**58** Aluminium (Al) For the determination of aluminium, quinolinol absorptiometry, flame atomic absorption spectrometry, electrothermal atomic absorption spectrometry, ICP atomic emission spectrometry or ICP mass spectrometry shall apply.

The ICP atomic emission spectrometry described in this clause is in consistency with the first edition of **ISO 11885** published in 1996.

NOTE : The International Standard corresponding to this test method is as follows:

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

ISO 11885:1996 Water quality – Determination of **33** elements by inductively coupled plasma atomic emission spectroscopy (MOD)

**58.1 Quinolinol absorptiometry** Add hydroxylammonium chloride and 1,10phenanthroline to slightly acidified sample in order to mask iron, add 8-quinolinol and ammonium acetate, and extract produced complex with chloroform. Then wash with ammonium chloride solution containing potassium cyanide to remove copper, nickel, cobalt or other which has been extracted in company with aluminium, and measure the absorbance of aluminium complex to determine aluminium.

Determination range: Al  $5 \mu g$  to  $50 \mu g$ 

Repeatability: 3% to 10%

- a) **Reagents** The following reagents shall be used:
  - 1) **Hydrochloric acid** (1+2), prepared using the hydrochloric acid specified in **JIS K 8180**.
  - 2) Ammonia solution (1+2), prepared using the ammonia solution specified in JIS K 8085.
  - 3) Sodium sulfate, specified in JIS K 8987.
  - 4) Hydroxylammonium chloride solution (100 g/L), prepared in accordance with 39.2 a) 3).
  - 5) Ammonium acetate solution (150 g/L) Dissolve 15 g of ammonium acetate specified in JIS K 8359 in water to make total 100 ml. Transfer this solution into a separatory funnel, add 5 ml of 8-quinolinol chloroform solution (dissolve 2 g of 8-quinolinol specified in JIS K 8775 in 100 ml of chloroform specified in JIS K 8322) and shake vigorously to mix. Allow to stand, and discard the chloroform layer. Repeat these procedures until the chloroform layer gives no coloring. Then, add 5 ml of chloroform to the water layer, shake vigorously to mix, allow to stand, and discard the chloroform layer in layer gives no coloring. Then, add 5 ml of chloroform to the water layer. Repeat these procedures until the water layer no longer shows yellow coloring. Filtrate the water layer through dried filter paper of Class 5 B, and remove droplets of chloroform remaining in solution.
  - 6) **Potassium cyanide-ammonium chloride solution** Dissolve 1.0 g of potassium cyanide specified in **JIS K 8443** with water to make total 500 ml.

Dissolve little by little ammonium chloride specified in **JIS K 8116** in this solution until the pH is 9.0 to 9.5. Refine this solution, similarly to the case of ammonium acetate solution in **5**), by washing with both 8-quinolinol chloroform solution and chloroform.

