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**Methods for chemical analysis of
solder**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Welding Engineering Society (JWES)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS Z 3910:2008** is replaced with this Standard.

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Methods for chemical analysis of solder

1 Scope

This Japanese Industrial Standard specifies the methods for determination of alloy components (tin, silver, antimony, bismuth, copper, indium, zinc, nickel, germanium and lead) and of impurities (lead, silver, antimony, bismuth, copper, indium, zinc, nickel, iron, aluminium, arsenic, cadmium and gold) in the solder.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS H 0301 *General rules for sampling, sample preparation and analytical testing of non-ferrous metals*

JIS H 0321 *General rules for inspection of non-ferrous metal materials*

JIS H 2105 *Pig lead*

JIS K 0050 *General rules for chemical analysis*

JIS K 0116 *General rules for atomic emission spectrometry*

JIS K 0119 *General rules for X-ray fluorescence analysis*

JIS K 0121 *General rules for atomic absorption spectrometry*

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 1105 *Argon*

JIS Z 2611 *General rules for photoelectric emission spectrochemical analysis of metal materials*

JIS Z 3001-1 *Welding and allied processes—Vocabulary—Part 1: General*

JIS Z 3001-3 *Welding and allied processes—Vocabulary—Part 3: Soldering and brazing*

JIS Z 3282 *Soft solders—Chemical compositions and forms*

JIS Z 8401 *Guide to the rounding of numbers*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS K 0211**, **JIS Z 3001-1** and **JIS Z 3001-3** apply.

4 General

4.1 General matters of chemical analysis

General matters of chemical analysis given in **JIS K 0050**, **JIS K 0116**, **JIS K 0119**, **JIS K 0121**, **JIS Z 2611**, and the following apply.

- a) For the water to be used for analytical procedure, the water of at least equal quality to A1 specified in Annex D of **JIS K 0050** shall be used.
- b) For reagents, titrant and standard solution of known concentration on the market may be used.

4.2 Precaution on safety

In chemical analysis, there are matters requiring sufficient precautions, such as the use and disposal of hazardous or dangerous chemicals, interference due to X-ray in X-ray fluorescence analysis, handling of high pressure gas in atomic absorption spectrometry, ICP atomic emission spectrometry and spark discharge atomic emission spectrometric analysis, ignition and extinction of flame. Since the acetylene gas to be used for atomic absorption spectrometry could produce an explosive acetylide by reacting with silver, etc., care shall be taken for prevention of hazards and for protection of the environment by taking such measures as washing a burner head with water each time of use.

5 Sampling, handling and weighing of analysis sample

5.1 Sampling

The sampling and preparation of sample shall be in accordance with **JIS H 0301** and **JIS H 0321**.

5.2 Handling of sample

The handling of sample shall be as follows.

- a) Store an analysis sample in a suitable glass vessel with a cover, and seal the vessel hermetically in order to prevent the contamination by foreign matters, etc.
- b) When oil, etc. is found on the surface of analysis sample, wash with ethanol or diethyl ether preliminarily to remove it.

5.3 Weighing of sample

The weighing of sample shall be as follows.

- a) Before weighing, make sure to agitate the analysis sample sufficiently so that it can represent the average composition, and to prevent contamination by foreign matters.
- b) Weigh the sample of the quantity specified in each analysis method to the nearest 0.1 mg, using a chemical balance or electronic scale.
- c) The amount of analysis sample depends on each analysis method.

6 Expression of analysis values

6.1 Analysis cycles

Repeat the parallel determination on the same sample two or more times.

6.2 Indication of analysis values

The analysis values shall be indicated as follows.

- a) Express the analysis values in percentage (mass fraction).
- b) Calculate the analysis values of alloy components to the second decimal place, average the two or more values, and round off to the first decimal place in accordance with Rule A of **JIS Z 8401**. For nickel, calculate the analysis values to the third decimal place, average the two or more values, and round off to the second decimal place in accordance with Rule A of **JIS Z 8401**. For germanium, calculate the analysis values to the fourth decimal place, average the two or more values, and round off to the third decimal place in accordance with Rule A of **JIS Z 8401**.
- c) For lead, silver, antimony, copper, bismuth, iron, arsenic, indium, gold and nickel, calculate the analysis values of impurities to the third decimal place, average the two or more values, and round off to the second decimal place in accordance with Rule A of **JIS Z 8401**. For zinc, aluminium and cadmium, calculate the values of impurities to the fourth decimal place, average the two or more values, and round off to the third decimal place in accordance with Rule A of **JIS Z 8401**.
- d) Decimal places of analysis values for alloy components and impurities shall be as given in Table 1.

