NOTES 6 The permissible limits of main coexisting substances are shown by the maximum ratio as follows.

$$NO_{3}^{-}, SO_{4}^{2-}, PO_{4}^{3-}: 10^{4}$$

 $F^{-}: 10^{2}$
 $Br^{-}: 10^{-2}$
 $I^{-}, CN^{-}, S^{2-}: 10^{-3}$

7 Potentiometric titration by chloride ion-selective electrode Place 100 ml of sample in a beaker, and adjust its pH to about 7. While measuring the electric potential according to the procedures in c) 2) to 4) using a chloride ion-selective electrode or silver ion-selective electrode, titrate the sample with 10 mmol/L to 100 mmol/L silver nitrate solution, and draw the titration curve to find an end point. When halide ion coexists in the sample, the inflection point shown on titration curve makes the order of iodide ion, bromide ion, and chloride ion. Obtain an end point from each inflection point, and calculate the concentration of each ion. One milliliter of 10 mmol/L silver nitrate solution is equivalent to 1.269 mg of iodide ion, 0.799 mg of bromide ion, and 0.354 5 mg of chloride ion.

35.3 Ion chromatography Chloride ion in a sample is determined by the ion chromatography. This method can also determine fluoride ion, nitrite ion, nitrate ion, phosphate ion, bromide ion and sulfate ion simultaneously or singly. However, for determination of nitrite ion, nitrate ion, phosphate ion or bromide ion, the test shall be immediately carried out without performing the treatment for storage as given in **3.3**. When the test cannot be carried out immediately, the sample shall be preserved in a dark place maintained at 0 °C to 10 °C and tested as quickly as possible.

The accuracy and precision of measurement vary, generally depending on the type of ion due to the influence of sample matrix. For checking this, recovery should be determined by addition of the reference solution.

Anion		With suppressor mg/L	Without suppressor mg/L
Chloride	(Cl ⁻)	0.1 to 25	0.5 to 25
Fluoride	(F ⁻)	0.05 to 20	0.1 to 20
Nitrous acid	(NO_2^{-})	0.1 to 25	0.5 to 25
Nitric acid	(NO_3^{-})	0.1 to 50	0.5 to 50
Phosphoric acid	(PO4 ³⁻)	0.1 to 50	0.5 to 50
Bromide	(Br ⁻)	0.1 to 50	0.5 to 50
Sulfuric acid	$({\rm SO_4^{2-}})$	0.2 to 100	1 to 100

Table 35.1 Example of determination range of anion according to ionchromatography

NOTE : The measuring range varies depending on the detector used, quantity of sample injection, exchange capacity of column and so on.

- a) **Reagents** The following reagents shall be used.
 - 1) Water, of A2 or A3 specified in JIS K 0557.
 - 2) **Eluent** Different eluents (¹⁶) are used, their choice depending on the type of detector used, and anion-exchanger packed in the separation column. Their resolution shall be checked before use in accordance with the procedure in NOTE 8 and if they can separate fluoride ion, chloride ion, nitrite ion, nitrate ion, phosphate ion, bromide ion and sulfate ion with the resolution of 1.3 or better, they are appropriate for use in this analysis.

Degas all the eluents or prepare eluents using degassed water. Take steps to avoid any renewed gas pick-up during operation.