- 7) 1,10-Phenanthroline solution (1g/L), prepared in accordance with 57.1 a)
  5).
- S) 8-Quinolinol solution (10 g/L) Add 5 ml of acetic acid specified in JIS K
   8355 onto 2 g of 8-quinolinol specified in JIS K 8775, heat it slightly for dissolving, and add water to make total 200 ml.
- 9) Chloroform, specified in JIS K 8322.
- 10) Aluminium reference solution (Al 0.1 mg/ml) Weigh 1.76 g of aluminium potassium sulfate-12 water [bis (sulfuric acid) potassium aluminium-water (1/12)] specified in JIS K 8255, dissolve it in 20 ml of hydrochloric acid (1+1) [specified in 57.1 a)1)], transfer to a 1000-ml volumetric flask, and add water to the mark. Alternatively, weigh 0.100 g of aluminium (99.9% min.) specified in JIS K 8069, dissolve it in 20 ml of hydrochloric acid (1+1)through heating, and after allowing to cool, transfer to a 1000-ml volumetric flask, and add water up to the mark.
- 11) Aluminium reference solution (Allμg/ml) Place 10 ml of aluminium reference solution (Al 0.1 mg/ml) in a 1000-ml volumetric flask, add 20 ml of hydrochloric acid (1+1) [specified in 57.1 a)1)], and add water up to the mark.
- b) **Implement and apparatus** The implement and apparatus to be used shall be as follows:
  - 1) Separatory funnel, of 200-ml capacity
  - 2) **Photometer** Spectrophotometer or photoelectric photometer
- c) **Operation** The operation shall be carried out as follows:
  - 1) Take a suitable volume  $(^2)$  (containing 5 µg to 50 µg as Al) of sample which has been treated as in clause  $5(^1)$ , add 1 ml of hydroxylammonium chloride solution (100 g/L) and 5 ml of 1,10-phenanthroline solution (1g/L), mix by shaking, and drip ammonia solution (1+2) to adjust its pH to about  $3.5(^3)$ .
  - 2) Add water to make total about 80 ml, and leave it to stand for about 15 min.
  - 3) Add 3 ml of 8-quinolinol solution (10 g/L) and 10 ml of ammonium acetate solution (150 g/L), and drip ammonia solution (1+2) to adjust the pH to 5.2 to 5.5<sup>(4)</sup>.
  - **4)** Transfer this solution into a separatory funnel, add water to make total about 100 ml. Add 10 ml (or 20 ml) of chloroform, shake vigorously to mix for about 1 min and allow to stand.
  - 5) Separate the chloroform layer, and transfer it into another separatory funnel. Add 25 ml of potassium cyanide-ammonium chloride solution, agitate, and leave to stand.
  - 6) Place the chloroform layer in a 30-ml ground-stoppered test tube, add about 1 g of sodium sulfate, and shake lightly to remove moisture.

- 7) Transfer a part of the chloroform layer into an absorption cell, and measure its absorbance near the wavelength of 390 nm using chloroform as a reference solution.
- 8) For a blank test, take about 70 ml of water, carry out the procedures in 1) to 7) to measure the absorbance, and correct the absorbance obtained on the sample.
- 9) Find the amount of aluminium on the working curve, and calculate the concentration of aluminium (Al mg/L) in the sample.
  - Notes (1) Among those specified in clause 5, the method in 5.3 can not be used. In the case of sample containing a minute amount of organic matter, the following method is permissible; add 5 ml of hydrochloric acid specified in JIS K 8180 per 100 ml of sample, and heat gently until the volume of solution concentrates to about  $\frac{1}{5}$  of the original volume.
    - <sup>(2)</sup> The amount of sample shall generally be 50 ml to 100 ml, and may be up to maximum of 500 ml.
    - <sup>(3)</sup> Use bromophenol blue test paper.
    - (4) Use bromocresol green test paper. If pH does not fall within the range of 5.2 to 5.5, adjust it with hydrochloric acid (1+2) or ammonia solution (1+2).
- d) Working curve The working curve shall be prepared as follows:
  - 1) Place several aliquots, increased stepwise from 5 ml to 50 ml, of aluminium reference solution (Al  $1\mu g/ml$ ) in as many separatory funnels, and add water to each of the solutions to make total about 70 ml. Carry out the procedures in c) 1) to 8), and plot the relation curve between the amount of aluminium (Al) and its absorbance.
    - NOTES 1 When fluoride ion is contained in sample, adding 36 mg of beryllium sulfate per 0.5 mg of fluoride ion can prevent the interference.
      - 2 If chromium is present, the extraction under pH of 5.2 to 5.5 shall be performed at as low a temperature as possible. Cooling can be done with the use of icy water.
      - 3 When a lot of manganese is contained, wash chloroform solution, in which complex was extracted, with acetic acid ammonium acetate solution of 7 or less pH (prepared by dissolving 173g of ammonium acetate specified in **JIS K 8359** in approximately 800 ml of water, adding 13 ml of acetic acid specified in **JIS K 8355**, and making it 1L with water) containing hydroxylammonium chloride, in order to eliminate manganese.
      - 4 When titanium, molybdenum, or others are included, the following shall be performed: remove copper, nickel, cobalt, or others by the procedures in c) 3), and furthermore wash the chloroform layer with the mixed solution of 25 ml of ammonia-alkalified ammonium chloride solution (50 g/L) of pH10 (prepared by dissolving 50 g of ammonium chloride specified in JIS K 8116 in about 500 ml of

