NOTE 9 In place of the operation in **c**), the following procedure may be performed using pressure-resistant heat-resistant screw-top test tube and a block heater.

1.1) If the sample has a pH of 5 to 9 and contains less than 12 ∝g of total phosphorus per 10 ml, take 10 ml of the sample in a pressure-resistant heat-resistant screw-top test tube (see NOTE 3 to **45.6**). If the sample contains 12 ∝g or more of total phosphorus, place a suitable volume (containing less than 25 ∝g as P) of the sample in a 20-ml volumetric flask, dilute to the mark with water, and use 10 ml of this solution.

1.2) When the sample has a pH outside the range of 5 to 9 and contains less than 12 ∝g of total phosphorus per 10 ml, place 10 ml of the sample in a pressure-resistant heat-resistant screw-top test tube, and neutralize the sample with sulfuric acid (1+35) or sodium hydroxide solution (40 g/L). Record the total volume (*b* ml) of both solutions used in this neutralization.

1.3) If the sample has a pH outside the range of 5 to 9 and contains 12 ∝g or more of total phosphorus per 10 ml, take a suitable volume of the sample (containing less than 25 ∝g as P) in a beaker or a similar container, neutralize with sulfuric acid (1+35) or sodium hydroxide solution (40 g/L), transfer to a 20-ml volumetric flask, dilute to the mark with water, and use 10 ml of this solution.

2) Add 2 ml of potassium peroxodisulfate solution (40 g/L), tightly stopper the bottle and mix.

3) Heat and decompose this solution for about 20 min by means of a block heater heated to 150 °C to 180 °C.

4.1) Take out the pressure-resistant heat-resistant screw-top test tube, and allow it to cool.

4.2) If sample contains a large amount of chloride ion, add 0.2 ml of sodium hydrogensulfite (50 g/L) to the solution after decomposition according to **c**) **4.2**). If this addition has been performed, correct for it in calculation of total phosphorus concentration.

4.3) If the solution after decomposition contains arsenic (V), referring to **c**) **4.3**), add 0.6 ml of sulfuric acid (1 mol/L) and 0.3 ml of sodium thiosulfate solution (7.65 g/L) to the solution after decomposition, allow to stand for 5 min to 10 min to change arsenic (V) into arsenic (III), and neutralize with sodium hydroxide solution (40 g/L). Take 10 ml of this supernatant solution and perform the procedure in **2**) of NOTE 10.

Note the total volume (ml) of sulfuric acid (1 mol/L), sodium thiosulfate solution (7.65 g/L) and sodium hydroxide solution (40 g/L) used, and use the value for correction in the calculation of total phosphorus.

5) For a blank test, carry out the procedures in **2**) to **4.1**) using 10 ml of water taken in a decomposing bottle.

46.3.1.2 Determination method The phosphate ion in the decomposed solution obtained according to **46.3.1.1** is determined by the molybdenum blue absorptiometry, and the total phosphorus concentration calculated.

Determination range: P 1.25 ∝g to 25 ∝g, Repeatability: 2 % to 10 %

a) **Reagents** The following reagents shall be used.

1) **Water**, as specified in **46.3.1.1 a**) **1**).

2) **Ascorbic acid solution (72 g/L)**, in accordance with **46.1.1 a**) **2**).

3) **Ammonium molybdate solution**, in accordance with **46.1.1 a**) **3**). Ammonium amidosulfate need not be added.

4) **Ammonium molybdate-ascorbic acid mixed solution**, in accordance with **46.1.1 a**) **4**).

5) **Phosphorus reference solution (P 50 ∝g/ml)** Heat potassium dihydrogen phosphate (for pH reference solution) specified in **JIS K 9007** at 105 °C ± 2 °C for about 2 h, and allow to cool in a desiccator. Dissolve 0.220 g of this in a small volume of water, transfer into a 1 000-ml volumetric flask, and add water to the mark. Store in a dark place at 0 °C to 10 °C.

6) **Phosphorus reference solution (P 5 ∝g/ml)** Place 10 ml of phosphorus reference solution (P 50 ∝g/ml) in a 100-ml volumetric flask, and add water to the mark. Prepare immediately before use.