- 3) **Reclaiming solution** The reclaiming solutions (¹⁷) are used when a suppressor is used, with their choice depending on the type of detector and suppressor used. Their performance shall be checked before use according to the procedure in NOTE 8, in combination with the separation column.
- 4) Chloride ion reference solution (Cl⁻ 1 mg/ml), in accordance with 35.2 a) 2).
- 5) **Chloride ion reference solution (Cl⁻ 0.1 mg/ml)** Place 10 ml of chloride ion reference solution (Cl⁻ 1 mg/ml) in a 100-ml volumetric flask, and add water to the mark.
- 6) Fluoride ion reference solution (F⁻ 1 mg/ml) Place sodium fluoride of the reference material for volumetric analysis specified in JIS K 8005 in a platinum dish, heat at 500 °C for about 1 h and allow to cool in a desiccator. Weigh out 2.210 g of NaF (100 %), dissolve in a small amount of water, transfer to a 1 000-ml volumetric flask and add water to the mark. Preserve this solution in a polyethylene bottle.
- 7) Fluoride ion reference solution (F^- 0.01 mg/ml) Place 1 ml of fluoride ion reference solution (F^- 1 mg/ml) in a 100-ml volumetric flask and add water to the mark.
- 8) Nitrite ion reference solution (NO₂⁻ 1 mg/ml) Heat sodium nitrite specified in JIS K 8019 at 105 °C to 110 °C for about 4 h, allow to cool in a desiccator, and then determine the purity of sodium nitrite (¹⁸). Take sodium nitrite equivalent to 1.500 g to NaNO₂ 100 %, dissolve in a small amount of water, transfer to a 1 000-ml volumetric flask and add water to the mark. Prepare this solution immediately before use.
- 9) Nitrite ion reference solution $(NO_2^- 0.1 \text{ mg/ml})$ Place 10 ml of nitrite ion reference solution $(NO_2^- 1 \text{ mg/ml})$ in a 100-ml volumetric flask and add water to the mark. Prepare this solution immediately before use.
- 10) Nitrate ion reference solution $(NO_3^- 1 \text{ mg/ml})$ Heat potassium nitrate specified in JIS K 8548 at 105 °C ± 2 °C for about 2 h and allow to cool in a desiccator. Take 1.631 g of this, dissolve in a small amount of water, and after transferring to a 1 000-ml volumetric flask, add water to the mark. Store this solution in a dark place at 0 °C to 10 °C.

- 11) Nitrate ion reference solution $(NO_3^- 0.1 \text{ mg/ml})$ Place 10 ml of nitrate ion reference solution $(NO_3^- 1 \text{ mg/ml})$ in a 100-ml volumetric flask and add water to the mark. Prepare this solution immediately before use.
- 12) **Phosphate ion reference solution** ($PO_4^{3-} 1 \text{ mg/ml}$) Heat potassium dihydrogen phosphate specified in **JIS K 9007** (for pH reference solution) at 105 °C ± 2 °C for about 2 h and allow to cool in a desiccator. Take 1.433 g of this, dissolve in water, and after transferring to a 1 000-ml volumetric flask, add water to the mark. Store this solution in a dark place at 0 °C to 10 °C.
- 13) **Phosphate ion reference solution** ($\mathbf{PO_4}^{3-}$ **0.1 mg/ml**) Place 10 ml of phosphate ion reference solution ($\mathbf{PO_4}^{3-}$ 1 mg/ml) in a 100-ml volumetric flask, and add water to the mark.
- 14) **Bromide ion reference solution (Br 1 mg/ml)** Heat potassium bromide specified in **JIS K 8506** at 105 °C for about 4 h and allow to cool in a desiccator. Take 1.489 g of this (1.00 g as bromide), dissolve in small amount of water, and after transferring to a 1 000-ml volumetric flask, add water to the mark.
- 15) **Bromide ion reference solution (Br 0.1 mg/ml)** Place 10 ml of bromide ion reference solution (Br 1 mg/ml) in a 100-ml volumetric flask and add water to the mark. Prepare this solution immediately before use.
- 16) Sulfate ion reference solution $(SO_4^{2-} 1 \text{ mg/ml})$ Heat potassium sulfate specified in JIS K 8962 at approximately 700 °C for about 30 min, and allow to cool in a desiccator. Take 1.815 g of this, dissolve in small amount of water, and after transferring to a 1 000-ml volumetric flask, add water to the mark.
- 17) Sulfate ion reference solution $(SO_4^{2^-} 0.1 \text{ mg/ml})$ Place 10 ml of sulfate ion reference solution $(SO_4^{2^-} 1 \text{ mg/ml})$ in a 100-ml volumetric flask and add water to the mark. Prepare this solution immediately before use.
 - Notes (¹⁶) When an ultraviolet absorption detector is to be used, the eluent shall be without absorption in ultraviolet part. If the detector is equipped with a suppressor, the eluent of carbonate base may be also used.
 - (¹⁷) For example, sulfuric acid (12.5 mmol/L) [prepared by making 25 ml of sulfuric acid (0.5 mol/L) (prepared by adding 30 ml of sulfuric acid specified in **JIS K 8951** little by little to 500 ml of water, cooling, and then making 1 L with water) 1 L with water] is used.
 - $(^{18})$ The determination of purity shall be in accordance with **JIS K 8019**.
- b) **Implements and apparatus** The following implements and apparatus shall be used.
 - 1) **Ion chromatograph**, of either the type combining a separation column with a suppressor (¹⁹) or the type consisting only of a separation column, which satisfies the following conditions and is capable of separating and determining fluoride ion, chloride ion, nitrite ion, nitrate ion, phosphate ion, bromide ion, and sulfate ion.
 - 1.1) **Separation column**, made of stainless steel or synthetic resin (²⁰), and packed with strongly basic anion exchanger (surface coated type or totally porous silica type) (²¹).