water, adding ammonia solution specified in **JIS K 8085** to attain the pH of about 10 and adding water to make 1L) and 2 ml of hydrogen peroxide specified in **JIS K 8230**.

- 5 Presence of iron up to 0.45 mg gives no influence in this method.
- 6 In the case of simultaneous determination of aluminium and iron, the following shall be performed:
  - Excluding the procedure in c) 1) in which 1 ml of hydroxylammonium chloride solution (100 g/L) and 5 ml of 1,10phenanthroline solution (1 g/L) are added, carry out the procedures in c) 2) to 7) to measure the absorbance A near the wavelength of 390 nm and absorbance B near 470 nm.

  - 3) On the working curve of iron (111) near the wavelength of 470 nm, find the amount of iron (III) corresponding to absor-Dance B', and calculate the concentration (Fe mg/L) of iron.
  - 4) Applying the amount of iron (111)corresponding to absorbance B' to the working curve of iron (III) near the wavelength of 390 nm, find absorbance C by iron (III) near the wavelength of 390 nm. Subtract the absorbance C from the absorbance A', and obtain the absorbance D by aluminium near the wavelength of 390 nm.
  - 5) Using absorbance D, find the amount of aluminium on the working curve near the wavelength of 390 nm, and calculate the concentration of aluminium (Al mg/L) in the sample.
  - 6) **Working curve** The working curve shall be prepared as follows:
  - 6.1) Place several aliquots, increased stepwise from 5 ml to 50 ml, of aluminium reference solution (Al 1μg/ml) in as many separatory funnels, and add water to each to make total about SO ml. Carry out the procedures in c) 3) to 6), and measure the absorbance near the wavelength of 390 nm.
  - 6.2) Separately, place several aliquots, increased stepwise from 0.5 ml to 10 ml of iron (III) reference solution (Fe 10 μg/ml)(\*) in as many separatory funnels, and add water to each to make total about SO ml. Carry out the procedures in c) 3) to 6), and measure the absorbance of each near the wavelengths of 470 nm and 390 nm.
  - 6.3) For a blank test, take about 80 ml of water, carry out the procedures in c) 3) to 6), measure each absorbance near the wavelengths of 470 nm and 390 nm, and correct the measured absorbances of aluminium reference solution and iron (III) reference solution. Plot the relation curve between the amount of aluminium (Al) and its absorbance given near the

wavelength of 390 nm, and relation curve between the amount of iron (Fe) and its absorbance near 470 nm and its absorbance near 390 nm.

Note (\*) Iron (III) reference solution (Fe 10 µg/ml) Weigh 8.63 g of ammonium iron (111) sulfate-12 water [bis (sulfuric acid) iron (III) ammonium-water (1/12)] specified in JIS K 8982, dissolve it in 20 ml of sulfuric acid (1+1) [specified in 5.4 a) 2)] and water, transfer it into a 1000-ml volumetric flask, and add water up to the mark. Transfer 10 ml of this iron (111) reference solution (Fe 1mg/ml) into a 1000-ml volumetric flask, add 10 ml of sulfuric acid (1-1) and add water up to the mark.

**58.2 Flame atomic absorption spectrometry** After pretreatment of the sample, spray it into an acetylene-dinitrogen monoxide flame, and measure the atomic absorption made by aluminium at the wavelength of 309.3 nm to determine aluminium.