Table 1 Decimal places of components

Designation of components	Alloy components	Impurities
Pb	1	2
Sb		
Bi		
Cu		
In		
Ag		
Zn		3
Sn		—
Ni	2	2
Ge	3	—
Au	—	2
As		
Fe		
Al		
Cd		3

6.3 Blank test

For analysis, perform the blank test to correct the measured value. However, it does not apply to the wavelength dispersive X-ray fluorescence analysis and spark discharge atomic emission spectrometric analysis.

7 Division of analysis methods

7.1 Lead (or nickel) reduction potassium iodate titration method

Lead (or nickel) reduction potassium iodate titration method applies to the determination of tin as alloy component.

7.2 Potassium thiocyanate titration method

Potassium thiocyanate titration method applies to the determination of silver as alloy component.

7.3 Potassium permanganate titration method

Potassium permanganate titration method applies to the determination of Type Sn95Sb5 antimony specified in **JIS Z 3282** and antimony as impurity in the lead-containing solder specified in Table 2. It is not applicable to Types Sn63Pb37E, Sn60Pb40E and Pb50Sn50E specified in **JIS Z 3282**.

7.4 Atomic absorption spectrometry

The atomic absorption spectrometry applies to the determination of impurities (except for arsenic) of lead-containing solder specified in Table 2, and determination of impurities (except for arsenic) of lead-free solder specified in Table 3 and some alloy components (Table 3).

7.5 Wavelength dispersive X-ray fluorescence analysis

The wavelength dispersive X-ray fluorescence analysis applies to the determination of alloy components of lead-containing solder specified in Table 2, determination of alloy components of lead-free solder specified in Table 3, and determination of lead as an impurity.

7.6 ICP atomic emission spectrometry

The ICP atomic emission spectrometry applies to the determination of impurities of lead-containing solder specified in Table 2 and some alloy components (Table 2), and determination of impurities of lead-free solder specified in Table 3 and some alloy components (Table 3).

7.7 Spark discharge atomic emission spectrometric analysis

The spark discharge atomic emission spectrometric analysis applies to the determination of impurities and alloy components of lead-containing solder specified in Table 2, determination of impurities and alloy components of lead-free solder specified in Table 3.

Table 2 Types of lead-containing solders and applicable analysis method

Type		Applicable analysis method													
Designation 1	Designation 2	Sn	Pb	Sb	Bi	Cd	Cu	Au	In	Ag	Al	As	Fe	Ni	Zn
Sn95Pb5	H95A	—	3,4,5	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn63Pb37	H63A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn63Pb37E	H63E	1,3,5	—	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn60Pb40	H60A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn60Pb40E	H60E	1,3,5	—	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb50Sn50	H50A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb50Sn50E	H50E	1,3,5	—	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb55Sn45	H45A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb60Sn40	H40A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb65Sn35	H35A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb70Sn30	H30A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb80Sn20	H20A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb90Sn10	H10A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb95Sn5	H5A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn57Pb40Bi3	H57Bi3A	1,3,5	—	1,2,4,5	3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn46Pb46Bi8	H46Bi8A	1,3,5	—	1,2,4,5	3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn43Pb43Bi14	H43Bi14 A	1,3,5	—	1,2,4,5	3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Sn62Pb36Ag2	H62Ag2A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
Pb97.5Ag1.5Sn1	H1Ag1.5A	1,3,5	—	1,2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5
For solders other than specified in JIS Z 3282 , it is permitted to select the designation of solder having the most similar composition, and to use the analysis method of that solder.															
NOTE : 1: titration method Sn: lead (or nickel) reduction potassium iodate titration method Ag: potassium thiocyanate titration method Sb: potassium permanganate titration method 2: atomic absorption spectrometry 3: wavelength dispersive X-ray fluorescence analysis 4: ICP atomic emission spectrometry 5: spark discharge atomic emission spectrometric analysis															