- 1.2) **Detector** Electrical conductivity detector or ultraviolet absorption detector. However, the ultraviolet absorption detector is used for the individual or simultaneous measurement of nitrite ion, nitrate ion and bromide ion.
- 1.3) Recorder, in accordance with JIS K 0127.
- 2) **Microsyringe**, of a suitable capacity of 50 μ l to 200 μ l, or an automatic injecting device.
 - Notes (¹⁹) The suppressor is for exchanging cation in eluent for hydrogen ion, and can be the cation-exchange membrane (including both membrane type and electrodialysis type) having ion-exchange capability sufficient for the cation concentration in eluent or can be packed with cation-exchanger with equal performance to the membrane. It is used together with a reclaiming solution. For the electrodialysis type, however, an effluent from the detector (a solution discharged from the detector) is used as the reclaiming solution.
 - (²⁰) For instance, polytetrafluoroethylene, polyetheretherketone, etc.
 - (²¹) See NOTE 8.
 - NOTE 8 The performance of ion chromatograph is such that its resolution (R) is 1.3 or more, and the resolution should be checked periodically. For obtaining the resolution, let flow the eluent at a specified flow rate (for example, 1 ml/min to 2 ml/min), prepare the anion mixed solution with the concentration of which the peak height of chromatogram becomes approximately the same degree, and prepare the chromatogram to calculate the resolution according to the following formula:

$$R = \frac{2 \times (t_{R2} - t_{R1})}{W_1 + W_2}$$

where, t_{R1} : retention time of the first peak (s)

 t_{R2} : retention time of the second peak (s)

 W_1 : peak width of the first peak (s)

 W_2 : peak width of the second peak (s)

- c) **Preparatory operation** The preparatory operation shall be carried out as follows.
 - 1) Filtrate the sample in accordance with **3.2**.
 - 2) When the sample has electrical conductivity of 10 mS/m (100 μ S/cm) (25 °C) or more, dilute it with water at a decided ratio to obtain a conductivity of 10 mS/m or less.
- d) **Procedure** The test procedure shall be as follows.
 - 1) Get an ion chromatograph ready for running, and flow the eluent in a separation column at a specified rate (for instance, 1 ml/min to 2 ml/min). In the case of an apparatus equipped with a suppressor, the reclaiming solution shall have been flown at a specified rate.

