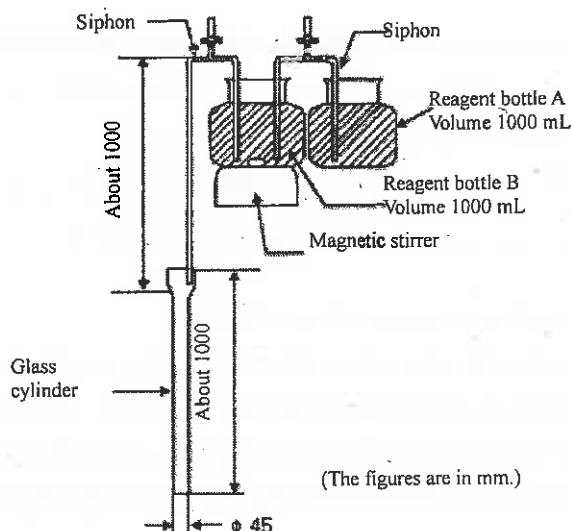


Fig. 3 Density gradient tube

Standard floats

Use hollow glass balls, which are 2 to 5 mm in diameter, the densities of which are accurately determined to the same digit as specified in the required measurement result.

Preparation of density gradient tube

Prepare a density gradient tube as follows.

- To determine the densities ρ_A and ρ_B ($\rho_A < \rho_B$) of two liquids which consist of the density gradient liquid by using the following equation.

$$\rho_A = \rho_B - \frac{2(\rho_B - \rho) \times V_B}{V}$$

ρ_A : Initial density (g/cm^3) of the liquid in glass container A

ρ_B : Initial density (g/cm^3) of the liquid in glass container B

ρ : Density (g/cm^3) of the liquid in the uppermost part of the density gradient tube

V_B : Initial volume (mL) of the liquid in glass container B

V : Total volume (mL) of the liquid in the density gradient tube

When measurements down to the third decimal place are required, prepare a density gradient tube of which density difference between the tube bottom and tube top does not exceed 0.2 g/cm^3 .

- Prepare two liquids having the densities specified in a) according to Tables 3, 4, and 5 and determine their densities according to Methods 1 to 3. Sufficiently deaerated liquid is used. When water is used, it is boiled or decompressed before use.
- Place the liquid of a lower density in the glass container A in Fig. 3 and the liquid of a higher density in the glass container B, make the level of both liquid equal, and connect with a siphon filled with the liquid of a lower density.
- While stirring the liquid in the glass container B with a stirrer, inject the liquid in the container into the glass cylinder along the wall of the cylinder at the rate of 20 mL/min or lower using a siphon.
- Perform procedures b) to d) at the specified temperature $t^\circ\text{C}$.
- Stopper the glass cylinder, place it slowly in a water bath maintained at the specified temperature $t^\circ\text{C}$,

and allow to stand for not less than 1 hour. When measurements down to the third decimal place are required, allow the density gradient tube to stand in a water bath maintained at $t^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$.

- g) Wet a standard float with the liquid in the glass container A, place it gently in the cylinder, stopper it, and use this as the density gradient tube. When measurements down to the third decimal place are required, more than one standard floats are appropriate per density difference of 0.01.
- h) After 24 hours, read the height of the gravity center of the standard float in the density gradient tube to 1 mm from graduations of the density gradient tube, and prepare a calibration curve indicating the relation between the density of the standard float and the graduation of the density gradient tube. The accuracy of the calibration curve should be ± 1 mm.
- i) If the calibration curve is not a smooth curve or markedly curved, repeat the above procedures from a) to h).

Density gradient liquid

A density gradient liquid should not dissolve or swell a solid sample. The two liquids which consist of a density gradient liquid should be compatible. Representative examples of density gradient liquids and the applicable density range are shown in Table 4. Reagents for density adjustment used for various mixtures are shown in Table 5.

Table 4 Examples of density gradient liquids

Liquids	Density range (g/cm^3)
Methanol / Benzyl alcohol	0.80-0.92
2-Propanol / Water	0.79-1.00
2-Propanol / Diethylene glycol	0.79-1.11
Ethanol / Carbon tetrachloride	0.79-1.59
Ethanol / Water	0.79-1.00
Toluene / Carbon tetrachloride	0.87-1.59
Water / Sodium bromide	1.00-1.41
Water / Calcium nitrite	1.00-1.60
Zinc chloride / Ethanol / Water	0.80-1.70
Carbon tetrachloride / 1,3-Dibromopropane	1.60-1.99
1,3-Dibromopropane / Ethylene bromide	1.99-2.18
Ethylene bromide / Bromoform	2.18-2.89
Carbon tetrachloride / Bromoform	1.60-2.89
2-Propanol / Methyl glycol acetate	0.79-1.00

Table 5 Examples of reagents for density adjustment

Reagent name	Density (g/cm^3)
n-Octane	0.70
Dimethylformamide	0.94
Ethane tetrachloride	1.59
Ethyl iodide	1.93

Procedure

Dry the sample sufficiently, prepare 3 samples from the identical sample, and used these samples as the samples for measurement. The samples should be stored in a desiccator with a desiccant. The length of the largest part of each sample should be not more than one-fourth the inside diameter of the density gradient tube and the volume of each sample should be not more than 0.5 cm^3 (usually, $2 \text{ to } 5 \text{ mm} \times 2 \text{ to } 5 \text{ mm}$). If the sample is a chemical fiber, deoil the sample as directed in Method 4, air-dry, and tie in a ring having a diameter of 3 mm.

When multiple samples are put in simultaneously and measured using the existing calibration curve, the size of each sample should be adjusted so that the increased level of liquid surface due to charge-in of all samples should not exceed 1 mm.

Wet three samples with the liquid having a lower density and place them gently in the density gradient tube, taking care to avoid formation of air bubbles. If the samples are fiber, allow the samples in the liquid with a low density to stand under reduced pressure of 0.7 kPa for 5 minutes or centrifuge at $2000 \text{ to } 3000 \text{ min}^{-1}$ for 2 to 3 minutes to expel any entrapped air bubbles.

After the sample reaches equilibrium and exists still, read the level of the gravity center to 1 mm from graduations of the density gradient tube. If the sample contacts with the standard float or the inner wall of the tube, perform the test again. Compare the readings and calibration curve, read the density corresponding to each sample to the same digit number as required accuracy.

Determine the mean density, ρ_T^t of three samples at the specified temperature $t^\circ\text{C}$, designate it as the density of the sample and round off to the same decimal place as that of the calibration density of the standard float. If the density gradient per mm of the density gradient tube exceeds the change in one, the last digit of the calibration density of the standard float, round off to one place higher than that of the calibration density of the standard float. Calculate the specific gravity of the sample d_s^t by using the following equation from the density determined above and the density of water ρ_W^t at $t^\circ\text{C}$ in Table 1, and round off to the same decimal place as that of the density.

$$d_s^t = \frac{\rho_T^t}{\rho_W^t}$$

19. Arsenic Limit Test

Arsenic Limit Test is a limit test for arsenic contained in a sample. The limit is expressed in terms of arsenic (III) trioxide (As_2O_3). In each monograph, the permissible limit for arsenic (as As_2O_3) is described in terms of ppm in parentheses.

Apparatus

Use the apparatus shown in the figure.

Method 2

Unless otherwise specified, weigh 1.0 g of the sample, and place it in a crucible of platinum, quartz, or porcelain. Add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 50), ignite the ethanol, and heat gradually to incinerate. If a carbonized residue still remains, moisten the residue with a little amount of nitric acid, and ignite again to incinerate. After cooling, add 3 mL of hydrochloric acid, heat on a water bath to dissolve the residue, and designate it as the sample solution.

Test solutions

Absorbing solution for hydrogen arsenide: Dissolve 0.50 g of silver N,N-diethyldithiocarbamate in pyridine to make 100 mL. Preserve this solution in a glass-stoppered bottle protected from light, in a cold place.

Standard Arsenic Stock Solution: Weigh accurately 0.100 g of finely powdered arsenic (III) trioxide dried at 105°C for 4 hours, and add 5 mL of sodium hydroxide solution (1 in 5) to dissolve. Add dilute sulfuric acid to neutralize, add further 10 mL of dilute sulfuric acid, and add freshly boiled and cooled water to make exactly 1000 mL.

Standard Arsenic Solution: Pipet 10 mL of Standard Arsenic Stock Solution, add 10 mL of dilute sulfuric acid, and add freshly boiled and cooled water to make exactly 1000 mL. Each mL of the solution contains 1 µg of arsenic (III) trioxide (As_2O_3). Prepare before use and preserve in a glass-stoppered bottle.

Procedure

Unless otherwise specified, proceed using apparatus shown in Figure.

Carry out the preparation of the standard color at the same time.