7) **Phosphorus reference solution (P 0.5 ∝g/ml)** Place 10 ml of phosphorus reference solution (P 5 ∝g/ml) in a 100-ml volumetric flask, and add water to the mark. Prepare immediately before use.

b) **Apparatus** The following apparatus shall be used.

1) **Photometer** Spectrophotometer or photoelectric photometer

c) **Procedure** The test procedure shall be as follows. If the decomposition described in NOTE 9 has been performed, carry out the determination according to NOTE 10.

1.1) Take a 25 ml aliquot of the supernatant solution obtained after decomposition as described in **46.3.1.1 c**) in a 25-ml measuring cylinder (with a stopper).

1.2) When turbidity is found in the supernatant solution, filtrate it through a Class 5 C filter paper or glass-fiber filter paper with 1 ∝m or smaller pore diameter, and use the filtrate after discarding the initial portion of 5 ml to 10 ml.

1.3) When precipitate of metal hydroxides is observed in the sample solution after decomposition, add sulfuric acid (1+35) [or if necessary, sodium hydroxide solution (40 g/L)] until the precipitate has been dissolved. Record the volume (ml) of both solutions added in this procedure, and use the value for correction in the calculation of total phosphorus concentration.

If the solution is turbid after dissolution of the precipitate, filter the solution.

2) Carry out the procedures in **2**) and **3**) of **46.1.1 c**), and measure the absorbance (2). When the concentration of total phosphorus in the solution is less than 0.1 mg/L, employ an absorption cell with an optical path length of 50 mm.

3) Take a 25 ml aliquot of the solution in the decomposing bottle obtained according to **46.3.1.1 c**) **5**), carry out the operation in **2**) to measure its absorbance, and correct the measured absorbance of the sample.

4) Determine the amount of phosphorus in the 25 ml aliquot of the solution taken in **1.1**) by reference to the working curve, and calculate the concentration of total phosphorus (P mg/L) in the sample according to either of the following formulae:

$$P = a \times \frac{60+b}{25} \times \frac{1\,000}{50} \qquad P = a \times \frac{60}{25} \times \frac{1\,000}{50} \times \frac{100}{v}$$

where, P : concentration of total phosphorus (P mg/L)

a : mass of total phosphorus in 25 ml of the solution taken in **1.1**) (mg)

b : sulfuric acid and sodium hydroxide solution required for neutralization in **46.3.1.1 c**) **1.2**) (ml)

v : volume of sample taken in a 100-ml volumetric flask in **1.1**) or **1.3**) of **46.3.1.1 c**) (ml)

Note (2) When the solution after decomposition contains arsenic (V), treat the solution according to NOTE 1 and reduce arsenic (V) to arsenic (III) by the following: Add 3 ml of sulfuric acid (1 mol/L) (prepared as described in NOTE 1) and 1.5 ml of sodium thiosulfate solution (7.65 g/L) (prepared as described in NOTE 1) to the solution after decomposition, and leave to stand for 5 min to 10 min until arsenic (V) is reduced to arsenic (III). Neutralize this solution according to the procedure in **46.1.1 c**) **1.2**). If this has been performed, note the total volume (ml) of sulfuric acid (1 mol/L), sodium thiosulfate solution (7.65 g/L) and sodium hydroxide solution (40 g/L) used in this operation, and use the value for correction in the calculation of total phosphorus.

d) **Working curve** The working curve shall be prepared as follows.

1) Place a series of aliquots of phosphorus reference solution (P 0.5 ∝g/ml) or phosphorus reference solution (P 5 ∝g/ml) so that the amount of phosphorus contained is 1.25 ∝g to 25 ∝g in as many 25-ml measuring cylinders (with stoppers), and add water to each to make 25 ml. Carry out the procedures in **2**) and **3**) of **46.1.1 c**), and measure the absorbance.

2) Separately, for a blank test, place 25 ml of water in a 25-ml measuring cylinder (with a stopper), carry out the procedures in **2**) and **3**) of **46.1.1 c**) to measure its absorbance, and correct the absorbance measured in **1**).

3) Plot the relation curve between the amount of phosphorus (P) in the 25-ml aliquot of sample and the absorbance.