- 2) Inject a specified amount (for instance, 50 μ l to 200 μ l) of the sample, which has been pretreated according to **c**), using a microsyringe (²²) into the ion chromatograph and record the chromatogram.
- 3) Note the detector reading (²⁴) of the peak (²³) corresponding to each target ion on the chromatogram.
- 4) If the sample has been diluted, perform a blank test by carrying out the procedures in 1) to 3) using the same amount of water as the sample, and correct the detector reading (²⁴) obtained on the sample using the obtained blank test value.
- 5) Determine the amount of each target ion by reference to the working curve, and calculate the concentration of each target ion (mg/L) in the sample.
 - Notes (²²) The same microsyringe as used in preparation of working curve shall be used.
 - (²³) Using a specified amount (50 μ l to 200 μ l) of anion mixed reference solution [for example, (F⁻ 5 μ g, Cl⁻ 10 μ g, NO₂⁻ 10 μ g, NO₃⁻ 10 μ g, PO₄³⁻ 10 μ g, Br⁻ 10 μ g and SO₄²⁻ 10 μ g)/ml], the chromatogram shall be recorded, and the position of the peak corresponding to the retention time of each ion shall have been checked.
 - (²⁴) Peak height or peak area.
- e) **Working curve** The working curve shall be prepared as follows.
 - 1) Take an adequate quantity of each of **a**) **4**) to **17**) to prepare the mixed diluted reference solution containing the measurement target ion and having a concentration higher than the measurement concentration range $(^{25})$.
 - 2) Dilute this mixed diluted reference solution in four to five steps and prepare the mixed diluted reference solutions for preparing the working curve.
 - 3) Carry out the procedures of **d**) **1**) to **4**) on the mixed diluted reference solution for preparation of working curves, and note (²⁶) the detector reading (²⁴) of the peak corresponding to each ion.
 - 4) Separately, for a blank test, carry out the procedures of **d**) **1**) to **4**) using water, and correct the detector reading value corresponding to each ion. Then, for respective ions, plot the concentration on the abscissa and the detector reading on the ordinate to prepare the relation curve $(^{27})$.
 - Notes (²⁵) In the case of determining single ion or several decided ions, both the mixed reference solution required and a mixed reference solution containing required anion should be prepared.
 - (²⁶) Example of allowable rate of respective interfering ions in each ion is shown in table 35.2.
 - (27) The working curve does not necessarily show linearity, depending on the composition of eluent and the type of measurement target ion.

NOTES 9 Interfering substance

a) The organic acid such as monocarboxylic acid and dicarboxylic acid can interfere with the determination of inorganic anion.

- b) With the use of the buffering eluent (for example, carbonate/ hydrogen carbonate), the determination is not interfered when the pH of sample is in the range from 2 to 9.
- c) When there is a great difference in concentrations among the anions (fluoride ion, chloride ion, nitrite ion, nitrate ion, phosphate ion, bromide ion and sulfate ion), typical crossing sensitivity interference can occur due to incomplete separation. The determination of chloride ion, when the concentration of fluoride ion is high, is susceptible to interference.

d) Influence of coexisting ion on each ion

- 1) When the concentration of chloride ion is 1 mg/L, nitrite ion of up to 200 mg/L will not interfere.
- 2) When the concentration of nitrite ion is 1 mg/L, chloride ion of up to 50 mg/L, bromide ion of up to 200 mg/L and sulfate ion of up to 500 mg/L will not interfere.
- 3) When the concentration of nitrate ion is 1 mg/L, bromide ion of up to 200 mg/L and sulfate ion of up to 500 mg/L will not interfere.
- 4) When the concentration of bromide ion is 1 mg/L, nitrite ion of up to 200 mg/L will not interfere.
- 5) When the concentration of sulfate ion is 1 mg/L, bromide ion of up to 200 mg/L and nitrate ion of up to 400 mg/L will not interfere.
- e) The determination of sulfate ion can be easily affected by interference of iodide ion or thiosulfate ion of high concentration.
- f) Sulfide ion, which can cause errors in determination of sulfate ion, shall be precipitated by adding zinc acetate solution, and then separated through filtration.
- NOTE 10 The performance of a separation column can deteriorate after repeated use and should be checked periodically according to the procedure in NOTE 8.

If the column performance is found to have deteriorated, prepare a solution of a concentration about 10 times that of the eluent and inject it into the separation column for cleansing. Check the performance according to NOTE 8, and if the performance has not recovered, replace the column with a new one.

Since presence of suspended matter or organic substances (protein, oils, or surfactants, etc.) in sample causes contamination of the column and gradual deterioration of the column performance, if the sample contains suspended matter, eliminate it according to the preparatory operation stated in \mathbf{c}) prior to test. In the case of a sample containing organic substances, filtrate it through an ultrafilter membrane to remove organic substances as completely as possible prior to test.