Place the sample solution in the generator bottle A, and, if necessary, wash down the solution in the bottle with a small quantity of water. Add 1 drop of methyl orange TS, and after neutralizing with ammonia TS, ammonia solution (28), or dilute hydrochloric acid, add 5 mL of diluted hydrochloric acid (1 in 2) and 5 mL of potassium iodide TS, and allow to stand for 2 to 3 minutes. Add 5 mL of acidic tin (II) chloride TS, and allow to stand for 10 minutes. Then add water to make 40 mL, add 2 g of zinc for arsenic analysis, and immediately connect the rubber stopper H fitted with B and C with the generator bottle A. Transfer 5 mL of the absorbing solution for hydrogen arsenide to the absorber tube D, insert the tip of C to the bottom of the absorber tube D, then, immerse the generator bottle A up to the shoulder in water maintained at 25°C and allow to stand for 1 hour. Disconnect the absorber tube, add pyridine to make 5 mL if necessary, and observe the color of the absorbing solution: the color produced is not more intense than the standard color.

Preparation of standard color: Measure accurately 2 mL of Standard Arsenic Solution in the generator bottle A. Add 5 mL of diluted hydrochloric acid (1 in 2) and 5 mL of potassium iodide TS, and allow to stand for 2 to 3 minutes. Add 5 mL of acidic tin (II) chloride TS, allow to stand at room temperature for 10 minutes, and then proceed as directed above. The color produced corresponds to 2 µg of arsenic (III) trioxide (As_2O_3) and is used as the standard.

Note: Apparatus, reagents, and test solutions used in the test should contain little or no arsenic. If necessary, perform a blank determination.

20. Melting Point Determination

The melting point is defined to be the temperature at which a crystalline substance melts during heating, when the solid phase and the liquid phase are in an equilibrium. However, it is conventionally defined to be the temperature at which the remaining solid sample melts completely when it is subjected to continuous heating and the change of the sample state that accompanies heating is accurately observed. Since a pure substance has an intrinsic melting point, it is used for the identification and/or confirmation of a substance and also as an indicator of the purity of a substance.

The melting point is the temperature measured by either of the following methods. Method 1 is applied to those substances of which the purity is comparably high and which can be pulverized, Method 2 to those substances which are insoluble in water and can not be readily pulverized, and Method 3 can be applied to polymer resins or synthetic fibers.

Method 1

This method is applied to those substances of which the purity is comparably high and which can be pulverized.

Apparatus

Use as shown in the figure. Alternatively, apparatus in which some of the procedures, such as stirring, heating, and cooling are automated, can be used.

Bath fluid: Usually use clear silicone oil having a viscosity of 50 to 100 mm²/s at an ordinary temperature.

Thermometer with an immersion line: There are six types of thermometers, Type 1- Type 6, which are specified by an appropriate measuring temperature range. For melting points lower than 50°C, use a thermometer Type 1; for 40°C to 100°C, Type 2; for 90°C to 150°C, Type 3; for 140°C to 200°C, Type 4; for 190°C to 250°C, Type 5; for 240°C to 320°C, Type 6.

Capillary tube: Use a hard glass capillary tube 120 mm long, 0.8 to 1.2 mm in inner diameter and 0.2 to 0.3 mm thick, with one end closed.

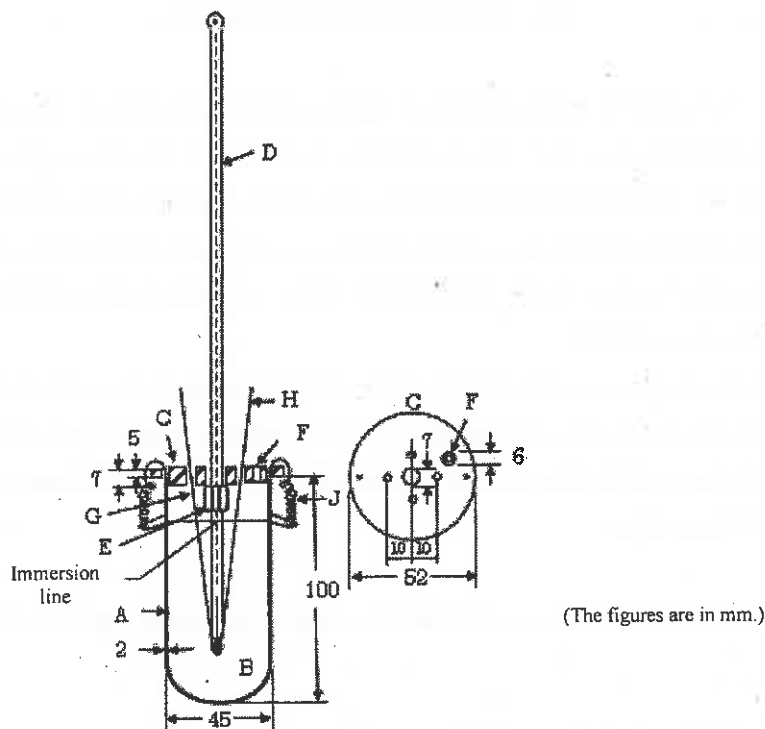


Figure Melting point determination device

- A: Heating vessel of hard glass
- B: Bath fluid
- C: Teflon stopper
- D: Thermometer with an immersion line
- E: Thermometer-fastening spring
- F: Vent for adjustment of the bath fluid volume
- G: Coil spring
- H: Capillary tube
- J: Spring for fastening Teflon stopper

Procedure

Pulverize the sample to a fine powder, and, unless otherwise specified, dry in a desiccator (silica gel) for 24 hours. When it is specified to do the test after drying, dry the sample under the conditions specified in the monograph before measurement.

Place the sample in a dried capillary tube H, and pack it tightly so as to form a layer about 2.5-3.5 mm high by dropping the capillary repeatedly, with the closed end of H down, through a glass tube, about 70 cm long, held vertically on a glass or porous plate.

Heat the bath fluid B until the temperature rises to about 10°C below the expected melting point, place the thermometer D in the bath with the immersion line at the same level as the meniscus of the bath fluid, and insert capillary tube H into a coil spring G so that the packed sample is placed in a position corresponding to the center of the mercury bulb of the thermometer D. Continue heating to raise the temperature at a rate of

approximately 3°C per minute until the temperature rises to 5°C below the expected melting point, then carefully regulate the rate of temperature increase to 1°C per minute.

Read the thermometer indication of the instantaneous temperature at which the sample liquefies completely and no solid is detectable in the capillary, and designate the indicated temperature as the melting point of the sample specimen.

System suitability test

Confirmation of the system suitability of the apparatus should be done periodically by using the Melting Point Standards. The Reference Standard is prepared for the suitability test of the apparatus when it is used with Type 2 - Type 5 thermometers, and consists of 6 highly purified substances: acetanilide, acetophenetidine, caffeine, sulfanilamide, sulfapyridine, and vanillin. The label shows the certified melting points of the respective substances (the end point of the melting change), *MP_f*. After selecting one of the thermometers and the appropriate Melting Point Standard based upon the expected melting point of a sample specimen, perform a melting point measurement of the selected Reference Standard, according to the above procedure. When the value of the obtained melting point of the Reference Standard is within $MP_f \pm 0.5^\circ\text{C}$ in the case of vanillin and acetanilide, within $MP_f \pm 0.8^\circ\text{C}$ in the case of acetophenetidine and sulfanilamide, and within $MP_f \pm 1.0^\circ\text{C}$ in the case of sulfapyridine and caffeine, the apparatus is assumed to be suitable. The above-mentioned measurement is repeated 3 times and the average is determined to be the melting point of the Reference Standard tested.

When the above suitability test criteria are not met in a certain melting point measurement system of an apparatus and a Reference Standard, do the test again, after checking the packing of the sample specimen into the capillary tube, the locations and positioning of the thermometer and the capillary tube, the heating and stirring of the bath fluid, and the control of the temperature increasing rate. When a melting point measurement system does not meet the suitability test criteria again after checking these measuring conditions, the thermometer with an immersion line should be calibrated again or replaced with a new one.

Method 2

This method is applied to substances such as fats, fatty acids, paraffins or waxes.

Apparatus

Instead of the apparatus specified in Method 1, use a water-containing beaker as a bath fluid and a heating vessel. In this measurement, total immersion mercury-filled thermometers can also be used in place of the thermometer with an immersion line. Furthermore, the capillary tube should be the same as specified in Method 1, except that both ends of the tube are open.

Procedure

Carefully melt the sample at as low a temperature as possible, and, taking care to prevent bubbles, introduce it into a capillary tube to a height of about 10 mm. Allow the capillary containing the sample to stand for 24 hours at below 10°C, or for at least 1 hour in contact with ice, holding the capillary so that the sample can not flow out. Then attach the capillary to the thermometer by means of a rubber band so that the absorbed sample is located at a position corresponding to the center of the mercury bulb. Adjust the capillary tube in a water-containing beaker to such a position that the lower edge of the sample is located 30 mm below the water surface. Heat the beaker with constant stirring until the temperature rises to 5°C below the expected melting point. Then regulate the rate of temperature increase to 1°C per minute. The temperature at which the sample begins floating in the capillary is taken as the melting point of the sample specimen.

Method 3

Melting point can be determined by "Thermal Analysis", either "Differential Thermal Analysis (DTA)" or "Differential Scanning Calorimetry (DSC)".

Thermal Analysis is a generic term for a variety of techniques to measure the physical properties of a substance as a function of temperature and/or time.