NOTE 10 If the decomposition described in NOTE 9 has been performed, analyze phosphate ion in the decomposed solution according to the following and determine the total phosphorus concentration in the sample.

1.1) Transfer a 10 ml aliquot of the supernatant solution of the decomposed solution to a 10-ml measuring cylinder (with a stopper).

1.2) If turbid, centrifuge the supernatant solution.

1.3) When precipitate of metal hydroxides is observed in the sample solution after decomposition, add sulfuric acid (1+35) [or if necessary, sodium hydroxide solution (40 g/L)] according to **46.3.1.2 c**) **1.3**) until the precipitate has been dissolved. Record the volume (ml) of both solutions added in this procedure, and use the value for correction in the calculation of total phosphorus concentration. If the solution is turbid after dissolution of the precipitate, centrifuge the solution.

2) Carry out the procedures in **2**) and **3**) of **46.1.1 c**) to measure the absorbance using 0.8 ml of ammonium molybdate-ascorbic acid mixed solution.

When the concentration of total phosphorus in the sample is less than 0.1 mg/L, employ an absorption cell with an optical path length of 50 mm.

3) Transfer 10 ml of the decomposed solution in the pressure-resistant heat-resistant screw-top test tube obtained after decomposition according to **5**) of NOTE 9 to a 10-ml measuring cylinder (with stopper), perform the procedure in **2**) to measure the absorbance and correct the measured absorbance of the sample.

4) Determine the amount of phosphorus in the 10-ml aliquot of supernatant solution by referring to the working curve, and calculate the concentration of total phosphorus (P mg/L) in the sample according to either of the following formulae:

$$P = a \times \frac{(12+b)}{10} \times \frac{1\,000}{10} \qquad P = a \times \frac{12}{10} \times \frac{1\,000}{10} \times \frac{20}{v}$$

where,
P : concentration of total phosphorus (P mg/L)
a : mass of total phosphorus in 10-ml aliquot of the solution taken in **1.1**) of NOTE 10 (mg)
b : sulfuric acid and sodium hydroxide solution required for neutralization in **1.2**) of NOTE 9 (ml)
v : volume of sample taken in **1.1**) or **1.3**) of NOTE 9 (ml)

NOTE 11 In the case of a sample whose phosphorus concentration is insufficient for an accurate determination, carry out either the concentration by heating described in the following or the solvent extraction described in **46.3.1.3**.

Place 100 ml to 250 ml of sample in a 200-ml to 500-ml beaker, add one or two drops of sulfuric acid (2+1) (prepared in accordance with **JIS K 8951**), and heat on a hot plate to concentrate to a volume of 50 ml or less. Neutralize this solution with sodium hydroxide solution (40 g/L), transfer into a decomposing bottle (previously marked at 50 ml level), add water to make 50 ml, and carry out the procedures in **2**) to **5**) of **46.3.1.1 c**), and **46.3.1.2 c**).

46.3.1.3 Determination by solvent extraction method By extracting the molybdenum blue, which has been colored according to **46.3.1.2 c**) after decomposition in **46.3.1.1 c**), with 2,6-dimethyl-4-heptanone [diisobutylketone (DIBK)], the concentration of trace of total phosphorus can be determined.

Determination range: P 0.25 ∝g to 6.25 ∝g, Repeatability: 2 % to 10 %

a) **Reagents** The following reagents shall be used.

1) **Water**, as specified in **46.3.1.1 a**) **1**).

2) **Ascorbic acid solution (72 g/L)**, in accordance with **46.1.1 a**) **2**).

3) **Ammonium molybdate solution**, in accordance with **46.1.1 a**) **3**).

4) **Ammonium molybdate-ascorbic acid mixed solution**, in accordance with **46.1.1 a**) **4**).

5) **Phosphorus reference solution (P 0.5 ∝g/ml)**, in accordance with **46.3.1.2 a**) **7**).

6) **2,6-Dimethyl-4-heptanone [diisobutylketone (DIBK)]**

b) **Implement and apparatus** The following implement and apparatus shall be used.

1) **Photometer** Spectrophotometer or photoelectric photometer

2) **Separatory funnel**, of 100-ml capacity.

c) **Procedure** The test procedure shall be as follows.