Table 3 Types of lead-free solders and applicable analysis method

Class		Applicable analysis method														
Designation 1	Designation 2	Sn	Pb	Sb	Bi	Cu	Au	In	Ag	Al	As	Cd	Fe	Ni	Zn	Ge
Sn95Sb5	S50	—	2,3,4,5	1,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn97Cu3	C30	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn99.3Cu0.7	C7	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn99.25Cu0.7Ni0.05	C7Ni	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	—
Sn95Ag5	A50	—	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Su92Cu6Ag2	C60A20	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn95Cu4Ag1	C40A10	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn99Cu0.7Ag0.3	C7A3	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn97Ag3	A30	—	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn96.5Ag3.5	A35	—	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn96.3Ag3.7	A37	—	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn98.3Ag1Cu0.7	A10C7	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn96.5Ag3Cu0.5	A30C5	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn95.5Ag4Cu0.5	A40C5	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn95.92Ag3.5Cu0.5Ni0.07Ge0.01	A35C5NiGe	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,3,4,5
Sn95.8Ag3.5Cu0.7	A35C7	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn95.5Ag3.8Cu0.7	A38C7	—	2,3,4,5	2,4,5	2,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn96Ag2.5Bi1Cu0.5	A25B10C5	—	2,3,4,5	2,4,5	3,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn96.5Bi1.6Ag1Cu0.7In0.2	B16A10C7N2	—	2,3,4,5	2,4,5	3,4,5	2,3,4,5	2,4,5	2,3,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn96.3Bi2Ag1Cu0.7	B20A10C7	—	2,3,4,5	2,4,5	3,4,5	2,3,4,5	2,4,5	2,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn92In4Ag3.5Bi0.5	N40A35B5	—	2,3,4,5	2,4,5	3,4,5	2,4,5	2,4,5	2,3,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn88In8Ag3.5Bi0.5	N80A35B5	—	2,3,4,5	2,4,5	3,4,5	2,4,5	2,4,5	2,3,4,5	1,3,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
Sn91Zn9	Z90	—	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	3,4,5	—
Sn89Zn8Bi3	Z80B30	—	2,3,4,5	2,4,5	3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	3,4,5	—
Bi58Sn42	B580	1,3,5	2,3,4,5	2,4,5	—	2,4,5	2,4,5	2,4,5	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—
In52Sn48	N520	1,3,5	2,3,4,5	2,4,5	2,4,5	2,4,5	2,4,5	—	2,4,5	2,4,5	4,5	2,4,5	2,4,5	2,4,5	2,4,5	—

Table 3 (concluded)

For solders other than specified in **JIS Z 3282**, it is permitted to select the designation of solder having the most similar composition, and to use the analysis method of that solder.

NOTE: 1: titration method

Sn: lead (or nickel) reduction potassium iodate titration method

Ag: potassium thiocyanate titration method

Sb: potassium permanganate titration method

2: atomic absorption spectrometry

3: wavelength dispersive X-ray fluorescence analysis

4: ICP atomic emission spectrometry

5: spark discharge atomic emission spectrometric analysis

8 Lead (or nickel) reduction potassium iodate titration method

8.1 Summary

Decompose a sample with sulfuric acid, add water, hydrochloric acid, antimony chloride (III) and lead or nickel, and heat to reduce tin (IV) to tin (II). Titrate with potassium iodate solution by using starch as an indicator.

8.2 Reagents

The reagents shall be as follows.

8.2.1 Hydrochloric acid

8.2.2 Sulfuric acid

8.2.3 Lead

A small piece of pig lead corresponding to special class specified in **JIS H 2105**.

8.2.4 Nickel cylinder or nickel wire

Nickel cylinder or nickel wire, which is reusable, with minimum purity 99.8 % (mass fraction), generally of the shape and dimensions shown in a) or b) of Figure 1.