Among the physical properties, phase transitions such as solid/liquid phase transition (melting, freezing) and crystal polymorphism or thermal behavior such as heat evolution or absorption accompanying thermal degradation or chemical reaction can be detected by the techniques of differential thermal analysis (DTA) or differential scanning calorimetry (DSC).

DTA is a method for detecting the thermal behavior of a specimen in terms of the temperature change, while DSC employs the heat quantity (enthalpy) change.

Apparatus

Apparatus for DTA or DSC is usually composed of a heating furnace, a temperature-controller, a detector, a device for controlling the atmosphere, and an indicator/recorder.

In a DTA apparatus, a sample specimen and an inert reference material placed in the heating furnace are heated or cooled at a constant rate, and the temperature difference evolved between the sample and reference material is detected continuously by a device such as a thermocouple and recorded as a function of time and/or temperature. As an inert reference material, α -Alumina for thermal analysis is usually adopted.

Two kinds of DSC apparatus, based upon different principles are available as shown below.

(1) Input compensation-type differential scanning calorimetry (Input compensation DSC)

A sample specimen and the reference material in twin furnaces are programmed to be heated or cooled at a constant rate, and the temperature difference between the sample and the reference, which is detected by a device such as a platinum resistance thermometer, is kept at null by controlling the heating unit with a compensation feed-back circuit. The instrument is designed to measure and record continuously the balance of thermal energy applied to each furnace as a function of temperature and/or time.

(2) Heat flux-type differential scanning calorimetry (Heat flux DSC)

A sample specimen and the reference material in twin furnaces are programmed to be heated or cooled at a constant rate, and the temperature difference between the sample and the reference is detected as a difference of heat flux and recorded as a function of temperature and/or time. In heat flux DSC, thermal conductors are adopted so that the heat flux between the sample and the heat reservoir is proportional to the temperature difference between them.

In usual DSC analysis, α -Alumina is used as a reference material, both in Input compensation DSC and in Heat flux DSC. But in some cases, an empty sample container can also be used without any reference material.

Procedure

A sample specimen and the reference material are put in sample pans, and the furnace is heated or cooled under a controlled temperature program. As the temperature changes, the temperature difference (DTA) or heat quantity change (DSC) that develops between the specimen and the reference is detected and recorded

continuously. Apparatus equipped with a data-processor is operated according to the instruction manual provided with the instrument.

A preliminary experiment is needed to determine the appropriate temperature range of measurement, within which a predicted physical change such as melting or polymorphic phase transition will occur, and to confirm that unpredicted thermal changes are not induced in a specimen in that temperature range. In this preliminary test, a wide temperature range (room temperature-the temperature at which degradation begins) can be scanned at a rapid heating rate (10-20°C/min). Thereafter, tests by DSC or DTA should be performed at a low heating rate, usually 2°C/min, in the chosen temperature range. However, when a clear heat change cannot be observed, such as in a case of glass-transition, the heating rate may be changed to a higher or a lower rate, as appropriate for the kind of physical change being observed. By analyzing the measured DTA-curve or DSC-curve, a quantity of heat change and/or a specific temperature (ignition, peak or end temperature) that accompanies a physical change, such as melting or polymorphic phase transition, can be obtained.

Calibration of apparatus

(1) Temperature calibration

Temperature calibration for DTA and/or DSC apparatus can be performed by using reference substances having an intrinsic thermal property, such as melting point of pure metals or organic substances, or phase transition point of crystalline inorganic salts or oxides. Melting points of Indium for thermal analysis and/or Tin for thermal analysis are usually employed for calibration.

(2) Heat-quantity calibration for DSC

For accurate estimation of a quantity of heat change (enthalpic change) of a sample specimen, caused by a certain physical change accompanying a temperature change, it is necessary to calibrate the apparatus by using appropriate reference substances. As indicated in the section of Temperature calibration, heat-quantity calibration for DSC apparatus can be performed by using appropriate reference substances having a known definite enthalpic change caused by such physical changes as melting of pure metals and/or organic substances, or phase transition of crystalline inorganic salts. Melting points of Indium for thermal analysis and/or Tin for thermal analysis are usually employed for calibration.

Notes on operating conditions

When DTA or DSC measurements are made, the following items must be recorded: sample size, discrimination of open or closed-type sample container, heating or cooling rate, measuring temperature range, and kind and flow rate of atmospheric gas.

21. Readily Carbonizable Substances Test

Readily Carbonizable Substances Test is a method to examine the minute impurities contained in samples, which are readily colored by addition of sulfuric acid.

Procedure

Use 94.5% to 95.5% sulfuric acid (sulfuric acid for readily carbonizable substances). Before use, wash the Nessler tubes thoroughly with sulfuric acid. Unless otherwise specified, proceed as follows. When the sample

is solid, place 5 mL of sulfuric acid in a Nessler tube, to which add a quantity of the finely powdered sample, little by little, as directed in the monograph, and dissolve it completely by stirring with a glass rod. When the sample is liquid, transfer a volume of the sample, as directed in the monograph, to a Nessler tube, add 5 mL of sulfuric acid, and mix by shaking. If the temperature of the content of the tube rises, cool the content; maintain it at the standard temperature, if the reaction may be affected by the temperature. Allow to stand for 15 minutes, and compare the color of the liquid with that of the matching fluid in the Nessler tube specified in the monograph, by viewing transversely against a white background.

When it is specified to heat the sample with sulfuric acid, transfer the sample and sulfuric acid to a Nessler tube, heat as specified, and compare the colors.

22. Standard Solutions, Matching Fluids for Color, Reference Standards, Reagents, Test Solutions, Measuring Instruments and Appliances

Standard Solutions for Volumetric Analysis are the solutions of reagent with an accurately known concentration, mainly used for the volumetric analysis.

Standard Solutions are used as the standard for the comparison in the tests specified in the JSNM.

Matching Fluids for Color are used as the reference for the comparison of color in the tests specified in the JSNM.

Reference Standards are the substances prepared to a specified purity or quality necessary with regard to their intended use as prescribed in monographs of the JSNM.

Reagents are the substances used for the tests specified in the JSNM. The reagents that are described as "standard reagent", "special class", "first class", "for pH determination", etc. in the JSNM meet the corresponding specifications under the Japan Industrial Standards (JIS). The tests for them shall be performed according to the test methods under the JIS. In the case where the reagent names in the JSNM differ from those of the JIS, the JIS names are given in the brackets. The reagents that are described as "JP reference standard" or "JP monograph" meet the specifications of the corresponding reference standards or monographs. In the case of the reagents that are described merely as test items, the corresponding test methods of the JP are to be applied.

Test Solutions are the solutions prepared for use in the tests of the JSNM.

Measuring Instruments are the instruments or machines used for measuring mass or volume in the tests of the JSNM.

Appliances are the instruments specified in order to make test conditions as consistent as possible in the tests of the JSNM.

(1) Volumetric Standard Solutions (VS)

Hydrochloric Acid, 0.5 mol/L

1000 mL of this solution contains 18.230 g of hydrochloric acid (HCl: 36.46).

Preparation: Dilute 45 mL of hydrochloric acid with water to make 1000 mL, and standardize the solution as follows:

Standardization: Weigh accurately about 0.5 g of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 50 mL of water, and titrate with the prepared hydrochloric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.5 mol/L hydrochloric acid VS = 26.50 mg of Na₂CO₃

Hydrochloric Acid, 0.2 mol/L

1000 mL of this solution contains 7.292 g of hydrochloric acid (HCl: 36.46).

Preparation: Dilute 18 mL of hydrochloric acid with water to make 1000 mL, and standardize the solution as follows:

Standardization: Weigh accurately about 0.15 g of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 30 mL of water, and titrate the solution with the prepared hydrochloric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.2 mol/L hydrochloric acid VS = 10.60 mg of Na₂CO₃

Hydrochloric Acid, 0.1 mol/L

1000 mL of this solution contains 3.6461 g of hydrochloric acid (HCl: 36.46).

Preparation: Before use, dilute 0.2 mol/L hydrochloric acid VS with water to make exactly 2 times the initial volume.

Each mL of 0.1 mol/L hydrochloric acid VS = 5.300 mg of Na₂CO₃

Hydrochloric Acid, 0.01 mol/L

1000 mL of this solution contains 0.36461 g of hydrochloric acid (HCl: 36.46).

Preparation: Before use, dilute 0.2 mol/L hydrochloric acid VS with water to make exactly 20 times the initial volume.

Potassium Permanganate, 0.02 mol/L

1000 mL of this solution contains 3.1607 g of potassium permanganate (KMnO₄: 158.03).

Preparation: Dissolve 3.2 g of potassium permanganate in water to make 1000 mL, and boil the solution for 15 minutes. Allow the solution to stand for at least 48 hours in a tightly stoppered flask, and filter it through a glass filter (G3 or G4). Standardize the solution as follows:

Standardization: Weigh accurately about 0.3 g of sodium oxalate (standard reagent), previously dried between 150°C and 200°C for 1 to 1.5 hours and allowed to cool in a desiccator (silica gel), transfer it to a 500 mL conical flask, dissolve in 30 mL of water, add 250 mL of diluted sulfuric acid (1 in 20), and warm the mixture between 30°C and 35°C. Transfer the prepared potassium permanganate solution to a burette, add quickly 40 mL of the solution under gentle stirring from the burette, and allow to stand until the red color of the mixture disappears. Warm the solution between 55°C and 60°C, and complete the titration with the potassium permanganate solution until a faint red color persists for 30 seconds. Add the last 0.5 to 1 mL dropwise before the end point, being particularly careful to allow the solution to be decolorized before the next drop is added. Calculate the molarity factor.