1) After the procedures in **1**) to **4**) of **46.3.1.1 c**), transfer the solution in a decomposing bottle into a 100-ml separatory funnel, wash the decomposing bottle with 10 ml of water, and join the washings to the funnel.

2) To this, add 5.5 ml of ammonium molybdate-ascorbic acid mixed solution, and allow it to stand at 20 °C to 40 °C for about 15 min.

3) Add 5 ml of 2,6-dimethyl-4-heptanone to the separatory funnel, and shake to mix for about 5 min.

4) After standing, discard the water layer, transfer a part of the 2,6-dimethyl-4-heptanone layer (if there is a turbidity due to water drops, etc., filtrate it swiftly through a dry filter paper) into an absorption cell, and measure its absorbance near the wavelength of 640 nm.

5) After the procedures in **46.3.1.1 c**) **5**), transfer the solution in the decomposing bottle into a 100-ml separatory funnel, wash the decomposing bottle with 10 ml of water, and join the washings to the separatory funnel. Carry out the procedures in **2**) to **4**) to measure the absorbance, and correct the measured absorbance of the sample. Determine the amount of total phosphorus by reference to the working curve, and calculate the concentration of total phosphorus (P mg/L) in the sample according to the following formula:

$$P = a \times \frac{1\,000}{V}$$

where, P : concentration of total phosphorus (P mg/L)
a : mass of total phosphorus measured (mg)
V : volume of sample (ml)

d) **Working curve** The working curve shall be prepared as follows.

1) Place a series of aliquots in stepwise increment ranging from 0.5 ml to 12.5 ml of the phosphorus reference solution (P 0.5 ∝g/ml) in as many 100-ml separatory funnels (previously marked at 70 ml level), add water to each of these solutions to make 70 ml, and measure the absorbance according to the procedure in **2**) to **4**) of **c**).

2) For a blank test, take 70 ml of water in a 100-ml separatory funnel, carry out the same procedures to measure its absorbance, and correct the measured absorbance of each phosphorus reference solution (P ∝g/ml), and plot the relation curve between the amount of phosphorus (P) taken and the absorbance.

46.3.2 Nitric acid-perchloric acid decomposition method After addition of nitric acid, the sample is heated to concentrate, and with addition of nitric acid and perchloric acid, heated again to decompose organic matter, etc. Phosphate ion in this solution is determined and the concentration of total phosphorus calculated. This method is applicable to samples containing a large amount of organic matters or samples containing hardly decomposable organic phosphorus compounds.

46.3.2.1 Decomposition method

a) **Reagents** The following reagents shall be used.

1) **Water**, as specified in **46.3.1.1 a**) **1**).

2) **Nitric acid**, specified in **JIS K 8541**.

3) **Perchloric acid**, specified in **JIS K 8223**.

4) **Sodium hydroxide solution (40 g/L)**, in accordance with **21 a**) **3**).

5) **Sodium hydroxide solution (200 g/L)**, in accordance with **38.1.1.1 a**) **3**).

6) ***p*-Nitrophenol solution (1 g/L)**, in accordance with **46.1.1 a**) **7**).

b) **Procedure** The test procedure shall be as follows.

1) Place 50 ml of sample in a beaker. In the case of a sample with low concentration of total phosphorus, the volume used shall be 50 ml or more, and in the case of a sample containing a large amount of chloride ion and high concentration of total phosphorus, the volume used shall be less than 50 ml.

2) Add nitric acid to make weak acidity, and heat gently on a hot plate to concentrate to a volume of 15 ml to 20 ml.

3) Add 2 ml to 5 ml of nitric acid, and heat again to concentrate to a volume of about 10 ml. Further add 2 ml of nitric acid, heat again until it concentrates to a volume of about 10 ml, and allow to cool.

4) Add gradually 5 ml of perchloric acid.

In the case of a sample containing a large amount of chloride ion, further addition shall be made so as to exceed the amount equivalent to that of the chloride ion. Continue heating on a hot plate, and when white fumes of perchloric acid start to form, cover the beaker with a watch glass and keep the perchloric acid refluxing inside of the beaker (3). When, even after this operation, organic matter is not decomposed and the color in the solution persists, repeat the procedure of adding 2 ml of nitric acid and heating the solution.