If the sample contains anions (such as iodide ion, chromate ion) which have strong affinity with the packing material in the separation column, the anions adsorb onto the packing material and cause gradual reduction of the separative performance of the column. In order to prevent this, prepare a solution of a concentration five to ten times that of the eluent, and inject it into the separation column, in a similar manner to the injection of sample, so as to clean the column.

If oxidizing substance or reducing substance coexists in the sample, it reduces the separative performance of the separation column, in which case dilution of the sample with water at a definite ratio is expected to prevent some of the adverse influence.

- NOTE 11 When the concentrations of nitrite ion, nitrate ion, and phosphate ion are expressed by nitrite nitrogen, nitrate nitrogen and phosphate phosphorus, the following conversion formulae are used:
 - a) Nitrite nitrogen (NO₂⁻-N mg/L) = Nitrite ion (NO₂⁻ mg/L) × 0.304 5
 - b) Nitrate nitrogen (NO₃⁻-N mg/L) = Nitrate ion (NO₃⁻ mg/L) \times 0.225 9
 - c) Phosphate phosphorus (PO₄³⁻-P mg/L) = Phosphate ion (PO₄³⁻ mg/L) \times 0.326 1

Table 35.2 Example of cross-sensitivity of anion

Mass conc	entr	Maximum allowable		
Solute/ir	nterf	concentration of		
			interfering ion*	
			mg/.	
Br ⁻ /Cl ⁻	1:	500	Cl-	500
Br^{-}/PO_4^{3-}	1:	100	PO_4^{3-}	100
Br ⁻ /NO ₃ ⁻	1:	50	NO ₃ -	100
$\mathrm{Br}^{-}/\mathrm{SO}_4^{2-}$	1:	500	SO_4^{2-}	500
Br ⁻ /SO ₃ ²⁻	1:	50		
Cl ⁻ /NO ₂ ⁻	1:	50	NO_2^-	5
Cl ⁻ /NO ₃ ⁻	1:	500	NO_3^-	500
$\text{Cl}^-/\text{SO}_4^{2-}$	1:	500	SO_4^{2-}	500
NO ₃ ⁻ /Br ⁻	1:	100	Br⁻	100
NO ₃ ⁻ /Cl ⁻	1:	500 (CD)	Cl-	500
	1:	$2 \ 000 \ (UV)$	Cl⁻	500
NO3 ⁻ /SO4 ²⁻	1:	500 (CD)	SO_4^{2-}	500
	1:	$1\ 000\ (UV)$	SO_4^{2-}	500
NO ₃ ⁻ /SO ₃ ²⁻	1:	50		
NO ₂ ⁻ /Cl ⁻	1:	250 (CD)	Cl ⁻ (CD)	100
	1:	10 000 (UV)	Cl ⁻ (UV)	500
NO2 ⁻ /PO4 ³⁻	1:	50	PO_4^{3-}	20
NO ₂ ⁻ /NO ₃ ⁻	1:	500	NO ₃ ⁻	500
NO2 ⁻ /SO4 ²⁻	1:	500 (CD)	SO_4^{2-}	500
	1:	$1 \ 000 \ (UV)$	SO_4^{2-}	500
PO_4^{3-}/Br^-	1:	100	Br ⁻	100
PO4 ³⁻ /Cl ⁻	1:	500	Cl-	500
PO4 ³⁻ /NO ³⁻	1:	500	NO_3^-	400
PO4 ³⁻ /NO ²⁻	1:	100	NO_2^-	100
PO4 ³⁻ /SO4 ²⁻	1:	100	SO_4^{2-}	500
PO4 ³⁻ /SO3 ²⁻	1:	50 **		
SO4 ²⁻ /Cl ⁻	1:	500	Cl-	500
SO4 ²⁻ /NO3 ⁻	1:	500	NO ₃ -	400
SO4 ²⁻ /SO3 ²⁻	1:	50 **		
$SO_4^{2-}/S_2O_3^{2-}$	1:	500		
SO_4^{2-}/I^-	1:	500		

(Detected by: electrical conductivity or direct ultraviolet absorption)

Notes * When the concentration of interfering substance exceeds the limit, the sample shall be diluted.