Each mL of 0.02 mol/L potassium permanganate VS = 6.700 mg of Na₂C₂O₄

Note: Store protected from light. This solution, if stored for a long period, should be restandardized.

Potassium Permanganate, 0.002 mol/L

1000 mL of this solution contains 0.31607 g of potassium permanganate (KMnO₄: 158.03).

Preparation: Before use, dilute 0.02 mol/L potassium permanganate VS with water to make exactly 10 times the initial volume.

Potassium Hydroxide, 0.1 mol/L

1000 mL of this solution contains 5.611 g of potassium hydroxide (KOH: 56.11).

Preparation: Dissolve 6.5 g of potassium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Shake the mixture thoroughly, and allow it to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 0.25 g of amidosulfuric acid (sulfamic acid)(standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly boiled and cooled water, and add 2 drops of bromothymol blue TS. Titrate with the prepared potassium hydroxide solution until it acquires a green color. Calculate the molarity factor.

Each mL of 0.1 mol/L potassium hydroxide VS =9.709 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Potassium Hydroxide-Ethanol, 0.5 mol/L

1000 mL of this solution contains 28.055 g of potassium hydroxide (KOH: 56.11).

Preparation: Dissolve 35 g of potassium hydroxide in 20 mL of water, and add aldehyde-free ethanol to make 1000 mL. Allow the solution to stand for 24 hours in a tightly stoppered bottle. Then quickly decant the supernatant liquid, and standardize the solution as follows:

Standardization: Measure exactly 25 mL of 0.25 mol/L sulfuric acid VS, add 50 mL of water, and titrate with the prepared potassium hydroxide-ethanol solution to calculate the molarity factor (indicator: 2 drops of phenolphthalein TS). In the indicator method, titrate until the solution acquires a light red color.

Note: Store in tightly stoppered bottles, protected from light. Standardize before use.

Potassium Hydroxide-Ethanol, 0.1 mol/L

1000 mL of this solution contains 5.611 g of potassium hydroxide (KOH: 56.11).

Preparation: Dissolve 7 g of potassium hydroxide in 20 mL of water, and add aldehyde-free ethanol to make 1000 mL. Allow the solution to stand for 24 hours in a tightly stoppered bottle. Then quickly decant the supernatant liquid, and standardize the solution as follows:

Standardization: Measure exactly 15 mL of 0.05 mol/L sulfuric acid VS, add 50 mL of water, and titrate with the prepared potassium hydroxide-ethanol solution to calculate the molarity factor (indicator: 2 drops of phenolphthalein TS). In the indicator method, titrate until the solution acquires a light red color.

Note: Store in tightly stoppered bottles, protected from light. Standardize before use.

Sodium Hydroxide, 1 mol/L

1000 mL of this solution contains 39.997 g of sodium hydroxide (NaOH: 40.00).

Preparation: Dissolve 42 g of sodium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Mix well the mixture, and allow to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 1.5 g of amidosulfuric acid (sulfamic acid)(standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly

boiled and cooled water, and titrate the solution with the prepared sodium hydroxide solution to calculate the molarity factor (indicator method: 2 drops of bromothymol blue TS). In the indicator method, titrate until the solution acquires a green color.

Each mL of 1 mol/L sodium hydroxide VS = 97.09 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Sodium Hydroxide, 0.5 mol/L

1000 mL of this solution contains 19.999 g of sodium hydroxide (NaOH: 40.00).

Preparation: Dissolve 22 g of sodium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Mix well the mixture, and allow to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 0.7 g of amidosulfuric acid (sulfamic acid) (standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly boiled and cooled water, and titrate the solution with the prepared sodium hydroxide solution to calculate the molarity factor (indicator method: 2 drops of bromothymol blue TS). In the indicator method, titrate until the solution acquires a green color.

Each mL of 0.5 mol/L sodium hydroxide VS = 48.55 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Sodium Hydroxide, 0.1 mol/L

1000 mL of this solution contains 3.9997 g of sodium hydroxide (NaOH: 40.00).

Preparation: Dissolve 4.5 g of sodium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Mix well the mixture, and allow to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 0.15 g of amidosulfuric acid (sulfamic acid) (standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly boiled and cooled water, and titrate the solution with the prepared sodium hydroxide solution to calculate the molarity factor (indicator method: 2 drops of bromothymol blue TS). In the indicator method, titrate until the solution acquires a green color.

Each mL of 0.1 mol/L sodium hydroxide VS = 9.709 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Sodium Thiosulfate, 0.2 mol/L

1000 mL of this solution contains 49.636 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$: 248.18).

Preparation: Dissolve 52 g of sodium thiosulfate and 0.2 g of anhydrous sodium carbonate in freshly boiled and cooled water to make 1000 mL, and standardize the solution as follows:

Standardization: Weigh accurately about 0.2 mg of potassium iodate (standard reagent), previously dried

between 120°C and 140°C for 2 hours and allowed to cool in a desiccator (silica gel), and transfer to an iodine flask. Dissolve it in 25 mL of water, add 4 g of potassium iodide and 10 mL of dilute sulfuric acid, and stopper the flask. After allowing the mixture to stand for 10 minutes, add 100 mL of water, and titrate the liberated iodine with the prepared sodium thiosulfate solution to calculate the molarity factor. When the solution assumes a pale yellow color as the end point is approached, add 3 mL of starch TS. Continue the titration until the blue color disappears. Perform a blank determination in the same manner, and make any necessary correction.

Each mL of 0.2 mol/L sodium thiosulfate VS = 7.133 mg of KIO₃

Note: This solution, if stored for a long period, should be restandardized.

Sodium Thiosulfate, 0.1 mol/L

1000 mL of this solution contains 24.818 g of sodium thiosulfate pentahydrate (Na₂S₂O₃·5H₂O: 248.18).

Preparation: Before use, dilute 0.2 mol/L sodium thiosulfate VS with freshly boiled and cooled water to make 2 times the initial volume.

Sodium Thiosulfate, 0.01 mol/L

1000 mL of this solution contains 2.4818 g of sodium thiosulfate pentahydrate (Na₂S₂O₃·5H₂O: 248.18).

Preparation: Before use, dilute 0.2 mol/L sodium thiosulfate VS with freshly boiled and cooled water to make 20 times the initial volume.

Sulfuric Acid, 0.25 mol/L

1000 mL of this solution contains 24.520 g of sulfuric acid (H₂SO₄: 98.08).

Preparation: Add slowly, under stirring, 15 mL of sulfuric acid to 1000 mL of water, allow to cool, and standardize the solution as follows:

Standardization: Weigh accurately about 0.4 g of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 50 mL of water, and titrate with the prepared sulfuric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.25 mol/L sulfuric acid VS= 26.50 mg of Na₂CO₃

Sulfuric Acid, 0.05 mol/L

1000 mL of this solution contains 4.904 g of sulfuric acid (H₂SO₄: 98.08).

Preparation: Add slowly, under stirring, 3 mL of sulfuric acid to 1000 mL of water, allow to cool, and standardize the solution as follows:

Standardization: Weigh accurately about 80 mg of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 30 mL of water, and titrate the solution with the prepared sulfuric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.05 mol/L sulfuric acid VS= 5.300 mg of Na_2CO_3

(2) Standard Solutions

Borate pH Standard Solution See pH Determination.

Calcium Hydroxide pH Standard Solution See pH Determination.

Carbonate pH Standard Solution See pH Determination.

Oxalate pH Standard Solution See pH Determination.

Phosphate pH Standard Solution See pH Determination.

Phthalate pH Standard Solution See pH Determination.

Standard Arsenic Solution Pipet 10 mL of Standard Arsenic Stock Solution, add 10 mL of dilute sulfuric acid, and add freshly boiled and cooled water to make exactly 1000 mL. Each mL of the solution contains 1 μg of arsenic (III) trioxide (As_2O_3). Prepare before use and preserve in a glass-stoppered bottle.

Standard Arsenic Stock Solution Weigh accurately 0.100 g of finely powdered arsenic (III) trioxide dried at 105°C for 4 hours, and add 5 mL of sodium hydroxide solution (1 in 5) to dissolve. Add dilute sulfuric acid to neutralize, add further 10 mL of dilute sulfuric acid, and add freshly boiled and cooled water to make exactly 1000 mL.

Standard Iron Solution Weigh exactly 86.3 mg of ammonium iron (III) sulfate dodecahydrate, dissolve in 100 mL of water, and add 5 mL of dilute hydrochloric acid and water to make exactly 1000 mL. Each mL of this solution contains 0.01 mg of iron (Fe).