5) After allowing to cool, add about 30 ml of water. If necessary, heat to dissolve soluble salts. When insoluble material still remains after heating, filtrate the solution through a Class 5 C filter paper or a glass-fiber filter paper with 1 μm or smaller pore diameter, wash the filter paper with a small volume of water, and put together the washings and the filtrate.

6) To the above solution, add three to five drops of *p*-nitrophenol solution (1 g/L) as an indicator. Add sodium hydroxide solution (200 g/L) and then sodium hydroxide solution (40 g/L) to neutralize until a faint yellow color develops. When this operation generates precipitate of metal hydroxide, stop the addition of sodium hydroxide solution (40 g/L) just before precipitation begins. If necessary, add sulfuric acid (1+35) [prepared in accordance with **30.1.1 a**) **2**)] for adjustment.

7) Transfer the solution into a 50-ml volumetric flask, and add water to the mark.

8) For a blank test, place the same volume of water as the sample collected in **1**) in a beaker, and carry out the procedures in **2**) to **7**).

Note (3) For some types of samples, the thermal decomposition using perchloric acid can involve the risk of explosion, and the following precautions should be taken.

- Thoroughly decompose easily oxidizable organic matter according to the procedures in **2**) and **3**) before adding perchloric acid.
- Ensure that the concentrated solution has been allowed to cool before adding perchloric acid.
- Ensure that thermal decomposition is performed under coexistence of perchloric acid and nitric acid.
- Do not dry up the concentrated liquid.

46.3.2.2 Determination method The phosphate ion in the decomposed solution obtained according to **46.3.2.1** is determined by the molybdenum blue absorptiometry, and total phosphorus concentration is calculated.

Determination of phosphate ion may be performed according to **46.1.4**. For samples with low concentration of phosphorus ion, carry out the procedure in **46.3.1.3** and calculate the concentration of total phosphorus concentration.

Determination range: P 1.25 μg to 25 μg, Repeatability: 2 % to 10 %

a) **Reagents** The following reagents shall be used.

1) **Water**, as specified in **46.3.1.1 a**) **1**).

2) **Ascorbic acid solution (72 g/L)**, in accordance with **46.1.1 a**) **2**).

3) **Ammonium molybdate solution**, in accordance with **46.1.1 a**) **3**).

4) **Ammonium molybdate-ascorbic acid mixed solution**, in accordance with **46.1.1 a**) **4**).

5) **Phosphorus reference solution (P 5 ∝g/ml)**, in accordance with **46.3.1.2 a**) **6**).

6) **Phosphorus reference solution (P 0.5 ∝g/ml)**, in accordance with **46.3.1.2 a**) **7**).

b) **Apparatus** The following apparatus shall be used.

1) **Photometer** Spectrophotometer or photoelectric photometer

c) **Procedure** The test procedure shall be as follows.

1) Place 25 ml of the solution in **46.3.2.1 b**) **7**) in a 25-ml measuring cylinder (with stopper). If 25 ml of the solution contains 25 ∝g or more of total phosphorus, use any suitable volume (*b* ml) that contains less than 25 ∝g of phosphorus, and add water to make 25 ml.

2) Carry out the procedures in **2**) and **3**) of **46.1.1 c**), and measure the absorbance.

3) For a blank test, place 25 ml of the solution in **46.3.2.1 b**) **8**) in a 25-ml measuring cylinder (with a stopper), perform the procedures in **1**) and **2**), and correct the measured absorbance of the sample.

4) Determine the amount of phosphorus in the aliquot taken in **1**) by referring to the working curve, and calculate the concentration of total phosphorus (P mg/L) in the sample according to the following formula:

$$P = a \times \frac{50}{b} \times \frac{1\,000}{v}$$

where, P : concentration of total phosphorus (P mg/L)

a : mass of total phosphorus in 25 ml or b ml aliquot taken in **1**) (mg)

b : volume of aliquot taken in **1**) (ml)

v : volume of sample (ml)

d) **Working curve** Prepare the working curve according to the same procedure as described in **46.3.1.2 d**).