** SO_3^{2-} , if present, always interferes.

36 Iodide ion (I^-) For the determination of iodide ion, the iodine-extraction absorptiometry or iodometry is applied.

36.1 Iodine-extraction absorptiometry Iodide ion is reacted with nitrite ion under sulfuric acidity, and the isolated iodine is extracted with chloroform, the absorbance of which is measured to determine iodine ion.

Determination range: $I^- 0.1 \text{ mg to } 5 \text{ mg}$

Repeatability: 3 % to 10 %

- a) **Reagents** The following reagents shall be used.
 - 1) Sulfuric acid (1+1), in accordance with 5.4 a) 2).
 - 2) Sodium nitrite, specified in JIS K 8019.
 - Urea solution (10 g/L), prepared by dissolving 1 g of urea specified in JIS K
 8731 in water to make 100 ml.
 - 4) Sodium sulfate, specified in JIS K 8987.
 - 5) Chloroform, specified in JIS K 8322.
 - 6) **Iodide ion reference solution (I⁻ 1 mg/ml)** Dissolve 1.310 g of potassium iodide specified in **JIS K 8913** in a small volume of water, transfer into a 1 000-ml volumetric flask, and add water to the mark.
 - 7) **Iodide ion reference solution (I⁻ 0.1 mg/ml)** Place 20 ml of iodide ion reference solution (I⁻ 1 mg/ml) in a 200-ml volumetric flask, and add water to the mark. Prepare this solution immediately before use.
- b) **Implement and apparatus** The following implement and apparatus shall be used.
 - 1) Separatory funnel, of capacity 100 ml.
 - 2) **Photometer** Spectrophotometer or photoelectric photometer
- c) **Procedure** The test procedure shall be as follows.
 - 1) Place a suitable volume (1)(2) (containing 0.1 mg to 5 mg as I⁻) of sample in a 100-ml separatory funnel, and add water to make about 50 ml.
 - 2) Add 1 ml of sulfuric acid (1+1) [if sample is alkaline, neutralize it by adding sulfuric acid (1+1)] and 0.5 g of sodium nitrite, and shake up.
 - 3) Add 10 ml of chloroform, and after shaking up vigorously for about 2 min, allow to stand.
 - 4) Transfer the chloroform layer into another 100-ml separatory funnel. Add again 10 ml of chloroform to the water layer to extract, and join the obtained chloroform layer to the above chloroform layer.
 - 5) Add 50 ml of urea solution (10 g/L) to the separatory funnel containing the chloroform, and shake up vigorously for about 2 min to wash the chloroform layer.

- 6) After allowing to stand for about 5 min, transfer the chloroform layer into a 50-ml Erlenmeyer flask with a ground stopper containing about 1 g of sodium sulfate, and shake up for dehydration.
- 7) Place a part of the chloroform layer into an absorption cell, and measure its absorbance at a wavelength near 515 nm using chloroform as a contrast solution.
- 8) For a blank test, using 50 ml of water, carry out the procedures in **2**) to **7**), and correct the measured absorbance of the sample.
- 9) Determine the amount of iodide ion by reference to the working curve, and calculate the concentration of iodide ion $(I^- mg/L)$ in the sample.
 - Notes (1) When the concentration of iodide ion is 2 mg/L or less, take a suitable volume of sample, make it alkaline by adding sodium hydroxide solution (200 g/L) (prepared by dissolving 20 g of sodium hydroxide specified in JIS K 8576 in water to make 100 ml), and heat gently to concentrate. If turbidity occurs during concentration, filtrate it, and then carry out the procedures from 1) on.
 - (²) When a lot of organic matter coexists in the sample, take 200 ml of the sample, add 2 ml or 3 ml of aluminium potassium sulfate solution (prepared by dissolving 5 g of aluminium potassium sulfate 12-water specified in **JIS K 8255** in water to make total 100 ml), and then add sodium hydroxide solution (50 g/L) (prepared by dissolving 5 g of sodium hydroxide specified in **JIS K 8576** in water to make 100 ml) until precipitate of aluminium hydroxide forms. After allowing to stand for about 5 min, filtrate it, and take a suitable volume of this filtrate to carry out the procedures from **1**) on.
- d) **Working curve** The working curve shall be prepared as follows.
 - 1) Place a series of aliquots in stepwise increment ranging from 1 ml to 50 ml of the iodide ion reference solution ($I^- 0.1 \text{ mg/ml}$) in as many 100-ml separatory funnels.
 - 2) Carry out the procedures in c) 1) to 8) to measure the absorbance, and plot the relation curve between the amount of iodide ion (I^{-}) and the absorbance.
 - NOTE 1 If the sample contains iodate ion, since the iodate ion reacts with iodide ion and produces iodine during acidification by sulfuric acid, iodate ion is entirely or partially determined as iodide ion. Bromide ion does not interfere.