Standard Lead Solution Measure exactly 10 mL of Standard Lead Stock Solution, and add water to make exactly 100 mL. Prepare before use. Each mL of this solution contains 0.01 mg of lead (Pb).

Standard Lead Stock Solution Weigh exactly 159.8 mg of lead (II) nitrate, dissolve in 10 mL of dilute nitric acid, and add water to make exactly 1000 mL. Prepare and store this solution using glass containers, free from soluble lead salts.

Standard Liquids for Calibrating Viscosimeters [JIS, Standard Liquids for Calibrating Viscosimeters (Z8809)]

Standard Lithium Solution for Atomic Absorption Spectrophotometry Weigh exactly 6.107 g of lithium chloride (anhydrous) and dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 1000 mL. Each mL of this solution contains 1.00 mg of lithium (Li).

Standard Vinyl Chloride Solution Transfer ethanol to a 200-mL volumetric flask in a volume of 2 to 3 mL smaller than the marked volume, stopper with a silicone rubber stopper and measure the mass. Inject about 200 mg of liquefied vinyl chloride through the silicone rubber stopper to dissolve in ethanol. Then, measure the weight, determine the increase in mass exactly, and define this value as "a." Add ethanol to make exactly 200 mL through the silicone rubber stopper, shake well to make uniform, and use this solution as the standard vinyl chloride stock solution. Cool the standard vinyl chloride stock solution in a methanol-dry ice bath, transfer 95 mL of cooled ethanol in a 100-mL volumetric flask in the same manner, add 1 mL of the standard stock solution while cooling, and allow to stand to room temperature. Then, add ethanol to make 100 mL, and use this solution as the vinyl chloride standard solution. The concentration of the standard stock solution is

$a/(100 \times 1.0567)$ ppm. Dilute the standard solution with ethanol to prepare standard solutions for calibration curve with a concentration of 0.1 to 10 ppm.

(3) Matching Fluids for Color

Cobaltous Chloride Colorimetric Stock Solution

Weigh 65 g of cobaltous chloride (cobalt (II) chloride hexahydrate), and dissolve in 25 mL of hydrochloric acid and water to make 1000 mL. Pipet 5 mL of this solution into a 250-mL iodine flask. Add 5 mL of hydrogen peroxide TS and 15 mL of a solution of sodium hydroxide (1 in 5), and boil for 10 minutes. Cool, and add 2 g of potassium iodide and 20 mL of diluted sulfuric acid (1 in 4). When the precipitate dissolves, titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 23.793 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$

According to the titration value, add diluted hydrochloric acid (1 in 40) to make a solution containing 59.5 mg of cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$: 237.93) in each mL, and use this solution as the colorimetric stock solution.

Copper Sulfate Colorimetric Stock Solution

Weigh 65 g of copper sulfate (copper (II) sulfate pentahydrate), and dissolve in 25 mL of hydrochloric acid and water to make 1000 mL. Pipet 10 mL of this solution into an iodine flask. Add 4 mL of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1 mol/L sodium thiosulfate (indicator: 1 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 24.968 mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

According to the titrated value, add diluted hydrochloric acid (1 in 40) to make a solution containing 62.4 mg of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$: 249.68) in each mL, and use this solution as the colorimetric stock solution.

Ferric Chloride Colorimetric Stock Solution

Weigh 55 g of ferric chloride (iron (III) chloride hexahydrate), and dissolve in 25 mL of hydrochloric acid and add water to make 1000 mL. Measure exactly 10 mL of this solution, transfer to an iodine flask, add 15 mL of water and 3 g of potassium iodide, stopper tightly, and allow to stand in a dark place for 15 minutes. Add 100 mL of water to the mixture, and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 27.03 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

According to the titrated value, add diluted hydrochloric acid (1 in 40) to make a solution containing 45.0 mg of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$: 270.30) in each mL, and use this solution as the colorimetric stock solution.

(4) Reference Standards, Reagents and Test Solutions (TS)

Acetanilide reference standard for melting point determination [JP Acetanilide reference standard for melting point determination]

Acetic acid 2-methoxyethyl $\text{CH}_3\text{COOCH}_2\text{CH}_2\text{OCH}_3$

Melting point: -65.1°C , Boiling point: 145°C

Acetic acid, dilute Dilute 6 g of acetic acid (100) with water to make 100 mL (1 mol/L).

Acetic acid, glacial See acetic acid (100).

Acetic acid (100) CH_3COOH [K8355, Acetic acid, Special class]

Acetic anhydride $(\text{CH}_3\text{CO})_2\text{O}$ [K8886, Special class]

Acetone CH_3COCH_3 [K8034, Special class]

Acetonitrile CH_3CN [K8032, Special class]

Acetonitrile for liquid chromatography CH_3CN Colorless clear liquid. Mixable with water.

Purity Ultraviolet light absorbing substances: Determine the absorbances at the following wavelengths as directed under Ultraviolet-visible Spectrophotometry, using water as the control: not more than 0.07 at 200 nm, not more than 0.046 at 210 nm, not more than 0.027 at 220 nm, not more than 0.014 at 230 nm and not more than 0.009 at 240 nm.

Acetophenetidine reference standard for melting point determination [JP Acetophenetidine reference standard for melting point determination]

Acetylene See dissolved acetylene.

Acidic stannous chloride TS See tin (II) chloride TS, acidic.

Acidic tin (II) chloride TS See tin (II) chloride TS, acidic.

Acrylic acid reference standard Acrylic acid (Special class). It contains not be less than 99.0% (Acrylic acid, $\text{C}_3\text{H}_4\text{O}_2$:72.06).

Assay: Weigh accurately about 1 g of this substance, add 20 mL of water, mix, and titrate with 1 mol/L of sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS).

Each mL of 1 mol/L sodium hydroxide VS = 72.06 mg $\text{C}_3\text{H}_4\text{O}_2$

Aldehyde-free ethanol See ethanol, aldehyde-free.

Alizarin S See alizarin red S.

Alizarin S TS See alizarin red S TS.

Alizarin red S $\text{C}_{14}\text{H}_7\text{NaO}_7\text{S}$ [K8057, Special class] Range of color change: pH (yellow) 3.7-5.2 (orange).

Alizarin red S TS Dissolve 0.1 g of alizarin red S in water to make 100 mL, and filter.

α -Alumina for thermal analysis $\alpha\text{-Al}_2\text{O}_3$ Prepared for thermal analysis.

Amidosulfuric acid (standard reagent) HOSO_2NH_2 [K8005, Amidosulfuric acid, Standard reagent for volumetric analysis] or [K8587, Special class]

Ammonia copper TS To 0.5 g of cupric carbonate monohydrate add 10 mL of water, triturate, and add 10 mL of ammonia solution (28).

Ammonia TS To 400 mL of ammonia solution (28) add water to make 1000 mL (10%).

Ammonium chloride NH_4Cl [K8116, Special class]

Ammonium chloride TS Dissolve 10.5 g of ammonium chloride in water to make 100 mL (2 mol/L).

Ammonium iron (III) sulfate dodecahydrate $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ [K8982, Ammonium iron (III) sulfate dodecahydrate, Special class]

Ammonium peroxodisulfate $(\text{NH}_4)_2\text{S}_2\text{O}_8$ [K8252, Special class]

Ammonium persulfate See ammonium peroxodisulfate.

Ammonium sodium hydrogenphosphate See ammonium sodium hydrogenphosphate tetrahydrate.

Ammonium sodium hydrogenphosphate tetrahydrate $\text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$ [K9013, Special class]

Ammonia solution (28) NH_3 [K8085, Ammonia Water, Special class, Specific gravity: about 0.90, Density:

0.908 g/mL, Content: 28–30%]

Ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$ [K8960, Special class]

Ammonium thiocyanate NH_4SCN [K9000, Special class]

Ammonium thiocyanate-cobalt nitrate TS See ammonium thiocyanate-cobalt (II) nitrate TS.

Ammonium thiocyanate-cobalt (II) nitrate TS Dissolve 17.4 g of ammonium thiocyanate and 2.8 g of cobalt (II) nitrate hexahydrate in water to make 100 mL.

Ammonium thiocyanate TS Dissolve 8 g of ammonium thiocyanate in water to make 100 mL (1 mol/L).

Anhydrous ethanol See ethanol (99.5).

Anhydrous sodium carbonate See sodium carbonate, anhydrous.

Anhydrous sodium sulfate See sodium sulfate, anhydrous.

Arsenic (III) trioxide As_2O_3 [K8044, Arsenic (III) trioxide, Special class]

Arsenic trioxide See arsenic (III) trioxide.

Barium chloride See barium chloride dihydrate.

Barium chloride dehydrate $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ [K8155, Special class]

Barium chloride TS Dissolve 12 g of barium chloride dihydrate in water to make 100 mL (0.5 mol/L).

Barium hydroxide See barium hydroxide octahydrate.

Barium hydroxide octahydrate $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ [K8577, Special class] Preserve in tightly stoppered bottles.

Benzene C_6H_6 [K8858, Special class]

Benzyl alcohol $\text{C}_6\text{H}_5\text{CH}_2\text{OH}$ Colorless liquid, having a faint, characteristic odor.