46.3.3 Nitric acid-sulfuric acid decomposition method After addition of nitric acid, the sample is heated to concentrate, and after addition of nitric acid and sulfuric acid, heated again to decompose organic matter and so on. Phosphate ion in this solution is determined, and the concentration of total phosphorus calculated. This method is applicable to samples containing a large amount of organic matters or samples containing hardly decomposable organic phosphorus compounds.

46.3.3.1 Decomposition method

a) **Reagents** The following reagents shall be used.

1) **Water**, as specified in **46.3.1.1 a**) **1**).

2) **Nitric acid**, as specified in **46.3.2.1 a**) **2**).

3) **Sulfuric acid (1+1)**, in accordance with **5.4 a**) **2**).

4) **Sodium hydroxide solution (40 g/L)**, in accordance with **21 a**) **3**).

5) **Sodium hydroxide solution (200 g/L)**, in accordance with **38.1.1.1 a**) **3**).

6) ***p*-Nitrophenol solution (1 g/L)**, in accordance with **46.1.1 a**) **7**).

b) **Procedure** The test procedure shall be as follows.

1) Carry out the procedures in **1**) and **2**) of **46.3.2.1 b**).

2) To the solution treated according to **1**), add 2 ml of sulfuric acid (1+1) and 2 ml to 5 ml of nitric acid.

In the case of a sample containing a large amount of chloride ion, further volume of sulfuric acid (1+1) shall be added so as to exceed the amount equivalent to that of the chloride ion. Heat it to concentrate until white fumes of sulfuric acid start to form, cover the beaker with a watch glass, and heat further for a short while until more intense fumes of sulfuric acid evolve, and allow to cool.

3) Add 5 ml of nitric acid to the above solution, and heat again until white fumes of sulfuric acid start to form. When, even after this operation, organic matter is not decomposed and the color in the solution persists, repeat the procedure of adding 2 ml of nitric acid and heating the solution.

4) After allowing to cool, add about 30 ml of water, and boil gently for about 10 min. When insoluble material still remains after heating, filtrate the solution through a Class 5 C filter paper or a glass-fiber filter paper with 1 ∝m or smaller pore diameter, wash the filter paper with a small volume of water, and put together the washings and the filtrate.

5) Carry out the procedures in **6**) and **7**) of **46.3.2.1 b**).

6) For a blank test, place the same volume of water as the sample collected **1**) in a beaker, and carry out the procedures in **2**) to **5**).

46.3.3.2 Determination method The phosphate ion in the decomposed solution obtained according to **46.3.3.1** is determined by the molybdenum blue absorptiometry, and the total phosphorus concentration calculated.

Determination of phosphate ion may be performed according to **46.1.4**. For samples with low concentration of phosphorus ion, carry out the procedure in **46.3.1.3** and calculate the concentration of total phosphorus concentration.

Determination range: P 1.25 ∝g to 25 ∝g, Repeatability: 2 % to 10 %

a) **Reagents** The following reagents shall be used.

1) **Water**, as specified in **46.3.1.1 a**) **1**).

2) **Ascorbic acid solution (72 g/L)**, in accordance with **46.1.1 a**) **2**).

3) **Ammonium molybdate solution**, in accordance with **46.1.1 a**) **3**).

4) **Ammonium molybdate-ascorbic acid mixed solution**, in accordance with **46.1.1 a**) **4**).

5) ***p*-Nitrophenol solution (1 g/L)**, in accordance with **46.1.1 a**) **7**).

6) **Phosphorus reference solution (P 5 ∝g/ml)**, in accordance with **46.3.1.2 a**) **6**).

7) **Phosphorus reference solution (P 0.5 ∝g/ml)**, in accordance with **46.3.1.2 a**) **7**).

b) **Apparatus** The following apparatus shall be used.

1) **Photometer** Spectrophotometer or photoelectric photometer

c) **Procedure** The test procedure shall be as follows.

1) Place 25 ml of the solution in **46.3.3.1 b**) **5**) in a 25-ml measuring cylinder (with stopper). If 25 ml of the solution contains 25 ∝g or more of total phosphorus, use any suitable volume (*b* ml) that contains less than 25 ∝g of phosphorus, and add water to make 25 ml.