36.2 Iodometry Iodide ion is oxidized by hypochlorous acid under pH 1.3 to pH 2.0 into iodate ion. After excess hypochlorous acid is decomposed by sodium formate under pH 3 to pH 7, potassium iodide is added, isolated iodine is titrated with sodium thiosulfate solution and iodide ion is determined.

Determination range: $I^- 0.1 \text{ mg or more}$

- a) **Reagents** The following reagents shall be used.
 - Hydrochloric acid (1+1), prepared using the hydrochloric acid specified in JIS K 8180.

- Hydrochloric acid (1+11), prepared using the hydrochloric acid specified in JIS K 8180.
- 3) Sodium hypochlorite solution (35 g/L effective chlorine) Determine (³) effective chlorine contained in sodium hypochlorite solution (7 % to 12 % of effective chlorine), and dilute it with water so as to contain the effective chlorine of 35 g/L. Prepare this solution immediately before use.
- 4) Sodium formate solution (400 g/L) Dissolve 40 g of sodium formate specified in JIS K 8267 in water to make 100 ml.
- 5) **Potassium iodide**, specified in **JIS K 8913**.
- 6) Methyl orange solution (1 g/L), in accordance with 24.1 a) 2).
- 7) Starch solution (10 g/L), in accordance with 19 a) 5).
- 8) 10 mmol/L sodium thiosulfate solution, in accordance with 19 a) 9).
 - Note (³) Place 10 ml of sodium hypochlorite solution (7 % to 12 % of effective chlorine) in a 200-ml volumetric flask, and add water to the mark. Take 10 ml of this solution in a 300-ml Erlenmeyer flask with a ground stopper, and add water to make about 100 ml. Add 1 g to 2 g of potassium iodide and 6 ml of acetic acid (1+1) [in accordance with **33.4 a**) **9.2.1**)], stopper tightly, shake up well, allow to stand in a dark place for about 5 min, and titrate with 50 mmol/L sodium thiosulfate solution [prepared by diluting twice the 0.1 mol/L sodium thiosulfate solution specified in **19 a**) **8**)]. When the solution turns faint yellow, add 1 ml of starch solution (10 g/L) as an indicator, and continue the titration until the blue color of resultant iodostarch disappears. Separately, for a blank test, carry out the same operation using 10 ml of water, and correct the value obtained by the titration. Calculate the amount of effective chlorine by the formula below:

$$N = a \times f \times \frac{200}{10} \times \frac{1\,000}{V} \times 0.001\,773$$

where,

- N: amount of effective chlorine (g/L)
- *a*: volume of 50 mmol/L sodium thiosulfate solution required for titration (ml)
- f: factor of 50 mmol/L sodium thiosulfate solution [use the factor of 0.1 mol/L sodium thiosulfate solution in **19 a**) **8**)]
- 0.001 773 : mass of effective chlorine corresponding to 1 ml of 50 mmol/L sodium thiosulfate solution (g)
 - V: volume of sodium hypochlorite solution (7 % to 12 % of effective chlorine) (ml)
- b) **Procedure** The test procedure shall be as follows.