Unit: mm

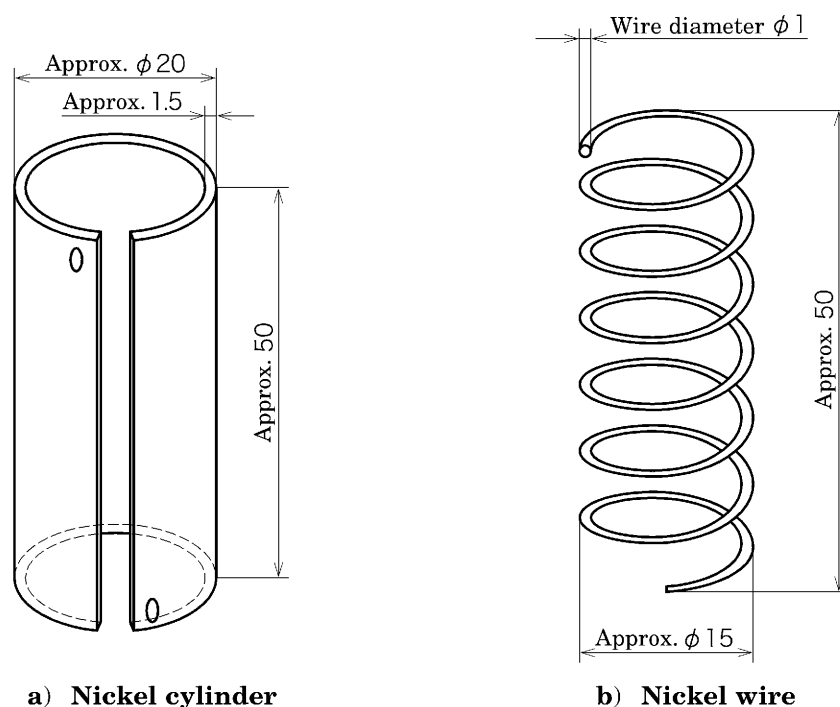


Figure 1 Nickel cylinder and nickel wire

8.2.5 Carbon dioxide

Use carbon dioxide with minimum purity 99.9 % (volume fraction). When carbon dioxide contains oxygen, remove it as follows: dissolve 45 g of copper chloride (I) in 300 ml of hydrochloric acid (1+1), add small amount of metallic copper scraps, seal hermetically, leave to stand for at least 24 h, and pass carbon dioxide through that solution.

8.2.6 Antimony chloride (III) solution Add 5 g of antimony chloride (III) to 500 ml of hydrochloric acid (1+1), and heat to dissolve.

8.2.7 Sodium hydrogen carbonate solution (saturated)

8.2.8 0.017 mol/L potassium iodate solution

Dissolve 1 g of sodium hydroxide and 10 g of potassium iodide in 200 ml of water, and add 3.567 g of potassium iodate to dissolve. Transfer the solution into a 1 000 ml volumetric flask with water, and dilute to the mark with water. One millilitre of this solution is equivalent to approximately 0.006 g of tin. Conduct the standardization according to the following procedure.

a) **Preparation of tin standard solution (Sn: 4 g/L)** Preparation of tin standard solution (Sn: 4 g/L) shall be in accordance with either of the following.

- 1) Weigh out 1.0 g of tin [minimum purity 99.99 % (mass fraction)] to the nearest 1 mg accurately, and transfer it into a tall beaker (300 ml). Add 20 ml of sulfuric acid, cover with a watch glass, and heat to decompose. Heat to vaporize sulfur adhering to the watch glass and the inner wall of beaker. Allow to cool, wash the bottom face of watch glass and the inner wall of beaker with water and hydrochloric acid (1+1), and remove the watch glass. Add 50 ml of hydrochloric acid (1+1) to dissolve salts. Cool it to room temperature, transfer the solution into a 250 ml volumetric flask with hydrochloric acid (1+1), and dilute to the mark with hydrochloric acid (1+1).
- 2) Weigh out 1.0 g of tin [minimum purity 99.99 % (mass fraction)] to the nearest 1 mg accurately, transfer it into a beaker (300 ml), and add 50 ml of hydrochloric acid. Heat to decompose on a water bath while bringing tin into contact with the platinum plate. Cool to room temperature, wash the bottom face of watch glass and the inner wall of beaker with hydrochloric acid (1+1), and remove the watch glass. Transfer the solution into a 250 ml volumetric flask by using hydrochloric acid (1+1), and dilute to the mark with hydrochloric acid (1+1).

b) **Standardization** Take accurately 25 ml of tin standard solution prepared in **1)** or **2)** of **a)**, and pour it into the Erlenmeyer flask (500 ml) of the equipment (**8.3**). Then, operate according to the procedures of **2)** to **6)** of **8.5 a)** or **2)** to **5)** of **8.5 b)**, and calculate the amount of tin corresponding to 1 ml of 0.017 mol/L potassium iodate solution according to the following formula.

$$f_1 = \frac{m_1}{V_1}$$

where, f_1 : amount of tin corresponding to 1 ml of 0.017 mol/L potassium iodate solution (g/ml)

m_1 : amount of tin weighed out in **1)** or **2)** of **a)** $\times$ 0.1

V_1 : amount of 0.017 mol/L potassium iodate solution used (ml)