Determination range: Al 5 mg/L to 100 mg/L

Repeatability: 2% to 10% (it can vary according to the type of apparatus used and measuring conditions)

- a) **Reagents** The following reagents shall be used:
  - 1) **Potassium chloride solution** (100 g/L) Dissolve 10 g of potassium chloride specified in JIS K 8121 in water to make 100 ml.
  - 2) Aluminium reference solution (Al 0.5 mg/ml) Weigh out 8.794 g of aluminium potassium sulfate-12 water [bis (sulfuric acid) potassium aluminiumwater (1/12)] specified in JIS K 8255, and dissolve it in 20 ml of hydrochloric acid (1+1)[specified in 57.1 a)1)], transfer it into a 1000-ml volumetric flask, and add water to the mark. Alternatively, weigh 0.500 g of aluminium (99.9% min.) specified in JIS K 8069, dissolve it in 30 ml of hydrochloric acid (1+1) by heating, allow to cool, transfer it into a 1000-ml volumetric flask, and add water to the mark.
- b) Apparatus The apparatus to be used shall be in accordance with 52.2 b).
- c) **Preparatory operation** The preparatory operation shall be carried out as follows:
  - 1) Treat the sample as shown in clause 5.
- d) **Operation** The operation shall be carried out as follows:
  - 1) Place a suitable amount (containing 0.5 mg to 10 mg as Al) of sample, which has been pretreated as specified in *c*), in a 100-ml volumetric flask, add 1 ml of hydrochloric acid specified in **JIS K 8180**, and add water to the mark.
  - 2) Transfer 50 ml of this solution into a dry beaker, and add 2 ml of potassium chloride solution (100 g/L).
  - 3) Introduce the sample obtained in 2) into the acetylene-dinitrogen monoxide flame<sup>(5)</sup>, and note the reading on the apparatus<sup>(6)</sup> at the wavelength of 309.3 nm.

- 4) For a blank test, using water of the same amount as the sample used in preparatory operation in c), carry out the operation in c) and d) 1) to 3) similarly to the sample, and correct the reading on the apparatus obtained on the sample.
- 5) Find the amount of aluminium on the working curve and calculate the concentration of aluminium (Al mg/L) in the sample.

Notes  $(\delta)$  Higher sensitivity is obtained if a fuel-rich flame is used.

- (6) Absorbance or its proportioned value.
- e) Working curve The working curve shall be prepared as follows:
  - 1) Place several aliquots, increased stepwise from 1 ml to 20 ml, of aluminium reference solution (Al 0.5 mg/ml) in as many 100-ml volumetric flasks. Add acid and hydrochloric acid (specified in **JIS K 8180**) to each of these to make the acid concentration same as that of the sample treated in **c**) 1) and add water up to the mark.
  - 2) Perform the procedure in **d**)**2**) and **3**).
  - 3) Separately, for a blank test, add acid and hydrochloric acid to water so that its acid concentration becomes the same as that of the sample treated in c)
    1), perform the procedures in d) 2) and 3) and correct the values of the sample previously obtained.
  - 4) Plot the relation curve between the amount of aluminium (Al) and the reading on the apparatus. Prepare the working curve at the time of measurement of sample.

**58.3 Electrothermal atomic absorption spectrometry** After pretreatment of the sample, atomize it in an electric furnace, and measure the atomic absorption made by aluminium at the wavelength of **309.3** nm to determine aluminium.

Determination range: Al 20  $\mu$ g/L to 200  $\mu$ g/L

Repeatability: 2% to 10% (it can vary according to the type of apparatus used and the measuring conditions.)

NOTE 7 See NOTE 6 of clause **52**.

- a) **Reagents** The following reagents shall be used:
  - 1) Water, in accordance with 52.3 a) 1).
  - 2) Nitric acid (1+1), prepared in accordance with 52.3 a) 2).
  - 3) Aluminium reference solution (Al 1 µg/ml) Place 5 ml of aluminium reference solution (Al 0.1 mg/ml) of 58.1 a) 10) in a 500-ml volumetric flask, add 10 ml of nitric acid (1-1), and add water to the mark. Prepare this solution immediately before use.
- b) Implements and apparatus, as specified in 52.3 b).
- c) **Preparatory operation** The preparatory operation shall be carried out as follows:
  - 1) Treat the sample as shown in clause **5**.