Specific gravity: d_{20}^{20} , 1.042 - 1.053, Purity: not less than 97.0%

Blue litmus paper See litmus paper, blue.

Boric acid H_3BO_3 [K8863, Boric acid, Special class]

Bromothymol blue $\text{C}_{27}\text{H}_{28}\text{Br}_2\text{O}_5\text{S}$ [K8842, Special class] Range of color change: pH (yellow) 6.0-7.6 (blue)

Bromothymol blue TS Dissolve 0.1 g of bromothymol blue in 100 mL of dilute ethanol, and filter if necessary.

Bromoform CHBr_3 (another name: tribromomethane) Colorless liquid. Preserve in a dark, cold place.

Melting point: -7°C , boiling point: $149 - 152^\circ\text{C}$, specific gravity: d^{15} 2.89

1-Butanol $\text{CH}_3(\text{CH}_2)_2\text{CH}_2\text{OH}$ [K8810, Special class]

n-Butanol See 1-butanol.

Caffeine reference standard for melting point determination [JP Caffeine reference standard for melting point determination]

Calcium chloride See calcium chloride dihydrate.

Calcium chloride dihydrate $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ [K8122, Special class]

Calcium chloride TS Dissolve 7.5 g of calcium chloride dihydrate in water to make 100 mL (0.5 mol/L).

Calcium hydroxide $\text{Ca}(\text{OH})_2$ [K8575, Special class]

Calcium hydroxide for pH determination [K8575, Special class] Calcium hydroxide prepared for pH determination. Use the saturated solution obtained at $23-27^\circ\text{C}$ of which pH is 12.45 at 25°C .

Calcium hydroxide TS To 3 g of calcium hydroxide add 1000 mL of cold distilled water, and occasionally shake the mixture vigorously for 1 hour. Allow to stand, and use the supernatant liquid (0.04 mol/L).

Calcium nitrite See calcium nitrate tetrahydrate.

Calcium nitrate tetrahydrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ [K8549, Special class]

Carbon tetrachloride CCl_4 [K8459, Special class]

Catechol $\text{C}_6\text{H}_4(\text{OH})_2$ White to gray granules or crystals. It is odorless or has a faint, characteristic odor. Melting point: 103°C - 107°C , Purity: not less than 98.0%.

Chloroform CHCl_3 [K8322, Special class]

Cobalt (II) chloride hexahydrate $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ [K8129, Special class]

Cobalt (II) nitrate hexahydrate $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [K8552, Special class]

Cobalt chloride See cobalt (II) chloride hexahydrate.

Cobaltus chloride See cobalt (II) chloride hexahydrate.

Cobaltous nitrate See cobalt (II) nitrate hexahydrate.

Copper (II) sulfate pentahydrate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ [K8983, Special class]

Cupric carbonate See cupric carbonate monohydrate.

Cupric carbonate monohydrate $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$ A blue to blue-green powder. It is insoluble in water, and dissolves foamingly in dilute acid. It dissolves in ammonia TS and shows a deep blue color.

Purity

(1) Chloride: Not more than 0.036%.

(2) Sulfate: Not more than 0.120%.

(3) Iron: Dissolve 5.0 g of this substance in excess ammonia TS and filter. Wash the residue with ammonia TS, dissolve in dilute hydrochloric acid, add excess ammonia TS and filter. Wash the residue with ammonia TS, and dry to constant mass: the residue is not more than 10 mg.

Cupric sulfate See copper (II) sulfate pentahydrate.

Dibasic sodium phosphate, anhydrous, for pH determination See disodium hydrogen phosphate for pH determination.

Dibenzylamine $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{NH}$ A colorless or yellowish clear liquid, practically insoluble in water, soluble in alcohol and ethanol. Purity: not less than 98.0%.

Specific gravity: 1.027-1.032.

1,2-Dibromoethane $\text{BrH}_2\text{CCH}_2\text{Br}$ Colorless liquid having a characteristic odor.

Melting point: 9°C - 10°C , Boiling point: 131°C - 132°C , d^0 2.21; d^{20} 2.18

1,3-Dibromopropane $\text{Br}(\text{CH}_2)_3\text{Br}$

Diethylene glycol $\text{HO}(\text{CH}_2\text{CH}_2\text{O})_2\text{H}$ Colorless and odorless liquid. Miscible with water and with ethanol (95).

Specific gravity: d_{20}^{20} 1.118-1.120

Diethyl ether $\text{C}_2\text{H}_5\text{OC}_2\text{H}_5$ [K8103, Special class]

Dilute acetic acid See acetic acid, dilute.

Dilute ethanol See ethanol, dilute.

Dilute hydrochloric acid See hydrochloric acid, dilute.

Dilute methylene blue TS See methylene blue TS, dilute.

Dilute nitric acid See nitric acid, dilute.

Dilute sulfuric acid See sulfuric acid, dilute.

N,N-Dimethylacetamide $\text{CH}_3\text{CON}(\text{CH}_3)_2$ A clear, colorless liquid.

Boiling point: 163-165°C, Specific gravity: 0.938-0.945, Water content: not more than 0.2% (0.1 g, Coulometric titration).

Purity: Perform the test with 3 µL of this substance as directed under Gas Chromatography according to the following conditions, and measure the area of each peak using automatic integration. Calculate the amount of N,N-Dimethylacetamide by the area percentage method: not less than 98.0%.

Operating conditions

Detector: Hydrogen flame-ionization detector

Column: A fused silica column, 0.25 mm in the inside diameter and 30 m in length, coated with polyethylene glycol 20M for gas chromatography 0.5 µm in thickness

Column temperature: The sample is injected at a constant temperature of about 70°C, keep this temperature for 1 minute, then raise to 200°C in a rate of 10°C per minute, and keep 200°C for 3 minutes.

Carrier gas: Helium

Flow rate (linear velocity): About 30 cm/sec

Time span of measurement: About 2 times as long as the retention time of N, N-dimethylacetamide.

p-Dimethylaminobenzaldehyde See 4-dimethylaminobenzaldehyde.

4-Dimethylaminobenzaldehyde (CH₃)₂NC₆H₄CHO [K8496, p-Dimethylaminobenzaldehyde, Special class]

N,N-Dimethylformamide HCON(CH₃)₂ [K8500, Special class]

Dimethylformamide See N,N-dimethylformamide.

Dimethylsulfoxide (CH₃)₂SO [K9702, Special class]

Dimethylsulfoxide for ultraviolet-visible spectrophotometry Colorless crystals or clear colorless liquid, having a characteristic odor.

It is highly hygroscopic.

Congealing point: Not less than 18.3°C

Purity: Read absorbance of this substance, immediately after saturating with nitrogen, using water as the blank as directed under Ultraviolet-visible Spectrophotometry: its value is not more than 0.20 at 270 nm, not more than 0.09 at 275 nm, not more than 0.06 at 280 nm, and not more than 0.015 at 300 nm. It exhibits no characteristic absorption between 260 nm and 350 nm.

Water content: Not more than 0.1.

Diphenylmethane diisocyanate C₁₅H₁₀O₂N₂ White to pale yellow solid. Soluble in benzene, toluene, acetone, and ether.

Specific gravity: d_4^{20} 1.19, Boiling point: 190°C (665 Pa)

Disodium hydrogen phosphate dodecahydrate Na₂HPO₄·12H₂O [K9019, Disodium hydrogen phosphate dodecahydrate, Special class]

Disodium hydrogen phosphate for pH determination Na₂HPO₄ [K9020, Disodium hydrogen phosphate, for pH standard solution]

Dissolved acetylene C₂H₂ [K1902]

Epichlorohydrin C₃H₅ClO Colorless liquid, having a pungent odor similar to chloroform, specific gravity of 1.1801, and boiling point of 117°C. Purity: not less than 99%.

Ethanol See ethanol (95).

Ethanol, aldehyde-free Transfer 1000 mL of ethanol (95) to a glass-stoppered bottle, add the solution prepared by dissolving 2.5 g of lead (II) acetate trihydrate in 5 mL of water, and mix thoroughly. In a separate

container, dissolve 5 g of potassium hydroxide in 25 mL of warm ethanol (95), cool, and add this solution gently, without stirring, to the first solution. After 1 hour, shake this mixture vigorously, allow to stand overnight, decant the supernatant liquid, and distil the ethanol.

Ethanol, anhydrous See ethanol (99.5).

Ethanol, dilute To 1 volume of ethanol (95) add 1 volume of water. C_2H_5OH Content: 47.45 to 50.00 vol%

Ethanol (95) C_2H_5OH [K8102, Special class]

Ethanol (99.5) C_2H_5OH [K8101, Special class]

Ether See diethyl ether.

Ethyl iodine See iodoethane.

Ethylene bromide See 1,2-dibromoethane.

Ferric ammonium sulfate See ammonium iron (III) sulfate dodecahydrate.

Ferric chloride See iron (III) chloride hexahydrate.

Fluorescence reference standard (1) Weigh exactly 7 mg of fluorescent brightener (Fluorescent-24) in a brown volumetric flask, add water to make exactly 500 mL, and store protected from light.

To 5 mL of this solution, add 0.1 g of sodium chloride and water to make 50 mL, immerse a filter paper (8 cm × 15 cm) into the resultant solution for 1 hour with occasional inverting and air-dry it on a filter paper.