2) Carry out the procedures in **2**) and **3**) of **46.1.1 c**), and measure the absorbance.

3) For a blank test, place 25 ml of the solution in **46.3.3.1 b**) **6**) in a 25-ml measuring cylinder (with a stopper), perform the procedure in **1**) to measure the absorbance, and correct the measured absorbance of the sample.

4) Determine the amount of phosphorus by reference to the working curve, and calculate the concentration of total phosphorus (P mg/L) in the sample according to the formula in **46.3.2.2 c**) **4**).

d) **Working curve** Prepare the working curve according to the same procedure as described in **46.3.1.2 d**).

46.3.4 Flow analysis The series of operations of hydrolysis or oxidization decomposition to turn phosphorus compounds etc. in the sample to phosphate ion and determination of phosphate ion by molybdenum blue absorptiometry by the same principle as in **46.3.1** is performed by the flow analysis method specified in clause **7** of **JIS K 0170-4** and the concentration of total phosphorus is determined.

Determination range: P 0.01 mg/L to 10 mg/L, Repeatability: 10 % max.

For details of the procedures and other matters of this method, refer to clause **7** of **JIS K 0170-4** excluding the methods given in **7.3.2** and **7.3.4** of the said standard.

Clause 47

Replace Note (9) by the following:

Note (9) When using the apparatus equipped with the device of high order spectral line, the high order spectral line may be used. As long as the sensitivity, precision and spectral interference are at a permissible level, other wavelength than this may be used.

Clause 47

Replace NOTE 5 by the following:

NOTE 5 With the apparatus capable of measuring simultaneously two or more spectrum lines with different wavelengths, an internal standard method can be applied. The procedure is as follows.

1) Place a suitable volume of sample in a 100-ml volumetric flask, add 10 ml of yttrium solution (Y 50 ∝g/ml) and add water to the mark.

2) Carry out the procedure of **c**) **1**) using this solution to measure the emission intensities of boron [249.773 (I) nm] and yttrium [371.029 (II) nm] and obtain the ratio between the emission intensities of boron and yttrium (*).

3) For the blank test, carry out the procedures in **1**) and **2**) using water in place of the sample to obtain the ratio between the emission intensities of boron and yttrium and correct the ratio obtained in **2**).

4) Determine the amount of boron by referring to the working curve and calculate the concentration of boron (B ∝g/L) in sample.

5) **Working curve** Place a series of aliquots in stepwise increment ranging from 0.1 ml to 40 ml of the boron reference solution (B 20 ∝g/ml) in as many 100-ml volumetric flasks, add 10 ml of yttrium solution (Y 50 ∝g/ml) and add water to each of them to the mark. Carry out the procedure in **2**) using these solutions, measure the emission intensities of boron and yttrium and obtain the ratio between the emission intensities of boron and yttrium. In addition, for a blank test, carry out the same operation using water in place of boron reference solution to obtain similarly the ratio between the emission intensities of boron and yttrium, and correct the ratio obtained by using boron reference solutions. Prepare the relation curve between the amount of boron and the ratio between the emission intensities of boron and yttrium.

6) **Preparation of yttrium solution (Y 50 ∝g/ml)** Take 0.318 g of yttrium (III) oxide, add 5 ml of highly purified nitric acid specified in **JIS K 9901**, heat to dissolve and then further boil to expel nitrogen oxide. After allowing to cool, transfer to a 250-ml volumetric flask and add water to the mark. Transfer 10 ml of this solution into a 200-ml volumetric flask and add water to the mark.

Note (*) See Notes to table 52.1. In the table, I and II indicate neutral line and ion line, respectively. Ion line is used for boron analysis because no suitable neutral line that can be used as an internal standard line is available.

Clause 47

Replace NOTE 6 by the following:

NOTE 6 Presence of boron in the solution introduced into the emission part causes larger memory effect than presence of other element. To ensure against this, the influence of the previous sample should be eliminated by spraying acid solution or water for a sufficient amount of time before spraying of the next solution. Further, since introduction of acid solution may cause boron memory to appear even after sufficient spraying of water, the signals of the next measurement should be checked for residual memory effect.