- d) **Operation** The operation shall be carried out as follows:
  - 1) Perform the procedure in **52.3 d) 1**), except that the atomization (<sup>7</sup>) temperature shall be **2** 200 "C to **3** 000 "C, and the wavelength of **309.3** nm shall be used.
  - 2) Perform the procedure in 52.3 d) 2) as a blank test.
  - 3) Find the amount of aluminium on the working curve and calculate the concentration of aluminium (Al  $\mu$ g/L) in the sample.

Note  $(^7)$  See Note  $(^9)$  of clause **52**.

- e) Working curve The working curve shall be prepared as follows:
  - 1) Place several aliquots, increased stepwise from 2 ml to 20 ml, of aluminium reference solution (Al  $1 \mu g/ml$ ) in as many 100-ml volumetric flasks. Add acid to each of these to make the acid concentration same as that of the sample treated in c) 1) and add water up to the mark. Perform the procedure in dj 1) on this solution.
  - 2) Perform the procedure in **52.3 e**) **2)** and **3)** and plot the relation curve between the amount of aluminium (Al) and the reading on the apparatus. Prepare the working curve at the time of measurement of sample.

**58.4 ICP atomic emission spectrometry** After pretreatment of the sample, spray it into an inductively coupled plasma through the sample introducing part, and measure the emission made by aluminium at the wavelength of **309.271** nm to determine aluminium. By this method, the elements given in table 58.1 can be simultaneously determined. Table 58.1 gives examples of measuring wavelength, determination range and repeatability of each target element.

<b>Table 58.1</b>	Example of measuring wavelength, determination ran		
	and repeatability*		

Target element	Measuring wavelength nm	Determination range µg/L	Repeatability %
Aluminium (Al)	309.271	80 to 4000	2 to 10
Chromium (Cr)	206.149	20 to 4 000	2 to 10
Molybdenum (Mo)	202.030	40 to 4 000	2 to 10
Vanadium (V)	309.311	20 to <b>2</b> 000	<b>2</b> to 10
Yttrium (Y) **	371.029	_	_

Notes \* May vary according to the type of apparatus used and measurement conditions.

- \*\* Internal standard element. Besides yttrium (Y) given above, indium (In) and ytterbium (Yb) can also be used.
- a) **Reagents** The following reagents shall be used:
  - 1) Nitric acid (1+1), prepared using nitric acid in JIS K 8541.
  - 2) Aluminium reference solution (Al 20 µg/ml) Place 10 ml of aluminium reference solution (Al 0.5 g/ml) in 58.2 a) 2) in a 250-ml volumetric flask, add 5 ml of nitric acid (1+1), and add water to the mark.