Fluorescence reference standard (2) Weigh exactly 7 mg of fluorescent brightener (Fluorescent-351) in a brown volumetric flask, add water to make exactly 500 mL, and store protected from light. To 0.35 mL of this solution, add 0.1 g of sodium chloride and water to make 50 mL, immerse a filter paper (8 cm × 15 cm) into the resultant solution for 1 hour with occasional inverting and air-dry it on a filter paper.

Fluorescent-24 $C_{40}H_{44}N_{12}Na_4O_{16}S_4$ Delayed-staining stilbene fluorescent brightener. Color index name: C.I. Fluorescent brightener-24 (C.I. No. 40650), Chemical name: Tetrasodium 4,4'-bis[[4-[bis(2-hydroxyethyl)amino]-6-[(3-sulphonato-phenyl)amino]-1,3,5-triazine-2-yl]amino]stilbene-2,2'-disulfonate (CAS No. 12224-02-1)

Fluorescent-351 $C_{28}H_{20}Na_2O_6S_2$ Stilbene fluorescent brightener Color index name: C.I. Fluorescent brightener-351 (FWA-5)(C.I.No.482200), Chemical name: Disodium 4,4'-Bis(2-sulfonatostyryl)biphenyl (CAS No. 27344-41-8)

Gauze Use medical gauze conforming to the requirements of Notification No. 0630001 of Secretary-General of Pharmaceutical and Food Safety Bureau, the MHLW dated June 30, 2005.

Glycerin $C_3H_8O_3$ [K8295, Special class] or [Concentrated Glycerin (JP monograph)]

Helium He, not less than 99.995 vol%

Heptane $CH_3(CH_2)_5CH_3$ [K9701, Special class]

n-Heptane See heptane.

Hexane for ultraviolet-visible spectrophotometry [K8848, Special class] When determining the absorbance of hexane as directed under Ultraviolet-visible Spectrophotometry, using water as the blank: not more than 0.10 at 220 nm and not more than 0.02 at 260 nm. It exhibits no absorption between 260 nm and 350 nm.

n-Hexane for ultraviolet-visible spectrophotometry See hexane for ultraviolet-visible spectrophotometry.

Hydrochloric acid HCl [K8180, Special class]

Hydrochloric acid, dilute Dilute 23.6 mL of hydrochloric acid with water to make 100 mL (10%).

0.1 mol/L Hydrochloric acid TS Dilute 100 mL of hydrochloric acid with water to make 1000 mL.

1 mol/L Hydrochloric acid TS Dilute 90 mL of hydrochloric acid with water to make 1000 mL.

Hydrogen peroxide (30) H_2O_2 [K8230, Hydrogen peroxide, Special class, Concentration: 30.0- 35.5%]

Hydrogen peroxide TS Dilute 1 volume of hydrogen peroxide (30) with 9 volumes of water. Prepare before use (3%).

Hydrogen peroxide water, strong See hydrogen peroxide (30).

Hydrogen sulfide H_2S Colorless, poisonous gas, heavier than air. It dissolves in water. Prepare by treating iron (II) sulfide with dilute sulfuric acid or dilute hydrochloric acid. Other sulfides yielding hydrogen sulfide with dilute acids may be used.

Hydroxylamine hydrochloride See hydroxylammonium chloride.

Hydroxylammonium chloride $\text{NH}_2\text{OH}\cdot\text{HCl}$ [K8201, Special class]

Hydroxylammonium hydrochloride See hydroxylammonium chloride.

Indium for thermal analysis Prepared for thermal analysis. Content: not less than 99.99%.

Iodine I [K8920, Iodine, Special class]

Iodine solution See iodine TS (0.05 mol/L).

Iodine TS Dissolve 14 g of iodine in 100 mL of a solution of potassium iodide (2 in 5), add 1 mL of dilute hydrochloric acid, and dilute with water to make 1000 mL (0.05 mol/L).

Storage: Preserve in light-resistant containers.

Iodoethane $\text{C}_2\text{H}_5\text{I}$ A colorless to a dark-brown, clear liquid, having diethyl ether-like odor. Distilling range 71.0-72.5°C, not less than 94 vol%.

Iron (III) chloride hexahydrate $\text{FeCl}_3\cdot 6\text{H}_2\text{O}$ [K8142, Special class]

Iron (II) sulfide FeS [K8948, for hydrogen sulfide development]

Isopropanol See 2-propanol.

Isopropyl alcohol See 2-propanol.

Isophorone diisocyanate $\text{C}_{12}\text{H}_{18}\text{N}_2\text{O}_2$ Colorless viscous liquid.

Isotonic sodium chloride solution [Isotonic Sodium Chloride Solution (JP monograph)]

Lead acetate See lead (II) acetate trihydrate.

Lead (II) acetate trihydrate $\text{Pb}(\text{CH}_3\text{COO})_2\cdot 3\text{H}_2\text{O}$ [K8374, Lead (II) acetate trihydrate, Special class]

Lead (II) acetate TS To 9.5 g of lead (II) acetate trihydrate add freshly boiled and cooled water to make 100 mL (0.25 mol/L). Preserve in tightly stoppered bottles.

Lead monoxide See lead (II) oxide.

Lead nitrate See lead (II) nitrate.

Lead (II) nitrate $\text{Pb}(\text{NO}_3)_2$ [K8563, Special class]

Lead (II) oxide PbO [K8090, Special class]

Lithium chloride LiCl White crystals or masses.

Identification: Perform the test as directed under Flame Coloration Test with this substance: a persistent red color develops.

Lithium chloride, anhydrous See lithium chloride.

Litmus paper, blue [K9071, Litmus paper, Blue litmus paper]

Litmus paper, red [K9071, Litmus paper, Red litmus paper]

Magnesium nitrate See magnesium nitrate hexahydrate.

Magnesium nitrate hexahydrate $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [K8567, Special class]

Magnesium sulfate See magnesium sulfate heptahydrate.

Magnesium sulfate heptahydrate $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ [K8995, Special class]

Magnesium sulfate TS Dissolve 12 g of magnesium sulfate heptahydrate in water to make 100 mL (0.5 mol/L).

Methanol CH_3OH [K8891, Special class]

Methylene blue $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S} \cdot 3\text{H}_2\text{O}$ [K 8897, Special class]

Methylene blue TS Dissolve 0.1g of methylene blue in 100 mL of ethanol, and filter if necessary.

Methylene blue TS, dilute Dilute 1 mL of methylene blue TS with water to make 100 mL.

Methylene iodide CH_2I_2 (another name: Diiodomethane) Yellow liquid.
Melting point: 6°C, boiling point: 181°C, specific gravity: d_{4}^{15} 3.3326

Methyl glycol acetate See acetic acid 2-methoxyethyl.

Methyl orange $\text{C}_{14}\text{H}_{14}\text{N}_3\text{NaO}_3\text{S}$ [K8893, Special class] Range of color change: pH (red) 3.1-4.4 (orange-yellow).

Methyl orange TS Dissolve 0.1 g of methyl orange in 100 mL of water, and filter if necessary.

Methyl red $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$ [K8896, Special class] Range of color change: pH (red) 4.2-6.2 (yellow).

Methyl red TS Dissolve 0.1 g of methyl red in 100 mL of ethanol (95), and filter if necessary.

Nitric acid HNO_3 [K8541, Special class, Concentration: 69 - 70%, Density: about 1.42 g/mL]

Nitric acid, dilute Dilute 10.5 mL of nitric acid with water to make 100 mL (10%).

Nitrogen N_2 [Nitrogen (JP monograph)]

Octadecylsilanized silica gel for liquid chromatography Prepared for liquid chromatography

n-Octane C_8H_{18}
Specific gravity: d_4^{20} 0.700 – 0.705
Purity: Perform the test with 2 μL of this substance as directed under Gas Chromatography according to the conditions in the Assay under Hypromellose. Measure each peak area by the automatic integration method, and calculate the amount of n-octane by the area percentage method: not less than 99.0%.

Perchloroethylene [K 1521, Tetrachloroethylene]

Phenolphthalein $\text{C}_{20}\text{H}_{14}\text{O}_4$ [K8799, Special class] Range of color change: pH (colorless) 8.3-10.0 (red)

Phenolphthalein TS Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95).

Phloroglucin See phloroglucinol dehydrate.

Phloroglucinol dehydrate $\text{C}_6\text{H}_3(\text{OH})_3 \cdot 2\text{H}_2\text{O}$ White to pale yellow crystals or crystalline powder. Melting point: 215-219°C (after drying), Loss on drying: 18.0-24.0% (1 g, 105°C, 1 hour)

Phosphorus (V) oxide P_2O_5 [K8342, Phosphrus (V) oxide, Special class]

Polyethylene glycol 20M for gas chromatography Prepared for gas chromatography.

Potassium bromide KBr [K8506, Special class]

Potassium bromide for infrared spectrophotometry Crush homocrystals of potassium bromide or potassium bromide, collect a powder passed through a No. 200 (75 μm) sieve, and dry at 120°C for 10 hours or at 500°C for 5 hours. Prepare tablets with this powder, and determine the infrared absorption spectrum: any

abnormal absorption does not appear.