- 3) Chromium reference solution (Cr0.1 mg/ml) Heat potassium dichromate for the reference material for volumetric analysis specified in JIS K 8005 at 150 °C for about 1h, and allow to cool in a desiccator. Weigh out 0.283 g of this with respect to  $100 \% \text{ K}_2 \text{Cr}_2 \text{O}_7$  and dissolve in small amount of water. Transfer into a 1000-ml volumetric flask, and add water to the mark.
- 4) Chromium reference solution (Cr  $10 \mu g/ml$ ) Place 50 ml of chromium reference solution (Cr 0.1 mg/ml) in a 500-ml volumetric flask, and after adding 10 ml of nitric acid (1+1), add water to the mark.
- 5) Molybdenum reference solution (Mo 0.1 mg/ml) Weigh out 0.184 g of hexaammonium heptamolybdate tetrahydrate specified in JIS K 8905 and dissolve in small amount of water. Transfer into a 1000-ml volumetric flask, and add water to the mark.
- 6) **Molybdenum reference solution (Mo 20 µg/ml)** Place 20 ml of molybdenum reference solution (Mo 0.1 mg/ml) in a 100-ml volumetric flask and add water to the mark.
- 7) Vanadium reference solution (V 0.1 mg/ml) Weigh out 0.230 g of ammonium vanadate (V) specified in JIS K 8747 to dissolve in 10 ml of sulfuric acid (1+1) [specified in 5.4 a)2)] and 200 ml of hot water. After allowing to cool, transfer into a 1 000-ml volumetric flask and add water to the mark.
- 8) Vanadium reference solution (V 10 µg/ml) Place 10 ml of vanadium reference solution (V 0.1 mg/ml) in a 100-ml volumetric flask, add 2 ml of nitric acid (1+1), and add water to the mark.
- b) Apparatus The apparatus to be used shall be in accordance with 52.4 b).
- c) **Preparatory operation** The preparatory operation shall be carried out as follows:
  - 1) Treat the sample as shown in **5.5**. The pretreatment given in **5.3** shall not be applied for the determination of chromium.
    - NOTE 8 When the pretreated sample has high concentration of sodium, potassium, calcium, magnesium, but has low concentration of aluminium, the following operation is permissible: Take 100 ml of sample and after performing the procedure of 58.1 c) 1) to 5) replacing chloroform with 3-methyl-1-butanol (isoamyl alcohol) specified in JIS K 8051, transfer the 3-methy-1-butanol layer into a ground stoppered test tube. In this case, the removal of water through addition of sodium sulfate as specified in 58.1 c) 6) may be omitted.
- d) **Operation** The operation shall be carried out in accordance with **52.4** dj. The emission intensity of each of the metal elements at the wavelengths shown in table 58.1 shall be measured.
- e) Working curve See 52.4 e)(<sup>8</sup>).
  - Note (8) When the preparatory operation is carried out according to NOTE S, and 3-methyl-1-butanol layer is sprayed as it is, the preparation of working curve shall be as follows:

Dilute the aluminium reference solution (Al  $20 \mu g/ml$ ) to an adequate concentration (Al  $1\mu g$  to  $4\mu g/ml$ ). Place several aliquots, increased stepwise, of this solution in as many 100-ml volumetric flasks, and after making 100 ml with water, carry out the operation of NOTE 8 and 52.4 d) 1) and 2) similarly to the sample. Prepare the relation curve between the amount of aluminium (Al) and the emission intensity.

## 58.5 ICP mass spectrometry See 52.5.

**59** Nickel (Ni) For the determination of nickel, the dimethylglyoxime absorptiometry, flame atomic absorption spectrometry, ICP atomic emission spectrometry or ICP mass spectrometry shall apply.

The flame atomic absorption spectrometry and the ICP atomic emission spectrometry described in this clause are in consistency with the first edition of **ISO 8288** published in 1986, and the first edition of **ISO 11885** published in 1996, respectively.

NOTE : The International Standards corresponding to these test methods are as follows:

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

ISO 8288:1986 Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods (MOD)

ISO 11885: 1996 Water quality – Determination of **33** elements by inductively coupled plasma atomic emission spectroscopy (MOD)

**59.1 Dimethylglyoxime absorptiometry** Add citrate to the sample, add ammonia solution to make it faint alkaline, and then add 2,3-butanedionedioxime (dimethylglyoxime) to produce nickel complex. Extract the produced nickel complex with chloroform, and then back-extract it with diluted hydrochloric acid. Add bromine and ammonia solution to the solution containing extracts to oxidize nickel, add again 2,3-butanedionedioxime, and measure the absorbance of reddish brown nickel complex to determine nickel.