Potassium bromate KBrO_3 [K8530, Special class]

Potassium bromate-potassium bromide TS Dissolve 1.4 g of potassium bromate and 8.1 g of potassium bromide in water to make 100 mL.

Potassium chloride KCl [K8121, Special class]

Potassium dihydrogen phosphate KH_2PO_4 [K9007, Potassium dihydrogen phosphate, Special class]

Potassium dihydrogen phosphate for pH determination KH_2PO_4 [K9007, Potassium dihydrogen

Potassium hydrogen phthalate for pH determination $\text{C}_6\text{H}_4(\text{COOK})(\text{COOH})$ [K8809, for pH standard solution]

Potassium hydroxide KOH [K8574, Special class]

Potassium hydroxide-ethanol TS Dissolve 10 g of potassium hydroxide in ethanol (95) to make 100 mL. Prepare before use.

Potassium iodide KI [K8913, Potassium iodide, Special class]

Potassium iodate (standard reagent) KIO_3 [K8005, Potassium iodate, Standard reagent for volumetric analysis]

Potassium iodide TS Dissolve 16.5 g of potassium iodide in water to make 100 mL. Preserve in light-resistant containers. Prepare before use (1 mol/L).

Potassium permanganate KMnO_4 [K8247, Special class]

Potassium tetraoxalate for pH determination See potassium trihydrogen dioxalate dihydrate for pH determination.

Potassium trihydrogen dioxalate dihydrate for pH determination $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ [K8474, Potassium trihydrogen dioxalate dihydrate for pH determination]

2-Propanol $(\text{CH}_3)_2\text{CHOH}$ [K8839, Special class]

Pyridine $\text{C}_5\text{H}_5\text{N}$ [K8777, Special class]

Red litmus paper See litmus paper, red.

Silica gel An amorphous, partly hydrated silicic acid occurring in glassy granules of various sizes. When used as a desiccant, it is frequently coated with a substance that changes color when the capacity to absorb water is exhausted. Such colored products may be regenerated by being heated at 110°C until the gel assumes the original color.

Loss on ignition: Not more than 6% (2 g, $950 \pm 50^\circ\text{C}$)

Water absorption: Not less than 31%. Weigh accurately about 10 g of this substance, and allow to stand for 24 hours in a closed container in which the atmosphere is maintained at 80% relative humidity with sulfuric acid having a specific gravity of 1.19. Weigh again, and calculate the increase in mass.

Siliceous earth [K8330, Siliceous earth, First class]

Siliceous earth for gas chromatography Prepared for gas chromatography.

Silicone oil Colorless clear liquid, having no odor. Viscosity 50-100 mm^2/s

Silver diethyldithiocarbamate See silver N, N-diethyldithiocarbamate.

Silver N,N-diethyldithiocarbamate $\text{C}_5\text{H}_{10}\text{AgNS}_2$ [K9512, Special class]

Silver nitrate AgNO_3 [K8550, Special class]

Silver nitrate TS Dissolve 17.5 g of silver nitrate in water to make 1000 mL (0.1 mol/L). Preserve in light-resistant containers.

Soda lime [K8603, for carbon dioxide absorption]

Sodium acetate See sodium acetate trihydrate.

Sodium acetate trihydrate $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ [K8371, Special class]

Sodium borate for pH determination See sodium tetraborate decahydrate for pH determination.

Sodium bromide NaBr [K8514, Special class]

Sodium carbonate, anhydrous Na_2CO_3 [K8625, Sodium carbonate, Special class]

Sodium carbonate for pH determination Na_2CO_3 [K8625, for pH standard solution]

Sodium carbonate (standard reagent) Na_2CO_3 [K8005, Sodium carbonate, Standard reagent for volumetric analysis]

Sodium chloride NaCl [K8150, Special class]

Sodium hydrogen carbonate for pH determination NaHCO_3 [K8622, for pH standard solution]

Sodium hydroxide NaOH [K8576, Special class]

Sodium hydroxide TS Dissolve 4.3 g of sodium hydroxide in water to make 100 mL (1 mol/L). Preserve in polyethylene bottles.

Sodium oxalate (standard reagent) $\text{C}_2\text{O}_4\text{Na}_2$ [K8005, Sodium oxalate, Standard reagent for volumetric analysis] or [K8528, Sodium oxalate, Special class]

Sodium sulfate, anhydrous Na_2SO_4 [K8987, Sodium sulfate, Special class]

Sodium sulfide enneahydrate $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ [K8949, Special class]

Sodium sulfide TS Dissolve 5 g of sodium sulfide enneahydrate in a mixture of 10 mL of water and 30 mL of glycerin. Or dissolve 5 g of sodium hydroxide in a mixture of 30 mL of water and 90 mL of glycerin, saturate a half volume of this solution with hydrogen sulfide, while cooling, and mix with the remaining half. Preserve in well-filled, light-resistant bottles. Use within 3 months.

Sodium tetraborate decahydrate for pH determination [K8866, Sodium tetraborate decahydrate for pH standard solution]

Sodium thiosulfate See sodium thiosulfate pentahydrate.

Sodium thiosulfate pentahydrate $\text{Na}_2\text{S}_2\text{O}_3\cdot 5\text{H}_2\text{O}$ [K8637, Special class]

Stannous chloride See tin (II) chloride dihydrate.

Stannous chloride TS, acidic See tin (II) chloride TS, acidic.

Starch [K8658, Starch, Special class]

Starch TS Triturate 1 g of starch with 10 mL of cold water, and pour the mixture slowly, with constant stirring, into 200 mL of boiling water. Boil the mixture until a thin, translucent fluid is obtained. Allow to settle, and use the supernatant liquid. Prepare before use.

Strong ammonia water See ammonia solution (28).

Strong ammonium water See ammonia solution (28).

Strong hydrogen peroxide water See hydrogen peroxide (30).

Styrene C_8H_8 Clear and colorless liquid.

Specific gravity: 0.902-0.910.

Purity Perform the test with 1 μL of this substance as directed under Gas Chromatography according to the following conditions. Measure each peak area by the automatic integration method and calculate the amount of styrene by the area percentage method: it shows the purity of not less than 99%.

Operating conditions

Detector: Thermal conductivity detector

Column: A glass column about 3 mm in inside diameter and about 2 m in length, packed with siliceous earth for gas chromatography (180 to 250 μm) coated with polyethylene glycol 20 M for gas chromatography in 10%

Column temperature: A constant temperature of about 100°C

Temperature of sample vaporization chamber: A constant temperature of about 150°C

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of styrene is about 10 minutes.

Time span of measurement: About twice as long as the retention time of styrene

Sulfamic acid (standard reagent) See amidosulfuric acid (standard reagent).

Sulfanilamide reference standard for melting point determination [JP Sulfanilamide reference standard for melting point determination]

Sulfapyridine reference standard for melting point determination [JP Sulfapyridine reference standard for melting point determination]

Sulfuric acid H_2SO_4 [K8951, Special class]

Sulfuric acid (94.5-95.5%) See sulfuric acid for readily carbonizable substances.

Sulfuric acid, dilute Cautiously add 5.7 mL of sulfuric acid to 10 mL of water, cool, and dilute with water to make 100 mL (10%).

Sulfuric acid for readily carbonizable substances To sulfuric acid, the content of which has previously been determined by the following method, add water cautiously, and adjust the final concentration to 94.5% to 95.5% of sulfuric acid (H_2SO_4). When the concentration is changed owing to absorption of water during storage, prepare freshly.

Assay: Weigh accurately about 2 g of sulfuric acid in a glass-stoppered flask rapidly, add 30 mL of water, cool, and titrate the solution with 1 mol/L sodium hydroxide VS (indicator: 2 to 3 drops of bromothymol blue TS).

Each mL of 1 mol/L sodium hydroxide VS = 49.04 mg H_2SO_4

Tetrahydrofuran $\text{CH}_2(\text{CH}_2)_2\text{CH}_2\text{O}$ [K9705, Special class]

Tetrahydrofuran for gas chromatography Use tetrahydrofuran prepared by distilling with iron (II) sulfate heptahydrate. Preserve in containers, in which the air has been displaced by nitrogen, in a dark, cold place.

Tin (II) chloride dihydrate $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ [K8136, Special class]

Tin (II) chloride TS, acidic Dissolve 8 g of Tin (II) chloride dihydrate in 500 mL of hydrochloric acid.

Preserve in glass-stoppered bottles. Use within 3 months.

Tin for thermal analysis [K8580, Tin, Special class, Content: not less than 99.99%]

Toluene $\text{C}_6\text{H}_5\text{CH}_3$ [K8680, Special class]

Vanillin reference standard for melting point determination [JP Vanillin reference standard for melting point determination]

Vinyl chloride $\text{C}_2\text{H}_3\text{Cl}$ Colorless gas. Boiling point: -14°C, Melting point: -160°C

Xylene $\text{C}_6\text{H}_4(\text{CH}_3)_2$ [K8271, First class]

Zinc for arsenic analysis Zn [K 8012, for arsenic analysis] Use granules of about 800 μm .