Determination range: Ni  $2 \mu g$  to  $50 \mu g$ 

Repeatability: 2% to 10%

- a) **Reagents** The following reagents shall be used:
  - 1) Hydrochloric acid (1+20), prepared using the hydrochloric acid specified in JIS K 8180.
  - 2) Ammonia solution (1+1) and (1+5), prepared using the ammonia solution specified in JIS K 8085.
  - Bromine water (saturated) Add 3 ml to 4 ml of bromine specified in JIS K 8529 to 100 ml of water, shake vigorously to mix, and after allowing to stand, employ its supernatant.
  - 4) **Diammonium hydrogen citrate solution** (100 g/L) Dissolve 10 g of diammonium hydrogen citrate specified in **JIS K 8284** in about 80 ml of water, adjust the pH to about 7 by dropping ammonia solution (1+1), and add water to make 100 ml.
  - 5) Phenolphthalein solution (5g/L), in accordance with NOTE 2 of clause 15.
  - 6) Ethanol solution of dimethylglyoxime (10 g/L) Dissolve 1g of dimethylglyoxime specified in **JIS K 8498** in ethanol (95) specified in **JIS K 8102** to make total 100 ml.

- 7) Sodium hydroxide solution of dimethylglyoxime (10g/L) Dissolve 1g of dimethylglyoxime specified in JIS K 8498 in sodium hydroxide solution (10 g/L) (prepared by using sodium hydroxide specified in JIS K 8576) and add sodium hydroxide solution (10 g/L) to make total 100 ml. If the undissolved is found, filtrate it.
- 8) Chloroform, as specified in JIS K 8322.
- 9) Nickel reference solution (Ni 0.1 mg/ml), in accordance with 52.4 a) 14).
- 10) Nickel reference solution (Ni 5  $\mu$ g/ml) Place 50 ml of nickel reference solution (Ni 0.1 mg/ml) in a 1000-ml volumetric flask, add 20 ml of nitric acid (1+1)[specified in 58.4 a) 1)] and add water to the mark.
- b) **Implement and apparatus** The implement and apparatus to be used shall be as follows:
  - 1) Separatory funnel
  - 2) **Photometer** Spectrophotometer or photoelectric photometer
- c) **Operation** The operation shall be carried out as follows:
  - 1) Place a suitable amount<sup>(1)</sup> (containing 2 µg to 50 µg as Ni) of the sample which has been pretreated as in clause **5** in a separatory funnel, and add 5 ml of diammonium hydrogen citrate solution (100 g/L) and 2 or **3** drops of phenolphthalein solution  $(5 \text{ g/L})^{(2)}$  as indicator. Then drip ammonia solution (1+5) until the solution turns faint red, and add a few drops more of ammonia solution (1+5) and water up to make total about 100 ml.
  - 2) Add 2 ml of ethanol solution of dimethylglyoxime (10 g/L) and 10 ml of chloroform, and after shaking vigorously for about 1 min and allowing to stand, transfer the chloroform layer into another separatory funnel. Add 5 ml of chloroform to the water layer, shake vigorously for about 1 min to extract, and after allowing to stand, take the chloroform layer, and join it to the above separatory funnel. Repeat this procedure once again.
  - 3) Add 10 ml to 20 ml of ammonia solution (I+50) (prepared using ammonia solution specified in **JIS K 8085)** to the separatory funnel containing the chloroform layer, shake for about 30 s, and after allowing to stand, transfer the chloroform layer to another separatory funnel.
  - 4) Add 10 ml of hydrochloric acid (1+20) to the separatory funnel containing the chloroform layer, shake vigorously for about 1 min, and back-extract nickel. After standing, transfer the chloroform layer into another separatory funnel. Add again 5 ml of hydrochloric acid (1+20) to the chloroform layer, and repeat back-extraction. Discard the chloroform layer, put this water layer together with the above water layer, and transfer it into a 25-ml volumetric flask.
  - 5) Add 2 ml of bromine water (saturated), shake to mix, and allow to stand for about 1 min. Neutralize it by adding ammonia solution (1+1), and after adding excessive 2 ml of ammonia solution (1+1), cool it to room temperature or below using running water.
  - 6) Add 2 ml of sodium hydroxide solution of dimethylglyoxime (10 g/L), shake it to have nickel color, and add water to the mark.